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Nickel Catalysis

Synthesis of Indanones and Spiroindanones by Diastereoselective Annulation Based on a Hydrogen Autotransfer Strategy

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Abstract: *An unprecedented nickel-catalyzed domino reductive cyclization of alkynes and o-bromoaryl aldehydes is described. The reaction features broad substrate scope and is tolerant of a variety of functional groups, providing straightforward access to biologically significant indanones and spiroindanone pyrrolidine derivatives in good yields with excellent regio- and diastereoselectivity. Preliminary mechanistic studies have shown that indanones are formed by the cyclization of o-bromoaryl aldehydes and alkynes to form indenol intermediates, followed by hydrogen autotransfer.*

Indanone and spiroindanone scaffolds are central structural motifs and can be found in a variety of biologically active natural products and pharmaceuticals,[1] such as pauciflorol F, pterosin B and C,[2] donepezil,[3] fredericamycin A[4] and exiguaquinol[5] (Figure 1). As a result, a considerable number of methods for their synthesis have been developed.[6] Transition-metal-catalyzed domino cyclization of *ortho*-carbonylated aryl halides and alkynes is one of the most straightforward methods to construct indenone scaffolds (Route a, Scheme 1a, top).[7] Rhodium-catalyzed direct C–H bond annulation reaction for the synthesis of indenones has also been reported (Route b, Scheme 1a, top).[8] In addition,

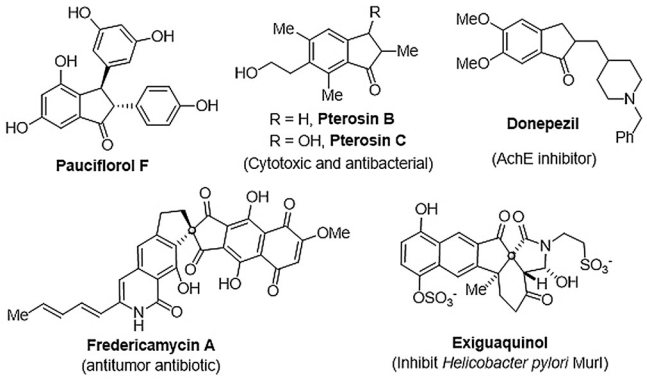

Figure 1. Representative natural products and pharmaceuticals containing indanone and spiroindanone cores.

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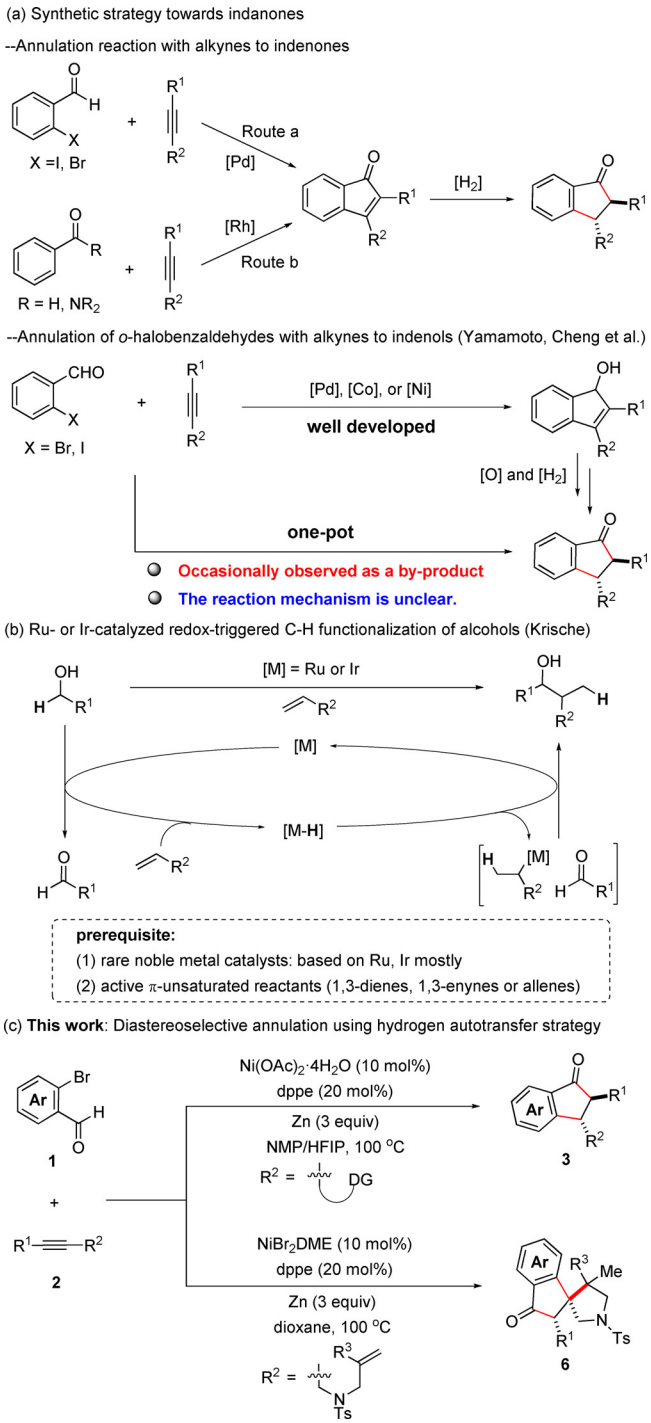


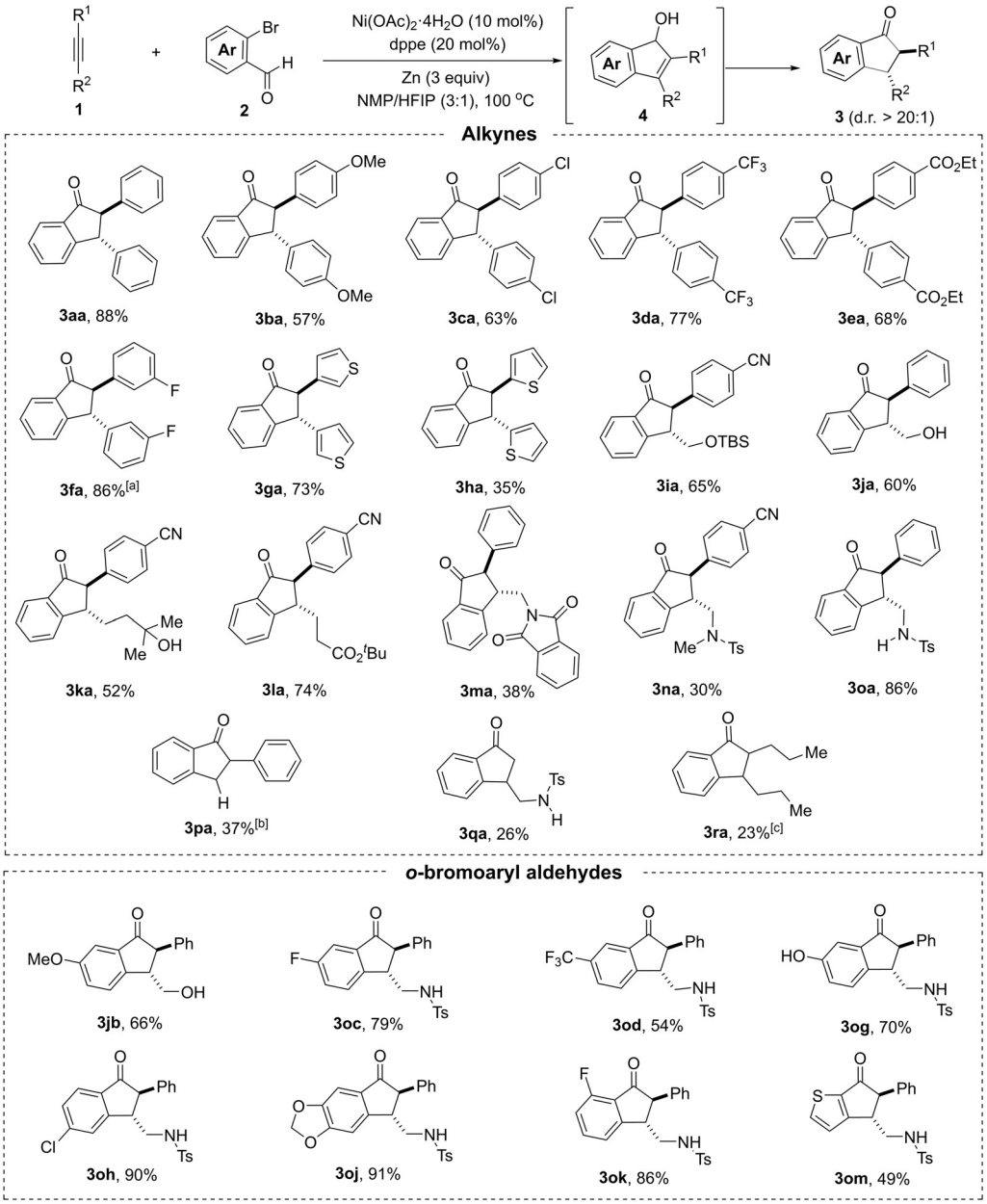
Scheme 1. Transition-metal-catalyzed domino reactions. DME = 1,2-dimethoxyethane, dppe = 1,2-bis(diphenylphosphanyl)ethane, NMP = 1-methyl-2-pyrrolidinone, HFIP = hexafluoroisopropyl alcohol.

Yamamoto[9] and Cheng[10] independently reported the annulation reaction of *ortho*-carbonylated aryl halides and alkynes for the synthesis of indenols (Scheme 1a, bottom). Conversion of indenones or indenols to indanones usually requires additional oxidation and/or reduction steps. Interestingly, indanones were occasionally observed as by-products in the one-pot reaction of *ortho*-carbonylated aryl halides and alkynes,[9b,10d] but the reaction mechanism for their formation remains unclear.

Meanwhile, the borrowing hydrogen catalysis, also known as hydrogen autotransfer, has emerged as an appealing tool for C–C bond formation in recent years.[11] Krische group developed the ruthenium- or iridium-catalyzed redox-triggered carbonyl addition reactions, in which hydrogen is transferred from the alcohols to active π-unsaturated reactants (1,3-dienes, 1,3-enynes or allenes) to afford aldehyde–organometal pairs, which combine to form the formal products of alcohol C–H functionalization (Scheme 1b).[12] In spite of notable progress, almost all borrowing hydrogen reactions have relied on the use of simple alcohols as starting materials and precious noble metals as catalysts, specifically those based on Ru and Ir. In addition, hydrogen transfer from alcohol to unactivated alkenes has rarely been demonstrated.[13]

Recently, we reported the nickel-catalyzed regioselective reductive coupling of two unsymmetrical internal alkynes for the synthesis of pentasubstituted 1,3-dienes.[14] The key to the success of this reaction was the use of hemilabile directing group strategy. We envisioned that the same strategy may also be used to control the regioselectivity in the cyclization reaction of *o*-carbonylated aryl halides and unsymmetrical alkynes. A more intriguing question thus arose: if the nickel-catalyzed regioselective cyclization reaction of *o*-carbonylated aryl halides with alkynes and the hydrogen autotransfer process could be combined into one synthetic platform, it would provide a more sustainable and attractive alternative for the synthesis of biologically significant indanones and spiroindanones (Scheme 1c).

Our study commenced with the reductive cyclization reaction of *o*-bromobenzaldehyde **2a** and diphenylacetylene **1a**. After systematically evaluating the reaction parameters



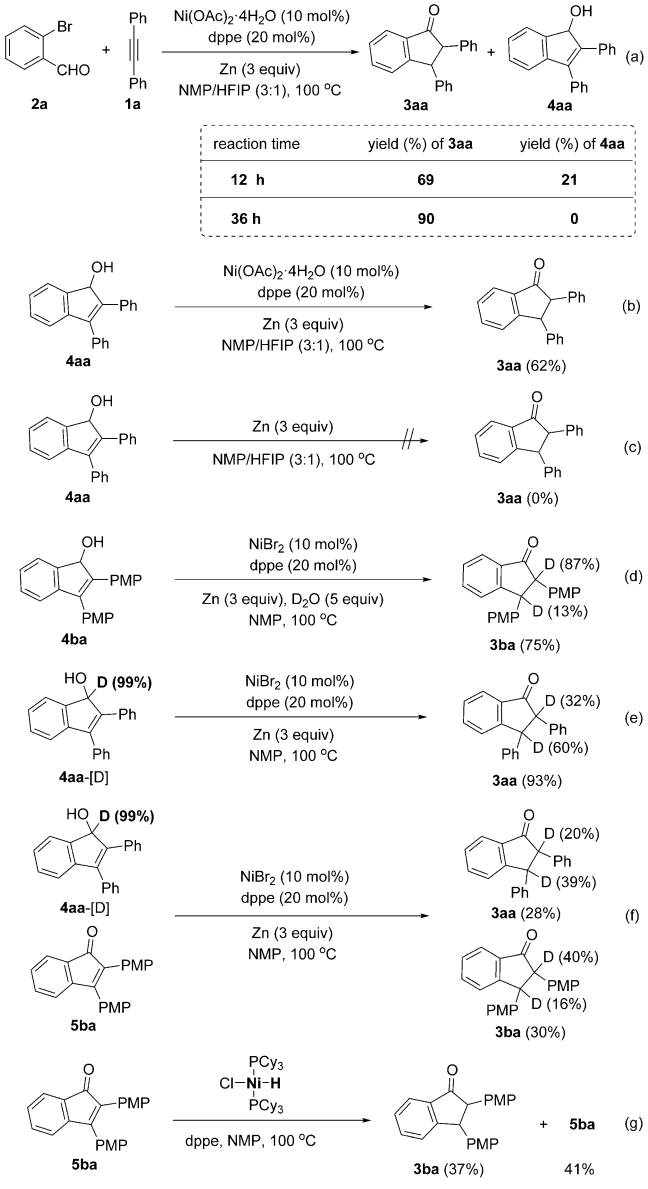
Scheme 2. Synthesis of indanones. Reaction conditions: **1** (0.1 mmol), **2** (0. 3 mmol), $Ni(OAc)_2{\cdot}4H_2O$ (10 mol%), dppe (20 mol%), Zn (0.3 mmol) in NMP (0.75 mL) and HFIP (0.25 mL) in a sealed tube at 100°C for 36 h. Yields of isolated products are given. [a] The reaction was conducted with 6 equivalents of **2a**. [b] **1p**: trimethyl(phenylethynyl)silane. [c] The reaction was conducted at 120°C; **4ra** was isolated in 35% yield. TBS = *tert*-butyldimethylsilyl, Ts = *p*-toluenesulfonyl.

(see the Supporting Information), we were pleased to find that a combination of $Ni(OAc)_2{\cdot}4H_2O$ (10 mol%) and dppe (20 mol%) afforded indanone **3aa** in 88% yield using Zn powder as reducing agent in NMP/HFIP (3:1) at 100 °C. The addition of HFIP could significantly improve the reaction efficiency. Lowering the temperature resulted in diminished reactivity. Control experiments revealed that both nickel catalyst and Zn powder were indispensable.

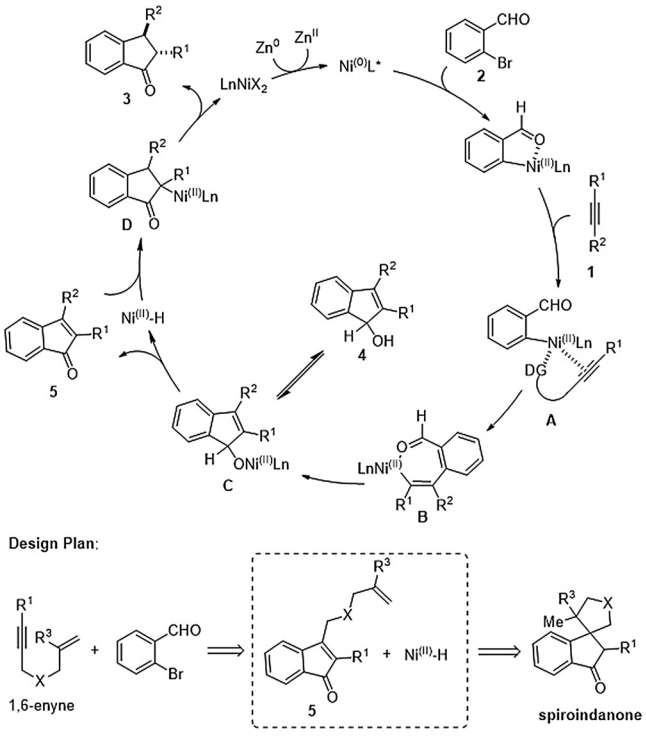
With the optimized reaction conditions in hand, we set out to explore the generality of our transformation (Scheme 2). Diarylalkynes bearing various substituent groups, such as OMe (**3ba**), Cl (**3ca**), CF_3 (**3da**), ester (**3ea**) or F (**3fa**) were well compatible with the reaction conditions. 2-Thiophene and 3-thiophene proceeded smoothly to furnish the target products **3ga**–**3ha** in moderate to good yields. For unsymmetrical internal alkynes **1i**–**1p**, the reactions with **2a** afforded the corresponding indanones **3ia**–**3pa** in moderate to good yields. In all cases, a single regioisomer was detected in the reaction mixture, where the alkyne carbon bearing aryl group is connected to the carbonyl group of **2**. Particularly informative was the chemoselectivity profile, as free hydroxyl groups (**3ja**–**3ka**), esters (**3la**), amides (**3ma**–**3na**), and free amine groups (**3oa**) could all be well-tolerated. Terminal alkyne was also a competent substrate to provide the desired product **3qa** with high regioselectivity. A dialkyl-substituted alkyne showed lower reactivity, mainly to obtain unconverted indenol (**4ra**, 35%).

The *o*-bromoaryl aldehydes bearing electron-donating or electron-withdrawing groups afforded the corresponding indanones **3jb**–**3og** in good yields with excellent regio- and diastereoselectivity. The presence of *ortho* substituent did not reduce the reaction efficiency (**3ok**). Interestingly, 3-bromothiophene-2-carbaldehyde proceeded smoothly to afford the desired bicycle **3om** in 49% yield.

A variety of mechanistic experiments were designed to shed light on the mechanism. Under standard conditions, the reaction of **1a** and **2a** was stopped after 12 hours. It was found that in addition to observing of an appreciable quantity of remaining aldehyde **2a**, the target **3aa** and indenol **4aa** were also isolated in 69% and 21% yield, respectively (Scheme 3a). Subsequently, pure indenol **4aa** was subjected to the standard reaction conditions, **3aa** was obtained in 62% yield (Scheme 3b). These results clearly indicate that indenol **4aa** is a key intermediate in the reaction. Control experiment further confirmed that no target product **3aa** was detected in the absence of nickel catalyst (Scheme 3c), thus suggesting that nickel catalyst is indispensable in the hydrogen autotransfer process.



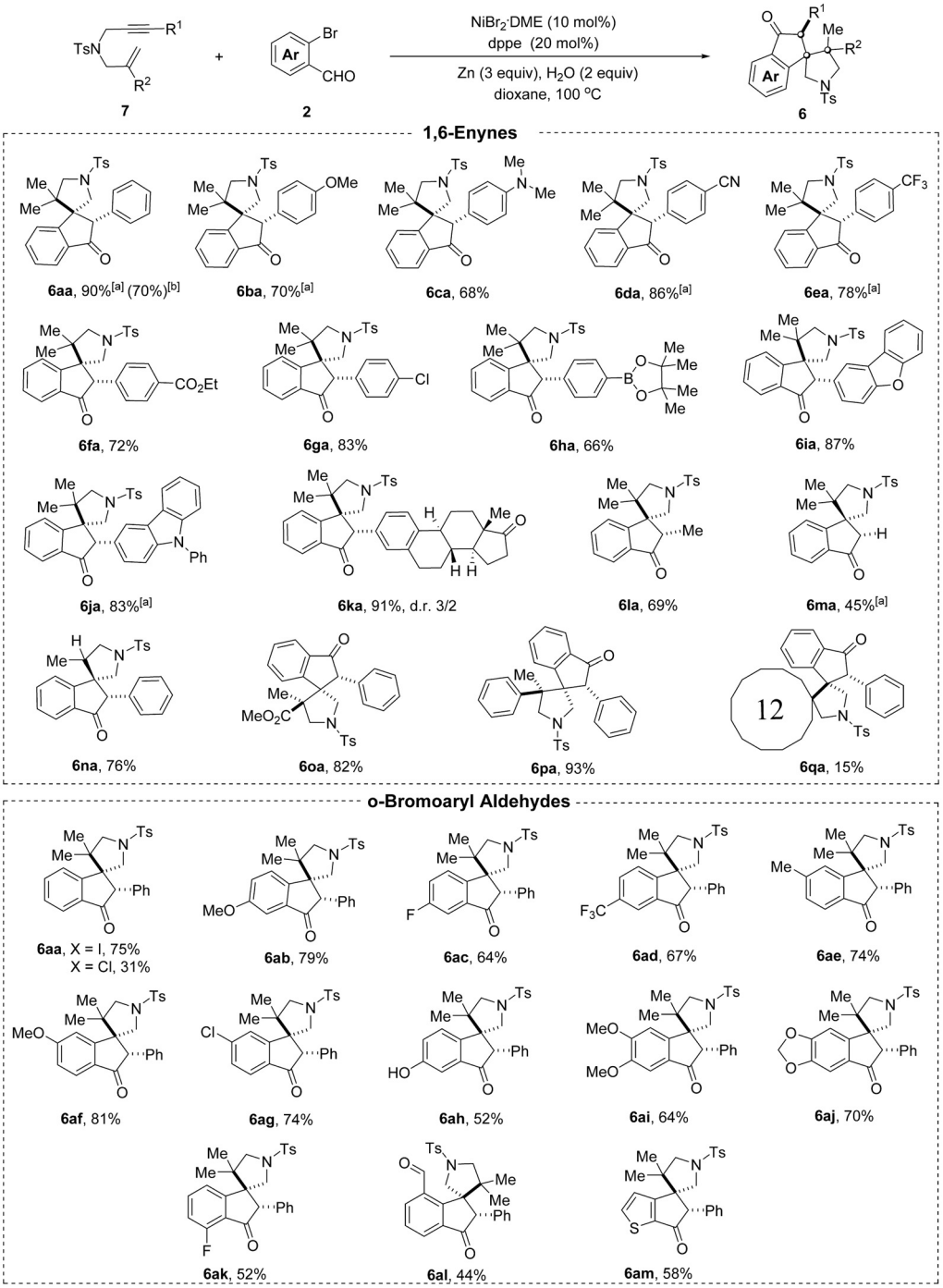
Scheme 3. Mechanistic study. Cy = cyclohexyl, PMP = *p*-methoxyphenyl.



Scheme 4. Proposed mechanism and further rational design.

The reaction of **4ba** and 5 equivalents of D_2O afforded the desired **3ba** in 75% yield, and 87% of the deuterium is incorporated into the α-position of ketone (Scheme 3d). Deuterium-labeled indenone **4aa**-[D] was synthesized and subjected to the reaction conditions, delivering the target product **3aa** in 93% yield. The deuterium originally on the aldehyde group was incorporated into the α- and β-position of ketone moiety in 32% and 60%, respectively (Scheme 3e). A crossover experiment was also designed to understand whether the hydrogen transfer is an intermolecular or an intramolecular process. A 1:1 mixture of indenol **4aa**-[D] and indenone **5ba** was subjected to the reaction conditions as shown in Scheme 3f, both the deuterium-labeled **3aa** and **3ba** were observed, thus suggesting that the intermolecular hydrogen transfer is feasible (Scheme 3f). Moreover, a structural defined Ni–H complex $(Cy_3P)_2NiClH$[15] was subjected to the stoichiometric reaction with indenone **5ba**, the desired **3ba** was indeed obtained in 37% yield (Scheme 3g). These results support that indenone **5** is a key intermediate, and Ni–H species is indeed formed during the reaction.

On the basis of the above results, a plausible mechanism was proposed as shown in Scheme 4.[10a] The catalytically active Ni^0 catalyst was generated in situ upon reduction by Zn^0. Oxidative addition of Ni^0 to *o*-bromoaryl aldehyde **2**, followed by migratory insertion into alkyne **1** gave seven-membered alkenyl–nickel intermediate **B**. The regioselectivity of migratory insertion was controlled by the hemilabile directing group. Nucleophilic addition of alkenyl–nickel species **B** to the aldehyde afforded alkoxynickel intermediate **C**, the subsequent β-H elimination would product indenone **5** and Ni–H species. The desired indanone **3** was finally afforded after hydronickelation of indenone **5** and the subsequent isomerization and hydrolysis.



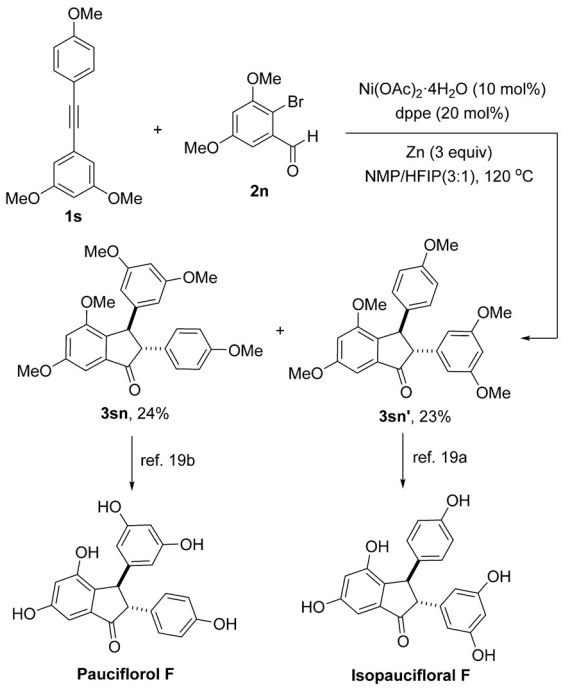
Scheme 5. Synthesis of spiroindanone pyrrolidines. Reaction conditions: **2** (0.1 mmol), **7** (0.2 mmol), $NiBr_2$·DME (10 mol%), dppe (20 mol%), Zn (0.3 mmol), H_2O (0.2 mmol) in dioxane (2 mL) in a sealed tube at 100°C for 72 h. Yields of isolated products are given; the diastereomeric ratio was >20:1 for all reactions. [a] The reaction was conducted with 3 equivalents of 1,6-enyne **7**. [b] The reaction was conducted on a 1.3 mmol scale with respect to **2a** with 1.3 equivalents of **7a**.

If the above mechanism is feasible, an intramolecular borrowing hydrogen cyclization of 1,6-enyne would provide a possibility for the synthesis of spiroindanones. Despite the privileged structure and utility of spirocycles in medicinal chemistry,[16] the catalytic stereoselective construction of spirocyclic skeletons with multiple stereocenters is still extremely challenging.[17] Therefore, we sought to further extend our strategy to the diastereoselective synthesis of spiroindanones.

After modifying the reaction conditions (see the Supporting Information), we were pleasure to find that a variety of 1,6-enynes **7** could undergo cyclization with **2a** to provide the desired spiroindanone pyrrolidine derivatives **6aa**–**6qa** in good yields with excellent diastereoselectivities (Scheme 5). Substitutions on the aromatic ring at the terminus of the alkyne with both electron-donating (methoxy and amino) as well as electron withdrawing groups (cyano, trifluoromethyl and ester) were well tolerated to furnish **6ba**–**6fa** in 68–86% yield. Remarkably, the presence of chlorine or borate on the aromatic ring could also be perfectly accommodated (**6ga**–**6ha**). Heteroaromatic-substituted 1,6-enynes, such as 3-dibenzofuran and 3-carbazole, were amenable substrates. Complex biologically important estrone derivative could also be successfully incorporated into the desired product **6ka** in 91% yield. Terminal and alkyl substituted 1,6-enynes were compatible with this transformation (**6la** and **6ma**). Alkenes bearing different substituents in the internal position of the double bond underwent the reaction smoothly, providing the corresponding spiroindanones **6na**–**6pa** with three stereogenic centers in high diastereoselectivity. Interestingly, the trisubstituted 12-membered internal alkene was also found to be reactive to furnish **6qa**, albeit in low yield. C- or O-tethered 1,6-enynes and 1,7-enyne failed to obtain the target product.

In addition to 1,6-enynes, a variety of *o*-bromoaryl aldehydes **2** were also explored (Scheme 5). Aryl bromide (90%) showed superior reactivity than aryl iodide (75%) and aryl chloride (31%). The substitution pattern and electronic properties of the substituents on the aromatic ring did not interfere with the reaction efficiency. Free hydroxyl substituted substrate proceeded smoothly, directly providing **6ah** in 52% yield. Again, 4,5-dimethoxy- or methylenedioxy-substituted *o*-bromoaryl aldehydes were well-tolerated (**6ai**–**6aj**). Even the more sterically hindered **2k** and **2l** were underwent the reaction efficiently (**6ak**–**6al**). Importantly, 3-bromothiophene-2-carbaldehyde was also applicable to the reaction to give the desired **6am** in good yield, which further demonstrated the merit of this protocol.

The utility of the protocol was further demonstrated in the concise synthesis of pauciflorol F and isopaucifloral F (Scheme 6). Pauciflorol F and isopaucifloral F are polyphenolic natural products derived from resveratrol and exhibit a wide range of biological activities, such as antioxidant, anti-HIV, antifungal, anti-inflammatory, antibiotic, and anticancer activities.[18] Many elegant strategies toward their synthesis have been developed to date.[19] The most efficient method was developed by Sarpong[20] by palladium-catalyzed Larock annulation, and pauciflorol F was synthesized in three steps from tolane and bromobenzaldehyde. As expected, the nickel-catalyzed borrowing hydrogen cyclization of unsymmetrical tolane **1s** and **2n** afforded a mixture of 1:1 regioisomers **3sn** and **3sn′**. Pauciflorol F and Isopaucifloral F could be obtained after demethylation according to known method.[19] Therefore, the synthesis of pauciflorol F and Isopaucifloral F can now be accomplished in only two steps.



Scheme 6. Concise formal synthesis of pauciflorol F and isopaucifloral F.

In conclusion, a nickel-catalyzed domino reductive cyclization of alkynes and *o*-bromoaryl aldehydes has been developed. The reaction is tolerant of a variety of functional groups, providing a straightforward access to indanones and spiroindanones in good yields with excellent regio- and diastereoselectivity. Preliminary mechanistic studies have shown that indanones were formed by a sequence involving the annulation of *o*-bromoaryl aldehydes and alkynes to form indenol intermediates, followed by hydrogen autotransfer.

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Conflict of interest

The authors declare no conflict of interest.

Keywords: diastereoselectivity · domino reactions · hydrogen autotransfer · indanones · spiro compounds

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