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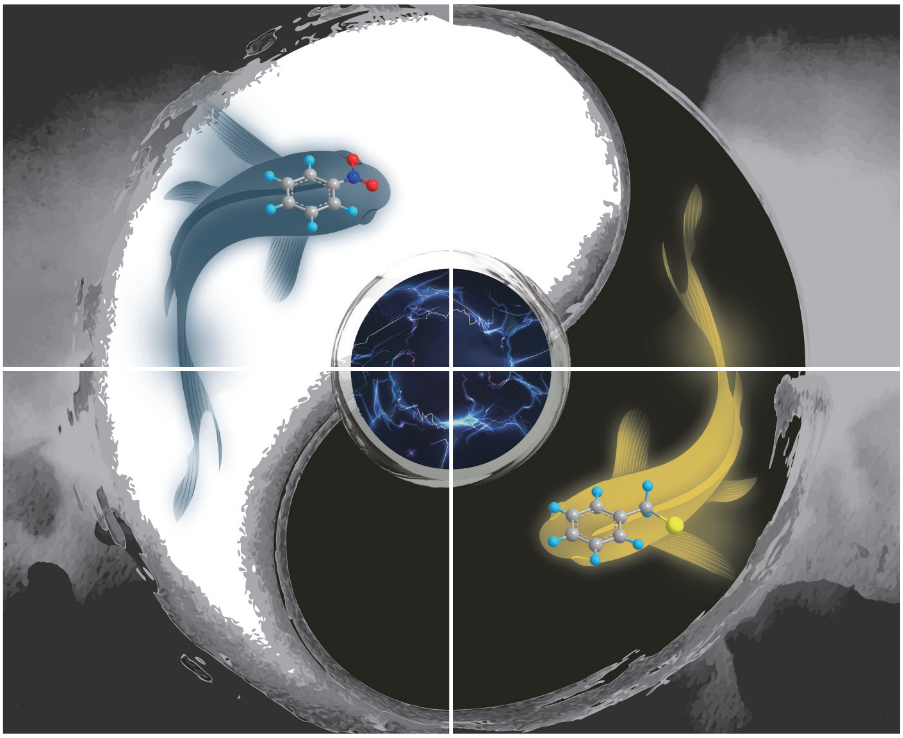

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Electrochemically driven reductive coupling of nitroarenes with alkyl bromides

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Electrosynthesis, which facilitates redox events under metal-catalyst free conditions, has garnered significant interest. Herein, we explore this promising methodology by introducing an application to the cross-coupling of nitroarenes with alkyl bromides. This approach enables the synthesis of aromatic tertiary amines from readily available and cost-effective starting materials under mild redox conditions. By leveraging the advantages of electrosynthesis, we achieved efficient transformations with good functional group compatibility, thereby contributing to the sustainable modification of valuable bioactive molecules.

Introduction

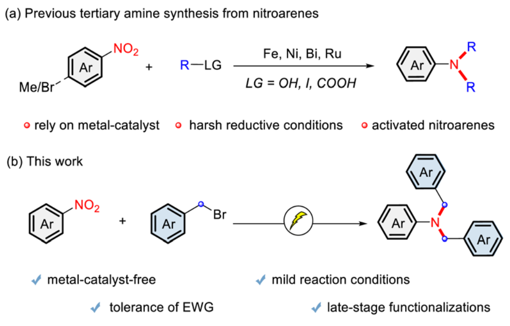
Amines are among the most pivotal motifs in chemistry,[1] especially aromatic tertiary amines, which are widely recognized as versatile building blocks[2] and fundamental constituents of various pharmaceuticals.[3,4] Consequently, the development of strategies for C–N bond formation,[5,6] particularly for the synthesis of aromatic tertiary amines, has attracted considerable attention.[7–13] While most C–N bond formations have been accomplished using amines as the nitrogen source, which are industrially produced from nitroarenes,[14] the sustainable strategy of employing nitroarenes for amine synthesis has attracted significant attention due to its step economy and cost efficiency.[15–30] Despite considerable achievements, the reductive amination of nitroarenes for the construction of tertiary amines remains underdeveloped,[31–34] with the examples reported thus far being largely limited to metal catalysts,[31–34] activated nitroarenes,[34] and harsh reductive conditions (Scheme 1a).[31,34]

The use of renewable and easily available electricity to create milder and greener redox conditions in comparison with traditional synthesis has provided a significant stimulus to organic synthesis.[35–41] However, although this strategy has been proven to be powerful in reductive transformations,[42–47] in strong contrast to C–C bond forming reactions, its application in C–N bond formation continues to be scarce, with limited examples reported for secondary amine construction.[48–54] Herein, we have developed an unprecedented metal-catalyst-free electro-reductive coupling of nitroarenes for the selective tertiary amine synthesis. The notable achievements of this work are as follows: (a) transition-metal-catalyst-free conditions; (b) mild reaction conditions; (c) tolerance toward electron-withdrawing groups; and (d) enabling late-stage functionalization of bioactive molecules (Scheme 1b).

Results and discussion

Optimization of reaction conditions

We initiated the optimization of the electroreductive coupling reaction using nitrobenzene (**1a**) and benzyl bromide (**2a**) as the model reactants in a user-friendly undivided cell (Table 1). Preliminary studies identified Ni foam and graphite felt (GF) as the optimal electrodes (entries 1–5). Furthermore, both the pyridine substitution pattern (entries 6–10) and the diboronate structure (entries 11 and 12) proved critical for this transformation. Further attempts to optimize the electrolytes and to lower the reaction temperature led to a decrease in efficiency (entries 13–15). A control experiment identified



Scheme 1 Methods for tertiary amine synthesis with nitroarene.

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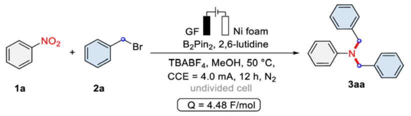
Table 1 Deviations from the optimized conditions[a]



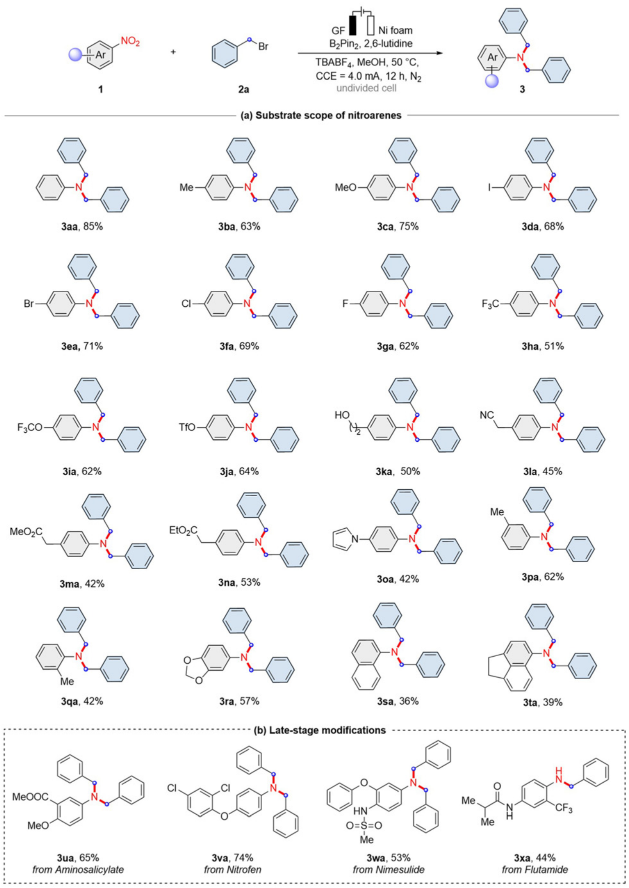
Entry	Variation from the standard conditions	Yield of **3aa** ^{b}/%
1	No change	92, 85^{c}
2	GF(+)/Cu(−)	71
3	GF(+)/Pt(−)	43
4	GF(+)/SS(−)	70
5	RVC(+)/Ni foam(−)	Trace
6	2,4,6-Collidine instead of 2,6-lutidine	64
7	Pyridine instead of 2,6-lutidine	Trace
8	2,6-Di-*tert*-butylpyridine instead of 2,6-lutidine	N.R.
9	DMAP instead of 2,6-lutidine	Trace
10	K_2CO_3 instead of 2,6-lutidine	N.R.
11	B_2Cat_2 instead of B_2Pin_2	64
12	Cy-Bpin instead of B_2Pin_2	11
13	TBAP instead of $TBABF_4$	63
14	$LiClO_4$ in place of $TBABF_4$	73
15	Room temperature	60
16	No electricity	N.R.

[a] Standard conditions: undivided cell, GF anode, Ni foam cathode, **1a** (0.40 mmol), **2a** (1.20 mmol), $TBABF_4$ (0.60 mmol), B_2Pin_2 (0.80 mmol), 2,6-lutidine (1.20 mmol), MeOH (4.0 mL), constant current (CCE) = 4.0 mA, 50 °C, 12 h, under an N_2 atmosphere. [b] Yields determined by GC analysis using 1,3,5-trimethoxybenzene as the internal standard. [c] Isolated yields. GF = graphite felt. B_2Cat_2 = bis(catecholato)diborane; Cy-Bpin = 2-cyclohexyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane.

the essential role of electricity in the reduction of nitroarenes (entry 16). Furthermore, current density, the amount of applied charge, and pH of the reaction mixture play a crucial role in the reaction outcome (Fig. S3–S5).[48] The optimal conditions were: current density of 1.0 mA cm^{-2}, applied charge of 4.48 F mol^{-1}, and pH 8.35. Further increase in current density or applied charge caused product decomposition, whereas lower values led to incomplete nitrobenzene conversion. Increased pH impeded proton acquisition, thereby suppressing nitrobenzene reduction, whereas decreased pH facilitated over-reduction, producing aniline.[55–59] Notably, a relative low amount of applied charge (4.48 F mol^{-1}) is required for this nitro reductive coupling reaction.[49–54] This may be attributed to the optimal pH closely matching the reduction potentials of nitrobenzene and benzyl bromide, thereby enabling high chemoselectivity at low current density while minimizing excessive energy consumption.

Robustness

With the optimal conditions established, we evaluated the versatility of the electroreductive coupling reaction using a variety of nitroarenes (Scheme 2). To our delight, nitroarenes bearing electron-donating (**3ba** and **3ca**) or electron-withdrawing groups were efficiently converted to the corresponding products (**3da**–**3ja**). Notably, redox-sensitive functional groups, such as hydroxy (**3ka**), nitrile (**3la**), ester (**3ma** and **3na**) and pyrrole (**3oa**), were well tolerated and provided the desired products in moderate yields, further demonstrating the mild redox conditions of the reaction. Moreover, other aromatic ring systems were also compatible with the coupling conditions (**3ra**, **3sa**, and **3ta**). Importantly, this novel approach can be applied to the late-stage modification of bioactive compounds such as aminosalicylate (**3ua**), nitrofen (**3va**), nimesulide (**3wa**), and flutamide (**3xa**).

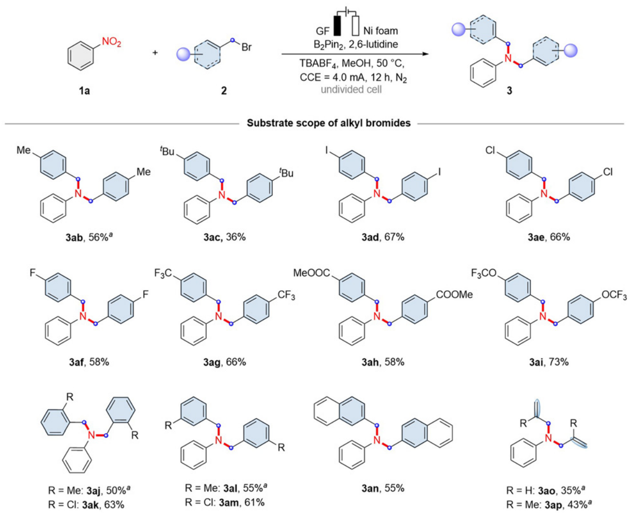


Scheme 2 Substrate scope of nitroarenes and late-stage modifications. Standard conditions: undivided cell, GF anode, Ni foam cathode, **1** (0.40 mmol), **2a** (1.20 mmol), $TBABF_4$ (0.60 mmol), B_2Pin_2 (0.80 mmol), 2,6-lutidine (1.20 mmol), MeOH (4.0 mL), constant current (CCE) = 4.0 mA, 50 °C, 12 h, under an N_2 atmosphere.

Encouraged by the successful electroreduction of nitroarenes, we next explored the scope of benzyl bromide substrates (Scheme 3). To our delight, benzyl bromide compounds containing EDGs (**3ab** and **3ac**) and EWGs (**3ad**–**3ai**) were found to be compatible with this reaction and successfully afforded the corresponding products in moderate to good yields. Notably, allyl bromides were also tolerated under the conditions (**3ao** and **3ap**).

Mechanistic studies

To elucidate the reaction mechanism, detailed mechanistic studies were performed. Studies with potential intermediates



Scheme 3 Substrate scope of alkyl bromides. Standard conditions: undivided cell, GF anode, Ni foam cathode, **1a** (0.40 mmol), **2** (1.20 mmol), TBABF$_4$ (0.60 mmol), B$_2$Pin$_2$ (0.80 mmol), 2,6-lutidine (1.20 mmol), MeOH (4.0 mL), constant current (CCE) = 4.0 mA, 50 °C, 12 h, under an N$_2$ atmosphere. ^{a} 24 h.

revealed that nitrosoarene (**Int-I**), *N*-phenylhydroxylamine (**Int-II**), and aniline (**Int-III**) could participate in product formation (Fig. 1a). However, 2,6-lutidine suppressed aniline formation during nitroarene reduction (Fig. 1b), disfavoring it as a major intermediate. This effect stems from the base-mediated pH increase that modulates protonation states of intermediates.[48] Furthermore, nitrosoarene is more easily reduced than the parent nitroarene,[48] and *N*-phenylhydroxylamine exhibited superior reactivity toward nitrosoarene under the standard conditions. Cyclic voltammetry showed that the addition of benzyl bromide eliminated the *N*-phenylhydroxylamine oxidation peak at −1.03 V (*vs.* Ag/AgCl, Fig. 1c). These results implicated *N*-phenylhydroxylamine as the key intermediate. *N*-Benzyl-*N*-phenylhydroxylamine and *N*-benzylaniline both gave high product yields under the standard conditions with or without an applied potential (eqn (1), Fig. 1d and Scheme S14a, SI). In contrast, reactions of *N*-benzyl-*N*-phenylhydroxylamine without B$_2$pin$_2$ afforded only 6% product yield (eqn (2), Fig. 1d), establishing B$_2$pin$_2$ as the reductant. Without 2,6-lutidine, product formation ceased while benzyl bromide underwent solvolysis (Schemes S12 and S13, SI). Considering benzyl bromide's lower reduction potential (−1.98 V *vs.* Ag/AgCl) relatively to that of nitroarene (−2.31 V *vs.* Ag/AgCl for reduction to *N*-phenylhydroxylamine, Fig. 1c), this effect could be rationalized by the ability of 2,6-lutidine to stabilize the highly reactive benzylboronic species generated through the electrolysis of benzyl bromide.[54] Competition experiments confirmed higher reactivity of electron-rich benzyl bromides (Scheme S17, SI). Finally, ^{11}B NMR analysis detected MeOBpin (**D**) formation, indicating favorable B$_2$pin$_2$ oxidation at the anode (Scheme S15, SI).

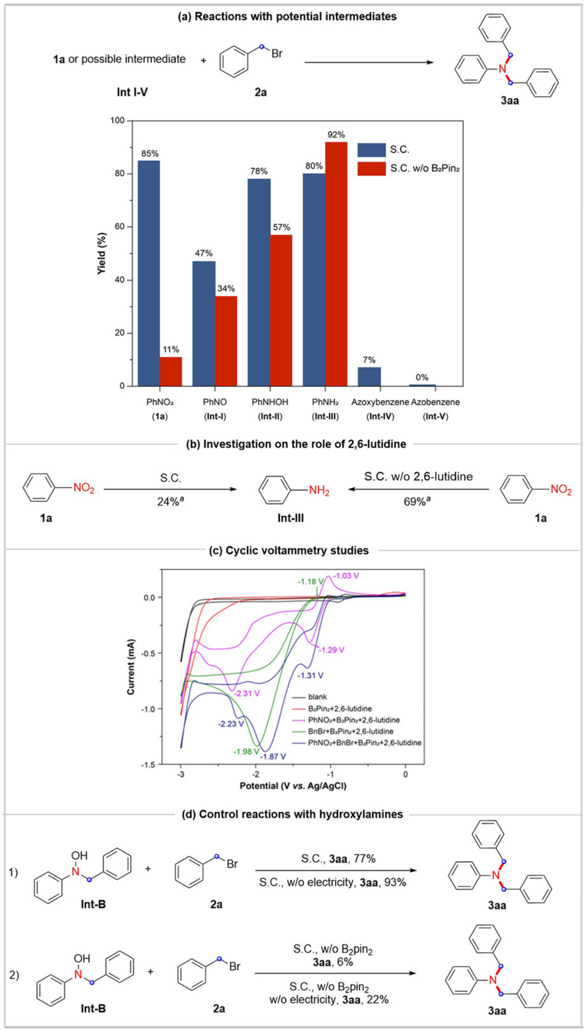
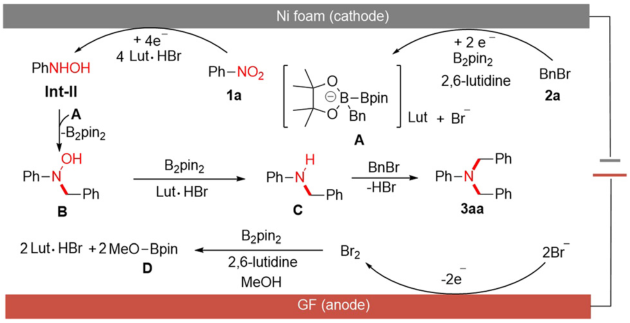


Fig. 1 Mechanistic studies. Standard conditions: undivided cell, GF anode, Ni foam cathode, nitrogen reagent (0.40 mmol), **2a** (1.20 mmol, if required), TBABF$_4$ (0.60 mmol), B$_2$Pin$_2$ (0.80 mmol), 2,6-lutidine (1.20 mmol), MeOH (4.0 mL), constant current (CCE) = 4.0 mA, 50 °C, 12 h, under an N$_2$ atmosphere. ^{a} Yields determined by GC-MS using 1,3,5-trimethoxy benzene as the internal standard.



Scheme 4 Proposed mechanism.

Based on literature reports[31–34,48–59] and experimental findings, the mechanism of the electrochemical reductive coupling was proposed (Scheme 4). Initially, nitroarene **1a** undergoes reduction at the cathode to generate *N*-phenylhydroxylamine (**Int-II**). Concurrently, benzyl bromide reacts with B_2pin_2 and 2,6-lutidine to afford intermediate **A**. Nucleophilic substitution between **Int-II** and **A** furnishes intermediate **B**, which subsequently undergoes further reduction and nucleophilic substitution to afford the final product **3aa**. At the anode, bromide is oxidized to bromine, and B_2pin_2 undergoes oxidation.

Conclusions

In summary, we have developed an efficient electrochemical-driven reductive coupling of nitroarenes and benzyl bromides for the synthesis of tertiary amines. This green and environmentally friendly strategy shows good compatibility with electron-withdrawing groups as well as functional groups, affording a series of tertiary amines in moderate to good yields. Mechanistic studies revealed a two-electron pathway, with *N*-phenylhydroxylamine identified as an important intermediate.

Author contributions

J. M. conceived the project and supervised the research. X. S., W. W., Z. D., J. L. and J. L. performed and analyzed the experiments. X. S., W. W., Z. D., J. L., J. L. and J. M. prepared the manuscript and the SI files.

Conflicts of interest

There are no conflicts to declare.

Data availability

The data supporting this article have been included as part of the supplementary information (SI). Supplementary information: experimental details and characterization data of all new compounds. See DOI: **https://doi.org/10.1039/d5qo01269d**.

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