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ORIGINAL PAPER

Catalytic systems containing *p*-toluenesulfonic acid monohydrate catalyzed the synthesis of triazoloquinazolinone and benzimidazoquinazolinone derivatives

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Abstract A variety of quinazolinones are readily prepared via one-pot three-component reaction in good to excellent yields. The desired products were obtained from the reaction of dimedone, various aldehydes with 2-aminobenzimidazole or 3-amino-1,2,4-triazole under mild reaction conditions using *p*-toluenesulfonic acid monohydrate as effective catalyst in acetonitrile as solvent. Starting from the corresponding available materials, this friendly and environmentally free-metal procedure has been successfully extended to the synthesis of triazoloquinazolinones and benzimidazoquinazolinones. The salient advantages of this method are mild reaction conditions, nontoxic and inexpensive catalyst, environmentally benign, high to excellent yields, shorter reaction times, easy operation, and no column chromatographic separation.

Keywords Triazoloquinazolinone · 3-Amino-1,2,4-triazole · 2-Aminobenzimidazole · *p*-Toluenesulfonic acid

Introduction

The growth of organic synthesis has been facilitated by the development of one-pot methods, since they generate less waste, minimize isolation of intermediates in multistep syntheses of complex molecular targets, and save time and minimize cost [1]. One-pot reactions can be classified roughly as cascade, domino, or tandem reactions [2–4]. Of one-pot synthetic strategies, multicomponent reactions (MCRs), leading to interesting heterocyclic scaffolds, are particularly useful for combinatorial chemistry as powerful tools [5] because of their valuable features such as atom economy, environmental friendliness, straightforward reaction design, and the opportunity to construct target compounds by the introduction of several diversity elements in a single chemical operation [6, 7].

Five- and six-membered heterocyclic compounds are important constituents that often exist in biologically active natural products and synthetic compounds of medicinal interest [8, 9]. Among them quinazolinones and 1,2,4-triazoloquinazolinone derivatives are known for diverse pharmacological activities as hypnotic [10], analgesic [11], neuroleptic [12], anticonvulsant [13], muscle relaxant [14], antifertility [15], latent leishmanicidal [16], antihistaminic [17–19], antihypertensive [11, 20–22], anti-inflammatory [23], anticancer [24], and anti-HIV [25]. In spite of their importance from a pharmaceutical, industrial, and synthetic point of view, a few methods for their preparation are reported in the literature. These include condensation reactions of dimedone, various aldehydes with 2-aminobenzimidazole or 3-amino-1,2,4-triazole in the presence of molecular iodine (I_2) [26], microwave [27], $H_6P_2W_{18}O_{62}\cdot 18H_2O$ [28], and refluxing in DMF [29, 30]. Some of these processes suffer from some limitations such as drastic reaction conditions, expensive reagents, and low-to-moderate yields, relatively long reaction times and high temperature. Therefore, the development of clean, high-yielding and environmentally friendly approaches is desirable.

The use of organic catalysts has received considerable attention in organic synthesis owing to their important advantages such as the possibility of performing reactions with acid-sensitive substrates, milder reaction conditions, and selectivity [31–33]. *p*-Toluenesulfonic acid

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monohydrate (*p*-TsOH·H_2O) has received considerable attention in organic synthesis owing to its advantages such as low cost and ease of handling and isolation [34–36]. Encouraged by the remarkable results obtained from above conditions, and also, in continuation of our ongoing green chemistry program that utilizes homogeneous systems [37–39] in various organic transformations, we report a simple, mild, and convenient procedure for effecting one-pot, three-component reaction of aryl aldehydes, dimedone and, 2-aminobenzimidazole/3-amino-1,2,4-triazole for preparation of triazolo/benzimidazolo-quinazolinone derivatives using *p*-toluenesulfonic acid monohydrate as a homogeneous catalyst under mild reaction conditions at 40–50 °C (Scheme 1).

Results and discussion

In the present study, the authors examine the catalytic behavior of *p*-toluenesulfonic acid as a catalyst for the coupling reaction, and to optimize the reaction conditions, we have performed a set of preliminary experiments on benzaldehyde, dimedone, and 2-aminobenzimidazole in acetonitrile at room temperature as a model reaction. The reaction was completed in 60 min when 15 mol % of *p*-TsOH·H_2O was used. Rate enhancement was observed when higher molar ratios were used but no significant improvement in the yield was observed. The results are shown in Table 1. As could be seen in Table 1, to determine the catalytic amount of *p*-toluenesulfonic acid, different ratios of the catalyst were examined and the admissible result was obtained from using 15 mol % of *p*-toluenesulfonic acid in acetonitrile (Table 1, entry 4). Also, it is noteworthy that the reaction did not progress even after 180 min in the absence of catalyst (Table 1, entry 1).

Next, we tested the influence of different temperatures on the synthesis of quinazolinones in the presence of *p*-toluenesulfonic acid (15 mol %) in acetonitrile, the results are summarized in Table 2. It was found that the best results were obtained at 40–50 °C, efficiency of 96 % was generated (Table 2, entry 3) at the higher temperature that did not increase the reaction yield (Table 2, entries 4, 5, and 6).

During the course of the screening of a variety of reaction conditions such as reaction temperature, the amount of the catalyst and solvent, we found that the use of acetonitrile as a solvent was essential for the efficient formation corresponding compounds. To estimate the generality and versatility of the catalyst, the same reaction was applied for the synthesis of 1,2,4-triazolo[5,1-*b*]quinolin-8(4*H*)-one derivatives by replacing 2-aminobenzimidazole with 3-amino-1,2,4-triazole. In this case, the products were obtained in excellent yields. To explore the scope of the procedure, we extended this reaction to various aromatic aldehydes in the presence of electron-withdrawing (NO_2, Cl, F) and electron-releasing (Me, OMe) substituents, both of which gave the desired product. We observed that the activity of 3-amino-1,2,4-triazole is better than 2-aminobenzimidazole. The results are shown in Table 3. Results show that the substituent groups did not play any significant role in the reactivity of the substrate.

In this procedure, the products were easily isolated by simple filtration; no need for column chromatography and further recrystallization. All known products were characterized by comparison of the melting points and the analytical data (IR, NMR) with those reported for authentic

Scheme 1

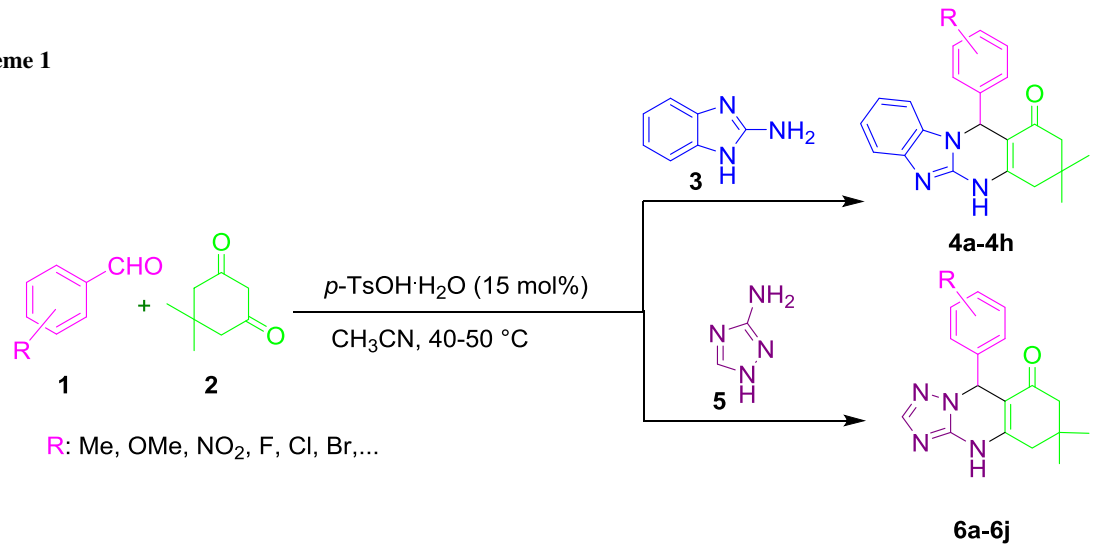
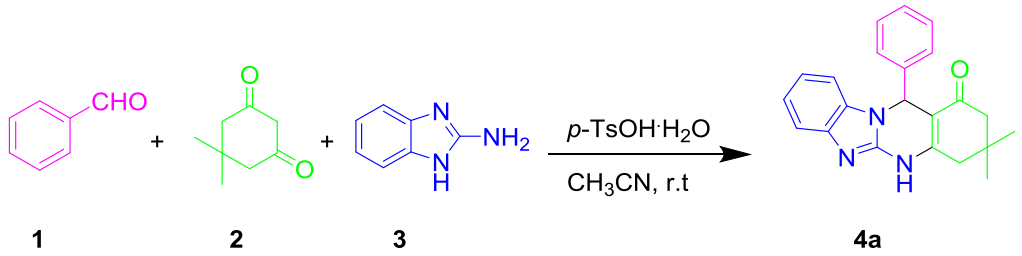


Table 1 Condensation reaction of 2-aminobenzimidazole, benzaldehyde, and dimedone in the presence of different loading of the catalyst at ambient conditions[a]



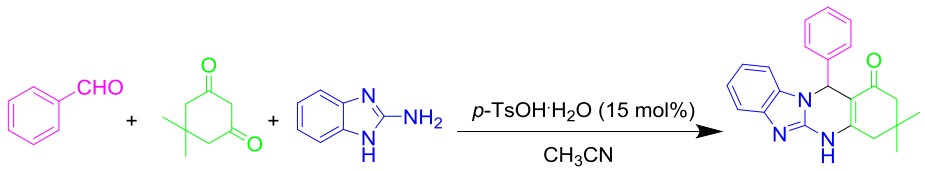
1 **2** **3** **4a**

Entry	Solvent/temp/ °C	Catalyst/mol %	Time/min	Yield/ %[b]
1	CH_3CN/rt	–	180	–
2	CH_3CN/rt	5	120	68
3	CH_3CN/rt	10	100	75
4	**CH_3CN/rt**	**15**	**60**	**87**
5	CH_3CN/rt	20	55	80
6	CH_3CN/rt	25	50	82
7	CH_3CN/rt	30	45	83
8	-/rt	15	180	–

[a] All reactions were carried out using 1 mmol benzaldehyde, 1 mmol dimedone, and 1 mmol 2-aminobenzimidazole with varying amounts of catalyst in 5 cm^3 CH_3CN at room temperature

[b] Yield refers to the pure isolated products

Table 2 Condensation reaction of 2-aminobenzimidazole, benzaldehyde, and dimedone in the presence of different loading of the catalyst at different temperature[a]



1 **2** **3** **4a**

Entry	Solvent	*T*/°C	Time/min	Yield/%[b]
1	CH_3CN	20–30 (r.t.)	60	87
2	CH_3CN	30–40	60	89
3	**CH_3CN**	**40–50**	**30**	**96**
4	CH_3CN	50–60	55	91
5	CH_3CN	60–70	50	90
6	CH_3CN	70–80	40	92

[a] All the reactions were carried out using 1 mmol benzaldehyde, 1 mmol dimedone, and 1 mmol 2-aminobenzimidazole with catalytic amount of catalyst in 5 cm^3 CH_3CN at different temperatures

[b] Yield refers to the pure isolated products

samples. The structure of new compounds **4h** and **6j** was deduced on the basis of IR, ^1H and ^{13}C NMR spectroscopy, mass spectrometry, and elemental analysis. For example, the mass spectrum of **4h** displayed the molecular ion peak (M^+) at $m/z = 344$, which is consistent with the proposed structure. The ^1H NMR spectrum of **4h** exhibited two singlet at $\delta = 0.91$ and 1.05 ppm for the geminal methyl protons. Four doublets were observed at 2.07 ($J = 16.0$ Hz), 2.26 ($J = 16.0$ Hz), 2.52 ($J = 16.0$ Hz), and 2.63 ($J = 16.0$ Hz) for the diastereotopic methylene

Table 3 Synthesis of 3,3-dimethyl-12-phenyl-3,4,5,12-tetrahydrobenzo[4,5]imidazo[2,1-*b*]quinazolin-1(2*H*)-one **4** and 1,2,4-triazolo[5,1-*b*]quinolin-8(4*H*)-one **6** derivatives in the presence of *p*-TsOH·H_2O

Entry	R	Amine	Product	Time/min	Yield/%[a]	M.p./°C	Lit. m.p./°C
1	H	**3**	**4a**	25	95	>300	>300 [28]
2	4-Br	**3**	**4b**	35	94	>300	>300 [28]
3	4-NO_2	**3**	**4c**	20	98	>300	>300 [29]
4	4-F	**3**	**4d**	40	87	>300	>300 [27]
5	4-OMe	**3**	**4e**	15	95	>300	>300 [29]
6	4-Cl	**3**	**4f**	18	91	>300	>300 [28]
7	2,4-Cl_2	**3**	**4g**	15	96	>300	>300 [26]
8	4-pyridin-	**3**	**4h**	25	97	>300	This work[b]
9	H	**5**	**6a**	30	96	248–250	248–250 [30]
10	4-OH	**5**	**6b**	45	89	305–306	307–309 [28]
11	4-Cl	**5**	**6c**	10	96	302–304	304–306 [26]
12	4-Me	**5**	**6d**	15	95	263–265	264–269 [28]
13	2,4-Cl_2	**5**	**6e**	18	97	320–322	323–325 [26]
14	2,4-$(OMe)_2$	**5**	**6f**	18	85	208–210	210–212 [26]
15	4-F	**5**	**6g**	20	90	256–258	258–260 [27]
16	3-NO_2	**5**	**6h**	14	98	265–267	266–269 [26]
17	2-Naphthyl-	**5**	**6i**	16	97	268–270	287–290 [26]
18	2,6-Cl_2	**5**	**6j**	15	98	306–308	This work[b]

[a] Yields refer to the pure isolated products

[b] The new compounds synthesized in this work

protons of H-4, H'-4, H-2 and H'-2, respectively. The methine proton of the central ring (H-12) was observed as a singlet at 6.47 ppm. The aromatic protons' resonance observed as the mixture of doublets and triplets at 6.97–8.45 ppm. Therefore, all the spectral data were in agreement with the proposed structure.

p-Toluenesulfonic acid exhibited remarkable reactivity as a homogenous organocatalyst and considerably accelerated the reactions. In the end, we turned our attention towards mechanistic studies for this transformation. On the basis of the proposed mechanism in the literature, and all our experimental results possible mechanism for this three-component reaction for the formation of benzimidazolo/1,2,4-triazoloquinazolinone derivatives **4a-4h** and **6a-6j** in the presence of *p*-toluenesulfonic is shown in Scheme 2. At first, the described transformations proceed via the initial formation of respective α,β-unsaturated carbonyl compounds **11** upon the loss of water molecule via Knoevenagel condensation. Next, the α,β-unsaturated carbonyl compounds, undergoing more nucleophilic endocyclic nitrogen attack in the 2-aminobenzimidazole **3**, yield the corresponding acyclic intermediate **12**. Therefore, the endocyclic nitrogen is more nucleophilic than the primary amino group [40, 41]. Further intermediate **12** undergoes intra-molecular cyclization giving rise to intermediate **13** and by the loss of water molecule to yield the corresponding quinazolinones **4**.

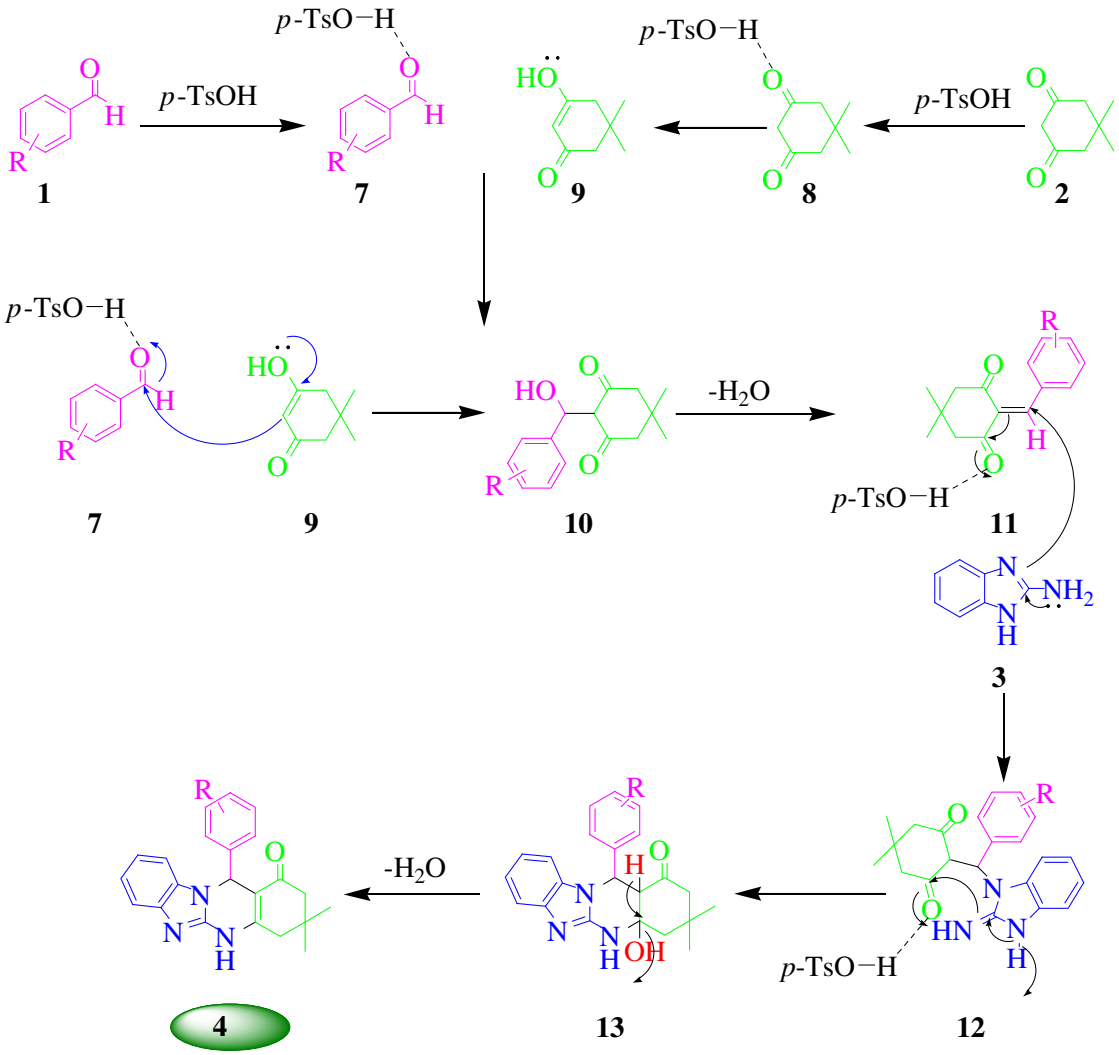
To show the merit of the present work in comparison with reported results in the literature, we compared results of *p*-TsOH·H_2O with molecular iodine (I_2) [26], microwave [27], $H_6P_2W_{18}O_{62}\cdot 18H_2O$ [28], and refluxing in DMF [29, 30] in the synthesis benzimidazolo/1,2,4-triazoloquinazolinone derivatives. As shown in Table 4, *p*-TsOH·H_2O can act as productive and effective catalyst with respect to reaction times and yields of products.

In summary, we have developed a novel, simple, convenient and efficient synthetic protocol for the synthesis of triazolo/benzimidazolo-quinazolinone derivatives using cheap, readily available and nontoxic *p*-toluenesulfonic acid monohydrate as a homogeneous catalyst in a catalytic amount. This reaction can be regarded as an efficient approach for the preparation of synthetically and pharmaceutically important quinazolinone, triazole, imidazole, pyrazole, and tetrazole systems. This one-pot reaction has some important advantages such as the easy workup procedure, simple and readily available precursors, nontoxic and inexpensive catalyst, and good to high yields. The products were collected easily by simple filtration and no need for further purification.

Experimental

Melting point and IR spectra of all compounds were obtained on an Electrothermal 9100 apparatus and a

Scheme 2



JASCO FT/IR-460 plus spectrometer, respectively. ^{1}H and ^{13}C NMR spectra of compounds were recorded on a Bruker DRX-400 Avance instrument in DMSO-d_6 or $CDCl_3$. The mass spectra for the new compounds were recorded on an Agilent Technology HP 5973 MSD mass spectrometer operating at an ionization potential of 70 eV. Elemental analyses for C, H, and N were determined using a Perkin-Elmer 2400 instrument. All commercial reagents were used without further purification.

General procedure for the synthesis of 3,4,5,12-tetrahydrobenzo[4,5]imidazo[2,1-b]quinazolin-1(2H).ones ***4*** *and 1,2,4-triazolo[5,1-b]quinazolin-8(4H)-ones* ***6*** *using p-TsOH·H_2O as the reaction medium*

A mixture of 2-aminobenzimidazole or 3-amino-1,2,4-triazole (1.0 mmol), arylaldehyde (1.0 mmol), dimedone (1.0 mmol), and 15 mol % *p*-TsOH·H_2O was stirred in 5 cm^3 acetonitrile as solvent at 40–50 °C for the appropriate time (Table 3). The progress of the reaction was monitored by TLC. After completion of the reaction, a thick precipitate was obtained. The solid product was filtered off and washed with acetonitrile and subsequently dried in air. The pure product was characterized by conventional spectroscopic methods.

3,3-Dimethyl-12-(4-pyridyl)-3,4,5,12-tetrahydrobenzo[4,5]-imidazo[2,1-b]quinazolin-1(2H).one (**4h**, $C_{21}H_{20}N_4O$)
White solid; yield 97 %; m.p.: >300 °C; IR (KBr): $\bar{\nu}$ = 3419, 3030, 2957, 2922, 1640, 1615, 1596, 1570, 1460, 1377, 748 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6): δ = 0.91 (s, 3H, CH_3), 1.05 (s, 3H, CH_3), 2.07 (d, J = 16.0 Hz, 1H, H-4), 2.26 (d, J = 16.0 Hz, 1H, H'-4), 2.52 (d, J = 16.0 Hz, 1H, H-2),

Table 4 Comparison result of *p*-TsOH·H_2O with molecular iodine (I_2) [26], microwave [27], $H_6P_2W_{18}O_{62}$·$18H_2O$ [28], and refluxing in DMF [29, 30] in the synthesis of benzimidazolo/ [1–4, 6, 7] triazoloquinazolinone derivatives

Entry	Comp.	Catalyst/conditions	Time/min	Yield/%
1	**4a**	I_2/CH_3CN, reflux	10	84.6
		Silica gel/microwave	3	95
		$H_6P_2W_{18}O_{62}$·$18H_2O/CH_3CN$, reflux	15	96
		DMF/reflux	–	65
		p-TsOH·H_2O/CH_3CN, 40–50 °C	25	95
2	**4c**	I_2/CH_3CN, reflux	10	97.1
		Silica gel/microwave	3	94
		$H_6P_2W_{18}O_{62}$·$18H_2O/CH_3CN$, reflux	10	99
		DMF/reflux	–	53
		p-TsOH·H_2O/CH_3CN, 40–50 °C	20	98
3	**6c**	I_2/CH_3CN, reflux	10	96.1
		Silica gel/microwave	4	93
		$H_6P_2W_{18}O_{62}$·$18H_2O/CH_3CN$, reflux	30	97
		DMF/reflux	–	65
		p-TsOH·H_2O/CH_3CN, 40–50 °C	10	96
4	**6d**	I_2/CH_3CN, reflux	–	–
		Silica gel/microwave	–	–
		$H_6P_2W_{18}O_{62}$·$18H_2O/CH_3CN$, reflux	40	91
		DMF/reflux	-	-
		p-TsOH·H_2O/CH_3CN, 40–50 °C	15	95
5	**6h**	I_2/CH_3CN, reflux	–	–
		Silica gel/microwave	5	96
		$H_6P_2W_{18}O_{62}$·$18H_2O/CH_3CN$, reflux	30	98
		DMF/reflux	–	–
		p-TsOH·H_2O/CH_3CN, 40–50 °C	14	98

2.63 (d, $J = 16.0$ Hz, 1H, H'-2), 6.47 (s, 1H, H-12), 6.97 (t, $J = 7.6$ Hz, 1H, Ar–H), 7.07 (t, $J = 7.6$ Hz, 1H, Ar–H), 7.23 (d, $J = 8.0$ Hz, 1H, Ar–H), 7.29 (d, $J = 5.6$ Hz, 2H, Ar–H), 7.40 (d, $J = 8.0$ Hz, 1H, Ar–H), 8.45 (d, $J = 8.0$ Hz, 2H, Ar–H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): $\delta = 27.0, 29.0, 32.7, 40.2, 50.2, 53.6, 105.3, 110.3, 117.5, 121.1, 122.4, 122.5, 132.1, 142.3, 145.6, 149.9, 150.2, 151.6, 193.0$ ppm; MS (EI, 70 eV): *m/z* (%) = 344 (M^+, 67), 301 (1), 298 (9), 266 (100), 236 (2), 210 (7), 182 (15), 155 (4), 129 (3), 105 (1), 83 (3), 51 (3).

9-(2,6-Dichlorophenyl)-5,6,7,9-tetrahydro-6,6-dimethyl-[1,2,4]triazolo[5,1-b]quinazolin-8(4H)-one
(**6j**, $C_{17}H_{16}Cl_2N_4O$).
White solid; yield 98 %; m.p.: 306–308 °C; IR (KBr): $\bar{\nu}$= 3396, 3132, 3085, 2956, 2923, 1650, 1641, 1580, 1546, 1441, 1369, 1255, 1191, 772 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6): $\delta = 0.99$ (s, 3H, CH_3), 1.04 (s, 3H, CH_3), 2.03 (d, $J = 16.0$ Hz, 1H, H-5), 2.20 (d, $J = 16.0$ Hz, 1H, H'-5), 2.41 (d, $J = 16.0$ Hz, 1H, H-7), 2.54 (d, $J = 20.0$ Hz, 1H, H'-7), 7.02 (s, 1H, H-9), 7.27 (d, $J = 4.0$ Hz, 1H, Ar–H), 7.29 (s, 1H, Ar–H), 7.48 (dd, $J = 4.0$, 8.0 Hz, 1H, Ar–H), 7.68 (s, 1H, H-2), 11.28 (s, 1H, NH) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): $\delta = 21.2, 27.2, 29.0, 32.3, 50.3, 55.4, 103.6, 125.9, 128.9, 130.3, 133.9, 134.2, 137.0, 147.9, 150.5, 152.4, 193.4$ ppm; MS (EI, 70 eV): *m/z* (%) = 363 (M^+, 9), 362 (22), 327 (100), 291 (67), 269 (1), 243 (8), 217 (31), 189 (3), 161 (23), 133 (8), 107 (5), 83 (8), 55 (8).

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