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A RESOURCEFUL MULTI COMPONENT CREATION OF NOVEL TRIAZOLOPYRIMIDINES

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ABSTRACT

Synthesis and natural action of new derivatives of triazolopyrimidines **(4a-j)** was achieved from diverse acetoacetamides, novel aldehyde and triazole using heating within 40 min. with good yield. The triazolopyrimidines of the products were sustained by FTIR, PMR and mass spectral data.

Key words: Pyrimidines, Acetoacetamides, Triazole abridgment synthesis.

INTRODUCTION

The condensation of a ring of 1,2,4-triazole and another one of pyrimidine gives rise to the formation of bicyclic heterocycles known as 1,2,4-triazolopyrimidines. Four different possibilities survive for the relative direction of both rings, so four different isomeric families of compounds are defined. Among these, 1,2,4-triazolo [1,5-a] pyrimidine derivatives are thermo-dynamically more stable and, thus, the most studied ones[1], a few of them being commercially offered. Revisions surveying the synthesis, reactivity, spectroscopic characterization and crystallographic studies of 1,2,4-triazolo [1,5-c] pyrimidines[2] 1,2,4-triazolo [4,3-a] pyrimidines[3], 1,2,4-triazolo [4,3-c] pyrimidines[4] and 1,2,4 triazolopyrimidines[5] have also been published.

The studies about the coordination chemistry of triazolopyrimidines have been exclusively focused till now in the 1,5-a series. These compounds, which are structurally similar and may be regarded as mimic of isomeric purines, have displayed a rich coordination chemistry, a considerable number of new compounds with interesting structural features having been characterized[6], including simple mononuclear compounds with monodentately coordinated ligands[7,8] and di or polynuclear compounds in which either the triazolopyrimidine ligand[9] or other auxiliary ligands[10] bridge the metal atoms.

From the standpoint of biological activity, fused heteroaromatic systems are often of much greater interest than the constituent monocyclic compounds. Recently, 1,2,4-triazolo [1,5-a] pyrimidines have aroused increasing attention from the chemical and biological view points, due to their diverse

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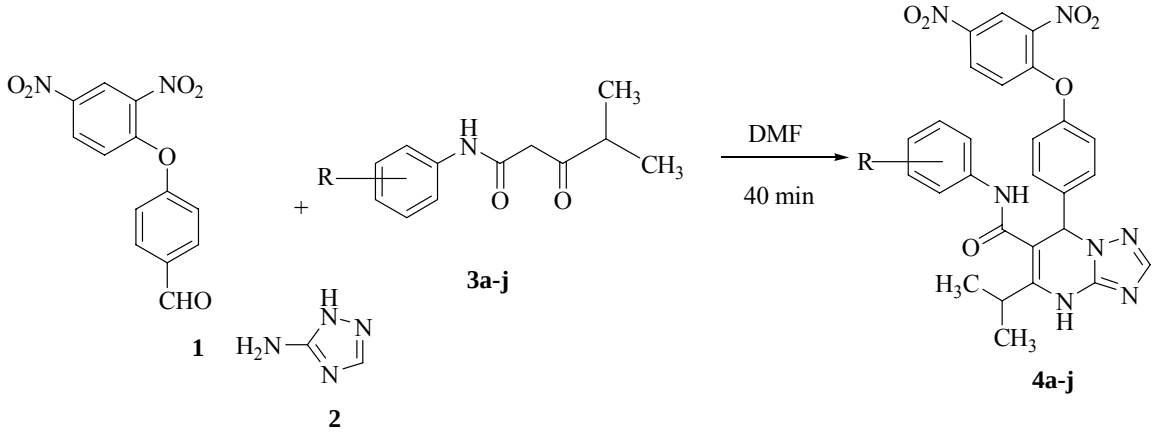
pharmacological activities, such as antitumor potency, inhibition of KDR kinase, antifungal effect and macrophage activation. They have proved to be promising anticancer agents with dual mechanisms of tubulin polymerization promotion as well as cyclin dependent kinases 2 inhibition. Some examples of published derivatives of 1,2,4-triazolo [1,5-a] pyrimidine with their biological activities.

To circumvent these problems, we have developed a new microwave assisted protocol for the synthesis of novel pyridmidines **(4a-j)** with the advantage of short reaction time, high yield and environmentally friendliness **(Scheme)**.



Scheme

EXPERIMENTAL

^{1}H NMR spectra were evidenced on Brukur spectrophotometer (400 MHz). Chemical shifts are expressed in units relative to TMS signal as internal reference. IR spectra were evidenced on FT-IR Shimadzu-FT-IR 8400 spectrophotometer on KBr pallets. Mass spectra were evidenced on GCMS QP2010 Gas Chromatograph. Thin Layer Chromatography was performed on silica gel-G using hexane: ethyl acetate and toluene: methanol solvent system.

Typical exaperimental procedure for the synthesis of triazolopyrimidines

A mixture of the 5-amino-1,2,4-triazole (2 mmol), an appropriate acetoacetaminde (2 mmol) and 4-(2,4-dinitrophenoxy)benzaldehyde (1 mmol) was refluxed in 0.5 mL of DMF for 40 min. After cooling, methanol (~20 mL) was added. The reaction mixture was allowed to stand overnight and then filtered to give the solid triazolopyrimidine products, which were crystallized from methanol.

7-(4-(2, 4-dinitrophenoxy) phenyl)-N-(3-chlorophenyl)-4, 7-dihydro-5-isopropyl-[1, 2, 4]-triazolo [1,5-a] pyrimidine-6-carboxamide

4a. m.p. 198^{o}C; white crystals; ^{1}H NMR (DMSO-d_6) δ ppm: 1.47 (s, 3H, H_a), 1.58 (s, 3H, H_b), 3.85 (m, 1H, H_c), (δ 6.26) (s, 1H, H_d), (δ 6.43-6.45) (d, 2H, $H_{ee'}$,), (δ 6.76-6.78) (d, 1H, H_f,), (δ 7.14-7.18) (t, 2H, $H_{gg'}$,), (δ 7.27-7.31) (t, 1H, H_h), (δ 7.37-7.45) (m, 3H, H_{i-k}), (δ 7.58-7.62) (dd, 2H, H_{lm}), (δ 7.71) (s, 1H, H_n), (δ 9.88) (s, 1H, H_o),(δ 10.29) (s, 1H, H_p). FT IR (cm^{-1}): 3259 (N-H stretching of secondary amine), 3013 (C-H stretching of aromatic ring), 2931 (C-H asymmetrical stretching of CH_3 group), 2886 (C-H asymmetrical stretching of CH_3 group), 1686 (C=O stretching of amide), 1613 (C=N stretching of triazole ring), 1564 (N-H deformation of pyrimidine ring), 1520 and 1478 (C=C stretching of aromatic ring), 1421 (C-H asymmetrical deformation of CH_3 group), 1410 (C-H symmetrical deformation of CH_3 group), 1330 (C-N stretching), 1274 (C-NO_2 symmetrical deformation of NO_2 group), 1247 (C-O-C stretching), 1028

(C-H in plane deformation of aromatic ring), 821 (C-H out of plane bending of 1,4-disubstituion), 730 (C-Cl stretching), Mass: m/z 548; Anal. Calcd. for $C_{27}H_{22}ClN_7O_6$: C, 55.80; H, 3.31; Cl, 6.47; N, 17.89; O, 17.52; Found: C, 55.05; H, 3.25; Cl, 6.24; N, 17.25; O, 17.03%.

7-(4-(2, 4-dinitrophenoxy) phenyl)-N-(4-fluorophenyl)-4,7-dihydro-5-isopropyl-[1, 2, 4] triazolo [1,5-a] pyrimidine-6-carboxamide

4b. m.p. 187°C; white crystals; ^{1}H NMR (DMSO-d_6) δ ppm: 1.37 (s, 3H, H_a), 1.48 (s, 3H, H_b), 3.75 (m, 1H, H_c), (δ 6.21) (s, 1H, H_d), (δ 6.60-6.70) (d, 2H, $H_{ee'}$,), (δ 6.72-6.79) (d, 1H, H_f,), (δ 7.11-7.16) (t, 2H, $H_{gg'}$), (δ 7.27-7.37) (dd'dd', 4H, $H_{hh'-ii'}$), (δ 7.49-7.54) (dd, 2H, H_{jk}), (δ 7.63) (s, 1H, H_l), (δ 9.07) (s, 1H, H_m), (δ 10.03) (s, 1H, H_n). FT IR (cm^{-1}): 3289 (N-H stretching of secondary amine), 3052 (C-H stretching of aromatic ring), 2921 (C-H asymmetrical stretching of CH_3 group), 2843 (C-H asymmetrical stretching of CH_3 group), 1667 (C=O stretching of amide), 1648 (C=N stretching of triazole ring), 1545 (N-H deformation of pyrimidine ring), 1522 and 1434 (C=C stretching of aromatic ring), 1404 (C-H asymmetrical deformation of CH_3 group), 1400 (C-H symmetrical deformation of CH_3 group), 1304 (C-N stretching), 1268 (C-NO_2 symmetrical deformation of NO_2 group), 1249 (C-O-C stretching), 1029 (C-H in plane deformation of aromatic ring), 836 (C-H out of plane bending of 1,4-disubstituion), 728 (C-Cl stretching), Mass: m/z 560; Anal. Calcd. for $C_{27}H_{12}FN_7O_6$: C, 57.96; H, 3.96; F, 3.40; N, 17.52; O, 17.16; Found: C, 57.56; H, 3.06; F, 3.00; N, 17.02; O, 17.00%.

7-(4-(2, 4-dinitrophenoxy) phenyl)-N-(4-chlorophenyl)-4, 7-dihydro-5-isopropyl-[1, 2, 4] triazolo [1,5-a] pyrimidine-6-carboxamide

4c. m.p. 188°C; white crystals; ^{1}H NMR (DMSO-d_6) δ ppm: 1.42 (s, 3H, H_a), 1.50 (s, 3H, H_b), 3.22 (m, 1H, H_c), (δ 6.22) (s, 1H, H_d), (δ 6.31-6.35) (d, 2H, $H_{ff'}$,), (δ 6.68-6.74) (d, 1H, H_g,), (δ 7.00-7.08) (t, 2H, $H_{hh'}$), (δ 7.13-7.31) (dd'dd', 4H, $H_{ii'-jj'}$), (δ 7.55-7.60) (dd, 2H, H_{kl}), (δ 7.79) (s, 1H, H_m), (δ 9.24) (s, 1H, H_n),(δ 10.00) (s, 1H, H_o). FT IR (cm^{-1}): 3159 (N-H stretching of secondary amine), 3052 (C-H stretching of aromatic ring), 2951 (C-H asymmetrical stretching of CH_3 group), 2853 (C-H asymmetrical stretching of CH_3 group), 1657 (C=O stretching of amide), 1622 (C=N stretching of triazole ring), 1505 (N-H deformation of pyrimidine ring), 1500 and 1484 (C=C stretching of aromatic ring), 1480 (C-H asymmetrical deformation of CH_3 group), 1400 (C-H symmetrical deformation of CH_3 group), 1354 (C-N stretching), 1258 (C-NO_2 symmetrical deformation of NO_2 group), 1209 (C-O-C stretching), 1009 (C-H in plane deformation of aromatic ring), 804 (C-H out of plane bending of 1,4-disubstituion), 709 (C-Cl stretching), Mass: m/z 576; Anal. Calcd. for $C_{27}H_{22}ClN_7O_6$: C, 56.30; H, 3.85; Cl, 6.16; N, 17.02; O, 16.67; Found: C, 56.10; H, 3.45; Cl, 6.06; N, 17.00; O, 16.27%.

7-(4-(2, 4-dinitrophenoxy) phenyl)-N-(4-nitrophenyl)-4, 7-dihydro-5-isopropyl-[1, 2, 4] triazolo [1,5-a] pyrimidine-6-carboxamide

4d. m.p. 181°C; white crystals; ^{1}H NMR (DMSO-d_6) δ ppm: 1.22 (s, 3H, H_a), 1.34 (s, 3H, H_b), 3.43 (m, 1H, H_c), (δ 6.30) (s, 1H, H_d), (δ 6.50-6.60) (d, 2H, $H_{ee'}$,), (δ 6.71-6.79) (d, 1H, H_f,), (δ 7.02-7.08) (t, 2H, $H_{gg'}$), (δ 7.15-7.34) (dd'dd', 4H, $H_{hh'-ii'}$), (δ 7.41-7.46) (dd, 2H, H_{jk}), (δ 7.56) (s, 1H, H_l), (δ 9.58) (s, 1H, H_m), (δ 10.02) (s, 1H, H_n). FT IR (cm^{-1}): 3210 (N-H stretching of secondary amine), 3012 (C-H stretching of aromatic ring), 2916 (C-H asymmetrical stretching of CH_3 group), 2813 (C-H asymmetrical stretching of CH_3 group), 1616 (C=O stretching of amide), 1613 (C=N stretching of triazole ring), 1515 (N-H deformation of pyrimidine ring), 1506 and 1423 (C=C stretching of aromatic ring), 1413 (C-H asymmetrical deformation of CH_3 group), 1400 (C-H symmetrical deformation of CH_3 group), 1345 (C-N stretching), 1253 (C-NO_2 symmetrical deformation of NO_2 group), 1229 (C-O-C stretching), 1028 (C-H in plane deformation of aromatic ring), 843 (C-H out of plane bending of 1,4-disubstituion), 738 (C-Cl stretching),

Mass: m/z 587; Anal. Calcd. for $C_{27}H_{22}N_8O_8$: C, 55.29; H, 3.78; N, 19.11; O, 21.82; Found: C, 55.09; H, 3.28; N, 19.01; O, 21.02%.

7-(4-(2, 4-dinitrophenoxy) phenyl)-N-(3-nitrophenyl)-4, 7-dihydro-5-isopropyl-[1, 2, 4] triazolo [1,5-a] pyrimidine-6-carboxamide

4e. m.p. 179^{o}C; white crystals; ^{1}H NMR (DMSO-d_6) δ ppm: 1.41 (s, 3H, H_a), 1.50 (s, 3H, H_b), 3.56 (m, 1H, H_c), (δ 6.03) (s, 1H, H_d), (δ 6.61-6.63) (d, 2H, $H_{ee'}$,), (δ 6.78-6.80) (d, 1H, H_f,), (δ 7.10-7.14) (t, 2H, $H_{gg'}$), (δ 7.18-7.20) (t, 1H, H_{hh}), (δ 7.23-7.31) (m, 3H, H_{i-k}), (δ 7.34-7.39) (dd, 2H, H_{lm}), (δ 7.83) (s, 1H, H_n), (δ 9.25) (s, 1H, H_o),(δ 10.13) (s, 1H, H_p).FT IR (cm^{-1}): 3260 (N-H stretching of secondary amine), 3020 (C-H stretching of aromatic ring), 2920 (C-H asymmetrical stretching of CH_3 group), 2815 (C-H asymmetrical stretching of CH_3 group), 1675 (C=O stretching of amide), 1625 (C=N stretching of triazole ring), 1514 (N-H deformation of pyrimidine ring), 1506 and 1482 (C=C stretching of aromatic ring), 1413 (C-H asymmetrical deformation of CH_3 group), 1400 (C-H symmetrical deformation of CH_3 group), 1323 (C-N stretching), 1221 (C-NO_2 symmetrical deformation of NO_2 group), 1203 (C-O-C stretching), 1002 (C-H in plane deformation of aromatic ring), 813 (C-H out of plane bending of 1,4-disubstituion), 728 (C-Cl stretching), Mass: m/z 558; Anal. Calcd. for $C_{27}H_{22}N_8O_8$: C, 55.29; H, 3.78; N, 19.11; O, 21.82; Found: C, 55.09; H, 3.38; N, 19.01; O, 21.32%.

7-(4-(2,4-dinitrophenoxy) phenyl)-N-(4-hydroxyphenyl)-4,7-dihydro-5-isopropyl-[1, 2, 4] triazolo [1,5-a] pyrimidine-6-carboxamide

4f. m.p. 176^{o}C; white crystals; ^{1}H NMR (DMSO-d_6) δ ppm: 1.31 (s, 3H, H_a), 1.40 (s, 3H, H_b), 3.11 (m, 1H, H_c), (δ 4.33) (s, 1H, H_d), (δ 6.32) (s, 1H, H_e), (δ 6.48-6.58) (d, 2H, $H_{ff'}$,), (δ 6.64-6.74) (d, 1H, H_g,), (δ 7.05-7.11) (t, 2H, $H_{hh'}$), (δ 7.16-7.29) (dd'dd', 4H, $H_{ii'-jj'}$), (δ 7.42-7.48) (dd, 2H, H_{kl}), (δ 7.62) (s, 1H, H_m), (δ 9.52) (s, 1H, H_n),(δ 10.11) (s, 1H, H_o). FT IR (cm^{-1}): 3286 (N-H stretching of secondary amine), 3084 (C-H stretching of aromatic ring), 2958 (C-H asymmetrical stretching of CH_3 group), 2868 (C-H asymmetrical stretching of CH_3 group), 1682 (C=O stretching of amide), 1681 (C=N stretching of triazole ring), 1585 (N-H deformation of pyrimidine ring), 1529 and 1462 (C=C stretching of aromatic ring), 1423 (C-H asymmetrical deformation of CH_3 group), 1401 (C-H symmetrical deformation of CH_3 group), 1354 (C-N stretching), 1245 (C-NO_2 symmetrical deformation of NO_2 group), 1234 (C-O-C stretching), 1031 (C-H in plane deformation of aromatic ring), 834 (C-H out of plane bending of 1,4-disubstituion), 728 (C-Cl stretching), Mass: m/z 558; Anal. Calcd. for $C_{27}H_{23}N_7O_7$: C, 58.17; H, 4.16; N, 17.59; O, 20.09; Found: C, 58.10; H, 4.10; N, 17.50; O, 20.00%.

7-(4-(2, 4-dinitrophenoxy) phenyl)-N-(2-chlorophenyl)-4, 7-dihydro-5-isopropyl-[1, 2, 4] triazolo [1,5-a] pyrimidine-6-carboxamide

4g. m.p. 183^{o}C; white crystals; ^{1}H NMR (DMSO-d_6) δ ppm: 1.43 (s, 3H, H_a), 1.52 (s, 3H, H_b), 3.11 (m, 1H, H_c), (δ 6.25) (s, 1H, H_d), (δ 6.71-6.73) (d, 2H, $H_{ee'}$,), (δ 6.78-6.80) (d, 1H, H_f,), (δ 7.00-7.04) (t, 2H, $H_{gg'}$), (δ 7.10-7.15) (t, 1H, H_h), (δ 7.20-7.24) (m, 5H, H_{i-l}), (δ 7.41-7.51) (dd, 2H, H_{mn}), (δ 7.52) (s, 1H, H_o), (δ 9.52) (s, 1H, H_p), (δ 10.02) (s, 1H, H_q). FT IR (cm^{-1}): 3225 (N-H stretching of secondary amine), 3013 (C-H stretching of aromatic ring), 2907 (C-H asymmetrical stretching of CH_3 group), 2844 (C-H asymmetrical stretching of CH_3 group), 1641 (C=O stretching of amide), 1602 (C=N stretching of triazole ring), 1514 (N-H deformation of pyrimidine ring), 1512 and 1454 (C=C stretching of aromatic ring), 1404 (C-H asymmetrical deformation of CH_3 group), 1401 (C-H symmetrical deformation of CH_3 group), 1376 (C-N stretching), 1264 (C-NO_2 symmetrical deformation of NO_2 group), 1246 (C-O-C stretching), 1022 (C-H in plane deformation of aromatic ring), 841 (C-H out of plane bending of 1,4-disubstituion), 722 (C-Cl stretching), Mass: m/z 576; Anal. Calcd. for $C_{27}H_{22}ClN_7O_6$: C, 56.30; H, 3.85; Cl, 6.16; N, 17.02; O, 16.67; Found: C, 56.10; H, 3.05; Cl, 6.00; N, 17.00; O, 16.27%.

7-(4-(2,4-dinitrophenoxy) phenyl)-N-(4-methoxyphenyl)-4,7-dihydro-5-isopropyl-[1, 2, 4] triazolo [1,5-a] pyrimidine-6-carboxamide

4h. m.p. 179°C; white crystals; ^{1}H NMR (DMSO-d_6) δ ppm: 1.40 (s, 3H, H_a), 1.56 (s, 3H, H_b), (δ 3.20) (s, 3H, H_c), 3.42 (m, 1H, H_d), (δ 3.46) (s, 2H, H_e), (δ 6.15) (s, 1H, H_f), (δ 6.61-6.63) (d, 2H, $H_{gg'}$,), (δ 6.78-6.82) (d, 1H, H_h,), (δ 7.01-7.05) (t, 2H, $H_{i'}$), (δ 7.08-7.10) (t, 1H, H_j), (δ 7.21-7.26) (m, 5H, H_{k-l}), (δ 7.41-7.51) (dd, 2H, H_{mn}), (δ 7.62) (s, 1H, H_o), (δ 9.66) (s, 1H, H_p), (δ 10.62) (s, 1H, H_q). FT IR (cm^{-1}): 3230 (N-H stretching of secondary amine), 3010 (C-H stretching of aromatic ring), 2953 (C-H asymmetrical stretching of CH_3 group), 2850 (C-H asymmetrical stretching of CH_3 group), 1660 (C=O stretching of amide), 1610 (C=N stretching of triazole ring), 1531 (N-H deformation of pyrimidine ring), 1500 and 1490 (C=C stretching of aromatic ring), 1431 (C-H asymmetrical deformation of CH_3 group), 1421 (C-H symmetrical deformation of CH_3 group), 1323 (C-N stretching), 1261 (C-NO_2 symmetrical deformation of NO_2 group), 1260 (C-O-C stretching), 1011 (C-H in plane deformation of aromatic ring), 831 (C-H out of plane bending of 1,4-disubstituion), Mass: m/z 572; Anal. Calcd. for $C_{28}H_{25}N_7O_7$: C, 58.84; H, 4.41; N, 17.15; O, 19.60; Found: C, 58.74; H, 4.21; N, 17.05; O, 19.40%.

7-(4-(2,4-dinitrophenoxy) phenyl)-N-(4-bromophenyl)-4,7-dihydro-5-isopropyl-[1, 2, 4] triazolo [1,5-a] pyrimidine-6-carboxamide

4i. m.p. 186°C; white crystals; ^{1}H NMR (DMSO-d_6) δ ppm: 1.33 (s, 3H, H_a), 1.46 (s, 3H, H_b), (δ 3.44) (s, 3H, H_c), (δ 6.03) (s, 1H, H_d), (δ 6.44-6.56) (d, 2H, $H_{ee'}$,), (δ 6.66-6.72) (d, 1H, H_f,), (δ 7.04-7.10) (t, 2H, $H_{gg'}$), (δ 7.16-7.36) (dd'dd', 4H, $H_{hh'-ii'}$), (δ 7.58-7.62) (dd, 2H, H_{jk}), (δ 7.80) (s, 1H, H_l), (δ 9.64) (s, 1H, H_m),(δ 10.20) (s, 1H, H_n). FT IR (cm^{-1}): 3227 (N-H stretching of secondary amine), 3011 (C-H stretching of aromatic ring), 2985 (C-H asymmetrical stretching of CH_3 group), 2858 (C-H asymmetrical stretching of CH_3 group), 1656 (C=O stretching of amide), 1603 (C=N stretching of triazole ring), 1556 (N-H deformation of pyrimidine ring), 1500 and 1453 (C=C stretching of aromatic ring), 1420 (C-H asymmetrical deformation of CH_3 group), 1401 (C-H symmetrical deformation of CH_3 group), 1344 (C-N stretching), 1223 (C-NO_2 symmetrical deformation of NO_2 group), 1200 (C-O-C stretching), 1050 (C-H in plane deformation of aromatic ring), 835 (C-H out of plane bending of 1,4-disubstituion), 732 (C-Br stretching), Mass: m/z 620; Anal. Calcd. for $C_{27}H_{22}BrN_7O_6$: C, 52.27; H, 3.57; Br, 12.88; N, 15.80; O, 15.47; Found: CC, 52.27; H, 3.57; Br, 12.88; N, 15.80; O, 15.42%.

7-(4-(2,4-dinitrophenoxy) phenyl)-N-(3-bromophenyl)-4,7-dihydro-5-isopropyl-[1, 2, 4] triazolo [1,5-a] pyrimidine-6-carboxamide

4j. m.p. 188°C; white crystals; ^{1}H NMR (DMSO-d_6) δ ppm: 1.33 (s, 3H, H_a), 1.46 (s, 3H, H_b), (δ 3.44) (s, 3H, H_c), (δ 6.31) (s, 1H, H_d), (δ 6.32-6.54) (d, 2H, $H_{ee'}$,), (δ 6.64-6.70) (d, 1H, H_f,), (δ 7.02-7.09) (t, 2H, $H_{gg'}$), (δ 7.22-7.30) (dd'dd', 4H, $H_{hh'-ii'}$), (δ 7.40-7.52) (dd, 2H, H_{jk}), (δ 7.74) (s, 1H, H_l), (δ 9.27) (s, 1H, H_m),(δ 10.34) (s, 1H, H_n). FT IR (cm^{-1}): 3212 (N-H stretching of secondary amine), 3021 (C-H stretching of aromatic ring), 2932 (C-H asymmetrical stretching of CH_3 group), 2852 (C-H asymmetrical stretching of CH_3 group), 1603 (C=O stretching of amide), 1587 (C=N stretching of triazole ring), 1537 (N-H deformation of pyrimidine ring), 1500 and 1450 (C=C stretching of aromatic ring), 1400 (C-H asymmetrical deformation of CH_3 group), 1390 (C-H symmetrical deformation of CH_3 group), 1350 (C-N stretching), 1228 (C-NO2 symmetrical deformation of NO2 group), 1212 (C-O-C stretching), 1048 (C-H in plane deformation of aromatic ring), 838 (C-H out of plane bending of 1,4-disubstituion), 723 (C-Br stretching), Mass: m/z 591; Anal. Calcd. for $C_{27}H_{22}BrN_7O_6$: C, 52.27; H, 3.57; Br, 12.88; N, 15.80; O, 15.47; Found: CC, 52.17; H, 3.47; Br, 12.78; N, 15.70; O, 15.42%.

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