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Cobalt-catalyzed reductive alkynylation to construct C(sp)-C(sp^3) and C(sp)-C(sp^2) bonds

Lei Wan[a], Yizhou Tong[a], Xi Lu[b,*], Yao Fu[a,*]

[a] Hefei National Research Center for Physical Sciences at the Microscale, iChEM, CAS Key Laboratory of Urban Pollutant Conversion, Anhui Province Key Laboratory of Biomass Clean Energy, University of Science and Technology of China, Hefei 230026, China
[b] Key Laboratory of Precision and Intelligent Chemistry, School of Chemistry and Materials Science, University of Science and Technology of China, Hefei 230026, China

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ABSTRACT

Transition-metal-catalyzed cross-electrophile coupling has emerged as a reliable method for constructing carbon–carbon bonds. Herein, we report a general method, cobalt-catalyzed reductive alkynylation, to construct C(sp)-C(sp^3) and C(sp)-C(sp^2) bonds. This presented reaction has a broad substrate scope, enabling the efficient cross-electrophile coupling between alkynyl bromides with alkyl halides and aryl or alkenyl (pseudo)halides. This presented reaction is conducted under mild conditions, tolerating many functional groups, thus suitable for the modification and synthesis of biologically active molecules.

Transition-metal-catalyzed cross-electrophile coupling has emerged as a reliable method for selectively constructing carbon–carbon bonds, featuring simple operation and mild condition characteristics. The type of electrophiles that can be utilized has marched swiftly, comprising C(sp^2)-C(sp^2), C(sp^2)-C(sp^3), C(sp^3)-C(sp^3) and others [1–11]. For example, Gosmini systematically investigated the cobalt-catalyzed cross-electrophile coupling between two different aryl or alkenyl (pseudo)halides [12–17]. Weix made impressive progress on the cross-coupling of aryl or alkenyl (pseudo)halides with alkyl (pseudo)halides using nickel catalysis [18–21]. Gong has contributed to nickel-catalyzed selective C(sp^3)-C(sp^3) formation with two different alkyl (pseudo)halides [22–24]. In contrast, reductive alkynylation was still underexplored to construct C(sp)-C(sp^x) bonds.

The efficient synthesis of alkynes has always been a concern for organic synthesis chemists [25–27]. The alkynyl group could be converted into many other functional groups, such as carbonyl, alkenyl, and alkyl groups [28–35]. The alkynyl group is a commonly found functional group in natural products, drug molecules, organic synthesis intermediates and organic conjugated materials (Fig. 1A). Canonical transition-metal-catalyzed Sonogashira reaction was reliable for constructing C(sp)-C(sp^2) bonds [36–43], while great progresses on alkynylation of unactivated alkyl electrophiles were achieved on recent years [44–46]. Meanwhile, reductive alkynylation using easily synthesized bromoalkynes was an expedient method for constructing C(sp)-C(sp^3) bonds (Fig. 1B) [47–54]. A pioneering study was reported by Weix and co-workers, which demonstrated the nickel-catalyzed reductive decarboxylative alkynylation of redox-active esters with bromoalkynes [18]. Another representative example was realized by Xu and co-workers, describing a dual nickel/photoredox catalytic system to enable the reductive cross-coupling between alkynyl bromides and α-bromo phosphonates [51]. Despite photoredox-catalyzed reductive alkynylation has been reported [55–57], only arylethynyl bromides could be used as the synthon due to the radical addition and then leaving-radical elimination mechanism (Fig. 1C) [58–60]. Therefore, a general method for reductive alkynylation is highly desirable.

Cobalt catalysis showed high efficiency for carbon–carbon bond formation [61–63], reflecting the solid ability to activate alkyl and aryl (pseudo)halides effectively. In continuous of our research interests on cobalt-catalyzed cross-coupling reactions with aliphatic electrophiles [64–67], we report a general method, cobalt-catalyzed reductive alkynylation, to construct C(sp)-C(sp^3) and C(sp)-C(sp^2) bonds (Fig. 1D). This presented reaction has a broad substrate scope and excellent functional group compatibility, enabling the efficient cross-electrophile coupling between alkynyl bromides with alkyl halides and aryl or alkenyl (pseudo)halides. This reaction provides a powerful tool for the late-stage modification of complex biologically active molecules.

* Corresponding authors.
E-mail addresses: luxi@mail.ustc.edu.cn (X. Lu), fuyao@ustc.edu.cn (Y. Fu).

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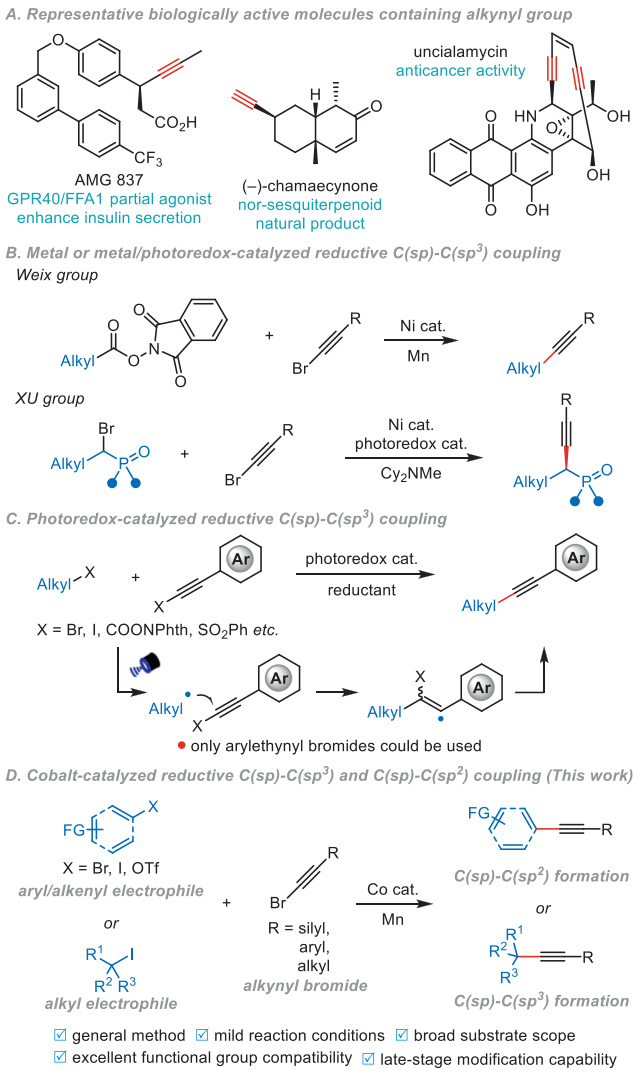
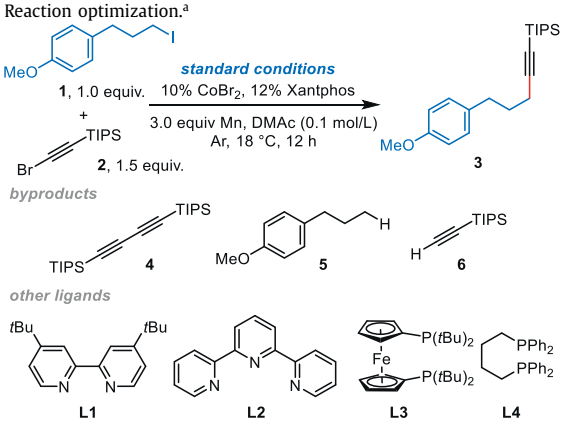


Fig. 1. Methods of reductive C(sp)-C(sp^3) cross-coupling. Tf, trifluoromethanesulfonyl; FG, functional group.

Table 1
Reaction optimization.[a]



Entry	Variants	GC yield (%)[b]
1	None	86 (82 [c])
2	12 mol% **L1** instead of Xantphos	16
3	12 mol% **L2** instead of Xantphos	17
4	12 mol% **L3** instead of Xantphos	7
5	12 mol% **L4** instead of Xantphos	<2
6	**1**, 1.0 equiv., **2**, 1.0 equiv.	74
7	**1**, 1.5 equiv., **2**, 1.0 equiv.	66
8	Alkyl bromide instead of **1**	37
9	DME as solvent	<2
10	THF as solvent	6
11	DMF as solvent	37
12	Zn instead of Mn	18
13	Without $CoBr_2$, Xantphos or Mn	n.d.
14	2.0 mmol scales	66 [c]

Xantphos, 4,5-bis(diphenylphosphino)-9,9-dimethylxanthene; TIPS, triisopropylsilyl; DMAc, *N*,*N*-dimethylacetamide; DME, 1,2-dimethoxyethane; THF, tetrahydrofuran; DMF, *N*,*N*-dimethylformamide; N.D. = no detected.

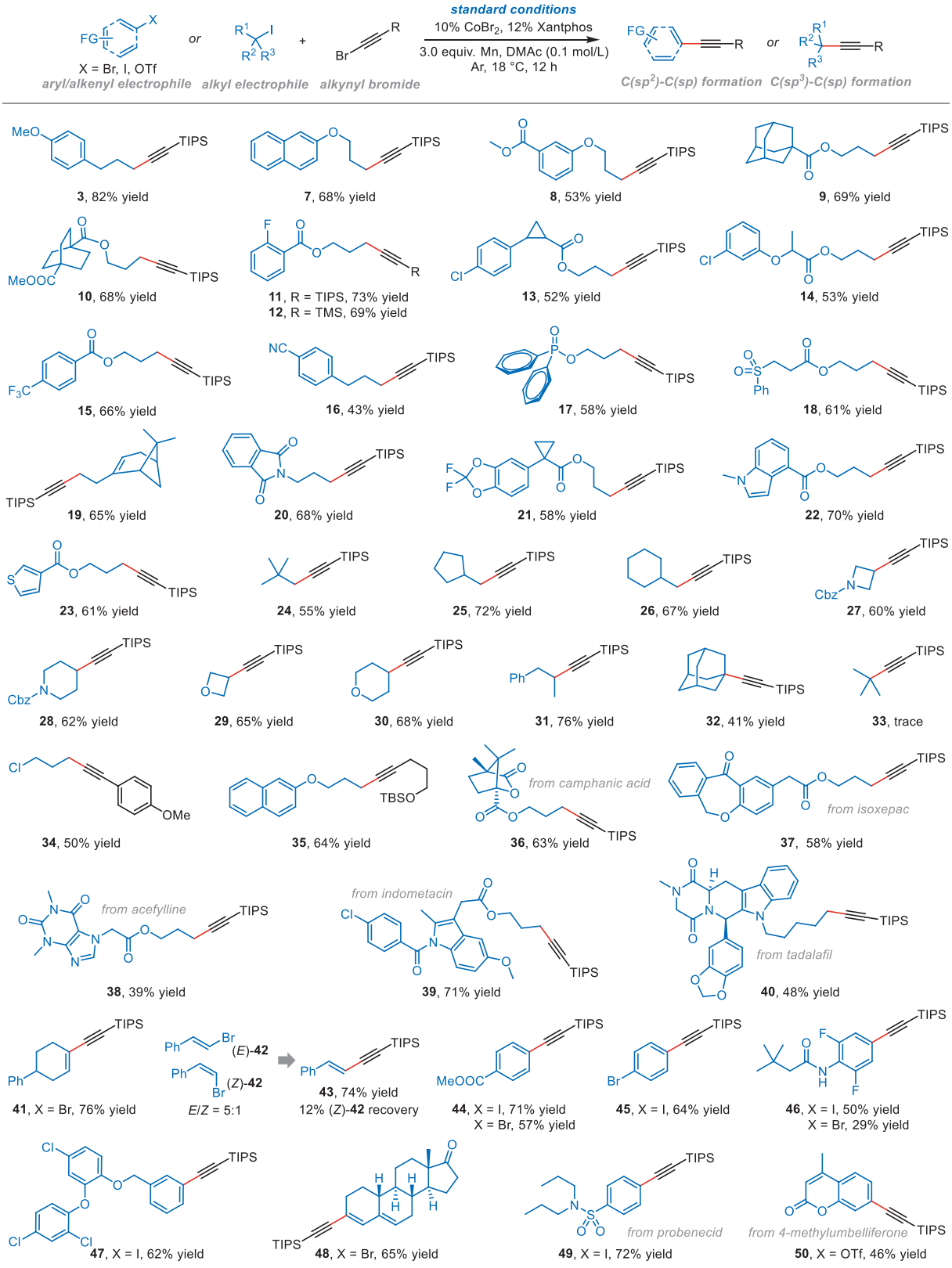
[a] Reactions were conducted under an argon atmosphere. Standard conditions: **1** (1.0 equiv.), **2** (1.5 equiv.), $CoBr_2$ (10 mol%), Xantphos (12 mol%), Mn powder (3.0 equiv.), DMA (0.1 mol/L), 18 °C, 12 h, 0.1 mmol scales.

[b] GC (gas chromatography) yield using triphenylmethane as an internal standard.

[c] Isolated yield.

As shown in Table 1, we began our study by screening the optimal reaction conditions using 1-(3-iodopropyl)-4-methoxybenzene (**1**) and (bromoethynyl)triisopropylsilane (**2**) as model substrates. We determined that the reductive alkynylation reaction underwent smoothly using a combination of 10 mol% $CoBr_2$ and 12 mol% Xantphos as the catalyst, 3.0 equiv. Mn powder as the reductant in DMAc, delivering the cross-coupling product **3** in 86% GC yield and 82% isolated yield (entry 1). The selection of bisphosphine ligand Xantphos was crucial for the success: bipyridine (**L1**), terpyridine (**L2**), and other bidentate bisphosphine ligands (**L3** and **L4**) gave out significantly decreased yields (entries 2-5). Careful analysis of the reaction mixture showed that the main byproducts were alkynyl bromide homo-coupling byproduct **4** and alkyl halide hydrodehalogenation byproduct **5**. Only trace amount of alkynyl bromide hydrodehalogenation byproduct **6** was observed. Changing the ratio of alkyl halide versus alkynyl bromide from 1:1.5 to 1:1 or 1.5:1 would decrease the coupling yields, which might be caused by the higher reactivity of alkynyl bromide substrates (entries 6 and 7). Alkyl bromide has a lower reactivity than the corresponding alkyl iodide (entry 8). Compared with the optimized solvent DMAc, the reaction shut down in low polarity solvents (entries 9 and 10) and delivered a much reduced yield in DMF (entry 11). We also evaluated the performance of Zn powder as the reductant; however, far inferior compared to Mn (entry 12). Control experiments proved that cobalt-catalyst, ligand, and Mn reductant were indispensable (entry 13). Finally, we conducted a larger scale reaction (2.0 mmol scales) under standard conditions (entry 14). The desired product **3** was obtained in a 66% isolated yield (435 mg product).

With the optimal reaction conditions, we sought to investigate the substrate scope of this cobalt-catalyzed reductive alkynylation reaction. As shown in Scheme 1, a broad scope of alkyl, alkenyl and aryl electrophiles was examined, providing moderate-to-good cross-coupling yields. Concerning alkyl electrophiles, both primary (**3**, **7**∼**26**) and cyclic or acyclic secondary (**27**∼**31**) alkyl iodides were suitable substrates. 1-Iodoadamantane (**32**), as an example of a tertiary alkyl halide with certain particularities, was also well suited. However, 2-iodo-2-methylpropane (**33**) could not be converted to the desired product because of β-H elimination side reactions. A variety of functional groups was accommodated under mild reductive cross-coupling reaction conditions, such as ether (**3**, **7**), ester (**8**∼**10**), aryl fluoride (**11**, **12**), aryl chloride (**13**, **14**), trifluoromethyl (**15**), cyano (**16**), phosphonate (**17**), sulfone (**18**) and carbamate (**27**, **28**) groups. This reaction exhibited good compatibility with many synthetically valuable heterocyclic compounds. For example, phthalimide (**20**), difluorobenzodioxole (**21**), indole (**22**) and thiophene (**23**) were compatible during the coupling process. Concerning C(sp^2) electrophiles, alkenyl bromide (**41**, **48**), aryl iodides (**44**∼**47**, **49**), aryl bromides (**44**, **46**), and aryl triflate (**50**) coupling partners all gave rise to the target products; the cou-



Scheme 1. Substrate scope. Standard conditions: aryl/alkenyl/alkyl electrophile (0.2 mmol, 1.0 equiv.), alkynyl bromide (0.3 mmol, 1.5 equiv.), $CoBr_2$ (0.02 mmol, 10 mol%), Xantphos (0.024 mmol, 12 mol%), Mn (0.6 mmol, 3.0 equiv.), DMAc (2.0 mL, 0.1 mol/L), 18 °C, 12 h. Isolated yields. Cbz, carbobenzoxy; TBS, *tert*-butyldimethylsilyl.

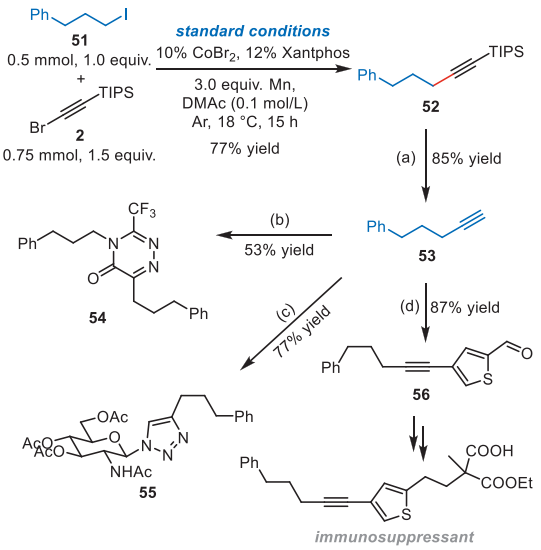


Fig. 2. Synthetic applications. (a) TBAF (0.7 mmol, 1.8 equiv), Et_2O (3.0 mL, 0.13 mol/L); (b) 3-phenylpropyl azide (0.43 mmol, 1.3 equiv.), $(CF_3CO)_2O$ (0.5 mmol, 1.5 equiv.), TFA (0.66 mmol, 2.0 equiv.), CuI (0.017 mmol, 5 mol%), THF (0.5 mL, 0.66 mol/L), 50 °C, 14 h; (c) peracetylated glucosamine azide (0.26 mmol, 0.78 equiv.), Cu_2SO_4 (0.052 mmol, 20 mol%), L-ascorbic sodium salt (0.104 mmol, 40 mol%), *t*-BuOH/THF/H_2O = 1:1:1 (6.0 mL, 0.43 mol/L); (d) 4-bromo-2-thiophenecarboxaldehyde (0.25 mmol, 0.75 equiv.), TFA (3.3 mmol, 10 equiv.), $(Ph_3P)_2PdCl_2$ (0.0083 mmol, 2.5 mol%), CuI (0.017 mmol, 5 mol%), THF (1.0 mL, 0.33 mol/L), 50 °C, 10 h. TBAF, tetrabutylammonium fluoride; TFA, trifluoroacetic acid; Ac, acetyl.

pling efficiency of aryl bromides was a slightly lower than that of the corresponding aryl iodides. We also examined acyclic alkenyl bromides. A mixture of (*E*)-(2-bromovinyl)benzene and (*Z*)-(2-bromovinyl)benzene was selected as a representative example. This mixture was successfully converted to the corresponding (*E*)-enyne product **43**, with the recovery of (*Z*)-alkenyl bromide. Alkynyl bromide containing other substituent groups rather than the silyl group could also be used, comprising aryl (**34**) and alkyl (**35**) substituted alkynyl bromides. The reductive C-C cross-coupling process between two electrophiles showed good chemoselectivity to many reactive sites. For instance, the alkenyl group (**19**) posed no problem without any hydrogenation or hydroalkylation byproducts observed. Without strong bases, the amide group (**46**) possessing a N-H bond was retained without significant *N*-alkylation side reactions. In the example of 1-bromo-4-iodobenzene (**45**), a phenylacetylene derivative carrying an aryl bromide group was obtained [68]. Due to the exemplary functional group tolerance, this reaction has the applicability to introduce alkyne fragments into complex molecules, such as multiply-functionalized drug molecules or natural products. Moderate-to-good yields were attainable in the reactions with derivatives of camphanic acid (**36**), isoxepac (**37**), acefylline (**38**), indomethacin (**39**), tadalafil (**40**) and probenecid (**49**), underscoring the practicability of this method in the modification of biologically active molecules [69,70].

We further demonstrated the utility of this reaction by employing it as a tool for synthesizing functional molecules (Fig. 2). Standard conditions were performed on the conversion of substrate **51**, generating the desired product **52** in a 77% isolated yield. Triisopropylsilyl protecting group could be easily removed under stirring in a TBAF solution, and terminal alkyne **53** was attained. The carbon-carbon triple bond could be transformed into various functional groups. For example, 3-trifluoromethyl-substituted 1,2,4-triazinone (**54**) could be synthesized by the trifluoroacetic anhydride promoted copper-catalyzed interrupted Click reaction [71]. In another example, peracetylated D-glucosamine triazole derivative (**55**) could be prepared through copper-catalyzed azide/alkyne cycloaddition, which might have applications as a low molecular weight gelator in the field of advanced soft materials [72]. Finally, an immunosuppressant intermediate **56** could be obtained *via* canonical Sonogashira reaction.

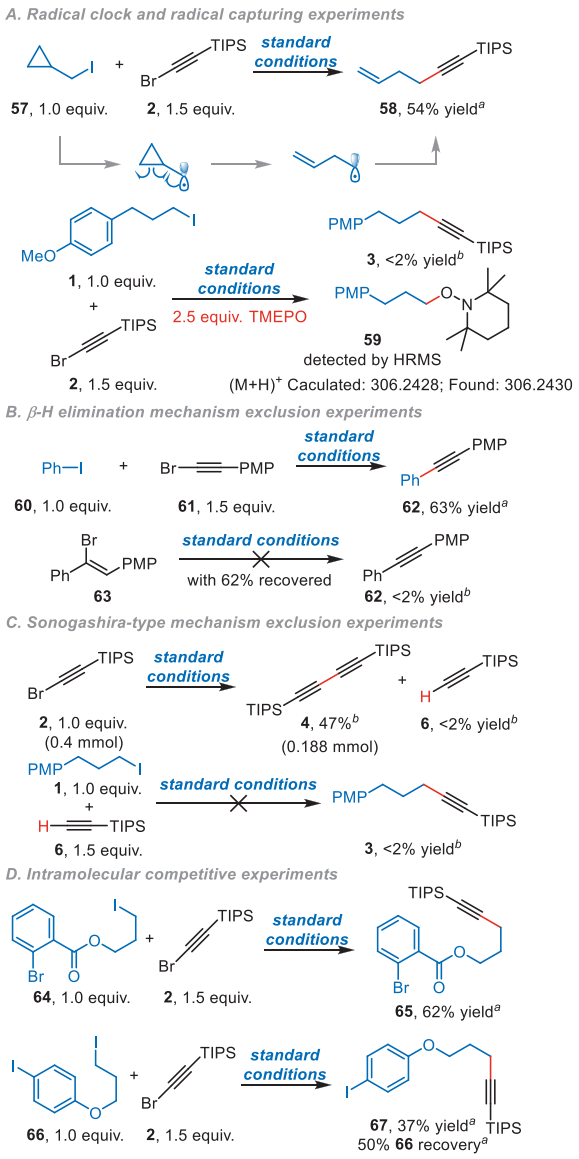


Fig. 3. Mechanistic studies. [a] Isolated yield. 0.2 mmol scales. [b] GC yield using triphenylmethane as an internal standard. TEMPO, 2,2,6,6-tetramethylpiperidinooxy; HRMS, high-resolution mass spectroscopy; PMP, 4-methoxyphenyl.

We conducted mechanistic studies to gain insights into this reductive cross-coupling reaction (Fig. 3). As shown in Fig. 3A, ring-opened product **58** was isolated in the reaction of (iodomethyl)cyclopropane (**57**). The model reaction would be inhibited after adding a radical inhibitor (TEMPO). In addition, HRMS confirmed the formation of radical trapping product **59**. Therefore, alkyl radicals might be the reaction intermediates during the transformation. Iodobenzene (**60**) reacted with 1-(bromoethynyl)-4-methoxybenzene (**61**) smoothly to deliver the product **62** under standard conditions. In comparison, alkenyl bromide **63** could not be converted to the anticipated product **62**, excluding the possibility of a radical addition and subsequent β-H elimination mechanism (Fig. 3B). In the absence of alkyl iodides, homo-coupling of alkynyl bromide dominated, with only a trace amount of alkynyl

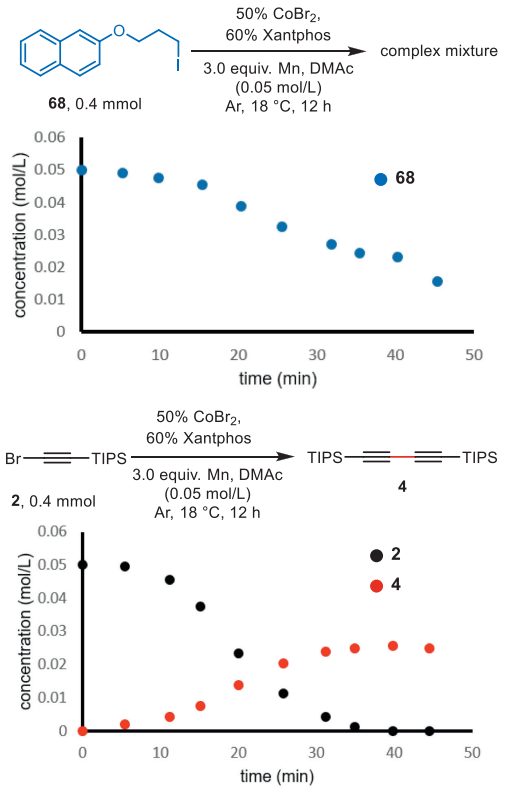


Fig. 4. Conversion rates comparison experiments.

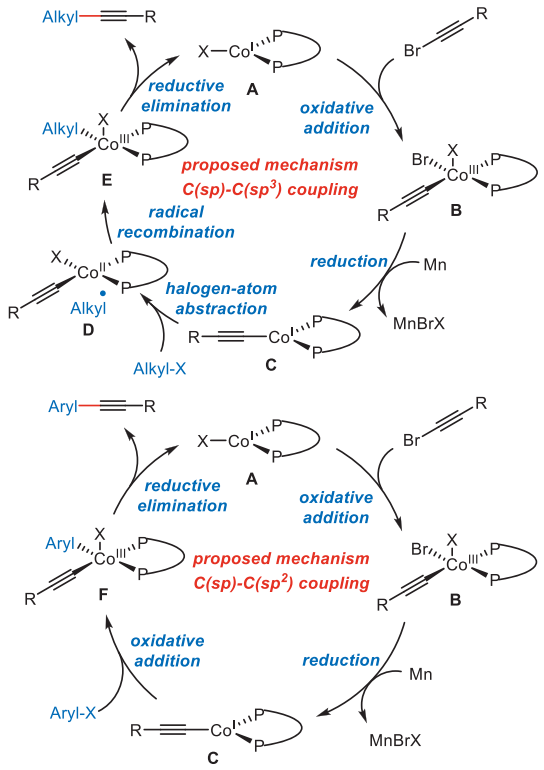


Fig. 5. Proposed mechanism.

bromide hydrodehalogenation byproduct **6** observed. In addition, we performed the standard conditions to terminal alkyne **6** and alkyl iodide **1**; however, we could not detect the production of compound **3**. The above results negated the possibility of an *in-situ* Sonogashira-type reaction (Fig. 3C). In the intramolecular competitive experiments (Fig. 3D), we performed the standard conditions to substrate **64** containing both aryl bromide and alkyl iodide, delivering the C(sp)-C(sp^3) coupling product **65** in a 62% isolated yield. Moreover, in the case of a substrate (**66**) containing both aryl and alkyl iodides, the reductive alkynylation occurred at the alkyl iodide site (**67**). The moderate yield was attributed to a low conversion ratio. The above results indicated that alkyl iodide has higher priority reactivity than aryl electrophiles. Finally, conversion rates comparison experiments in Fig. 4 also demonstrated that alkynyl bromide was hyperactive and might have priority in the oxidative addition step.

Based on relevant literature and our experimental results [14,73-78], we proposed the mechanism in Fig. 5. The initial $Co^{I}X$ (**A**) catalyst could be generated by reducing pre-catalyst $CoBr_2$ by Mn. Alkynylcobalt species (**B**) was formed through the oxidative addition of alkynyl bromide to $Co^{I}X$ (**A**). Then, alkynylcobalt species **B** underwent a reduction process to access another lower valent alkynylcobalt species **C**. The subsequent halogen-atom abstraction and radical recombination (for alkyl electrophiles) or oxidative addition (for aryl or alkenyl electrophiles) provided Co^{III} species (**E** or **F**). Finally, the reductive elimination on Co^{III} species (**E** or **F**) produced the target product and regenerated the initial catalyst **A**. However, other catalytic cycles involving different valence variations and transport processes could not be ruled out.

In summary, a cobalt-catalyzed reductive alkynylation reaction has been developed. This reaction uses relatively inexpensive and low-toxicity cobalt salts as catalysts, easily synthesized alkynyl bromides as alkynylation reagent and Mn powder as reductant. This reaction enables the efficient construct of C(sp)-C(sp^3) and C(sp)-C(sp^2) bonds, thus providing a general method for the synthesis of internal alkyne compounds. The C(sp^3) coupling partner scope covers primary, secondary and tertiary alkyl iodides. Aryl or alkenyl iodides, bromides and triflates are all suitable C(sp^2) coupling partners. This presented reaction is conducted under mild conditions, tolerating many functional groups, thus suitable for the modification and synthesis of biologically active molecules. Mechanism studies rule out radical addition and subsequent β-H elimination and *in-situ* Sonogashira-type reaction mechanisms. A step-by-step oxidative addition and reduction mechanism has been suggested. Developing an asymmetric reductive alkynylation reaction is processed stepwise in our lab.

Declaration of competing interest

The authors declare that they have no competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

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