Designed Synthesis of 2-(2,4-dinitrophenyl)-8,8-Dimethyl-5-Phenyl-5,7,8,9-Tetrahydro-6H-[1,3,4] Thiadiazolo[2,3-B] Quinazolin-6-One Promoted by $ZrOCl_2$

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Abstract — A simple and an efficient general procedure have been enhanced for the synthesis of 5-phenyl-5,7,8,9-tetrahydro-6H-[1,3,4] thiadiazolo[2,3-b]quinazolin-6-onefused analogous and biological properties. This analogous can be synthesized from compound (3) with dimedone and arylaldhyde in the presence of Lews acid catalyst ($ZrOCl_2$) and the compound (3) can be prepared by the 2,4-dinitrobenzoic acid with thiosemicarbaide in the presence protic acid medium. All products have been characterized by ^{1}H NMR and ^{13}C NMR spectroscopy, and mass spectrometry. This method provides versatile advantages of organo-catalyst such as economical, nontoxic, highly stable, readily and easily available as well as solvent-free reaction conditions.

Keywords: 5-(4-Nitrophenyl)-1, 3, 4-Thiadiazol-2-Amine, Dimedone, Aryl Aldehyde, $ZrOCl_2$ Bioevluation

I. INTRODUCTION

Organic compounds with a ring structure that includes atoms other than carbon, such as nitrogen, sulfur, or oxygen, are known as heterocyclic compounds. Because of their intriguing physiological characteristics [1], a number of five-membered aromatic systems with three heteroatoms arranged symmetrically have been investigated. These systems include azole, pyrole, thiazole, thiadiazole, oxadiazole, triazene, and others. They also display a wide range of biological activities.

Amongst five-membered sulphur-nitrogen heterocycles 1, 3, 2-dithiazoles have attracted the largest attention due to their important physical and biological properties Thiadiazole is a heterocyclic compound featuring both two-nitrogen atom and one sulfur atom as part of the aromatic five membered rings. Thiadiazole and related compounds are called 1, 3, 4-thiadiazole (two nitrogen and one other hetero atom in a five-membered ring). The others four isomeric forms of thiadiazole are occurred in nature as 1, 2, 3-thiadiazole; 1,2,5-thiadiazole; 1,2,4-thiadiazole. 1, 3, 4-thiadiazoles are important compounds.

A large number of thiadiazoles have been patented in the medical field for the treatment of a wide variety of diseases and some of them have become commercial products. A large number of thiadiazoles have been patented in the medical field for the treatment of a wide variety of diseases and some of them have become commercial products compounds in medicinal chemistry because of its therapeutic values. Due to presence of –N=C-S moiety in its structure1,3,4-thiadiazoles exhibit various biological activities[2-5],antimicrobial activities[6-12], antihyperlipidemic agents[13], anticancer agents[14-18], Analgesic, Anti-inflammatory, Anti-Bacterial and AntiTubercular Activity[19], antimicrobial and antioxidant agents[20], anticorrosion[21], According to previous literature reputed by the use of various catalysts such as Green Catalytic Approaches [22], iodine-catalyzed [23], Ionic Liquid [24], Hypervalent Iodine (III) [25], I2/CuCl2 [26], HI/DMSO[27], TBAI[28], Mn(II) (29)

In continuation of our on-going research towards the development of novel procedure for organic transformation by using alternative protocols and by considering the significance of 5-(4-nitrophenyl)-1,3,4-thiadiazol-2-amine derivatives, herein we wish to report, a simple, efficient procedure for the synthesis of 5-(4-nitrophenyl)-1,3,4-thiadiazol-2-amine derivatives using inexpensive $ZrOCl_2$ as organo-catalyst under ethanol solvent conditions.

II. MATERIALS AND METHODS

A. *Experimental*

All the chemicals and solvents were supplied by S. D. Fine and Sigma-Aldrich and Spectrochem Pvt. Ltd. The solvents were distilled and dried before use as required. The reactions were monitored with the help of thin-layer chromatography using precoated aluminium sheets silica gel 0.2 mm layer thickness by using solvent systems Ethylacetae: hexane (3:7). The spots were visualized under UV lamp and the melting points of the newly synthesized compounds were estimated and are uncorrected using one end open capillary tubes. Both ^{1}H NMR ($CDCl_3$) and ^{13}C NMR spectra of the synthesized compounds were performed with Bruker Avance-II400 NMR spectrometer operating at 100 MHz. and Chandigarh. Chemical shifts were measured relative to internal standard TMS and are reported in (δ ppm). Mass spectra of the synthesized compounds were measured at MAT 120.

B. *(4-NITROPHENYL)-1,3,4-THIADIAZOL-2-AMINE(3):*

Take dry and clean four necks RBF. The mixture of 4-nitro benzoic acid (10mmol) and semithiocarbazide (10mmol) dissolved in the ethanol (20mL) and few drops of phosphoric acid (H_3PO_4) into RBF at room temperature which is also fitted on the magnetic stirrer containing hot plate. The reaction mixture continuous carried the reaction for 4 hrs. at 65^{0}C . The progress of the reaction was checked by the TLC (EtOAc: n-hexane = 4:6). After all the reactants were consumed and then cooled the reaction mixture at RT. The crude dissolved in ethyl acetate and washed with as saturated solution of sodium bicarbonate and separated the ethyl acetate layer and also washed with water separated the organic layer. The organic layer can be distilled off under vacuums and solid compound obtained.

Palered, Yueld-91%, M.P-154.156^{0}C;^{1}HNMR(400MHz,$CDCl_3$)δppm:8.412-8.094(m,4H), 7.245 (s,2H,NH_2);^{13}CNMR(100MHz,$CDCl_3$)δppm:175.23,160.35, 148.95, 139.87, 128.77, 127.65'; Molecular formula:

$C_8H_6N_4O_2S$; Molecular weight: 226.19 (M); Elemental analysis: C-43.24,H-2.27,N-25.21;

C. GENERAL PROCEDURE FOR THE SYNTHESIS OF 2-(2,4-DINITROPHENYL)-8,8-DIMETHYL-5-PHENYL-5,7,8,9-TETRAHYDRO-6H-[1,3,4]THIADIAZOLO[2,3-B] QUINAZOLIN-6-ONE

A mixture of 5-(4-nitrophenyl)-1,3,4-thiadiazol-2-amine(10mmol),5,5-dimethylcyclo hexane-1,3-dione (dimedone) (10mmole) with substituted aromatic aldehyde (10mmol) and molecular iodine (10 mol%) in ethanol was refluxed at 80^0C for 3 h. The progress of reaction monitored by TLC and kept at room temperature. The solid needles like crystal are obtained in round bottom flask and filter over filter paper. The product was purified by recrystallization from ethanol.

1) Compound (6a):

Pale red; Yield-82%; m.p-208-210^0C ; 1H NMR (400 MHz, $CDCl_3$) δppm: 9.052(s, Ar-H), 8.623-8.062(m,2H), 7.326-7.274(M,5H,Ar-H), 4.317(s,1H,-CH-),2.114(s,2H), 1.662(s,2H), 0.926(s,6H,$(CH_3)_2$);^{13}C NMR(100 MHz,$CDCl_3$) δppm: 197.92, 158.06, 153.26, 147.76, 146.06, 141.07, 138.96, 132.03, 131.96, 129.62, 129.16, 128.76, 127.07, 127.62, 119.67, 63.27, 46.62, 38.76, 30.82, 27.07;Molecular formula : $C_{23}H_{19}N_5O_5S$, Molecular weight : 476.92(M-H);Elemental analysis : Calculated; C-57.85,H-4.01,N-14.67; Obtained: C-57.80, H-4.00, N-14.75

2) COMPOUND (6b):

Pale red; Yield-85%; m.p-214-216^0C ; ^{1}HNMR (400 MHZ,$CDCl_3$) δppm: 9.762(s,1H), 8.462(s,1H,AR-H),8.316-8.036(m,2H,Ar-H),6.916-6.726(m,4H,Ar-H),4.365(s,1H,-CH), 2.106 (s,2H,-CH),1.636(s,2H,-CH),0.956 (s,6H,-CH_2);^{13}C NMR (100 MHZ,$CDCl_3$) δppm: 193.36, 159.06, 153.6, 148.36, 145.62, 142.02, 135.39, 133.36, 131.26, 130.02, 129.72, 128.32, 127.32, 126.02, 121.06, 63.32, 49.62,38.72, 30.80, 27.62; Molecular formula :$C_{23}H_{19}N_5O_6S$, Molecular weight : 494.63 (M+H) ;Elemental analysis : Calculated C-55.98, H-3.88, N-14.19; Obtained: C-55.90, H-3.86, N-14.27.

3) Compound (6c):

Pale red; Yield-88%; m.p-224-226^0C; ^{1}H NMR (400 MHz, $CDCl_3$) δppm: 8.864(s, 1H,AR-H),8.546-8.336(m,2H,AR-H),6.964-6.762(m,2H),4.467(s,1H,-CH),3.712(s,6H),3.646(s, 3H) ;^{13}C NMR (100 MHz,$CDCl_3$) δppm: 192.68, 157.71, 150.64, 148.76, 145.66, 141.76, 138.36, 135.24, 133.34, 131.62, 130.07, 129.64, 121.16, 120.02, 63.33, 49.76, 38.78, 30.56,27.32,26.99;Molecularformula;$C_{26}H_{25}N_5O_8S$,;MolecularWeight:568.79(M+H); Elemental analysis : Calculated: C-55.02,H-4.44, N-12.35;Obtained: C-54.96, H-4.42, N-12.45

4) Compound (6d):

Palered;Yield-85%;m.p-224-226^0C;^{1}HNMR(400MHz,$CDCl_3$)δppm:9.062(s,8.696-8.556 (m,2H),7.256-7.036(m,4H),4.316(s,-CH-),2.096(s,2H),1.962(s,2H),0.967(s,6H);^{13}CNMR (100MHz,$CDCl_3$) δppm: 196.71, 158.04, 150.26, 148.37, 146.66, 143.02, 138.37, 132.66, 131.02, 130.37,129.28, 128.72, 128.36, 127.62, 119.19, 64.62, 49.19, 37.36, 30.27, 28.72,26.06;Molecularformula: $C_{23}H_{18}ClN_5O_5S$, Molecular weight : 513.38(M+2);Elemental analysis : Calculated; C-53.96, H-3.54, N-13.68; Obtained: C-53.90, H-3.53, N-13.76.

5) Compound (6e):

Pale red; Yield-84%; m.p-225-227^0C; ^{1}HNMR(400MHz,$CDCl_3$)δppm:8.916(s,1H), 8.713-8.562(t,2H,m,ar), 7.817-7.567(4H,m,ar), 4.417(s,1H,-C4), 2.097(s,2H), 1.762(s,2H), 0.915(6H,s);^{13}C NMR (100 MHz,$CDCl_3$) δppm: 195.37, 159.37, 152.68, 148.76, 146.07, 141.18, 139.09, 131.18, 130.96, 129.46, 128.92, 128.41, 127.77, 126.66, 121.78, 63.46, 49.17, 38.03, 30.87, 26.96; Molecular formula :$C_{23}H_{18}N_5O_5S$, Molecular weight :557.21(M+2);Elemental analysis : Calculated ;C– 49.55, H – 3.26, N -12.59; Obtained: C-49.48, H-3.25, N-12.65

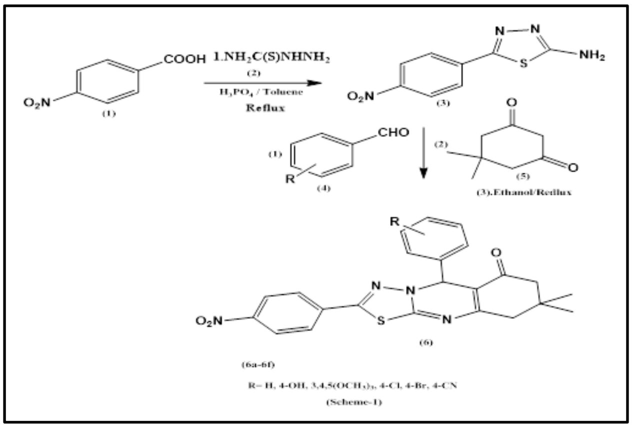
6) Compound (6f):

Pale red; Yield-86%; m.p-231-233^0C;^{1}H NMR (400 MHz,$CDCl_3$) δppm: 8.743(s,1H,ar), 8.356-8.047(m,2H,Ar-H),7.618-7.317(m,4H,Ar-H), 4.267(s,-CH), 2.046(2H,s), 1.734(s,2H), 0.966(s,6H$(CH_3)_2$);^{13}C NMR (100 MHz,$CDCl_3$) δppm: 197.92, 161.09, 155.26, 149.38, 145.07, 143.26, 142.19, 131.16, 130.07, 129.86, 128.75, 128.18, 127.02, 121.77, 118.06, 115.73, 62.46, 50.71, 38.47, 30.57, 28.04,17.15;Molecularformula: $C_{24}H_{18}N_6O_5S$, Molecular weight : 503.34(M+H);Elemental analysis : Calculated; C-57.37, H-3.61, N-16.70; Obtained: C-57.31, H-3.60, N-16.77

III. RESULTS AND DISCUSSION:

We have discussed the optimization of reaction conditions. We focus on the influence of solvent, temperature, reaction time and catalytic amount of molecular $ZrOCl_2$ iodine in model reaction. Therefore, the significant systematic optimization with ethanol, methanol, dichloromethane, acetonitrile could be discussed. It indicated that the solvent has significant effect in yield and reaction time. It excellent result was found in reaction carried out in acetonitrile. We have carried out the model reaction using different stoichiometric amount of catalyst and catalyst screening results are summarized in. It was found that excellent yield was able by using 5 mol% of molecular iodine in acetonitrile refluxed at 80°C for 4h. Further increase in catalyst quantity beyond 5 mol% did not increase the yield of product extremely.

The persuade of investigated with different parameters on the model reaction, we revolved our consideration towards the synthesis of thiadiazolo [2,3-b]quinazolin-6-(7H)-one derivatives from 2-amino5-phenyl 1,3,4-thiadiazole, 5,5-dimethyl cyclohexane-1,3-done (dimedone) and a variety of substituted aromatic aldehydes as shown scheme-1. Firstly, we gone through the 2-amino-5-phenyl 1,3,4-thiadiazole secured with both electron- donating and electron- withdrawing groups and it was gratifying notified respective product obtained excellent yield. We examine the scope of series of aldehydes tethered with both electron-poor and electron-rich groups. It was heart-warming to observe that in both cases, the corresponding products were obtained with excellent yields. All synthesized products well characterized by spectroscopic techniques.



The reaction condition of these titled was optimized at different catalyst, different loaded the catalyst and the various solvent are applied. The height yield of the compounds obtained in presence of zinc oxide (ZnO) catalyst than oxidative related catalyst such as titanium dioxide (TiO_2), zirconium oxychloride ($ZrOCl_2$), Iodine whereas different amount of catalyst utilized during the reaction (Table-I).

Entry	Catalyst	Time (hrs)	Yield (%)
1	ZnO	4	51
2	TiO_2	4	62
3	$ZrOCl_2$	4	88
4	I_2	4	72

Table I: The optimization of the various for titled synthesis (6c):

The various solvents were applied during the reaction that were evaluated (DMF, Isopropanol acetonitrile, ethanol, methanol, cyclohexane) in the model reaction. It was found to be the best medium for the reaction, with 88% product yield and was therefore used as the solvent for subsequent reactions on the merits of higher yield, green nature and easy work-up.

Entry	Catalyst	Time (hrs)	Yield (%)
1	DMF	4	46
2	IPA	4	58
3	CH_3CN	4	51
4	EtOH	4	88
5	MeOH	4	68

Table II: The Optimization of the solvent on titled derivatives $ZrOCl_2$ (6c):

Various spectral analytical techniques such as ^{1}H-NMR, 13CNMR and mass spectra were employed titled derivatives of chemical structure of quinazolinones derivatives. ^{1}H-NMR exhibited multiple of six protons at δ (ppm) 3.712 of methoxy group, aromatic protons multiple for at δ (ppm) 8.743-7.036 and singlet protons δ (ppm) 0.926-0.967 respectively. Mass spectra revealed molecular ion (M+2 for bromo and Chloro derivatives peak in satisfactory intensity and 13CNMR of keto group appears at 197.21.

IV. CONCLUSIONS:

In conclusion, we have approached a convenient protocol for the synthesis titled derivatives (6a-6f) starting material from 5-(4-Nitrophenyl)-1,3,4-Thiadiazol-2-Amine with aromatic aldehyde in the presence of transition metal halides good yield. The scope of the catalyst and advantages for the synthesis of desired derivatives.

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CONFLICT OF INTEREST

The authors declare no conflict of interest

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