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An Efficient and Ecofriendly Synthesis of 3,7-Diaryltetrahydro-1*H*,5*H*-[1,2,4]triazolo[1,2-*a*][1,2,4]triazole-1,5-dithiones

A.M.M. Mallikarjunaswamy[1], Bhuvaneshwari S. Vijaya[1], Gopal L. Khatik[2], Sidharth Chopra[3,4], Abdul Akhir[3], Deepanshi Saxena[3] and Vipin A. Nair[1,*]

[1]Department of Chemistry, REVA University, Yelahanka, Bangalore-560064, India
[2]Department of Medicinal Chemistry, National Institute of Pharmaceutical Education and Research, Raebareli-226002, India
[3]Division of Microbiology, CSIR-Central Drug Research Institute, Sitapur Road, Sector 10, Janakipuram Extension, Lucknow-226031, India
[4]Academy of Scientific and Innovative Research, Ghaziabad-201002, India

*Corresponding author: Tel: +91 80 9021190211, Extn. 225; E-mail: vipina.nair@reva.edu.in

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An efficient and environmentally friendly method was developed for the synthesis of 3,7-diaryltetrahydro-1*H*,5*H*-[1,2,4]triazolo[1,2-*a*]-[1,2,4]triazole-1,5-dithiones by reacting aldazines with KSCN and HCl in an ethanolic medium. The products were obtained in excellent yields within short duration of time, along with the advantage of ease of isolation by precipitation. The reaction mechanism substantiates the formation of a protonated intermediate, which facilitates an accelerated nucleophilic addition. Among the compounds synthesized, the 3,7-*bis*(4-chlorophenyl)tetrahydro-1*H*,5*H*-[1,2,4]triazolo[1,2-*a*][1,2,4]triazole-1,5-dithione demonstrated remarkable antibacterial properties.

Keywords: Triazolo[1,2-*a*][1,2,4]triazole-1,5-dithiones, Aldazines, Thiocyanate, Protonation, Ecofriendly.

INTRODUCTION

Potassium thiocyanate (KSCN) is widely used for introducing sulfur-containing functionalities and serve as an important auxiliary in various organic transformations and synthetic sequences [1]. For instance, dialkyl acetylenedicarboxylates react with indane-1,3-dione in the presence of catalytic amount of KSCN to afford 4-oxo-3,4-dihydro-2*H*-indeno[1,2-*b*]furan-2,3-dicarboxylates [2]. The one-pot reaction was performed under mild conditions in acetone medium. Copper catalyzed reactions of *o*-bromobenzamide derivatives with KSCN in water proceeds *via* tandem C-S and N-S bond formation to give benzisothiazol-3(2*H*)-one derivatives [3]. The α-azidochalcones on reaction with potassium thiocyanate along with potassium persulfate afforded 2,4,5-trisubstituted oxazoles [4]. The same starting materials gave 2-aminothiazoles when ferric nitrate was used with KSCN. Oxiranes have been efficiently converted to thiiranes by treating with KSCN in PEG-400 medium [5]. The synthesis of thiiranes was also accomplished from oxiranes in acetonitrile medium by treating with KSCN in the presence of catalytic amount of $LiClO_4$ at room temperature [6].

In one-pot reaction of supported reagent system KSCN/SiO_2–RNH_3OAc/Al_2O_3, allyl bromides reacted with KSCN/SiO_2 to form allyl isothiocyanates which subsequently reacted with RNH_3OAc/Al_2O_3 to form allyl thioureas [7]. However, extension of this strategy to α-haloketones resulted in the formation of intermediate α-thiocyanatoketones upon reaction with KSCN/SiO_2 and their subsequent reaction with RNH_3OAc/Al_2O_3 gave 2-aminothiazoles [8]. Regioselective ring opening of aziridines was achieved with KSCN using β-cyclodextrin as the catalyst in water at room temperature [9]. Nevertheless, the ring opening of aziridines with KSCN in silica-water under heating conditions afforded ring expansion products [10]. The reactions of 1,3-disubstituted imidazolium salts with KSCN under microwave-assisted solvent free conditions provided 1,3-disubstituted imidazol-2-thiones [11]. Microwave-assisted efficient synthesis of alkyl thiocyanates was achieved in aqueous medium by nucleophilic substitution reactions of alkali thiocyanates with halides or tosylates [12]. The reaction occurs in the absence of phase transfer catalysts and a variety of functional groups are tolerated. In aqueous medium, oxidation of potassium thiocyanate releases cyanide ions which can react

with tertiary amines to form α-aminonitriles [13]. The reaction of aromatic 1,4-diazabuta-1,3-dienes with KSCN in ethereal acetic acid gave the corresponding perhydro[4,5-*d*]imidazol-2,5-dithiones [14]. The reaction involves a direct nucleophilic addition of thiocyanate anion to the protonated 1,4-diazabuta-1,3-diene.

Keeping in mind these facts, the present work discusses a highly effective and environment friendly synthesis of 3,7-diaryltetrahydro-1*H*,5*H*-[1,2,4]triazolo[1,2-*a*] [1,2,4]triazole-1,5-dithiones from aldazines, using KSCN and HCl in ethanolic medium.

EXPERIMENTAL

All chemicals were purchased from commercial suppliers and used as received. The reactions were performed in oven-dried glass wares under appropriate atmosphere. The reactions were monitored by thin-layer chromatography (TLC) on 0.25 mm Merck Silica gel 60 F_{254} plates using UV light for visualization. IR spectra were recorded on a Bruker Alpha II FTIR spectrophotometer. NMR spectra were recorded on a Jeol ECZ 400R spectrometer (^{1}H at 400 MHz, ^{13}C at 100 MHz) using DMSO-d_6 as solvent with TMS as the internal standard. Chemical shifts (δ) are reported relative to residual solvent signals (DMSO-d_6, 2.5 ppm for ^{1}H NMR and 39.5 ppm for ^{13}C NMR). The melting points are uncorrected. High resolution mass spectrometry was carried out in an ESI quadrupole time of flight Agilent Mass spectrometer.

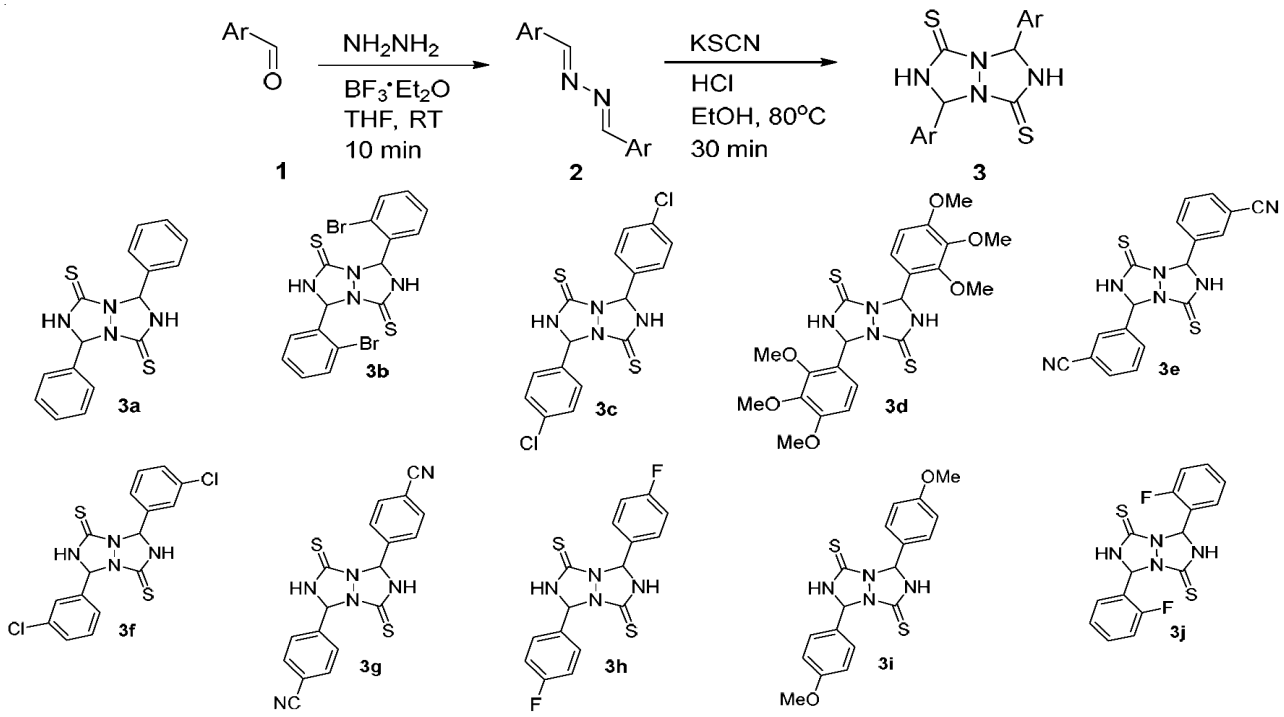
Synthesis of 3,7-diaryltetrahydro-1*H*,5*H*-[1,2,4]triazolo-[1,2-*a*][1,2,4]triazole-1,5-dithione (3a-j): To a solution of KSCN (1 mmol) in ethanol (10 mL), HCl (0.5 mL) was added and stirred at room temperature for 5 min. The aldazine (1.0 equiv.) was added to the reaction mixture and refluxed at 80 ºC for 15-20 min. The progress of the reaction was monitored by TLC. The product that precipitated out of the reaction mixture was filtered, washed successively with ice-cold water and diethyl ether, and recrystallized (**Scheme-I**).

3,7-Diphenyltetrahydro-1*H*,5*H*-[1,2,4]triazolo[1,2-*a*]-[1,2,4]triazole-1,5-dithione (3a): Time: 20 min, yield: 98%; m.p. 189-190 ºC; IR (KBr, ν_{max}, cm^{-1}): 3117.79, 2957.23, 1578.39, 1484.27, 1380.17, 1339.43, 1254.43, 1193.47, 1141.53, 1049.64, 981.09, 864.71, 818.69, 778.70, 644.83; ^{1}H NMR (400 MHz, DMSO-d_6) δ ppm: 11.38 (s, 2H), 7.35-7.43 (m, 10H), 6.80 (s, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ ppm: 184.56, 137.35, 129.49, 129.41, 126.21, 76.89; HRMS (ESI): *m/z* calcd. for $C_{16}H_{14}N_4S_2$ [M+H]$^+$: 326.0660; found: 327.0744.

3,7-*bis*(2-Bromophenyl)tetrahydro-1*H*,5*H*-[1,2,4]-triazolo[1,2-*a*][1,2,4]triazole-1,5-dithione (3b): Time: 15 min, yield: 96%; m.p.: 185-188 ºC; IR (KBr, ν_{max}, cm^{-1}): 3189.06, 1495.28, 1338.35, 1245.40, 1208.03, 1178.65, 1026.53, 992.42, 946.19, 839.96, 751.44, 686.59, 623.57; ^{1}H NMR (400 MHz, DMSO-d_6) δ ppm: 11.45 (s, 2H), 7.68-7.72 (m, 2H), 7.43-7.49 (m, 2H), 7.32-7.38 (m, 4H), 7.13 (d, J = 0.6 Hz, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ ppm: 185.07, 135.77, 133.90, 132.18, 129.36, 128.47, 122.39, 77.26; HRMS (ESI): *m/z* calcd. for $C_{16}H_{12}Br_2N_4S_2$ [M+H]$^+$: 481.8870; found: 482.8962.

3,7-*bis*(4-Chlorophenyl)tetrahydro-1*H*,5*H*-[1,2,4]-triazolo[1,2-*a*][1,2,4]triazole-1,5-dithione (3c): Time: 20 min, yield: 95%; m.p.: 197-200 ºC; IR (KBr, ν_{max}, cm^{-1}): 3174.65, 2838.14, 1701.63, 1593.67, 1489.49, 1410.55, 1332.91, 1246.73, 1213.05, 1171.22, 1090.25, 1013.75, 829.27, 679.72, 577.73; ^{1}H NMR (400 MHz, DMSO-d_6) δ ppm: 11.43 (s, 2H), 7.47-7.52 (m, 4H), 7.35-7.39 (m, 4H), 6.83 (s, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ ppm: 184.56, 136.37, 134.43, 129.57, 128.23, 76.50; HRMS (ESI): *m/z* calcd. for $C_{16}H_{12}Cl_2N_4S_2$ [M+H]$^+$: 393.9880; found: 394.9964.

3,7-*bis*(2,3,4-Trimethoxyphenyl)tetrahydro-1*H*,5*H*-[1,2,4]triazolo[1,2-*a*][1,2,4]triazole-1,5-dithione (3d): Time:



Scheme-I: Synthesis of 3,7-diaryltetrahydro-1*H*,5*H*-[1,2,4]triazolo[1,2-*a*][1,2,4]triazole-1,5-dithiones

20 min, yield: 96%; m.p.: 155-158 ºC; IR (KBr, ν_{max}, cm^{-1}): 3167.47, 2838.65, 1593.27, 1510.30, 1460.30, 1420.47, 1332.36, 1236.12, 1202.39, 1126.77, 997.42, 833.71, 869.07; ^{1}H NMR (400 MHz, DMSO-d_6) δ ppm: 11.40 (s, 2H), 6.52-6.79 (m, 6H), 3.72 (s, 12H), 3.62 (d, *J* = 2.8 Hz, 6H); ^{13}C NMR (100 MHz, DMSO-d_6) δ ppm: 184.07, 153.87, 153.09, 138.43, 137.27, 102.92, 76.88, 60.67, 56.22; HRMS (ESI): *m/z* calcd. for $C_{22}H_{26}N_4O_6S_2$ $[M+H]^+$: 506.1294; found: 507.1432.

3,3′-(3,7-Dithioxotetrahydro-1*H*,5*H*-[1,2,4]triazolo [1,2-*a*][1,2,4]triazole-1,5-diyl)dibenzonitrile (3e): Time: 30 min, yield: 92%; m.p.: 175-177 ºC; IR (KBr, ν_{max}, cm^{-1}): 3162.24, 2822.92, 1493.80, 1271.75, 1212.34, 1158.03, 992.35, 953.25, 831.12, 679.43, 606.07; ^{1}H NMR (400 MHz, DMSO-d_6) δ ppm: 11.53 (s, 2H), 7.86-7.90 (m, 2H), 7.80 (t, *J* = 1.6 Hz, 2H), 7.71 (dt, *J* = 7.9, 1.4 Hz, 2H), 7.63-7.68 (m, 2H), 6.94 (s, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ ppm: 184.55, 139.00, 133.55, 131.09, 131.02, 130.03, 118.75, 112.03, 76.50; HRMS (ESI): *m/z* calcd. for $C_{18}H_{12}N_6S_2$ $[M+H]^+$: 376.0565; found: 377.0650.

3,7-*bis*(3-Chlorophenyl)tetrahydro-1*H*,5*H*-[1,2,4]-triazolo[1,2-*a*][1,2,4]triazole-1,5-dithione (3f): Time: 20 min, yield: 96%; m.p.: 194-196 ºC; IR (KBr, ν_{max}, cm^{-1}): 3127.07, 2971.31, 1570.69, 1515.87, 1436.88, 1350.94, 1264.63, 1226.31, 1093.77, 847.70, 774.94, 658.78, 625.55, 592.09; ^{1}H NMR (400 MHz, DMSO-d_6) δ ppm: 11.45 (s, 2H), 7.47-7.53 (m, 4H), 7.35-7.40 (m, 4H), 6.83 (s, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ ppm: 184.52, 139.79, 134.06, 131.64, 129.77, 126.13, 124.91, 76.48; HRMS (ESI): *m/z* calcd. for $C_{16}H_{12}Cl_2N_4S_2$ $[M+H]^+$: 393.9880; found: 394.9959.

4,4′-(3,7-Dithioxotetrahydro-1*H*,5*H*-[1,2,4]triazolo-[1,2-*a*][1,2,4]triazole-1,5-diyl)dibenzonitrile (3g): Time: 30 min, yield: 91%; m.p.: 161-163 ºC; IR (KBr, ν_{max}, cm^{-1}): 3194.06, 2823.66, 2306.41, 1619.46, 1524.91, 1492.85, 1350.00, 1247.46, 1216.43, 1186.61, 1094.03, 1018.83, 807.02, 731.29, 695.14, 667.01, 573.06; ^{1}H NMR (400 MHz, DMSO-d_6) δ ppm: 11.59 (s, 2H), 7.87-7.94 (m, 4H), 7.51-7.60 (m, 4H), 6.94 (s, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ ppm: 184.61, 160.88, 142.36, 133.56, 129.33, 127.14, 76.84; HRMS (ESI): *m/z* calcd. for $C_{18}H_{12}N_6S_2$ $[M+H]^+$: 376.0565; found: 377.0644.

3,7-*bis*(4-Fluorophenyl)tetrahydro-1*H*,5*H*-[1,2,4]-triazolo[1,2-*a*][1,2,4]triazole-1,5-dithione (3h): Time: 25 min, yield: 97%; m.p.: 167-170 ºC; IR (KBr, ν_{max}, cm^{-1}): 3140.23, 1603.90, 1510.65, 1338.96, 1250.80, 1224.30, 1183.18, 1155.67, 1094.58, 1012.81, 857.03, 832.05, 658.23, 596.50; ^{1}H NMR (400 MHz, DMSO-d_6) δ ppm: 11.41 (s, 2H), 7.38-7.43 (m, 4H), 7.22-7.29 (m, 4H), 6.81 (s, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ ppm: 184.52, 139.81, 131.64, 126.18, 124.96, 76.88; HRMS (ESI): *m/z* calcd. for $C_{16}H_{12}F_2N_4S_2$ $[M+H]^+$: 362.0471; found: 363.0550.

3,7-*bis*(4-methoxyphenyl)tetrahydro-1*H*,5*H*-[1,2,4]-triazolo[1,2-*a*][1,2,4]triazole-1,5-dithione (3i): Time: 20 min, yield: 98%; m.p.: 131-133 ºC; IR (KBr, ν_{max}, cm^{-1}): 3399.46, 3155.58, 2895.66, 2831.79, 1605.57, 1486.47, 1359.42, 1301.88, 1244.80, 1166.78, 1082.04, 1023.79, 959.48, 827.08, 607.28; ^{1}H NMR (400 MHz, DMSO-d_6) δ ppm: 11.24 (s, 2H), 7.25-7.29 (m, 4H), 6.94-6.98 (m, 4H), 6.72 (s, 2H), 3.70-3.72 (m, 6H); ^{13}C NMR (100 MHz, DMSO-d_6) δ ppm: 184.51, 160.29, 129.44, 127.71, 114.80, 77.15, 55.80; HRMS (ESI): *m/z* calcd. for $C_{18}H_{18}N_4O_2S_2$ $[M+H]^+$: 386.0871; found: 387.0954.

3,7-*bis*(2-Fluorophenyl)tetrahydro-1*H*,5*H*-[1,2,4]-triazolo[1,2-*a*][1,2,4]triazole-1,5-dithione (3j): Time: 15 min, yield: 94%; m.p.: 179-182 ºC; IR (KBr, ν_{max}, cm^{-1}): 3142.10, 2877.28, 2818.00, 1619.56, 1586.24, 1483.75, 1453.56, 1346.00, 1251.64, 1193.86, 1168.28, 1091.39, 997.53, 860.65, 752.73, 679.80, 627.09, 571.23; ^{1}H NMR (400 MHz, DMSO-d_6) δ ppm: 11.37 (s, 2H), 7.43-7.50 (m, 2H), 7.23-7.33 (m, 6H), 7.04 (s, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ ppm: 184.46, 158.72, 132.45, 128.19, 125.74, 124.15, 116.78, 72.34; HRMS (ESI): *m/z* calcd. for $C_{16}H_{12}F_2N_4S_2$ $[M+H]^+$: 362.0471; found: 363.0553.

Antibacterial activities: The synthesized 3,7-diaryltetrahydro-1*H*,5*H*-[1,2,4]triazolo[1,2-*a*][1,2,4]triazole-1,5-dithiones were screened against *Escherichia coli*, *Staphylococcus aureus*, *Klebsiella pneumoniae*, *Acinetobacter baumannii* and *Pseudomonas aeruginosa*. The antimicrobial activity of the compounds was evaluated according to the guidelines of the National Committee for Clinical Laboratory Standards using agar disk diffusion method [15]. The colony was inoculated in MHBII and incubated to get the culture. The optical density was measured at 600 nm, followed by dilution to achieve ~ 10^6 CFU/mL. The compounds were tested from 64-0.5 mg/L in two-fold serial dilution with 2.5 µL of each concentration added to a well of 96-well microtiter plate. Bacterial suspension of 97.5 µL was then added to each well containing either test compound or control. After the incubation of the plates, MICs were determined.

RESULTS AND DISCUSSION

The synthesis of perhydro[1,2,4]triazolo[1,2-*a*][1,2,4]-triazole-1,5-dithiones was achieved by the condensation of various aldazines and KSCN and Brønsted acid HCl in a polar protic medium (**Scheme-I**). Accordingly, HCl was added to potassium thiocyanate taken in EtOH medium and stirred for 5 min. The reaction mixture was treated with benzaldazine, and refluxed for 20 min. Reactions were then performed to evaluate the amount of HCl required to obtain the optimum yield. At a concentration of 0.1 mL HCl, the reaction took 2 h to complete with a yield of 75%. Increasing the concentration of HCl decreased the reaction time to 20 min (Table-1). With 0.5 mL HCl an optimum yield of 98% was obtained in 20 min. Further increase in concentration did not alter the reaction.

TABLE-1
EFFECT OF DIFFERENT HCl CONCENTRATIONS ON KSCN MEDIATED SYNTHESIS OF TITLED COMPOUND

Amount of HCl (mL)	Reaction time	Yield (%)
0.1	2.0 h	75
0.3	1.5 h	80
0.5	20 min	98
1.0	20 min	98
2.0	20 min	98

Benzaldazine 1.0 mmol, KSCN 1.0 mmol, EtOH 10 mL, Temp. 80 °C.

The reactions were then performed using different solvents to examine their influence (Table-2). Polar aprotic solvents such as DMSO and DMF did not favour the reaction. Among the ether solvents, dioxane gave an appreciable yield, while the reaction with THF was not satisfactory. Whereas the reaction

TABLE-2
EFFECT OF DIFFERENT SOLVENTS ON KSCN MEDIATED SYNTHESIS OF TITLED COMPOUND

Solvent	Time	Yield (%)
Dimethyl sulfoxide	2.0 h	85
Dimethyl formamide	2.0 h	80
Dioxane	1.0 h	90
Tetrahydrofuran	1.5 h	70
Acetonitrile	50 min	82
Ethanol	20 min	98

Benzaldazine 1.0 mmol, KSCN 1.0 mmol, HCl 0.5 mL, Solvent 10 mL, Temperature reflux.

took 2 h to complete in DMSO, the time was reduced to 50 min in acetonitrile. More gratifying was the reaction which completed in 20 min with the polar protic medium ethanol.

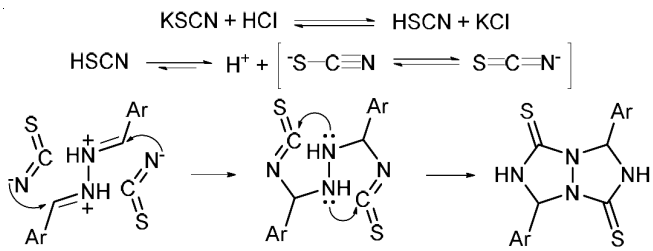
The effect of temperature under the standardized solvent condition was examined by carrying out the reaction at 0, 30, 40, 60 and 80 ºC. The reaction was sluggish under cold conditions and took approximately 18 h to afford 50% yield of the product at room temperature. However, increasing the temperature witnessed an increase in the rate of the reaction (Table-3) and the reaction was complete in 2 h at 60 ºC. As expected, under reflux condition, the reaction rate was the highest and the product was formed in 20 min. A possible enhancement of the rate of the reaction under the conditions discussed thus far could be attributed to the protonation of aldazines. It is quite likely that in presence of HCl, the thiocyanate anion gets transformed to thiocyanic acid in appreciable amounts and a hydrogen bonding would be established between the dissociated protons of the thiocyanic acid and aldazine, setting the stage for a nucleophilic attack by a thiocyanate anion. This would be evident and deemed appropriate from the better kinetics of

TABLE-3
EFFECT OF DIFFERENT TEMPERATURES ON KSCN MEDIATED SYNTHESIS OF TITLED COMPOUND

Temperature (°C)	Reaction time	Yield (%)
0	24 h	No product
30	18 h	50
40	12 h	70
60	2 h	80
80	20 min	98

Benzaldazine 1.0 mmol, KSCN 1.0 mmol, HCl 0.5 mL, EtOH 10 mL

the reaction. A subsequent attack by the second thiocyanate anion on the other half of protonated aldazine would pave the way for the formation of bicyclic product (**Scheme-II**). It is also worth to emphasize that the reaction was not catalytic in nature under the concentration of HCl (11.6 N) employed. The



Scheme-II: Formation of 3,7-diaryltetrahydro-1*H*,5*H*-[1,2,4]triazolo[1,2-*a*][1,2,4]triazole-1,5-dithiones

reaction mechanism substantiates the formation of a protonated intermediate, which facilitates an accelerated nucleophilic addition leading to products in less time and good yields.

Antibacterial activity: The synthesized 3,7-*bis*(4-chloro phenyl)tetrahydro-1*H*,5*H*-[1,2,4]triazolo[1,2-*a*][1,2,4]-triazole-1,5-dithione (**3c**) showed an appreciable activity (MIC 4 µg/mL) against *Staphylococcus aureus* (compared with levofloxacin, MIC 0.25 µg/mL).

Conclusion

3,7-Diaryltetrahydro-1*H*,5*H*-[1,2,4]triazolo[1,2-*a*][1,2,4]-triazole-1,5-dithiones were synthesized by a highly efficient and ecofriendly procedure from aldazines using KSCN and HCl in an ethanolic medium. The products were obtained in excellent yields and short duration of time, along with the advantage of ease of isolation by precipitation. Among the synthesized compounds, 3,7-*bis*(4-chlorophenyl)tetrahydro-1*H*,5*H*-[1,2,4]triazolo[1,2-*a*][1,2,4]triazole-1,5-dithione (**3c**) demonstrated biologically activity against *Staphylococcus aureus*.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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