## Palladium-Catalyzed Novel Arylations of Cyclic β-Bromo α,β-Unsaturated Aldehydes with Triarylbismuths as Multicoupling Organometallic Nucleophiles

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**Abstract:** Cross-coupling arylations of cyclic  $\beta$ -bromo  $\alpha$ , $\beta$ -unsaturated aldehydes were carried out with triarylbismuths as atomefficient multicoupling organometallic reagents under palladiumcatalyzed conditions. These reactions afforded the corresponding arylated alkenes in an efficient manner with good to high yields.

Key words: palladium catalysis, cyclic  $\beta$ -bromo  $\alpha$ , $\beta$ -unsaturated aldehyde, cross-coupling, triarylbismuths, tetrasubstituted alkenes

Palladium-catalyzed coupling reactions are often employed in the synthesis of complex molecular systems.<sup>1</sup> In particular, regio- and stereoselective synthesis of tetrasubstituted alkenes is an important aspect in organic synthesis. There is a constant demand for new strategies for the synthesis of these systems. Tetrasubstituted olefins are useful precursors in the synthesis of a variety of natural products.<sup>2</sup> These structures are also used for the synthesis of molecular structures such as phenanthrene<sup>3</sup> and quinoline derivatives<sup>4</sup> involving palladium-mediated Ullmann cross-couplings.

Similarly,  $\beta$ -halo  $\alpha$ ,  $\beta$ -unsaturated aldehydes represent an important class of compounds in organic synthesis. These compounds are useful in cycloaddition reactions of enedivne and ene-allene functionalities.<sup>5,6</sup> However, these substrates have not been much investigated in coupling reactions with organometallic reagents under metal catalysis.<sup>3</sup> We envisaged that such coupling with triarylbismuths under palladium-catalyzed conditions would provide a unique opportunity for chemoselective vinylic arylations leading to synthetically useful molecular scaffolds under facile reaction conditions. The advantage associated with triarylbismuth compounds is that they serve as atom-efficient multicoupling organometallic nucleophiles in reactions with three equivalents of an organic electrophile.<sup>7</sup> We have therefore studied the coupling reactions of cyclic  $\beta$ -bromo  $\alpha,\beta$ -unsaturated cyclic aldehydes with triarylbismuths under palladium-catalyzed conditions and the results are described herein.

The coupling reactivity of  $\beta$ -bromo  $\alpha$ , $\beta$ -unsaturated cyclic aldehydes with triarylbismuths was studied through systematic screening under palladium-catalyzed conditions. The 1-bromo-3,4-dihydronaphthalene-2-carbaldehyde (**1a**)

required for the coupling studies was prepared from  $\alpha$ -tetralone following the literature procedure.<sup>5h</sup> The investigation was carried out using 1-bromo-3,4dihydronaphthalene-2-carbaldehyde (**1a**) with triphenylbismuth (**1b**) under different conditions to obtain 1-phenyl-3,4-dihydronaphthalene-2-carbaldehyde (**2a**) and these results are summarized in Table 1.

 Table 1
 Screening Conditions<sup>a,b</sup>

	$ \begin{array}{c}  & \text{Br} \\  & \text{CHO} \\  & + \text{BiPh}_3 \xrightarrow{[Pd]} \text{Ph-Ph} + (1) \\  & 1b & 1c \\ 1a \\ \end{array} $		Ph	СНО
Entry	Catalyst, base, solvent, temperature	1a	1c	2a <sup>c,d</sup>
1	PdCl <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> , K <sub>2</sub> CO <sub>3</sub> , DMF, 90 °C	5	8	83 (75)
2	PdCl <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> , Na <sub>2</sub> CO <sub>3</sub> , DMF, 90 °C	15	5	73 (65)
3	PdCl <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> , Cs <sub>2</sub> CO <sub>3</sub> , DMF, 90 °C	8	6	80 (70)
4	PdCl <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> , K <sub>3</sub> PO <sub>4</sub> , DMF, 90 °C	2	2	93 (96)
5	PdCl <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> , K <sub>3</sub> PO <sub>4</sub> , NMP, 90 °C	3	3	88 (80)
6	PdCl <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> , K <sub>3</sub> PO <sub>4</sub> , DMA, 90 °C	3	16	81 (80)
7	PdCl <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> , K <sub>3</sub> PO <sub>4</sub> , 1,4-dioxane, 90 °C	51	5	30 (21)
8	PdCl <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> , K <sub>3</sub> PO <sub>4</sub> , DMF, 25 °C	44	7	26 (17)
9	PdCl <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> , K <sub>3</sub> PO <sub>4</sub> , DMF, 90 °C	11	5	80 (75) <sup>e</sup>
10	PdCl <sub>2</sub> (PPh <sub>3</sub> ) <sub>2</sub> , no base, DMF, 90 °C	32	0	42 (33)
11	no catalyst, K <sub>3</sub> PO <sub>4</sub> , DMF, 90 °C	72	3	0

<sup>a</sup> Conditions: BiPh<sub>3</sub> (1 equiv, 0.25 mmol), 1-bromo-3,4-dihydronaphthalene-2-carbaldehyde (3.3 equiv, 0.825 mmol), base (2 equiv), PdCl<sub>2</sub>(PPh<sub>3</sub>)<sub>2</sub> (0.09 equiv, 0.0225 mmol), solvent (3 mL), 1 h.

<sup>b</sup> GC conversions of the crude reaction mixture.

<sup>c</sup> Isolated yields are given in parenthesis.

<sup>d</sup> Yields are calculated with respect to 3 equiv of cross-coupling product (0.75 mmol) as 100% yield.

<sup>e</sup> The reaction was carried out with 1 equiv of K<sub>3</sub>PO<sub>4</sub> base.

The initial screening of  $PdCl_2(PPh_3)_2$  as catalyst with a set of different bases was studied to check their efficacy in *N*,*N*-dimethylformamide (DMF) to give the product **2a** (Table 1, entries 1–4). From this study,  $K_3PO_4$  base was found to be a better choice in comparison with other bases

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studied (Table 1, entry 4), although it should be noted that bases such as K<sub>2</sub>CO<sub>3</sub>, Na<sub>2</sub>CO<sub>3</sub> and Cs<sub>2</sub>CO<sub>3</sub> also furnished the product 2a in reasonably good yields. Further study was carried out in different solvents (Table 1, entries 5-7). This study revealed DMF as the better solvent among other polar aprotic solvents studied such as N-methyl-2pyrrolidone (NMP) and N,N-dimethylacetamide, while 1,4-dioxane was found to be less effective. A coupling reaction carried out under room temperature condition provided lower product conversion (Table 1, entry 8). Further study with one equivalent of base produced 75% of coupled product (Table 1, entry 9). A control reaction without base furnished the cross-coupling product in lower yield (Table 1, entry 10), while reaction without catalyst failed to provide the desired cross-coupling product (Table 1, entry 11). As shown in the Table 1, the reaction also produced homocoupled biphenyl as a side product in varied amounts under the conditions studied. This is due to the facile homocoupling reactivity of triarylbismuths under the palladium-catalyzed conditions.<sup>8</sup> Unreacted bromide was also seen in these screening reactions and the amount varied with respect to formation of the cross-coupling product. Finally it emerged that PdCl<sub>2</sub>(PPh<sub>3</sub>)<sub>2</sub> with K<sub>3</sub>PO<sub>4</sub> base in DMF solvent was the most suitable combination to obtain product 2a in 96% isolated yield in one hour reaction time (Table 1, entry 4). It is noteworthy that the overall process involves three couplings from triphenylbismuth with three equivalents of bromoaldehyde to give three equivalents of cross-coupling product **2a** and these couplings were completed in a short reaction time.

This encouraged us to extend the study to assess the scope of these couplings using  $PdCl_2(PPh_3)_2$  (0.09 equiv),  $K_3PO_4$  base (2 equiv) in DMF solvent at 90 °C, for one hour as the optimized conditions.<sup>9,10</sup> As shown in Table 2, the cross-coupling reactions of a variety of functionalized triarylbismuths were studied with both 1-bromo-3,4-dihydronaphthalene-2-carbaldehyde and 1-bromo-3,4-dihydro-6-methoxynaphthalene-2-carbaldehyde compounds.

Regardless of the nature of triarylbismuths employed, the reactions afforded good to excellent yields of cross-coupled products. For example, electronically divergent triarylbismuths with both electron-rich and electrondeficient aryl groups fared well under the established palladium protocol condition (Table 2, entries 1-17). It should be highlighted that three equivalents of either 1bromo-3,4-dihydronaphthalene-2-carbaldehyde or 1-bromo-3,4-dihydro-6-methoxynaphthalene-2-carbaldehyde reacted very efficiently with one equivalent of triarylbismuth in a short time. We also examined the crosscoupling reaction using tris(thiophenyl-2-yl)bismuth and this afforded the corresponding product in high yield (Table 2, entry 18). Additionally, the sterically bulky tris(2-naphthyl)bismuth provided the corresponding coupled product in 89% yield (Table 2, entry 19).

Table 2 Cross-Couplings with Different Triarylbismuth Compoundsa-d



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 Table 2
 Cross-Couplings with Different Triarylbismuth Compounds<sup>a-d</sup> (continued)

	$-Bi$ + $R^2$	HO <u>PdCl₂(PPh₃)₂ (0.09 equiv)</u> K₃PO₄ (2 equiv) DMF, 90 °C, 1h				
(1 equiv)	(3 equiv)	D 111 1	(3 equiv)			X' 11 (0)
6 7	BIAI3 Bi-(C)-OMe) 3	Bromoaldenyde	Product OMe CHO R <sup>2</sup>	$R^{2} = H$ $R^{2} = OMe$	2d 3c	76 86
8	Bi	Br CHO	OMe CHO		2e	85
9 10	BiOEt) <sub>3</sub>	Br CHO	R <sup>2</sup> CHO	$R^2 = H$ $R^2 = OMe$	2f 3d	86 86
11 12	Bi-Oi-Pr) <sub>3</sub>		O <sup>i</sup> Pr CHO	$R^2 = H$ $R^2 = OMe$	2g 3e	95 85
13 14	Bi-CI) <sub>3</sub>		CI CHO R <sup>2</sup> CHO	$R^2 = H$ $R^2 = OMe$	2h 3f	83 76
15 16	BiF) <sub>3</sub>		F CHO	$R^2 = H$ $R^2 = OMe$	2i 3g	89 85
17	Bi	Br CHO	CF3 CHO		2j	68

 Table 2
 Cross-Couplings with Different Triarylbismuth Compounds<sup>a-d</sup> (continued)



<sup>a</sup> Reaction conditions: BiAr<sub>3</sub> (0.25 mmol, 1 equiv), bromoaldehyde (0.825 mmol, 3.3 equiv),  $K_3PO_4$  (0.5 mmol, 2 equiv), PdCl<sub>2</sub>(PPh<sub>3</sub>)<sub>2</sub> (0.0225 mmol, 0.09 equiv), DMF (3 mL), 90 °C, 1 h.

<sup>b</sup> Isolated yields are based on three couplings, i.e. 3 equiv of cross-coupling product (0.75 mmol) as 100% yield.

<sup>c</sup> Biaryl as homocoupled products were formed in minor amounts.

<sup>d</sup> All products were characterized by <sup>1</sup>H NMR, <sup>13</sup>C NMR, HRMS (ESI) and IR analyses.

Next we turned our attention to investigate the reactivity of  $\beta$ -bromo- $\alpha$ , $\beta$ -cycloalkenyl aldehydes with triarylbismuths.<sup>9,10</sup> As shown in Table 3, these bromoaldehydes also led to moderate to good yields of the coupled products. For example, reaction using (*Z*)-2-bromocyclo-1octenecarbaldehyde furnished a moderate yield of the coupling product (Table 3, entry 1). Both 2-bromo-5-*tert*butylcyclohex-1-enecarbaldehyde and 2-bromocyclopent-1-enecarbaldehyde reacted well in coupling reactions with a variety of triarylbismuth compounds under the established conditions (Table 3, entries 2–12). Once again, all the reactions furnished the corresponding crosscoupling products in a short reaction time.

Coupling products such as the 1-aryl-3,4-dihydronaphthalene derivatives are important intermediates in the synthesis of biologically active compounds,<sup>11,12</sup> useful as oral anti-fertility agents, in the treatment of diabetic complications and in combating HIV infections.

The cross-coupling of  $\beta$ -bromo  $\alpha$ , $\beta$ -unsaturated aldehydes with triarylbismuths is expected to follow a reaction pathway similar to that proposed by Stille et al. for the coupling reactions of vinylic substrates under palladiumcatalyzed conditions.<sup>13</sup> It is well established that the oxidative addition of organic halides to zero-valent palladium catalyst is a facile process.<sup>13a</sup> Based on this, it is proposed that the initial oxidative addition of vinyl halide to palladium(0) forms the organopalladium(II) intermediate **B** (Scheme 1). This, upon transmetalation with BiAr species would form the intermediate **C**. This is expected to undergo a facile reductive elimination to deliver the arylated product **D**. As the reaction involves transfer of three aryl groups during the reaction cycle, the possibility of aryl transfer from BiAr<sub>3</sub>, Ar<sub>2</sub>BiX or ArBiX<sub>2</sub> is not ruled out.



Scheme 1 Proposed catalytic cycle

The regeneration of  $BiAr_3$  from the disproportion reaction of arylbismuth halides generated in situ during the reaction could be another possibility for aryl transfer in the transmetalation step.<sup>8</sup>

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Table 3 Arylations of  $\beta$ -Bromoalkenyl-1-carbaldehydes<sup>a-d</sup> (continued)



<sup>a</sup> Reaction conditions: BiAr<sub>3</sub> (0.25 mmol, 1 equiv), bromoaldehyde (0.825 mmol, 3.3 equiv),  $K_3PO_4$  (0.5 mmol, 2 equiv), PdCl<sub>2</sub>(PPh<sub>3</sub>)<sub>2</sub> (0.0225 mmol, 0.09 equiv), DMF (3 mL), 90 °C, 1 h.

<sup>b</sup> Isolated yields are based on three couplings i.e. 3 equiv of cross-coupling product (0.75 mmol) as 100% yield.

<sup>c</sup> Biaryl as homocoupled products were formed in minor amounts.

<sup>d</sup> All products were characterized by <sup>1</sup>H NMR, <sup>13</sup>C NMR, HRMS (ESI) and IR analyses.

In conclusion a novel coupling method is described for the synthesis of functionalized cyclic tetrasubstituted olefin derivatives. This efficient protocol uses triarylbismuths as atom-efficient multicoupling nucleophiles with three equivalents of cyclic  $\beta$ -bromo  $\alpha$ , $\beta$ -unsaturated aldehydes in a synthetically versatile and a useful method under palladium-catalyzed conditions. The multicoupling ability of triarylbismuths has been demonstrated to be facile, as all coupling reactions were complete within one hour giving high yields of coupling products.

**Supporting Information** for this article is available online at http://www.thieme-connect.com/ejournals/toc/synlett.

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- (9)Representative Procedure: An oven-dried Schlenk tube under a nitrogen atmosphere was charged with 1-bromo-3,4dihydronaphthalene-2-carbaldehyde (0.825 mmol, 0.196 g) followed by triphenylbismuth (0.25 mmol, 0.11 g), K<sub>3</sub>PO<sub>4</sub> (0.50 mmol, 0.106 g), PdCl<sub>2</sub>(PPh<sub>3</sub>)<sub>2</sub> (0.0225 mmol, 0.0158 g) and anhyd DMF (3 mL). The reaction mixture was stirred in an oil bath at 90 °C for 1 h. Then the contents were cooled to r.t. and the reaction was quenched with  $H_2O(10 \text{ mL})$  and the mixture was extracted with EtOAc ( $3 \times 15$  mL). The combined organic extracts were washed with  $H_2O$  (2 × 10 mL), brine (10 mL), dried over anhyd MgSO<sub>4</sub> and concentrated. The crude product mixture thus obtained was purified by silica gel column chromatography using 1% EtOAc-petroleum ether as eluent to obtain 1-phenyl-3,4dihydronaphthale-2-carbaldehyde(2a) as a pale yellow solid (0.169 g, 96%). All the products were characterized by <sup>1</sup>H NMR, <sup>13</sup>C NMR, IR spectroscopic data and HRMS (ESI).
- (10) In all the coupling reactions, 0.3 equiv of bromoaldehydes was employed in excess. However, the product yields were calculated based on the three couplings from triarylbismuths. Thus, 0.75 mmol of the cross-coupled product corresponds to 100% yield.
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