Palladium-Indium-Indium(III) Chloride-Mediated Allyl Cross-Coupling Reactions Using Allyl Acetates

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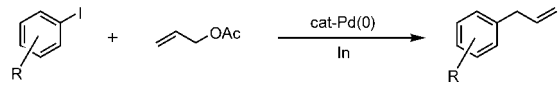
Abstract: Allylindiums *in situ* generated by reductive transmetalation of π-allylpalladium(II) complexes, obtained from allyl acetates and palladium(0) catalyst, with indium and indium(III) chloride are effective nucleophilic cross-coupling partners in Pd-catalyzed allyl cross-coupling reactions with a variety of electrophilic cross-coupling partners.

Keywords: allyl acetate; allylation; cross-coupling; indium; palladium

The widespread natural occurrence and potential for further functionalization of allylaromatic compounds have promoted a continued interest in synthetic methods for the introduction of allyl groups onto the aromatic nucleus.[1] Friedel–Crafts alkylation,[2] Claisen rearrangement,[3] substitution reactions of allyl halides with various aromatic organometallic reagents,[4] and the transition metal-mediated cross-couplings represent the most powerful allylation methods.[5] Among these, the Pd-catalyzed cross-coupling reaction has attracted much attention in the area of allylation methods. One of the most frequently used methods for allyl cross-coupling reactions involves the use of allylstannanes as coupling partners, which has attracted much attention as a result of their availability, air- and moisture-stability, and their compatibility with a variety of functional groups.[6] Although allylstannanes are generally accessible, such procedures are sometimes inadequate as the requisite allylmetals are difficult to obtain. The direct conversion of readily available allylic substrates to allylmetal species is desirable. Generally, the allylating reagents have been limited to allyl halides, particularly allyl bromides and iodides. Allyl chloride has been used with an iodide salt *via in situ* generated allyl iodide. Therefore, overcoming these shortcomings is a prerequisite for developing new allylmetals as coupling partners. Recently, a new method for the preparation of allylindiums *via* a reductive transmetalation[7] of π-allylpalladiums(II) complexes has been studied and then many successful examples of the Pd-In-mediated allylation of aldehydes,[8] imines,[9] and oximes[10] were reported. In connection with our current research interest in the synthetic utility of organoindium reagents,[11] we have reported Pd-catalyzed cross-coupling reactions of *in situ* generated allylindiums from allyl halides and indium with a variety of electrophilic coupling partners.[5 g, h] Our continuing efforts in the area of organoindium chemistry led us to find that allyl acetates, which are easily derived from available allyl alcohols, can be effectively utilized in the Pd-catalyzed allyl cross-coupling reaction, *via* π-allylpalladium(II) complexes and their transmetalation with In-$InCl_3$, in the presence of a catalytic amount of $Pd(Ph_3P)_4$ (Scheme 1).



Scheme 1.

We first investigated various experimental conditions in the model reaction using 4-*n*-butyliodobenzene (**1**) with allyl acetate (**2**) (Table 1). Of the solvents tested (DMF, THF, CH_3CN, and THF-H_2O), DMF was optimum. Among the catalysts examined, $Pd(Ph_3P)_4$ was the most suitable in terms of the yields. Other palladium complexes, such as $PdCl_2$, $Pd(OAc)_2$, $Pd(CH_3CN)_2Cl_2$, $Pd(PhCN)_2Cl_2$, and $Pd_2dba_3CHCl_3$ in the presence of Ph_3P produced 4-allyl-*n*-butylbenzene (**3**) in good yields. The combination of In and $InCl_3$ as a reductive transmetalating agent for π-allylpalladiums(II) complexes gave the allyl cross-coupling product in good yield.[8j] However, the desired product was obtained in only 30% yield with InI (entry 13).[8c] Also, the reaction was found to proceed efficiently in the presence of LiCl as an additive (entries 6, 9 and 10).[5g] A tertiary alkylamine was necessary to reduce side products such as *n*-butylbenzene and 4,4′-di-(*n*-butyl)-1,1′-biphenyl. Of the amines tested, *N*,*N*-dimethyl-*n*-butylamine gave the

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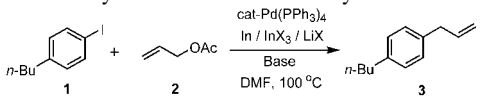
Table 1. Optimization of Pd-In-mediated cross-coupling reaction of 4-*n*-butyliodobenzene with allyl acetate.



1 2 3

Entry	InX_n	LiX	Base	Time [h]	Isolated Yield [%][a] 3	4-*n*-Bu-PhH[b]	(4-*n*-Bu-Ph)$_2$[b]
1[c]	$InCl_3$	LiCl	-	18	21	74	
2[d]	$InCl_3$	LiCl	Et_3N	42	14 21[e] (28)		
3	-	LiCl	Et_3N	18	58	15	15
4	$InCl_3$	LiCl	-	18	43	26	11
5	$InCl_3$	-	Et_3N	24	21	19	8
6	$InCl_3$	LiCl	Et_3N	6	54	11	27
7	$InBr_3$	LiCl	Et_3N	6	47	9	33
8	InI_3	LiCl	Et_3N	18	21 (41)	10	23
9	$InCl_3$	LiBr	Et_3N	18	36	15	41
10	$InCl_3$	LiI	Et_3N	18	16 (43)	29	6
11[f]	$InCl_3$	LiCl	K_2CO_3	18	21	35	33
12[d]	InI	LiCl	-	16	2 (95)	35	33
13[d]	InI	LiCl	*n*-BuNMe$_2$	16	30 (55)		
14	$InCl_3$	LiCl	*n*-BuNMe$_2$	9	65	10	10
15[c]	$InCl_3$	LiCl	*n*-BuNMe$_2$	16	31 (52)	7	

[a] Reactions performed in the presence of 10 mol % Pd(PPh_3)$_4$, 2.0 equivs. of In, 0.5 equivs. of InX_3, 3.0 equivs. of LiX, and 2.0 equivs. of base, unless otherwise noted. Numbers in parenthesis indicate the recovered yield of 4-*n*-butyliodobenzene.

[b] GC yield.

[c] 5 mol % Pd(PPh_3)$_4$ was used.

[d] Indium metal was not used.

[e] *trans*-1,3-Bis(4-*n*-butylphenyl)propene.

[f] 1.0 equiv. of K_2CO_3 was used.

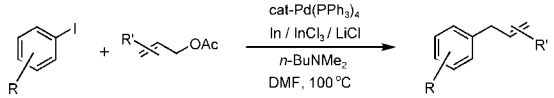
best result (entries 4, 6, and 14).[12] Inorganic bases resulted in low yields (entry 11). Of the reaction conditions examined, the best results were obtained with 10 mol % Pd(Ph_3P)$_4$, 2.0 equivs. of In, 0.5 equivs. of $InCl_3$, 3.0 equivs. of LiCl and 2 equivs. of *N*,*N*-dimethyl-*n*-butylamine in DMF at 100 °C for 9 h, which provided 4-allyl-*n*-butylbenzene (**3**) in 65% yield (entry 14). The use of allyl chloride, allyl alcohol, and acrolein diethyl acetal instead of allyl acetate in the Pd-In-mediated allyl cross-coupling reactions produced the desired compound **3** in 32%, 5% and 0% yields, respectively.

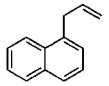
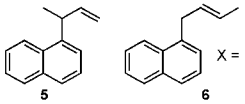
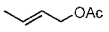
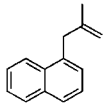
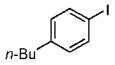
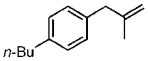
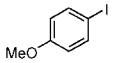
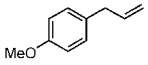
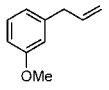
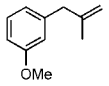
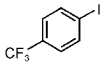
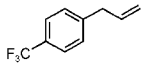
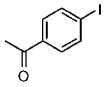
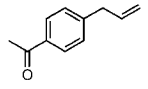
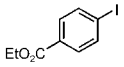
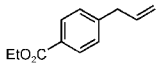
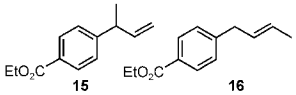
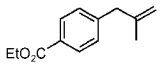
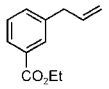
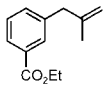
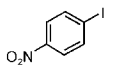
As shown in Table 2, this new catalytic protocol for the Pd-mediated allyl cross-coupling reactions of aryl halides with *in situ* generated allylindiums from allyl acetates in the presence of Pd-In-$InCl_3$ could be applied to a broad range of substrates, and it was found that a diverse range of functional groups were compatible with the reaction conditions. For example, aryl iodides bearing *n*-butyl (entry 7) and methoxy (entries 8–10) as electron-donating groups and trifluoromethyl (entry 11), acetyl (entry 12), ethoxycarbonyl (entries 13–17), and nitro (entry 18) as electron-withdrawing groups smoothly reacted with allyl acetate in the presence of Pd(0)-In-$InCl_3$ to give the corresponding allylaromatic compounds in moderate to good yields. These results mean that electron density on the aromatic ring does not seriously effect the efficiency of the reactions. Aryl iodides are more reactive than aryl bromides and give a higher yield of the cross-coupling product (entries 1 and 2). However, the triflate of 1-naphthol afforded 1-allylnaphthalene (**4**) in 33% yield under optimized conditions (entry 3). It should be noted that the present conditions could be applied to carbonyl-bearing aryl iodides (entries 12–17). Vinyl triflates worked equally well with *in situ* generated allylindium from allyl acetate, producing the allylic compound **21** in 84% yield (entry 19). α-Bromostyrene was treated with allyl acetate to give a 59% yield of 2-phenyl-1,4-pentadiene (**22**) and a 7% yield of *trans*-1-phenyl-1,4-pentadiene (**23**) (entry 20). For allyl acetates as coupling partners, the presence of a methyl substituent at the α, β or γ position affected both reaction rates and product yields. The use of allyl acetate and 2-methylallyl acetate in the allyl cross-coupling reaction gave the corresponding allylic compounds in good yields. Reaction of 1-iodonaphthalene with allyl acetate gave the allyl cross-coupling product (**4**) in 81% yield under the optimized conditions (entry 2). 1-Iodonaphthalene was treated with 2-methylallyl acetate to produce **7** in 74% yield (entry 6). Subjecting 1-iodonaphthalene to the allylindium derived from the corresponding acetate of 3-buten-2-ol afforded the desired products **5** and **6** in 57% yield, but the product **5** resulting from α-attack predominated (α:γ = 3.7:1) (entry 4). Although reaction of 1-iodonaphthalene with crotyl acetate gave the allyl cross-coupling products (**5** and **6**) in 46% yield, the regioselectivity of this reaction was similar to that of 1-methyl-2-propenyl acetate to provide **5** as the major compound (entry 5).[5g,h]

Although the mechanism of the present reaction has not been established, a possible reaction mechanism for the Pd-In-mediated allyl cross-coupling reaction of aryl iodides with allyl acetate is shown in Scheme 2. The initially formed π-allylpalladium(II) complex undergoes reductive transmetallation by In and $InCl_3$ to give the allylindium(III) species, which subsequently undergoes cross-coupling with the aryl iodide in the presence of Pd(0) catalyst to yield allylarenes. The present reaction is one of the good examples in which the Pd(0) catalyst is simultaneously involved to two catalytic cycles in a one-pot reaction (Scheme 2).

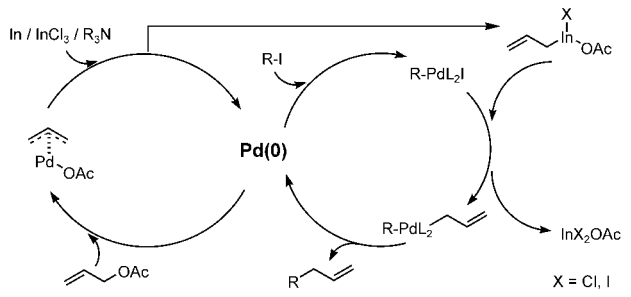
In conclusion, *in situ* generated allylindiums derived from the reductive transmetalation of π-allylpalladium(II) complexes, obtained from a variety of allyl acetates and a Pd(0) catalyst, with In and $InCl_3$ are effective nucleophilic cross-coupling partners in Pd-catalyzed cross-coupling reactions. A variety of electrophilic cross-coupling partners such as aryl iodides, vinyl bromide and triflates participate in this reaction. The present method complements existing synthetic methods as a result of advantageous features such as the ready availability of allyl acetates, the high reaction efficiency and operational simplicity.

Table 2. Pd-In-mediated allyl cross-coupling reactions of electrophilic coupling partners with allyl acetates.



Entry	Electrophiles	Allyl Acetates	Products		Isolated Yield [%][a]	Other Products[b]
1				X = Br	35 (41)	
2				I	81	
3			**4**	OTf	33 (50)	
4					57[c]	ArH 23% ArAr 7%
5			**5** **6**	X = I	46[d]	ArH 27% ArAr 18%
6				**7** X = I	74	ArH 12% ArAr 3%
7				**8**	59[e] 62	ArH 10% ArAr 24% ArH 14% ArAr 9%
8				**9**	66	ArAr 21%
9				**10**	75	ArAr 9%
10				**11**	61	ArAr 21%
11				**12**	60[f]	ArAr 20%
12				**13**	64 (4[a], 21[h])	
13				**14**	80[f]	ArAr 12%
14				**15** **16**	57[f][i]	ArAr 17%
15				**17**	63[f]	ArAr 19%
16				**18**	63[j] 80	ArH 12% ArAr 10% ArAr 8%
17				**19**	53[j] 57	ArAr 29% ArAr 22%
18				**20**	74[f][k]	
19				**21**	84	
20				**22** **23**	66[l] (23)	

[a] Reactions performed in the presence of 10 mol % $Pd(PPh_3)_4$, 2.0 equivs. of In, 0.5 equivs. of $InCl_3$, 3.0 equivs. of LiCl, and 2.0 equivs. of *n*-$BuNMe_2$, unless otherwise noted. Numbers in parenthesis indicate recovered yield of starting material. [b] GC yield. [c] Ratio of 5 to 6 = 3.7 : 1. [d] Ratio of **5** to **6** = 2.3 : 1. [e] Et_3N was used. [f] 5 mol % $Pd(PPh_3)_4$ was used. [g] 2-(4-Allylphenyl)-4-penten-2-ol. [h] 4,4′-Bis(2-hydroxy-4-penten-2-yl)-1,1′-biphenyl. [i] Ratio of **15** to **16** = 2.9 : 1. [j] *n*-$BuNMe_2$ was not used. [k] Base was not used. Reaction proceeded at 25 °C. [l] Ratio of **22** to **23** = 8.4 : 1.



Scheme 2. Plausible mechanism for Pd-catalyzed allyl cross-coupling reaction.

Experimental Section

Typical Experimental Procedure

To a suspension of indium (114.8 mg, 1.0 mmol), indium(III) chloride (55.29 mg, 0.25 mmol), lithium chloride (63.5 mg, 1.5 mmol), and $Pd(PPh_3)_4$ (57.3 mg, 10 mol %) in DMF (1 mL) was added *N,N*-dimethylbutylamine (101.2 mg, 2.0 mmol) and allyl acetate (250.3 mg, 2.5 mmol) at 35 °C under a nitrogen atmosphere. After 30 min, 1-iodonaphthalene (127.0 mg, 0.5 mmol) in DMF (1 mL) was added and the mixture was stirred at 100 °C for 9 h. The reaction mixture was quenched with $Na_2S_2O_3$ (saturated aqueous). The aqueous layer was extracted with ether (3 × 20 mL), and the combined organic layers were washed with water and brine, dried with $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography using *n*-hexane to give 1-allylnaphthalene; yield: 68.5 mg (81%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.01 (d, *J* = 8.08 Hz, 1H), 7.83 (t, *J* = 7.76 Hz, 1H), 7.72 (d, *J* = 6.89 Hz, 1H), 7.50–7.42 (m, 2H), 7.39 (d, *J* = 7.25 Hz, 1H), 7.32 (d, *J* = 6.89 Hz, 1H), 6.16–6.65 (m, 1H), 5.11–5.06 (m, 2H), 3.82 (d, *J* = 6.30 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 135.13, 133.27, 132.01, 130.15, 126.84, 125.13, 124.44, 123.96, 123.77, 123.68, 122.19, 114.33, 35.43; IR (film): ν = 3003, 2916, 1684, 1606, 1414, 1358 cm^{-1}; HR-EI-MS: calcd. for $C_{13}H_{12}$: 168.0939; found: 168.0934.

Acknowledgements

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