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Ni-Catalyzed Dimerization and Arylation of Diarylacetylenes with Arylboronic Acids

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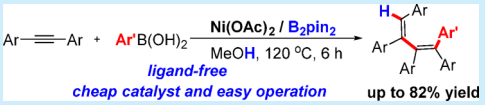
Supporting Information

ABSTRACT: A new, facile, and efficient protocol for the synthesis of polysubstituted conjugated 1,3-dienes through Ni-catalyzed tandem dimerization/cross-coupling reaction of diarylacetylenes and arylboronic acids in the presence of a catalytic amount of B_2pin_2 has been developed. A series of arynes and arylboronic acids with different substituents participated well in this catalytic system, affording a variety of useful conjugated 1,3-dienes.



The conjugated 1,3-dienes are an important class of molecules which are prevalent in a variety of biologically active natural compounds and pharmaceuticals.[1] Due to their unique reactivities, they are also vital building blocks for the synthesis of diverse carbocycles, heterocycles, and lactams.[2] Several common synthetic approaches including olefination of carbonyl compounds,[3] transition-metal-catalyzed cross-coupling reactions,[4] rearrangement, reduction, and metathesis of enynes,[5] as well as the Wittig reactions of allylic phosphonium ylides with aldehydes[6] have been developed with the aim of gaining conjugated dienes. Generally, linear dienes such as **A** can be prepared easily, whereas synthesis of polysubstituted conjugated dienes, such as **B**, which are a kind of efficient luminescent material processing various potential applications, is more challenging (Figure 1).[7,8] Several common methods to

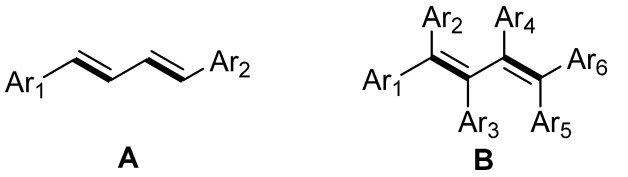
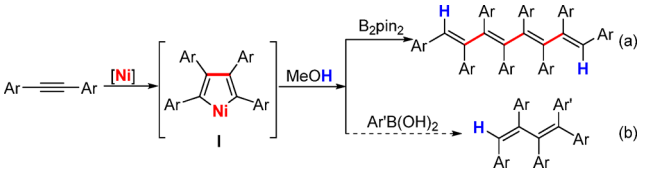


Figure 1. Linear (A) and polysubstituted (B) diene.

prepare the polysubstituted conjugated dienes, such as dienyl rearrangement, alkaline desilylation, allene isomerization, and aryne dimerization, have been reported.[5e,9] However, those approaches almost always involve noble metal catalysts such as Pd,[8c,9e] Au,[9b,c] Ru[5e] which are sensitive to air and moisture, leading to not only difficult operation, but also high cost. Hence, it is desirable and challenging to explore inexpensive and green reagents as well as efficient methods to prepare the polysubstituted conjugated 1,3-dienes.

Ni-catalyzed reductive cross-coupling reactions between two organic electrophiles have emerged as effective methods for C−C bond construction and have drawn increasing attention.[10,11] As active organic electrophiles, alkynes have been used as coupling partners for the Ni-catalyzed cross-coupling reactions.[11] In 2015, our group reported a Ni-catalyzed reductive tetramerization of diarylacetylenes with B_2pin_2 as an environmental and efficient reductant.[12] Mechanistic studies disclosed that the five-membered nickelacycle species was involved as a key intermediate. Based on the resulting mechanistic insight and inspired by previous reports,[9d,12,13] we reasoned that the alkenyl-Ni species would be transferred to the desired polysubstituted dienes with arylboronic acids (Scheme 1).

Scheme 1. Strategies for Synthesis of Polysubstituted Dienes



Herein, we report a Ni-catalyzed tandem dimerization/cross-coupling reaction of diarylacetylenes with arylboronic acids, providing an efficient method to prepare a series of polysubstituted conjugated 1,3-dienes.

At the start of our studies, we used 1,2-diphenylethyne (**1a**) and (4-methoxyphenyl)boronic acid (**2a**) as the model substrates to test the viability of our hypothesis, and the reaction was performed at 120 °C for 12 h. To our delight, the reaction smoothly took place in the presence of $Ni(COD)_2$ and gave the desired product **3aa** in 31% yield (Table 1, entry 1). However, $Ni(COD)_2$ is not only expensive, but also sensitive to air and moisture. Therefore, we wished to use cheap, stable Ni-precursor and environmental B_2pin_2 as catalyst and reductant,

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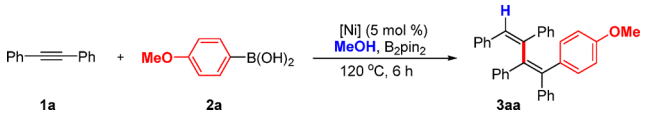
Table 1. Optimization of Reaction Conditions[a]



1a 2a 3aa

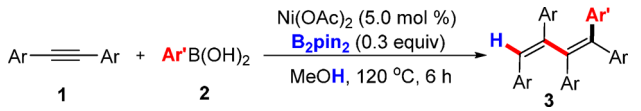
entry	[Ni]	B_2pin_2 (equiv)	solvent	yield (%)
1	$Ni(COD)_2$	0	MeOH	31
2	$NiSO_4$	0.5	MeOH	<5
3	$NiBr_2$	0.5	MeOH	0
4	$Ni(NO_3)_2$	0.5	MeOH	17
5	$Ni(COD)_2$	0.5	MeOH	64
6	$Ni(OAc)_2$	0.5	MeOH	80
7	$Ni(OAc)_2$	0.3	MeOH	78
8	$Ni(OAc)_2$	0.2	MeOH	66
9	$Ni(OAc)_2$	0.1	MeOH	44
10	$Ni(OAc)_2$	0.05	MeOH	18
11		0.3	MeOH	0
12	$Ni(OAc)_2$	0	MeOH	0
13	$Ni(OAc)_2$	0.3	EtOH	40
14	$Ni(OAc)_2$	0.3	^{n}PrOH	30
15	$Ni(OAc)_2$	0.3	^{i}PrOH	<5
16[b]	$Ni(OAc)_2$	0.3	MeOH	81
17[c]	$Ni(OAc)_2$	0.3	MeOH	82
18[d]	$Ni(OAc)_2$	0.3	MeOH	75
19[e]	$Ni(OAc)_2$	0.3	MeOH	81
20[f]	$Ni(OAc)_2$	0.3	MeOH	72

[a]General conditions: **1a** (178 mg, 1.0 mmol), **2a** (182 mg, 1.2 mmol), [Ni] (0.05 mmol, 5 mol %), solvent (2.0 mL), 120 °C, 12 h, isolated yield. [b]120 °C, 18 h. [c]120 °C, 6 h. [d]120 °C, 3 h. [e]150 °C, 6 h. [f]90 °C, 6 h.

respectively, to optimize the reaction conditions. First, screening of the Ni precursors revealed that $Ni(OAc)_2$ gave the best result. When the reaction was catalyzed by $Ni(OAc)_2$ in the presence of 0.5 equiv of B_2pin_2 in MeOH at 120 °C for 12 h, the desired product **3aa** was isolated in 80% yield as a single regio- and stereoisomer (Table 1, entry 6). Several other Ni catalysts, such as $NiSO_4$, $NiBr_2$, and $Ni(NO_3)_2$, showed much lower catalytic activities under the same reaction conditions (Table 1, entries 2–4). It is worth noting that the reaction proceeded smoothly with $Ni(COD)_2$ as catalyst in the presence of B_2pin_2 and afforded **3aa** in 64% yield, which indicated that B_2pin_2 played an important role in this catalytic system. Additionally, the impact of B_2pin_2 over the reactivity of this reaction was investigated, and it was found that the loading of B_2pin_2 was crucial to obtain good yields (Table 1, entries 6–10). Furthermore, when the loading of B_2pin_2 was decreased to 0.1 equiv, a moderate yield was still obtained. It is important to mention that no desired product was observed under identical conditions in the absence of B_2pin_2 or $Ni(OAc)_2$ (Table 1, entries 11 and 12). During the exploration of the ligand effects for this reaction, we discovered that the reaction was virtually stopped when phosphine ligand was introduced into the catalytic system (see the Supporting Information). Further optimization of the reaction conditions revealed that the choice of alcohol solvent was vital to the present catalytic system. Screening of some representative alcohols demonstrated that the most efficient catalysis was furnished in MeOH, affording the desired product in 78% isolated yield (Table 1, entries 7 and 13–15). Finally, the effects of time and temperature on the reaction were also investigated; the best result could be obtained in 82% yield when the reaction was conducted at 120 °C for 6 h (Table 1, entries 16–20). It is worth mentioning that the reaction is highly selective: only (1*Z*,3*Z*)-isomer **3aa** was observed in all cases.

With the optimized reaction conditions identified, the generality of the Ni-catalyzed cross-coupling reaction of diarylacetylenes with arylboronic acids was investigated. The scope of the arylboronic acids was first examined by the adoption of 1,2-diphenylethyne as the coupling partner, and the results are summarized in Table 2. Various arylboronic acids

Table 2. Substrate Scope of Arylboronic Acids and Arynes[a]



1 2 3

entry	Ar	Ar	Ar′	yield (%)
1	Ph	Ph	4-$OCH_3C_6H_4$	**3aa** (82)
2	Ph	Ph	3-$OCH_3C_6H_4$	**3ab** (62)
3	Ph	Ph	4-$OCF_3C_6H_4$	**3ac** (77)
4	Ph	Ph	Ph	**3ad** (70)
5	Ph	Ph	3-$CH_3C_6H_4$	**3ae** (63)
6	Ph	Ph	4-$CH_3C_6H_4$	**3af** (65)
7	Ph	Ph	3,5-$(CH_3)_2C_6H_3$	**3ag** (78)
8	Ph	Ph	4-FC_6H_4	**3ah** (72)
9	Ph	Ph	3-FC_6H_4	**3ai** (65)
10	Ph	Ph	4-ClC_6H_4	**3aj** (60)
11	3-$CH_3C_6H_4$	3-$CH_3C_6H_4$	4-$OCH_3C_6H_4$	**3ba** (63)
12	4-$CH_3C_6H_4$	4-$CH_3C_6H_4$	4-$OCH_3C_6H_4$	**3ca** (74)
13	3-$OCH_3C_6H_4$	3-$OCH_3C_6H_4$	4-$OCH_3C_6H_4$	**3da** (49)
14	4-$OCH_3C_6H_4$	4-$OCH_3C_6H_4$	4-$OCH_3C_6H_4$	**3ea** (62)
15	3,5-$(CH_3)_2C_6H_3$	3,5-$(CH_3)_2C_6H_3$	4-$OCH_3C_6H_4$	**3fa** (58)
16	4-FC_6H_4	4-FC_6H_4	4-$OCH_3C_6H_4$	**3ga** (70)
17[b]	Ph	4-$CH_3C_6H_4$	4-$OCH_3C_6H_4$	**3ha** (72)
18[b]	Ph	CH_3	4-$OCH_3C_6H_4$	**3ia** (31)

[a]General conditions: **1** (1.0 mmol), **2** (1.2 mmol), $Ni(OAc)_2$ (0.05 mmol, 5 mol %), B_2pin_2 (0.3 mmol), MeOH (2.0 mL), 120 °C, 6 h, isolated yield. [b]**3ha** and **3ia** were a mixture of regioisomers.

containing both electron-rich and electron-deficient functional groups on the phenyl ring participated well in this catalytic reaction, affording the desired product in good yields. Typical functional groups such as methyl, methoxy, fluoro, chloro, and trifluoromethoxyl were well tolerated under the reaction conditions. For instance, the substrates with electron-rich groups at the *para*- or *meta*-position on the phenyl ring proceeded smoothly to afford the corresponding products in good yields (Table 2, entries 1–7). Furthermore, the substrates bearing electron-deficient halogen groups, such as F and Cl, on the *meta*- or *para*-position of the phenyl ring were also transformed into the desired products under the standard conditions (Table 2, entries 8–10). Meanwhile, disubstituted arylboronic acid was also a suitable substrate which worked well in the reaction and provided the desired product **3ag** in 78% yield (Table 2, entry 7). Unfortunately, substrates with strong electron-withdrawing groups such as CN, CF_3, and substrates with substituents on the *ortho*-position of the phenyl ring were not applicable in this transformation.

Next, the scope of various diarylacetylenes was examined for this reaction. As shown in Table 2, diarylacetylenes bearing electron-rich functional groups proceeded well to give the corresponding products in good yields. For instance, the substrates with electron-rich functional groups such as CH_3 and

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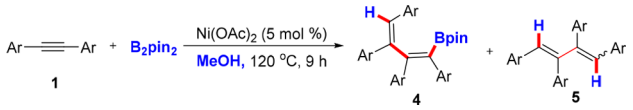
OCH_3 were compatible with the present catalytic system (Table 2, entries 11−15). In addition, the fluorine substituted diarylacetylene **1g** also proceeded smoothly during this cross-coupling reaction (Table 2, entry 16), whereas the substrates with strong electron-withdrawing functional groups including Cl, Br, CF_3 were not applied in this reaction. Meanwhile, the steric hindrance on the benzene ring of diarylacetylenes had a slight effect on the reactivities (Table 2, **3ba** vs **3ca**, **3da** vs **3ea**). Furthermore, the unsymmetrical arynes could also work well under the present catalytic system and gave the desired product **3ha** and **3ia**, but with lower regioselectivities (Table 2, entries 17 and 18). Finally, the structure of **3ae** was confirmed by single-crystal X-ray analysis, and other products were tentatively assigned by analogy.

In addition, we were pleased to find that the product **4a** was obtained in 46% yield in the presence of 1 equiv of B_2pin_2 under similar reaction conditions (Table 3, entry 1). Mean-

Table 3. Ni-Catalyzed Reaction of Diarylacetylenes with B_2pin_2[a]



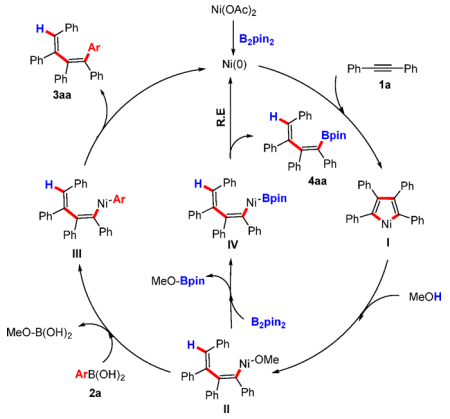
entry	Ar	Ar	yield (%)	
1	Ph	Ph	**4a** (46)	**5a** (40)
2	3-$CH_3C_6H_4$	3-$CH_3C_6H_4$	**4b** (32)	**5b** (30)
3	4-$CH_3C_6H_4$	4-$CH_3C_6H_4$	**4c** (44)	**5c** (37)
4	4-FC_6H_4	4-FC_6H_4	**4d** (38)	**5d** (32)
5[b]	Ph	4-$CH_3C_6H_4$	**4e** (48)	**5e** (36)

[a]General conditions: **1** (1.0 mmol), B_2pin_2 (1.0 mmol), $Ni(OAc)_2$ (0.05 mmol, 5 mol %), MeOH (2.0 mL), 120 °C, 9 h, isolated yield. [b]**4e** and **5e** were the mixture of regioisomers.

while, the diphenylethyne dimerization reaction occurred in this reaction and afforded the corresponding **5a** in 40% yield as a mixture with different stereoisomers. Further optimization of this reaction parameters, such as temperature, time, solvents and ligands, did not significantly improve the selectivity (see the SI). Subsequently, we investigated several simple diarylacetylenes, and the results are shown in Table 3. Obviously, the substrates with functional groups, such as CH_3 and F, could participate the present catalytic system, affording the corresponding products in the range of 32%−44% yields (Table 3, entries 2 and 3). Moreover, the unsymmetrical diarylacetylene also gave the desired product **4e** in 48% yield with lower regioselectivity (Table 3, entry 5). Finally, the structure of **4c** and one isomer of **5a** were further validated by single-crystal X-ray diffraction analysis.

On the basis of the above results and previous reports,[9d,12,13] a plausible reaction pathway for the Ni-catalyzed cross-coupling reaction of diarylacetylenes and arylboronic acids is proposed in Scheme 2. Initially, reduction of Ni(II) precatalyst by B_2pin_2 gave a catalytically active Ni(0) species. Subsequently, the Ni(0) species could undergo oxidative cycloaddition with diphenylethyne (**1a**) to generate five-membered nickelacycle **I**, followed by protonation with MeOH to produce the key intermediate **II**. The crowded environment promotes the Ni-mediated *cis*−*trans* isomerization to form intermediate **II**.[13] The consequent transmetalation of **II** with arylboronic acid (**2a**) took place to form intermediate **III**. Then, reductive elimination of **III** gave the desired product **3aa** and regenerated

Scheme 2. Plausible Reaction Mechanism



the active catalyst for the next catalytic cycle. On the other hand, the intermediate **II** could also react with B_2pin_2 in the absence of arylboronic acid by transmetalation to produce intermediate **IV**, which underwent reductive elimination to afford **4aa**.

In summary, we have successfully developed a new, facile, and efficient method for the preparation of polysubstituted conjugated 1,3-dienes through a Ni-catalyzed tandem dimerization/cross-coupling reaction of diarylacetylenes with arylboronic acids in the presence of B_2pin_2. A series of arynes and arylboronic acids with different substituents proceeded well in the catalytic system, affording a variety of useful conjugated 1,3-dienes. In view of the readily available starting materials and simple operation, this method may become a useful protocol for the synthesis of polysubstituted conjugated 1,3-dienes. Further investigations on application of products and detailed mechanism of this reaction are currently underway in our group.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.7b03398.

Experimental procedures and compound characterization data (PDF)

Accession Codes

CCDC 1582910−1582912 contain the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.

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Notes

The authors declare no competing financial interest.

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