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Photoinduced and palladium-catalyzed hydrogen atom transfer triggered 1,2-difunctionalization of 1,3-dienes with hydroxamides

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The discovery of novel catalysis modes to generate a significant increase in structural complexity from readily available reactants is a fundamental goal in modern organic synthesis. Here, we report a photoinduced palladium-catalyzed hydrogen atom transfer triggered 1,2-difunctionalization of conjugated dienes. Without the employment of exogeneous photosensitizers and external oxidants, the cascade reaction realized the integration of remote functionalization of various C(sp^3)−H bonds and selective difunctionalization of 1,3-dienes with 100% atom efficiency, allowing for the synthesis of structurally diverse amides with up to 90% yields. Given the prevalence of amides in pharmaceuticals and natural products, the current protocol has provided an efficient means to access highly functionalized amides from readily available carboxylic acid derivatives and 1,3-dienes.

photoinduced palladium catalysis, C–H activation, hydrogen atom transfer, 1, 3-dienes, allylation

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The development of new sustainable and atom-economical synthetic strategies employing readily available feedstock substrates is a fundamental goal of modern synthetic chemistry [1,2]. In this context, selective difunctionalization of 1,3-dienes, a class of readily available versatile building blocks, enables rapid access to complex molecules and has therefore attracted increasing interest [3–9]. In particular, palladium catalysis has been extensively studied in this field and successfully applied in many selective functionalization reactions of 1,3-dienes [3,7,10–12]. As briefly summarized in Figure 1a, most of the examples involve a Pd(0)/Pd(II) catalytic cycle that incorporates oxidative addition, alkene insertion and nucleophilic attack on the π-allyl palladium intermediate [3,7,13–16]. Recently, breakthroughs in photochemistry have revealed that alkyl halides and analogs could generate open-shell species under photoinduced palladium catalysis [17–21], enabling processes such as remote desaturation [22,23], alkyl Heck reaction [24–27], and others [28–30]. More recently, Glorius's group [31,32] and Gevorgyan's group [33] successfully established radical carbofunctionalization of 1,3-dienes by means of photoinduced palladium catalysis (Figure 1b). Employing *N*-hydroxyphthalimide esters as the precursor for alkyl radical and nitrogen-centered nucleophiles, Glorius's group [32] developed 1,4-aminoalkylation of dienes. With alkyl bromides and alkyl iodides as the radical precursors, Glorius's group [31] and Gevorgyan's group [33] independently developed 1,4- and 1,2-carbofunctionalization reactions of dienes. Despite these promising advances, the potential of difunctionalization of 1,3-dienes *via* excited-state palladium catalysis, whereas the palladium catalyst has dual roles by harvesting light and catalyzing the chemical transformation, has been far from well developed. Given the unique advantages of photoinduced palladium catalysis, the search for novel reaction modes in 1,3-diene functionalization is highly desired. Herein, we report a light-induced palladium-catalyzed process that combines remote C−H functionalization enabled by

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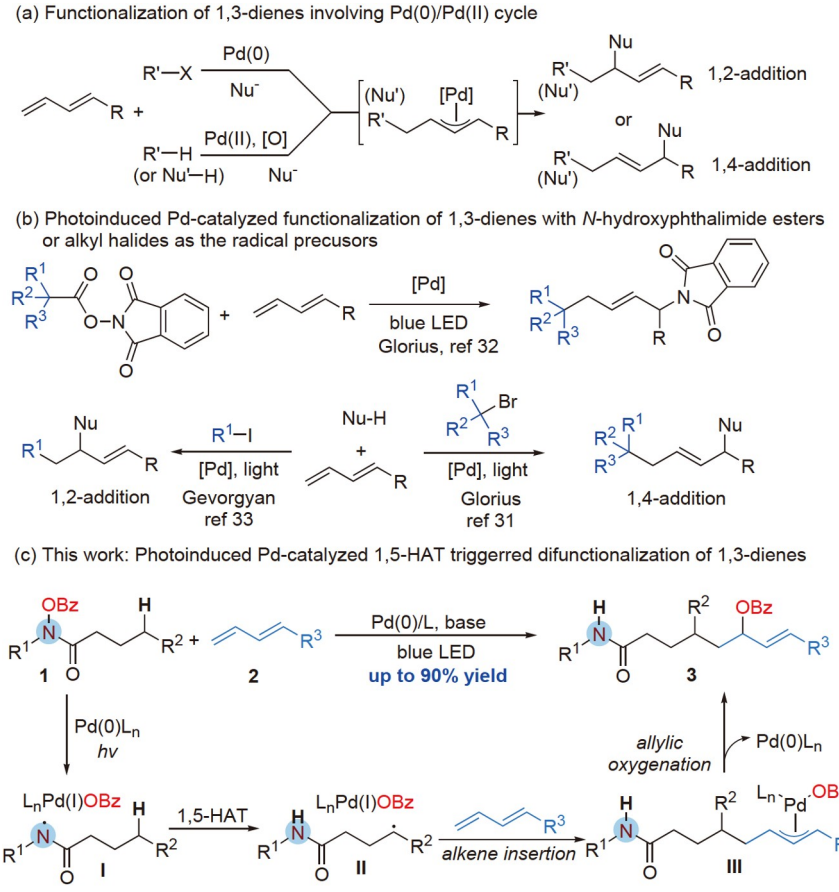


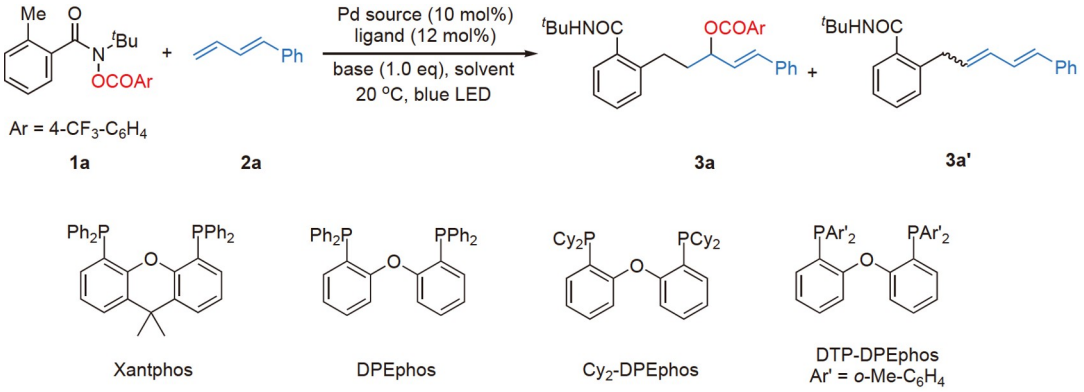
Figure 1 Palladium-catalyzed difunctionalization of 1,3-dienes (color online).

intramolecular hydrogen atom transfer (HAT) [34–37] and selective 1,2-carbonoxygenation of dienes (Figure 1c). The transformation is proposed to consist of the generation of an amidyl radical **I** [38,39] together with Pd(I) species from amide derivative **1** under visible light irradiation, 1,5-HAT to form translocated alkyl radical **II** and addition of the radical to diene **2** to give π-allyl palladium intermediate **III**, which undergoes allylic oxygenation [40–42] to give functionalized amide derivative **3**. Considering the prevalence of amides in natural products and pharmaceuticals, it is anticipated that the present method may simplify the synthesis and structural elaboration of amide-containing targets with readily available dienes.

Due to their excellent structural tunability and easy accessibility, hydroxylamine derivatives have recently been exploited as nitrogen radical precursors in visible light photochemistry [43,44]. Specifically, carboxylic acid-derived *O*-acyl hydroxylamides have been utilized for the reductive generation of amidyl radicals aided by photocatalysts such as Eosin Y and Ir-based photocatalysts [45–47]. Very recently, Yu and co-workers [23] reported a photoinduced and palladium-catalyzed remote desaturation reaction of hydroxylamides, exhibiting the capability of excited Pd(0) catalysts for the reductive generation of amidyl radicals from hydroxylamide derivatives. Despite the desaturation side reaction, we anticipated that under proper conditions with an adequate palladium catalyst, the translocated alkyl radical **II** would undergo addition to the diene substrate to give a π-allyl palladium intermediate and deliver the final product **3** (Figure 1c).

At the outset of the investigation, to verify our hypothesis and simplify the reaction sequence by avoiding the β-H elimination process, *O*-acyl hydroxamide **1a** (Ar = *p*-$CF_3C_6H_4$) was chosen as the model substrate for the reaction with diene **2a** (Table 1). Gratifyingly, amide **3a** bearing an allyl alcohol ester moiety could indeed be obtained in 63% yield with excellent 1,2-selectivity and *E*/*Z* selectivity under 5 W blue LED irradiation in the presence of $Pd(PPh_3)_4$ (10 mol%) as the catalyst and K_2CO_3 as the base in 1,4-dioxane at 20 °C (entry 1). A small amount of diene **3a′** was also observed as the side product (entry 1). The combination of $Pd(PPh_3)_4$ as the Pd source and additional ligands were subsequently investigated. The use of dppf and BINAP rendered the cascade reaction completely unproductive (entries 2 and 3). While Xantphos provided product **3a** in only 52% yield, DPEphos-type ligands all exhibited superior performance for the reaction (entries 5−7). The use of DTP-DPEphos bearing *ortho*-Me substituted phenyls proved to be optimal ligand for

Table 1 Optimization of catalysts and reaction conditions [a)]



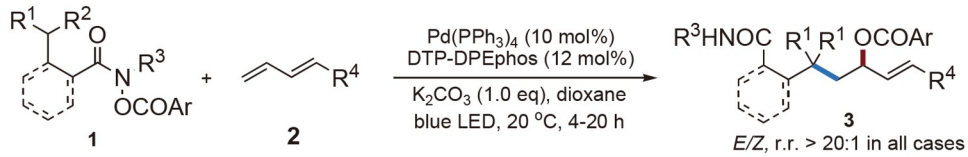
Entry	Catalyst/ligand	Base	Solvent	Yield (%)[b)]	**3a**/**3a′**
1	$Pd(PPh_3)_4$/–	K_2CO_3	dioxane	63	10/1
2	$Pd(PPh_3)_4$/dppf	K_2CO_3	dioxane	ND	–
3	$Pd(PPh_3)_4$/*rac*-BINAP	K_2CO_3	dioxane	ND	–
4	$Pd(PPh_3)_4$/Xantphos	K_2CO_3	dioxane	52	10/1
5	$Pd(PPh_3)_4$/DPEphos	K_2CO_3	dioxane	76	10/1
6	$Pd(PPh_3)_4$/Cy_2-DPEphos	K_2CO_3	dioxane	66	>20/1
7	$Pd(PPh_3)_4$/DTP-DPEphos	K_2CO_3	dioxane	80	>20/1
8	$Pd(PPh_3)_4$/DTP-DPEphos	Na_2CO_3	dioxane	43	17/1
9	$Pd(PPh_3)_4$/DTP-DPEphos	Cs_2CO_3	dioxane	50	10/1
10	$Pd(PPh_3)_4$/DTP-DPEphos	K_3PO_4	dioxane	66	12/1
11	$Pd(PPh_3)_4$/DTP-DPEphos	NEt_3	dioxane	64	>20/1
12	$Pd(PPh_3)_4$/DTP-DPEphos	–	dioxane	65	20/1
13	$Pd(PPh_3)_4$/DTP-DPEphos	K_2CO_3	THF	64	10/1
14	$Pd(PPh_3)_4$/DTP-DPEphos	K_2CO_3	DCM, MeCN or DMF	trace	–
15[c)]	–/–	K_2CO_3	dioxane	ND	–
16[d)]	$Pd(PPh_3)_4$/DTP-DPEphos	K_2CO_3	dioxane	ND	–

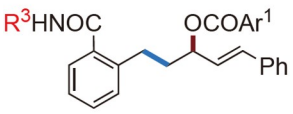
a) Unless indicated otherwise, the reaction was carried out in the scale of **1a** (0.2 mmol), **2a** (0.1 mmol), palladium precatalyst (10 mol %), ligand (12 mol %), base (0.1 mmol) in 2.0 mL of solvent at 20 °C under the irradiation of a 5 W blue LED for 20 h. b) Isolated yield of **3a**. c) The reaction was carried out without the palladium catalyst. d) The reactions were carried out in the dark at 50 or 100 °C.

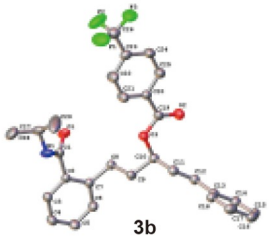
the reaction, providing **3a** in 80% yield (entry 7). Replacement of K_2CO_3 with other bases, such as Na_2CO_3, Cs_2CO_3, K_3PO_4, NEt_3 could not give improved results (entries 8−11). Since the overall transformation features a 100% atom efficiency, without any acid or salt waste byproduct, the reaction could also be performed without a base, albeit with slightly lower yield (entry 12). Investigation of the solvent effect revealed that the reaction favors ether-type solvents (entry 13), with 1,4-dioxane still proved to be the optimal reaction media. Other common solvents, such as dichloromethane (DCM), MeCN and *N*,*N*-dimethylformamide (DMF), all resulted in unproductive reactions (entry 14). Control experiments showed that the palladium catalyst and light are both essential for the cascade reaction (entries 15 and 16). No product could be observed even at elevated reaction temperature (50 or 100 °C) without blue light irradiation (entry 16).

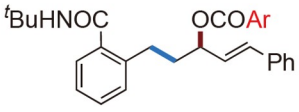
Having established the optimized reaction conditions, we first investigated the scope of *O*-acyl hydroxyamides **1** for the reaction with diene **2a** (Table 2). 2-Methylbenzoic acid-derived hydroxyamides with different alkyl groups on the nitrogen atom, *e.g.*, isopropyl and cyclohexyl, could also undergo the cascade reaction, albeit with lower yields compared with model substrate bearing a *tert*-butyl group (**3a**−**3c**). The structure of **3b** was confirmed by single-crystal X-ray chromatography analysis (See Supporting Information online for details). Replacement of the 4-(trifluoromethyl) phenyl group in the benzoate moiety of **1a** to phenyl or other aryl groups could also lead to smooth reactions, providing functionalized amide **3d**−**3g** in 64%−80% yield. Substrates **1** bearing an electron-donating group at the benzene core with different substitution patterns were well tolerated, as exemplified by the synthesis of **3h**−**3k** in 63%−85% yields. Meanwhile, substrates bearing electron-withdrawing groups, *e.g.*, 4-F, and 4-Cl, are less favored (**3l**, **3m** *vs*. **3i**). At this

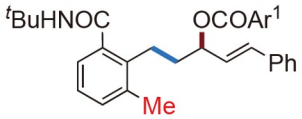
Table 2 Substrate scope for amide derivatives and 1,3-dienes [a)]

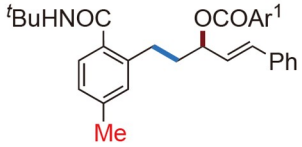


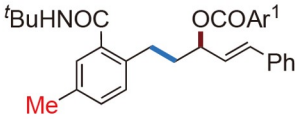

3a, R^3 = ^{t}Bu, 80%
3b, R^3 = ^{i}Pr, 62%
3c, R^3 = Cy, 54%
Ar^1 = (4-CF_3-C_6H_4)

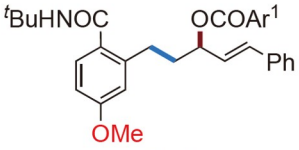

3b

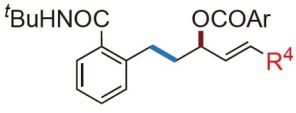

3d, Ar^2 = Ph , 80%
3e, Ar^3 = 4-Me-C_6H_4 , 64%
3f, Ar^4 = 4-OMe-C_6H_4, 76%
3g, Ar^5 = 4-Cl-C_6H_4, 70%

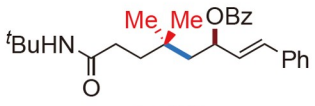

3h, 85%

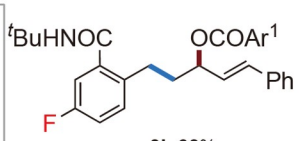

3i, 72%

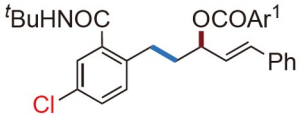

3j, 85%

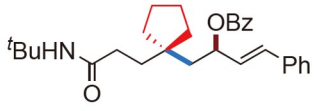

3k, 63%

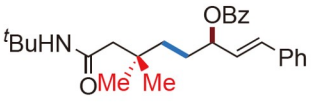

3n, R^4 = 4-Me-C_6H_4, 57%
3o, R^4 = 4-OMe-C_6H_4, 82%
3p, R^4 = 4-F-C_6H_4, 84%
3q, R^4 = 4-Cl-C_6H_4, 74%
3r, R^4= 2-Me-C_6H_4, 85%
3s, R^4= 2-OMe-C_6H_4, 56%
3t, R^4 = 2-Cl-C_6H_4, 77%
3u, R^4 = 3-Me-C_6H_4, 80%
3v, R^4 = 2-Naphthyl, 85%
3w, R_4 = CO_2Me, 23%
(for **3w**, Ar = Ar^4, for others, Ar = Ar^1)

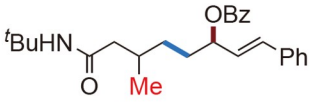

3x, 90%

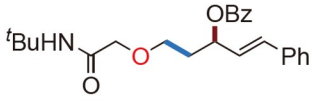

3l, 60%

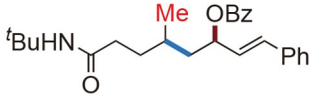

3m, 37%

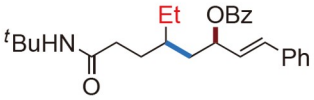

3y, 82%

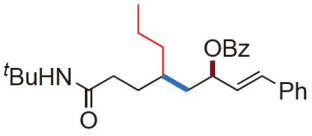

3z, 40%

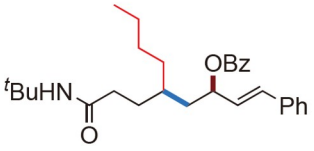

3aa, 37%, d.r. = 1:1,

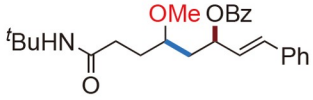

3ab, 88%

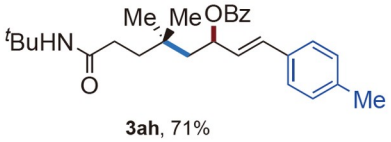

3ac, 87%, d.r. = 1:1

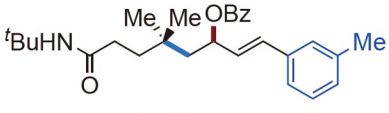

3ad, 78%, d.r. = 1:1

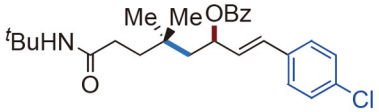

3ae, 65%, d.r. = 1:1

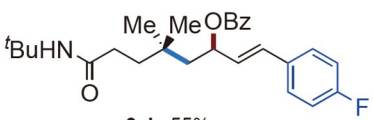

3af, 87%, d.r. = 1:1

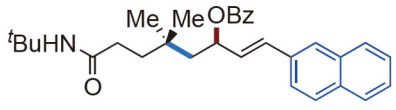

3ag, 63%, d.r. = 1:1


3ah, 71%


3ai, 78%


3aj, 55%


3ak, 55%


3al, 41%

a) Unless indicated otherwise, the reaction was carried out in the scale of **1a** (0.2 mmol), **2a** (0.1 mmol), palladium precatalyst (10 mol%), ligand (12 mol %), base (0.1 mmol) in 2.0 mL of solvent at 20 °C under the irradiation of blue LED (3, 5 or 7 W) for 4−20 h (See Supporting Information online for details). Yields based on isolated products.

stage, a variety of dienes were subjected to the reactions with *O*-acyl hydroxyamides **1a**. The reactions of phenyl substituted 1,3-dienes bearing electron-donating or electron-withdrawing groups proceeded smoothly to afford the corresponding allylic esters in 57%−85% yield with excellent regio- and *E*/*Z* selectivity, regardless of the substitution pattern of the benzene ring (**3n**−**3v**). 2,4-Pentadienoic acid methyl ester could also undergo the current cascade reaction to afford the desired product (**3w**). The low yield is due to the generation of diene side-product, probably from the β-elimination process of the π-allyl palladium complex or the allyl radical intermediate.

Next, *O*-acyl hydroxyamides derived from alkyl carboxylic acids with unactivated C−H bonds were tested for the reactions with diene **2a** under the standard reaction conditions. Delightedly, δ-C(sp^3)−H (relative to the nitrogen atom) functionalized amide product **3x** could be obtained in 90% yield with excellent regio- and *E*/*Z* selectivity when 4-methylpentanoic acid-derived *O*-benzoyl hydroxyamide was employed. Related substrate, wherein the transferred hydrogen originates from a carbocycle, *e.g.*, cyclopentane, also underwent a smooth reaction with diene **2a** to form products **3y**. Besides substrates involving the abstraction of tertiary or benzyl-type (**1a**) C−H bonds, substrates bearing unactivated primary δ-C−H bonds were also tolerated (albeit with lower efficiency), as demonstrated by the formation of **3z** and **3aa**. Notably, the formation of product **3z** also demonstrated that steric bulk adjacent to the abstracted site could be tolerated. Product **3aa** exhibited that the hydrogen abstraction would exclusively occur at the δ-position of the nitrogen atom even in the presence of a more active tertiary C−H bond. Primary C−H bonds with an adjacent oxygen atom could be alkylated more efficiently, as shown by the formation of adduct **3ab** in 88% yield. In addition, functionalization of the secondary δ-C−H bonds could also take place efficiently, thus leading to adducts **3ac**−**3ag** bearing two stereocenters in 63%−87% yields. A number of dienes were then examined for the reactions with 4-methylpentanoic acid-derived *O*-benzoyl hydroxyamide (**3ah**−**3al**). Generally, 1-aryl-substituted dienes with both electron-donating and -withdrawing groups on the benzene were suitable substrates, while the electron-rich substrates were slightly more favored.

The addition of 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) to the model reaction of **1a** and **2a** under otherwise standard conditions completely suppressed the formation of **3a**, while radical coupling product **4** was isolated in 60% yield. The observation supported the presence of a benzyl radical species resulting from a 1,5-HAT process. Moreover, the subjection of substrate **5** into the reaction with **2a** led to compound **6** in 27% yield, likely through a ring-opening, addition to the diene and allylic oxygenation process. The formation of compound **6** confirmed the presence of an alkyl radical intermediate (Figure 2).

Based on these mechanistic studies and relevant literatures, the proposed mechanism is presented in Figure 3. Initially, SET process between substrate **1** and the excited Pd(0) catalyst leads to the formation of amidyl radical **I** together with a Pd(I) species with the benzoate as the counter anion. An alkyl radical species **II** is then generated through a 1,5-HAT process. At this stage, side product alkenes might be generated through β-hydride elimination of an alkyl Pd(II) species [23]. In our case, the alkyl radical undergoes addition to diene **2** to give allyl radical **III′**. Recombination of radical **III'** and Pd(I) gives π-allylic Pd(II) intermediate **III** with a benzoate anion, which delivers the final allylic substitution product **3** after nucleophilic attack by a carboxylate anion. Notably, either intermediate **III** or **III′** might undergo elimination process to give dienes as the side product.

To illustrate the synthetic utility of the reaction, a series of transformations of the amide product bearing an allylic ester moiety were performed using **3x** as an illustration (Figure 4). Hydrogenation of **3x** in the presence Pd/C delivered ester **7** in 78% yield. Hydrolysis of the ester moiety of **3x** provided alcohol **8** in 91% yield. Ozonolysis of the double bond in **3x** readily gave aldehyde **9** in 80% yield. The allyl benzoate moiety could also undergo Tsuji-Trost reaction [48] in the

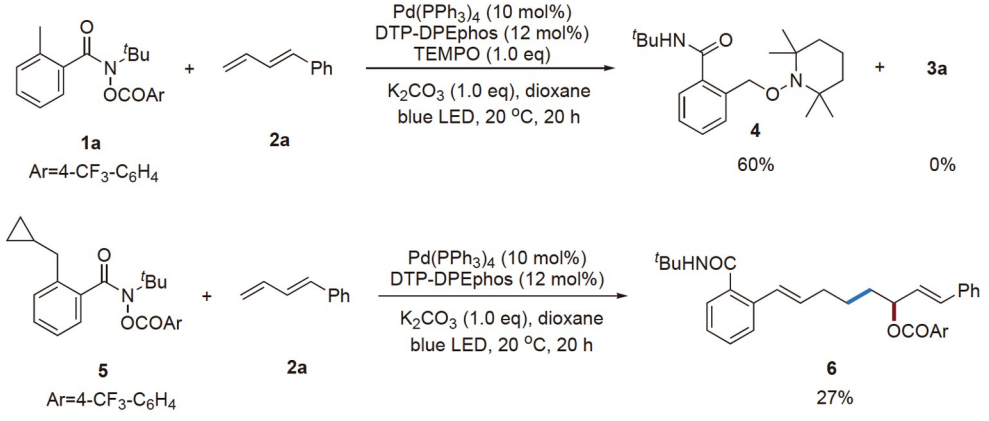


Figure 2 Mechanistic investigation.

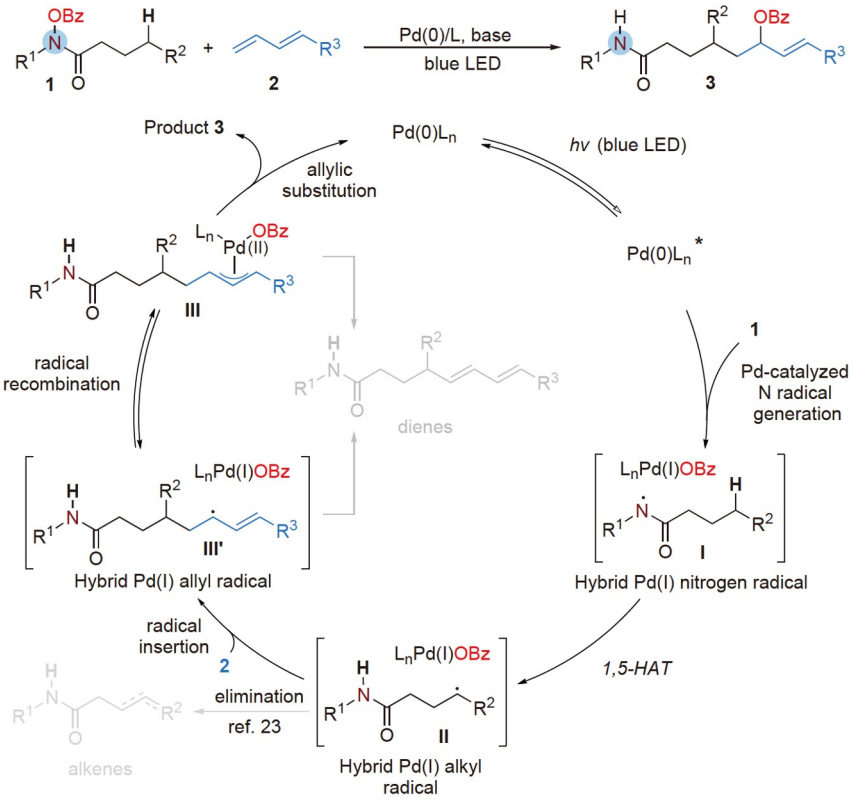


Figure 3 Proposed mechanism (color online).

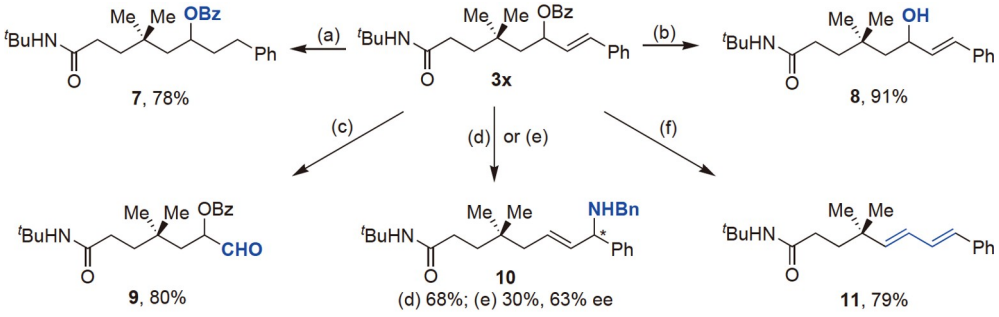


Figure 4 Derivation reactions of product **3x**. (a) Pd/C, H_2, MeOH, rt, 1 h; (b) K_2CO_3, MeOH, rt, 10 h; (c) O_3, CH_2Cl_2, −78 °C; (d) Pd_2dba_3/dppf, $BnNH_2$, $MeNO_2$, rt; (e) Pd_2dba_3/Trost's ligand, $BnNH_2$, $MeNO_2$, rt; (f) $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$, DPEphos, Et_3N, CsF, THF, rt (color online).

presence of a Pd(0) catalyst. Employing benzylamine as the nucleophile, the allylic amination could selectively occur at the carbon adjacent to the phenyl group to give compound **10**. Asymmetric allylic amination of **3x** was also preliminarily, affording the chiral allylic amine in 30% with 63% ee. Substituted 1,3-dienes are valuable building blocks in organic synthesis. Treatment of **3x** within In the presence of a $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$ and DPEPhos under basic conditions, **3x** could be transformed in to substituted diene **11** in 79% yield. Besides these examples, the *N*-*tert*-butyl amide moiety could also be hydrolyzed in acidic conditions [45].

In summary, we have successfully developed an efficient photoinduced Pd-catalyzed hydrogen atom transfer triggered regioselective 1,2-difunctionalization of 1,3-dienes. This unprecedented protocol afforded a diverse range of highly functionalized amides with 100% atom efficiency from readily available *O*-acyl hydroxyamides and 1,3-dienes. The cascade reaction proceeds through amidyl radical formation, 1,5-HAT, the addition of the resulted alkyl radical to 1,3-diene, formation of π-allyl palladium intermediate and regioselective allylic oxygenation process. Significantly, substrates bearing primary, secondary and tertiary $C(sp^3)$–H bonds at the δ-position of the nitrogen atom could all be readily functionalized. The current reaction has provided an efficient means to access functionalized amide-containing targets with readily available dienes.

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Conflict of interest The authors declare no conflict of interest.

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