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1,1-Disubstituted olefin synthesis *via* Ni-catalyzed Markovnikov hydroalkylation of alkynes with alkyl halides†

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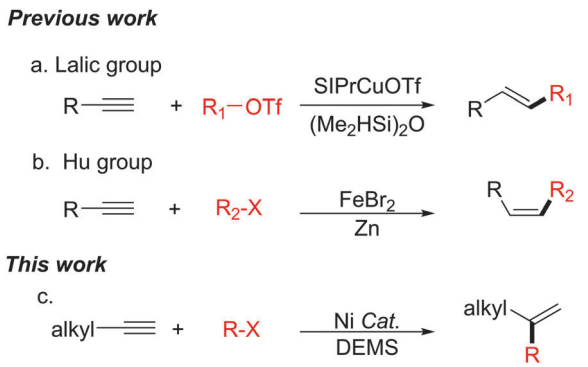
A Ni-catalyzed Markovnikov hydroalkylation of alkynes with alkyl halides is described. The reaction proceeds smoothly without the use of sensitive organometallic reagents and shows good functional-group compatibility, enabling the efficient synthesis of a variety of 1,1-disubstituted olefins. It also provides a straightforward approach for the modification of complex organic molecules.

Olefins are ubiquitous in materials, as well as the chemical and pharmaceutical industries.[1,2] Undoubtedly, numerous methods have been developed to date for their synthesis. Traditional methods, such as elimination reactions, the reduction of alkynes, condensation reactions, the Julia–Kocienski reaction *etc.* are still valuable in organic synthesis. In particular, the Wittig-type reaction is one of the most import reactions employed for the construction of olefins in modern organic synthesis.[3,4] Over the past few decades, significant progress has been made in the development of catalytic methods, for example, the semi-hydrogenation of alkynes, Heck reactions, olefin metathesis, and transition metal-catalyzed cross-coupling reactions of alkenyl halides or alkenyl metal reagents.[5] Recently, direct intermolecular hydrocarbonation between alkynes and electrophilic alkyl reagents have represented one of the most attractive approaches for the synthesis of olefins due to the wide availability of both starting materials.[6] Moreover, alkynes are an important synthon and structural motif in a variety of natural products, bioactive molecules, and functional materials.[7] It is worth pointing out that this approach may provide a potential route for the control of regio- and stereoselectivity. It also allows the synthesis of a diverse array of substituted olefins. Nevertheless, while there are many methods for the cross-coupling of alkynes with π-electrophiles, such as carbon dioxide,[8] formamides,[9] aldehydes,[10] ketones,[11] enones,[12] and imines,[13] hydroalkylation cross-coupling reactions of alkynes with alkyl (σ) electrophiles are rare.[14] Recently, the Lalic group reported the synthesis of (*E*)-alkenes *via* copper-catalyzed hydroalkylation of terminal alkynes with alkyl triflates (Scheme 1a).[15] Hu and coworkers realized the synthesis of (*Z*)-alkenes *via* the iron-catalyzed reductive coupling of terminal arylalkynes with alkyl halides (Scheme 1b).[16] However, these two elegant works provided efficient approachs for the hydroalkylation of alkynes with anti-Markovnikov regioselectivity, which afford 1,2-disubstituted olefins.



Scheme 1 Cross-couplings reactions for the hydroalkylation of alkynes with alkyl electrophiles.

In a continuation of our studies about the transition metal-catalyzed cross-coupling reactions of electrophilic alkyl reagents,[17] we now report the first example of a nickel-catalyzed hydroalkylation of terminal alkynes with Markovnikov regioselectivity using non-activated alkyl halides (Scheme 1c). The reaction has excellent Markovnikov regioselectivity. This newly developed reaction shows good functional-group compatibility due to its mild conditions, thus enabling the efficient synthesis of a variety of 1,1-disubstituted olefins from readily available alkynes and alkyl halides. It also provides a method for the modification of complex organic molecules.

To probe the feasibility of hydroalkylation cross-coupling reactions of terminal alkynes with Markovnikov regioselectivity, we began our study by choosing the reaction of (3-iodobutyl)benzene

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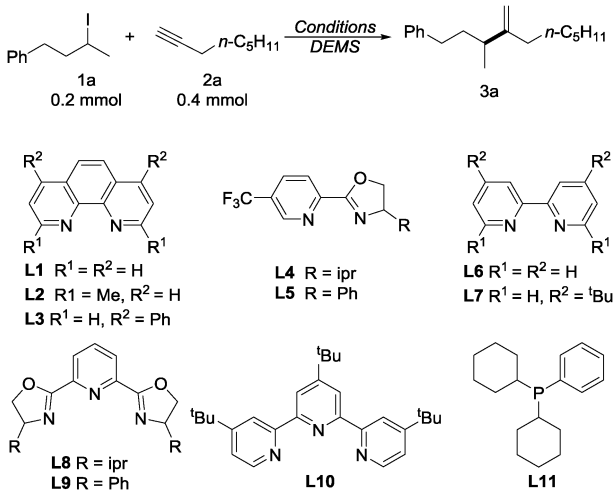
Table 1 Optimization of the reaction conditions



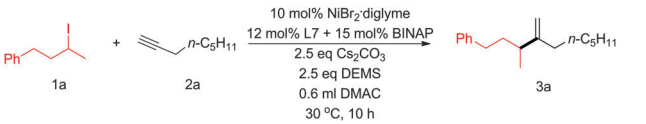
Entry	Catalyst (10 mol%)	Ligand (12 mol%)	Base (2.5 equiv.)	Solvent (0.6 mL)	Yield% **3a**[a]
1	$NiBr_2$·diglyme	**L1**	Cs_2CO_3	DMAC	10
2	$NiBr_2$·diglyme	**L2**	Cs_2CO_3	DMAC	7
3	$NiBr_2$·diglyme	**L3**	Cs_2CO_3	DMAC	4
4	$NiBr_2$·diglyme	**L4**	Cs_2CO_3	DMAC	6
5	$NiBr_2$·diglyme	**L5**	Cs_2CO_3	DMAC	5
6	$NiBr_2$·diglyme	**L6**	Cs_2CO_3	DMAC	50
7	$NiBr_2$·diglyme	**L7**	Cs_2CO_3	DMAC	65
8[b]	**$NiBr_2$·diglyme**	**L**	**Cs_2CO_3**	**DMAC**	**90 (84)**[d]
9	$NiBr_2$·diglyme	**L8**	Na_2CO_3	DMAC	35
10	$NiBr_2$·diglyme	**L9**	Cs_2CO_3	DMAC	2
11	$NiBr_2$·diglyme	**L10**	Cs_2CO_3	DMAC	2
12	$NiBr_2$·diglyme	**L11**	Cs_2CO_3	DMAC	5
13[c]	—	**L8**	Cs_2CO_3	DMAC	Trace

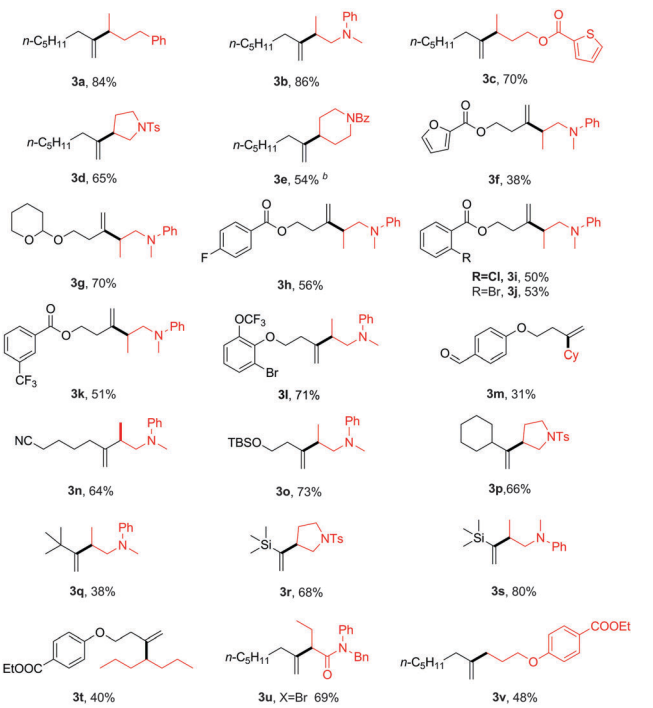
[a] The reaction was carried out at 30 °C for 10 h under an Ar atmosphere. 2.5 equiv. diethoxymethylsilane was used as a hydride donor. Yields determined by GC analysis used benzophenone as an internal standard (from an average of two GC runs). [b] **L7** and 15 mol% BINAP as ligand. [c] Without $NiBr_2$·diglyme. [d] Yield of isolated product. DMAC = *N*,*N*-dimethylacetamide, DMF = *N*,*N*-dimethylformamide, DMSO = dimethyl sulfoxide. THF = tetrahydrofuran.

(**1a**) and commercially available 1-octyne (**2a**) as a model (Table 1). The exploration commenced by carrying out screening for the ligand. When using $NiBr_2$·diglyme as a catalyst, 1,10-phenanthroline as a ligand, diethoxymethylsilane as a hydride donor and DMAC as a solvent, the desired product was observed albeit in a low yield of 10% (entry 1). This finding encouraged us to test other ligands in the phenanthroline family (entries 2 and 3), such as C2-substituted (**L2**) and C4-substituted (**L3**) phenanthroline. However, the amount of desired product obtained was still very low. We then used pyrox ligands (**L4–L5**) instead of phenanthroline-based ligands (entries 4 and 5). Disappointingly, this change was not useful for improving the yield. Gratifyingly, a bipyridine ligand **L6** exhibited much better reactivity and the GC yield increased to 50% (entry 6). Next we chose 4,4′-di-*tert*-butyl-2,2′-bipyridine (**L7**) as the ligand, resulting in a slight increase in the yield (entry 7). Unexpectedly, on the basis of **L7**, we added an additional 15 mol% BINAP and we obtained optimal reaction conditions (90% GC yield and an 84% isolated yield, entry 8). If only using BINAP as a ligand, the reaction efficiency was poor (see ESI†). We also tested various tri-nitrogen (**L8–10**) and monodentate phosphine ligands, for example **L11**, but only **L8** gave a moderate yield (entries 9–12). Finally, a control experiment indicated that the reaction completely shut down without the addition of nickel (entry 13). At the beginning of the optimization experiments, in order to achieve the Markovnikov regioselectivity for the hydroalkylation reaction, we also screened a series of bases, silanes and solvents (see ESI†).

With the optimized conditions in hand, we explored the scope of the Markovnikov hydroalkylation reaction. As shown in Table 2, our protocol exhibited an excellent chemoselectivity profile with many alkynes and non-activated alkyl halides containing different functional groups successfully converted into the desired products in modest to excellent yields (31–86%). Both acyclic and cyclic alkyl halides can be transformed. Furthermore, heterocycles, for example, thiophene (**3c**), pyrrolidine (**3d**), piperidine (**3e**), furan (**3f**), and tetrahydropyran (**3g**) are tolerated as either of the two coupling substrates. Due to the mild reaction conditions, this hydroalkylation is compatible with many synthetically relevant functional groups such as fluoride (**3h**), trifluoromethyl (**3k**), trifluoromethoxy (**3l**), ketal, amide (**3e**), sulfonamide (**3d**), and amine groups. Aryl–X (X = Cl, Br) bonds (**3i**, **3j**, **3l**) did not hinder the reaction, so it is possible to conduct additional

Table 2 Scope of the cross-coupling reaction[a,b]



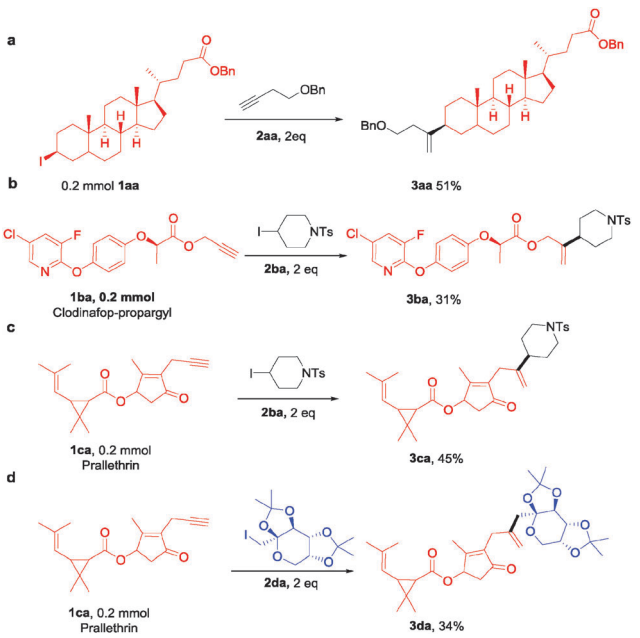


[a] The reactions were conducted on a 0.2 mmol scale at 30 °C. Yields of the isolated products are after 10 h. [b] 12 mol% ^{i}Pr-pybox, 2.5 equiv. Na_2CO_3, 3 equiv. DEMS, 0.2 mL DMF and 0.4 mL DMSO were used. TBS = *tert*-butyldimethylsilyl, TMS = trimethylsilyl, Bz = benzoyl, DEMS = diethoxymethylsilane and Ts = 4-toluenesulfonyl.

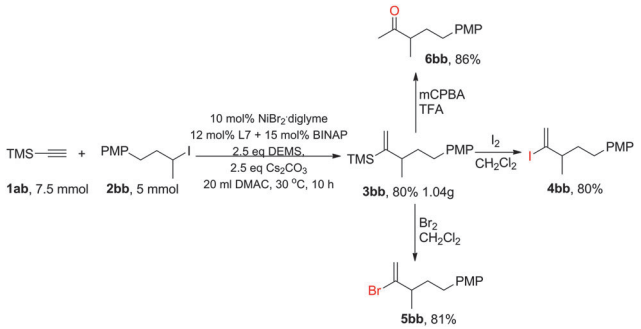
cross-coupling reactions at the halogenated positions. Some base-sensitive functional groups such as silyl (**3o**), ester, and nitrile (**3n**) groups are well tolerated. Even more active groups, such as benzaldehydes (**3m**), were compatible in the reaction. Large sterically hindered alkynes, for example, ethynylcyclohexane (**3p**), 3,3-dimethyl-1-butyne (**3q**), and trimethylsilylacetylene (**3r**, **3s**), still showed a remarkable selectivity. Activated secondary alkyl halides, for example, α-bromo amides (**3u**), were also good substrates with the exception of alkyl iodides. When using aryl alkynes, such as 1-ethynyl-4-methoxybenzene, as substrates, the regioselectivity and yield were disappointing (see ESI†).

We next demonstrated the usefulness of this regioselective Markovnikov hydroalkylation for the late-stage modification of complex biologically active molecules (Scheme 2). The treatment of lithocholic acid derivative **1aa** with **2aa** afforded the product **3aa** in moderate yield (Scheme 2a). Modification of clodinafop-propargyl (**1ba**) with **2ba** resulted in the formation of **3ba** (Scheme 2b), tolerating the pyridine, aryl–F, Cl, and ester groups. Prallethrin (**1ca**), which has been widely used in pesticides, could be smoothly converted to the desired product. Modification of prallethrin (**1ca**) with **2ba** resulted in the formation of **3ca** in 45% yield (Scheme 2d). In addition, we tested the cross-coupling of prallethrin with a fructose derivative (**2da**), and the highly complex desired product was obtained in 34% yield (Scheme 2e), thus offering a facile route to access the alkenylation of carbohydrates, which is of great interest in the life sciences.

To demonstrate the scalability of the Ni-catalyzed Markovnikov hydroalkylation, we also performed the reaction on a gram scale, which afforded **3bb** in 80% yield (Scheme 3). Furthermore, vinylsilane reagents are, in general, highly stable owing to their



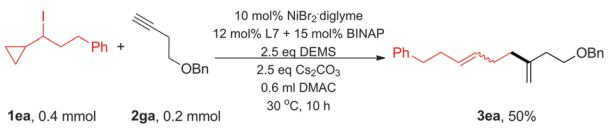
Scheme 2 The modification of complex molecules. Conditions: (a) 10 mol% $NiBr_2$·diglyme, 12 mol% **L7**, 15 mol% BINAP, 3.0 equiv. DEMS, 3.0 equiv. Cs_2CO_3, 1 mL DMAC, 30 °C, 10 h. (b–d) 15 mol% $NiBr_2$·diglyme, 20 mol% **L7**, 3.0 equiv. DEMS, 3.0 equiv. Cs_2CO_3, 1.2 mL DMAC, 30 °C, 10 h.



Scheme 3 A gram-scale cross-coupling reaction and the transform of vinyltrimethylsilane.

less polarised C–Si bonds. They are key intermediates in a variety of processes in materials science, polymer science, and organic chemistry.[18] Moreover, not only can the reported reaction be used to conduct cross-couplings, but it can also easily transform vinylsilanes to other atoms, such as bromine, iodine, sulphur, oxygen *etc.* Therefore, it could be widely used in organic synthesis. As mentioned above, we transformed the vinylsilane to bromine, iodine, and oxygen in good yields as shown in Scheme 3.

To gain preliminary understanding of the reaction mechanism, several experiments were conducted. First, when we added 1.0 equiv. of radical trapping agent, 2,2,6,6-tetramethylpiperidineoxy (TEMPO), the reaction was largely inhibited (for more details, see ESI†). Next, we performed a radical ring opening experiment between ((but-3-yn-1-yloxy)methyl)benzene (**2ea**) and (3-cyclopropyl-3-iodopropyl)-benzene (**1ea**) (eqn (1)) and we obtained the completely open product. The above observations are consistent with a radical-type mechanism for the alkyl halide. Nonetheless, a detailed possible mechanism for this reaction is not clear at present. Further studies are on-going in our group.



(1)

In summary, for the first time, we have developed a practical method for the formation of $C(sp^2)$–$C(sp^3)$ bonds through the Ni-catalyzed hydroalkylation of terminal alkynes with Markovnikov regioselectivity using non-activated alkyl halides under reductive conditions. This new reaction provides a useful approach to the synthesis of 1,1-disubstituted olefins using readily available alkynes as nucleophilic precursors. It does not need any sensitive organometallic reagents and exhibits good compatibility with many synthetically important functional groups. It also could be used for the modification of biologically relevant molecules. Our next challenge is to screen ligands to induce enantioselectivity in these regioselective Ni-catalyzed hydroalkylation cross-coupling reactions.

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