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EFFICIENT SYNTHESIS OF 1, 2, 4-TRIAZOLO [3, 4 -B]-1, 3, 4 - THIADIZOLES DERIVATIVES

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ABSTRACT:

Derivatives of 3,6-disubstituted1,2,4-triazolo-[3,4-b]-1,3,4-thiadiazoles containing pyridine moiety was 4-Amino-5-pyridin-4-yl-4*H*-[1,2,4]triazole-3-thiol and 4-Amino-5-pyridin-3-yl-4*H*-[1,2,4] triazole-3-thiol synthesized by cyclization using substituted aromatic carboxylic acid in presence of $POCl_3$. Derivatives of 3,6-disubstituted1,2,4-triazolo-[3,4-b]-1,3,4-thiadiazoles containing pyridine moiety was 4-Amino-5-pyridin-4-yl-4*H*-[1,2,4]triazole-3-thiol and 4-Amino-5-pyridin-3-yl-4*H*-[1,2,4] triazole-3-thiol synthesized by cyclization using substituted aromatic carboxylic acid in presence of $POCl_3$.

Keywords: Triazolo, Thiadiazoles, $POCl_3$, Pyridine, Cyclization, Carboxylic acid, substituted aromatic carboxylic acid.

1. INTRODUCTION

Heterocyclic compounds are the most important and maintain a vigorous field in organic chemistry. They play a significant role in the drug design and synthesis process. 1,2,4-triazole is a significant heterocyclic compound that has attracted the attention of synthetic chemists. Many substituted triazole and its fused heterocyclic compounds have exposed various biological activities [1-10]. Triazoles and their derivatives exhibit importance in bactericides, pesticides and fungicidesproperties [11–13]. Some compounds have been developed as commercial medicinal drugs (Fig.5.1), such as Fluconazole, Itraconazole, Ravuconazole, and Voriconazole wonderful to their diverse properties, 1, 2, 4-triazole derivatives may develop into one of the focuses in drug research [14-17].

Several heterocyclic derivatives containing pyridine fused with triazole were associated with diverse pharmacological properties such as antimicrobial [18-19], antifungal [20], and antitumor [21]. In addition, there is an application in light-emitting electrochemical cells. Similar to triazole thiadiazole is also an important class of heterocyclic compounds showing various important biological activities such as antibacterial[22], antimicrobial[23-25], anti-tubercular[26-27], anticancer[28-31], insecticidal[32], antileishmanial[33], antitrypanosomal[34], anti-inflammatory[35], cytotoxicity[36-37], antioxidant activities[37], anti-HIV[38], antitumor agents[39]. In addition, they are also used as a light emitting diodes [40].1, 2, 4-triazolo [3, 4-b]-1, 3, 4-thiadiazoles, a fused triazole and thiadiazoles are some important nitrogen-containing heterocyclic compounds having pharmacological activity.

Experimental

General

All reactions were performed in electric oven-dried glassware under atmospheric pressure. All the starting reagents, materials, and solvents used were of analytical grade (AR) and used as it is received by commercial suppliers. The melting point of the synthesized compounds was taken on a precision melting point apparatus (DBK instrument), and all are uncorrected. A Nicolet 400D spectrometer was used to obtain the compounds' infrared (IR) spectra, and a Bruker 400MHz spectrometer was used to record their 1H NMR and 13C spectra in DMSO or CDCl3 solvent. A mass spectrum was recorded on a Waters ZQ-4000 spectrometer. The yield of the synthesized compounds mentioned is for isolated products. Pre-coated TLC on silica gel plates with a 2 mm thickness was used to monitor the reaction's progress while employing n-hexane and ethyl acetate as the solvent system. In addition to a UV chamber, an iodine chamber was used to visualise the location.

General Procedure for the Synthesis of Potassium Dthiocarbazinate (2a-B)

Substituted acid hydrazide 1a-b (0.01 mol) was reacted in alcoholic potassium hydroxide (0.08 g KOH in 20 mL aqueous ethanol, 1.5 mol) by using carbon disulphide (0.01 mol) for a period of 4.5 h. The isolated potassium dithiocarbazinate2a-b salt was removed, dried, and used without additional purification for the subsequent step (yield: 80%). After the reaction was finished, the precipitate filter was collected, washed with anhydrous diethyl ether (10 mL), dried, and utilised without additional purification for the next step.

Synthesis of 4-Amino-5-Pyridin-4-Yl-4*H*-[1, 2, 4] Triazole-3-Thiol and 4-Amino-5-Pyridin-3-Yl-4*H*-[1, 2, 4] Triazole-3-Thiol (3a-B).

A mixture of potassium dithiocarbamate (0.1 mol) (2a-b) in hydrazine hydrate (80 %, 10 mL) was heated for reflux for 9 h. After the evolution of H_2S gas, a clear reaction mass was observed. The reaction mass was added to ice-cold water and the precipitate was formed by the

addition of dilute (4N) HCl solution. The obtained solid was filtered out washed with water and recrystallized with 50% ethanol.

General Procedure for Synthesis of 6-(Substituted)-1, 2, 4-Triazolo-[3, 4-B]-1, 3, 4-Thiadiazoles (4a–J)

An equimolar mixture of 4-amino-5-diphenylmethyl-4*H*-1,2,4-triazole-3-thiol **(3a-b)** (0.01 mol) and aromatic acids (0.01 mol) in phosphorus oxychloride (10 mL) was refluxed for 4 h. The reaction mixtures were cooled to room temperature and then gradually poured onto crushed ice with stirring. The mixtures were allowed to stand overnight and the solids separated out were filtered, treated with dilute sodium hydroxide solution and washed thoroughly with cold water. The compound so obtained was dried and recrystallized with ethanol (4a-j).

Spectral Data

6-(2-(Trifluoromethyl)Pyridin-3-Yl)-3-Phenyl-[1,2,4]Triazolo[3,4-B][1,3,4]Thiadiazole (4a)

Melting Point: 218^{0}C. IR 1650(C=N), 639 (C-S-C), 2895 (C-H aromatic), 1272 (N-N=C triazolo thiadiazole), 1195 (C-F). ^{1}H NMR (400 MHz, DMSO, δ ppm): 7.28 (d, 1H, ArH), 7.42 (dd, 2H, ArH), 7.51(d, 2H, ArH), 7.82 (d, 1H, ArH), 8.35 (dd, 1H, ArH), 8.37 (d, 1H, ArH).

6-(2,4-Difluorophenyl)-3-Phenyl-[1,2,4]Triazolo[3,4-B][1,3,4]Thiadiazole(4b).

Melting Point: 230^{0}C. IR 1601(C=N), 685 (C-S-C), 3175 (C-H aromatic), 1272 (N-N=C triazolo thiadiazole), 1195 (C-F). ^{1}H NMR (400 MHz, DMSO, δ ppm): 7.42 (d, 1H, ArH,), 7.50 (dd, 2H, ArH), 7.52 (d, 2H, ArH), 8.27 (s, 1H), 8.48 (d, 1H, ArH), 9.01 (d, 1H, ArH).

6-(3-Methyl-Pyridin-4-Yl)-3-Phenyl-[1,2,4]Triazolo[3,4-B][1,3,4]Thiadiazole(4c).

Melting Point: 210^{0}C. IR 1601(C=N), 685 (C-S-C), 3175 (C-H aromatic), 1272 (N-N=C triazolo thiadiazole), ^{1}H NMR (400 MHz, DMSO, δ ppm): 1.98 (s, 3H, -CH_3), 7.40 (d, 1H,ArH), 7.52 (dd, 2H, ArH), 7.54 (d, 2H, ArH),8.270 (s, 1H, ArH),8.46 (d, 1H, ArH), 9.00 (d, 1H, ArH).

3-Phenyl-6-M-Toly-[1,2,4]Triazolo[3,4-B][1,3,4]Thiadiazole(4d).

Melting Point: 213^{0}C. IR 1601(C=N), 685 (C-S-C), 3175 (C-H aromatic), 1272 (N-N=C triazolo thiadiazole), ^{1}H NMR (400 MHz, DMSO, δ ppm): 1.89 (s, 3H, -CH_3), 7.18 (d, 2H,ArH), 7.50 (dd, 2H, ArH), 7.56 (d, 2H, ArH),8.20 (s, 1H, ArH),8.50 (d, 1H, ArH), 9.12 (d, 1H, ArH).

6-(2-Methoxy-Phenyl)-3-Phenyl-[1,2,4]Triazolo[3,4-B][1,3,4]Thiadiazole(4e).

Melting Point: 240^{0}C. IR 1601(C=N), 685 (C-S-C), 3175 (C-H aromatic), 1272 (N-N=C triazolo thiadiazole), 1100 (O-CH_3). ^{1}H NMR (400 MHz, DMSO, δ ppm): 2.30 (s, 3H, -OCH_3), 7.38 (d, 1H, ArH), 7.50 (dd, 2H, ArH), 7.58 (d, 2H, ArH), 8.22 (d, 2H, ArH), 8.48 (d, 1H, ArH), 9.20 (d, 1H, ArH).

6-(3-Methoxy-Phenyl)-3-Phenyl-[1,2,4]Triazolo[3,4-B][1,3,4]Thiadiazole(4f).

Melting Point: 246^{0}C. IR 1601(C=N), 685 (C-S-C), 3175 (C-H aromatic), 1272 (N-N=C triazolo thiadiazole), 1100 (O-CH_3). ^{1}H NMR (400 MHz, DMSO, δ ppm): 2.3 (s, 3H, -OCH_3), 7.42 (d, 1H, ArH), 7.53 (dd, 2H, ArH), 7.59 (d, 2H, ArH), 8.00 (s, 1H, ArH), 8.42 (d, 2H, ArH), 9.03 (d, 1H, ArH).

6-(3-Fluoro-Pyridin-4-Yl)-3-Phenyl-[1,2,4]Triazolo[3,4-B][1,3,4]Thiadiazole(4g).
Melting Point: 232^{0}C. IR 1601(C=N), 685 (C-S-C), 3175 (C-H aromatic), 1272 (N-N=C triazolo thiadiazole), 1100 (O-CH_3), 1195 (C-F). ^{1}H NMR (400 MHz, DMSO, δ ppm): 7.42 (d, 1H, ArH), 7.53 (dd, 2H, ArH), 7.55 (d, 2H, ArH), 8.27 (s, 1H, ArH), 8.49 (d, 1H, ArH), 9.11 (d, 1H, ArH).

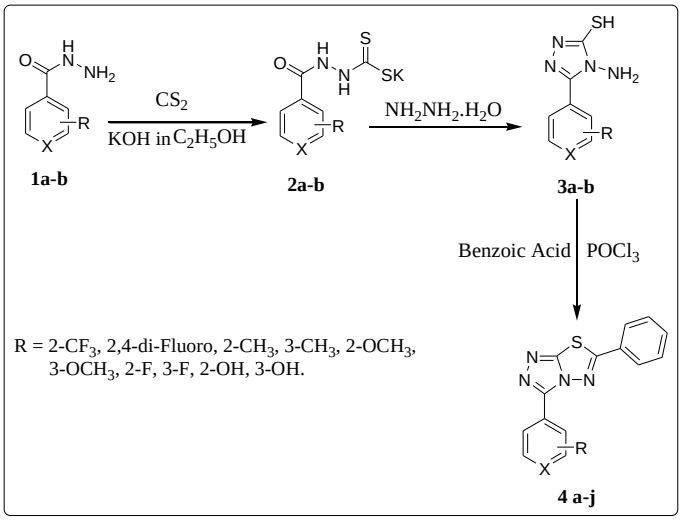
6-(3-Fluoro-Phenyl)-3-Phenyl-[1,2,4]Triazolo[3,4-B][1,3,4]Thiadiazole(4h).
Melting Point: 234^{0}C. IR 1601(C=N), 685 (C-S-C), 3175 (C-H aromatic), 1272 (N-N=C triazolo thiadiazole), 1100 (O-CH_3), 1195 (C-F). ^{1}H NMR (400 MHz, DMSO, δ ppm): 7.38 (d, 1H, ArH), 7.51 (dd, 2H, ArH), 7.53 (d, 2H, ArH), 8.27 (s, 1H, ArH), 8.50 (d, 2H, ArH), 9.04 (d, 1H, ArH).

2-(3-Phenyl)-[1,2,4]Triazolo[3,4-B][1,3,4]Thiadiazole(4i).
Melting Point: 237^{0}C. IR 1601(C=N), 685 (C-S-C), 3175 (C-H aromatic), 1272 (N-N=C triazolo thiadiazole), 1100 (O-CH_3), 3300 (OH). ^{1}H NMR (400 MHz, DMSO, δ ppm): 7.48 (d, 1H, ArH), 5.20 (s, 1H, -OH), 7.50 (dd, 2H, ArH), 7.54 (d, 2H, ArH), 8.29 (d, 2H, ArH), 8.42 (d, 1H, ArH), 9.11 (d, 1H, ArH).

3-(3-Phenyl)-[1,2,4]Triazolo[3,4-B][1,3,4] Thiadiazole(4j).
Melting Point: 204^{0}C. IR 1601(C=N), 685 (C-S-C), 3175 (C-H aromatic), 1272 (N-N=C triazolo thiadiazole), 1100 (O-CH_3), 3300 (OH). ^{1}H NMR (400 MHz, DMSO, δ ppm): 5.20 (s, 1H, -OH), 7.32 (d, 1H, ArH), 7.52 (dd, 2H, ArH), 7.65 (d, 2H, ArH), 8.30 (s, 1H, ArH), 8.50 (d, 2H, ArH), 9.20 (d, 1H, ArH).

2. RESULT AND DISCUSSION

Present synthesis involves the synthesis of aimed derivatives are shown in scheme 4.13. The compound (2a-b) was prepared by reaction acid hydrazide (1 a-b) with carbon disulphide in ethanolic potassium hydroxide. Required substituted pyridine-4-yl-4*H*-[1, 2, 4] triazole-3-thiol) (3a-b) was synthesized from corresponding potassium dithiocarbazates refluxing with aqueous hydrazine hydrate (80%). The resultant substituted pyridine-4-yl-4*H*-[1,2,4]triazole-3-thiol) (3a-b) was further converted into fluoro substituted -[1,2,4] triazolo [3,4-b][1,3,4] thiadiazole4(a-j) using fluoro substituted aromatic acid in the presence of phosphorus oxychloride (Table 1).



Scheme 1. Synthesis of fluoro substituted - [1, 2, 4] triazolo [3, 4-b][1,3,4] thiadiazole4(a-j)

Table 1: Substitution scope of synthesized derivatives 4a-j.

Entry	R=Substituents (X=C / N)	Reaction Time (h)	M. P. ^{O}C.	% Yield (Isolated)
4a	2-CF_3 (X=N)	3.5	218	78
4b	2,4-di-F (X=C)	4.0	230	82
4c	2-CH_3 (X=N)	2.5	210	88
4d	3-CH_3 (X=C)	3.0	213-215	80
4e	2-OCH_3 (X=C)	4.0	240	72
4f	3-OCH_3 (X=C)	4.5	246-248	79
4g	2-F (X=N)	3.0	232	81
4h	3-F (X=C)	3.0	234	88
4i	2-OH (X=C)	3.5	237-238	84
4j	3-OH (X=C)	4.0	204-206	80

The IR spectra exhibit an absorption band at 1650for C=N, 639 (C-S-C), 2895 (C-H aromatic), 1272 (N-N=C triazole thiadiazole), 1195 (C-F) confirm the cyclization of synthesized compound (4a). The disappearance of absorption peaks for NH_2 and SH stretching is observed. The ^{1}H NMR characteristic peaks were observed at δ 8.19–9.51 as multiplet for four aromatic protons and singlet observed for 1H proton between fluorine atoms. Fig. 1 represents the graph of per cent yields of the synthesized compounds.

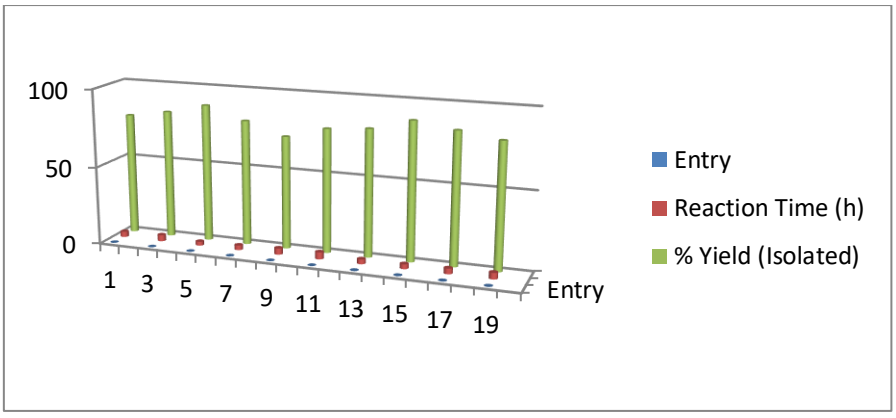
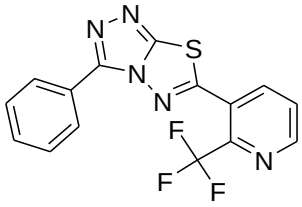
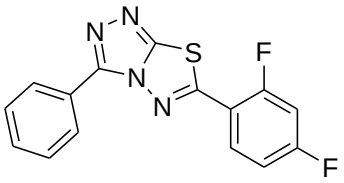


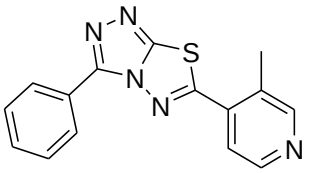
Fig. 1 Graphical representation for the synthesis of 4a – 4j.



6-(2-(trifluoromethyl)pyridin-3-yl)-3-phenyl-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazole (4a)

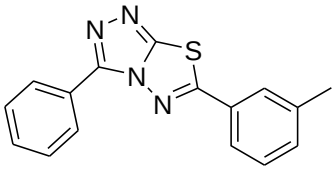


6 -(2,4-difluorophenyl)-3-phenyl-[1,2,4] triazolo[3,4-b][1,3,4]thiadiazole**(4b)**



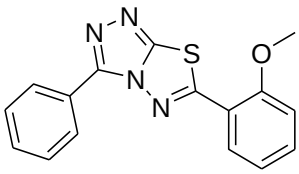
6-(3-Methyl-pyridin-4-yl)-3-phenyl-[1,2,4]triazolo[3,4-*b*][1,3,4]thiadiazole

4c



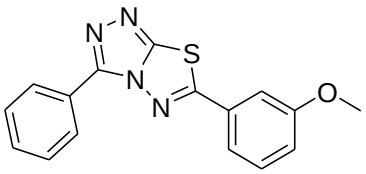
3-Phenyl-6-*m*-tolyl-[1,2,4]triazolo[3,4-*b*][1,3,4]thiadiazole

4d



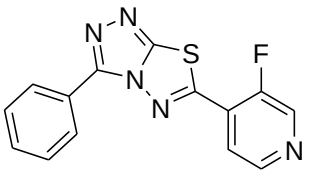
6-(2-Methoxy-phenyl)-3-phenyl-[1,2,4]triazolo[3,4-*b*][1,3,4]thiadiazole

4e



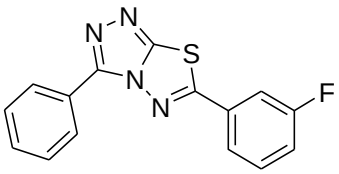
6-(3-Methoxy-phenyl)-3-phenyl-[1,2,4]triazolo[3,4-*b*][1,3,4]thiadiazole

4f



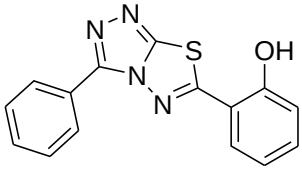
6-(3-Fluoro-pyridin-4-yl)-3-phenyl-[1,2,4]triazolo[3,4-*b*][1,3,4]thiadiazole

4g



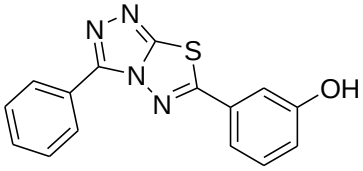
6-(3-Fluoro-phenyl)-3-phenyl-[1,2,4]triazolo[3,4-*b*][1,3,4]thiadiazole

4h



2-(3-Phenyl-[1,2,4]triazolo[3,4-*b*][1,3,4]thiadiazol-6-yl)-phenol

4i



3-(3-Phenyl-[1,2,4]triazolo[3,4-*b*][1,3,4]thiadiazol-6-yl)-phenol

4j

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