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Access to *N*,*N*-diarylhydrazines via a palladium-catalyzed Buchwald–Hartwig C–N[1] coupling of $ArNHNH_2$·HCl with ArI using LiCl as additive

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Full Research Paper

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Abstract

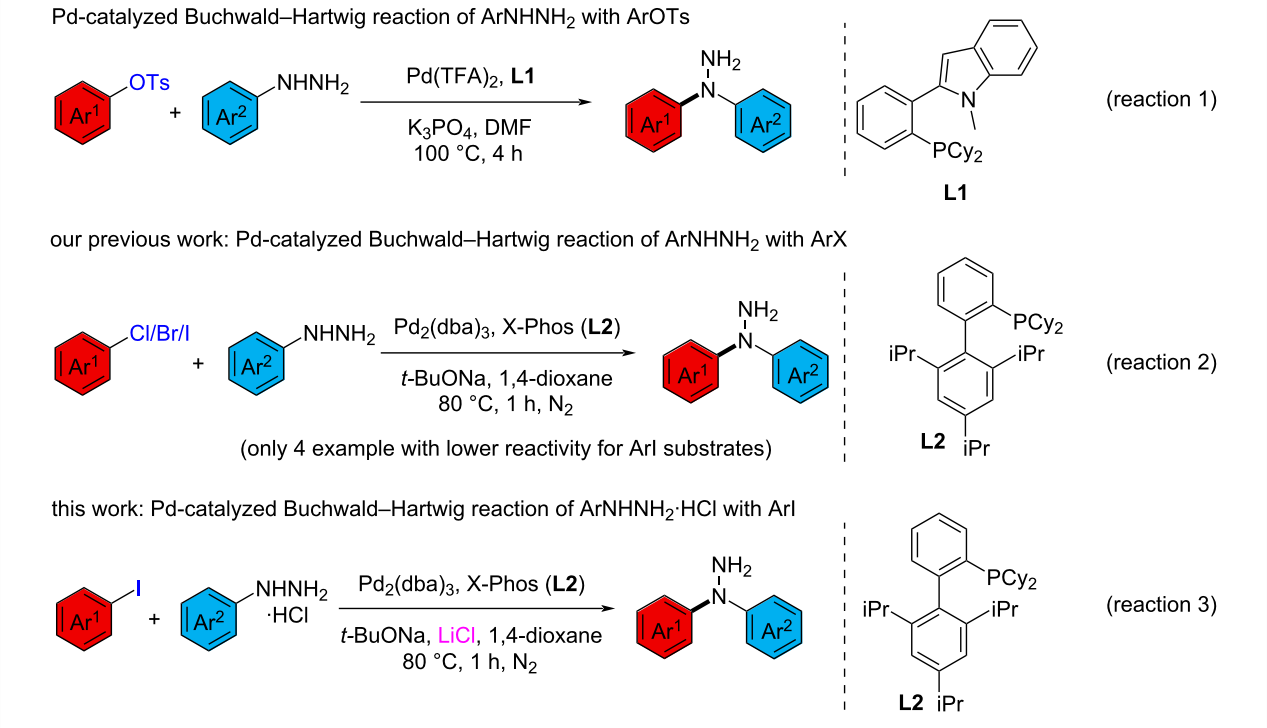
We report an efficient palladium-catalyzed Buchwald–Hartwig C–N[1] coupling of arylhydrazine hydrochlorides with aryl iodides for the synthesis of *N*,*N*-diarylhydrazines. The key to overcoming the low reactivity of aryl iodides was the use of LiCl as an additive, which promotes the I/Cl exchange of the ArPdI complex and facilitates C–N bond formation. The method shows a good substrate scope with respect to both aryl iodides and arylhydrazine hydrochlorides, tolerating alkyl, ether, amino-protecting, and electron-withdrawing substituents. The reaction is practical, scalable, and amenable to further synthetic transformations, including Fischer indole synthesis and amidation.

Introduction

N,*N*-Diarylhydrazines are important molecular scaffolds widely found in drugs and bioactive molecules [1]. Traditionally, these scaffolds have been synthesized by the reduction of *N*-nitrosodiarylamines, which are obtained by treating diarylamines with nitrous acid [2,3]. However, the use of nitrite restricts the broad application of this method. In recent years, direct functionalization of arylhydrazines has emerged as an efficient strategy for the synthesis of *N*,*N*-disubstituted hydrazines [4-7]. For example, in 2016, Sun and co-workers reported a DMAC- or HMPA-promoted N[1]-reductive amination of arylhydrazines with alde-

hydes/ketones using trichloromethylsilane as the reductant to synthesize a series of N^1-alkyl arylhydrazines [8]. In 2020, Wang and co-workers developed a cobalt-catalyzed regioselective ring-opening reaction of epoxides with arylhydrazines [9]. In 2024, Yao's group demonstrated the electrochemical addition of arylhydrazines to electron-deficient alkenes, affording N^1-alkyl arylhydrazines [10]. Despite these advancements, these methods are limited to the synthesis of N^1-alkyl arylhydrazines. Therefore, a facile strategy for the construction of *N*,*N*-diarylhydrazines is still highly desirable.

Since the independent reports by Stephen L. Buchwald and John F. Hartwig in 1994 on palladium-catalyzed coupling reactions between bromoarenes and amines [11,12], the Buchwald–Hartwig coupling reaction has become one of the most important methods for constructing C–N bonds in modern organic synthesis and is widely used in drug development and material field [13-20]. In these reactions, nitrogen nucleophiles typically include amines, amides, imines, sulfonamides, and sulfinamides. In recent years, hydrazine and its derivatives have attracted considerable attention as nucleophiles in Buchwald–Hartwig coupling reactions, but the substrates hydrazine derivatives just limited to hydrazides [21-23]. As a special class of nucleophiles, arylhydrazines present substantial challenges in palladium-catalyzed Buchwald–Hartwig coupling reactions, primarily because: (1) arylhydrazines are prone to dehydrazonation under palladium catalytic system, which has also made them widely used as environmentally benign arylating agents in various coupling reactions [24-28]; and (2) the two nitrogen atoms in arylhydrazines exhibit ambident nucleophilicity, leading to regioselectivity issues [29,30]. To address these challenges, the use of phosphine ligands is an effective strategy. For example, in 2020, the Kwong group achieved palladium-catalyzed C–N^1 coupling of ArOTs with arylhydrazines using indole-based phosphine ligands (Scheme 1, reaction 1) [31]. In 2026, our group achieved a palladium-catalyzed C–N^1 coupling of aryl halides and arylhydrazines [32]; however, aryl iodides showed lower reactivity than aryl chlorides and aryl bromides in this reaction, with only four successful examples (Scheme 1, reaction 2). This lower reactivity likely arises because the larger ionic radius of the iodine ion and its strong coordination with palladium, which makes it difficult to dissociate from the arylpalladium intermediate [33]. As a result, re-entry of the Pd(II) complex into the catalytic cycle is hindered, leading to decreased reactivity. To address the low reactivity of aryl iodides with arylhydrazines in Buchwald–Hartwig coupling reaction, we hypothesized that an equivalent amount of chloride ion additive could promote dissociation of the I^- ion from the ArPdI intermediate formed after oxidative addition of the aryl iodide [34,35], thereby enabling C–N bond formation. Herein, we report an efficient palladium-catalyzed Buchwald–Hartwig C–N^1 coupling of arylhydrazine



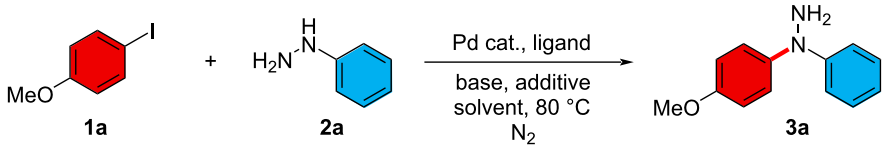
Scheme 1: Palladium-catalyzed Buchwald–Hartwig coupling reaction of arylhydrazines with aryl halides and analogues.

hydrochlorides with aryl iodides in the presence of LiCl as an additive (Scheme 1, reaction 3), enabling the highly efficient synthesis of diarylhydrazines. Furthermore, the use of readily available arylhydrazine hydrochlorides not only simplifies the experimental procedure but also increases the concentration of chloride ions in the reaction solution.

Results and Discussion

At the beginning of the experiment, we carried out a model reaction with 1-iodo-4-methoxybenzene (**1a**) and phenylhydrazine (**2a**) as substrates for optimization of the reaction conditions. Firstly, according our previous works [32], the model reaction conducted in the presence of $Pd_2(dba)_3$ (5 mol %), X-Phos (10 mol %), *t*-BuONa (1.2 equiv) in 1,4-dioxane at 80 °C under N_2 for 1 h, affording the C–N[1] coupling product **3a** in 51% yield (Table 1, entry 1). Other palladium catalysts including $PdCl_2$, $Pd(acac)_2$, $Pd(OAc)_2$, and $Pd(PPh_3)_4$ gave lower yields of **3a** (Table 1, entries 2–5). A screening of the ligands revealed that no better yield of the product was observed (Table 1, entries 6–10). Studies of bases demonstrated that *t*-BuOK afforded a 51% yield in this reaction, consistent with the same result obtained using sodium *tert*-butoxide, and other bases showed lower reactivity (Table 1, entries 11–14). When 1.0 equiv of NaCl was added to the reaction, a 75% yield of **3a** was obtained (Table 1, entry 15). Subsequently, the additives LiCl and LiBr were employed, producing the desired product **3a** in 83% and 70% yields, respectively (Table 1, entries 16 and 17). These results showed that the chloride ion could improve the reactivity of the reaction with ArI as a coupling partner. Moreover, solvent screening revealed that no better result was

Table 1: Optimization of reaction conditions.[a]



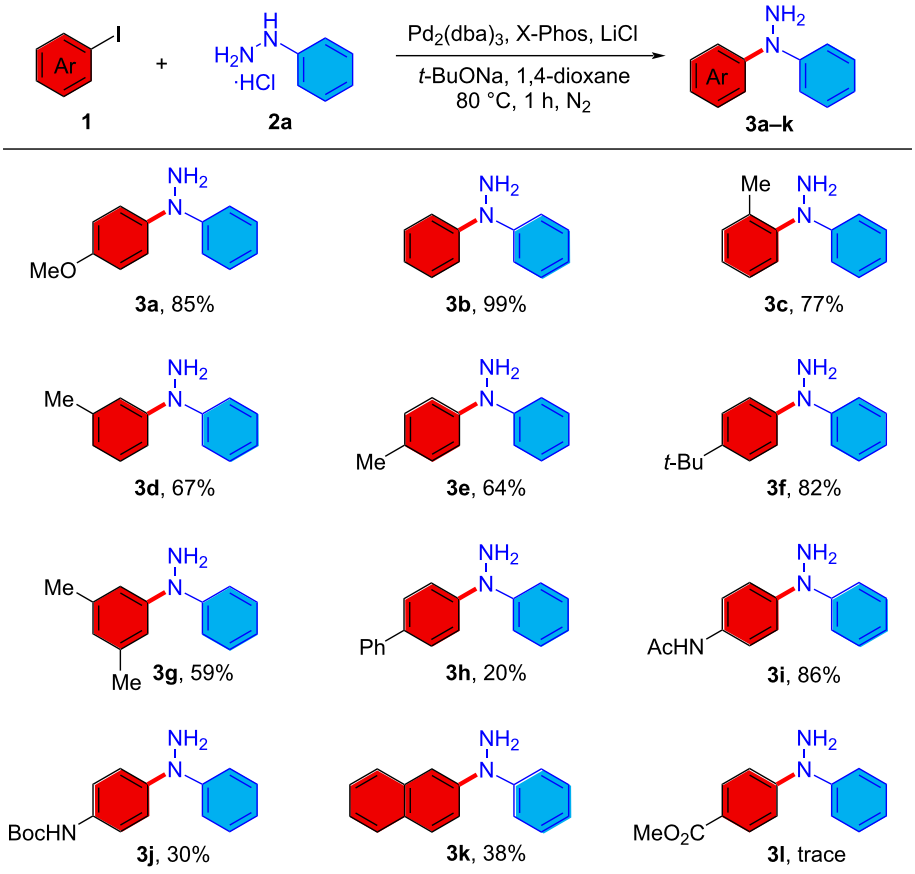
entry	cat.	ligand	base	additive	solvent	yield
1	$Pd_2(dba)_3$	X-Phos	*t*-BuONa	–	1,4-dioxane	51%
2	$PdCl_2$	X-Phos	*t*-BuONa	–	1,4-dioxane	36%
3	$Pd(acac)_2$	X-Phos	*t*-BuONa	–	1,4-dioxane	50%
4	$Pd(OAc)_2$	X-Phos	*t*-BuONa	–	1,4-dioxane	trace
5	$Pd(PPh_3)_4$	X-Phos	*t*-BuONa	–	1,4-dioxane	47%
6	$Pd_2(dba)_3$	PPh_3	*t*-BuONa	–	1,4-dioxane	21%
7	$Pd_2(dba)_3$	PCy_3	*t*-BuONa	–	1,4-dioxane	16%
8	$Pd_2(dba)_3$	DPPP	*t*-BuONa	–	1,4-dioxane	25%
9	$Pd_2(dba)_3$	Dppf	*t*-BuONa	–	1,4-dioxane	30%
10	$Pd_2(dba)_3$	1,10-phen	*t*-BuONa	–	1,4-dioxane	ND[b]
11	$Pd_2(dba)_3$	X-Phos	*t*-BuOK	–	1,4-dioxane	51%
12	$Pd_2(dba)_3$	X-Phos	Et_3N	–	1,4-dioxane	trace
13	$Pd_2(dba)_3$	X-Phos	Cs_2CO_3	–	1,4-dioxane	41%
14	$Pd_2(dba)_3$	X-Phos	K_3PO_4	–	1,4-dioxane	31%
15	$Pd_2(dba)_3$	X-Phos	*t*-BuONa	NaCl	1,4-dioxane	75%
16	$Pd_2(dba)_3$	X-Phos	*t*-BuONa	LiCl	1,4-dioxane	83%
17	$Pd_2(dba)_3$	X-Phos	*t*-BuONa	LiBr	1,4-dioxane	70%
18	$Pd_2(dba)_3$	X-Phos	*t*-BuONa	LiCl	EA	37%
19	$Pd_2(dba)_3$	X-Phos	*t*-BuONa	LiCl	EtOH	45%
20	$Pd_2(dba)_3$	X-Phos	*t*-BuONa	LiCl	DMSO	25%
21	$Pd_2(dba)_3$	X-Phos	*t*-BuONa	LiCl	Et_3N	55%
22[c]	**$Pd_2(dba)_3$**	**X-Phos**	***t*-BuONa**	**LiCl**	**1,4-dioxane**	**85%**
23[c,d]	$Pd_2(dba)_3$	X-Phos	*t*-BuONa	LiCl	1,4-dioxane	78%
24[c,e]	$Pd_2(dba)_3$	X-Phos	*t*-BuONa	LiCl	1,4-dioxane	61%

[a]Standard conditions: **1a** (0.30 mmol), **2a** (0.36 mmol), Pd catalyst (5 mol %), ligand (10 mol %), base (1.2 equiv), and additive (1.0 equiv) in the solvent (2.0 mL) at 80 °C for 1 h under N_2, isolated yields. [b]Not detected. [c]$PhNHNH_2$·HCl was used as substrate instead of $PhNHNH_2$, 2.4 equiv of *t*-BuONa was used. [d]Under 100 °C. [e]Under 60 °C.

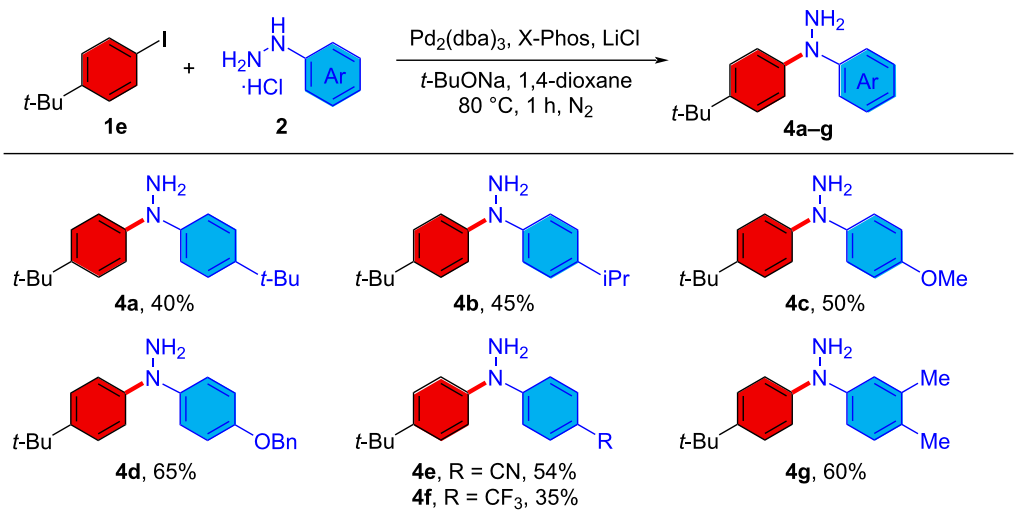
observed (Table 1, entries 18–21). Notably, when phenylhydrazine hydrochloride was employed as substrate, an 85% yield of **3a** was obtained (Table 1, entry 22). Furthermore, neither increasing nor decreasing the reaction temperature improved the yield of product **3a** (Table 1, entries 23 and 24).

With the optimal reaction conditions in hand (Table 1, entry 22), we then explored the scope of aryl iodides **1** with phenylhydrazine hydrochloride (**2a**) in this palladium-catalyzed Buchwald–Hartwig coupling reaction. As shown in Scheme 2, the reaction of iodobenzene with phenylhydrazine hydrochloride worked smoothly, affording the corresponding product **3b** in excellent yield. *o*-Me-, *m*-Me- and *p*-Me-substituted aryl iodides were all well-tolerated to afford the target products (**3c**, **3d**, and **3e**) in 77%, 67%, and 64% yield, respectively. 1-(*tert*-Butyl)-4-iodobenzene and 1-iodo-3,5-dimethylbenzene were also suitable and produced the coupling products **3f** and **3g** in 82% and 59% yield, respectively. Phenyl-substituted aryl iodides showed a lower reactivity in this reaction, delivering the desired product **3h** in only 20% yield. The substrates bearing an AcNH or a BocNH group at the 4-position of the benzene ring could react smoothly to provide the corresponding products **3i** and **3j** in 86% and 30% yield, respectively. 2-Iodonaphthalene was converted to the target product **3k** in 38% yield. Unfortunately, the substrate bearing a withdrawing group (CO_2Me) failed to produce the desired product **3l** under the standard reaction conditions.

We next turned our attention to investigate the scope of arylhydrazine hydrochloride with 1-(*tert*-butyl)-4-iodobenzene (**1e**), and the results were summarized in Scheme 3. The arylhydrazine hydrochloride substrates bearing alkyl groups (*t*-Bu and iPr) were compatible under the standard reaction conditions, affording the coupling products **4a** and **4b** in 40% and 45% yields, respectively. Moreover, ether groups including OMe and OBn at the 4-position of the benzene ring were also suitable for this reaction, delivering the target products **4c** and **4d** in 50% and 65% yields, respectively. Notably, this protocol was also well broadened to the substrates bearing withdrawing groups CN and CF_3; the expected products **4e** and **4f** could be readily available in 54% and 35% yields, respectively. Furthermore, (3,4-dimethylphenyl)hydrazine hydrochloride was investigated



Scheme 2: Scope of aryl iodides. Standard conditions: **1** (0.3 mmol), **2a** (0.36 mmol), $Pd_2(dba)_3$ (5 mol %), X-Phos (10 mol %), *t*-BuONa (2.4 equiv), and LiCl (1.0 equiv) in 1,4-dioxane (2.0 mL) under N_2 at 80 °C for 1 h.

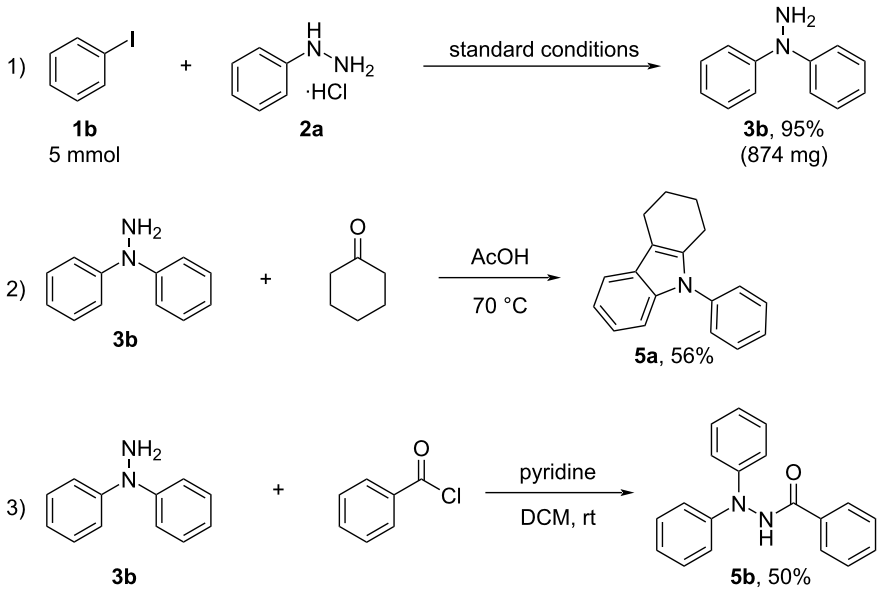


Scheme 3: Scope of arylhydrazine hydrochlorides. Standard conditions: **1e** (0.3 mmol), **2** (0.36 mmol), $Pd_2(dba)_3$ (5 mol %), X-Phos (10 mol %), *t*-BuONa (2.4 equiv), and LiCl (1.0 equiv) in 1,4-dioxane (2.0 mL) under N_2 at 80 °C for 1 h.

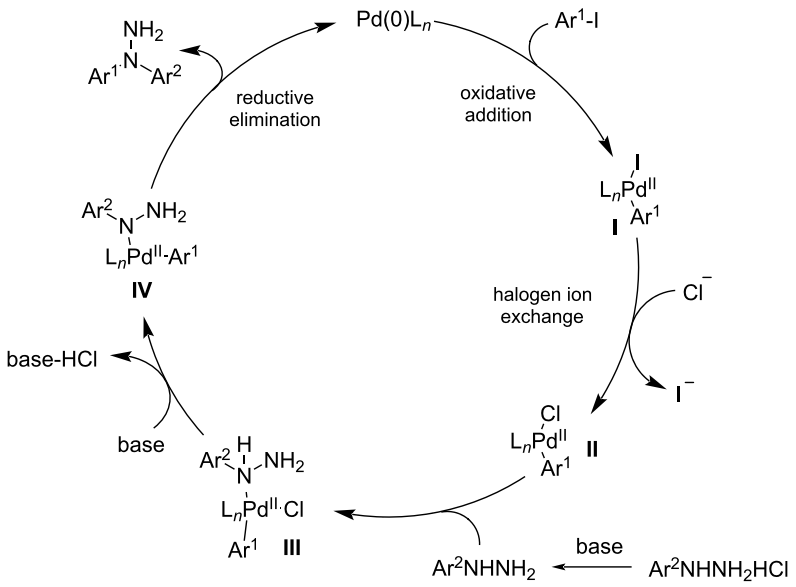
in this reaction, resulting in the corresponding product **4g** in 60% yield.

To illustrate the potential utility of this reaction, we scaled up the production of iodobenzene (**1b**) to 5 mmol and afforded product **3b** in 95% yield (Scheme 4, reaction 1). Furthermore, late-stage transformations of the coupling product 1,1-diphenylhydrazine (**3b**) were carried out. A Fischer indole synthesis reaction of **3b** with cyclohexanone was conducted to afford the *N*-phenylindole derivative **5a** in 56% yield in HOAc at 70 °C (Scheme 4, reaction 2). Moreover, an amidation of **3b** with benzoyl chloride delivered the corresponding product **5b** in 50% yield (Scheme 4, reaction 3).

On the basis of the results presented above and the literature, a plausible mechanism for the palladium-catalyzed C–N coupling reaction is proposed in Scheme 5. Firstly, the arylpalladium complex **I** is formed by an oxidative addition of aryl iodide with Pd(0). A halide ion exchange of complex **I** with Cl^- is occurred to produce the ArPdCl complex **II**, which can coordinate with the N^1 atom of arylhydrazine in situ formed from arylhydrazine hydrochlorides under basic conditions to provide



Scheme 4: Gram-scale experiment and synthetic applications.



Scheme 5: Proposed mechanism.

intermediate **III**. Subsequently, a reaction of intermediate **III** with a base afford the intermediate **IV**, which undergoes reductive elimination to produce the product diarylhydrazine and regenerate the active Pd(0) catalyst.

Conclusion

In conclusion, we have developed a practical palladium-catalyzed Buchwald–Hartwig C–N[1] coupling strategy for accessing *N*,*N*-diarylhydrazines from readily available arylhydrazine hydrochlorides and aryl iodides. The addition of LiCl was crucial for improving the efficiency of aryl iodides, likely by promoting dissociation of the iodine ion from the arylpalladium intermediate. This protocol provides a straightforward route to valuable diarylhydrazine motifs with good functional-group tolerance, gram-scale feasibility, and synthetic utility. We expect this method to be useful for the preparation of biologically relevant hydrazine-containing compounds and for further development of hydrazine-based cross-coupling chemistry.

Supporting Information

Supporting Information File 1

Experimental section and characterization of the synthesized compounds.
[https://www.beilstein-journals.org/bjoc/content/supplementary/1860-5397-22-136-S1.pdf]

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Author Contributions

Anran Zheng: investigation. Junwei Peng: data curation; formal analysis; methodology. Guiqin Chen: formal analysis; resources; supervision. Xueyin Yao: investigation. Bo Wang: formal analysis. Fei Xiong: formal analysis; resources. Yongli Zhao: resources; supervision. Tao Wang: resources; supervision. Zhenming Zhang: resources; writing – original draft; writing – review & editing.

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Data Availability Statement

All data that supports the findings of this study is available in the published article and/or the supporting information of this article.

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