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Electrosynthesis of 1*H*- and 2*H*-indazoles by reduction of *o*-nitrobenzylamines, a versatile and sustainable access

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Indazoles represent an important pharmacologically active class of compounds. However, reliable and sustainable pathways from simple substrates remain scarce. We report an easily applicable and sustainable electrochemical synthesis of various 1*H*- and 2*H*-indazoles *via* direct reduction of *o*-nitrobenzylamines, avoiding stoichiometric reagents and elevated temperatures. The protocol enables access to 50 distinct structures with isolated yields up to 96% and a broad tolerance for functional groups and heterocycles. Additionally, we demonstrate electrochemical post-functionalization, access to APIs and their precursors, as well as robust scalability.

Introduction

Over the past decade, the percentage of approved small molecule drugs containing a nitrogen-based heterocycle has steadily increased, with recent reports stating up to 80% of newly approved medications.[1,2] Within these scaffolds, indazoles account for a particularly interesting class, as indicated by their three appearances in the top 200 small molecule drugs by retail sales in 2025.[3] Active pharmaceutical ingredients (APIs) containing an indazole structure exhibit a wide range of bioactivity, such as anti-cancer, anti-inflammatory, anti-tubercular, anti-diabetic, as well as anti-bacterial and anti-fungal effects.[4–8] Representative examples include the marketed APIs pazopanib,[9,10] and niraparib.[11] In addition, various indazole derivatives with promising properties are under investigation (see Scheme 1A).[12]

In contrast to their aforementioned bioactivity, indazoles are rarely encountered in nature, including only a few reports, like the one of the plant *Nigella sativa*.[7] This scarcity underscores the necessity for reliable synthetic pathways to access such structures. Hence, the development of general, efficient, and sustainable methods for the synthesis of indazoles remains highly desirable.

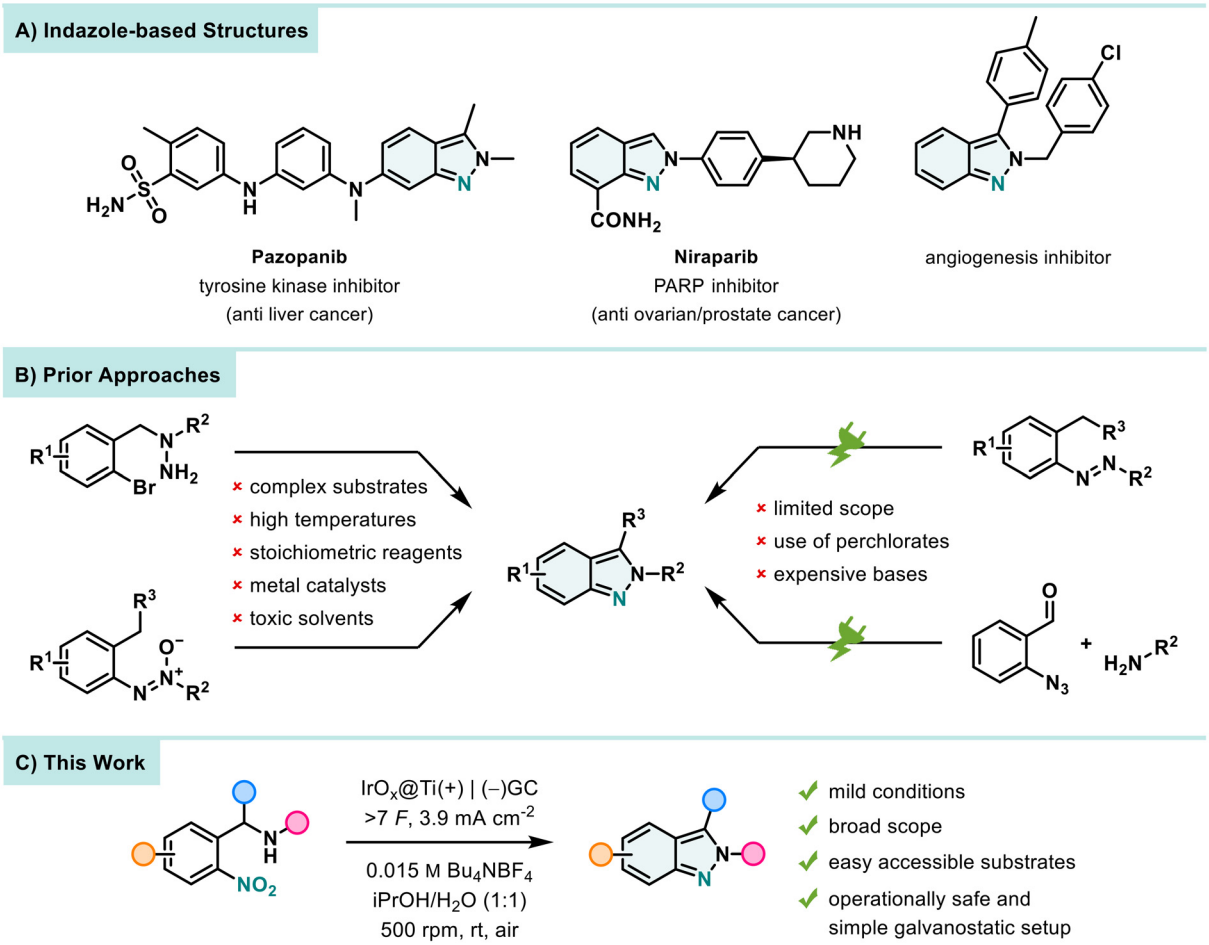
Commonly, indazoles are prepared by thermochemical approaches, including base-mediated condensations,[13] 2,3-dipolar cycloaddition/decarboxylation,[14] as well as transition-metal catalysis (*e.g.*, Pd, Rh, or Cu).[15–17] However, many of these methods require extensive substrate synthesis, harsh, and energy-intensive conditions (*e.g.*, elevated temperatures), as well as frequent use of toxic reagents in stoichiometric amounts (see Scheme 1B). These factors make them difficult to control, generate substantial chemical waste, and entail significant safety concerns.

As a powerful alternative that circumvents these concerns, synthetic electrochemistry has re-emerged as an engaging, safer, and more sustainable approach.[18–25] The use of electric current as an inexpensive, clean, and renewable energy source omits the administration of stoichiometric oxidants/reducing agents and additional catalysts, thereby considerably reducing chemical waste generation and enabling milder conditions coupled with mitigation of safety risks associated with traditional methods. As a consequence, electro-organic synthesis provides efficient and more sustainable routes to value-added products.[26–29] Practical advantages include inherent safety and the potential for cost-efficient scalability.

Furthermore, synthetic electrochemistry has proven highly valuable for generating N-heterocycles.[30–38] In particular, the electroreduction of nitro compounds allows tunable reactivity by adjusting the employed parameters (*e.g.*, electrodes, current density, pH), providing access to a broad variety of heterocycles.[39–49]

Up to now, reported electrochemical routes to form indazoles face limitations concerning safety in technical operations.[50] For example, the oxidation-induced cyclization of

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Scheme 1 A) Important indazole structures of pazopanib, niraparib and an angiogenesis inhibitor; (B) prior described conventional and electrochemical approaches towards indazoles with possible limitations; and (C) our newly developed electrochemical nitro reduction protocol.

azobenzenes employs perchlorates as supporting electrolyte, requires heating of the mixture, and evolves hydrogen gas at the cathode, which are considered unfavorable combinations at elevated temperatures.[51] On the other hand, reports on electrochemical hydrazone cyclization reactions are limited by the use of highly fluorinated solvents like hexafluoroisopropanol (HFIP), evolving hydrogen, and, in addition, are restricted to 1*H*-indazoles and often demand an inert atmosphere.[52,53] Other strategies, on one hand, involve aryl azides and costly bases, while others only yield protected 1*H*-indazoles *via* an indirect iodide to iodine redox mediator.[54,55] In contrast, accessing indazoles *via* electroreduction has only been scarcely investigated. To our knowledge, Moinet *et al.* reported a redox flow setup, operated potentiostatically, with a relatively narrow scope (see Scheme 1B).[56] Moreover, most strategies yield *N*-arylated indazoles, omitting the chemical space of alkylated products.

Prompted by these limitations, we designed a safer, operationally simple electrochemical protocol to create indazoles from readily available *o*-nitrobenzylamines in an undivided cell setup (see Scheme 1C).

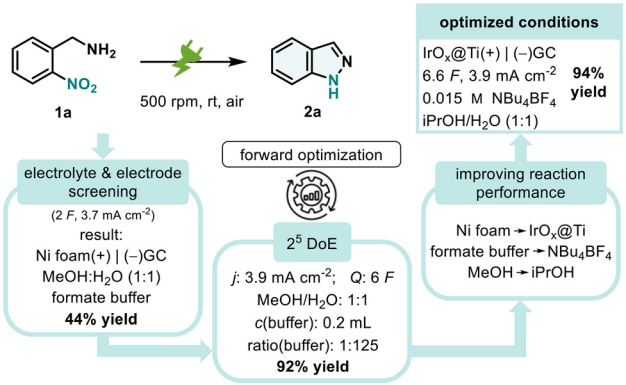
Results and discussion

Guided by knowledge derived in our group on nitroarene reductions, we optimized the conditions for the unsubstituted substrate **1a**.[39,41,42] Our aim was to obtain an operationally simple undivided cell setup, selectively targeting the nitroso product by reduction to favor the indazole formation (see Scheme 2). The specific formation of the electrophilic nitroso species enables an intramolecular attack of the benzylic amine, followed by condensation to the desired indazole. Precise control is inevitable, since the over-reduced hydroxylamine could act as electricity shuttle, causing the need of additional amount of applied charge.

In theory, a neutral medium and low current densities should favor the partial reduction to the nitroso species.[39] Initial parameter evaluation was conducted in 5 mL screening cells[57] using a glassy carbon (GC) anode and boron-doped diamond (BDD) cathode, while applying an amount of applied charge of 2 *F* (theoretical amount) and 3.7 mA cm^{-2}.

First, we screened different solvent systems, while concurrently balancing sufficient substrate solubility, toxicity, and

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Scheme 2 Optimization study with the unsubstituted substrate **1a** to form indazole **2a** (more details see SI).

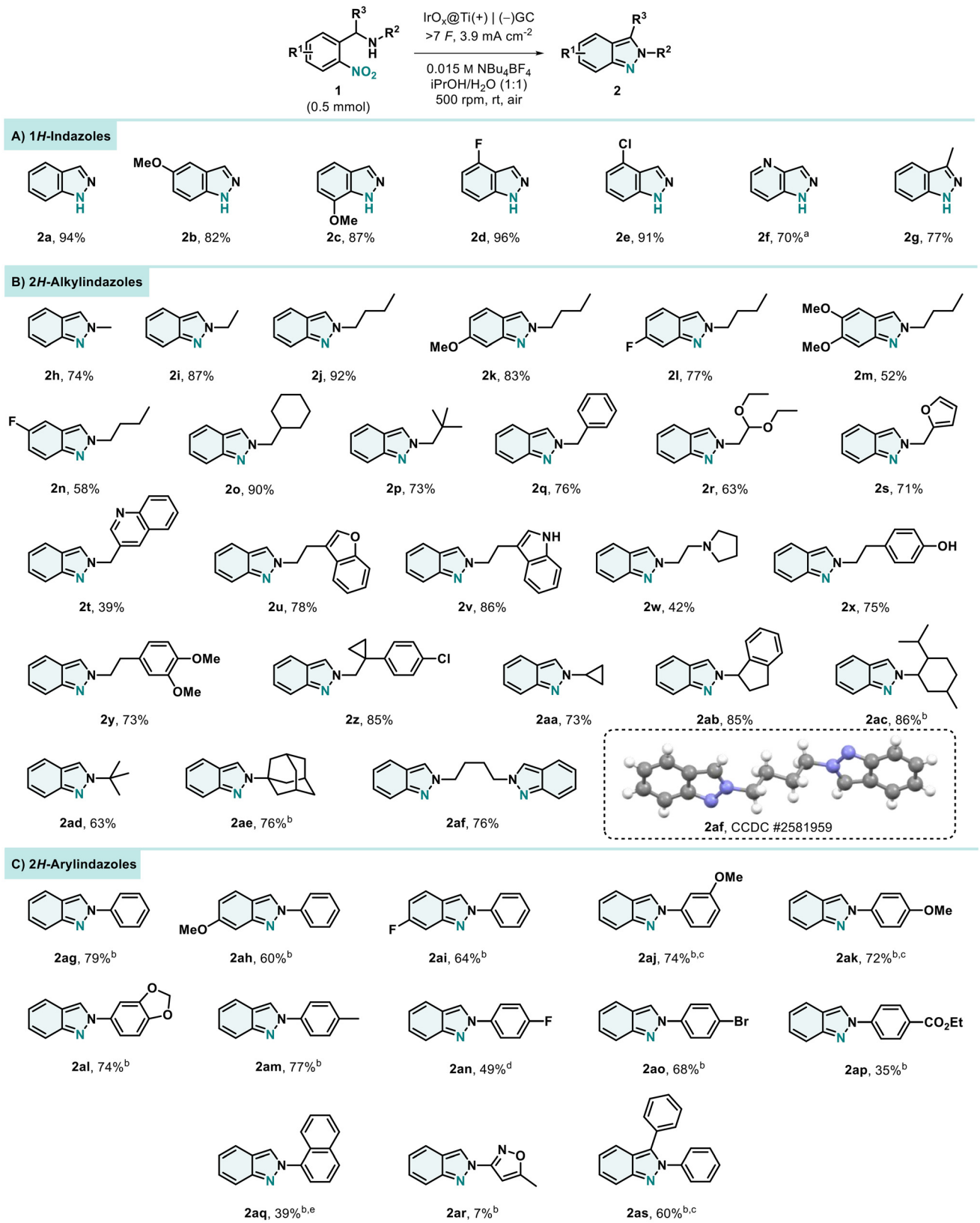
conductivity (mixtures of MeOH, H_2O, MeCN and acetone), alongside supporting electrolytes (formate/acetate buffers, Bu_4NBF_4, H_2SO_4 and NaOH, see SI for details). A formate buffer in a 1 : 1 MeOH/H_2O mixture was selected for further optimization. We then evaluated a variety of electrode materials (BDD cathode for anode screening and graphite anode for cathode screening). As a result, GC evolved as the best cathode (robust, easy to clean) and nickel foam as the best anode. After determination of the categorical parameters, a Design of Experiment (DoE), a method to investigate multifactor interactions of parameters, was employed to optimize towards maximum yields (92%; 5 factors, interaction between buffer concentration and ratio, solvent ratio, amount of applied charge, and current density).[29,58] Nevertheless, free nickel ions were detected in the reaction mixture, while microscopic inspection confirmed anode fouling, causing reconsideration of the counter reaction.[59] To minimize unfavored oxidations of solvent/supporting electrolyte, dimensionally stable anode (DSA) materials were tested to improve clean oxygen evolution reaction (OER).[60] IrO_x@Ti proved as the best substitute. Simultaneously, the solvent was exchanged to iPrOH, which is less volatile and generated less by-products and Bu_4NBF_4 as supporting electrolyte. A brief re-screening was performed, confirming IrO_x@Ti and GC as anode and cathode, respectively, in this medium, further boosting the yield of **2a** (see Scheme 2 and SI for more details).

With the optimized conditions in hand, the unsubstituted model substrate yielded 1*H*-indazole **2a** in 94% yield. On that basis, we evaluated different core-substituted primary amines (see Scheme 3A), where electron-donating methoxy derivatives were achieved in 82% and 87% yield (**2b** and **2c**). Halogenated substrates, which are often prone to dehalogenation, were well tolerated, achieving yields of 91–96% (**2d** and **2e**). Gratifyingly, the more challenging synthesis of a pyrazolopyridine derivative (**2f**, 8.5 *F* and C_{gr} anode, see SI for more details) was achieved in 70% yield. Furthermore, substitutions in C3 position, as shown in case of indazole **2g**, proved to be successful.

To broaden the scope, the cyclization performance of secondary amines, which suppress rearrangement to 1*H*-indazoles and furnish 2*H*-indazoles instead, was investigated. Two representative substrates (*N*-butyl **1j** and *N*-phenyl **1ag**) were used and the conditions (supporting electrolyte and electrodes) were re-evaluated, in particular considering poor solubility of organic substrates in aqueous mixtures. The previously obtained optimized conditions proved to be most efficient, while alternative ones increased by-product formation (see SI for NMR comparisons). Performed control experiments confirmed, that no conversion occurs in the absence of electric current alongside the stability of the generated indazoles when more charge is applied. Furthermore, no transformation to the respective indazole was observed when starting from the *o*-nitrobenzylimine. Subsequently, the scope was expanded to generate various 2*H*-indazoles, while 7 *F* for alkyl- and 12 *F* for aryl-substituted amines were applied (conversion monitored by UPLC-MS).

First off, we evaluated aliphatic *N*-substituents, including linear chains, sterically more demanding and bridged hydrocarbons. Linear substitutions (Me, Et, and Bu) yielded indazoles **2h–2j** in yields of 74–92%. Butyl-substitution in combination with core-substitutions (methoxy and fluoride) was additionally examined. *meta*-Substitutions to the nitro group showed comparable yields (**2k** for *m*-OMe and **2l** for *m*-F), while *para*-substituted substrates displayed reduced yields of 52–58% (**2m** for *p*-OMe and *m*-OMe and **2n** for *p*-F). Next, methylene-bridged hydrocarbon systems (cyclohexyl **2o**, *tert*-butyl **2p**, and phenyl **2q**) were tested, validating the robustness of the method with yields reaching 90%. An acetal protected aldehyde, which can readily be cleaved and then further reacted, was obtained in 63% yield (**2r**). Furthermore, we evaluated heterocyclic substrates (furfuryl **2s**, quinolines **2t**, benzofurans **2u**, and indole from tryptamine **2v**), proving that bridged heterocycles are well tolerated within the protocol. However, ethylene pyrrolidinyl functionalized indazole (**2w**) suffered losses during the work-up, due to high aqueous solubility, diminishing the yield to 42%. Bridged free phenol (**2x**), veratrole (**2y**), a relevant biological scaffold, and a more complex cyclopropylchlorophenyl substituent (**2z**) were readily tolerated with yields of 73–85%. Next, sterically more demanding alkyl substituents were examined (cyclic **2aa–2ac**, *tert* butyl **2ad** and adamantyl **2ae**), resulting in yields of 63–86%. Notably, a symmetric butyl-bridged substrate underwent smooth double cyclization, yielding bis-indazole (**2af**) in 76% yield. Formation of **2af** was unambiguously confirmed by X-ray analysis of a single crystal (see Scheme 3B). Worth to mention, is that the smaller and shorter the respective substituent, the more volatile the indazole.

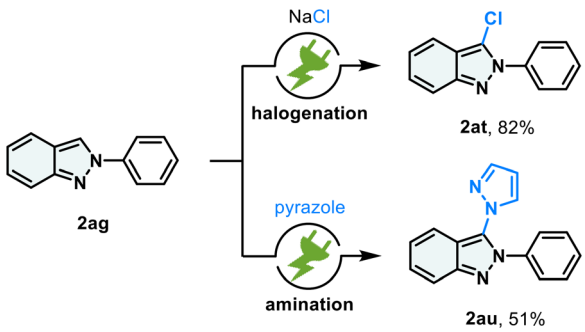
As next step, less nucleophilic arylamines were investigated regarding their cyclization properties, but as mentioned afore, 12 *F* had to be applied for full conversion of the substrates. The phenyl-substituted model substrate (**2ag**) was cyclized with 79%, while *meta* core-substituted derivatives (OMe **2ah** and F **2ai**) showed decreased yields. Some substrates suffered from poor solubility. Thus, the ratio of iPrOH/H_2O in the reaction for the electron-donating methoxy substituents (**2aj** and **2ak**) was increased to 3 : 2, while a benzodioxolyl (**2al**) and



Scheme 3 Overview of the scope (0.5 mmol) of 1*H*- and 2*H*-indazoles with isolated yields as well as the molecular structure of indazole **2af** determined by X-ray analysis of a single crystal (ORTEP image ellipsoid at 50% probability; CCDC number: 2581959; bottom); [a] 8.5 *F*, C_{gr} cathode (more details see SI); [b] 12 *F*; [c] iPrOH/H_2O (3 : 2); [d] 17 *F*, [e] iPrOH/H_2O (7 : 3).

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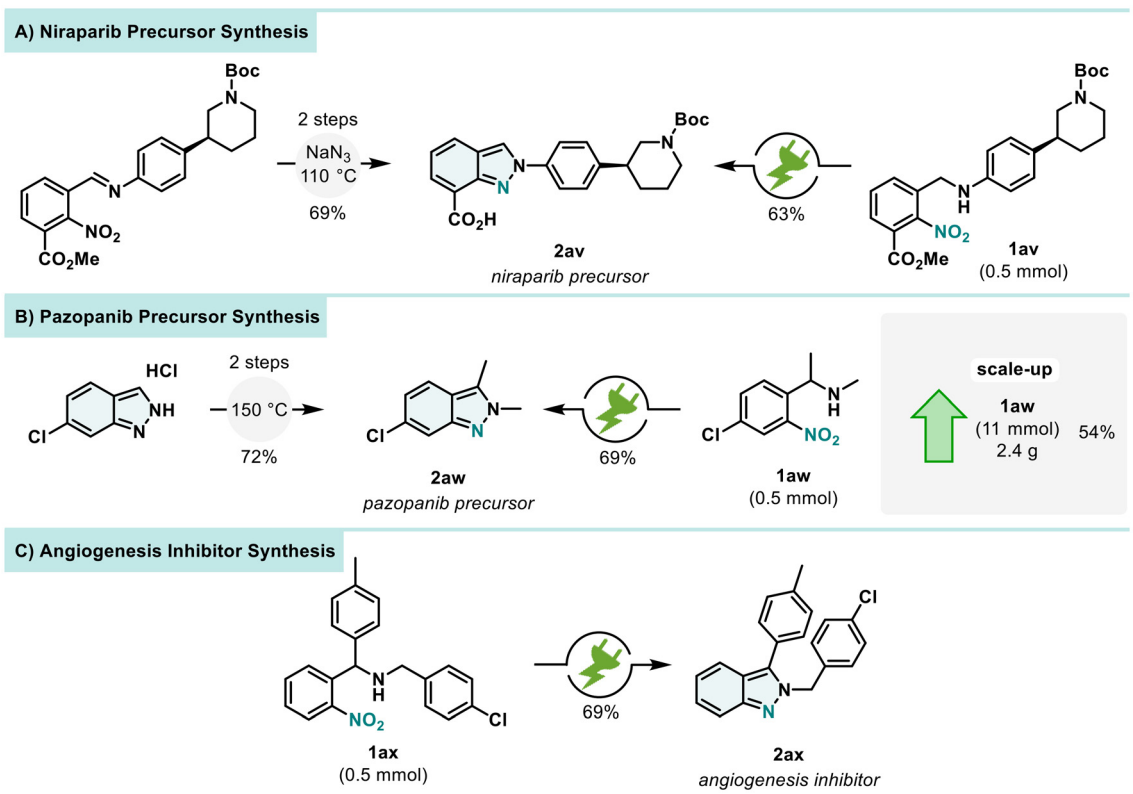
p-Me arylamine (**2am**) were kept at 1 : 1, resulting in similar yields of around 75%. More electron-poor systems had a significant decrease in yield, due to the reduced nucleophilicity of aniline derivatives. As a result, *para*-F substituted aniline indazole (**2an**) was formed in 41%. Applying an additional amount of 5 *F* raised the yield to 49%, but considering the cost-efficiency, 12 *F* were deemed more effective. Similarly, the reduced reactivity was also observed for *p*-Br, *p*-COOEt (**2ao** and **2ap**), and naphthyl-substituted indazoles (**2aq**) with respective yields of 68, 35 and 39% (iPrOH/H_2O (7 : 3) for **2aq**). Nevertheless, direct substitution with an isoxazole moiety (**2ar**) only yielded 7%, a result which was attributed to decomposition and dissociation of the initial substrate. Indazole (**2as**) was obtained in 60%, adding another substrate to the 3*H*-substituted indazoles within the protocol (see Scheme 3C).



Scheme 4 Electrochemical post-functionalization of **2ag**; chlorination (**2at**, top)[62] and amination (**2au**, bottom).[63]

Many obtained products are 2*H*-indazoles, offering attractive opportunities for C3 post-functionalization.[61] Multiple electrochemical transformations, such as halogenation or amination have been reported, only requiring minor deviations to our applied system.[62,63] Accordingly, **2ag** was successfully converted to **2at** (electrochemical chlorination) in 82% and **2au** (electrochemical amination) in 51% yield, following literature described protocols (see Scheme 4).[62,63] This was also shown by subsequent reactions without in between workup, where after initial electrolysis, simple solvent removal, electrode exchange, and addition of new supporting electrolyte and solvent mixture enabled direct post-functionalization and broadening the applicability (see SI for more details).

With the scope established, we selected several API targets to further showcase the applicability of our designed method. As noted above, niraparib (distributed as Zejula) is a prominent indazole-derived API. Indazole **2av** is a key precursor in reported process routes towards niraparib and was obtained by our method in 63%.[64] This mild, sustainable electrochemical pathway offers a viable alternative concerning the use of reactive substrates and elevated temperatures in conventional routes. Worth to mention, our electrochemical approach fur-



Scheme 5 (A) Comparison of conventional[64] and electrochemical synthesis of a niraparib precursor (**2av**); (B) comparison of conventional[65] and electrochemical synthesis of a pazopanib precursor (**2aw**) and scale-up and (C) electrochemical synthesis of an angiogenesis inhibitor (**2ax**).

nishes the carboxylic acid directly, without any further saponification step (see Scheme 5A). Pazopanib (distributed as Votrient) is another renowned representative with indazole **2aw** as key intermediate.[65] Reported routes rely on several elevated temperature steps, while our method enables access to **2aw** in a shorter route, allowing for literature known follow-up transformations (see Scheme 5B). Scalability of the substrate was shown as well as for indazole **2j**, in which case the substrate concentration was significantly increased (see SI for further details), showcasing the overall potential and practicability for processes in industry. Yields can possibly be improved by shifting into flow chemistry, where residence time on the electrodes can be reduced. In addition, our method afforded an angiogenesis inhibitor (**2ax**) in 69% yield (see Scheme 5C).

Conclusions

In conclusion, we have developed a versatile, operationally simple electrochemical protocol for accessing a wide range of indazoles under mild, sustainable conditions, hence, circumventing harsh reagents and complex setups. The method furnishes 50 diverse structures with isolated yields up to 96%, displaying a broad functional group and heterocycle tolerance. Additionally, it facilitates direct electrochemical post-functionalization, expanding the chemical space for drug discovery. On top of this, we provide alternative synthetic routes to intermediates of the APIs niraparib and pazopanib, highlighting the procedures relevance for future drug design. The process is robust and scalable, hence enabling multigram-scale preparation of indazoles, proving it suitable for the synthesis of a vast pool of fine chemicals.

Author contributions

B. W. and S. R. W. conceived the idea of the project. C. K. conducted the initial optimization experiments. E. F. R. P. and D. D. synthesized starting materials, conducted the scope experiments, planned and carried out the synthesis of the APIs and their analogues, and analyzed the results. E. F. R. P., D. D., S. B. B. and S. R. W. wrote and reviewed the manuscript. S. B. B., B. W. and S. R. W. supervised the project and acquired funding. T. W. performed XRD measurements. All authors discussed and commented on the manuscript.

Conflicts of interest

The authors declare no conflicts of interest.

Data availability

The data supporting this article have been included as part of the supplementary information (SI). It contains detailed protocols, characterization of starting materials and products as well as spectra. Supplementary information is available. See DOI: **https://doi.org/10.1039/d6qo01360k**.

CCDC 2581959 contains the supplementary crystallographic data for this paper.[66]

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