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# Direct functionalization of benzylic and non-benzylic C($sp^3$)–H bonds *via* keteniminium ion initiated cascade [1,5]-hydrogen transfer/cyclization

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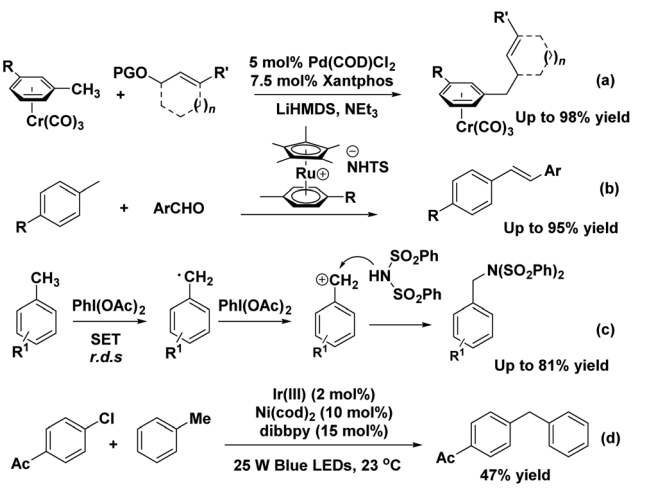
Incredibly electrophilic keteniminium ions generated *in situ* were employed as powerful intermediates for cascade [1,5]-hydride transfer/cyclization, *via* which inert benzylic and non-benzylic C($sp^3$)–H bonds without a directing group were directly functionalized to furnish complex polycycles in a single operation.

Over the past few decades, the transition-metal-catalyzed direct functionalization of notoriously unreactive C($sp^3$)–H bonds has emerged as a powerful methodology in organic synthesis due to its step economy, atom economy, and potential application for the late-stage functionalization of complex organic molecules.[1] However, this elegant approach generally suffers from high reaction temperature, low chemoselectivity and regioselectivity, narrow substrate scope, and indispensability of toxic transition metal catalysts. Additionally, specific directing groups on the substrates are required to facilitate the C($sp^3$)–H activation process,[2] thus diminishing the general applicability of the methods.

Among various C($sp^3$)–H bond functionalizations, the direct activation of the inert benzylic primary C–H bond in toluene and the non-benzylic C($sp^3$)–H bond without a directing group is particularly intriguing chemistry, which remains a formidable challenge for organic chemists. This chemistry has received considerable research interest from the organic chemistry community and a variety of protocols have been developed, which mainly rely on transition metal catalysis and a radical strategy.[3] Benzylic methyl groups in arenes can be activated *via* deprotonation–functionalization through the $\eta^6$-complexation of the phenyl moiety to a cationic transition metal complex, such as Cp*Ru[3f] and chromium tricarbonyl complexes.[4] Due to the electron-withdrawing character of the tricarbonylchromium or Cp*Ru, the acidity of the benzylic proton can be dramatically increased, furnishing the benzylic anions after deprotonation, which would serve as a surrogate for hard benzylic organometallic reagents. Walsh *et al.* utilized this strategy elegantly to accomplish the allylation of toluene-derived pronucleophiles (Scheme 1, a). However, this strategy is not ideal due to the high toxicity of chromium salt, the employment of which would cause severe environmental problems.[4] A similar approach was employed by Takemoto *et al.* for the direct catalytic dehydrative condensation between the benzylic C–H bonds of toluene/*p*-xylene and aromatic aldehydes using ruthenium-sulfonamide as the activator (Scheme 1, b), which provides highly atom-economical access to stilbenes.[3f]

Radical strategies can also be employed for direct benzylic C–H bond functionalization *via* H-abstraction by a radical generated from radical initiators such as DTBP[3i–l], TBHP, *etc.*,[3m,n] and in most cases the reactions proceed under metal catalyst-free conditions. Hypervalent iodine(III) has also been employed as a single electron oxidant for directive benzylic C–H bond




Scheme 1 Traditional approaches for the directive functionalization of benzylic primary C–H bonds in toluene without a directing group.

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functionalization under transition-metal-free conditions (Scheme 1, c).[3d] The radicals for the H-abstraction of benzylic C–H bonds can be generated by photocatalysis as well, *e.g.* chlorine radicals. Doyle *et al.* disclosed a C($sp^3$)–H cross-coupling of toluene enabled by the catalytic generation of chlorine radicals by nickel and photoredox catalysis, with aryl chlorides as both the cross-coupling partners and the chlorine sources (Scheme 1, d).[5] Murakami *et al.* employed a similar catalytic system consisting of nickel/dtbbpy for the photo-induced direct benzylic C–H arylation of toluene derivatives with aryl bromides under irradiation with UV light, in which the chlorine radical was generated with $NiCl_2$ as the chlorine source.[6] Although the inert benzylic C–H bond can be functionalized directly *via* these strategies, they still suffer from some apparent disadvantages, *e.g.* the indispensability of toxic transition metal catalysts, or low atom economy.

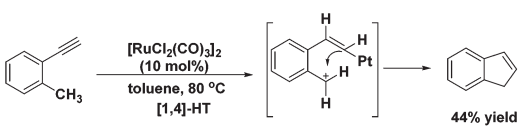
In addition to the traditional C($sp^3$)–H bond activation protocols mentioned above, the cascade [1,*n*]-hydride transfer/cyclization, which was initially termed '*tert*-amino effect', opens a new avenue for the direct functionalization of C($sp^3$)–H bonds.[7] The redox-neutral and highly atom-economical characteristics make it an attractive and ideal alternative for C($sp^3$)–H bond activation compared with the traditional protocols. Although this strategy has been elegantly exploited for the construction of various cyclic compounds, it still suffers from a number of lethal drawbacks. Firstly, a high temperature is a prerequisite for a decent yield due to the high energy barrier of the hydride transfer step. Secondly, the products furnished *via* this strategy are quite limited which are restricted to tetrahydroquinoline and chroman in most cases. Thirdly, the types of hydrogen donors are comparatively limited, which are limited to the heteroatom-containing ones in most cases. The benzylic C–H bond[3l,8] and non-benzylic C($sp^3$)–H bond[9] can also serve as hydrogen donors; the successful examples however are comparatively rare.

Chatani *et al.* once reported the cycloisomerization of 1-alkyl-2-ethynylbenzenes catalysed by $PtCl_2$, $PtCl_4$ and $[RuCl_2(CO)_3]_2$ for preparing substituted indenes with an alkynophilic transition metal activated alkyne as a hydride acceptor (Scheme 2).[10] Remarkably, the benzylic primary C–H bond could participate in this cascade process as a hydride donor to deliver indene in 44% yield.

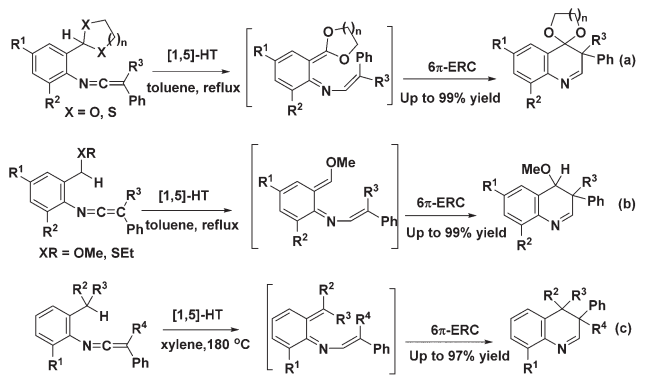
Alajarin *et al.* described a variety of cascade [1,5]-hydrogen transfer/cyclizations with the electrophilic ketenimine[11] and (thio)acetal[12]/(thio)ether[13]/arylmethines[8f] as a hydrogen acceptor and donors, respectively, which furnished the sterically congested 3,4-dihydroquinolines under thermal conditions alone (Scheme 3, a–c). Mechanistically, these 3,4-dihydroquinolines were produced *via* the 6π-electrocyclic ring closure of the 1,3,5-hexatriene intermediates generated *via* a [1,5]-hydride shift. Due to the comparatively low reactivity of the ketenimine, a high temperature was required for decent yields.

The non-benzylic C($sp^3$)–H bond can also serve as a hydride donor in the cascade hydride transfer/cyclization. Akiyama *et al.* reported a cascade [1,5]-hydride transfer/cyclization with non-benzylic methine and benzylidene barbituric acid as the hydride donor and acceptor, respectively, which furnish the desired tetraline in excellent yield using 3 mol% $Sc(OTf)_3$ as the catalyst (3 mol%) (Scheme 4, a).[14] They also reported another cascade process, in which non-benzylic aliphatic methine and the tosyl imine generated *in situ* were employed as the hydride donor and acceptor, respectively (Scheme 4, b).[8d] Although the reaction was heated to a high temperature with 30 mol% $Sc(OTf)_3$ loaded for a prolonged reaction time, the tetrahydroisoquinoline was obtained only in 32% yield, which might be attributed to the low reactivity of non-benzylic methine.

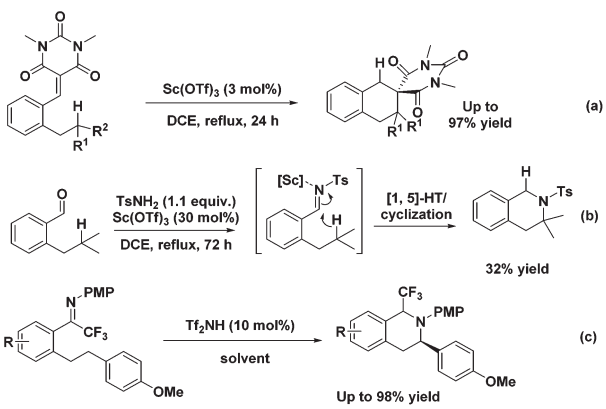
The same group described a Brønsted acid-catalyzed benzylic C–H bond functionalization as well with highly electrophilic trifluoromethyl ketimine employed as a hydride acceptor,[8e] which furnished 3-aryl-1-trifluoromethyl-tetra-




**Scheme 2** Cascade process for the synthesis of indene and tetrahydro-isoquinolines with benzylic C–H bonds as the hydride donors.




**Scheme 3** The electrophilic ketenimine as the hydride donor for the synthesis of 3,4-dihydroquinolines.




**Scheme 4** Non-benzylic C($sp^3$)–H bonds as the hydride donors.

hydroisoquinolines in high yield (Scheme 4, c). Notably, the diastereoselectivities could be readily tuned by a suitable selection of solvents and substituents on the nitrogen atom.

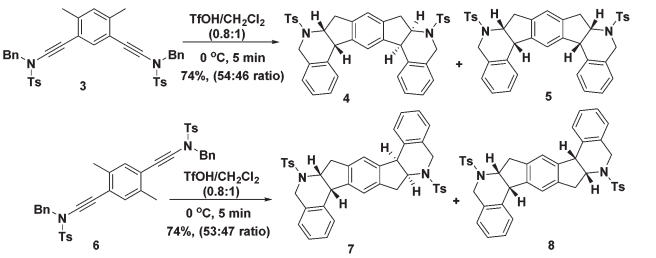
Ynamides bearing an electron-withdrawing group on the nitrogen atom have emerged as key and versatile building blocks due to their combined stability and peculiar reactivity.[15] The alkyne moiety is activated through an attached nitrogen atom bearing an electron-withdrawing group, which thus has both electrophilic and nucleophilic properties. The electronic properties and the reactivities of ynamides can be easily tuned by the selection of the electron withdrawing groups attached to nitrogen, thus making them highly versatile synthons for the syntheses of N-containing products. For instance, keteniminium ions can be exploited as incredibly electrophilic species with short life and high energy, which are conveniently generated *via* the treatment of ynamides with strong Brønsted acids.

Due to the ubiquity of polycycle frameworks in a large number of natural products and bioactive molecules, a variety of polycyclization strategies have been well developed for the syntheses of complex polycyclic molecules,[16] among which the cationic one is probably the most powerful at present.[17] Although the traditional cascade [1,*n*]-hydride transfer/cyclization has been recognized as an elegant cyclization strategy, only one full-carbon or hetero-cycle can be constructed.

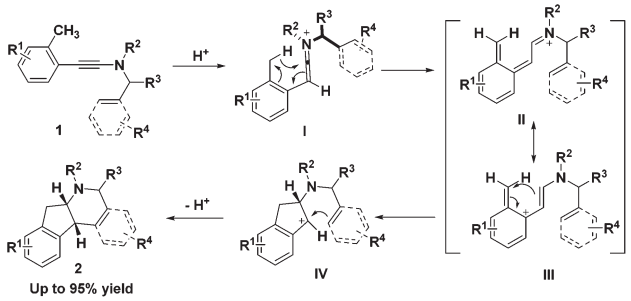
Recently, Evano *et al.* disclosed a novel and efficient keteniminium-initiated cationic polycyclization of ynamides which afforded the complex polycyclic nitrogen heterocycles **2** diastereoselectively, possessing up to three contiguous stereocenters and seven fused cycles in a single operation with TfOH or $Tf_2NH$ as promoters (Scheme 5).[8g,h] Compared with traditional cascade [1,5]-hydride transfer/cyclization, this work features three fascinating characteristics, the first one of which is that the multiple fused rings could be facilely constructed *via* double cyclization. The second one is that the inert C–H bond of benzylic methyl in toluene analogues can be readily functionalized *via* the cascade [1,5]-hydrogen transfer/cyclization. Thirdly, the cascade process was accomplished at low temperature (0 °C), which is in sharp contrast to the traditional thermal transformations. Mechanistically, the cascade process is initiated by the protonation of the electron-rich alkyne of ynamide **1**, yielding a highly reactive *N*-tosyl-keteniminium ion **I** which triggers a [1,5]-sigmatropic hydrogen shift to furnish the conjugated iminium **II** (in resonance with the bis-allylic carbocationic form **III**). The first cyclization would then occur in the manner of the Nazarov reaction by a 4π conrotatory electrocyclization, affording the carbocationic intermediate **IV**. Subsequently, the second cyclization of **IV** would operate through an intermolecular Friedel–Crafts-type reaction to deliver the final polycycle product **8**. When ethyl was used as a hydride donor instead of methyl, a comparatively lower yield was observed with complete diastereoselectivity and with three contiguous stereocenters established (Scheme 5).

The double cationic cascade polycyclization also proceeded smoothly upon the treatment of substrates **3** and **6** with triflic acid, which delivered the heptacyclic nitrogen heterocycles **4**/**5** and **7**/**8**, respectively, in good yields albeit with low diastereoselectivities (Scheme 6).

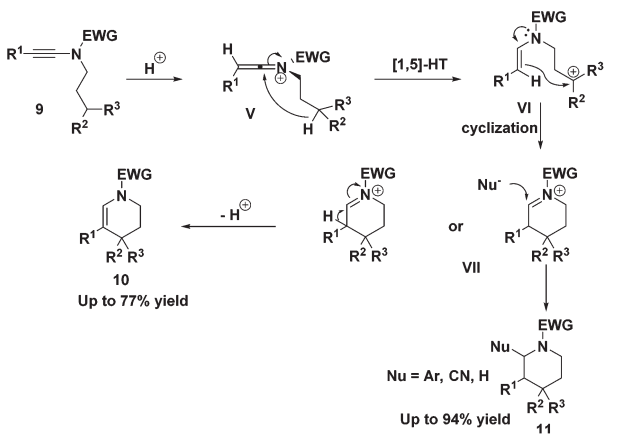
Evano *et al.* also demonstrated that the non-benzylic C($sp^3$)–H bond could be directly functionalized *via* the [1,5]-hydride shift/cyclization sequence with keteniminium ions as hydride acceptors, which provided straightforward and divergent access to tetrahydropyridines **10** or piperidines **11** in a single operation (Scheme 7).[9b] The cascade process could be accomplished in 15 min at −60 °C with TfOH as the catalyst. Mechanistically, the keteniminium ions generated *via* the protonation of the ynamide **9** initiate the [1,5]-hydride shift at the outset, furnishing the carbocationic intermediate **V**.



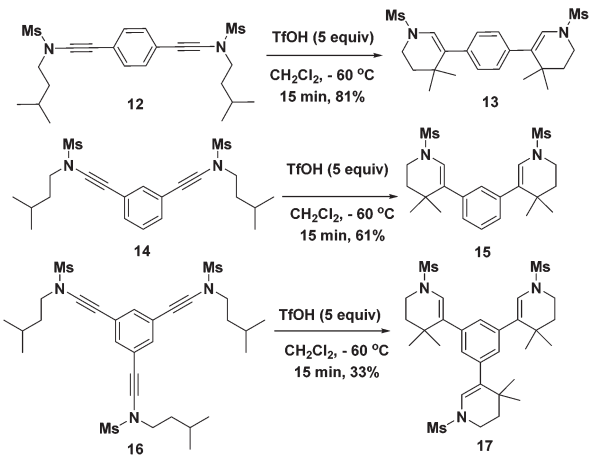

**Scheme 6** Double polycyclization from bis-ynamides.




**Scheme 5** Cascade cationic polycyclization initiated by keteniminium ions.




**Scheme 7** Keteniminium ions initiated cascade [1,5]-hydride transfer/cyclization for the synthesis of tetrahydropyridines and piperidines.




**Scheme 8** Double and triple hydride-shift/cyclization sequences.

Subsequently, an intramolecular cyclization of **I** occurs between the enamide moiety and carbocation moiety, leading to the intermediate **VI**. Then the tetrahydropyridine **10** is finally afforded *via* the elimination of a proton from the cyclic iminium ion **VII**. The iminium ionic intermediate **VII** can also be trapped by the exogenous nucleophile to furnish the substituted piperidines **11**. Similarly, the reactive iminium ionic intermediate **VII** can be reduced with triethylsilane as the reductant.

The double and triple cascade cyclizations initiated by keteniminium ions were also successful with bis-ynamides **12**, **14**, and tris-ynamides **16** subjected to the optimized conditions (Scheme 8), furnishing the corresponding bis-tetrahydropyridines **13**, **15** and tris-tetrahydropyridines **17**, respectively, in good yields, which further demonstrated the robustness and efficiency of this strategy.

## Conclusions

We have highlighted the great power of the incredibly electrophilic keteniminium ions which initiate the cascade [1,5]-hydride transfer/cyclization for the direct functionalization of benzylic primary C–H bonds in toluene and non-benzylic C($sp^3$)–H bonds without a directing group to construct complex poly-heterocycles in a single operation. In sharp contrast to the traditional approaches, this strategy features higher step economy, metal-free conditions and low reaction temperature, and highly complex products, which unambiguously demonstrated its robustness and efficiency.

## Conflicts of interest

There are no conflicts to declare.

## Acknowledgements

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