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Enyne synthesis through a modified Sonogashira cross-coupling reaction catalyzed by cyclopalladated complexes

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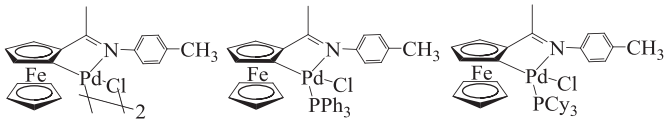
ABSTRACT

A series of conjugated enynes were successfully synthesized by the palladacycle-catalyzed modified Sonogashira cross-coupling reaction of β-bromostyrene and terminal alkynes. The reaction proceeds smoothly in DMSO at 40 °C to give the corresponding products in moderate to excellent yields. This catalytic system is tolerant to a broad range of functional groups on the substrates. Moreover, the products were furnished as specific *E* isomers. We also found that the product of the reaction between (*E*)-β-bromostyrene and 2-methyl-3-butyn-2-ol is diarylated enyne in the presence of excess Cs_2CO_3.

1. Introduction

The conjugated enyne is a structure of high interest owing to its presence in many natural products and biologically active compounds[1]. For example, the skeleton of conjugated enyne is present in the molecule of *trans*-ku-mausyne, Calicheamycin, and Terbinafine[2]. So development of a method for synthesis of these compounds becomes one of the major directions in organic chemistry. Usually Sonogashira cross-coupling reaction[3–7] catalyzed by the palladium complexes was used to prepare them. Moreover, there were large numbers of publications, concerning Sonogashira reaction catalyzed by other metal complexes, e.g., Ni[8], Cu(I),[9] and Cu(II)[10] complexes, but the reactions usually proceed under severe reaction conditions or with phosphorous-containing additives[11–13].

Currently, the catalyst of cyclopalladated complexes became one of the most popular catalysts in catalytic chemistry[14–16]. Especially, the palladium complexes containing ferrocenyl fragment have been the most developed and extensively studied in our laboratory[17–20], owing to their structural versatility, stability, and easily synthetic accessibility. They have a high catalytic activity in Heck[21], Suzuki,[22,23] and other reactions[24–26]. However, these palladium complexes have not applied in modified Sonogashira reaction using β-bromostyrene as substrate. So in this paper, we will report the results of palladacycle-catalyzed cross-coupling reaction of β-bromostyrene with terminal alkynes, which is a modified Sonogashira reaction by using the palladacycles **I**, **II** or **III** as a catalyst.



2. Results and discussion

For optimization of the reaction conditions, we chose the cross-coupling of (*E*)-β-bromostyrene with phenylactylene as model reaction, and the effects of the base, catalyst, solvent, copper salt additive (Cu(I)X) and temperature were examined. The results were summarized in Table 1. It was found that this reaction proceeded in the presence of weak base Cs_2CO_3 (entry 4) giving product **3a**. Similar results have also been reported by other authors[27], and in this case, the ion Cs^+ could be considered as an auxiliary component for the reaction. In addition, without palladium complex, CuI appeared no catalytic activity (entry 8). Otherwise, the yield of product **3a** catalyzed by Cat.**I** reached to 70% at 100 °C (entry 6). We found that the polarity of aprotic solvents have dramatic influence on Sonogashira reaction, i.e., the stronger polarity of the solvent, the higher yield of the reaction (entries 6 and 10–13). However,

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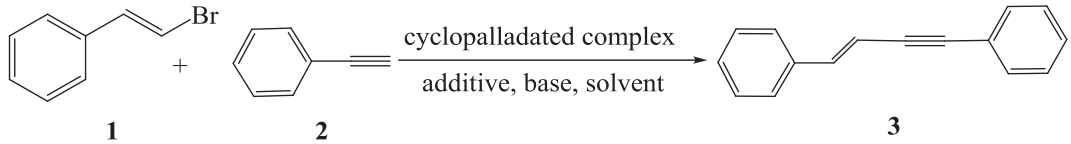
Table 1
Optimization of the reaction conditions for the formation of **3a**[a]



1 **2** **3**

Entry	Catalyst	Additive	Base	Solvent	T (°C)	Yield[e] [%]
1	Cat.**III**	CuI	K_2CO_3	DMF	100	12
2	Cat.**III**	CuI	CsF	DMF	100	NR[b]
3	Cat.**III**	CuI	NaOAc	DMF	100	NR
4	Cat.**III**	CuI	Cs_2CO_3	DMF	100	52
5	Cat.**III**	CuI	K_3PO_4	DMF	100	NR[b]
6	Cat.**I**	CuI	Cs_2CO_3	DMF	100	70
7	Cat.**II**	CuI	Cs_2CO_3	DMF	100	65
8	—	CuI	Cs_2CO_3	DMF	100	NR[b]
9	Cat.**I**	CuBr	Cs_2CO_3	DMF	100	76
10	Cat.**I**	CuBr	Cs_2CO_3	DMSO	100	86
11	Cat.**I**	CuBr	Cs_2CO_3	1,4-Dioxane	100	22
12	Cat.**I**	CuBr	Cs_2CO_3	THF	66	21
13	Cat.**I**	CuBr	Cs_2CO_3	Acetone	65	64
14	Cat.**I**	CuBr	Cs_2CO_3	MeOH	57	38
15	Cat.**I**	CuCl	Cs_2CO_3	DMSO	100	73
16	Cat.**I**	CuI	Cs_2CO_3	DMSO	100	82
17	Cat.**I**	—	Cs_2CO_3	DMSO	100	17
18	Cat.**I**	CuBr	Cs_2CO_3	DMSO	120	77
19	Cat.**I**	CuBr	Cs_2CO_3	DMSO	80	80
20	Cat.**I**	CuBr	Cs_2CO_3	DMSO	60	82
21	**Cat.I**	**CuBr**	**Cs_2CO_3**	**DMSO**	**40**	**91**
22	Cat.**I**	CuBr	Cs_2CO_3	DMSO	30	27
23	Cat.**I**[c]	CuBr	Cs_2CO_3	DMSO	40	57
24	Cat.**I**[d]	CuBr	Cs_2CO_3	DMSO	40	85

The values in bold represent the the optimized condition for the studied reaction.
[a] Reaction conditions: catalyst (1 mol %), additive (20 mol %), base (1 mmol), (*E*)-β-bromostyrene (0.5 mmol), phenylacetylene (0.75 mmol), solvent (3 mL), 24 h, N_2.
[b] No reaction.
[c] Catalyst (0.5 mol %).
[d] Catalyst (2 mol %).
[e] Isolated yield.

using the protic solvents, yield of product **3a** decreased drastically to 38% (entry 14). In the absence of CuX, the reaction proceeded slowly (24 h) with a low yield (17%, entry17), and the optimal activity of CuX for this reaction is CuBr (entries 10 and 15–16). Furthermore, it was found that the reaction temperature has certain influence on the reaction yields, but this relationship is not completely regular (entries 10 and 18–22). If the loading of catalyst **I** was reduced (0.5 mol %, entry 23) or increased (2 mol %, entry 24), the corresponding yields were reduced. Therefore, the optimized conditions of palladacycle-catalyzed cross-coupling reaction of (*E*)-β-bromostyrene with terminal alkynes includes the following parameters: β-bromostyrene (0.5 mmol), terminal alkyne (0.75 mmol), Cat.**I** (1 mol %), CuBr (20 mol %), Cs_2CO_3 (1 mmol), DMSO (3 mL), N_2, at 40 °C in 24 h.

Under these optimized conditions, we examined the substrate scope. The results indicated that the cyclopalladated complex I constituted a highly efficient catalyst for Sonogashira reaction of bromostyrene with terminal alkynes (Table 2). Both the electron-rich (deactivated) and electron-poor (activated) terminal alkynes could be converted to the desirable products **3a**–**i** (entries 1–9), but yield of products containing electron-rich groups (entries 1–6) is appreciably higher than the products containing electron-poor groups (entries 7–9). Moreover, the location of the radical contained in terminal alkynes also influences on the yield of the products. Product yields of *p*-substituted terminal alkynes are always higher than that of *o*- and *m*-substituted compounds. However, the (*Z*)-β-bromostyrene **1b** could not react with phenylacetylene **2a** (entry 10).

Despite the conditions of this reaction have been optimized, it was found that catalyst **I** in this case does not have any catalytic activity in Sonogashira reaction between (*E*)-β-bromostyrene and alkyl terminal alkynes (Table 3, entries 1–3). Using the catalyst **II**, the reactions of (*E*)-β-bromostyrene with alkyl terminal alkynes**2k** and **2l** proceeded smoothly, and the corresponding yield of these reactions rapidly increased to 75% and 89% (entries 4–6) However, when we studied the reaction of (*E*)-β-bromostyrene with 2-methyl-3-butyn-2-ol (**2m**), it was found that the product of this reaction was compound **3m**. We assumed that this reaction might proceed with formation of unstable monoarylated enyne **4** at first and then excess Cs_2CO_3 led to the formation of compound **3m**[28] (Fig. 1).

3. Conclusion

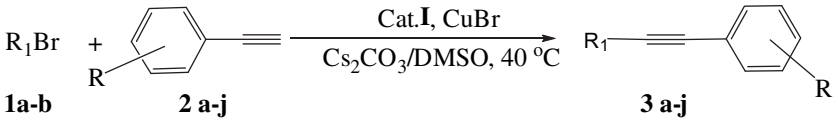
In summary, we have found a mild and efficient catalytic system for the direct synthesis of conjugated enynes by using the palladacycle-catalyzed coupling of β-bromostyrene and alkynes. We have studied Sonogashira coupling reaction with the palladacycle catalysts, which were synthesized and discovered as a popular catalyst in our laboratory. This catalytic system has wide functional-group tolerance on substrates. This novel catalytic system provides a very useful supplement to the known methods for enyne synthesis in terms of economy, ecology, and easy workup.

4. Experimental

4.1. General methods

^1H NMR and ^{13}C NMR spectra were recorded on a Bruker DPX 400 instrument using $CDCl_3$ as the solvent and TMS as the internal

Table 2
Preparation of enynes from bromostyrene with terminal alkynes[a]



1a-b **2 a-j** **3 a-j**

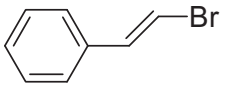
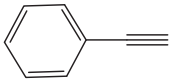
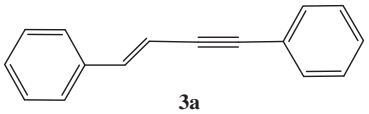
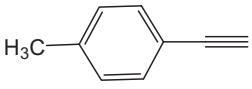
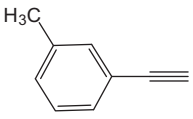
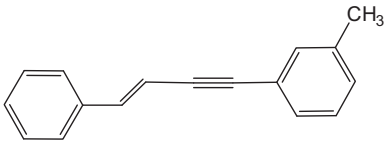
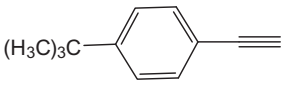
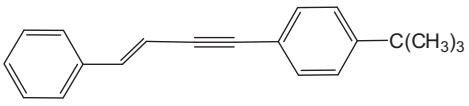
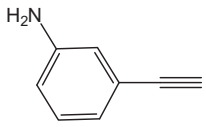
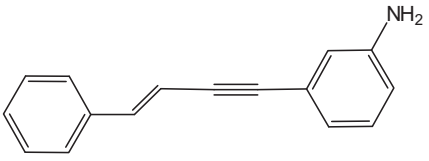
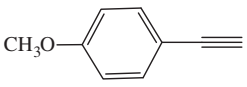
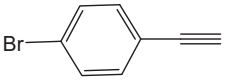
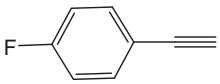
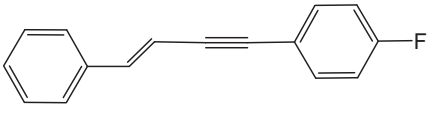
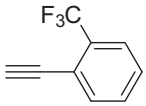
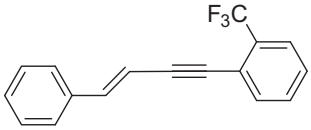
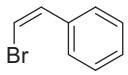
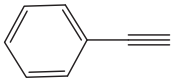
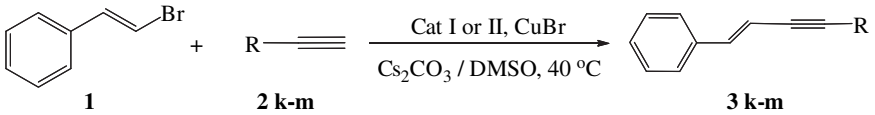
Entry	Catalyst	R_1Br	Alkyne	Product	Yield[c] [%]
1	Cat.**I**	 **1a**	 **2a**	 **3a**	91
2	Cat.**I**	**1a**	 **2b**	 **3b**	94
3	Cat.**I**	**1a**	 **2e**	 **3c**	83
4	Cat.**I**	**1a**	 **2d**	 **3d**	75
5	Cat.**I**	**1a**	 **2e**	 **3e**	82
6	Cat.**I**	**1a**	 **2f**	 **3f**	81
7	Cat.**I**	**1a**	 **2g**	 **3g**	48
8	Cat.**I**	**1a**	 **2h**	 **3h**	77

Table 2 (*continued*)

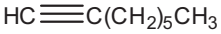
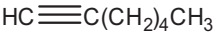
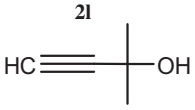
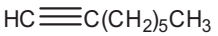
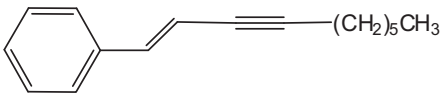
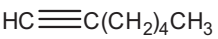
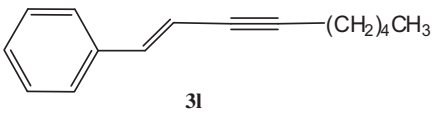
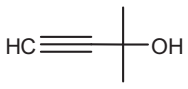
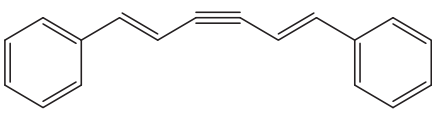
Entry	Catalyst	R_1Br	Alkyne	Product	Yield[c] [%]
9	Cat.**I**	**1a**	 **2i**	 **3i**	49
10	Cat.**I**	 **1b**	 **2a**	—	NR[b]

[a] Reaction conditions: β-bromostyrene (0.5 mmol), terminal alkyne (0.75 mmol), catalyst (1 mol %), CuBr (20 mol %), Cs_2CO_3 (1 mmol) in DMSO (3 mL) at 40 °C for 24 h, N_2.
[b] No reaction.
[c] Isolated yield.

Table 3
Preparation of enynes from (*E*)-β-bromostyrene with terminal alkynes[a]



1 **2 k-m** **3 k-m**

Entry	Catalyst	Alkyne	Product	Yield [%]
1	Cat.**I**	 **2k**	—	NR[b]
2	Cat.**I**	 **2l**	—	NR[b]
3	Cat.**I**	 **2m**	—	NR[b]
4	Cat.**II**	 **2k**	 **3k**	75
5	Cat.**II**	 **2l**	 **3l**	89
6	Cat.**II**	 **2m**	 **3m**	77

[a] Reaction conditions: (*E*)-β-bromostyrene (0.5 mmol), terminal alkyne (0.75 mmol), catalyst (1 mol %), CuBr (20 mol %), Cs_2CO_3 (1 mmol) in DMSO (3 mL) at 40 °C for 24 h, N_2.
[b] No reaction.

standard. EA data were recorded on Elementar Vario ELIIIspectrometer. Melting points were measured using a WC-1 microscopic apparatus and are uncorrected. Preparative TLC was performed on dry silica gel plates developed with petroleum/EtOAc.

All solvents were purified by the standard methods. (*E*)-β-bromostyrene and all the alkynes were purchased from commercial sources and generally used without further purification. (*Z*)-β-Bromostyrene was prepared according to the reported procedure[29].

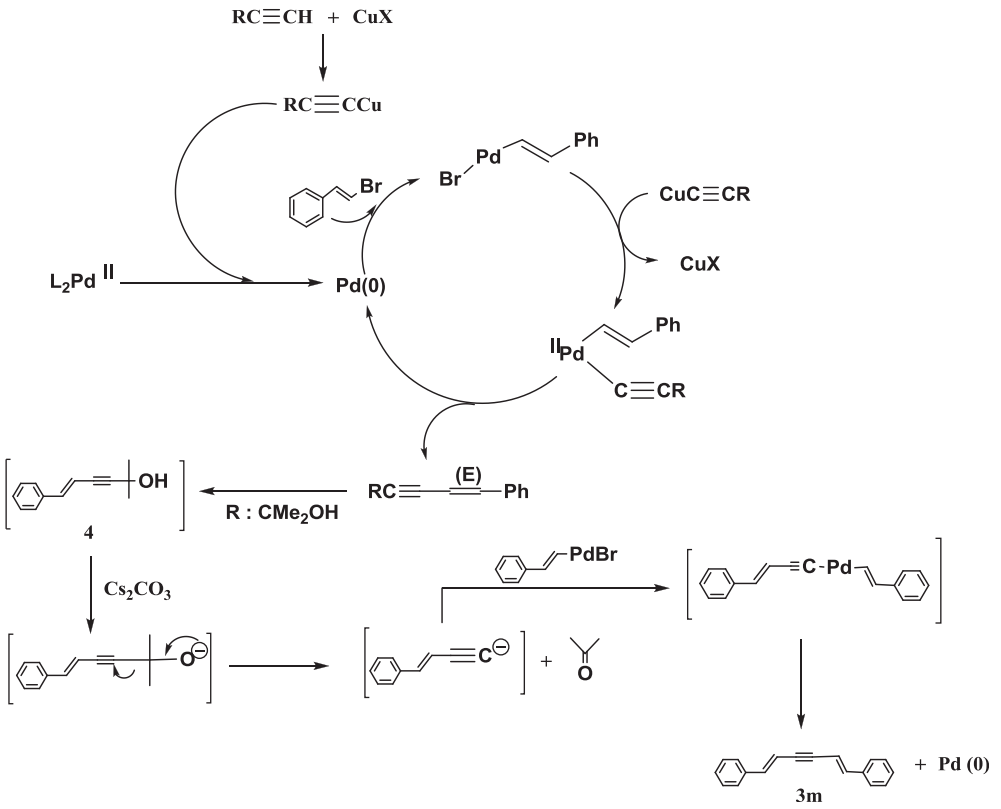


Fig. 1. The mechanism of Cat.**II** catalyzed cross-coupling reaction of (*E*)-β-bromostyrene with 2-methyl-3-butyn-2-ol.

All of the products **3a**–**m** except **3c**, **3d**, and **3i** were known and the purified products were identified by comparison of melting points, ^{1}H NMR or ^{13}C NMR spectra with the literatures.

4.2. General procedure for synthesis of enynes

To a Schlenk tube was charged with catalyst (1 mol%), CuBr (20 mol%), Cs_2CO_3 (1 mmol). The vessel was then evacuated and backfilled with nitrogen. (*E*)-β-Bromostyrene (1.0 mmol), the terminal alkyne (1.2 mmol), and DMF (3 mL) were successively added. The reaction tube was quickly sealed, and the contents were stirred at 40 °C for 24 h. Then, the reaction mixture was dissolved in H_2O and extracted with CH_2Cl_2. The combined organic layer was dried ($MgSO_4$). The product was further purified by silica gel column chromatography.

*4.2.1. (E)-1,4-Diphenylbut-1-en-3-yne (**3a**)*[30]. White solid, mp 96–97 °C (lit. 96–97 °C); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.47 (d, *J*=7.6 Hz, 2H, aromatic hydrogens), 7.41 (d, *J*=7.2 Hz, 2H, aromatic hydrogens), 7.33–7.26 (m, 6H, aromatic hydrogens), 7.03 (d, *J*=16.4 Hz, 1H, CH=), 6.37 (d, *J*=16.0 Hz, 1H, CH=); ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.2, 136.3, 131.5, 128.7, 128.6, 128.3, 128.2, 126.3, 123.4, 108.1 (CH=CH group and aromatic carbons), 91.8 (C≡), 88.9 (C≡).

*4.2.2. (E)-1-Phenyl-4-tolylbut-1-en-3-yne (**3b**)*[31]. White solid, mp 74–75 °C (lit. 75–76 °C); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.25–7.45, 7.10–7.17 (2 m, 9H, aromatic hydrogens), 7.01 (d, *J*=16.14 Hz, 1H, CH=), 6.37 (d, *J*=16.14 Hz, 1H, CH=), 2.35 (s, 3H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.84, 138.33, 136.41, 132.37, 131.40, 129.11, 128.70, 126.24, 120.31, 108.30 (CH=CH group and aromatic carbons), 91.97 (C≡), 88.24 (C≡), 21.48 (CH_3). Anal. Calcd for $C_{17}H_{14}$ (218.29): C, 93.54; H, 6.46. Found: C, 93.47; H, 6.40%.

*4.2.3. (E)-1-Phenyl-4-m-methylphenylbut-1-en-3-yne (**3c**).* Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37–7.44, 7.26–7.35, 7.15–7.23, 7.05–7.13 (m, 9H, aromatic hydrogens), 7.02 (d, *J*=16.14 Hz, 1H, CH=), 6.36 (d, *J*=16.14 Hz, 1H, CH=), 2.31 (s, 3H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.06, 137.94, 136.31, 132.04, 129.07, 128.67, 128.56, 128.52, 128.20, 126.24, 123.18, 108.19 (CH=CH group and aromatic carbons), 91.95 (C≡), 88.55 (C≡), 21.17 (CH_3). Anal. Calcd for $C_{17}H_{14}$ (218.29): C, 93.54; H, 6.46. Found: C, 93.45; H, 6.50%.

*4.2.4. (E)-1-Phenyl-4-p-tretbutylphenylbut-1-en-3-yne (**3d**).* White solid, mp 82–84 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38–7.44, 7.26–7.37 (m, 9H, aromatic hydrogens), 7.02 (d, *J*=16.45 Hz, 1H, CH=), 6.38 (d, *J*=16.45 Hz, 1H, CH=), 1.31 (s, 9H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$): δ 151.48, 140.84, 136.44, 131.24, 128.71, 128.49, 126.25, 125.34, 120.38, 108.36 (CH=CH group and aromatic carbons), 91.97 (C≡), 88.25 (C≡), 34.77(CMe_3), 31.16 ($3CH_3$). Anal. Calcd for $C_{20}H_{20}$ (260.37): C, 92.26; H, 7.74. Found: C, 92.15; H, 7.72%.

*4.2.5. (E)-1-Phenyl-4-p-methoxyphenylbut-1-en-3-yne (**3f**)*[32]. White solid, mp 49–51 °C (lit. 48–50 °C); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38–7.44, 7.25–7.36, 6.80–6.87 (3 m, 9H, aromatic hydrogens), 7.00 (d, *J*=16.14 Hz, 1H, CH=), 6.37 (d, *J*=16.14 Hz, 1H, CH=), 3.81 (s, 3H, CH_3O); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.60, 140.44, 136.49, 132.97, 128.71, 128.43, 126.21, 115.54, 114.02, 108.40 (CH=CH group and aromatic carbons), 91.84 (C≡), 87.65 (C≡), 55.28 (CH_3).

*4.2.6. (E)-1-Phenyl-4-m-aminophenylbut-1-en-3-yne (**3e**)*[33]. Brown solid, mp 88–89 °C (lit. 88–89 °C); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38–7.44, 7.26–7.36, 7.08–7.14, 6.85–6.92, 6.75–6.82, 6.59–6.68 (6 m, 9H, aromatic hydrogens), 7.02 (d, *J*=16.45 Hz, 1H, CH═), 6.89 (d, *J*=7.31 Hz, 1H, CH═), 3.99–3.05 (br s, 2H, NH_2); ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.22, 141.07, 136.34, 129.24, 128.68, 128.52, 126.24, 124.03, 121.99, 117.66, 115.28, 108.20 (CH═CH group and aromatic carbons), 91.99 (C≡), 88.26 (C≡).

*4.2.7. (E)-1-Phenyl-4-p-bromophenylbut-1-en-3-yne (**3g**)*[31]. Yellow solid, mp 113–114 °C (lit. 113–114 °C); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.49–7.40, 7.38–7.27 (2 m, 9H, aromatic hydrogens), 7.04 (d, *J*=16.45 Hz, 1H, CH═), 6.35 (d, *J*=16.45 Hz, 1H, CH═); ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.73, 136.17, 132.89, 131.61, 128.76, 126.34, 122.38, 107.78 (CH═CH group and aromatic carbons), 90.59 (C≡), 90.03 (C≡).

*4.2.8. (E)-1-Phenyl-4-p-fluorophenylbut-1-en-3-yne (**3h**)*[34]. White solid, mp 83–85 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38–7.49, 7.26–7.23, 6.98–7.08 (3 m, 10H, aromatic hydrogens and CH═ group), 6.36 (d, *J*=16.45 Hz, 1H, CH═); ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.68, 141.33, 136.26, 133.40, 133.32, 128.66, 119.51, 115.75, 115.53, 107.93 (CH═CH group and aromatic carbons), 90.61 (C≡), 88.55 (C≡); Anal. Calcd for $C_{16}H_{11}F$ (222.08): C, 86.46; H, 4.99. Found: C, 86.95; H, 5.06%.

*4.2.9. (E)-1-Phenyl-4-o-trifluoromethylphenylbut-1-en-3-yne (**3i**)*. Yellow oil, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60–7.72, 7.44–7.54, 7.29–7.44 (3 m, 9H, aromatic hydrogens), 7.10 (d, *J*=16.14 Hz, 1H, CH═), 6.42 (d,*J*=16.14 Hz, 1H, CH═), ^{13}C NMR (100 MHz, $CDCl_3$): δ 136.10, 135.07, 133.77, 132.62, 131.37, 129.50, 128.78, 128.48, 127.80, 126.50, 125.92, 125.87, 107,67 (CH═CH group and aromatic carbons), 94.46 (C≡), 87.42 (C≡); Anal. Calcd for $C_{17}H_{11}F_3$ (272.08): C, 74.99; H, 4.07. Found: C, 74.42; H, 4.14%.

*4.2.10. (E)-1-Phenyl-4-octynylbut-1-en-3-yne (**3k**)*[35]. Yellow oil, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.21–7.40 (m, 5H, aromatic hydrogens), 6.87 (d, *J*=16.63 Hz, 1H, CH═), 6.16 (d, *J*=16.63 Hz, 1H, CH═), 2.32–2.40, 1.51–1.61, 1.37–1.50, 1.23–1.36 (4 m, 10H, CH_2), 0.90 (t, *J*=6.85 Hz, 3H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$): δ 139.90, 136.53, 128.62, 128.18, 126.01, 108.84 (CH═CH group and aromatic carbons), 93.06 (C≡), 79.64 (C≡), 31.34, 28.72, 28.59, 22.53, 19.62 (CH_2), 14.04 (CH_3).

*4.2.11. (E)-1-Phenyl-4-heptynylbut-1-en-3-yne (**3l**)*[36]. Yellow oil, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.22–7.41 (m, 5H, aromatic hydrogens), 6.87 (d, *J*=16.63 Hz, 1H, CH═), 6.16 (d, *J*=16.63 Hz, 1H, CH═), 2.41–2.32, 1.66–1.51, 1.49–1.28 (3 m, 8H, CH_2), 0.92 (t, *J*=7.10 Hz, 3H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$): δ 139.91, 136.53, 128.60, 128.16, 126.00, 108.85 (CH═CH group and aromatic carbons), 93.08 (C≡), 79.67 (C≡), 31.11, 28.49, 22.24, 19.62 (CH_2), 14.00 (CH_3).

*4.2.12. 1,6-Diphenyl-1,5-hexadien-3-yne (**3m**)*[37]. Yellow solid, mp 57–59 °C (lit. 58–60 °C); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38–7.46, 7.28–7.37 (m, 10H, aromatic hydrogens), 6.98 (d, *J*=15.65 Hz, 2H, CH═), 6.35 (d, *J*=15.65 Hz, 2H, CH═); ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.12, 136.31, 128.72, 128.61, 126.28, 108.23(CH═CH group and aromatic carbons), 93.08 (C≡).

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