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Stereoselective *in situ* assembled hydrofunctionalization and cascade

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ABSTRACT

Hydrofunctionalization of unsaturated hydrocarbons *via* transition metal catalysis is a powerful route to prepare allyl skeletons, but is limited to mono- and two-component transformation models. Here we describe a novel protocol for the unprecedented three-component hydrofunctionalization *via in situ* formed diene species. Both amines and stabilized carbon nucleophiles undergo the assembled hydrofunctionalization with various olefins and alkenyl bromides through Pd-catalyzed tandem Heck coupling and outer-sphere allylation, generating allylic C–N and C–C bonds in reasonable yields and with excellent regioselectivities. In particular, the combination of assembled hydrofunctionalization and following derivatizations enables new access to a series of valuable substituted cyclic skeletons. Preliminary mechanistic studies support the *in situ* formation of critical conjugated diene intermediate for sequential hydrofunctionalization

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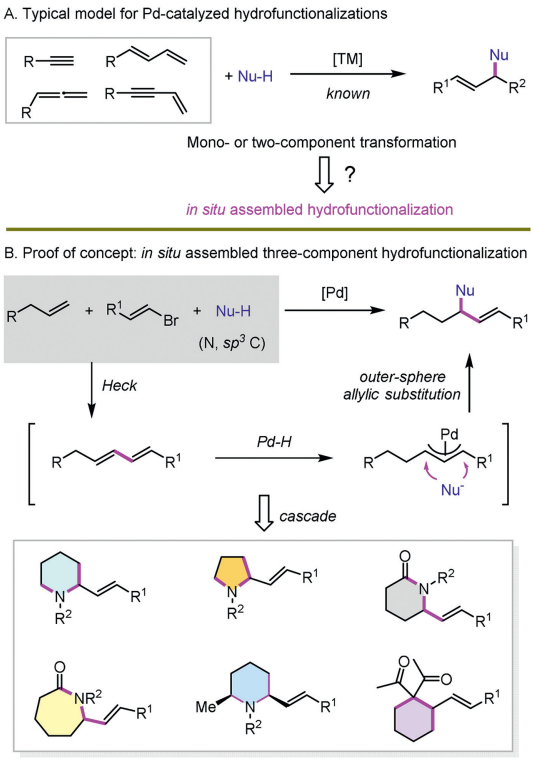
Transition metal-catalyzed hydrofunctionalization of unsaturated hydrocarbons *via* η^3-allyl metal species has emerged as a powerful strategy to construct allyl skeletons [1–12]. A series of typical nucleophiles have been demonstrated suitable for the preparation of allylic C–C, C–N, C–O, C–S and C–P bonds. In contrast to typical Tsuji-Trost reaction, this route usually features the ready availability of both substrates, 100% atom economy and mild reaction conditions. Unsaturated hydrocarbons, including alkynes, dienes, allenes and enynes are most commonly used to react with nucleophiles *via* an intramolecular or intermolecular route (Scheme 1A) [13–76]. However, different from the well explored mono- and two-component hydrofunctionalization models, a three-component hydrofunctionalization *via in situ* assembling pathway from simple alkenes is unknown.

Due to the inability to form critical η^3-allyl metal species, alkenes as feedstock compounds could not be adopted directly for hydrofunctionalization. It can be imagined that if the alkene could be *in situ* transformed into a typical unsaturated substrate, such as conjugated diene, then hydrofunctionalization might occur readily. A few elegant studies explored related pathways *via* Pd- [77–79] or Ni-catalyzed [80] three-component cross couplings of alkenes with cyclic vinyl triflates and aryl boronic acids or B_2pin_2, all of which, however, focused on inner-sphere reductive elimination coupling route using boron reagents. A different process involving general nucleophiles through outer-sphere allylic substitution pathway is unexplored.

Following our long-standing interest in hydrofunctionalization areas [32–34,39,52,58,63,70–74], we envisioned that the combination of *in situ* formation of conjugated diene from the reaction of simple alkene and alkenyl bromide *via* Pd-catalyzed Heck reaction and following hydrofunctionalization with common nucleophiles might provide a potential route for the unprecedented *in situ* assembled three-component hydrofunctionalization (Scheme 1B). However, this design is not straightforward. The perfect merger of Heck reaction and hydrofunctionalization is challenging, due to competing direct coupling between alkenyl electrophiles and nucleophiles, and the balanced basicity for transmetallation, β-H elimination and allylic substitution. The stereocontrol is another potential issue, especially considering Heck reaction usually requires a high reaction temperature [81–82]. Herein, we report the first stereoselective *in situ* assembled three-component hydrofunctionalization *via* Pd catalysis. A series of simple olefins react with alkenyl bromides and amines or carbon nucleophiles smoothly to provide diverse allyl skeletons in moderate to good yields and with high regioselectivities. In addition, the combination of assembled hydrofunctionalization and cascade processes enables convenient access to various intriguing substituted cyclic scaffolds, including

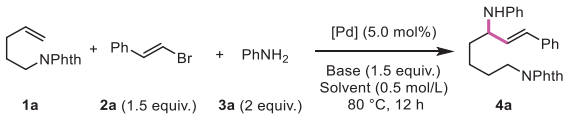
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Scheme 1. Pd-catalyzed hydrofunctionalizations of unsaturated bonds.

Table 1
Reaction development for assembled hydrofunctionalization.[a]



1a **2a** (1.5 equiv.) **3a** (2 equiv.) **4a**

Entry	[Pd]	Solvent	Base	Yield (%)
1	$Pd(OAc)_2$	THF	$KHCO_3$	5
2	$Pd(OAc)_2$	DCM	$KHCO_3$	Trace
3	$Pd(OAc)_2$	CH_3CN	$KHCO_3$	Trace
4	$Pd(OAc)_2$	DMF	$KHCO_3$	Trace
5	$Pd(OAc)_2$	PhEt	$KHCO_3$	3
6	$Pd(OAc)_2$	DMSO	$KHCO_3$	46
7	$Pd(PPh_3)_4$	DMSO	$KHCO_3$	29
8	$Pd[P(^tBu)_3]_2$	DMSO	$KHCO_3$	32
9	$Pd(PPh_3)_2Cl_2$	DMSO	$KHCO_3$	40
10	$Pd_2(dba)_3$	DMSO	$KHCO_3$	53
11[b]	$Pd_2(dba)_3$	DMSO	$KHCO_3$	<48
12	$Pd_2(dba)_3$	DMSO	K_2CO_3	48
13	$Pd_2(dba)_3$	DMSO	Na_2CO_3	40
14	$Pd_2(dba)_3$	DMSO	K_3PO_4	44
15	$Pd_2(dba)_3$	DMSO	Et_3N	10
16[c]	$Pd_2(dba)_3$	DMSO	Mixture	63
17[c,d]	$Pd_2(dba)_3$	DMSO	Mixture	<54
18[c,e]	$Pd_2(dba)_3$	DMSO	Mixture	71

[a] The reaction was carried out in 0.10 mmol scale. The yield was determined by ^1H NMR.
[b] A series of ligands were used and see Supporting information for details.
[c] Both K_2CO_3 (1.5 equiv.) and $KHCO_3$ (1.0 equiv.) were used.
[d] At 60 or 100 °C.
[e] The reaction time was 24 h and isolated yield was shown.

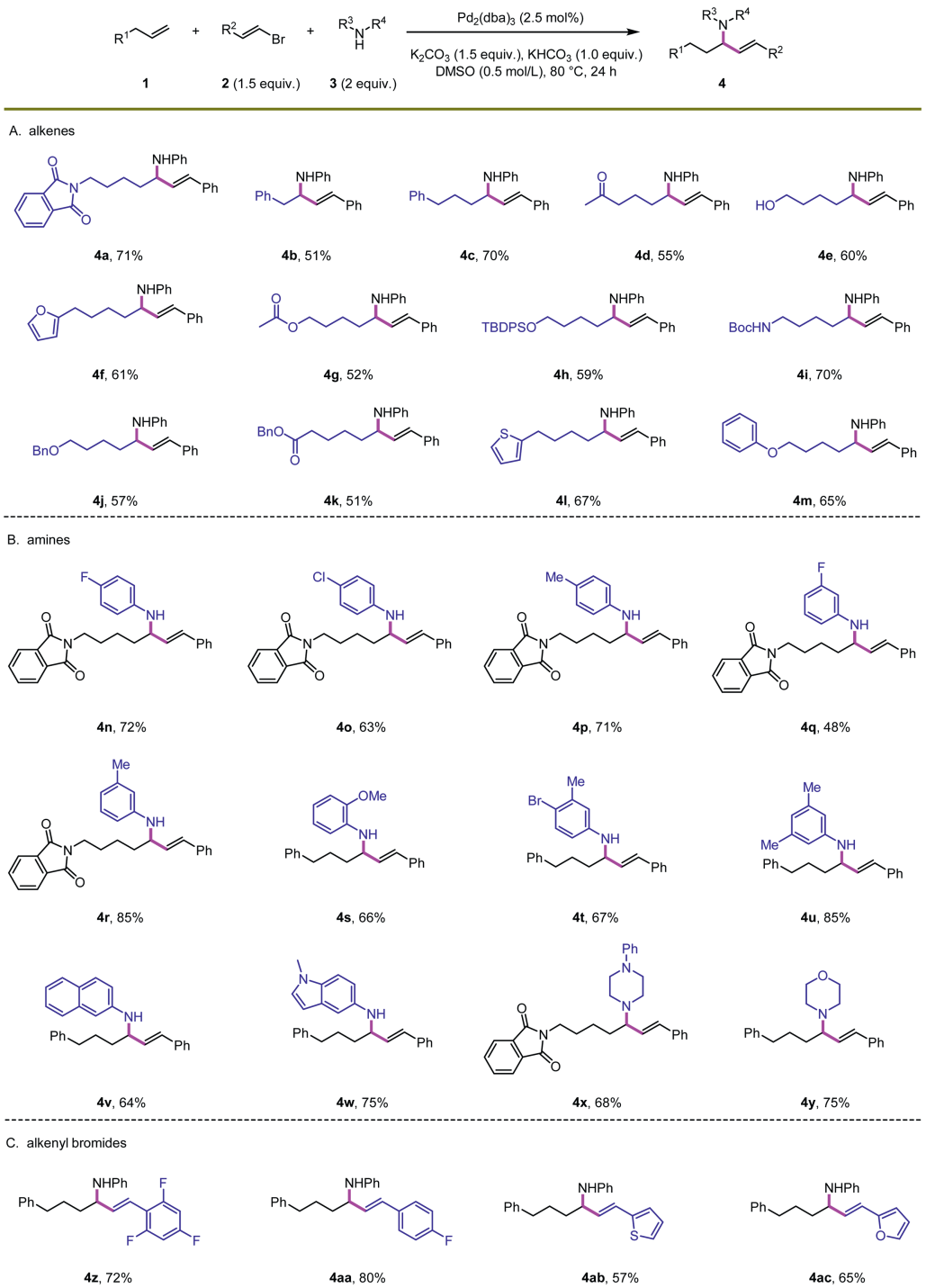
piperidines, pyrrolidines, piperidones, azepan-2-ones, and cyclohexanes.

We initiated studies of the assembled hydrofunctionalization by conducting reactions with olefin **1a**, alkenyl bromide **2a**, and aniline **3a** as the substrates, inorganic $KHCO_3$ as the base, and $Pd(OAc)_2$ as the catalyst at 80 °C for 12 h (Table 1). A set of solvents were evaluated first. However, low reaction yield of **4a** was detected along with most substrates recovered when using both nonpolar and polar solvents (entries 1-5 and see Supporting information for details). Only with DMSO as the solvent, product **4a** was observed in 46 % yield (entry 6). The evaluation of a couple of palladium sources indicated that $Pd_2(dba)_3$ as the catalyst was slightly favorable to the reaction (entries 7-10). In this context, we expected that different ligands might help regulate the reactivity and stereoselectivity. Various ligands were thus examined but all led to the erosion of the yield of **4a** (entry 11 and see Supporting information for details), which suggested that DBA (dibenzylideneacetone) was the optimal ligand for Pd metal. Next, weak inorganic bases generally showed comparable reactivity, but strong inorganic bases and organic bases were unfavorable to the transformation (entries 12-15 and see Supporting information for details). Nevertheless, with the adoption of K_2CO_3 and $KHCO_3$ as mixed bases, **4a** was provided in an increased yield (entry 16). Finally, both higher and lower temperatures led to the decrease of reactivity (entry 17), but extended reaction time to 24 h was effective and furnished the optimal reaction yield of **4a** in 71 % (entry 18). It should be noted that no regioisomers were detected for these trials.

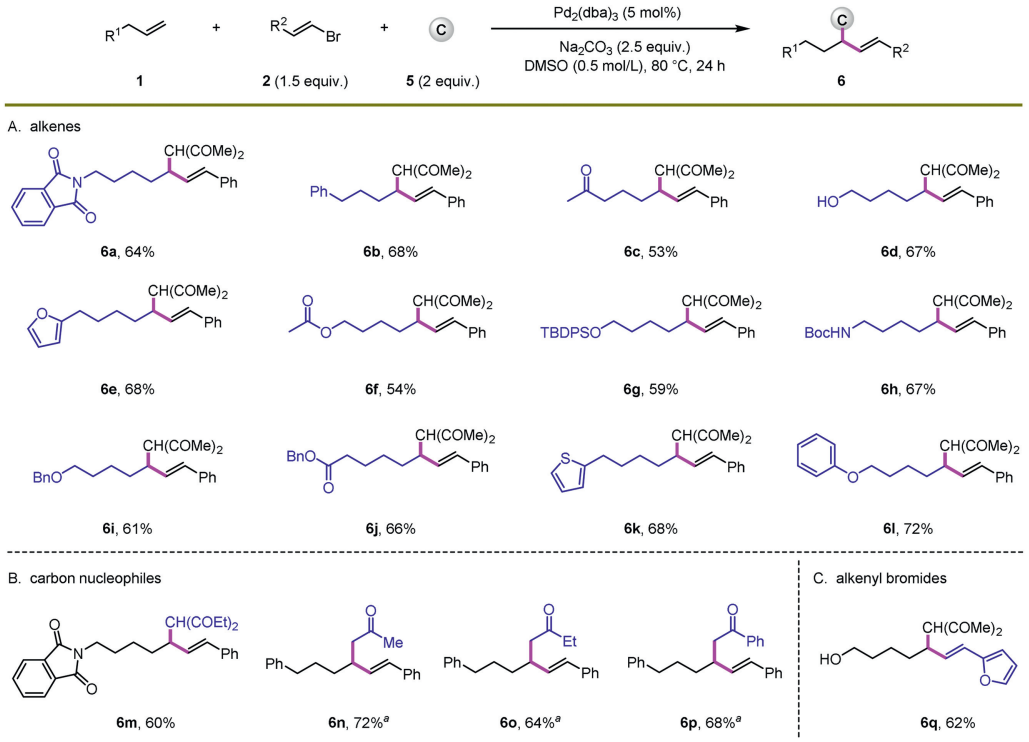
Having identified conditions for three-component hydrofunctionalization, the scope of the transformation was examined, and the results are summarized in Scheme 2. First, a series of terminal olefins bearing diverse functional groups showed high compatibility with the reaction. For example, alkenes containing amide, aryl, ketone, hydroxyl, heteroaryl, ester, OTBDPS, NHBoc, and ether units in the alkenes were well tolerated and afforded corresponding products **4a**–**4m** in 51 %–71 % yield and with excellent regioselectivity (Scheme 2A). In particular, the unaffected functional groups such as ketone, hydroxyl and ester units in the alkenes provided further modification space with the formed nucleophilic secondary amines (**4d**, **4e**, **4k**).

Arylamines featuring different electronic and steric characters were also suitable for the *in situ* assembled hydrofunctionalization (Scheme 2B). Substituents including F, Cl, alkyl, and ether at varying positions in the arylamines exhibited good tolerance in the reaction with alkene **1a** and alkenyl bromide **2a**, generating allylamines in moderate to good yields (**4n**–**4s**). Arylamines containing multiple substituents and large ring system and heteroaryl amines were all good coupling partners in the three-component reaction (**4t**–**4w**). In addition to arylamines, alkyl amines were evaluated as well and furnished similarly good reactivity and stereocontrol during the reaction (**4x**–**4y**). Finally, a couple of diverse aryl vinyl bromides were tested and gave corresponding products **4z**–**4ac** in 57 %–80 % yield (Scheme 2C). It should be noted that only one regioisomer was detected for each case. It should be noted that alkyl-substituted alkenyl bromide was not suitable for the protocol at present (see Supporting information for failed trials).

To demonstrate the generality of the assembled hydrofunctionalization concept, hydroalkylation through this *in situ* assembled process was established simultaneously (Scheme 3). With the use of Na_2CO_3 as the base instead of K_2CO_3 and $KHCO_3$ in the conditions for hydroamination, assembled hydroalkylation reaction proceeded smoothly. Alkenes containing various functional groups, such as amide, aryl, ketone, hydroxyl, furyl, ester, OTBDPS, NHBoc, ether, and thienyl units, reacted with alkenyl bromide **2a** and acetylacetone **5a** well, furnishing assembled allyl skeletons **6a**–**6l** in reasonable yields and with excellent regioselectivities (Scheme 3A). Other carbon nucleophiles were also suitable for the transformation (Scheme 3B, **6m**–**6p**). In particular, with the combination of assembled hydrofunctionalization and decarboxylation, a set of β-alkenyl ketones were prepared readily *via* a one-pot reaction. In addition, a heteroaryl vinyl bromide underwent the trans-



Scheme 2. Scope of assembled hydroamination. The reaction was conducted in 0.1 mmol scale. Isolated yield.



Scheme 3. Scope of assembled hydroalkylation. Isolated yield. Prepared through assembled hydroalkylation and decarboxylation. See Supporting information for details.

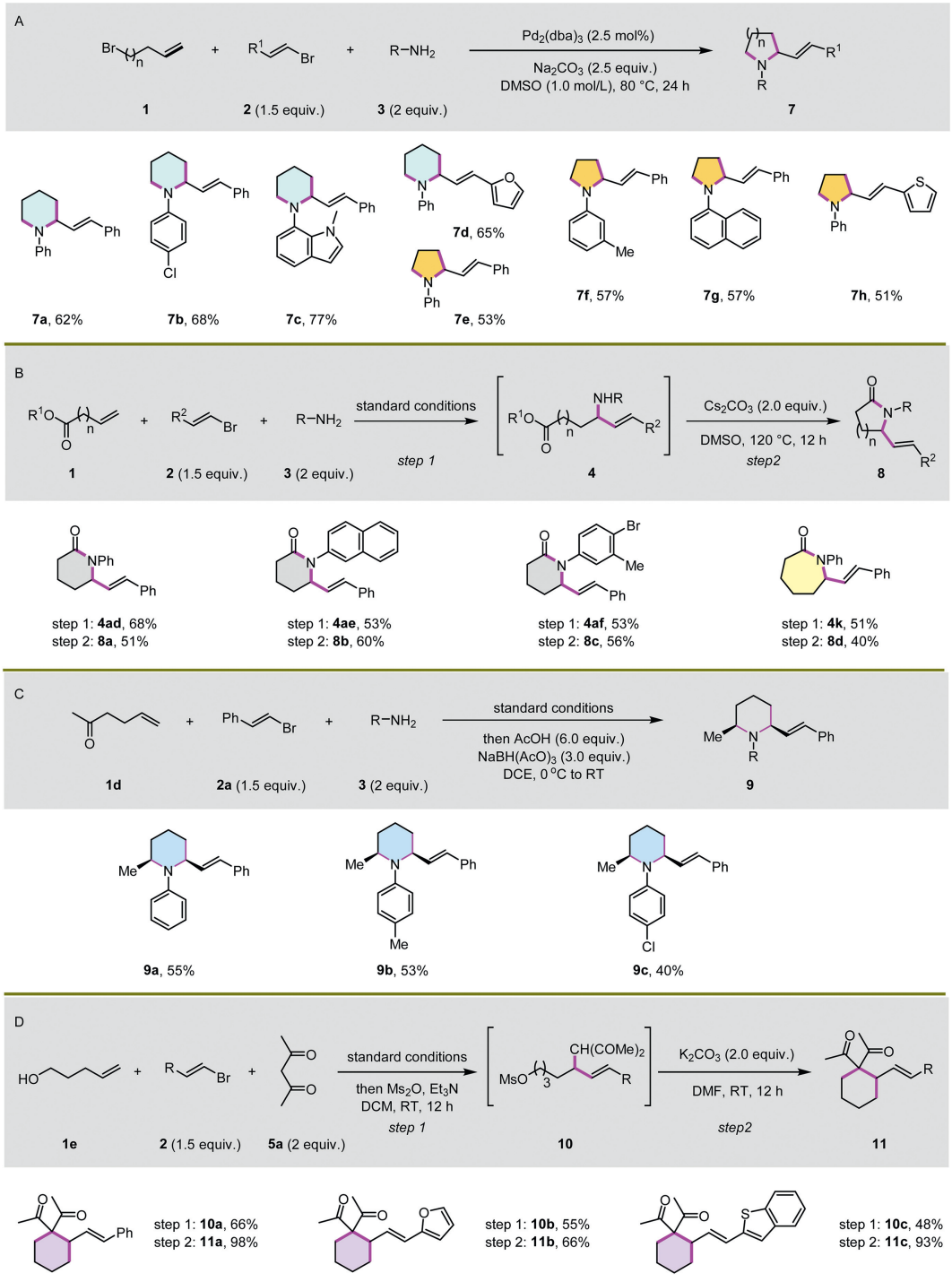
formation smoothly, furnishing **6q** in 62 % yield as a single regioisomer (Scheme 3C).

Considering the broad functional group tolerance, we hypothesized that a combination of assembled hydrofunctionalization and various precursor formation or sequential downstream transformations might construct valuable scaffolds conveniently from readily available substrates (Scheme 4). First, with the use of alkene containing a terminal Br atom, alkenyl bromide and amine as substrates, a series of substituted saturated *N*-heterocycles were achieved facilely (Scheme 4A). For example, piperidines and pyrrolidines bearing different substituents were obtained in good yields through this one-pot process (**7a–7h**). Similarly, by using ester-derived alkenes, piperidones and azepan-2-ones were readily achieved *via* sequential assembled hydroamination and amide formation (Scheme 4B, **8a–8d**). Furthermore, multiple substituted piperidines **9a–9c** could be easily synthesized from the reaction of alkene **1d**, phenyl vinyl bromide **2a** and arylamine **3** through assembled hydroamination and reductive amination cascade pathway (Scheme 4C). Finally, substituted cyclohexanes **11a–11c** were prepared from pent–4-en-1-ol **1e** *via* assembled hydroalkylation and nucleophilic substitution (Scheme 4D). These designed cascade reactions provide new routes to build various cyclic scaffolds, highlighting the value and power of the assembled hydrofunctionalization concept.

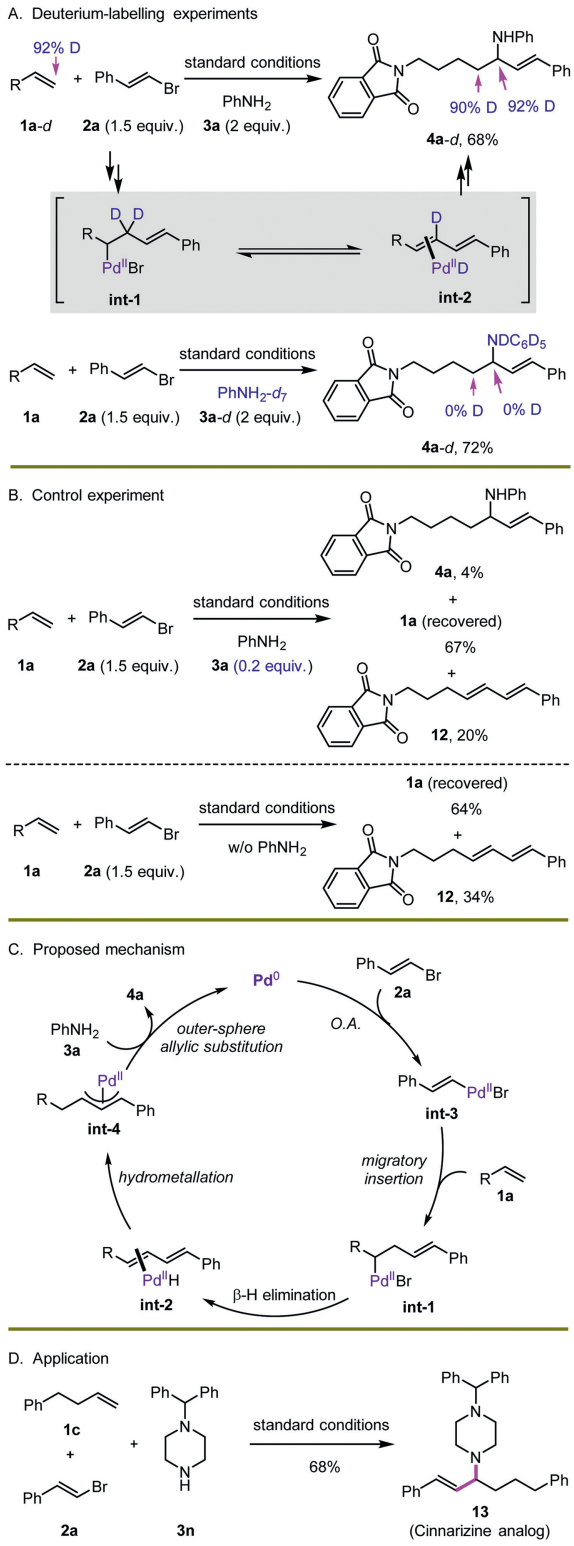
To elucidate the mechanistic pathway of the designed assembled hydrofunctionalization, a couple of experiments were carried out (Scheme 5). When deuterated alkene **1a**-*d* was adopted under standard conditions, product **4a**-*d* was observed in 68 % yield (Scheme 5A, top). In this case, both allylic and homoallylic positions were observed in high deuteration. This result indicated that a Heck coupling occurred first to generate a diene intermediate **int-2** and Pd-D species. The reinsertion of Pd-D with diene species introduced deuterium atom to the homoallylic position of product **4a**-*d*. When deuterated phenylamine was used as nucleophile, no deuteration was observed in the skeleton of prepared product **4a**-*d* (Scheme 5A, bottom). This fact suggested that the formed Pd-D species *via* β-H elimination was tightly associated with formed diene intermediate. To further support the potential diene formation step, a control experiment with the use of only 0.2 equiv. of **3a** as limited nucleophile was conducted (Scheme 5B, top). In this case, product **4a** was observed in 4 % yield, along with substrate **1a** recovered in 67 % yield and diene intermediate **12** in 20 % yield. Without the use of amine nucleophile **3a**, diene **12** was still detected in 34 % yield along with 64 % yield of **1a**. All these results demonstrated that a Heck coupling was involved in the design.

Based on these experiments, a plausible mechanistic pathway was described in Scheme 5C. The low-valent Pd was oxidized by alkenyl bromide first to form alkenyl-Pd species **int-3**, which reacted with olefin **1a** to provide **int-1** *via* migratory insertion. The following β-H elimination from **int-1** gave diene intermediate **int-2**, and then generated electrophilic allyl-Pd species **int-4**. Finally, an outer-sphere allylic substitution from arylamine **3a** provided the product **4a** and regenerated Pd catalyst. In addition, the present protocol could be easily applied to the concise synthesis of Cinnarizine analog **13** from the reaction of simple substrates **1c**, **2a**, and **3n**, highlighting the practical value of the methodology (Scheme 5D) [83].

In conclusion, a three-component model for *in situ* assembled hydrofunctionalizations is established *via* palladium catalysis, different from conventional mono- and two-component reaction model. Both assembled hydroamination and hydroalkylation are developed through this tandem Heck coupling and hydrofunctionalization pathway. Various allyl skeletons are prepared in moderate to good yields and with high regioselectivities from the reaction of readily available olefins, alkenyl bromides and common nucleophiles. The combination of assembled hydroamination and different downstream transformations provide convenient routes to access a series of valuable substituted cyclic scaffolds, including piperidines, pyrrolidines, piperidones, azepan-2-ones, and cy-



Scheme 4. Assembled hydrofunctionalization cascade. Isolated yield. See Supporting information for detailed reaction conditions.



Scheme 5. Preliminary mechanistic studies and application.

clohexanes. Preliminary studies support the diene-involved mechanistic pathway.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

CRediT authorship contribution statement

Li-Hong Xiong: Investigation. **Liang Chen:** Investigation. **Zhi-Tao He:** Writing – review & editing, Writing – original draft, Supervision, Project administration, Funding acquisition, Conceptualization.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2025.111921.

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