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Molecular Iodine Mediated One-Pot Three-Component Reaction for Synthesis of Thiadiazolo [2, 3-*b*] Quinozolin-6(7H)-ones and their Antimicrobial Activity

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ABSTRACT: Thiadiazolo [2,3-b] quinazolinone derivatives are significantly synthesized by one-pot manner. Herein, we describe a three-component reaction system of 5-aryl-1,3,4-thiadiazolo-2amine, dimedone moiety and substituted aromatic aldehyde with a massive addition of molecular iodine. Interestingly, in present work, we focused on more efficient and convenient synthesis, such as easy isolation of compound, mild conditions and excellent yield. All synthesized compounds are characterized by spectral analysis and further ahead for their antimicrobial activity.

KEYWORDS: Dimedone, 1,3,4-thiadiazole, Aromatic aldehyde, Acetonitrile, Molecular iodine

■ Introduction

Multicomponent reaction (MCR) is a three or more components system which is accessible to react in the single reaction media to furnish desired product. Multicomponent reactions (MCRs) [1] are widely used in organic synthesis and computational chemistry for their molecular diversity and high economic efficiency. It minimizes the number of steps and having lesser time of synthesis in reaction and it is comparable with green chemistry [2]. The N, S-containing fused heterocyclic compound play an important role in pharmaceutical due to their potent physiological properties [3]. Interestingly benzothiazole[4-5], quinazolines [6] and 1,3,4-thiadiazole derivatives are involved in biological application. These compounds have a crucial role or widely used in biological and pharmacological properties such as antifungal [7], anticancer [8], antibacterial [9], antiviral [10], anti-inflammatory [11] and anti-HIV [12]. Few fused thiadiazolo quinazoline derivatives having convenient biological activities shown in **figure-1**, such as 5-(4-bromo phenyl)-5H [1,3,4] thiadiazolo [2,3-*b*] quinazoline-2-sulfonamide enhance anticancer activity (I), 6,7,8,9-tetrahydro 5H [1,3,4] thiadiazolo [2,3-*b*] quinazoline-5-one reveal anti-inflammatory activity(II) and 5-(4-bromophenyl)-5H -[1,3,4] thiadiazole [2,3-*b*] quinazoline-2-sulfonamide derivative(III) exposed antifungal activity.

In reports mention, thiadiazolo [2,3-*b*] quinazolin-6-(7H) one was reported in acidic tetrabutyl ammonium hydrogen sulphate (Bu_4NHSO_4) [13] acts as phase transfer catalyst in *p*-toluenesulfonic acid-mediated three-component reaction in water mediated for the synthesis of novel thiadiazolo-[2,3-b] quinazolin-6(7H)-one[14].

In literature survey, we observed that lewis acids are used for various organic transformations due to their non-metallic, nontoxic character and easy availability in nature. Interestingly, molecular iodine will be one of the best choice catalysts in the construction of various multicomponent reactions. In the present work, we synthesized biologically active compounds such as thiadiazolo [2,3-b] quinazolin-6-(7H) one derivative

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via the reaction of 2-amino-5-phenyl 1,3,4-thiadiazole(1a), with substituted aromatic aldehydes (2a-x) and dimedone (3) with molecular iodine in acetonitrile refluxed at 80 ^{0}C (Scheme 1). According to literature survey we developed few molecules containing N & S fused heterocycles which is more feasible in antimicrobial activity.

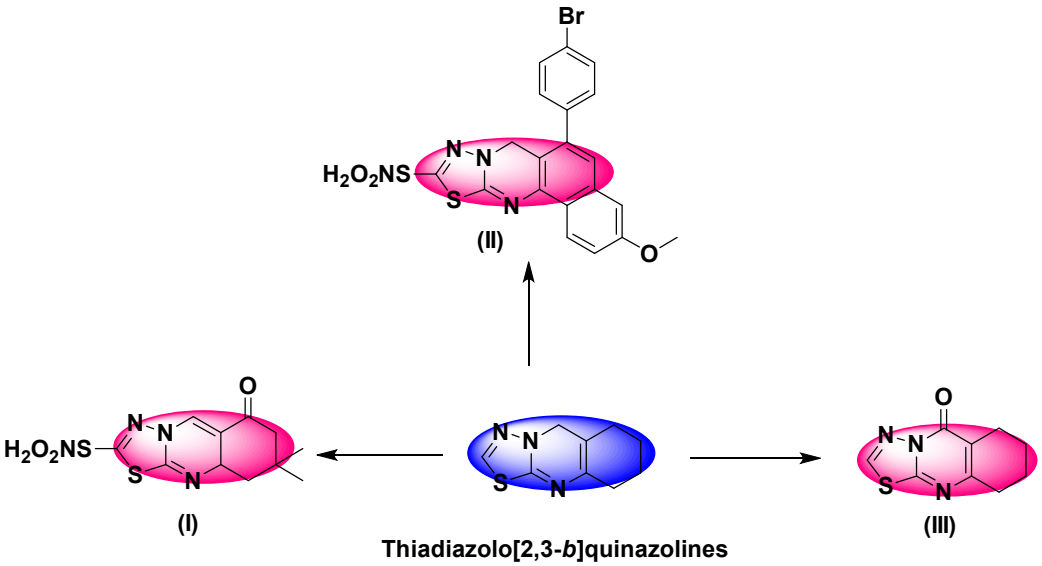
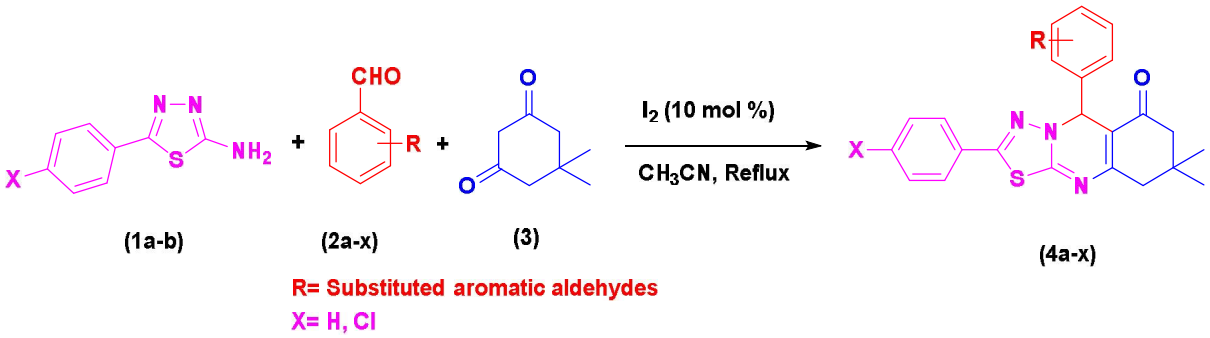


Figure 1. Biological active some structures of thiadiazolo quinazoline derivatives.

Results and Discussion

In this report we discuss the synthesis of different thiadiazolo-quinazoline analogous in general reaction and synthesis showed in one pot manner. Initially we started the synthesis of thiadiazolo [2,3-*b*] quinazolin-6-(7H)-one, from 2-amino-5-phenyl 1,3,4-thiadiazole (1a), dimedone (3) it was refluxed at independently in acetonitrile and iodine (10 mol%) with different substituted benzaldehydes (2a) (**Scheme 1**).



Scheme1. Synthesis of thiadiazolo[2,3-b]quinazoline-6-(7H)-one.

We have discussed the optimization of reaction conditions. We focus on the influence of solvent, temperature, reaction time and catalytic amount of molecular iodine in model reaction (**Table 1**). Further, more systematic optimization with dichloromethane, acetonitrile (**Table1, Entry 2-9**). It indicated that the solvent has significant effect in yield and reaction time. It excellent result was found in reaction carried out

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in acetonitrile (**Table 1, Entry 9**). We have carried out the model reaction using different stoichiometric amount of catalyst and catalyst screening results are summarized in (**Table 2)**. It was found that excellent yield was able by using 10 mol% of molecular iodine in acetonitrile refluxed at 80°C for 3h (**Table 2, Entry 5**). Further increase in catalyst quantity beyond 10 mol% did not increase the yield of product extremely (**Table 2, Entry 6**).

Table 1. Optimization of reaction for the synthesis of thiadiazolo [2,3-*b*] quinazolin-6-(7H)-one (**4a**). [a]

Entry	Catalyst (mol %)	Solvent	Temperature (°C)	Time (h)	Yield (%) [b]
1	-	-	r.t.	24	No
2	I_2 (10)	Toluene	r.t.	24	10
3	I_2(10)	THF	r.t.	24	18
4	I_2(10)	DMF	r.t.	24	30
5	I_2(10)	H_2O	r.t.	24	38
6	I_2(10)	CH_3OH	r.t.	24	35
7	I_2(10)	CH_2Cl_2	r.t.	20	47
8	I_2(10)	CH_3CN	r.t.	12	65
9	I_2(10)	CH_3CN	80	03	80
10	I_2(10)	-	80	12	32

[a]***Reaction conditions:*** 2-amino-5-phenyl 1,3,4-thiadiazole (1 mmol), dimedone (1 mmol) with substituted benzaldehydes (1 mmol) and Molecular iodine in acetonitrile (5mL) were refluxed at 80°C.[b] Isolated yields. nd = not determined.

Table 2: Optimization Study for the amount of Molecular iodine.[a]

Entry	Amount of Catalyst (mol %)	Temperature (°C)	Time (h)	Yield % [b]
1	-	-	r.t.	24
2	I_2 (10)	Toluene	r.t.	24
3	I_2(10)	THF	r.t.	24
4	I_2(10)	DMF	r.t.	24
5	I_2(10)	H_2O	r.t.	24
6	I_2(10)	CH_3OH	r.t.	24

[a]***Reaction conditions:*** 2-amino-5-phenyl 1,3,4-thiadiazole (1 mmol), dimedone (1 mmol) with substituted benzaldehydes (1 mmol) and Molecular iodine in acetonitrile (5mL) were refluxed at 80°C.[b] Isolated yields

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-amino-5-phenyl 1,3,4-thiadiazole, 5,5-dimethyl cyclohexane-1,3-done (dimedone) and a variety of substituted aromatic aldehydes as summarized in Table 3. 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 heartwarming to observe that in both cases, the

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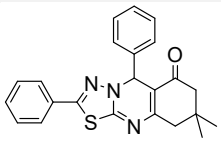
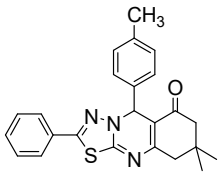
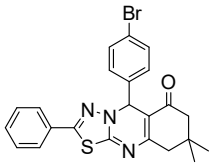
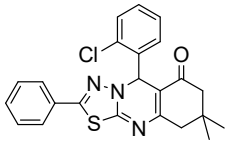
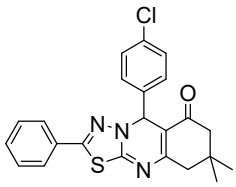
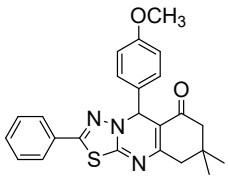
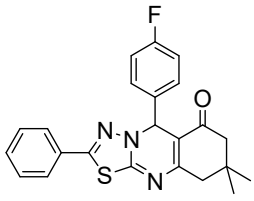
corresponding products were obtained with excellent yields (**Table 3**). All synthesized products well characterized by spectroscopic techniques.

Table 3. Three component condensation of 2-amino-5-phenyl 1,3,4-thiadiazole / 2-amino-5-(4'-chlorophenyl)-1, 3, 4-thiadiazole (1a-b) dimedone (3) with substituted benzaldehydes (2a-x) [a]

Entry	Compound (code)	Product (4a-x)	Time (h)	Yield (%)[b]	M.P. (°C)
1	**4a**		2.5	70	197-199
2	**4b**		2.0	80	204-206
3	**4c**		3.0	80	206-208
4	**4d**		3.5	78	203-205
5	**4e**		4.0	80	214-216
6	**4f**		2.0	78	184-186
7	**4g**		4.5	62	208-210

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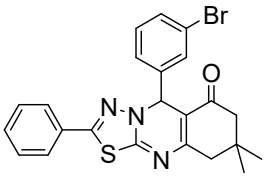
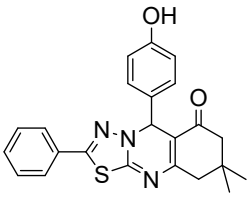
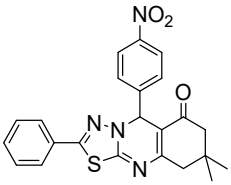
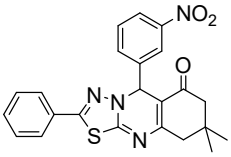
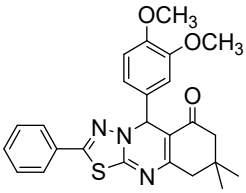
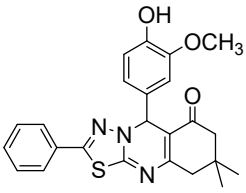
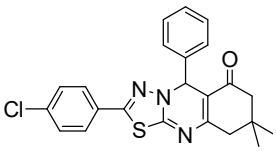
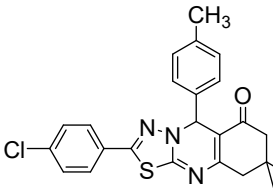
8	**4h**		3.0	66	218-220
9	**4i**		2.5	73	211-113
10	**4j**		4.5	59	201-203
11	**4k**		3.0	60	207-210
12	**4l**		3.5	66	192-194
13	**4m**		2.5	68	213-215
14	**4n**		3.5	78	205-207
15	**4o**		3.0	80	201-203

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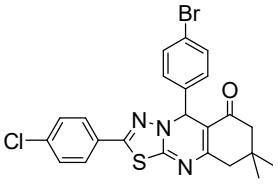
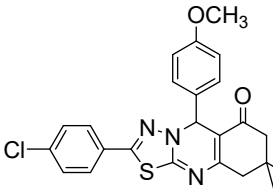
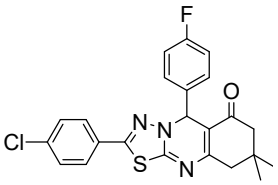
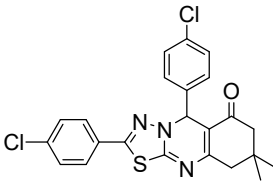
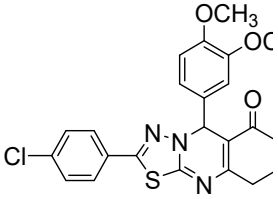
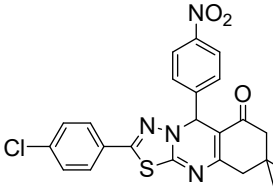
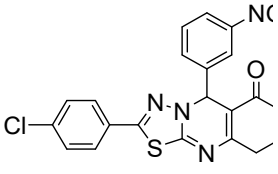
16	**4p**		2.5	80	274-276
17	**4q**		3.5	72	217-219
18	**4r**		4.5	57	222-225
19	**4s**		3.5	67	197-199
20	**4t**		3.0	69	190-192
21	**4u**		3.5	58	210-212
22	**4v**		4.5	59	225-227

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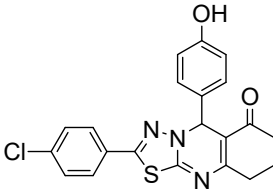
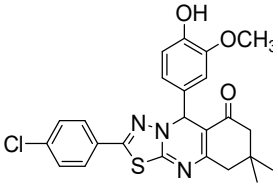
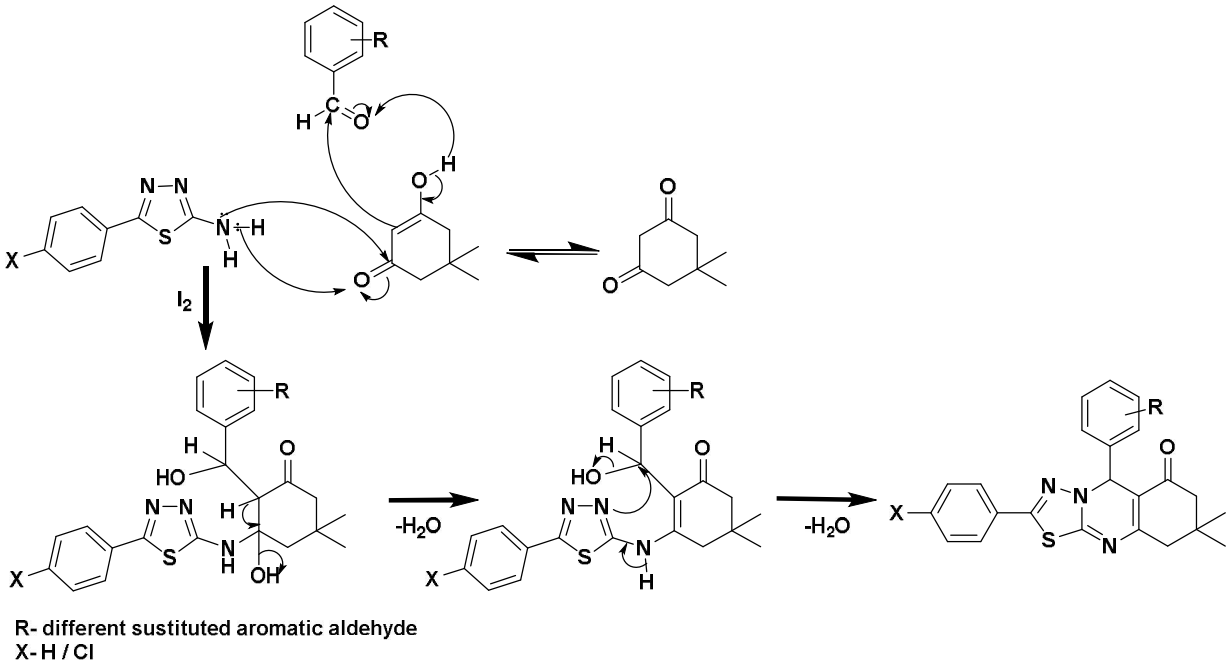
23	**4w**		2.5	69	217-219
24	**4x**		3.0	65	229-231

[a]***Reaction conditions:*** (**1**a-b) (1 mmol), (**3**) (1 mmol) with (**2**a-x) (1 mmol) and Molecular iodine in acetonitrile (5 mL) were refluxed at 80°C.[b] Isolated yields.

The plausible mechanism of molecular iodine synthesis of thiadiazolo[2,3-*b*]quinazolin-6-(7H)-one from 2-amino-5- phenyl 1,3,4-thiadiazol, 5,5-dimethyl cyclohexane-1,3-done (dimedone) with substituted benzaldehydes throughout Knovenagel-Michael addition condensation followed dehydration and cyclization is proposed as below, (**Scheme 2).**



Scheme 2. Plausible mechanism for the synthesis of thiadiazolo[2,3-b]quinazoline-6-(7H)-one.

Antimicrobial Activity

The synthesized thiadiazole derivative were screened for their antibacterial activity against Gram-positive bacteria (*S. aureus*) and Gram-negative bacteria,(*E. coli and P. Vulgaris*) using the Penicillin and Streptomycin as standard drugs. All synthesized thiadiazole derivative are

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screened for their biological evaluation like antibacterial activity against Gram-positive bacteria (S-aureus) and gram negative bacteria as compounds **4e, 4f, and 4i** showed good antibacterial activity against *P.Vulgaris*, distinguish with penicillin and streptomycin. The compounds **4b, 4e, 4p, 4t,** are having good antibacterial activity against *S. aureus* as compared to penicillin and streptomycin. The compounds **4c, 4f,** showed good antibacterial activity against *E.coli* as compared to penicillin and streptomycin. The compounds **4e, 4l, 4i, 4f, 4h, 4o, 4p,** showed good antibacterial activity against *P. Vulgaris* as compared to penicillin and streptomycin.

Table 4. Antibacterial Activity & Antifungal Activity of tested compounds (4a-x)

Entry	Compound Code	R-	X-	Antibacterial Activity Zone of inhibition* (mm) (activity index)std			Antifungal Activity Zone of inhibition* (mm) (activity index)std		
				S. Aureus	*E. Coli*	*P. Vulgaris*	*A. fumigatus*	*A. niger*	*C. neoformans*
1	**4a**	Ar-H	H	ND	10	ND	12	ND	ND
2	**4b**	4-CH_3	H	ND	10	07	13	ND	ND
3	**4c**	4-Br	H	ND	**13**	ND	12	ND	ND
4	**4d**	4-Cl	H	ND	12	10	ND	**12**	10
5	**4e**	2-Cl	H	11	11	**16**	10	ND	ND
6	**4f**	4-OCH_3	H	ND	**13**	13	12	**12**	**12**
7	**4g**	4-F	H	ND	ND	12	12	ND	ND
8	**4h**	3-Br	H	ND	**13**	13	ND	10	ND
9	**4i**	4-OH	H	ND	Nil	12	12	ND	ND
10	**4j**	4-NO_2	H	09	07	11	10	ND	ND
11	**4k**	3-NO_2	H	ND	ND	10	ND	ND	ND
12	**4l**	3,4-di OCH_3	H	ND	ND	13	11	ND	ND
13	**4m**	3OCH_3,4OH	H	11	**13**	10	11	11	10
14	**4n**	Ar-H	Cl	10	10	**16**	**16**	10	10
15	**4o**	4-CH_3	Cl	11	10	12	11	10	**12**
16	**4p**	4-Br	Cl	11	ND	ND	10	**12**	10
17	**4q**	4-OCH_3	Cl	10	10	11	10	ND	ND
18	**4r**	4-F	Cl	10	10	10	**24**	ND	ND
19	**4s**	4-Cl	Cl	11	11	Nil	11	ND	ND
20	**4t**	3,4-di -OCH_3	Cl	11	11	12	**20**	12	ND
21	**4u**	4-NO_2	Cl	10	10	12	09	10	ND
22	**4v**	3-NO_2	Cl	10	ND	ND	09	10	ND
23	**4w**	4-OH	Cl	**12**	10	10	12	ND	11
24	**4x**	3OCH_3,4OH	Cl	**12**	11	12	14	ND	10

ND: Not detected

The synthesized thiadiazole derivative was screened for their antifungal activity against *A.niger, A.fumigatus and C. neoformans* using the *Nystatin* as standard drugs. The compounds **4e, 4l and 4h)**

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showed good antibacterial activity against *A.niger*, after distingwishes with *Nystatin*. The **compounds 4b, 4e, 4p, 4t,** possessing good antibacterial activity against *A.fumigatus* distingwishes with *Nystatin*. The compounds **4c, 4f,** showed good antibacterial activity against *E.coli* as compared to penicillin and streptomycin. The compounds **4e, 4l, 4i, 4f, 4n, 4o, 4p** had a good antibacterial activity against *C. neoformans* as compared to Nystatin (**Table 4**).

■ Summary and Outlook

All thiadiazolo-quinazoline derivatives are synthesized in one pot manner via cyclo-condensation reaction. Herein we used the precursor like substituent thiadiazole, dimedone and substituted aromatic aldehyde of reaction is mediated with very chief and easily available catalyst molecular iodine in acetonitrile. All reaction offered with excellent yield, also it is non-chromatographic purification & easy for isolation & these compounds screened for Antimicrobial & Antifungal activity.

■ Experimental

All the chemical ware purchased from Sigma-Aldrich without any purification solvent from sd fine chemicals. All the reaction was monitored by TLC. NMR spectra were recorded on Bruker Avance-400 MHz spectrometer in DMSO-d6 as a solvent with tetramethyl silane (TMS) as an internal standard. Mass spectrometer recorded on Shimadzu QP 2010 GCMS. IR spectra were recorded on Shimadzu IR-470 spectrophotometer using KBr discs.

General Procedure for the Synthesis of 5-substituted derivative of 2,5-diphenyl-8,9-dihydro-5H-[1,3,4] thiadiazolo [2,3-*b*] quinazolin-6(7*H*)-one (4a-x):

A mixture of 2-amino-5-phenyl 1,3,4-thiadiazole (**1 a-b** ,1 mmol), 5,5-dimethyl cyclohexane-1,3-dione or dimedone (**3**, 1mmole) with substituted aromatic aldehyde (**2a-x** ,1mmol) and molecular iodine (10 mol%) in acetonitrile was refluxed at 80 ^{0}C 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 recrystalization from ethanol.

Spectral Analysis of Some Compounds (4a-x):

8,9-dihdro-8,8-dimethyl-2-phenyl-5-p-tolyl-5*H* [1,3,4] thiadiazolo [2,3-*b*] quinazolin-6(7H)-one (4b): Yellow solid (80%), M.P.= 204-206 ^{0}C.^{1}H-NMR (DMSO, 300 MHz) δ (ppm) = 0.96 (s, 3H); 1.03 (s, 3H); 2.04-2.10 (d, 1H); 2.19 (s, 1H); 2.23 (s, 3H); 2.46 (s, 2H); 6.32 (s, 1H); 7.12-7.14 (d, 2H); 7.20-7.23 (d, 2H); 7.48-7.71 (m, 5H). IR (KBr) υ$^-$ =2927, 2889, 1642, 1589, 1504, 1373 cm^{-1}. MS (EI): 402 (m+H)$^+$.

5-(4-bromophenyl) 8,9-dihdro-8,8-dimethyl-2-phenyl-5*H* [1,3,4] thiadiazolo [2,3-*b*] quinazolin-6(7H)-one (4c): Yellow solid (80%), M.P.= 206-208 ^{0}C.^{1}H-NMR (DMSO, 400 MHz) *δ* (ppm) = 0.95 (s, 3H); 1.03 (s, 3H); 2.06-2.11 (d, 1H); 2.19-2.25 (d, 1H); 2.46 (s, 2H); 6.38 (s, 1H); 7.28-7.31 (d, 2H); 7.69-7.73 (d, 2H); 7.48-7.56 (m, 5H).^{13}C-NMR (DMSO, 100 MHz): 27.08,28.66, 32.24, 44.49, 59.46, 126.34, 128.17, 129.29, 129.43, 131.52, 131.93, 142.00, 154.00, 161.10, 194.19 ppm.IR (KBr) υ =3058, 2970, 2889, 1643, 1585, 1504, 1373 cm^{-1}. MS(EI): 466 (m+H)$^+$, 468 (m+H+2)$^+$.

5-(2-chlorophenyl) 8,9-dihdro-8,8-dimethyl-2-phenyl-5*H* [1,3,4] thiadiazolo [2,3-*b*] quinazolin-6(7H)-one(4d): Yellow solid (78%), M.P.= 203-205 ^{0}C. ^{1}H-NMR (DMSO, 300 MHz) *δ* (ppm) = 0.95 (s, 3H); 1.04 (s, 3H); 2.02-2.07 (d, 1H); 2.18-2.23 (d, 1H); 2.47 (s, 2H); 6.77 (s, 1H); 7.26-7.36 (d, 2H); 7.41-7.55 (m, 5H); 7.64-7.67 (d, 2H). IR (KBr) υ =2950, 2866, 1631, 1585, 1500, 1377 cm^{-1}. MS (EI): 422 (m)$^+$, 424 (m+2)$^+$.

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5-(4-chlorophenyl) 8,9-dihdro-8,8-dimethyl-2-phenyl-5-p-tolyl-5*H* [1,3,4] thiadiazolo [2,3-*b*] quinazolin-6 (7H)-one(4e): Yellow solid (80 %), M.P.= 214-216 ^{0}C. ^{1}H-NMR (DMSO, 300 MHz) δ (ppm) = 0.95 (s, 3H); 1.03 (s, 3H); 2.04-2.10 (d, 1H); 2.19 (d, 1H); 2.23 (s, 2H); 2.46 (s,3H); 6.31 (s, 1H); 7.12-7.14 (d, 2H); 7.20-7.22 (d, 2H); 7.56-7.59 (d, 2H); 7.70-7.73 (d, 2H). ^{13}C-NMR (DMSO, 100 MHz): 20.63, 27.00, 28.77, 32.23, 44.45, 50.11, 59.70, 109.93, 126.96, 127.14, 128.03, 129.13, 129.49, 136.38, 137.63, 138.90,151.80, 158.21, 164.03, 194.19 ppm. IR (KBr) υ =2954, 2869, 1639, 1585, 1481, 1373 cm^{-I}. MS (EI): 435 (m)$^{+}$, 437 (m+2)$^{+}$.

2-(4-chlorophenyl)-8,9-dihdro-8,8-dimethyl-5*H* [1,3,4]thiadiazolo [2,3-*b*] quinazolin-6(7H)-one(4n): Yellow solid (78%), M.P.= 205-207 ^{0}C. ^{1}H-NMR (DMSO, 300 MHz) δ (ppm) = 0.99 (s, 3H); 1.04 (s, 3H); 2.09 (d, 1H); 2.20-2.14 (d, 1H); 2.52-2.27 (s, 2H); 6.38 (s, 1H); 7.36-7.11 (m, 5H); 7.59-7.51 (d, 2H); 7.76-7.71 (d, 2H); ^{13}C-NMR (DMSO, 100 MHz): 26.08, 29.66, 34.24, 45.12, 56.16, 124.34, 127.17, 129.42, 130.04, 131.93, 148.00, 152.00, 161.10, 194.6 ppm. IR (KBr) υ =2951, 1673, 1595, 1529, 1379 cm^{-I}. MS (EI): 422 (m+H)$^{+}$.

2-(4-chlorophenyl)-8,9-dihdro-8,8-dimethyl-5-p-tolyl-5*H* [1,3,4]thiadiazolo [2,3-*b*] quinazolin-6(7H)-one(4o): Yellow solid (80%), M.P.= 201-203 ^{0}C. ^{1}H-NMR (DMSO, 300 MHz) δ (ppm) = 0.96 (s, 3H); 1.02 (s, 3H); 2.18-2.05 (d, 1H); 2.45-2.24 (m, 2H); 6.32 (s, 1H); 7.23-7.11 (m, 4H); 7.58-7.57 (d, 2H); 7.74-7.71 (d, 2H). IR (KBr) υ =2954, 1648, 1586, 1450, 1378 cm^{-I}. MS (EI): 435 (m+H) +.

5-(4-bromophenyl)-2-(4-chlorophenyl)-8,9-dihdro-8,8-dimethyl-5*H*[1,3,4]thiadiazolo [2,3-*b*] quinazolin-6(7H)-one(4p): Yellow solid (80%), M.P.= 274-276 ^{0}C. ^{1}H-NMR (DMSO, 300 MHz) δ (ppm) = 0.97 (s, 3H); 1.04 (s, 3H); 2.08 (d, 1H); 2.14-2.20 (d, 1H); 2.26 (s, 2H); 6.40 (s, 1H); 7.32-7.34 (d, 2H); 7.54-7.56 (d, 2H); 7.57-7.60 (d, 2H); 7.74-7.76 (d, 2H). IR (KBr) υ =2954, 2889, 1643, 1581, 1477, 1377 cm^{-I}. MS (EI): 499 (m)$^{+}$, 501 (m+2)$^{+}$.

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