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An electrochemical multicomponent [3 + 1 + 1] annulations to synthesize polysubstituted 1,2,4-triazoles



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1. Introduction

The development of green and sustainable ways for the construction of chemical products is a fundamental goal in modern organic synthesis [1]. Multicomponent reactions provide a new method for constructing fine chemicals. Multicomponent reactions are a prominent approach for the generation of structurally complex and diverse scaffolds from simple building blocks, thereby affording the advantages of operational simplicity, high chemical efficiency, convergence and atom economy [2-6]. However, the development of multicomponent reactions used to construct aromatic N-heterocyclic compounds remains a daunting task for synthetic chemists because, with an increase in the number of starting materials, multicomponent reactions may suffer from inefficiency

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ABSTRACT

An electrochemical multicomponent [3 + 1 + 1] annulation used for the synthesis of 1,3,5-trisubstituted 1,2,4-triazoles has been developed under transition-metal-, acid-, base- and external-oxidant-free conditions. In an undivided cell, a variety of hydrazones and aldehydes smoothly participate in this transformation to furnish a range of substituted 1,2,4-triazoles. The reaction constitutes a new transformation from hydrazones, aldehydes and NH4OAc into 1,2,4-triazoles, providing a unifying, simple and environmentally friendly approach to the currently available methods.

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and additional competing side-reactions [7–9]. Thus, the exploration of multicomponent reactions for the synthesis of aromatic Nheterocycles is still a crucial challenge.

In this context, 1,2,4-triazoles are omnipresent aromatic Nheterocyclic motifs in numerous biologically active compounds and they also have widespread applications in biological and pharmaceutical fields as well as materials science [10-14]. Given their applications, several strategies have been implemented for the synthesis of 1,2,4-triazoles [15-23]. Traditional methods for the preparation of substituted 1,2,4-triazoles include the Pellizzari and Einhorn-Brunner reactions [24]. More recent methods involve transition-metal-catalysed or transition-metal-free dehydrogenative annulations. Although the previously reported protocols have certain merits of their own, still they suffer from many shortcomings such as the use of a super-stoichiometric amount of reagents or oxidants, low chemo-selectivity, narrow functional group tolerance, multiple reaction steps and the production of copious waste. Transition-metal-, acid-, base-, external-oxidant-free and stepeconomical processes for efficient synthesis of substituted 1,2,4triazoles are in demand.

Organic electrochemistry represents an environmentally benign and sustainable method using electrons as reagents. Using





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electrons as mass-free reagents, the use of a stoichiometric amount of a chemical oxidant can be avoided, thereby eliminating the production of waste [25-29]. Over the past decade, electrochemical synthesis has attracted increasing attention and has been subjected to considerable development [30-39]. The electrochemical multicomponent reactions dramatically shorten the pathway to construct complex ring systems. Recently, Yuan and coworkers reported an electrosynthesis of 1.5-disubstituted and 1aryl 1,2,4-triazoles via an electrochemical multicomponent reaction [40]. However, a strong base was used in this method and 1,2,4-triazoles bearing alkene or alkyne substituents were not found to be viable products. Thus, an electrosynthesis of 1,2,4triazole products bearing alkene or alkyne substituents via a convergent route under transition-metal-, acid-, base- and external-oxidant-free conditions has remained elusive. Herein, we report the successful development of an electrochemical multicomponent [3 + 1 + 1] annulation used for the synthesis of 1,3,5trisubstituted 1,2,4-triazoles from hydrazones, aldehydes and NH₄OAc. Advantageously, this method proceeds in a transitionmetal-, acid-, base- and external oxidant-free fashion to provide a variety of functionalized 1,2,4-triazoles (Scheme 1).

2. Results and discussion

We chose 1-benzylidene-2-phenylhydrazine 1a, benzaldehyde 2a and NH₄OAc as model substrates to perform our study. To optimise the electrolytic conditions, the electrolysis was carried out at a constant current in an undivided cell equipped with a graphite felt (GF) anode and cathode. The reaction was conducted using 1.0 equiv of 2a and 3.0 equiv of NH₄OAc at 40 °C in the presence of 20 mol% LiClO₄ and 20 mol% ⁿBu₄NI (TBAI). When the reaction was carried out in MeCN under a constant current (23 mA) in the undivided cell, the [3+1+1] annulation product **3aa** was obtained in 86% yield (Table 1, entry 1). A shorter reaction time was insufficient form the product 3aa effectively (entry 2). Several other mediators, including NaI, HI, NH₄I and ⁿBu₄NBr were subsequently investigated and they were found to be less effective than TBAI (entries 3–7). The variation of electrodes had the biggest influence on this reaction. Carbon-based materials showed good activity at both the anode and cathode (entries 8, 9), which is attributed to the high surface area of the porous carbon materials. Both increasing or decreasing the current afforded the final product in lower yield (entries 10, 11). A solvent screen revealed that MeCN was the most suitable solvent for this transformation (entries 12–15). As for the choice of the electrolyte, ⁿBu₄NBF₄ and ⁿBu₄NPF₆ exhibited lower efficiencies than LiClO₄ (entries 16, 17). Lowering the reaction temperature decreased the yield (entry 18). A relatively low yield was obtained when the reaction was conducted under an air atmosphere (entry 19). Performing the reaction with NH₄HCO₃ resulted in a decreased yield (entry 20). A control experiment indicated that galvanization was necessary for this transformation



Scheme 1. The electrochemical multicomponent reaction for the synthesis of 1,2,4-triazole derivatives.

Table 1

Optimization of the reaction conditions ^{*a*}.

Ph N H Ph +	Ph [^] O	+ NH ₄ OAc	GF(+) GF(-) I = 23 mA	Ph N-N II Ph
1a	2a		undevided cell	3aa

Entry	Variation from the standard reaction conditions	Yield ^b (%)
1	None	86
2	3 h instead of 6 h	59
3	NaI instead of TBAI	70
4	HI instead of TBAI	66
5	NH ₄ I instead of TBAI	71
6	ⁿ Bu ₄ NBr instead of TBAI	53
7	NIS instead of TBAI	n.d.
8	Pt(+) Pt(-) instead of $GF(+) GF(-)$	23
9	C(+) C(-) instead of $GF(+) GF(-)$	70
10	20 mA instead of 23 mA	54
11	26 mA instead of 23 mA	67
12	DMSO instead of MeCN	25
13	DMF instead of MeCN	33
14	DCE instead of MeCN	30
15	EtOH instead of MeCN	Trace
16	ⁿ Bu ₄ NBF ₄ instead of LiClO ₄	72
17	ⁿ Bu ₄ NPF ₆ instead of LiClO ₄	67
18	R.T. instead of 40 °C	57
19	Air instead of Ar	75
20	NH ₄ HCO ₃ instead of NH ₄ OAc	14
21	No current	n.d.
22	Large-scale reaction	76

^a Reaction conditions: GF anode, GF cathode, constant current = 23 mA, **1a** (0.3 mmol), **2a** (0.3 mmol), NH₄OAc (0.9 mmol), TBAI (20 mol%), LiClO₄ (0.1 M), MeCN (6 mL), Ar, 40 $^{\circ}$ C, undivided cell. ^b Isolated yield.

(entry 21). The scale of this reaction between **1a**, **2a** and NH₄OAc was increased to 10 mmol and the desired product **3aa** was obtained in 76% yield (entry 22).

To demonstrate the applicability of this transformation, a variety of hydrazones 1 were then applied to react with benzaldehyde 2a and NH₄OAc under the optimized reaction conditions (Table 2). The reaction proceeded successfully with respect to the hydrazones prepared from various substituted aromatic aldehydes. A variety of hydrazones substituted at the para position with electron-donating alkyl or alkoxy groups on the benzene ring were viable in the reaction (3 ab, 3ac). It is particularly noteworthy that functional groups such as F, Cl, Br, CN and CO₂Me were well-tolerated in the reaction (3ad-3ah). Regarding the steric hindrance in the substrate, an ortho-substituted starting material was also tolerated and provided the desired 1,2,4-triazole in 50% yield (3ai). Moreover, substrate 1j also reacted smoothly and the desired product 3aj was obtained in 48% yield. A substrate bearing a furan ring was successfully applied in the reaction, furnishing the 1,2,4-triazole product in 52% yield. Interestingly, hydrazones derived from an unsaturated aldehyde or aliphatic aldehyde could participate in the reaction (**3 al**, **3am**). To highlight the utility of this transformation, hydrazones derived from different aromatic hydrazines and benzaldehyde were subjected to the reaction and good to excellent product yields were observed (3an-3ap).

Next, the scope of the reaction with respect to the aldehyde was investigated. As shown in Table 3, the substituents on the phenyl ring of the benzaldehydes studied had no obvious effect on the reaction and the expected 1,2,4-triazole products were produced in good to high yield. The electrolysis reaction exhibited excellent compatibility with the electron-donating groups on the phenyl ring, thereby providing a high yield of substituted 1,2,4-triazoles

Table 2

Scope of hydrazones **1** used in the reaction ^a.



^{*a*} Reaction conditions: GF anode, GF cathode, constant current = 23 mA, **1** (0.3 mmol), **2a** (0.3 mmol), NH₄OAc (0.9 mmol), TBAI (20 mol%), LiClO₄ (0.1 M), MeCN (6 mL), Ar, 40 °C, undivided cell. ^b Isolated yield.

(**3ba**, **3ca**). Substrates bearing electron-withdrawing groups on the phenyl ring produced good product yields (**3da**, **3ea**). Steric hindrance did not affect the reaction yields, employing *ortho*- and *meta*-substituted benzaldehydes as the starting materials, the reactions occurred smoothly and gave the cyclization products in good to high yields.

(**3fa, 3ga**). Notably, the 1-naphthaldehyde gave its desired product **3ha** in 51% yield. Apart from benzaldehyde **2a**, thiophene-2-carbaldehyde **2i** gave the corresponding product **3ia** in 67% yield. The use of methyl 2-oxoacetate **2j** gave the desired product in 49% yield. Importantly, aliphatic aldehydes were also compatible with the reaction system (**3ka, 3la**). Alkenyl- and alkynyl-substituted aldehydes also displayed good activity under the conditions (**3ma, 3na**). Unfortunately, paraformaldehyde did not deliver the corresponding 1,2,4-triazole product under the optimal reaction conditions.

To gain further information on the reaction mechanism, several control reactions were performed. The reaction of **1a** with **2a** and NH₄OAc were conducted in the presence of a radical inhibitor (2.0 equiv), such as 2,6-di-*tert*-butyl-4-methylphenol, hydroquinone and 2,2,6,6-tetramethyl-1-piperidinyloxy. The reaction was completely suppressed. These results suggest that a radical process may be involved in this transformation. As expected, when the

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Table 3

Scope of aldeaydes **2** used in the reaction.^{*a*}.



^{*a*} Reaction conditions: GF anode, GF cathode, constant current = 23 mA, **1** (0.3 mmol), **2a** (0.3 mmol), NH₄OAc (0.9 mmol), TBAI (20 mol%), LiClO₄ (0.1 M), MeCN (6 mL), Ar, 40 °C, undivided cell. ^b Isolated yield.

reaction of **1a** with **2a** was carried out in the presence of a stoichiometric amount of iodine as an oxidant, no desired product was obtained at 40 °C. Cyclic voltammetry experiments were performed to investigate the possible reaction mechanism. In the TBAI case (Figure S1, curve e), an obvious oxidation peak occurred at 0.45 V vs Ag/AgCl (SCE), which was due to the electro-oxidation of I⁻ ions. These results suggest that the electrochemical oxidation of I⁻ at the anode was the main process.

Based on these observations and previous studies [18,37], a plausable mechanism was proposed, as shown in Scheme 2. At the anode, the electrochemical oxidation of I⁻ leads to the formation of iodine radicals. At the same time, 1a undergoes tautomerization to give compound A. Thereafter, an iodine radical reacts with A to afford intermediate **B**. Meanwhile, phenylmethanimine **D** is formed through the condensation of benzaldehyde **2a** with NH₄OAc. Anodic oxidation of **B** may give complex **C** and nucleophilic attack of intermediate **D** leads to intermediate **E**, which undergoes tautomerization to give F. With the aid of an iodine radical, F is readily transformed into intermediate G, which will undergo a similar process to the previous pathway to give H. Then, the intramolecular attack of the nitrogen atom provides I. Finally, I gives the desired aromatization product 3aa via an oxidative dehydrogenation reaction. Alternatively, the nucleophilic attack of NH₃ on complex C gives intermediate J. The reaction of benzaldehyde 2a with J forms intermediate E.



Scheme 2. Plausible mechanism for the reaction.

3. Conclusion

In summary, we introduced a novel sustainable multicomponent 1,2,4-triazole synthesis. Hydrazones, aldehydes and NH₄OAc are assembled in three-component reactions. Our electrochemical synthetic method is compatible with a wide range of hydrazones and aldehydes. Readily available raw materials, good product yields, scalability and an experimentally convenient catalytic process make this protocol practical and attractive.

4. Experimental section

4.1. General materials and methods

All glassware was oven dried at 110 °C for hours and cooled down under vacuum. Unless otherwise noted, materials were obtained from commercial suppliers and used without further purification. The instrument for electrolysis was dual display potentiostat (DJS-292 B) (made in China). Both of the anode eleccathodic electrode trode and were carbon rod (15 mm \times 15 mm \times 0.2 mm). Thin layer chromatography (TLC) employed glass 0.25 mm silica gel plates. Flash chromatography columns were packed with 200-300 mesh silica gel in petroleum (bp. 60–90 °C). ¹H and ¹³C NMR data were recorded with Bruker Advance III (400 MHz) spectrometers with tetramethylsilane as an internal standard. All chemical shifts (δ) are reported in ppm and coupling constants (*J*) in Hz.

4.2. General procedure for synthesis of 6-aryl (heteroaryl) substituted phenanthridines 3

In an oven-dried undivided three-necked bottle (15 mL) equipped with a stir bar, hydrazones **1** (0.30 mmol), aldehydes **2** (0.30 mmol), NH₄OAc (0.9 mmol), TBAI (0.06 mmol), LiClO₄ (0.05 mmol) and CH₃CN (5 mL) were combined and added. The bottle was equipped with graphite felt as the anode and graphite felt as the cathode and was then charged with argon. The reaction mixture was stirred and electrolyzed at a constant current of 23 mA for 6 h at 40 °C. When the reaction was finished, the reaction mixture was diluted in 40 mL ethyl acetate, washed with a saturated solution of brine (2 × 10 mL), dried (Na₂SO₄) and concentrated in vacuum, and the resulting residue was purified by silica gel column chromatography (hexane/ethyl acetate) to afford the products desired product.

Large-scale experimental procedure: In an oven-dried undivided three-necked bottle (100 mL) equipped with a stir bar, 1-benzylidene-2-phenylhydrazine **1a** (10.0 mmol), benzaldehyde **2a** (10.0 mmol), NH₄OAc (30.0 mmol), TBAI (2.0 mmol), LiClO₄

(1 mmol) and CH₃CN (50 mL) were combined and added. The bottle was equipped with graphite felt as the anode and cathode and was then charged with argon. The reaction mixture was stirred and electrolyzed at a constant current of 23 mA for 16 h at 40 °C. When the reaction was finished, the reaction mixture was diluted in 80 mL ethyl acetate, washed with a saturated solution of brine (2 × 20 mL), dried (Na₂SO₄) and concentrated in vacuum, and the resulting residue was purified by silica gel column chromatography (hexane/ethyl acetate) to afford the products desired product.

1,3,5-Triphenyl-1H-1,2,4-triazole (3aa): yield: 86%; 76.6 mg, light yellow solid; ¹H NMR (400 MHz, DMSO-*d*₆) δ : 8.15 (dd, J = 8.0 Hz, 1.6 Hz, 2H), 7.54-7.50 (m, 7H), 7.49-7.46 (m, 3H), 7.45-7.39 (m, 3H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ : 160.7, 154.5, 137.9, 130.5, 130.1, 129.5, 129.2, 128.8, 128.7, 128.6, 127.6, 126.0, 125.8; LRMS (EI 70 ev) m/z (%): 297 (M⁺, 100); HRMS m/z (ESI) calcd for C₂₀H₁₆N₃ (M + H)⁺ 298.1338, found 298.1335.

1,5-Diphenyl-3-p-tolyl-1H-1,2,4-triazole (3 ab): yield: 69%; 64.4 mg, light white solid; ¹H NMR (400 MHz, DMSO- d_6) δ : 8.02 (d, J = 7.6 Hz, 2H), 7.51-7.40 (m, 10H), 7.33 (d, J = 8.0 Hz, 2H), 2.37 (s, 3H); ¹³C NMR (100 MHz, DMSO- d_6) δ : 160.8, 154.4, 139.1, 137.9, 130.1, 129.5, 129.4, 129.2, 128.7, 128.6, 127.7, 127.6, 126.0, 125.8, 21.0; LRMS (EI 70 ev) m/z (%): 311 (M⁺, 100); HRMS m/z (ESI) calcd for C₂₁H₁₈N₃ (M + H)⁺ 312.1495, found 312.1490.

3-(4-Methoxyphenyl)-1,5-diphenyl-1H-1,2,4-triazole (**3ac**): yield: 70%; 68.7 mg, light yellow solid; ¹H NMR (400 MHz, DMSO- d_6) δ : 8.07 (dd, J = 6.8 Hz, 2.0 Hz, 2H), 7.52-7.48 (m, 5H), 7.48-7.45 (m, 3H), 7.44-7.39 (m, 2H), 7.08 (dd, J = 6.8 Hz, 2.0 Hz, 2H), 3.82 (s, 3H); ¹³C NMR (100 MHz, DMSO- d_6) δ : 160.7, 160.3, 154.3, 138.0, 130.0, 129.5, 129.1, 128.7, 128.6, 127.7, 127.5, 125.7, 123.0, 114.2, 55.2; LRMS (EI 70 ev) m/z (%): 327 (M⁺, 100); HRMS m/z (ESI) calcd for C₂₁H₁₈N₃O (M + H)⁺ 328.1444, found 328.1440.

3-(4-Fluorophenyl)-1,5-diphenyl-1H-1,2,4-triazole (3ad): yield: 49%; 46.3 mg, light yellow solid; ¹H NMR (400 MHz, DMSO- d_6) δ : 8.17 (dd, J = 8.8 Hz, 5.6 Hz, 2H), 7.53-7.45 (m, 8H), 7.43-7.39 (m, 2H), 7.36-7.32 (m, 2H); ¹³C NMR (100 MHz, DMSO- d_6) δ : 164.2 (d, J = 244.9 Hz), 159.9, 154.6, 137.8, 130.2, 129.5, 129.3, 128.7, 128.6 (d, J = 37.0 Hz), 128.2, 127.5, 127.0 (d, J = 2.8 Hz), 125.8, 115.9 (d, J = 21.7 Hz); LRMS (EI 70 ev) m/z (%): 315 (M⁺, 100); HRMS m/z(ESI) calcd for C₂₀H₁₅FN₃ (M + H)⁺ 316.1244, found 316.1240.

3-(4-Chlorophenyl)-1,5-diphenyl-1H-1,2,4-triazole (3ae): yield: 62%; 61.6 mg, light yellow solid; ¹H NMR (400 MHz, DMSO- d_6) δ : 8.13 (dd, J = 6.4 Hz, 1.6 Hz, 2H), 7.57 (dd, J = 6.8 Hz, 2.0 Hz, 2H), 7.52-7.45 (m, 8H), 7.43-7.39 (m, 2H); ¹³C NMR (100 MHz, DMSO- d_6) δ : 159.8, 154.7, 137.8, 134.2, 130.2, 129.5, 129.3, 129.3, 129.0, 128.7, 128.6, 127.7, 127.4, 125.8; LRMS (EI 70 ev) m/z(%): 331 (M⁺, 100); HRMS m/z (ESI) calcd for C₂₀H₁₅ClN₃ (M + H)⁺ 332.0949, found 332.0951.

3-(4-Bromophenyl)-1,5-diphenyl-1H-1,2,4-triazole (3af): yield: 62%; 69.8 mg, light yellow solid; ¹H NMR (400 MHz, DMSO- d_6) δ : 8.07-8.04 (m, 2H), 7.73-7.70 (m, 2H), 7.53-7.45 (m, 8H), 7.44-7.40 (m, 2H); ¹³C NMR (100 MHz, DMSO- d_6) δ : 159.8, 154.7, 137.8, 131.9, 130.3, 129.7, 129.6, 129.4, 128.7, 128.6, 128.0, 127.4, 125.8, 122.9; LRMS (EI 70 ev) *m/z* (%): 375 (M⁺, 100); HRMS *m/z* (ESI) calcd for C₂₀H₁₅BrN₃ (M + H)⁺ 376.0443, found 376.0450.

4-(1,5-Diphenyl-1H-1,2,4-triazol-3-yl)benzonitrile (3 ag): yield: 55%; 53.1 mg, light yellow solid; ¹H NMR (400 MHz, DMSO-*d*₆) δ : 8.28 (dd, *J* = 6.8 Hz, 2.0 Hz, 2H), 7.99 (dd, *J* = 6.8 Hz, 1.6 Hz, 2H), 7.55-7.47 (m, 8H), 7.45-7.41 (m, 2H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ : 159.3, 155.1, 137.7, 134.7, 133.1, 130.4, 129.7, 129.6, 128.8, 128.7, 127.3, 126.7, 125.9, 118.7, 111.9; LRMS (EI 70 ev) *m/z* (%): 322 (M⁺, 100); HRMS *m/z* (ESI) calcd for C₂₁H₁₅N₄ (M + H)⁺ 323.1291, found 323.1295.

Methyl 4-(1,5-diphenyl-1H-1,2,4-triazol-3-yl)benzoate (3ah): yield: 73%; 77.7 mg, light yellow solid; ¹H NMR (400 MHz, DMSO- d_6) δ : 8.27 (dd, J = 6.8 Hz, 1.6 Hz, 2H), 8.11 (dd, J = 6.4 Hz, 1.6 Hz, 2H), 7.55-7.41 (m, 10H), 3.89 (s, 3H); ¹³C NMR (100 MHz, DMSO- d_6) δ : 166.0, 159.8, 155.0, 137.8, 134.7, 130.4, 130.3, 129.9, 129.7, 129.5, 128.8, 128.7, 127.4, 126.3, 125.9, 52.3; LRMS (EI 70 ev) m/z (%): 355 (M⁺, 100); HRMS m/z (ESI) calcd for C₂₂H₁₈N₃O₂ (M + H)⁺ 356.1393, found 356.1397.

3-(2-Chlorophenyl)-1,5-diphenyl-1H-1,2,4-triazole (3ai): yield: 50%; 49.7 mg, light yellow solid; ¹H NMR (400 MHz, DMSO- d_6) δ : 8.04-8.02 (m, 1H), 7.64-7.62 (m, 1H), 7.54-7.47 (m, 10H), 7.45-7.41 (m, 2H); ¹³C NMR (100 MHz, DMSO- d_6) δ : 159.4, 153.9, 137.8, 131.7, 131.5, 131.5, 130.9, 130.8, 130.3, 129.6, 129.4, 128.8, 128.7, 127.4, 127.4, 125.8; LRMS (EI 70 ev) m/z (%): 331 (M⁺, 100); HRMS m/z (ESI) calcd for C₂₀H₁₅ClN₃ (M + H)⁺ 332.0949, found 332.0956.

3-(3-Chlorophenyl)-1,5-diphenyl-1H-1,2,4-triazole (3aj): yield: 48%; 47.7 mg, light yellow solid; ¹H NMR (400 MHz, DMSO- d_6) δ: 8.09-8.06 (m, 2H), 7.56-7.51 (m, 6H), 7.51-7.47 (m, 3H), 7.47-7.40 (m, 3H); ¹³C NMR (100 MHz, DMSO- d_6) δ: 159.4, 154.8, 137.8, 133.7, 132.5, 131.0, 130.4, 129.7, 129.6, 129.5, 128.8, 128.7, 127.4, 125.9, 125.5, 124.6; LRMS (EI 70 ev) m/z (%): 331 (M⁺, 100); HRMS m/z (ESI) calcd for C₂₀H₁₅ClN₃ (M + H)⁺ 332.0949, found 332.0953.

3-(Furan-2-yl)-1,5-diphenyl-1H-1,2,4-triazole (3ak): yield: 52%; 44.8 mg, brown red oil; ¹H NMR (400 MHz, DMSO-*d*₆) δ : 7.87-7.86 (m, 1H), 7.53-7.40 (m, 10H), 7.07 (dd, *J* = 3.6 Hz, 0.8 Hz, 1H), 6.68 (dd, *J* = 3.6 Hz, 2.0 Hz, 1H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ : 154.3, 154.3, 145.7, 144.2, 137.7, 130.3129.6, 129.3, 128.7, 128.7, 127.3, 125.8, 111.8, 109.9; LRMS (EI 70 ev) *m/z* (%): 287 (M⁺,100); HRMS *m/z* (ESI) calcd for C₁₈H₁₄N₃O (M + H)⁺ 288.1131, found 288.1136.

1,5-Diphenyl-3-styryl-1H-1,2,4-triazole (3 al): yield: 51%; 49.4 mg, light yellow solid; ¹H NMR (400 MHz, DMSO-*d*₆) δ : 7.72 (d, *J* = 7.2 Hz, 2H), 7.66 (d, *J* = 16.4 Hz, 1H), 7.52-7.47 (m, 5H), 7.46-7.39 (m, 7H), 7.36-7.32 (m, 1H), 7.29 (d, *J* = 16.4 Hz, 1H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ : 160.5, 154.2, 137.9, 136.0, 133.8, 130.2, 129.6, 129.2, 128.9, 128.8, 128.7, 128.6, 127.6, 127.1, 125.7, 117.5; LRMS (EI 70 ev) *m*/*z* (%): 323 (M⁺, 100); HRMS *m*/*z* (ESI) calcd for C₂₂H₁₈N₃ (M + H)⁺ 324.1495, found 324.1490.

3-*tert*-**Butyl-1,5-diphenyl-1H-1,2,4-triazole (3am)**: yield: 80%; 66.5 mg, white solid; ¹H NMR (400 MHz, DMSO- d_6) δ : 7.49-7.45 (m, 3H), 7.44-7.35 (m, 7H), 1.39 (s, 9H); ¹³C NMR (100 MHz, DMSO- d_6) δ : 170.7, 153.4, 138.0, 129.9, 129.4, 128.8, 128.6, 128.6, 128.0, 125.6, 32.5, 29.5; LRMS (EI 70 ev) m/z (%): 277 (M⁺, 100); HRMS m/z (ESI) calcd for C₁₈H₂₀N₃ (M + H)⁺ 278.1651, found 278.1649.

1-(4-Methoxyphenyl)-3,5-diphenyl-1H-1,2,4-triazole (3an): yield: 74%; 72.6 mg, light yellow solid; ¹H NMR (400 MHz, DMSO-*d*₆) δ: 8.13-8.10 (m, 2H), 7.54-7.50 (m, 5H), 7.49-7.40 (m, 5H), 7.06 (dd, J = 6.8 Hz, 2.4 Hz, 2H), 3.81 (s, 3H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ: 160.4, 159.6, 154.5, 130.8, 130.6, 130.0, 129.5, 128.8, 128.7, 128.6, 127.7, 127.4, 125.9, 114.6, 55.5; LRMS (EI 70 ev) m/z (%): 327 (M⁺, 100); HRMS m/z (ESI) calcd for C₂₁H₁₈N₃O (M + H)⁺ 328.1444, found 328.1438.

1-(4-Chlorophenyl)-3,5-diphenyl-1H-1,2,4-triazole (3ao): yield: 55%; 54.6 mg, light yellow solid; ¹H NMR (400 MHz, DMSO- d_6) δ : 8.13 (dd, J = 8.4 Hz, 2.0 Hz, 2H), 7.60 (dd, J = 6.8 Hz, 2.4 Hz, 2H), 7.54-7.45 (m, 10H); ¹³C NMR (100 MHz, DMSO- d_6) δ : 160.9, 154.7, 136.7, 133.6, 130.3, 129.6, 129.6, 128.9, 128.9, 128.8, 128.7, 127.5, 127.4, 126.0; LRMS (EI 70 ev) m/z (%): 331 (M⁺, 100); HRMS m/z (ESI) calcd for C₂₀H₁₅ClN₃ (M + H)⁺ 332.0949, found 332.0950.

1-(4-Bromophenyl)-3,5-diphenyl-1H-1,2,4-triazole (3ap): yield: 61%; 68.6 mg, brown yellow solid; ¹H NMR (400 MHz, DMSO- d_6) δ : 8.13 (dd, J = 8.4 Hz, 2.0 Hz, 2H), 7.73 (dd, J = 6.4 Hz, 2.0 Hz, 2H), 7.54-7.52 (m, 3H), 7.50-7.47 (m, 4H), 7.47-7.42 (m, 3H); ¹³C NMR (100 MHz, DMSO- d_6) δ : 160.9, 154.7, 137.1, 132.5, 130.3, 130.3, 129.6, 128.9, 128.8, 128.7, 127.7, 127.4, 126.0, 122.1; LRMS (EI 70 ev) m/z (%): 375 (M⁺, 100); HRMS m/z (ESI) calcd for C₂₀H₁₅BrN₃ $(M + H)^+$ 376.0443, found 376.0449.

1,3-Diphenyl-5-p-tolyl-1H-1,2,4-triazole (3ba): yield: 77%; 71.8 mg, light yellow solid; ¹H NMR (400 MHz, DMSO-*d*₆) δ : 8.13 (d, *J* = 7.6 Hz, 2H), 7.52-7.47 (m, 8H), 7.40 (d, *J* = 8.0 Hz, 2H), 7.22 (d, *J* = 7.6 Hz, 2H), 2.31 (s, 3H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ : 160.6, 154.6, 140.0, 138.0, 130.5, 129.5, 129.5, 129.2, 128.8, 128.7, 128.6, 126.0, 125.8, 124.7, 20.9; LRMS (EI 70 ev) *m/z* (%): 311 (M⁺, 100); HRMS *m/z* (ESI) calcd for C₂₁H₁₈N₃ (M + H)⁺ 312.1495, found 312.1488.

5-(4-Methoxyphenyl)-1,3-diphenyl-1H-1,2,4-triazole (**3ca**): yield: 71%; 69.7 mg, light yellow solid; ¹H NMR (400 MHz, DMSO-*d*₆) δ: 8.13-8.11 (m, 2H), 7.54-7.51 (m, 4H), 7.49-7.43 (m, 6H), 6.97 (dd, J = 6.8 Hz, 2.0 Hz, 2H), 3.77 (s, 3H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ: 160.5, 160.5, 154.4, 138.1, 130.6, 130.2, 129.5, 129.4, 129.2, 128.8, 126.0, 125.8, 119.7, 114.1, 55.3; LRMS (EI 70 ev) m/z (%): 327 (M⁺, 100); HRMS m/z (ESI) calcd for C₂₁H₁₈N₃O (M + H)⁺ 328.1444, found 328.1443.

5-(4-Fluorophenyl)-1,3-diphenyl-1H-1,2,4-triazole (3da): yield: 70%; 66.2 mg, light yellow solid; ¹H NMR (400 MHz, DMSO-*d*₆) δ:8.14-8.11 (m, 2H), 7.59-7.56 (m, 2H), 7.55-7.51 (m, 4H), 7.50-7.45 (m, 4H), 7.30-7.24 (m, 2H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ: 162.8 (d, *J* = 247.0 Hz), 160.7, 153.7, 137.8, 131.2, 131.1, 130.4, 129.6 (d, *J* = 2.7 Hz), 129.3, 128.8, 126.0, 125.8, 124.4 (d, *J* = 3.1 Hz), 115.7 (d, *J* = 22.0 Hz); LRMS (EI 70 ev) *m/z* (%): 315 (M⁺,100); HRMS *m/z* (ESI) calcd for C₂₀H₁₅FN₃ (M + H)⁺ 316.1244, found 316.1241.

5-(4-Chlorophenyl)-1,3-diphenyl-1H-1,2,4-triazole (3ea): yield: 66%; 65.5 mg, light yellow solid; ¹H NMR (400 MHz, DMSO- d_6) δ : 8.13 (dd, J = 8.4 Hz, 1.6 Hz, 2H), 7.55-7.52 (m, 3H), 7.51-7.47 (m, 9H); ¹³C NMR (100 MHz, DMSO- d_6) δ : 160.8, 153.6, 137.7, 135.1, 130.5, 130.3, 129.7, 129.6, 129.4, 128.9, 128.8, 126.4, 126.0, 125.8; LRMS (EI 70 ev) m/z (%): 331 (M⁺, 100); HRMS m/z (ESI) calcd for C₂₀H₁₅ClN₃ (M + H)⁺ 332.0949, found 332.0954.

1,3-Diphenyl-5-o-tolyl-1H-1,2,4-triazole (3fa): yield: 75%; 70.0 mg, light yellow solid; ¹H NMR (400 MHz, DMSO-*d*₆) δ : 8.16-8.13 (m, 2H), 7.54-7.47 (m, 3H), 7.45-7.35 (m, 7H), 7.32-7.24 (m, 2H), 2.10 (s, 3H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ : 160.7, 154.4, 137.4, 137.0, 130.5, 130.2, 130.2, 129.5, 129.3, 128.8, 128.5, 128.1, 126.0, 125.9, 124.1, 19.3; LRMS (EI 70 ev) *m/z* (%): 311 (M⁺, 100); HRMS *m/z* (ESI) calcd for C₂₁H₁₈N₃ (M + H)⁺ 312.1495, found 312.1491.

1,3-Diphenyl-5-m-tolyl-1H-1,2,4-triazole (3ga): yield: 65%; 60.6 mg, light yellow solid; ¹H NMR (400 MHz, DMSO-*d*₆) δ : 8.13 (d, *J* = 7.6 Hz, 2H), 7.54-7.43 (m, 9H), 7.29-7.24 (m, 2H), 7.19-7.17 (m, 1H), 2.28 (s, 3H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ : 160.7, 154.6, 138.1, 138.0, 130.8, 130.6, 130.5, 129.6, 129.5, 129.3, 129.3, 128.9, 128.5, 127.5, 126.0, 125.8, 20.9; LRMS (EI 70 ev) *m*/*z* (%): 311 (M⁺, 100); HRMS *m*/*z* (ESI) calcd for C₂₁H₁₈N₃ (M + H)⁺ 312.1495, found 312.1493.

5-(Naphthalen-1-yl)-1,3-diphenyl-1H-1,2,4-triazole (3ha): yield: 51%; 53.1 mg, brown red oil; ¹H NMR (400 MHz, DMSO- d_6) δ : 8.21-8.19 (m, 2H), 8.09 (dd, J = 29.2 Hz, 8.0 Hz, 2H), 7.82 (d, J = 8.4 Hz, 1H), 7.63 (dd, J = 7.2 Hz, 1.2 Hz, 1H), 7.58-7.49 (m, 6H), 7.36-7.30 (m, 5H); ¹³C NMR (100 MHz, DMSO- d_6) δ :160.9, 153.6, 137.4, 133.0, 130.8, 130.6, 130.5, 129.6, 129.2, 129.2, 128.9, 128.6, 128.4, 127.3, 126.6, 126.1, 125.6, 125.1, 124.8, 124.3; LRMS (EI 70ev) m/z (%): 347 (M⁺,100); HRMS m/z (ESI) calcd for C₂₄H₁₈N₃ (M + H)⁺ 348.1495, found 348.1499.

1,3-Diphenyl-5-(thiophen-2-yl)-1H-1,2,4-triazole (3ia): yield: 67%; 60.9 mg, white yellow solid; ¹H NMR (400 MHz, DMSO- d_6) δ : 8.11-8.08 (m, 2H), 7.75 (dd, J = 5.2 Hz, 1.2 Hz, 1H), 7.66-7.62 (m, 5H), 7.53-7.47 (m, 3H), 7.07 (dd, J = 4.8 Hz, 3.6 Hz, 1H), 6.99 (dd, J = 3.6 Hz, 1.2 Hz, 1H); ¹³C NMR (100 MHz, DMSO- d_6) δ : 160.6, 149.9, 137.5, 130.3, 130.2, 130.1, 129.9, 129.6, 129.6, 128.9, 128.8, 128.0, 127.0, 126.0; LRMS (EI 70 ev) m/z (%): 303 (M⁺, 100); HRMS m/z(ESI) calcd for C₁₈H₁₄N₃S (M + H)⁺ 304.0902, found 304.0908.

Methyl 2,5-diphenyl-2H-1,2,4-triazole-3-carboxylate (3ja):

yield: 49%; 41.0 mg, light brown red solid; ¹H NMR (400 MHz, DMSO- d_6) δ : 8.10-8.08 (m, 2H), 7.66-7.64 (m, 2H), 7.58-7.50 (m, 6H), 3.84 (s, 3H); ¹³C NMR (100 MHz, DMSO- d_6) δ : 160.7, 157.4, 145.4, 137.9, 130.0, 129.7, 129.6, 129.0, 128.9, 126.1, 125.9, 52.9; LRMS (EI 70 ev) m/z (%): 279 (M⁺, 100); HRMS m/z (ESI) calcd for C₁₆H₁₄N₃O₂ (M + H)⁺ 280.1080, found 280.1084.

1,3-Diphenyl-5-propyl-1H-1,2,4-triazole (3ka): yield: 78%; 61.5 mg, light yellow solid; ¹H NMR (400 MHz, DMSO- d_6) δ : 8.07 (dd, J = 8.4 Hz, 2.0 Hz, 2H), 7.62-7.42 (m, 8H), 2.80 (dd, J = 10.0 Hz, 7.6 Hz, 2H), 1.76-1.67 (m, 2H),0.88 (t, J = 7.4 Hz, 3H); ¹³C NMR (100 MHz, DMSO- d_6) δ : 160.2, 156.7, 137.2, 130.8, 129.5, 129.2, 128.9, 128.7, 125.8, 125.1, 27.8, 20.4,13.5; LRMS (EI 70 ev) m/z (%): 263 (M⁺, 100); HRMS m/z (ESI) calcd for C₁₇H₁₈N₃ (M + H)⁺ 264.1495, found 264.1498.

5-cyclopropyl-1,3-diphenyl-1H-1,2,4-triazole (**3la**): yield: 65%; 50.9 mg, light yellow oil; ¹H NMR (400 MHz, DMSO-*d*₆) δ : 7.90 (dd, *J* = 1.6 Hz, 1.2 Hz, 2H), 7.57 (d, *J* = 7.6 Hz, 2H), 7.50 (t, *J* = 7.6 Hz, 2H), 7.41 (dd, *J* = 7.2 Hz, 5.2 Hz, 1H), 7.35-7.30 (m, 3H), 1.92-1.87 (m, 1H), 1.01-0.98 (m, 2H), 0.97-0.92 (m, 2H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ : 160.0, 158.3, 137.1, 130.7, 129.6, 129.3, 128.8, 128.7, 125.9, 124.7, 9.2, 7.3; LRMS (EI 70 ev) *m/z* (%): 261 (M⁺, 100); HRMS *m/z* (ESI) calcd for C₁₇H₁₆N₃ (M + H)⁺ 262.1344, found 262.1341.

1,3-Diphenyl-5-vinyl-1H-1,2,4-triazole (3ma): yield: 61%; 45.2 mg, light yellow oil; ¹H NMR (400 MHz, DMSO-*d*₆) δ : 8.11-8.09 (m, 2H), 7.65-7.57 (m, 5H), 7.53-7.45 (m, 3H), 6.68 (dd, *J* = 16.8 Hz, 10.8 Hz, 1H), 6.40 (dd, *J* = 17.2 Hz, 2.0 Hz, 1H), 5.75 (dd, *J* = 11.2 Hz, 2.0 Hz, 1H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ : 160.7, 152.7, 136.6, 130.4, 129.7, 129.5, 129.3, 128.8, 126.0, 125.4, 124.0, 121.7; LRMS (EI 70 ev) *m*/*z* (%): 247 (M⁺, 100); HRMS *m*/*z* (ESI) calcd for C₁₆H₁₄N₃ (M + H)⁺ 248.1182, found 248.1185.

5-Ethynyl-1,3-diphenyl-1H-1,2,4-triazole (3na): yield: 52%; 38.2 mg, light yellow solid; ¹H NMR (400 MHz, DMSO-*d*₆) δ : 8.08 (dd, *J* = 8.0 Hz, 2.0 Hz, 2H), 7.84-7.82 (m, 2H), 7.65-7.61 (m, 2H), 7.59-7.49 (m, 4H), 5.08 (s, 1H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ : 161.1, 137.9, 136.5, 130.0, 129.6, 129.5, 129.4, 129.0, 126.1, 123.3, 89.5, 71.1; LRMS (EI 70 ev) *m/z* (%): 245 (M⁺, 100); HRMS *m/z* (ESI) calcd for C₁₆H₁₂N₃ (M + H)⁺ 246.1025, found 246.1028.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.tet.2021.132111.

Supporting information available

Characterization data of **3** including the ¹H and ¹³C NMR data.

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