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Platinum-catalyzed allylation of aminonaphthalenes with allylic acetates in water

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ABSTRACT

The activation of C–O bonds in allylic acetates in water as a suspension medium has been accelerated by carrying out the reactions in the presence of platinum complexes associated with ligands. The platinum-catalyzed allylation of aminonaphthalenes using allylic acetates gave the corresponding *N*-allylic aminonaphthalenes in good yields.

1. Introduction

A principal goal of organometallic chemistry is the catalytic synthesis of organic compounds by using the chemistry of organic ligands covalently bound to transition metals. Transition metals, in particular palladium and rhodium, have been workhorse elements in many commercialized catalytic processes that include hydrogenations, hydroformylations, acetic acid production, and other C–C and C–H bond forming processes.[1–4] Transition metal η^3-allyl complexes, as well as transition metal σ-alkyl complexes, play important roles as active species and key intermediates in many reactions catalyzed by transition metal complexes.[5] The palladium-catalyzed allylation is a powerful tool for C–C, C–N, and C–O bond formation, which has been widely applied to organic chemistry.[6–8] The processes have been shown to proceed by attack of nucleophiles on intermediate η^3-allylpalladium(II) complexes generated by oxidative addition of allylic compounds including halides,[9–11] esters,[12–20] carbonates,[21–25] carbamates,[26,27] phosphates,[28,29] and related derivatives[30–33] to a Pd(0) complex. Aromatic amines have not been used commonly in allylic amination, presumably because they are less nucleophilic than the more commonly used benzylamine or stabilized anionic nitrogen nucleophiles.[34,35] We have reported our attempts and some successful applications of a process involving the C–O bond cleavage with direct use of allylic alcohols catalyzed by palladium complexes in the presence of $Ti(O^iPr)_4$ in benzene.[36–39] Organic reactions in water have recently attracted much attention, because water is a safe and economical substitute for conventional organic solvent.[40–46] Thus, development of atom-economical reactions in water is one of the most important goals of synthetic chemistry. However, platinum-catalyzed allylation has attracted little attraction.[47–50] Herein, we will describe a novel catalysis of platinum complex, which mediates *N*-allylation of aminonaphthalenes with allylic acetates using water as solvent.

2. Results and discussion

The platinum-catalyzed allylation of 1-aminonaphthalene with allyl acetate was investigated under various conditions (Scheme 1). When a mixture of 1-aminonaphthalene (**1a**, 2 mmol) and allyl acetate (**2a**, 2.4 mmol) was stirred in the presence of catalytic amounts of $Pt(acac)_2$ (0.02 mmol) and PPh_3 (0.08 mmol) in water at room temperature for 30 min, *N*-allyl-1-naphthylamine (**3a**) was formed in only 17% yield (entry 1 in Table 1). The reaction, under reflux, increased the yields of products **3a** and *N*,*N*-diallyl-1-naphthylamine (**4a**) to 90 and 6%, respectively (entry 3). The reaction gave 59% yield under reflux for 15 min (entry 4). As expected, increasing the relative amount of allyl acetate favored the formation of the desired diallylated product **4a** (entries 5 and 6). The reaction did not occur in the absence of the platinum species (entry 7) or phosphine ligand (entry 8). Using MeOH and EtOH as solvents gave only low yields of products (entries 9 and 10). Among the platinum catalysts including $Pt(acac)_2$ (entry 3), *cis*-$PtCl_2(PhCN)_2$ (entry 11), *cis*-$PtCl_2(PPh_3)_2$ (entry 12), $PtCl_2$ (entry 13), PtI_2 (entry

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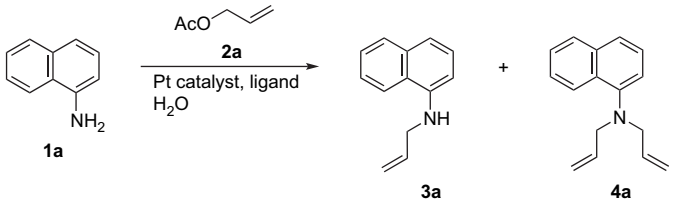


Scheme 1. Allylation of 1-aminonaphthalene (**1a**) with allyl acetate (**2a**).

14), $Pt(CN)_2$ (entry 15), $O[Si(CH_3)_2C{=}CH_2]_2Pt$ (entry 16), $Pt(CH_2{=}CH_2)(PPh_3)_2$ (entries 17 and 18), and $Pt(PPh_3)_4$ (entries 19 and 20) were used. $Pt(acac)_2$, $O[Si(CH_3)_2C{=}CH_2]_2Pt$, and $Pt(CH_2{=}CH_2)(PPh_3)_2$ were found to be the superior. The use of $O[Si(CH_3)_2C{=}CH_2]_2Pt$ as catalyst was cheaper than palladium reagents and could give good results. However, using $Pt(CH_2{=}CH_2)(PPh_3)_2$ or $Pt(PPh_3)_4$ with extra PPh_3 as catalyst increased the yield of products (entries 18 and 20). The catalytic reactivity of the phosphine ligands is likely due to improved catalyst stability. In the presence of various monodentate ligands including PPh_3, Bu_3P, $(2\text{-}MeC_6H_4)_3P$, $(3\text{-}MeC_6H_4)_3P$, $(4\text{-}MeC_6H_4)_3P$, $(4\text{-}FC_6H_4)_3P$, $(4\text{-}ClC_6H_4)_3P$, $(4\text{-}MeOC_6H_4)_3P$, $[2,4,6\text{-}(MeO)_3C_6H_2]_3P$, $(2\text{-furyl})_3P$, and $(2\text{-pyridyl})Ph_2P$ (entries 3 and 21–30) showed that PPh_3 (entry 3), $(3\text{-}MeC_6H_4)_3P$ (entry 23), $(4\text{-}MeC_6H_4)_3P$ (entry 24), $(4\text{-}FC_6H_4)_3P$ (entry 25), $(4\text{-}ClC_6H_4)_3P$ (entry 26), and $(2\text{-furyl})_3P$ (entry 29) were the most effective ligands. Using $O[Si(CH_3)_2C{=}CH_2]_2Pt$ associated with $(4\text{-}FC_6H_4)_3P$ as catalyst gave 99% yield (entry 31). The bidentate ligands such as dppm, dppb, and dpph gave worst results (entries 32–34).

Results for N-allylation of a number of aminonaphthalenes (**1b**–**l**) with allyl acetate (**2a**) using $Pt(acac)_2$ and PPh_3 in water are summarized in Table 2. 2-Methy-1-aminonaphthalene (**1b**) gave mono- and diallylated products in high yields (entry 1). 4-Bromo-1-aminonaphthalene (**1c**) also reacted to give the *N*-allylated products in moderate yields (entry 2). Using aminonaphthalenes having strong electron-withdrawing groups, such as the cyano group, under reflux for 3 h, only the monoallylated product *N*-allyl-1-(4-cyanonaphthyl)amine (**3d**) was obtained, however, in a very low yield (entry 3). These differences in reactivity could be related to the nucleophilicity of the corresponding aminonaphthalene. The lower yield observed may arise from the nature of the cyano group. The more acidic 1-amino-4-cyanonaphthalene (**1d**) is probably less reactive in attack on the π-allyl complex. Thus, 4-nitro-1-aminonaphthalene (**1e**) and 5-amino-1-naphthalenesulfonic acid (**1f**) under reflux for 3 h gave no products (entries 4 and 5).

Table 1
Platinum-catalyzed allylation of 1-aminonaphthalene (**1a**) with allyl acetate (**2a**)[a]

Entry	Platinum catalyst	Ligand	Yield (%)[b] (**3a**+**4a**)	Yield (%)[b] of **3a**	Yield (%)[b] of **4a**
1[c]	$Pt(acac)_2$	PPh_3	17	17	
2[d]	$Pt(acac)_2$	PPh_3	28	27	1
3	$Pt(acac)_2$	PPh_3	96	90	6
4[e]	$Pt(acac)_2$	PPh_3	59	53	6
5[f]	$Pt(acac)_2$	PPh_3	83	79	4
6[g]	$Pt(acac)_2$	PPh_3	99	54	45
7	—	PPh_3	0		
8	$Pt(acac)_2$	—	0		
9[h]	$Pt(acac)_2$	PPh_3	2	2	
10[i]	$Pt(acac)_2$	PPh_3	5	5	
11	*cis*-$PtCl_2(PhCN)_2$	PPh_3	79	73	6
12	*cis*-$PtCl_2(PPh_3)_2$	PPh_3	81	75	6
13	$PtCl_2$	PPh_3	85	79	6
14	PtI_2	PPh_3	16	16	
15	$Pt(CN)_2$	PPh_3	2	2	
16	$O[Si(CH_3)_2C{=}CH_2]_2Pt$	PPh_3	89	76	13
17	$Pt(CH_2{=}CH_2)(PPh_3)_2$	—	77	74	3
18	$Pt(CH_2{=}CH_2)(PPh_3)_2$	PPh_3	95	90	5
19	$Pt(PPh_3)_4$	—	37	37	
20	$Pt(PPh_3)_4$	PPh_3	87	80	7
21	$Pt(acac)_2$	Bu_3P	3	3	
22	$Pt(acac)_2$	$(2\text{-}MeC_6H_4)_3P$	11	3	8
23	$Pt(acac)_2$	$(3\text{-}MeC_6H_4)_3P$	95	82	13
24	$Pt(acac)_2$	$(4\text{-}MeC_6H_4)_3P$	96	90	6
25	$Pt(acac)_2$	$(4\text{-}FC_6H_4)_3P$	99	71	28
26	$Pt(acac)_2$	$(4\text{-}ClC_6H_4)_3P$	99	74	25
27	$Pt(acac)_2$	$(4\text{-}MeOC_6H_4)_3P$	61	56	5
28	$Pt(acac)_2$	$[2,4,6\text{-}(MeO)_3C_6H_2]_3P$	1	1	
29	$Pt(acac)_2$	$(2\text{-Furyl})_3P$	98	77	21
30	$Pt(acac)_2$	$(2\text{-Pyridyl})Ph_2P$	68	63	5
31	$O[Si(CH_3)_2C{=}CH_2]_2Pt$	$(4\text{-}FC_6H_4)_3P$	99	76	23
32	$Pt(acac)_2$	Dppm[j]	3	3	
33	$Pt(acac)_2$	Dppb[k]	8	8	
34	$Pt(acac)_2$	Dpph[l]	11	11	

[a] Reaction conditions: **1a** (2 mmol), **2a** (2.4 mmol), Pt catalyst (0.02 mmol), and PPh_3 (0.08 mmol) in water (5 mL) were refluxed for 30 min.
[b] Isolated yield.
[c] Stirred at room temperature.
[d] Stirred at 50 °C.
[e] Reflux for 15 min.
[f] 1.6 mmol of **2a** was used.
[g] 4 mmol of **2a** was used.
[h] Using MeOH as solvent.
[i] Using EtOH as solvent.
[j] 1,3-Bis(diphenylphosphino)methane.
[k] 1,4-Bis(diphenylphosphino)butane.
[l] 1,6-Bis(diphenylphosphino)hexane.

Table 2
Reaction of aromatic amines (**1b–l**) with allyl acetate (**2a**)[a]

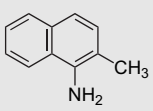
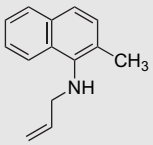
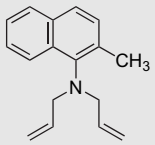
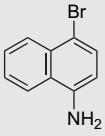
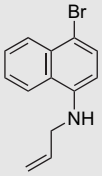
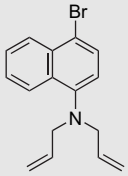
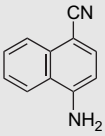
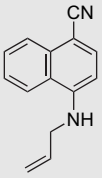
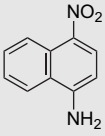
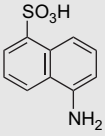
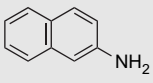
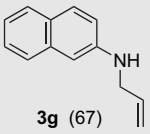
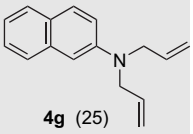
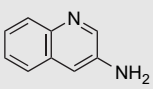
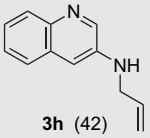
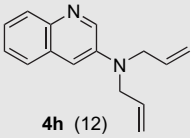
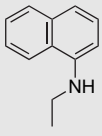
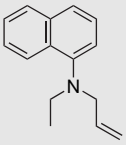
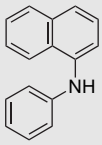
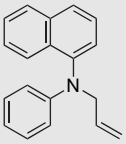
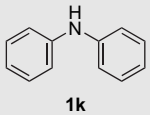
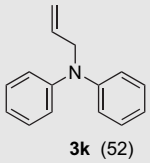
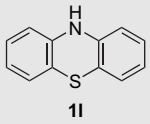
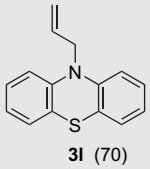
Entry	1	Yields[b] (%)	
1	 **1b**	 **3b** (58)	 **4b** (34)
2[c]	 **1c**	 **3c** (52)	 **4c** (2)
3[d]	 **1d**	 **3d** (11)	
4[d]	 **1e**	—	—
5[d]	 **1f**	—	—
6	 **1g**	 **3g** (67)	 **4g** (25)
7[c]	 **1h**	 **3h** (42)	 **4h** (12)
8	 **1i**	 **3i** (75)	
9[d]	 **1j**	 **3j** (20)	

Table 2 (*continued*)

Entry	1	Yields[b] (%)
10[c]	 **1k**	 **3k** (52)
11[d]	**1k**	**3k** (85)
12[d]	 **1l**	 **3l** (70)

[a] Reaction conditions: **1** (2 mmol), **2a** (2.4 mmol), $Pt(acac)_2$ (0.02 mmol), and PPh_3 (0.08 mmol) in water (5 mL) were refluxed for 30 min.
[b] Isolated yield.
[c] Reflux for 1 h.
[d] Reflux for 3 h.

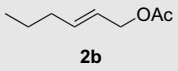
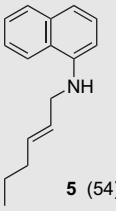
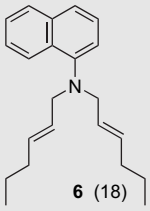
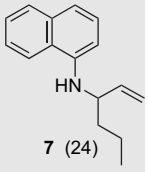
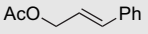
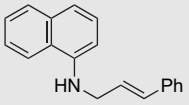
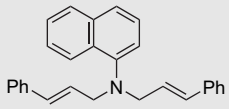
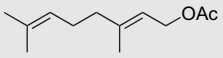
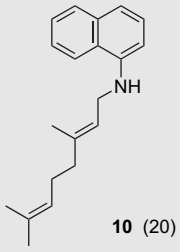
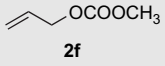
2-Aminonaphthalene (**1g**) gave mono- and diallylated products **3g** and **4g** in the yields of 67 and 25%, respectively (entry 6). 3-Aminoquinoline (**1h**) and *N*-ethyl-1-aminonaphthalene (**1i**) reacted to give the *N*-allylated products in moderate yields (entries 7 and 8). The sterically more demanding secondary aminonaphthalene (**1j**) gave lower yields (entry 9). Secondary aromatic amines, such as diphenylamine (**1k**) and phenothiazine (**1l**), also reacted to give the *N*-allylamine in moderate yield (entries 10 and 12). Increasing the refluxing time to 3 h, diphenylamine (**1i**) could give good yields of products (entry 11).

The results for amination of a number of derivatives of allylic alcohols **2b–f** with 1-aminonaphthalene (**1a**) using $Pt(acac)_2$ and PPh_3 are summarized in Table 3. Amination of *trans*-2-hexen-1-yl acetate (**2b**) gave mixtures of monoallylated, diallylated, and regioisomeric aminonaphthalenes **5**, **6**, and **7** in yields of 54, 18, and 24%, respectively (entry 1 in Table 3). These products may all be derived from the same π-allyl intermediate, which can be attacked at either the C-1 or C-3 position. The reaction is considered to proceed via π-allylplatinum intermediates. The loss of the stereochemistry of the starting acetate **2b** is due to a rapid $\sigma \leftrightarrows \eta^3 \leftrightarrows \sigma$ interconversion of the π-allyl intermediate compared to the rate of amination of this intermediate. In the reaction of aromatic allylic acetate **2c**, the corresponding monoallylated and diallylated products were formed in overall 99% yields (entry 2). Amination of geranyl acetate (**2d**) gave only monoallylated product **10** in 20% yield (entry 3). Increasing the refluxing time to 3 h could give the product **10** in 56% yield (entry 4). With the allyl chloride (**2e**), the reaction produces only **3a** in 23% yield (entry 5). Allyl carbonate (**2f**) gave products in high yields (entry 6).

3. Conclusion

In summary, we have developed a catalytic system that enables reactions of aromatic amines with allylic acetates as allylating agents in water. This is a simple and efficient route for C–N bond formation. The reaction did not occur in the absence of the phosphine ligand or platinum catalyst. The amination of allylic acetates worked well with aminonaphthalenes, giving generally good yields of the corresponding allylic aminonaphthalenes. Aminonaphthalenes with steric constraints gave lower chemical yields.

Table 3
Reaction of 1-aminonaphthalene (**1a**) with allylic compounds (**2b–f**)[a]

Entry	2	Yields[b] (%)		
1	 **2b**	 **5** (54)	 **6** (18)	 **7** (24)
2	 **2c**	 **8** (66)	 **9** (33)	
3	 **2d**	 **10** (20)		
4[c]	**2d**	**10** (56)		
5	 **2e**	**3a** (23)		
6	 **2f**	**3a** (68)	**4a** (31)	

[a] Reaction conditions: **1a** (2 mmol), **2** (2.4 mmol), $Pt(acac)_2$ (0.02 mmol) and PPh_3 (0.08 mmol) in water (5 mL) were refluxed for 30 min.
[b] Isolated yield.
[c] Reflux for 3 h.

4. Experimental

4.1. General considerations

IR absorption spectra were recorded on a Perkin–Elmer System 2000 FT-IR spectrophotometer. Proton and carbon-13 NMR were measured with a Unity-400 or Mercury Plus-400 spectrometer. Carbon multiplicities were obtained from DEPT experiments. Chemical shifts (δ) and coupling constants (Hz) were measured with respect to TMS or chloroform-d_1. MS and high-resolution mass spectra (HRMS) were taken on a Thermo-Finnigan trace GC or Finnigan MAT-95XL instrument, with a direct inlet system.

4.2. General procedure for the platinum-catalyzed allylation of aminonaphthalenes. Reaction with 1-aminonaphthalene (1a)

A mixture of 1-aminonaphthalene (**1a**) (286 mg, 2 mmol), allyl acetate (**2a**) (240 mg, 2.4 mmol), $Pt(acac)_2$ (7.8 mg, 0.02 mmol), and PPh_3 (21 mg, 0.08 mmol) in water (5 mL) was refluxed for 30 min. After the mixture was cooled to room temperature, water and brine were added. The organic materials were extracted with dichloromethane, dried over magnesium sulfate, and concentrated under vacuum. Column chromatography (ethyl acetate/*n*-hexane 1:4) of the residue afforded **3a** and **4a** in 90 and 6% yields, respectively.

Products **3a**,[50] **4a**,[50] **3b**,[50] **4b**,[50] **3c**,[50] **4c**,[50] **3d**,[50] **3g**,[50] **4g**,[50] **3h**,[50] **4h**,[50] **3i**,[50] **3j**,[50] **3k**,[39] **3l**,[39] **5**,[50] **6**,[50] **7**,[50] **8**,[50] and **9**[50] are known.

4.2.1. *N-(3,7-Dimethyl-2E,6-octadienyl)-1-naphthylamine (**10**)*

Light yellow thick oil. IR (KBr): ν 3422 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ 1.62 (s, 3H, CH_3), 1.70 (s, 3H, CH_3), 1.74 (s, 3H, CH_3), 2.06–2.17 (m, 4H, $CH_2\times2$), 3.88 (d, J=6.8 Hz, 2H, CH_2), 4.21 (br s, 1H, NH), 5.09–5.14 (m, 1H, vinyl H), 5.46–5.50 (m, 1H, vinyl H), 6.65 (d, J=7.2 Hz, 1H, ArH), 7.25 (d, J=8.0 Hz, 1H, ArH), 7.36 (t, J=8.0 Hz, 1H, ArH), 7.40–7.45 (m, 2H, ArH), 7.77–7.83 (m, 2H, ArH). ^{13}C NMR (100 MHz, $CDCl_3$): δ 16.4 (CH_3), 17.7 (CH_3), 25.7 (CH_3), 26.4 (CH_2), 39.5 (CH_2), 42.2 (CH_2), 104.5 (CH), 117.2 (CH), 119.9 (CH), 121.3 (CH), 123.4 (C), 123.9 (CH), 124.6 (CH), 125.6 (CH), 126.6 (CH), 128.6 (CH), 131.7 (C), 134.2 (C), 139.5 (C), 143.5 (C). EIMS: m/z 279 (M^+), 264, 236, 222, 210, 197, 180, 168, 154, 143, 127, 115, 89, 77. EI-HRMS calcd for $C_{20}H_{25}N$: 279.1987, found: 279.1987.

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