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Olefination of Electron-Deficient Alkenes with Allyl Acetate: Stereo- and Regioselective Access to (2*Z*,4*E*)-Dienamides

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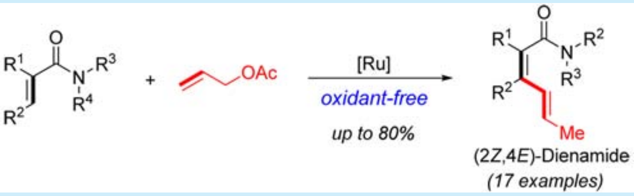
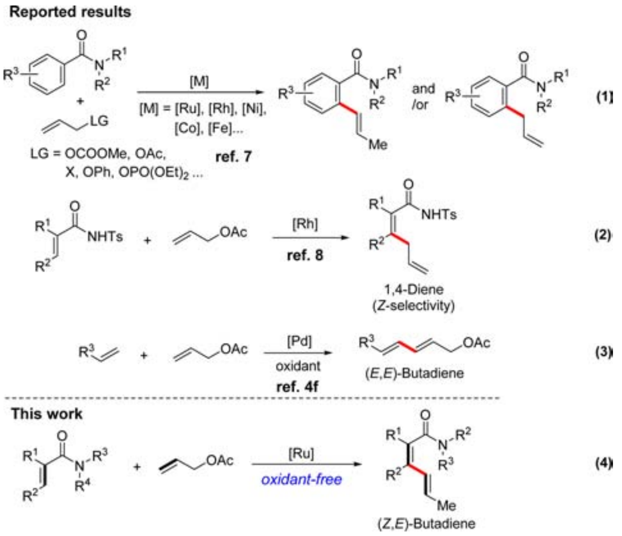
Supporting Information

ABSTRACT: A Ru-catalyzed direct olefination of electron-deficient alkenes with allyl acetate via C−H bond activation is disclosed. By using *N*,*N*-disubstituted aminocarbonyl as the directing group, this external oxidant-free protocol resulted in high reaction efficiency and good stereo- and regioselectivities, which opens a novel synthetic passway for access to (*Z*,*E*)-butadiene skeletons.



Butadienes are widely occurring functionalities commonly found in many natural products, drugs, and bioactive molecules.[1] In this regard, chemists have made intense efforts in the design and development of efficient, selective, and practical synthetic methodology for the synthesis of 1,3-butadienes.[2,3] Recently, following Ishii's pioneering work,[4a] a series of direct cross-couplings between two alkenes via double-vinylic C−H bond activation have been developed, representing an efficient and simple strategy for the synthesis of butadienes with highly atomic and step economy. Among them, the palladium-catalyzed direct cross-coupling of simple alkenes attracted a great deal of attention, which proceeded via alkenyl Pd species like Heck reaction generally providing (*E*,*E*) configured butadienes.[4,5] For example, Liu developed a Pd-catalyzed 1,3-butadiene synthesis by direct cross-coupling of alkenes and allyl acetate (Scheme 1, eq 3).[4f,g] Meanwhile, the rhodium- or ruthenium-catalyzed functional group directed cross-coupling between alkenes provided butadienes with excellent (*Z*,*E*)-stereoselectivities due to the olefinic C−H cyclometalation event, representing a complementary method for (*E*,*E*)-butadiene synthesis.[6] To date, some directing groups have been successfully introduced to the cross-coupling between alkenes, such as enolate,[6b] amide,[6c] ester,[6d,e] and phosphate,[6f] providing a series of valuable and functionalized 1,3-butadienes. However, this directed cross-coupling reaction is not developed, as there are still limited directing groups in practical use; in particular, a stoichiometric amount of oxidant is usually required to maintain the catalytic cycle, which produced a large amount of organic and inorganic waste. Furthermore, using electron-rich and internal alkenes as coupling partners is very rare. Thus, it is highly desirable to develop more efficient and oxidant-free cross-coupling reactions.

Scheme 1. Transition-Metal-Catalyzed Olefination and Allylation by C−H Activation with Allyl Derivatives



Allyl acetate and its derivatives have been deeply investigated in organic transformations as a popular and powerful allylation and olefination reagent, including transition-metal-catalyzed cross-coupling, Friedel−Crafts allylation of electronic-rich arenes, and catalytic C−H activation of electron-deficient arenes (Scheme 1, eq 1).[7] More recently, Loh group developed a rhodium(III)-catalyzed C−H allylation of electron-deficient alkenes with allyl acetates, delivering 1,4-dienes (Scheme 1, eq 2).[8] Meanwhile, the Kanai group reported a dehydrative direct C−H allylation with allylic alcohols by using a Co(III) catalyst.[9] Interestingly, Snaddon and co-workers disclosed a novel cooperative catalysis-based method for the direct asymmetric α-allylation of acyclic esters.[10] Very recently, the Ackermann group reported several novel allylation examples using inexpensive Fe, Co, or Mn catalyst.[11] These reports have made breakthroughs in allylation or olefination reactions, but double-bond migration and poor regioselectivity were usually inevitable and difficult to control. Hence, developing more efficient and selective reaction patterns of allyl acetates to

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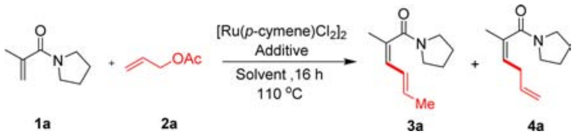
construct valuable molecules is still highly desirable. With our interest in novel 1,3-butadiene synthesis via C−H olefination, we assumed that allyl acetate could be introduced to the directed allylation of electron-deficient alkenes, first delivering 1,4-diene, and then the desired 1,3-butadiene of the thermodynamically more stable product could be formed after migratory isomerization of the C−C double bond,[7] forming the diene product which cannot be obtained by cross-coupling using electron-rich alkenes, such as propene, as coupling partners.[6] Herein, we report an inexpensive Ru(III)-catalyzed olefinic C−H bond olefination of acrylamides with allyl acetates in the absence of any oxidant, which opens a new synthetic route for the access of valuable (*Z,E*)-butadienes (Scheme 1, eq 4).

To evaluate the feasibility of our proposal, the reaction of readily available acrylamide **1a** and allyl acetate **2a** was chosen as a model to investigate catalytic conditions (Table 1).

Table 1. Optimization of Catalytic Conditions[a]



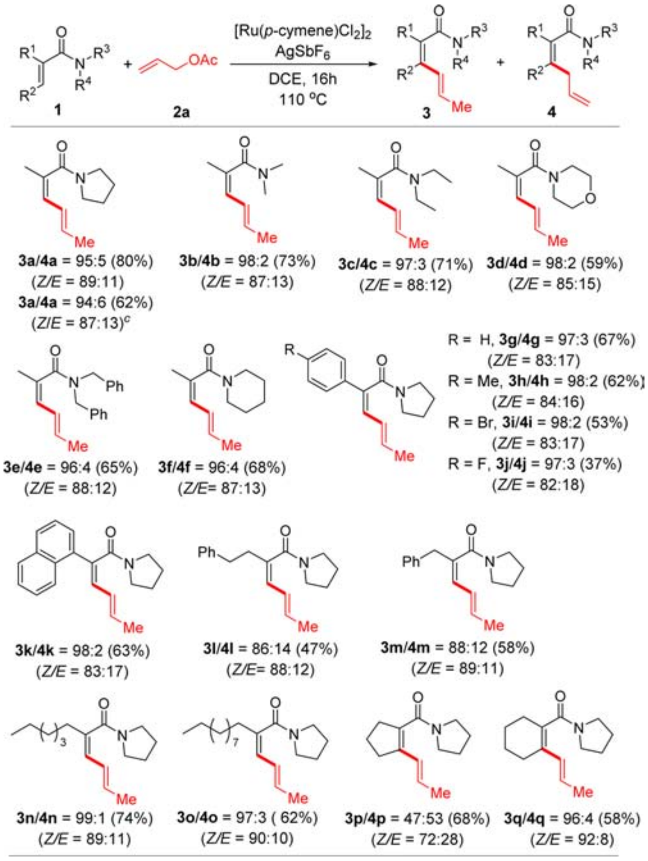
entry	additive	solvent	yield[a] (%)	*Z*/*E*	3a/4a
1	$AgSbF_6$	*t*-AmOH	10	86:14	68:32
2	$AgSbF_6$	DMF	**0**		
3	$AgSbF_6$	dioxane	8	89:11	>99:1
4	$AgSbF_6$	THF	21	>99:1	77:23
5	$AgSbF_6$	MeCN	0		
6	**$AgSbF_6$**	**DCE**	**80**	**88:12**	**95:5**
7[c]	$AgSbF_6$	DCE	45	>99:1	46:54
8[d]	$AgSbF_6$	DCE	33	98:2	92:8
9		DCE	0		
10	Ag_2CO_3	DCE	0		
11	KPF_6	DCE	0		
12	$Cu(OAc)_2$	DCE	0		

[a]Reaction conditions: **1a** (0.2 mmol), **2a** (0.4 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (3 mol %), and an additive (20 mol %) at 110 °C in a specific solvent (1 mL), under argon, 16 h. [b]Yields are of products **3a** and **4a**, *Z*/*E* ratio of **3a**, and the regioisomeric ratio of **3a**/**4a** was calculated by ^{1}H NMR. [c]The reaction was performed at 90 °C. [d]The reaction was performed at 130 °C. DMF: *N*,*N*-dimethylformamide.

Commercially available and inexpensive [Ru(*p*-cymene)Cl_2]$_2$ was chosen as the catalyst, using $AgSbF_6$ as an additive which may abstract chloride to generate cationic ruthenium complex for electrophilic C−H bond activation.[12] After screening a series of representative solvents, we were pleased to find that the product yield was dramatically improved to 80% with good selectivity (*Z*/*E* = 88:12)[13] when the reaction was performed in 1,2-dichloroethane (DCE), providing a trace amount of allylation product **4a** (**3a**/**4a** = 95:5) (Table 1, entry 6). However, other solvents, such as THF and *t*-AmOH, greatly hindered the reaction, while strong polar solvent, such as MeCN and DMF, provided no product (Table 1, entries 1−5). Moreover, lower temperature (90 °C) resulted in decreased yield but facilitated the allylation process, while elevating the temperature (130 °C) enhanced the olefination process with a decreased yield presumably due to degradation (Table 1, entries 7 and 8). Complex [Ru(*p*-cymene)Cl_2]$_2$ itself could not deliver the corresponding butadiene **3a** (Table 1, entry 9). Other additives, such as Ag_2CO_3, KPF_6, and $Cu(OAc)_2$, were also screened, but all of them failed to assist the ruthenium complex to form the product (Table 1, entries 10−12).

With the optimized catalytic conditions in hand, we turned to explore various *N*-substituted acrylamides **1** with allyl acetate **2a** to make (2*Z*,4*E*)-dienamides. As shown in Scheme 2, the differently *N*-substituted acrylamides **1** also reacted well with allyl acetate, providing the desired 1,3-butadienes **3a−f** in good stereoselectivities (*Z*/*E* up to 88:12). This cross-coupling reaction also proceeded smoothly when performed on gram scale as described in the synthesis of **3a**, and the product yield was obtained in 62% without a decrease in stereo- and regi-selectivity, showing the robustness of this method.[14]

Scheme 2. Scope of Differently Substituted Acrylamides[a,b]



[a]Reactions conditions: **1** (0.2 mmol), **2a** (0.4 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (3 mol %), $AgSbF_6$ (20 mol %) in DCE (1.0 mL) at 110 °C, 16 h. [b]The yields indicated are of isolated products **3** and **4**; *Z*/*E* ratio of **3** and the regioisomeric ratio **3**/**4** were calculated by ^{1}H NMR spectroscopy. [c]The reaction was performed on a 0.5 g scale.

Encouraged by these results, we turned our attention to further enlarge the scope of the cross-coupling reaction to other representative aliphatic and aromatic group tethered *α*- and/or *β*-substituted acrylamides (Scheme 2, **3g−q**). Installation of a phenyl ring to the *α*-position of acrylamide showed limited influence on the reaction, and the desired product swere isolated in 67% yield with excellent regioselectivity (**3g**/**4g** = 97:3), but the stereoselectivity decreased slightly (*Z*/*E* = 83:17). Valuable functional groups such as Br, F, or Me could be well tolerated, although the product yields decreased when an electron-withdrawing group introduced (Scheme 2, **3h−j**). A larger aromatic ring such as naphthalene-subsitituted acrylamide also provided good results (Scheme 2, **3k**). Other alkyl groups such as benzyl- and hexyl-tethering substrates also reacted well with good regioselectivity and *Z*/*E* selectivity (Scheme 2, **3l**−**o**). The reactivity of *α*,*β*-disubstituted acrylamides **1** was also examined. Acrylamide **1** bearing a cyclopentenyl unit reacted well, but the yield of allylation product **4p** increased remarkably. Interestingly, acrylamide embedded with a cyclohexenyl moiety exhibited excellent regioselectivity and stereoselectivity, forming trace amounts of 1,4-diene **4q**.[15]

Next, we turned to examine the reactivity of different allyl derivatives (Scheme 3).[16] Other allyl carboxylic esters, such as allyl hexanoate **2b**, allyl methacrylate **2c**, allyl phenoxyacetate **2d**, and allyl 3,3,3-trifluoropropanoate **2e**, were also tested, showing some decreased reactivity compared to allyl acetate **2a**. In contrast, allyl methyl carbonate **2f** is more inactive for olefination and allylation, forming product in only 24% yield. It is worth noting that allyl iodide **2g** did not exhibit any reactivity toward acrylamide, suggesting chelation of acetate oxygen to the metal is crucial in the catalytic process.[7,8]

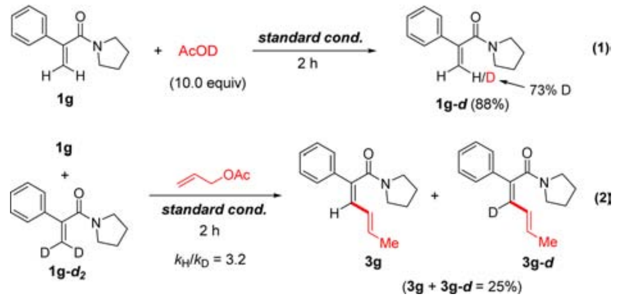
Scheme 3. Scope of Different Allyl Derivatives[a,b]

	3a	4a
2a, R = Me	80%, *Z*/*E* = 88:12	5%
2b, R = *n*-Hex	64%, *Z*/*E* = 86:14	10%
2c, R = $CH_3C{=}CH_2$	56%, *Z*/*E* = 83:17	< 5%
2d, R = CH_2OPh	61%, *Z*/*E* = 86:14	8%
2e, R = CH_2CF_3	35%, *Z*/*E* = 77:23	ND
2f, R = OCH_3	24%, *Z*/*E* = 86:14	< 5%
2g, allyl iodide	NR	

[a]Reactions conditions: **1a** (0.2 mmol), **2** (0.4 mmol), $[Ru(p\text{-cymene})Cl_2]_2$ (3 mol %), $AgSbF_6$ (20 mol %) in DCE (1.0 mL) at 110 °C, 16 h. [b]The yields indicated are isolated and total yields of **3a** and **4a**; *Z*/*E* ratio of **3a** and the yield of **4a** were determined by ^1H NMR.

In addition, to investigate the reaction mechanism, two deuterium-labeled experiments were conducted (Scheme 4). If the acrylamide **1g** was subjected to a standard catalytic system in the presence of acetic acid-d_4 (10.0 equiv) without allyl acetate, the cationic ruthenium species led to *Z*-selective H/D exchange on acrylamide, and *E*-selective H/D exchange was not observed, thereby indicating a reversible cyclometalation event (Scheme 4, eq 1).[6−8] Moreover, a kinetic isotope effect (KIE) of k_H/k_D = 3.2 was observed in the intermolecular isotopic study, suggesting the olefinic C−H bond metalation step is probably involved in the rate-determining step (Scheme 4, eq 2).[6e,f]

Scheme 4. Deuterium-Labeled Experiments



In conclusion, we have disclosed a stereo- and regioselective method for the olefination of acrylamides with allyl acetate. This ruthenium-based catalytic system opens a new synthetic route for access to valuable (2*Z*,4*E*)-dienamides.

■ ASSOCIATED CONTENT

Ⓢ Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.6b02229.

Experimental procedures and spectral data for all new compounds (PDF)

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Notes

The authors declare no competing financial interest.

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(13) The *cis*-structure was confirmed by the NOESY NMR analysis, indicating the amido group directed C−C bond formation step in the catalytic cycle (refs 7−9.)

(14) Secondary and primary amides were also tested, but none of them delivered the olefination or allylation product.

(15) Unfortunately, β-substituted acrylamide, such as crotonamide, and plain acrylamide without any substituent did not exhibit any reactivity even at elevated temperature.

(16) Other branched allyl acetates were also examined. α- or β-substituted allyl acetates were completely inert for cross-coupling, while γ-substituted allyl acetate afforded only trace amounts of product.

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