

Pd-Catalyzed Allylic Substitution Using Nucleophilic Amines: Access to Functionalized Mono- and Bis-*N*-Allyl Synthons

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Abstract: We here report a catalytic strategy to enable the decarboxylative allylic amination of vinyl cyclic carbonates using various aliphatic and nucleophilic amines. The use of a protic medium and chelating diphosphine ligands are the main drivers towards chemoselective allylic amine formation, thereby minimizing undesired ligand-driven complex speciation and aminolysis of the involved substrate. This improved approach amplifies the repertoire of allylic amine synthons that can be prepared from a variety of substrate combinations.

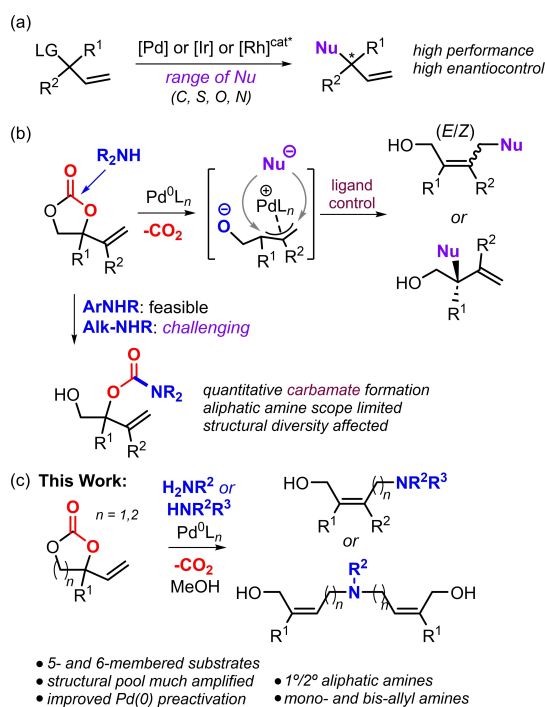
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Introduction

Metal-catalyzed allylic substitution chemistry plays a central role in the development of compounds of use in fine chemical synthesis and towards the discovery of bioactive compounds.^[1–3] Allylic substitutions are versatile C–X bond formation reactions that are typically promoted by transition metal catalysts derived from Pd,^[4] Ir^[5] or Rh.^[6] In the last decades, these conversions have been advanced to challenging asymmetric ones giving access to compounds with elusive (carbon) stereocenters (Scheme 1a).^[7] We have been interested in Pd-mediated allylic aminations enabled by substrates known as vinyl cyclic carbonates (VCCs, Scheme 1b).^[8] VCCs feature a carbonate leaving group and extrude CO₂ upon allylic substitution. These formal decarboxylative catalytic processes are, however, limited in scope as VCCs themselves undergo

undesired ring-opening in the presence of *nucleophilic* amines producing linear carbamates as the predominant product even in the presence of a Pd precatalyst.^[9] This significantly limits the structural scope of the allylic amines that can be prepared from VCCs. In addition, often related catalytic aminations also show similar limitations (i.e., catalyst poisoning/deactivation, undesired side-reactions) when such reactive substrates are present.^[10]

In the course of our ongoing research, we observed that the in situ combination of Pd₂(dba)₃ and (monodentate) phosphoramidite ligands gave rise to mixtures of components.^[4,11] This aligns with previous work in the area that showed that Pd-based catalyst speciation is a function of the dynamic and competitive coordination behavior of dba.^[12] We surmised that such a speciation could influence the relative rates of allylic amination versus undesired aminolysis of VCCs



Scheme 1. (a) Asymmetric synthesis of branched allylic amines; (b) Preparation of stereodefined linear analogues from VCCs as allylic precursors with current limitations; (c) This work.

(leading to linear carbamates). We thus set out to provide a more defined and well-behaved Pd(0) complex for VCC activation and formation of the key Pd(II)–allyl complex. Here we show that upon combining Pd(OAc)₂ and bidentate DPEPhos as catalyst in a protic solvent, these previous issues are circumvented. More specifically, active Pd(0) complex formation in the newly developed protocol allows to prepare new types of previously inaccessible allylic amines with stereodefined trisubstituted C=C bonds forged from both five- and six-membered vinyl carbonates and a range of primary and secondary aliphatic amines.^[13]

Results and Discussion

At the onset of the screening phase, we used six-membered cyclic carbonate **1a**^[14] and aniline **2a** as benchmark substrates to test our hypothesis. Based on previous reports,^[4a,8] we compared various Pd precursors and ligands (Table 1), and solvents to monitor the *chemo*-selectivity of the reaction. In this first approximation, we focused on simple aniline as the amine to compare to our previous results in terms of overall performance and stereo-outcome. The amination of **1a** with **2a** proceeds with different degrees of success judging from the product mixtures that were attained. Different combinations of solvents (Table S1 and S6), Pd precursors (Table S2), ligands (Table 1, entries 1–

Table 1. Screening of various conditions towards the preparation of allylic amine **3a**.

Entry ^[a]	[Pd]/L	Conv. (%) ^[b]	Solv./Add.	3a ^[b] (%)	3ac ^[b] (%)
1 ^[c]	Pd1/L1	63	DMF	26	< 1
2 ^[d]	Pd1/L1	47	THF	35	5
3	Pd1/L1	> 99	MeOH	64	8
4	Pd2/L1	71	MeOH	30	19
5	Pd3/L1	84	MeOH	37	6
6	Pd4/L1	> 99	MeOH	70	3
7	Pd4/L2	> 99	MeOH	50	9
8 ^[e]	Pd4/L3	43	MeOH	15	2
9	Pd4/L1	> 99	MeOH ^[g]	80	4
10 ^[f]	Pd4/L1	> 99	MeOH ^[g]	79	4

[Pd1]: Pd(OAc)₂-bis-sulfoxide (White)
[Pd2]: Pd₂(dba)₃·CHCl₃
[Pd3]: Pd(PPh₃)₄
[Pd4]: Pd(OAc)₂

L1:
L2:
L3:

^[a] Reaction conditions: carbonate **1a** (0.10 mmol), aniline **2a** (1.5 equiv., 0.15 mmol), [Pd] (2 mol%), **L** (5 mol%), solvent (200 μ L), r.t., 15 h.

^[b] Determined by ¹H NMR (CDCl₃) using mesitylene as internal standard.

^[c] Also **3aa** (6%) was observed.

^[d] Also **3ab** (10%) was observed.

^[e] Also **3ab** (4%) was formed.

^[f] Carbonate **1a** (0.20 mmol), aniline **2a** (1.5 equiv., 0.30 mmol), [Pd] (2 mol%), **L** (5 mol%), solvent (200 μ L), additive (20 mol%), r.t., 15 h.

^[g] Using 20 mol% NEt₃ as additive.

10 and Table S3), additives (Table S4) and relative amounts of **1a** and **2a** (Table S5) were examined.

We first used the White Pd-precursor (**Pd1**) and DPEPhos **L1** (entry 1) and found that the target product **3a** was only formed in 26% NMR yield. Together with **3a**, a low amount of a ketone-based byproduct (**3aa**, 6%) was noted. Changing the solvent to THF (entry 2) did improve the yield for **3a** (35%). However, under these latter conditions other types of byproducts, *viz.* the diene **3ab** (10%) and bis-allyl derivative **3ac** (5%), were formed. By switching to MeOH as a medium, a much-improved yield for **3a** (entry 3, 64%) was obtained. Building on this latter observation, we then further varied both the [Pd]

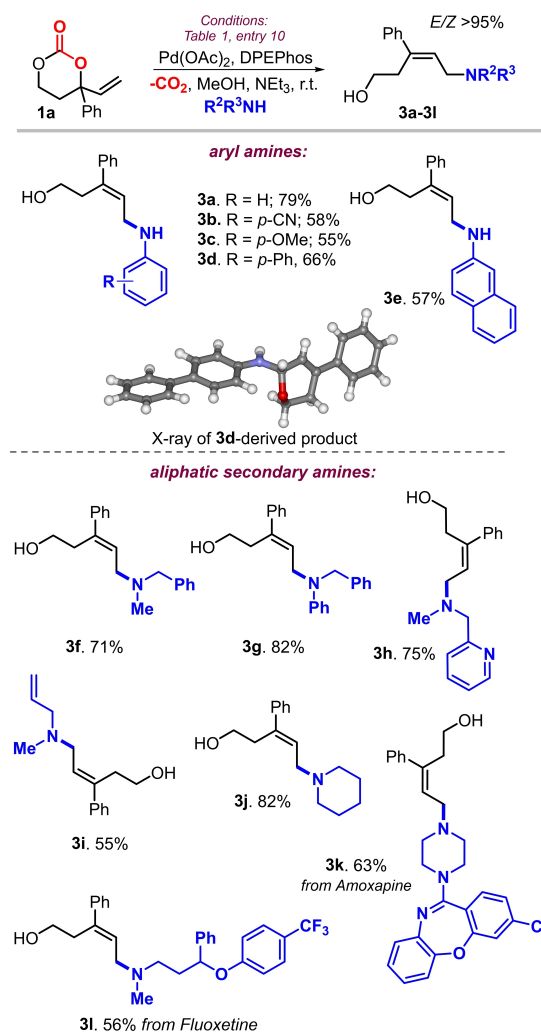
precursor and **L** (**Pd1–Pd4**, **L1–L3**). We found eventually that the best conditions involved applying simple Pd(OAc)₂ and DPEPhos in MeOH (entries 9 and 10) producing **3a** in good yield (79–80%) and with high chemo-selectivity. These results can be rationalized by assuming that MeOH hydrogen-bonds to the amine reagent thereby reducing its ability to coordinate to the Pd-species (cf., its deactivation) present during pre-activation and productive catalysis.

The stereochemistry of **3a** (here formally *E*) is similar to the ones we previously determined when converting five-membered VCCs being formerly rationalized by experimental and computational analysis. This manifold involves a sequence of decarboxylation, amine activation by a basic palladacyclic intermediate and nucleophilic attack on the terminal carbon center of the allyl unit.^[15] This suggests that the allylic amination process can be extended to larger-ring type vinyl cyclic carbonates without affecting stereocontrol.

We continued to investigate the scope of this allylic amination combining a small selection of aromatic, and a much larger portion of aliphatic amines with carbonate **1a** (Scheme 2, **3a–3l**). We were pleased to find that a wide variety of amine and carbonate reagents could be used under the optimized reaction conditions producing a structurally diverse set of synthons that were previously not accessible. In the case of **3d**, we were able to support the proposed stereochemistry by X-ray analysis.^[16]

Specifically, after initially assessing that aromatic amines are productive substrates leading to appreciable yields of allylic amines **3a–3e** (up to 79%), we turned our focus to secondary aliphatic amines with different degrees of structural complexity. Overall, compounds **3f–3l** were easily obtained in yields ranging from 55–82% based on the use of either cyclic or acyclic amines. This part of the scope also included secondary amines based on bioactive scaffolds such as Amoxapine (**3k**, 63%) and Fluoxetine (**3l**, 56%).

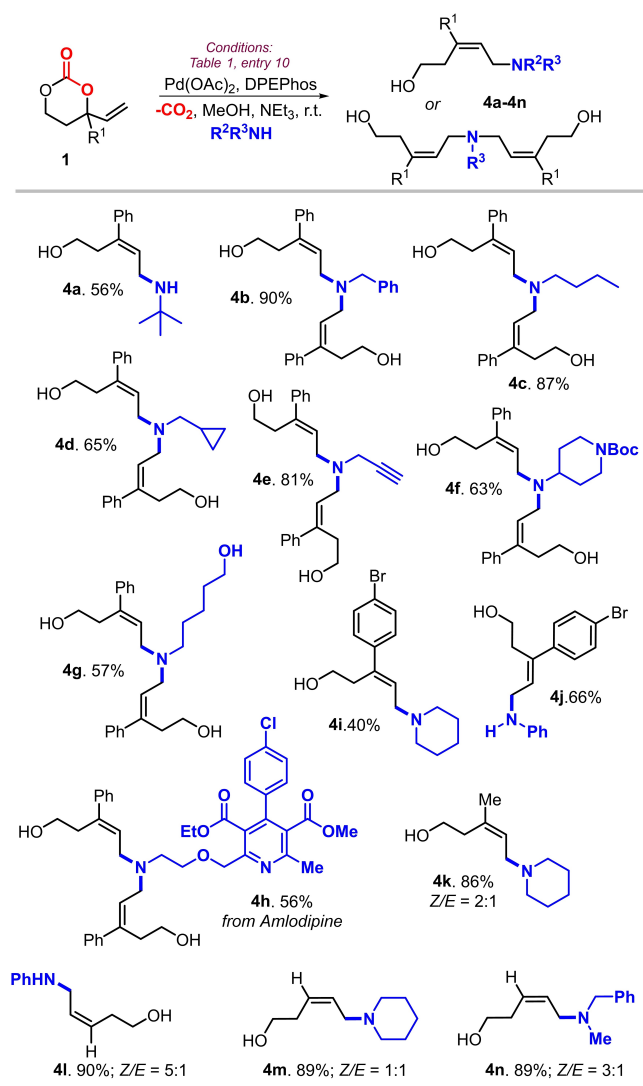
In a second embodiment of the scope, we focused (mostly) on the use of various amines and some other six-membered cyclic carbonates to widen the diversity of the protocol (Scheme 3). Six-membered cyclic carbonates of type **1** also react productively with primary aliphatic amines although in most cases double allylation takes place since the mono-allylated intermediate is more nucleophilic than the starting amine. In all these cases, a different carbonate-to-amine stoichiometry was applied (i. e., 2:1: see the SI for details) to maximize the yield of the bi-allylated amine product. Only in cases of very bulky reagents such as *tert*-butyl amine (**4a**, 56%), mono-allylation is predominant. Smooth access to diallylated products **4b–4h** was straightforward producing the targets in yields ranging from 56–90%. The procedure allows to introduce, apart from the allyl groups, orthogonal functionalities such as alkynes (**4e**), (protected) amine



Scheme 2. Synthesis of allylic amines **3a–3l** from aromatic and secondary aliphatic amines.

groups (**4f**), free alcohols (**4g**) and more complex drug fragments (Amlodipine, **4h**). Variations in the cyclic carbonate substrate were also feasible (**4i–4n**). Combination of a cyclic carbonate comprising a 4-bromo-aryl group with either cyclohexyl amine or aniline in this protocol afforded **4i** (40%) and **4j** (66%).

A cyclic carbonate having a methyl-substitution is also productive and was converted into allylated **4k** (86%) in high yield. The latter reaction proceeded with lower stereocontrol, which is frequently noted with alkyl/non-substituted cyclic carbonates (cf., R¹ = Me, H). This is connected with the lower electronically stabilizing character of alkyl versus aryl groups resulting in various propagating Pd(allyl) species.^[16] The presence of aryl groups causes favorable charge delocalization upon forming the Pd(II) pi-allyl intermediate, while alkyl groups present on the allyl ligands could lead to more dynamic speciation of Pd(allyl) species (*endo* vs *exo*, and η^1 versus η^3). We believe



Scheme 3. Scope of products based on the use of primary amines and various carbonate precursors.

that the latter could more easily lead to multiple Pd(allyl) species advancing towards the allyl-amine product with on average lower stereocontrol. When R¹ is “H” in the carbonate reagent, allylic amination occurs in high yield (**4l–4n**, 89–90%) but with lower stereocontrol and the Z/E ratios ranged from 1:1 (**4m**) to 5:1 (**4l**).

To unambiguously demonstrate the higher nucleophilic character of the intermediate mono-allylated amine product, we performed further experiments using cyclic carbonate **1a** and 1,1-diphenylmethanamine under the optimized reaction conditions but choosing varying amounts of the amine reagent (Table 2). A clear influence of the relative amount of amine reagent is noted (entries 1–5) with the yield of mono-allyl product **4o** ranging between 25 and 46%, while at the same time the diallylated product **4p** is

Table 2. Screening of various amounts of 1,1-diphenylmethanamine in the catalytic amination of **1a**.

Entry ^[a]	amine (equiv)	t (h)	Conv ^[b] 1a (%)	Yield ^[b] 4o, 4p (%)	4o:4p
1	1.5	15	>99	25, 50	1:2
2	0.8	15	>99	25, 68	1:2.7
3	3	6	>99	38, 44	1:1.15
4	3	3	>99	42, 40	1.05:1
5	3	1.5	60	46, 14	3:1

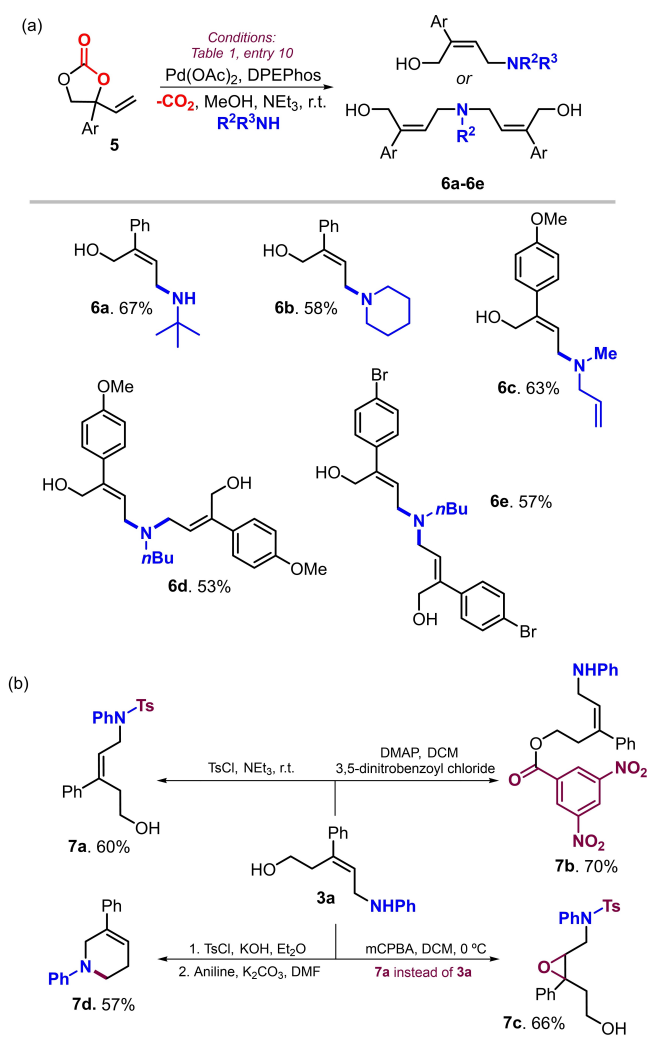
^[a] For the reaction conditions see Table 1, entry 10.

^[b] Determined by ¹H NMR (CDCl₃) using mesitylene as internal standard.

formed in 40–68% with reaction times > 3 h. Only when a short reaction time (1.5 h) is used at incomplete consumption of **1a** (60%, entry 5), a 3:1 ratio of **4o:4p** is accomplished. These results underscore the intrinsic challenge of achieving mono-allylation with more nucleophilic amines.^[18]

Similar reactivity was observed with 5-membered vinyl cyclic carbonates **5**^[19] producing mono- and bis-allylated compounds **6a–6e** in appreciable to high yields (Scheme 4a) and overall with excellent stereocontrol (Z/E > 95:5). Apart from using primary (**6a, 6d+e**) and secondary aliphatic amines (**6b+6c**), again variations in the carbonate precursor **5** further allowed to diversify the scope of the anticipated allylated amine products.

Next, we selected a typical allylic amine product (**3a**) to demonstrate that post-synthetic diversification can be easily realized. When **3a** is treated with tosyl chloride, N-tosylation takes place delivering **7a** in 60% yield. O-esterification of **3a** using 3,5-dinitrobenzoyl chloride affords **7b** in good yield (70%). Whereas direct oxidation of **3a** gave an intractable reaction mixture possibly due to undesired N-oxidation, we then probed the epoxidation of **7a** that should have a much less nucleophilic nitrogen center. This latter protocol provided smooth access to the epoxide derivative **7c**, which was isolated as a single diastereoisomer in 66% yield. By slight modification of the tosylation procedure (cf., **7a**), cyclized product **7d** was successfully obtained in 57% yield. The formation of this product can be explained by a sequence that involves O-tosylation followed by intramolecular cyclization involving the N-nucleophile.



Scheme 4. Use of five-membered precursors and post-synthetic diversification.

Conclusion

In summary, we here disclose an allylic amination manifold using VCCs and nucleophilic amines that was previously not feasible. Key to the development of the presented protocol is the efficient and relatively fast formation of Pd(0) in the presence of a bidentate, chelating ligand (here DPEPhos). As a result, efficient activation of the allylic precursor (VCC) takes place preventing parasitic ring-opening aminolysis of the substrate. Our process combines VCCs of different ring-sizes with functional primary, secondary and aromatic amines (31 examples in total, but easily extendable) creating thereby a plethora of novel and potentially useful building blocks for synthetic applications.

Experimental Section

Typical Procedure for the Allylation of Amines

A screw-capped vial was charged with the respective vinyl cyclic carbonate **1** or **5** (0.2 mmol, 1 equiv.), the amine reagent (0.3 mmol, 1.5 equiv), Pd(OAc)₂ (0.9 mg, 0.004 mmol, 2 mol%), DPEPhos (5.4 mg, 0.01 mmol, 5 mol%), Et₃N (5.6 μL , 0.04 mmol, 20 mol%) and MeOH (200 μL). The reaction mixture was stirred at room temperature for 15 h. Then, the product was purified by flash chromatography affording the corresponding product. All analytical data and copies of NMR spectra for each new product is provided in the SI.

X-ray Analysis Studies

The measured crystals of were stable under atmospheric conditions; nevertheless, they were treated under inert conditions immersed in perfluoro-polyether as protecting oil for manipulation. Data Collection: measurements were made on a Bruker-Nonius diffractometer equipped with an APPEX II 4 K CCD area detector, a FR591 rotating anode with MoK α radiation, Montel mirrors and a Kryoflex low temperature device ($T = -173^\circ\text{C}$). Full-sphere data collection was used with ω and ϕ scans. Programs used: Data collection Apex2 V2011.3 (Bruker-Nonius 2008), data reduction SAINT + Version 7.60 A (Bruker AXS 2008) and absorption correction SADABS V. 2008-1 (2008). Structure Solution: SHELXTL Version 6.10 (Sheldrick, 2000) was used. Structure Refinement: SHELXTL-97-UNIX VERSION.

Crystallographic data for the derivative of **3d**: $\text{C}_{46}\text{H}_{42}\text{N}_2\text{O}_2$, $M_r = 654.81$, triclinic, $P-1$, $a = 5.6469(13) \text{ \AA}$, $b = 16.014(4) \text{ \AA}$, $c = 18.706(4) \text{ \AA}$, $\alpha = 82.035(4)^\circ$, $\beta = 84.074(8)^\circ$, $\gamma = 89.674(7)^\circ$, $V = 1666.3(6) \text{ \AA}^3$, $Z = 2$, $\rho = 1.305 \text{ mg M}^{-3}$, $\mu = 0.079 \text{ mm}^{-1}$, $\lambda = 0.71073 \text{ \AA}$, $T = 100.18 \text{ K}$, $F(000) = 696$, crystal size = $0.40 \times 0.03 \times 0.03 \text{ mm}$, $\theta(\text{min}) = 2.21^\circ$, $\theta(\text{max}) = 58.644^\circ$, 8943 reflections collected, 8943 reflections unique ($R_{\text{sigma}} = 0.0970$), $\text{GoF} = 1.038$, $R_1 = 0.0778$ and $wR_2 = 0.2136$ [$I > 2\sigma(I)$], $R_1 = 0.1095$ and $wR_2 = 0.2426$ (all indices), min/max residual density = $-0.40/0.46$ [$\text{e} \cdot \text{ \AA}^{-3}$].

CCDC-2354470 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/structures.

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