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Ligand-promoted reductive coupling between aryl iodides and cyclic sulfonium salts by nickel catalysis

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ABSTRACT

Developing applicable methods to forge linkages between sp^3 and sp^2-hydridized carbons is of great significance in drug discovery. We show here a new, Ni-catalyzed reductive cross-coupling reaction that forms Csp^3-Csp^2 bonds from aryl iodides and cyclic sulfonium salts. Notably, Csp^3-Csp^2 bonds can be forged selectively at the iodine-bearing carbon of bromo(iodo)arenes which is usually recognized as a huge challenge under the catalytic reductive cross-coupling (CRCC) conditions. Experimental and computational mechanistic studies support $LNi^{I}Ar$ as an active species, while the untraditional *anti*-Markovnikov selective alkylation of asymmetric sulfonium salts is determined by the oxidative S-substitution of sulfonium salts with $LNi^{I}Ar$. This protocol further expands the range of alkyl electrophiles under the CRCC conditions and provides a new strategy for the construction of Csp^3-Csp^2 bonds.

The degree of saturation of organic molecules has a great influence on their physical properties, such as crystallinity and aqueous solubility [1–4]. Recent research has shown that an increased fraction of sp^3-hydridized carbons, and thus reducing its planarity, is an effective approach to make the compound more drug-like [5–7]. Consequently, there is a continued interest in developing concise and widely applicable methods to forge linkages between sp^3 and sp^2-hydridized carbons in drug discovery [8,9]. Traditionally, transition metal-catalyzed cross-coupling reactions between arylmetallic species and alkyl halides, and transition metal-catalyzed cross-coupling of aryl halides with alkylmetallic reagents, are the two classical methods for constructing Csp^3-Csp^2 bonds (Scheme 1a) [10–19]. Over the past decade, the nickel-catalyzed reductive cross-coupling reactions have emerged as a powerful strategy to forge Csp^3-Csp^2 bonds in the presence of a terminal reductant. In this field, the pioneering work has been reported by Périchon [20], Knochel [21], Gosmini [22], Lipschutz [23], Jacobi von Wangelin [24], Weix, [8,25–28] MacMillan, [29,30] Buchwald, [31] Molander [32,33], Gong [34,35], and others [36–41]. Notably, in this respect, the alkylation reagents mainly focus on alkyl halides [42–45], alkyl trifluoroborates [46,47], ammonium alkylsilicates [48,49], alkyl Katritzky salts [33], *N*-(acyloxy)phthalimides [50], alkyl acids [51,52], and alkyl sulfinate salts [53], and so on [54,55] (Scheme 1b). Although great achievements have been made in this field, the stoichiometric organometallic reagents, harsh reaction conditions, and unavailability of starting materials restricted their wide applications in organic synthesis. Therefore, it is still highly desirable to develop more robust methods to access Csp^3-Csp^2 bonds directly from stable and readily available starting materials under catalytic reductive cross-coupling (CRCC) conditions.

Sulfonium salts are an important class of sulfur-containing reagents, which have long been used as precursors to sulfur ylides and play a vital role in the construction of cyclic compounds as one-carbon building blocks [56]. On the other hand, the nucleophilic Markovnikov ring opening reactions of sulfonium salts with nucleophiles have also been sporadically reported (Scheme 1c) [57,58]. In recent years using sulfonium salts as radical precursors for constructing C–C bonds and C–heteroatom bonds has been well developed [59–72]. However, the reductive coupling reactions using sulfonium salts as electrophiles are rarely reported. In 2021, Ritter and co-workers initially reported an elegant palladium-

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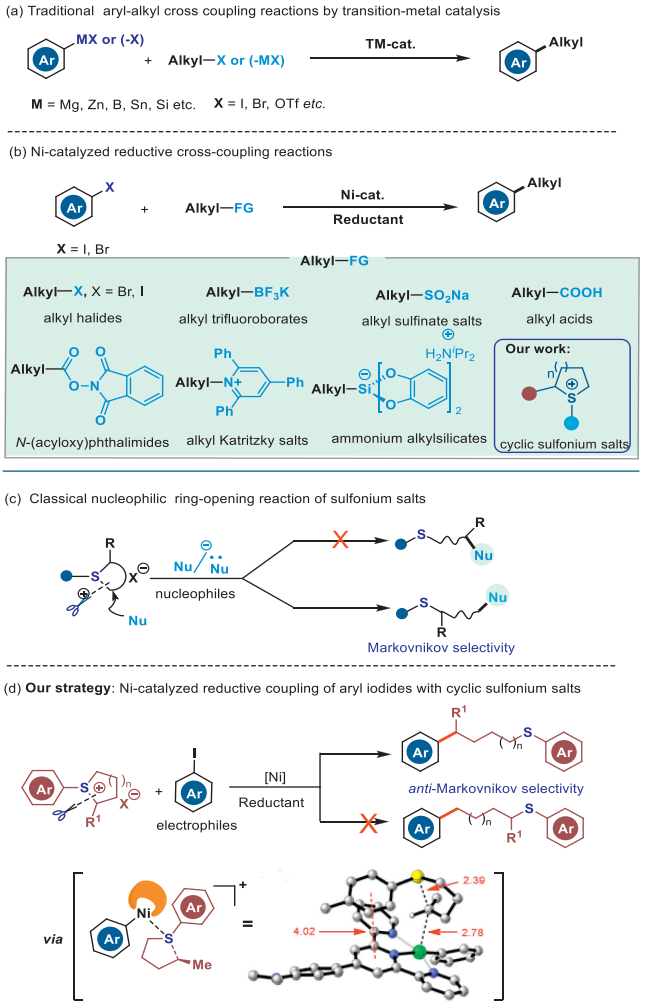
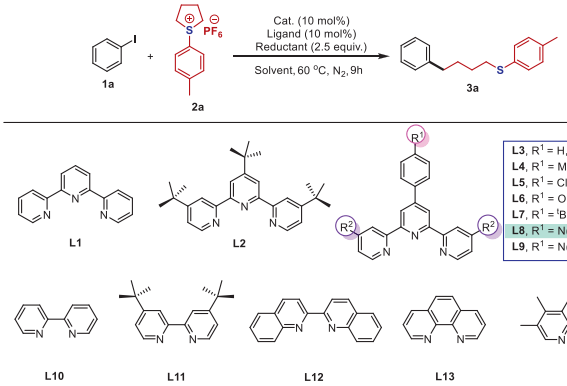


Scheme 1. (a) Traditional aryl-alkyl cross-coupling reactions by transition-metal catalysis. (b) Ni-catalyzed reductive cross-coupling reactions. (c) Classical nucleophilic ring-opening reaction of sulfonium salts. (d) Invention of a new Ni-catalyzed reductive coupling of aryl iodides with cyclic sulfonium salts.

catalyzed reductive electrophile cross-coupling of aryl thianthrenium salts with alkyl iodides for the construction of $Csp^3–Csp^2$ bonds under mild conditions [73,74]. However, developing novel reductive coupling modes of sulfonium salts as electrophiles still remains a huge challenge.

The aryl alkyl thioether skeletons are ubiquitous in natural products, pharmaceuticals, and biologically active molecules [75–77]. Therefore, the development of new reaction modes of sulfonium salts and the introduction of alkylthio fragments into organic molecules is of great significance in the field of drug development. With the insight obtained from our recent experimental results in the radical type ring-opening reactions with sulfonium salts [78,79], we envision that $Csp^3–Csp^2$ bonds could be formed starting from readily available aryl iodides and cyclic alkyl(aryl)sulfonium salts under CRCC conditions. We speculate that, during the C–S bond cleavage of alkyl sulfonium salts, the combination of bulky tridentate ligand and radical-containing nickel complexes possibly obstacles the coordination of alkyl moiety to nickel center and affords an alkyl radical species. In this scenario, different to the nucleophilic ring opening, *anti*-Markovnikov selectivity might be realized (Scheme 1d). To the best of our knowledge, the *anti*-Markovnikov selective ring-opening reaction of cyclic alkyl(aryl)sulfonium salts under CRCC conditions has never been documented to date, and how to avoid the $Csp^2–S$ bond cleavage under Ni catalysis will be a huge challenge for our protocol [59].

Table 1
Screening for the optimal conditions.[a]

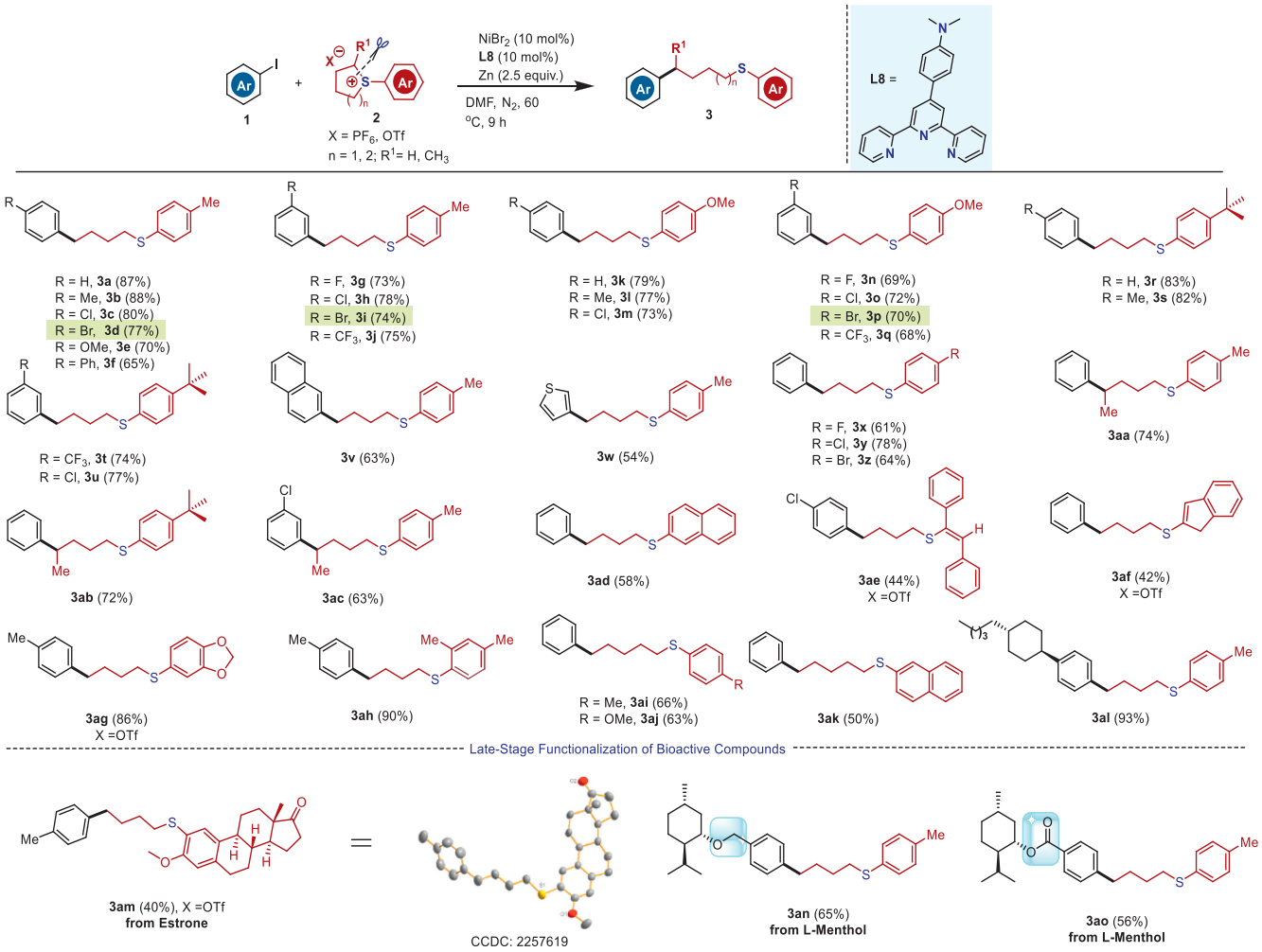


Entry	Catalyst	Ligand	Solvent	Reductant	Yield (%)[b]
1	$NiBr_2$	**L1**	DMF	Zn	75
2	$NiCl_2$(dppe)	**L1**	DMF	Zn	71
3	$NiCl_2$	**L1**	DMF	Zn	69
4	NiI_2	**L1**	DMF	Zn	66
5	$Ni(OAc)_2·4H_2O$	**L1**	DMF	Zn	40
6	$NiBr_2$	**L2-L9**	DMF	Zn	40–87
7	$NiBr_2$	**L10-L14**	DMF	Zn	Trace-23
8	$NiBr_2$	**L8**	NMP	Zn	45
9	$NiBr_2$	**L8**	DMSO	Zn	67
10	$NiBr_2$	**L8**	Toluene	Zn	Trace
11	$NiBr_2$	**L8**	THF	Zn	Trace
12	$NiBr_2$	**L8**	DMF	Mn	50
13	$NiBr_2$	**L8**	DMF	Mg	35
14	$NiBr_2$	**L8**	DMF	DEMS	0
15	$NiBr_2$	**L8**	DMF	TES	0
16	$NiBr_2$	**L8**	DMF	Zn	40[c]
17	$NiBr_2$	**L8**	DMF	Zn	87[d]
18	$NiBr_2$	**L8**	DMF	Zn	86[e]

[a] Reaction conditions: **1a** (0.2 mmol), **2a** (0.3 mmol), catalyst (10 mol%), ligand (10 mol%), reductant (2.5 equiv.), solvent (2 mL), N_2, 60 °C, 9 h. DEMS = Diethoxymethylsilane. TES = Triethoxysilane.
[b] Isolated yield.
[c] At 50 °C.
[d] At 70 °C.
[e] At 80 °C.

To validate our hypothesis, our investigation of this CRCC transformation was performed between iodobenzene **1a** and sulfonium salt **2a**. As shown in Table 1, five nickel catalysts such as $NiBr_2$, $NiCl_2$(dppe), $NiCl_2$, NiI_2, and $Ni(OAc)_2·H_2O$ were examined in DMF in the presence of 2.5 equiv. of Zn powder at 60 °C (entries 1–5), and $NiBr_2$ provided the highest yield 75% (entry 1). Next, various ligands including tridentate ligands (**L1**–**L9**) and bidentate ligands (**L10**–**L14**) were investigated, and 4′-(*p*-dimethylaminophenyl)–2,2′:6′,2″-terpyridine **L8** showed the highest activity, affording the desired product **3a** in 87% yield with the corresponding homocoupling product **3b'** in a trace amount (entries 6 and 7, see Supporting information for details). Then, we tested various solvents including DMF, NMP, DMSO, toluene, THF, and DMF was superior to the others (compare entries 6, 8–11). Furthermore, other reductants, including Mn, Mg, DEMS and TES were examined and resulted in lower productivity than Zn (entries 12–16). Finally, controlled experiments show that reducing the temperature can lead to a decrease in the yield of the target product, and accelerate the temperature cannot improve the yield (entrie 16–18).

With the optimized reaction conditions in hand, we next began to investigate the scope and generality of the nickel-catalyzed reductive alkylation of aryl iodides with cyclic alkyl(aryl)sulfonium salts, and the results are listed in Scheme 2. We were pleased



Scheme 2. The substrate scope. Reaction conditions: **1** (0.2 mmol), **2** (0.3 mmol), $NiBr_2$ (10 mol%), **L8** (10 mol%), Zn (2.5 equiv.), DMF (2 mL), N_2, 60 °C, 9 h. Isolated yield. Unless otherwise noted, X = PF_6. ORTEP drawing at 50% ellipsoid probability.

to find that diverse aryl iodides **1** containing either electron-rich or electron-poor groups smoothly reacted with sulfonium salts **2** affording the corresponding sulfur-containing alkylation products in moderate to good yields. The structure of the alkylation products was determined by the single crystal X-ray diffraction study of compound **3am**. It is worth mentioning that a range of bromo(iodo)arenes bearing iodo and bromo groups in the *meta*- and *para*-positions reacted in good yields to afford the desired products with excellent chemoselectivity (**3d**, **3i** and **3p**). A heterocycle iodide such as 3-iodothiophene delivered the product **3w** in 54% yield. Of note that this reductive cross-coupling transformation has a different regioselectivity from the nucleophilic ring-opening reaction, the alkylation products were obtained in an *anti*-Markovnikov pathway at a higher steric hindrance site (**3aa**–**3ac**). Experimental and computational mechanistic studies support $LNi^{I}Ar$ as an active species, while the untraditional *anti*-Markovnikov selective alkylation of asymmetric sulfonium salts is determined by the oxidative S-substitution of sulfonium salts with $LNi^{I}Ar$ (Fig. 1). Further exploration on the scopes and limits of the synthetic application are in progress. To our delight, the styrene-substituted sulfonium salt was also suitable for the reaction affording **3ae** in 44% yield. In addition, six-membered cyclosulfonium salts could also efficiently participate in the catalytic system delivering the desired products in good yields (**3ai**–**3ak**). Biologically relevant molecules such as estrone and L-menthol were well functionalized with moderate yields (**3am**–**3ao**) which demonstrated the practical application of the reaction. Unfortunately, we found that treatment of iodobenzene **1a** with thianthrenium salts or acyclic aryl sulfonium salts under the standard reaction conditions delivered no desired products (see Supporting information for details). Further exploration on the scopes and limits of the synthetic application are in progress. A lot of functional groups were tolerated in the present transformation, such as halogen substituents, methyl, and methoxy, leaving ample room for further modifications.

We explored the synthetic utility of this reductive cross-coupling protocol. The gram-scale reaction was performed between **1a** and **2a**, and the reaction gave **3a** in 85% isolated yield without loss of reactive efficiency (Scheme 3a). In addition, the sulfoxide product **4**, sulfone product **5** and analogues sulfoximine product **6** were efficiently synthesized under different oxidative conditions (Schemes 3b-d). Thus, this simple Ni-catalyzed reductive cross-coupling protocol could be served as an alternative method for the synthesis of sulfur-containing organic derivatives.

To gain insight into the mechanism of this reaction, several control experiments were conducted. Firstly, the reaction did not proceed in the absence of Zn (Scheme 4a). Furthermore, when the reaction was performed using $Ni(cod)_2$ as the catalyst, no alkylation product **3b** was detected, but the alkyl dimer **3b'** was obtained as the major product (Scheme 4b). These preliminary re-

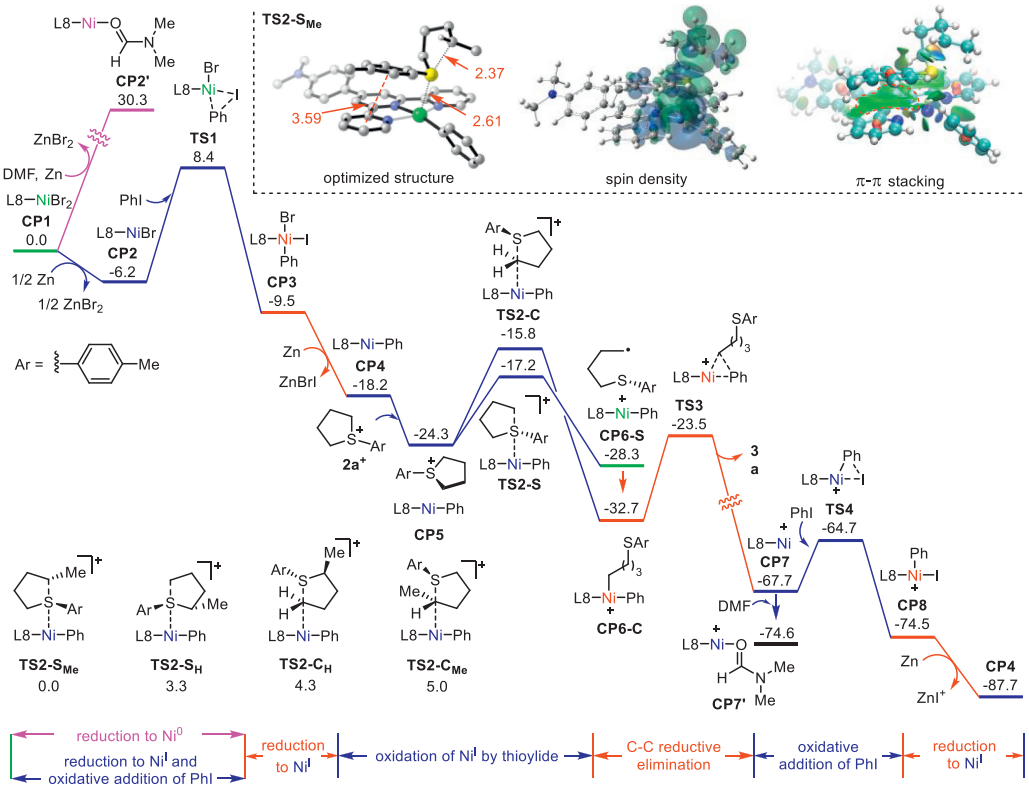
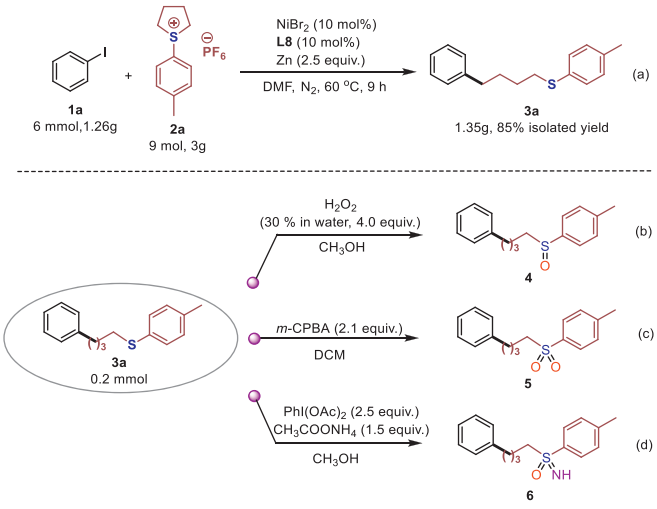
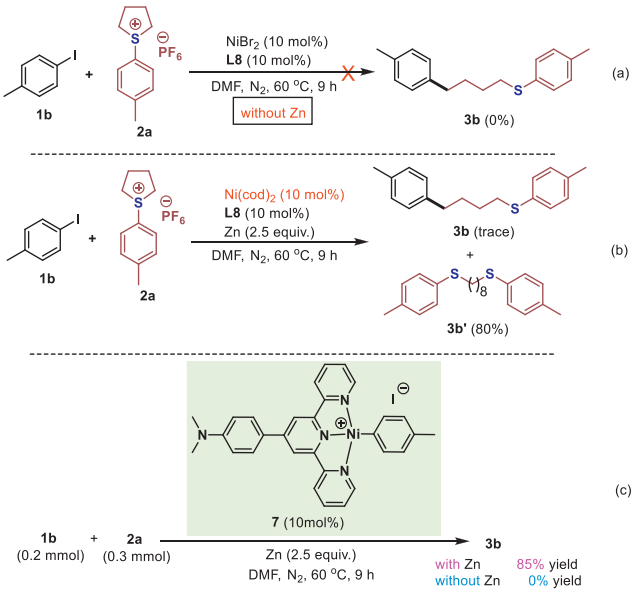


Fig. 1. Calculated solution-phase Gibbs free energy profile of Ni-catalyzed cross-coupling of PhI and sulfonium salt **2a** (in kcal/mol). Optimized structure of **TS2-S$_{Me}$** was provided with most of hydrogen atoms omitted for clarity. Bond distances and the minimum distance between the geometric centers of the benzene ring on S atom and the pyridine rings of ligands were given in angstrom (red). Mulliken spin density and plot of noncovalent interaction were made at the isosurface value of 0.0004 and 0.03, respectively. Pink, blue, green and red were used to denote Ni^0, Ni^I, Ni^{II} and Ni^{III}, respectively.



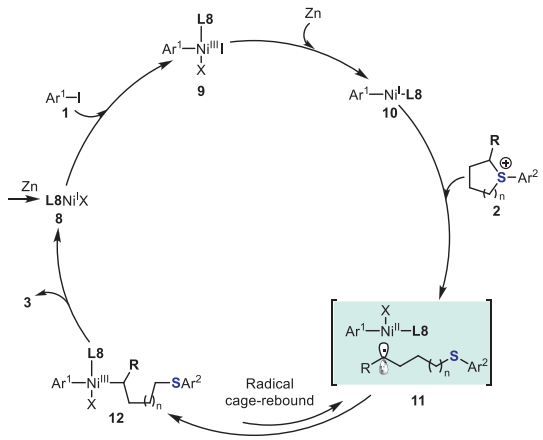
Scheme 3. Synthetic applications.



Scheme 4. Investigations of the reaction mechanism.

sults suggest that the transformation is not initiated by nickel(II) or nickel(0) catalyst, it could occur with Ni(I) species, which is in accordance with the previous reports [20]. To further elucidate the reaction pathway, a tridentate Ni(II) complex **7** was obtained by reaction of Ni(cod)$_2$ with methyl 4-iodotoluene in the presence of **L8**. No desired **3b** was detected for the reactions of **1b** with **2a** using **7** as the precatalyst, in the absence of **Zn**. While, with Zn, **3b** was obtained in 85% yield (Scheme 4c). We reason that it is likely that the complex **7** was reduced by Zn to **L8**Ni(I)-Ar, which reacts with **2a** to give Ar-Ni(III)alkyl prior to the reductive elimination delivering **3b**.

Furthermore, the mechanistic details were investigated with the aid of density functional theory (DFT) calculations at the level of B3LYP-D3/SMD (solvent = DMF)/SDD&def2-TZVP//B3LYP-D3/SMD (solvent = DMF)/LANL2DZ+*p*&6–31G(d) (see Supporting information for more computational details). As shown in Fig. 1, reduction of NiBr$_2$ complex **CP1** to Ni^0 complex **CP2′** with Zn is highly endergonic by 34.8 kcal/mol, being significantly less favored than the reduction to Ni(I) complex **CP2**. This result is consistent with



Scheme 5. Possible reaction pathway.

our control experiment which excluded a Ni^0-Ni^{II} catalytic process (Scheme 4b). Very recently, the experimental mechanistic study from Li also indicates that the reduction of NiI_2 precatalyst by Zn generates Ni^I species rather than Ni^0 species [20]. From **CP2**, oxidative addition of PhI goes through **TS1** to afford Ni^{III} complex **CP3** which is then again reduced to Ni^I−pH complex **CP4** by Zn. Thereafter, sulfonium cation $\mathbf{2a}^+$ can oxidize the Ni^I species *via* C-substitution transition state **TS2-C** or S-substitution transition state **TS2-S**. For $\mathbf{2a}^+$, **TS2-S** is slightly favored over **TS2-C** by 1.4 kcal/mol and generates a Ni^{II} complex **CP6-S** that contains an alkyl radical (Fig. S1 in Supporting information for the plot of Mulliken spin density). The bonding of the alkyl radical to Ni^{II} center forms the more stable Ni^{III} complex **CP6-C**, from which $C(sp^3)$−$C(sp^2)$ reductive elimination can occur *via* **TS3**. The resulting Ni^I complex **CP7** undergoes oxidative addition with another PhI *via* **TS4**, followed by reduction with Zn to **CP4** to restart the catalytic cycle. According to the calculated energy profile, the oxidation of Ni^I−pH complex **CP4** determines the regioselectivity for asymmetric sulfonium salts (Scheme 2, **3aa**−**3ac**). Taking the starting material of product **3aa** as the model substrate, the S-substitution transition states (**TS2-S_{Me}** and **TS2-S_H**) also have lower energies than the C-substitution transition states (**TS2-C_{Me}** and **TS2-C_H**). This phenomenon possibly results from the fact that the benzene ring on the S atom is closer to the pyridine rings of ligand **L8** in **TS2-S_{Me}** and **TS2-S_H** as reflected by the minimum distance of their geometric centers (Fig. 1 and Fig. S1). Thus a stronger π-π stacking effect is expected in **TS2-S_{Me}** and **TS2-S_H** (Fig. 1 and Fig. S1 for plots of noncovalent interaction). On the other hand, **TS2-S_{Me}** is more stable than **TS2-S_H** by 3.3 kcal/mol, supporting the superiority of C−S bond cleavage at the alkyl-substituted C-terminal. It was found that remarkable Mulliken spin density spreads on the cleaving carbon in **TS2-S_{Me}** and **TS2-S_H** with the absolute values of 0.495 and 0.561, respectively, contributing to the lower energy of **TS2-S_{Me}** that owns a more stable carbon radical.

According to the mechanistic studies, a proposed catalytic cycle is proposed in Scheme 5. Firstly, the reduction of **L8**Ni^{II} precatalyst by Zn generates Ni^I species **8**. Then, oxidative addition of aryl iodides **1** to Ni^I gives a Ni^{III} species **9** that is subjected to reduction by stoichiometric Zn leading to Ar^1−Ni^I−**L8** complex **10**. Subsequently, the complex **10** acts as a reducing agent and delivers an electron to **2**, thus giving a formal Ar^1−Ni^{III}−$C(sp^3)$ species **12** that undergoes an in-cage radical/Ni rebound **11** [80]. Finally, reductive elimination of the species **11** affords the desired cross-coupling product **3** while regenerating the Ni^I catalyst to restart the next catalytic cycle.

In conclusion, we have successfully developed an efficient method for Csp^3−Csp^2 bonds formation based on a Ni-catalyzed reductive cross-coupling protocol using cyclic sulfonium salts as alkylation reagents. This reaction tolerates a broad scope of functional groups and provides the corresponding aryl alkyl thioethers in moderate to good yields. This method further expands the range of alkyl electrophiles under the CRCC conditions and provides a new strategy for the construction of Csp^3−Csp^2 bonds. This protocol also provides a new avenue to aryl alkyl thioethers using cyclic sulfonium salts as alkylthio fragments surrogates. Importantly, the utility of this method was proven by late-stage functionalization of bioactive molecules.

Declaration of competing interest

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

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2024.109732.

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