



Palladium-catalyzed cross-couplings of allylic carbonates with triarylbismuths as multi-coupling atom-efficient organometallic nucleophiles

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ABSTRACT

Allylic carbonates were efficiently cross-coupled with triarylbismuths under palladium catalysis. Using the optimized protocol, arylations of various allylic carbonates were carried out with triarylbismuths to afford high yields of 1,3-disubstituted propenes in regio- and chemo-selective manner. Triarylbismuths were employed as multi-coupling atom-efficient organometallic nucleophiles in sub-stoichiometric amounts in all the reactions.

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1. Introduction

The metal catalyzed reactions of allylic substrates are useful for a variety of synthetic transformations [1–10]. The reactivity profile of allylic carbonates differ from other allylic substrates in coupling reactions [11–22]. The coupling reaction of allylic carbonate in the presence of palladium catalyst usually involves π -allylpalladium(II) intermediate species [17] through the initial oxidative addition of allylic carbonate to Pd(0) with subsequent loss of carbon dioxide. The transmetalation of π -allylpalladium(II) with organometallic nucleophile followed by reductive elimination delivers the cross-coupled product. Here, the utilization of allylic carbonate as coupling partner is advantageous from the view point that the *in situ* formed alkoxide is expected to activate the organometallic reagent during transmetalation step [17]. Thus, allylic carbonates serve as facile substrates in coupling reactions with organometallic nucleophiles. The cross-coupling reactions of various allylic substrates with organo-boron, -tin, -silicon, -indium, etc. reagents were reported under palladium-catalyzed conditions [11–22]. However, recently triarylbismuths have been demonstrated to be useful as multi-coupling organometallic nucleophiles for atom-efficient couplings with organic electrophiles [23–36]. In particular, the palladium-catalyzed cross-couplings of allyl bromide [37,38] with triarylbismuths were reported earlier. Recently, we have also demonstrated the arylation of various allylic acetates

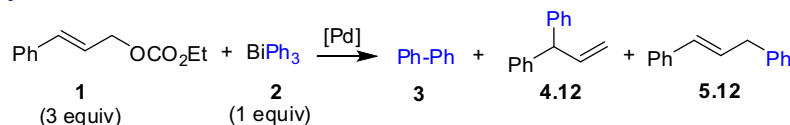
with triarylbismuths under palladium catalysis [39]. During this study, a more facile cross-coupling reactivity of allylic acetates in comparison with allylic bromides was observed. This prompted us to extend this study to allylic carbonate substrates. Herein, we report the cross-coupling study of allylic carbonates with triarylbismuth under palladium-catalyzed conditions.

2. Results and discussion

The initial studies were carried out using cinnamyl carbonate and triphenylbismuth as model substrates under different conditions and these studies are summarized in Table 1. The coupling reaction with cinnamyl carbonate was found to be facile to furnish the corresponding allylic arylation product, **5.12** as major one along with minor regio-isomeric compound, **4.12**. However, biphenyl was also formed as homo-coupling product from triphenylbismuth in minor amounts (Table 1). This was expected as triarylbismuths were known to give bi-aryls in the presence of palladium catalyst [36].

The screening carried out with different palladium precursors using K_3PO_4 base in *N,N*-dimethylformamide (DMF) provided moderate conversion to **5.12** (Table 1, entries 1–3). However, the reaction with NET_3 as base delivered somewhat better conversion in comparison with pyridine (Table 1, entries 4 and 5). Reaction at 90 °C with 1 equiv. of NET_3 furnished **5.12** in 74% isolated yield (Table 1, entry 6). This reaction was further tried in different solvents to improve the cross-coupling yield (Table 1, entries 7–11). From this, *N*-Methyl-2-pyrrolidone (NMP) solvent was found to be more

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Table 1
Screening with cinnamyl carbonate.^{a–c}

Entry	Catalytic conditions	1 (%)	2 (%)	3 (%)	4.12 (%)	5.12 (%)
1	PdCl ₂ (MeCN) ₂ , K ₃ PO ₄ (4), DMF, 60 °C, 2.5 h	67	18	9	0	0
2	Pd(PPh ₃) ₄ , K ₃ PO ₄ (4), DMF, 60 °C, 2.5 h	3	–	32	0	26
3	PdCl ₂ (PPh ₃) ₂ , K ₃ PO ₄ (4), DMF, 60 °C, 2.5 h	23	–	11	0	66
4	PdCl ₂ (PPh ₃) ₂ , Et ₃ N (1), DMF, 60 °C, 1 h	15	–	13	0	71
5	PdCl ₂ (PPh ₃) ₂ , Pyridine (1) DMF, 90 °C, 1 h	22	–	16	0	62
6	PdCl ₂ (PPh ₃) ₂ , Et ₃ N (1), DMF, 90 °C, 1 h	–	–	6	2	78 (74)
7	PdCl ₂ (PPh ₃) ₂ , Et ₃ N (1), DME, 90 °C, 1 h	68	17	12	0	0
8	PdCl ₂ (PPh ₃) ₂ , Et ₃ N (1), THF, 90 °C, 1 h	60	30	7	0	0
9	PdCl ₂ (PPh ₃) ₂ , Et ₃ N (1), MeCN, 90 °C, 1 h	19	–	12	4	64
10	PdCl ₂ (PPh ₃) ₂ , Et ₃ N (1), DMA, 90 °C, 1 h	18	–	12	4	65
11	PdCl ₂ (PPh ₃) ₂ , Et ₃ N (1), NMP, 90 °C, 1 h	10	–	5	2	81(76)
12	PdCl ₂ (PPh ₃) ₂ , Et ₃ N (2), NMP, 90 °C, 1 h	5	1	6	3	78(71)
13	PdCl ₂ (PPh ₃) ₂ , Et ₃ N (3), NMP, 90 °C, 1 h	4	1	8	2	79(71)
14	No catalyst, Et ₃ N (1), DMF, 90 °C, 1 h	96	–	4	0	0
15	PdCl ₂ (PPh ₃) ₂ , no base, DMF, 90 °C, 1 h	32	–	6	0	43

^a Conditions: BiPh₃ (1 equiv., 0.50 mmol), Cinnamyl carbonate (3.5 equiv., 1.75 mmol), base (1–4 equiv.), palladium catalyst (0.09 equiv., 0.045 mmol), solvent (6 mL).

^b Conversions are based on GC analysis of the crude reaction mixture with respect to triphenylbismuth. Isolated yields are given in parentheses and, considering 3 equiv. of cross-coupling product (1.5 mmol) as 100% yield.

effective in comparison with 1,2-dimethoxyethane (DME), tetrahydrofuran (THF), acetonitrile and *N,N*-dimethylacetamide (DMA) solvents. At this stage, increase in the amount of NEt₃ to 2 or 3 equiv. did not improve the product yield further (Table 1, entries 12 and 13). A control reaction without palladium catalyst did not furnish allylic arylation product (Table 1, entry 14). Another reaction without NEt₃ also furnished lower conversion to **5.12** (Table 1, entry 15). From the above screening it was found that the reaction is facile in polar aprotic solvents such as DMF and NMP. Formation of **4.12** as regio-isomer was also observed under certain conditions in minor amounts. Overall this investigation revealed that the protocol involving PdCl₂(PPh₃)₂ catalyst, NEt₃ base (1 equiv.) in NMP solvent at 90 °C is very effective for the facile coupling of cinnamyl carbonate with triphenylbismuth. Notably, the coupling reaction was completed in short reaction time affording high yield of arylation product. Out of DMF and NMP solvents we have chosen NMP as our choice for further study. In these reactions 3.5 equiv. of cinnamyl carbonate was employed although 3 equiv. of cinnamyl carbonate is sufficient to react with three phenyl groups of triphenylbismuth. It was also gratifying that triphenylbismuth was effectively coupled with 3 equiv. of allylic carbonates under the established protocol.

To elaborate the scope, coupling reactions of a variety of triaryl-bismuths with (*E*)-3-*p*-tolylallyl carbonate were conducted under the optimized conditions (Table 2).

The arylations using electronically divergent triaryl-bismuths as organometallic nucleophiles were found to furnish high yields of products. The regio-selectivities at the allylic position were high in all cases with the formation of corresponding 1,3-disubstituted propenes as major products. However in some reactions, the other regio-isomer was also formed in minor amounts. Noteworthy is that the novel reactivity of functionalized triaryl-bismuths and their facile reactivity under the coupling conditions (Table 2, entries 8 and 9). Importantly, trithiophen-2-ylbismuth as multi-coupling atom-efficient reagents also furnished high yield of arylation product (Table 2, entry 11).

The versatility of the coupling reaction was also assessed with a variety of allylic carbonates and the results are summarized in

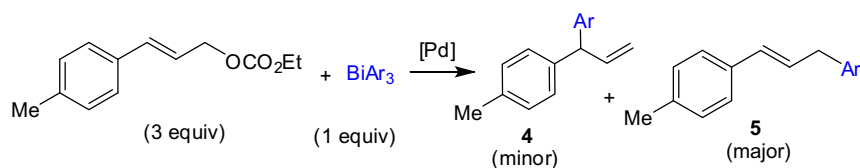
Table 3. This study revealed an efficient coupling reactivity of various functionalized allylic carbonates. Importantly, cinnamyl carbonates substituted with bromo- and chloro- groups provided chemo-selective allylic arylations in high yields. The cross-coupling of 3-cyclohexylallyl carbonate also furnished the regio-selective arylation with different triaryl-bismuth reagents.

The above results revealed us the following: (i) facile cross-couplings of allylic carbonates with triaryl-bismuths; (ii) the high reactivity of electronically divergent triaryl-bismuths with allylic carbonates; (iii) the atom-efficient reactivity of triaryl-bismuths as multi-coupling organometallic nucleophiles with allylic carbonates; (iv) fast reactivity of couplings in 1 h involving three C–C couplings using 3 equiv. of allylic carbonates; and (v) novel multi-coupling ability of triaryl-bismuths in comparison with other organometallic nucleophiles in allylic arylation. Overall, the high reactivity of triaryl-bismuths for cross-coupling with allylic carbonate was established under the established conditions. The products obtained in these reactions, i.e. 1,3-disubstituted propenes are important skeletons found in natural products [40–43]. These compounds are also useful for a variety of synthetic transformations [44–49].

A tentative catalytic cycle proposed for this reaction is given in Fig. 1. This is similar to the earlier mechanism proposed by Hiyama and co-workers with allylic carbonates [17]. As noted before in the introduction, the catalytic cycle is expected to go through π -allylpalladium alkoxide intermediate, **A**.

This intermediate upon transmetalation with triaryl-bismuth provides allylpalladium intermediates **B** and **C** which upon reductive elimination delivers arylated products **D** and **E**. The alkoxide as counter anion in **A** was proposed to activate aryl-bismuth species during transmetalation, as we have not used extra base to activate the Ar–Bi bond of triaryl-bismuths [28]. As the coupling sequence involves three aryl transfers from triaryl-bismuth, the *in situ* generated species such as Ar₂BiOEt/ArBi(OEt)₂ are proposed to involve in the transmetalation steps of subsequent catalytic cycles. This is in tune with the facile coupling reactivity of organobismuth alkoxide under palladium catalysis [50,51]. Thus, triaryl-bismuths were involved as multi-coupling

Table 2
Couplings with different triarylbismuths.^{a–c}



Entry	Triarylbismuth	GC ratio	1,3-Diarylpropene, 5	Yield (%)
1		94/4		80
2		85/15		70
3		91/9		64
4		94/6		76
5		95/5		74
6		79/21		67
7		100/0		78
8		100/0		90
9		100/0		70
10		100/0		70
11		100/0		80

^a Conditions: BiAr₃ (1 equiv., 0.50 mmol), allylic carbonate (3.5 equiv., 1.75 mmol), PdCl₂(PPh₃)₂ (0.09 equiv., 0.045 mmol), NEt₃ (1 equiv., 0.50 mmol); NMP (6 mL), 90 °C, 1 h

^b Isolated yields of **5** based on three couplings from triarylbismuth, i.e. 3 equiv. of cross-coupling product (3 × 0.5 = 1.5 mmol) as 100% yield. All products were characterized by ¹H, ¹³C NMR, IR, HRMS and in comparison with the literature data.

^c In general, homo-coupling bi-aryls from triarylbismuths are formed in all the reactions and the amount varied with respect to the degree of the cross-coupling reaction. Minor isomer **4** was not isolated as it is formed in small amounts.

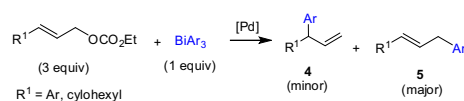
organometallic nucleophiles in these coupling reactions with allylic carbonates for three C–C couplings. However, homo-coupling reactivity of triarylbismuths under palladium-catalyzed conditions [36] is an unavoidable side pathway leading to formation of bi-aryls as minor products.

In conclusion, we have demonstrated an atom-efficient cross-coupling reactivity of triarylbismuths as multi-coupling organometallic nucleophiles with allylic carbonate under palladium-catalyzed conditions.

3. Experimental

3.1. General

All the reactions have been conducted under nitrogen atmosphere using an oven dried Schlenk tube using anhydrous solvent conditions. Allylic carbonates were prepared using standard methods [52–54]. Triarylbismuths were made following the literature procedure [55]. Anhydrous solvents such as *N,N*-dimethylformam-

Table 3
Couplings of allylic carbonates with triarylbismuths.^{a-c}

Entry	Allylic carbonate	Triarylbismuth	GC ratio	1,3-diarylpropene, 5	Yield (%)
12			90/10		76
13			85/15		74
14			83/17		72
15			93/7		75
16			90/10		72
17			88/12		73
18			92/8		73
19			87/13		71
20			86/14		70
21			90/10		70
22			86/14		64
23			87/13		70
24			98/2		75
25			88/12		71
26			89/11		70
27			94/6		85
28			90/10		80
29			90/10		77
30			88/12		70
31			91/9		68
32			91/9		71
33			90/10		77

^a Conditions: BiAr₃ (1 equiv., 0.50 mmol), allylic carbonate (3.5 equiv., 1.75 mmol), PdCl₂(PPh₃)₂ (0.09 equiv., 0.045 mmol), NEt₃ (1 equiv., 0.50 mmol); NMP (6 mL), 90 °C, 1 h.

^b Isolated yields of **5** based on three couplings from triarylbismuth, i.e. 3 equiv. of cross-coupling product (3 × 0.5 = 1.5 mmol) as 100% yield. All products were characterized by ¹H, ¹³C NMR, IR, HRMS and in comparison with literature data.

^c In general, homo-coupling bi-aryls from triarylbismuths are formed in all the reactions and the amount varied with respect to the degree of the cross-coupling reaction. Minor isomer **4** was not isolated as it is formed in small amounts.

ide (DMF) and *N*-methylpyrrolidone (NMP) were distilled using standard drying procedures. Column chromatography was per-

formed using silica gel (100–200 mesh). NMR spectra were recorded in CDCl₃ using JEOL 400 MHz spectrometer. IR spectra

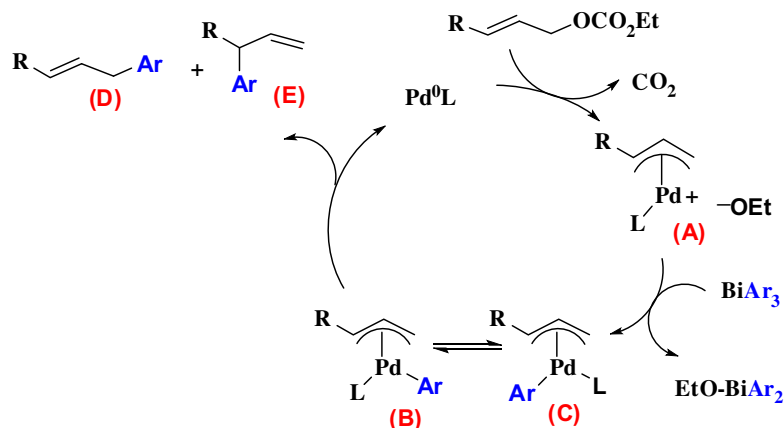


Fig. 1. Proposed mechanism.

were recorded using Bruker FT-IR spectrometer and HRMS were measured using WATERS LCMS spectrometer.

3.2. Representative procedure for cross-coupling of allylic carbonate with triaryl bismuth

To an oven dried Schlenk tube, cinnamyl carbonate (1.75 mmol, 361 mg) was charged followed by Et₃N (0.5 mmol, 51 mg), triphenylbismuth (0.5 mmol, 220 mg), PdCl₂(PPh₃)₂ (0.045 mmol, 31.5 mg) and NMP (6 mL) under nitrogen atmosphere. The reaction mixture was stirred in an oil bath at 90 °C for 1 h. After the reaction time, the contents were cooled to room temperature, quenched with 10 mL water and extracted with ethyl acetate (3 × 20 mL). The combined organic extract was washed with water (2 × 10 mL), brine (20 mL) and dried over anhydrous MgSO₄. The organic extract was concentrated under reduced pressure. The crude product was purified by column chromatography using petroleum ether as eluent to afford 1,3-diphenylpropene, **5.12** (221 mg, 76%).

5.1. (*E*)-1-(4-Methylphenyl)-3-phenylpropene [56]: Colourless oil. Yield, 80%. ¹H NMR (400 MHz, CDCl₃) δ 7.18–7.36 (m, 7H), 7.14 (d, 2H, *J* = 8.04 Hz), 6.47 (d, 1H, *J* = 15.84 Hz), 6.30–6.38 (m, 1H), 3.57 (d, 2H, *J* = 6.60 Hz), 2.36 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 140.32, 136.80, 134.67, 130.90, 129.17, 128.64, 128.43, 128.14, 126.10, 125.99, 39.32, 21.13. IR (KBr) 3026, 2920, 1723, 1452, 1270, 1177, 816, 750, 699 cm⁻¹.

5.2. (*E*)-1,3-bis(4-Methylphenyl)propene [57]: White solid. M.P 42–43 °C. Yield, 70%. ¹H NMR (400 MHz, CDCl₃) δ 7.09–7.29 (m, 8H), 6.42 (d, 1H, *J* = 15.88 Hz), 6.26–6.33 (m, 1H), 3.50 (d, 2H, *J* = 6.60 Hz), 2.34 (s, 3H), 2.33 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 137.22, 136.72, 135.57, 134.75, 130.67, 129.12, 128.51, 128.45, 125.96, 38.90, 21.11, 20.99. IR (KBr) 3022, 2920, 1723, 1514, 1271, 1177, 1109, 1039, 810, 698 cm⁻¹. HRMS (ESI) calcd. for C₁₇H₁₈ [M–H]⁺, 221.1336; found, 221.1330.

5.3. (*E*)-1-(4-Methylphenyl)-3-(4-methoxyphenyl)propene [56]: Colourless oil. Yield, 64%. ¹H NMR (400 MHz, CDCl₃) δ 7.27 (d, 2H, *J* = 8.04 Hz), 7.11–7.19 (m, 4H), 6.88 (d, 2H, *J* = 8.52 Hz), 6.43 (d, 1H, *J* = 15.60 Hz), 6.27–6.34 (m, 1H), 3.82 (s, 3H), 3.50 (d, 2H, *J* = 6.60 Hz), 2.35 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 158.00, 136.74, 134.71, 132.33, 130.58, 129.59, 129.19, 128.62, 125.99, 113.88, 55.26, 38.41, 21.12. IR (KBr) 3063, 2939, 2853, 1598, 1511, 1267, 1149, 1045, 967, 775 cm⁻¹. HRMS (ESI) calcd. for C₁₇H₁₈O [M]⁺, 238.1358; found, 238.1355.

5.4. (*E*)-1-(4-Methylphenyl)-3-(4-fluorophenyl)propene [56]: Light yellow oil. Yield, 76%. ¹H NMR (400 MHz, CDCl₃) δ 7.28 (d, 2H, *J* = 8.00 Hz), 7.20–7.23 (m, 2H), 7.13 (d, 2H, *J* = 7.84 Hz), 6.99–7.04 (m, 2H), 6.43 (d, 1H, *J* = 15.88 Hz), 6.26–6.33 (m, 1H), 3.53

(d, 2H, *J* = 6.84 Hz), 2.35 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 161.47 (d, *J* = 243.00 Hz), 136.94, 135.91, 134.56, 131.09, 130.01, 129.19, 127.91, 126.01, 115.15 (d, *J* = 22.00 Hz), 38.45, 21.10. IR (KBr) 3024, 2920, 1602, 1508, 1221, 1156, 967, 798, 704 cm⁻¹. HRMS (ESI) calcd. for C₁₆H₁₅F [M+H]⁺, 227.1236; found, 227.1236.

5.5. (*E*)-1-(4-Methylphenyl)-3-(4-chlorophenyl)propene [56]: Colourless oil. Yield, 74%. ¹H NMR (400 MHz, CDCl₃) δ 7.24–7.28 (m, 4H), 7.16 (d, 2H, *J* = 8.32 Hz), 7.10 (d, 2H, *J* = 8.40 Hz), 6.41 (d, 1H, *J* = 15.84 Hz), 6.21–6.29 (m, 1H), 3.50 (d, 2H, *J* = 6.60 Hz), 2.32 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 138.77, 137.02, 134.50, 131.91, 131.37, 129.98, 129.22, 128.54, 127.48, 126.04, 38.59, 21.12. IR (KBr) 3023, 2921, 1726, 1489, 1089, 1014, 966, 803 cm⁻¹. HRMS (ESI) calcd. for C₁₆H₁₅Cl [M+H]⁺, 243.0941; found, 243.0940.

5.6. (*E*)-1-(4-Methylphenyl)-3-(3-methoxyphenyl)propene: Colourless oil. Yield, 67%. ¹H NMR (400 MHz, CDCl₃) δ 7.24–7.30 (m, 3H), 7.13 (d, 1H, *J* = 8.08 Hz), 6.88 (d, 2H, *J* = 7.56 Hz), 6.79–6.83 (m, 2H), 6.47 (d, 1H, *J* = 15.80 Hz), 6.29–6.39 (m, 1H), 3.83 (s, 3H), 3.55 (d, 2H, *J* = 6.60 Hz), 2.36 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 159.78, 141.97, 136.80, 134.70, 131.01, 129.16, 127.94, 126.02, 121.03, 114.35, 111.51, 55.13, 39.34, 21.10. IR (KBr) 3023, 2919, 2833, 1598, 1511, 1261, 1148, 1045, 967, 775 cm⁻¹. HRMS (ESI) calcd. for C₁₇H₁₈O [M+H]⁺, 239.1436; found, 239.1436.

5.7. (*E*)-1-(3-Methylphenyl)-3-(4-methylphenyl)propene: Colourless oil. Yield, 78%. ¹H NMR (400 MHz, CDCl₃) δ 7.29 (d, 2H, *J* = 8.28 Hz), 7.08–7.22 (m, 6H), 6.30–6.47 (m, 2H), 3.53 (d, 2H, *J* = 6.84 Hz), 2.36 (s, 3H), 2.35 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 140.23, 138.02, 136.76, 134.71, 130.77, 129.15, 128.32, 126.83, 125.99, 125.63, 39.27, 21.36, 21.11. IR (KBr) 3023, 2922, 2855, 1702, 1607, 1488, 1452, 1094, 813 cm⁻¹. HRMS (ESI) calcd. for C₁₇H₁₈ [M–H]⁺, 221.1336; found, 221.1336.

5.8. (*E*)-2-Methyl-2-[4-(3-*p*-tolyl-allyl)-phenyl]-[1,3]dioxolane: Yellow oil. Yield, 90%. ¹H NMR (400 MHz, CDCl₃) δ 7.44 (d, 2H, *J* = 8.08 Hz), 7.28 (d, 2H, *J* = 7.56 Hz), 7.25 (d, 2H, *J* = 7.80 Hz), 7.13 (d, 2H, *J* = 7.80 Hz), 6.46 (d, 1H, *J* = 15.88 Hz), 6.28–6.33 (m, 1H), 4.06 (t, 2H, *J* = 6.60 Hz), 3.81 (t, 2H, *J* = 6.60 Hz), 3.56 (d, 2H, *J* = 6.84 Hz), 2.35 (s, 3H), 1.68 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 141.23, 139.97, 136.83, 134.70, 131.02, 129.17, 128.40, 128.01, 126.01, 125.40, 108.86, 64.42, 39.00, 27.60, 21.09. IR (KBr) 3024, 2987, 2888, 1704, 1609, 1410, 1255, 1198, 1039, 1212, 871, 757 cm⁻¹. HRMS (ESI) calcd. for C₂₀H₂₂O₂ [M–H]⁺, 293.1547; found, 293.1547.

5.9. (*E*)-2-[4-(3-*p*-Tolyl-allyl)-phenyl]-[1,3]dioxolane: Yellow oil. Yield, 70%. ¹H NMR (400 MHz, CDCl₃) δ 7.43 (d, 2H, *J* = 7.80 Hz), 7.24–7.27 (m, 4H), 7.11 (d, 2H, *J* = 7.80 Hz), 6.43 (d, 1H, *J* = 16.36 Hz), 6.24–6.32 (m, 1H), 5.81 (s, 1H), 4.14 (t, 2H, *J* = 6.84 Hz), 4.02–4.05 (m, 2H), 3.54 (d, 2H, *J* = 6.60 Hz), 2.32 (s,

3H). ^{13}C NMR (100 MHz, CDCl_3) δ 141.42, 136.84, 134.69, 131.07, 130.01, 129.18, 128.67, 127.92, 126.57, 126.01, 103.75, 65.26, 39.06, 21.09. IR (KBr) 3024, 2921, 2853, 1697, 1605, 1261, 1272, 1045, 1212, 967, 816 cm^{-1} . HRMS (ESI) calcd. for $\text{C}_{19}\text{H}_{20}\text{O}_2$ $[\text{M}+\text{H}]^+$, 281.1547; found, 281.1542.

5.10. (*E*)-2-Methoxy-6-(3-*p*-tolyl-allyl)-naphthalene: Light yellow oil. Yield, 70%. ^1H NMR (400 MHz, CDCl_3) δ 7.68 (d, 2H, $J = 8.28$ Hz), 7.60 (s, 1H), 7.33 (dd, 2H, $J = 1.68, 8.28$ Hz), 7.27 (d, 2H, $J = 8.04$ Hz), 7.09–7.14 (m, 3H), 6.45 (d, 1H, $J = 15.88$ Hz), 6.33–6.40 (m, 1H), 3.90 (s, 3H), 3.66 (d, 2H, $J = 6.60$ Hz), 2.32 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 157.33, 136.82, 135.49, 134.79, 133.22, 131.02, 129.19, 128.97, 128.27, 127.95, 126.87, 126.57, 126.05, 118.70, 105.78, 55.26, 39.26, 21.09. IR (KBr) 3054, 3020, 2961, 1631, 1508, 1421, 1228, 1195, 1033, 855, 816 cm^{-1} . HRMS (ESI) calcd. for $\text{C}_{21}\text{H}_{20}\text{O}$ $[\text{M} + \text{H}]^+$, 289.1598; found, 289.1594.

5.11. (*E*)-2-(3-*p*-Tolyl-allyl)-thiophene: Light brown oil, Yield, 80%. ^1H NMR (400 MHz, CDCl_3) δ 7.29 (d, 2H, $J = 8.08$ Hz), 7.18 (dd, 1H, $J = 1.20, 5.12$ Hz), 7.14 (d, 2H, $J = 7.80$ Hz), 6.98 (dd, 1H, $J = 3.40, 5.12$ Hz), 6.88 (dd, 1H, $J = 1.00, 3.40$ Hz), 6.32–6.52 (m, 2H), 3.75 (d, 2H, $J = 6.60$ Hz), 2.36 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 143.33, 137.03, 134.39, 131.21, 129.19, 127.10, 126.87, 126.11, 124.57, 123.65, 33.31, 21.13. IR (KBr) 3023, 2919, 1512, 1438, 1180, 966, 847, 817 cm^{-1} . HRMS (ESI) calcd. for $\text{C}_{14}\text{H}_{14}\text{S}$ $[\text{M}-\text{H}]^+$, 213.0743; found, 213.0727.

5.12. (*E*)-1,3-Diphenyl propene [12]: Colourless oil, Yield, 76%. ^1H NMR (400 MHz, CDCl_3) δ 7.25–7.42 (m, 10H), 6.51 (d, 1H, $J = 15.88$ Hz), 6.37–6.45 (m, 1H), 3.60 (d, 2H, $J = 6.56$ Hz). ^{13}C NMR (100 MHz, CDCl_3) δ 140.14, 137.45, 131.05, 129.19, 128.64, 128.47, 127.07, 126.15, 126.10, 39.32. IR (KBr) 3060, 3026, 1600, 1494, 1452, 1428, 965, 741, 697 cm^{-1} .

5.13. (*E*)-3-(4-methylphenyl)-1-phenyl propene [58]: Colourless oil, Yield, 74%. ^1H NMR (400 MHz, CDCl_3) δ 7.15–7.39 (m, 9H), 6.47 (d, 1H, $J = 15.64$ Hz), 6.33–6.40 (m, 1H), 3.53 (d, 2H, $J = 6.36$ Hz), 2.35 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 137.46, 135.64, 130.77, 129.47, 129.14, 128.51, 128.45, 127.01, 126.06, 125.53, 38.89, 21.00. IR (KBr) 3024, 2921, 1600, 1513, 1495, 1449, 1022 cm^{-1} . HRMS (ESI) calcd. for $\text{C}_{16}\text{H}_{16}$ $[\text{M}]^+$, 208.1252; found, 208.1258.

5.14. (*E*)-1-phenyl-3-(4-methoxyphenyl)propene [12]: Light yellow oil, Yield, 72%. ^1H NMR (400 MHz, CDCl_3) δ 7.21–7.40 (m, 5H), 7.20 (d, 2H, $J = 8.52$ Hz), 6.89 (d, 2H, $J = 8.56$ Hz), 6.47 (d, 1H, $J = 15.88$ Hz), 6.34–6.41 (m, 1H), 3.83 (s, 3H), 3.53 (d, 2H, $J = 6.36$ Hz). ^{13}C NMR (100 MHz, CDCl_3) δ 158.03, 137.50, 132.14, 130.69, 129.64, 129.56, 128.45, 127.01, 126.07, 113.87, 55.25, 38.42. IR (KBr) 3001, 2955, 2835, 1606, 1512, 1248, 1176, 1033, 829, 698 cm^{-1} .

5.15. (*E*)-1-(3-Bromophenyl)-3-phenylpropene: Light yellow oil. Yield, 75%. ^1H NMR (400 MHz, CDCl_3) δ 7.14–7.52 (m, 9H), 6.38–6.39 (m, 2H), 3.56 (d, 2H, $J = 4.16$ Hz). ^{13}C NMR (100 MHz, CDCl_3) δ 139.67, 130.96, 129.97, 129.61, 128.97, 128.64, 128.54, 126.30, 124.76, 122.70, 39.25. IR (KBr) 3062, 3028, 1722, 1570, 1451, 1252, 1070, 783, 746, 699 cm^{-1} . HRMS (ESI) calcd. for $\text{C}_{15}\text{H}_{13}\text{Br}$ $[\text{M}-\text{H}]^+$, 271.0128; found, 271.0125.

5.16. (*E*)-1-(3-Bromophenyl)-3-(4-methylphenyl)propene: Light yellow oil. Yield, 72%. ^1H NMR (400 MHz, CDCl_3) δ 7.54–7.55 (m, 2H), 7.36 (dd, 2H, $J = 0.96, 7.80$ Hz), 7.17–7.30 (m, 4H), 6.43 (d, 1H, $J = 15.88$ Hz), 6.36–6.40 (m, 1H), 3.56 (d, 2H, $J = 3.44$ Hz), 2.38 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 139.71, 136.57, 135.82, 131.28, 129.94, 129.38, 128.98, 128.53, 124.73, 122.69, 38.83, 21.02. IR (KBr) 3054, 2922, 2856, 1687, 1344, 1265, 1020, 741 cm^{-1} . HRMS (ESI) calcd. for $\text{C}_{16}\text{H}_{15}\text{Br}$ $[\text{M}]^+$, 286.0357; found, 286.0358.

5.17. (*E*)-1-(3-Bromophenyl)-3-(4-methoxyphenyl)propene: Light yellow oil, Yield, 73%. ^1H NMR (400 MHz, CDCl_3) δ 7.11–7.32 (m, 6H), 6.84–6.88 (m, 2H), 6.33–6.41 (m, 2H), 3.80 (s, 3H), 3.48 (d, 2H, $J = 4.88$ Hz). ^{13}C NMR (100 MHz, CDCl_3) δ 158.16,

135.64, 132.72, 132.55, 131.85, 129.57, 128.05, 127.01, 126.73, 113.98, 55.28, 38.65. IR (KBr) 2999, 2954, 2930, 1606, 1511, 1176, 995 cm^{-1} . HRMS (ESI) calcd. for $\text{C}_{16}\text{H}_{15}\text{BrO}$ $[\text{M}]^+$, 302.0306; found, 302.0306.

5.18. (*E*)-1-(4-Bromophenyl)-3-phenylpropene [12]: Light yellow oil, Yield, 73%. ^1H NMR (400 MHz, CDCl_3) δ 7.42 (d, 2H, $J = 6.84$ Hz), 7.32 (d, 2H, $J = 7.32$ Hz), 7.21–7.27 (m, 5H), 6.32–6.42 (m, 2H), 3.54 (d, 2H, $J = 4.64$ Hz). ^{13}C NMR (100 MHz, CDCl_3) δ 139.77, 136.38, 131.53, 130.13, 129.84, 128.64, 128.51, 127.63, 126.26, 120.73, 39.29. IR (KBr) 3061, 3028, 2923, 1722, 1591, 1489, 1266, 1070, 1029, 796, 700 cm^{-1} .

5.19. (*E*)-1-(4-Bromophenyl)-3-(4-methylphenyl)propene: Light yellow solid, Yield, 71%. M.P. 47–49 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.50 (d, 2H, $J = 8.08$ Hz), 7.42 (d, 2H, $J = 8.52$ Hz), 7.13–7.27 (m, 4H), 6.31–6.41 (m, 2H), 3.52 (d, 2H, $J = 5.12$ Hz), 2.35 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 136.66, 136.45, 135.78, 131.51, 130.45, 129.61, 129.19, 128.51, 127.61, 120.66, 38.87, 21.00. IR (KBr) 3020, 2888, 1901, 1510, 1482, 1397, 1069 cm^{-1} . HRMS (ESI) calcd. for $\text{C}_{16}\text{H}_{15}\text{Br}$ $[\text{M}-\text{H}]^+$, 285.0284; found, 285.0267.

5.20. (*E*)-1-(4-Bromophenyl)-3-(4-methoxyphenyl)propene: Light yellow oil, Yield, 70%. ^1H NMR (400 MHz, CDCl_3) δ 7.42 (d, 2H, $J = 8.52$ Hz), 7.23 (d, 2H, $J = 8.56$ Hz), 7.16 (d, 2H, $J = 8.80$ Hz), 6.88 (d, 2H, $J = 8.76$ Hz), 6.31–6.40 (m, 2H), 3.82 (s, 3H), 3.49 (d, 2H, $J = 4.88$ Hz). ^{13}C NMR (100 MHz, CDCl_3) δ 158.16, 136.50, 131.81, 131.53, 130.63, 129.56, 127.63, 120.68, 113.99, 55.33, 38.41. IR (KBr) 3026, 2953, 2833, 1701, 1607, 1510, 1486, 1245, 1176, 1071, 1035, 967, 827 cm^{-1} . HRMS (ESI) calcd. for $\text{C}_{16}\text{H}_{15}\text{BrO}$ $[\text{M}]^+$, 302.0306; found, 302.0304.

5.21. (*E*)-1-(4-Chlorophenyl)-3-phenylpropene [12]: Pale yellow oil, Yield, 70%. ^1H NMR (400 MHz, CDCl_3) δ 7.24–7.35 (m, 9H), 6.41 (d, 1H, $J = 15.84$ Hz), 6.31–6.38 (m, 1H), 3.56 (d, 2H, $J = 6.08$ Hz). ^{13}C NMR (100 MHz, CDCl_3) δ 139.86, 136.00, 132.67, 130.03, 129.85, 128.62, 128.53, 127.31, 126.26, 39.29. IR (KBr) 3026, 2926, 1600, 1452, 1429, 1093 cm^{-1} .

5.22. (*E*)-1-(4-Chlorophenyl)-3-(4-methylphenyl)propene: White solid. M.P. 43–45 °C. Yield, 64%. ^1H NMR (400 MHz, CDCl_3) δ 7.34–7.50 (m, 4H), 7.13–7.26 (m, 4H), 6.39 (d, 1H, $J = 15.88$ Hz), 6.29–6.36 (m, 1H), 3.51 (d, 2H, $J = 6.08$ Hz), 2.34 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 136.72, 136.00, 135.77, 132.54, 130.29, 129.56, 129.20, 128.57, 128.52, 127.27, 38.86, 21.00. IR (KBr) 3020, 2920, 2888, 1487, 1450, 1332, 1091 cm^{-1} . HRMS (ESI) calcd. for $\text{C}_{16}\text{H}_{15}\text{Cl}$ $[\text{M}-\text{H}]^+$, 241.0790; found, 241.0794.

5.23. (*E*)-1-(4-Chlorophenyl)-3-(4-methoxyphenyl)propene [12]: Colourless oil. Yield, 70%. ^1H NMR (400 MHz, CDCl_3) δ 7.22–7.35 (m, 4H), 7.18 (d, 2H, $J = 8.56$ Hz), 6.89 (d, 2H, $J = 8.56$ Hz), 6.41 (d, 1H, $J = 15.88$ Hz), 6.31–6.38 (m, 1H), 3.83 (s, 3H), 3.50 (d, 2H, $J = 5.84$ Hz). ^{13}C NMR (100 MHz, CDCl_3) δ 158.10, 136.01, 132.55, 131.83, 130.45, 129.57, 129.47, 128.58, 127.27, 113.93, 55.26, 38.39. IR (KBr) 2999, 2995, 2835, 1606, 1511, 1301, 1248, 1176, 1090, 1034, 827 cm^{-1} .

5.24. (*E*)-1-(3-Chlorophenyl)-3-phenylpropene: Light yellow oil. Yield, 75%. ^1H NMR (400 MHz, CDCl_3) δ 7.18–7.36 (m, 9H), 6.38–6.45 (m, 2H), 3.56 (d, 2H, $J = 4.40$ Hz). ^{13}C NMR (100 MHz, CDCl_3) δ 139.70, 139.38, 134.36, 130.91, 129.70, 128.64, 127.01, 126.29, 124.31, 123.76, 39.25. IR (KBr) 3062, 3027, 2925, 1595, 1572, 1196, 1077, 785, 747, 699 cm^{-1} . HRMS (ESI) calcd. for $\text{C}_{15}\text{H}_{13}\text{Cl}$ $[\text{M}-\text{H}]^+$, 227.0633; found, 227.0637.

5.25. (*E*)-1-(3-Chlorophenyl)-3-(4-methylphenyl)propene: Light yellow oil. Yield, 71%. ^1H NMR (400 MHz, CDCl_3) δ 7.15–7.35 (m, 8H), 6.36–6.39 (m, 2H), 3.52 (d, 2H, $J = 4.88$ Hz), 2.36 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 139.39, 136.57, 135.80, 134.39, 131.19, 129.47, 128.52, 126.93, 126.03, 124.27, 38.82, 21.00. IR (KBr) 3023, 2981, 2921, 1698, 1428, 1264, 1077, 785, 749, 691 cm^{-1} . HRMS (ESI) calcd. for $\text{C}_{16}\text{H}_{15}\text{Cl}$ $[\text{M}-\text{H}]^+$, 241.0790; found, 241.0782.

5.26, (*E*)-1-(3-Chlorophenyl)-3-(4-methoxyphenyl)propene: Light yellow oil. Yield, 70%. ^1H NMR (400 MHz, CDCl_3) δ 7.18–7.36 (m, 4H), 7.17 (d, 2H, $J = 8.28$ Hz), 6.88 (d, 2H, $J = 8.76$ Hz), 6.34–6.43 (m, 2H), 3.83 (s, 3H), 3.50 (d, 2H, $J = 4.64$ Hz); ^{13}C NMR (100 MHz, CDCl_3) δ 158.12, 139.40, 134.40, 131.68, 131.34, 129.58, 126.94, 126.02, 124.28, 113.95, 55.27, 38.35. IR (KBr) 2997, 2990, 2839, 1600, 1510, 1312, 1248, 1174, 1095, 1036, 820 cm^{-1} . HRMS (ESI) calcd. for $\text{C}_{15}\text{H}_{13}\text{ClO}$ $[\text{M}]^+$, 258.0811; found, 258.0814.

5.27, (*E*)-1-(2-Chlorophenyl)-3-phenylpropene: Pale yellow oil. Yield, 85%. ^1H NMR (400 MHz, CDCl_3) δ 7.53 (dd, 1H, $J = 1.68$, 7.56 Hz), 7.17–7.37 (m, 8H), 6.90 (d, 1H, $J = 15.64$ Hz), 6.32–6.40 (m, 1H), 3.63 (d, 2H, $J = 7.08$ Hz). ^{13}C NMR (100 MHz, CDCl_3) δ 139.83, 135.57, 132.72, 132.10, 129.60, 128.53, 128.12, 127.34, 126.73, 126.26, 39.55. IR (KBr) 3061, 3027, 1469, 1452, 1051, 965, 748, 698 cm^{-1} . HRMS (ESI) calcd. for $\text{C}_{15}\text{H}_{13}\text{Cl}$ $[\text{M}-\text{H}]^+$, 227.0633; found, 227.0627.

5.28, (*E*)-1-(2-Chlorophenyl)-3-(4-methylphenyl)propene: Light yellow oil. Yield, 80%. ^1H NMR (400 MHz, CDCl_3) δ 7.52 (d, 2H, $J = 7.56$ Hz), 7.36 (d, 2H, $J = 7.56$ Hz), 7.14–7.27 (m, 4H), 6.88 (d, 1H, $J = 15.60$ Hz), 6.30–6.38 (m, 1H), 3.58 (d, 2H, $J = 7.08$ Hz), 2.36 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 136.74, 135.75, 132.71, 132.39, 129.57, 129.21, 128.51, 128.05, 127.09, 126.73, 39.13, 21.00. IR (KBr) 3020, 2925, 2856, 1467, 1436, 1264, 1033, 966, 749 cm^{-1} . HRMS (ESI) calcd. for $\text{C}_{16}\text{H}_{15}\text{Cl}$ $[\text{M}]^+$, 242.0862; found, 242.0860.

5.29, (*E*)-1-(2-Chlorophenyl)-3-(4-methoxyphenyl)propene: Light yellow oil. Yield, 77%. ^1H NMR (400 MHz, CDCl_3) δ 7.52 (d, 2H, $J = 7.56$ Hz), 7.35 (d, 2H, $J = 7.56$ Hz), 7.19 (d, 2H, $J = 8.32$ Hz), 6.84–6.89 (m, 3H), 6.29–6.36 (m, 1H), 3.79 (s, 3H), 3.56 (d, 2H, $J = 7.08$ Hz). ^{13}C NMR (100 MHz, CDCl_3) δ 158.16, 135.64, 132.71, 132.55, 131.85, 129.57, 128.05, 127.01, 126.73, 113.98, 55.28, 38.65. IR (KBr) 2931, 2905, 2833, 1609, 1511, 1300, 1245, 1176, 1034, 966, 752 cm^{-1} . HRMS (ESI) calcd. for $\text{C}_{16}\text{H}_{15}\text{ClO}$ $[\text{M}]^+$, 258.0811; found, 258.0813.

5.30, (*E*)-1-(3-methoxyphenyl)-3-phenylpropene [59]: Colourless oil, Yield, 70%. ^1H NMR (400 MHz, CDCl_3) δ 7.20–7.35 (m, 4H), 6.91–6.98 (m, 4H), 6.78 (dd, 1H, $J = 2.44$, 8.26 Hz), 6.33–6.47 (m, 2H), 3.81 (s, 3H), 3.57 (d, 2H, $J = 6.36$ Hz). ^{13}C NMR (100 MHz, CDCl_3) δ 159.77, 140.07, 138.92, 130.95, 129.55, 128.66, 126.18, 118.80, 112.83, 111.33, 55.16, 39.28. IR (KBr) 3028, 2937, 2835, 1721, 1598, 1490, 1455, 1263, 1043, 752, 699 cm^{-1} . HRMS (ESI) calcd. for $\text{C}_{16}\text{H}_{16}\text{O}$ $[\text{M}+\text{H}]^+$, 225.1279; found, 225.1275.

5.31, (*E*)-1-Cyclohexyl-3-phenylpropene [60]: Colourless oil. Yield, 68%. ^1H NMR (400 MHz, CDCl_3) δ 7.21–7.34 (m, 5H), 5.46–5.60 (m, 2H), 3.35 (d, 2H, $J = 5.36$ Hz), 1.96–2.01 (m, 1H), 1.57–1.77 (m, 6H), 1.07–1.34 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 141.21, 138.10, 128.43, 128.27, 126.10, 125.80, 40.62, 39.08, 33.10, 26.20, 26.08. IR (KBr) 2922, 2850, 1725, 1510, 1440, 1175, 960, 846 cm^{-1} . HRMS (ESI) calcd. for $\text{C}_{15}\text{H}_{20}$ $[\text{M}]^+$, 200.1565; found, 200.1560.

5.32, (*E*)-1-Cyclohexyl-3-(4-methylphenyl)propene [61]: Colourless oil. Yield, 71%. ^1H NMR (400 MHz, CDCl_3) δ 7.12–7.29 (m, 4H), 5.47–5.58 (m, 2H), 3.33 (d, 2H, $J = 5.60$ Hz), 2.36 (s, 3H), 1.94–2.01 (m, 1H), 1.66–1.77 (m, 6H), 1.13–1.30 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 138.13, 137.82, 135.22, 128.96, 128.31, 126.38, 40.60, 38.65, 33.10, 26.20, 26.08, 20.97. IR (KBr) 2925, 2852, 1720, 1513, 1449, 1178, 968, 843 cm^{-1} . HRMS (ESI) calcd. for $\text{C}_{16}\text{H}_{22}$ $[\text{M}]^+$, 214.1721; found, 214.1721.

5.33, (*E*)-1-Cyclohexyl-3-(4-methoxyphenyl)propene: Colourless oil. Yield, 77%. ^1H NMR (400 MHz, CDCl_3) δ 7.11 (d, 2H, $J = 8.28$ Hz), 6.85 (d, 2H, $J = 8.52$ Hz), 5.43–5.55 (m, 2H), 3.82 (s, 3H), 3.28 (d, 2H, $J = 5.64$ Hz), 1.92–1.99 (m, 1H), 1.57–1.74 (m, 6H), 1.08–1.29 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 157.79, 137.78, 133.28, 129.34, 126.55, 113.75, 55.24, 40.57, 38.16, 33.13, 26.21, 26.08. IR (KBr) 2925, 2851, 1607, 1511, 1448, 1246, 1175,

1035, 968, 848 cm^{-1} . HRMS (ESI) calcd. for $\text{C}_{16}\text{H}_{22}\text{O}$ $[\text{M}-\text{H}]^+$, 229.1598; found, 229.1554.

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Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2010.03.010.

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