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Witkop–Winterfeldt oxidation using electrochemically generated *para*-periodate

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We report a simple protocol for the Witkop–Winterfeldt oxidation of tetrahydrocarbazoles and a tetrahydro- γ -carboline using electrochemically generated *para*-periodate ($\text{Na}_3\text{H}_2\text{IO}_6$). A substrate scope with 17 examples and yields up to 87%, including successful 20- and 40-fold scale-ups, together with periodate recovery and reuse, demonstrates the feasibility of this novel one-pot/one-step protocol.

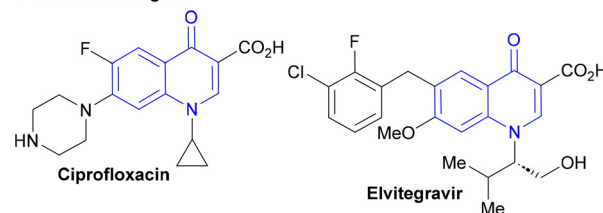
Introduction

The 4-quinolone scaffold is an important structural motif found in many synthetic drugs such as fluoroquinolones (*e.g.*, ciprofloxacin, levofloxacin, and marbofloxacin), a class of broad-spectrum antibacterial agents.¹ Other prominent examples of 4-quinolones include elvitegravir,² an integrase inhibitor used to treat human immunodeficiency virus (HIV) infection, and ivacaftor,³ a therapeutic agent approved for cystic fibrosis. Although less abundant in nature than the 2-quinolone class, the 4-quinolone scaffold is found in a few natural alkaloids with diverse biological activities (Fig. 1).^{4–11} For instance, the alkaloid Waltherione C, reported in several *Malvaceae* species such as *Waltheria indica*, *Melochia umbellata*, and *Melochia odorata*, exhibits notable biological activities including antitrypanosomal effects against *Trypanosoma cruzi*,⁶ cytotoxicity toward P-388 murine leukemia cells,⁷ and significant *in vitro* anti-HIV activity.⁸ Interveninol,⁹ isolated from the cultured broth of *Nocardia* sp. ML96-86F2, displays both anti-tumor and anti-*Helicobacter pylori* activities. Punarnavine¹⁰ (also known as Lunamarine), obtained from *Boerhaavia diffusa* (Nyctaginaceae), possesses immunomodulatory and anti-

angiogenic properties. Ervaoffline J,¹¹ an iboga-type alkaloid, was recently isolated from the aerial parts of *Ervatamia officinalis*. Therefore, due to the versatility of the quinolone skeleton, the development of efficient synthetic methods for its synthesis and/or structural modification is of great interest in both organic and medicinal chemistry.

Oxidative cleavage of aromatic rings is a process that occurs frequently in Nature (*e.g.*, the enzymatic oxidation of tryptophan to *N*-formylkynurenine).¹² In 1951, Witkop reported the first chemical oxidative cleavage of the C2–C3 double bond in indoles using catalytic Pt/O₂ oxidation as well as autoxidation.¹³ Subsequently, various oxidants such as chromic oxide,¹⁴ peracids (*e.g.*, *m*-CPBA),¹⁴ sodium *meta*-periodate (NaIO_4),¹⁴ ozone,¹⁴ oxone-halide,¹⁵ H₂O₂/UHP,¹⁶ and singlet oxygen¹⁴ have been examined for the Witkop oxidation. Remarkably, Winterfeldt demonstrated that treatment with *t*-BuOK/O₂¹⁷ or NaH/O₂¹⁸ in DMF directly converts 2,3-disubstituted indole derivatives, such as 1,2,3,4-tetrahydro- β -carbolines, into quinolones through a one-pot Witkop-oxidation/Camps-cyclization sequence (Scheme 1a, A–B).

4-Quinolone drugs



4-Quinolone natural products

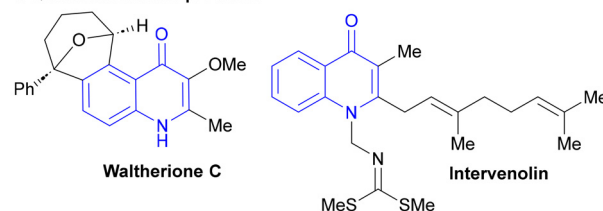
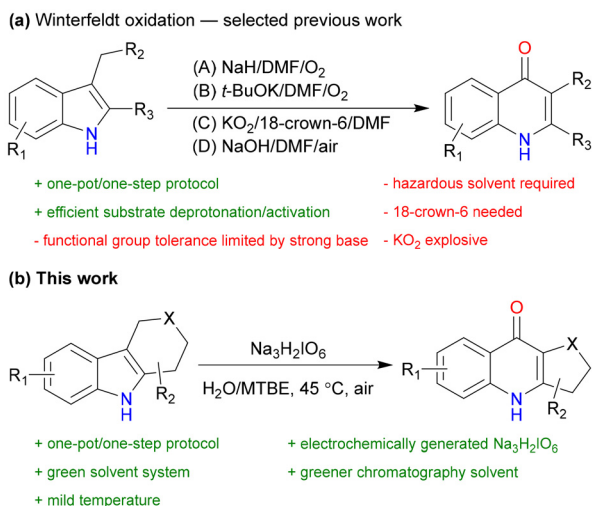


Fig. 1 Selected 4-quinolone involving drugs and natural products.

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Scheme 1 Synthesis of quinolones *via* the Winterfeldt oxidation and a novel Na₃H₂IO₆-mediated approach.

Subsequently, Speier introduced the weakly basic potassium superoxide (KO₂) as an oxidizer for the conversion of indoles to quinolones (Scheme 1a, C).¹⁹ In 2003, Jiang and co-workers extended this protocol to β -carboline, including acyl-substituted derivatives, demonstrating that KO₂ can overcome the strong-base limitation of the original Winterfeldt protocol (Scheme 1a, C).²⁰ Although effective, the KO₂ method uses 18-crown-6 in the hazardous solvent DMF together with a highly reactive and potentially explosive oxidizer, making scale-up challenging, even though it has been reported on a 200 g scale.²⁰ In 2012, Hu and co-workers demonstrated that the Winterfeldt oxidation of tetrahydro- γ -carbolines to dihydropyrrolo[3,2-*b*]quinolones could be achieved using NaOH in DMF at room temperature under an air atmosphere (9 examples, 38–94% yields) (Scheme 1a, D).²¹ While this protocol avoids the use of strong bases such as NaH and *t*-BuOK, as well as the KO₂/18-crown-6 system, it still relies on DMF as the reaction medium. More recently, photocatalytic approaches for Witkop–Winterfeldt-type transformations have also been reported under mild conditions (visible-light irradiation, room temperature, and an air or O₂ atmosphere).^{22,23} In 2020, Brasholz and co-workers described a catalytic cascade singlet oxygenation of tetrahydrocarbazoles and perhydrocyclohepta[*b*]indoles to afford chiral tricyclic perhydropyrido- and perhydroazepino[1,2-*a*]indoles, using chrysazine (1,8-dihydroxyanthraquinone) or Rose Bengal as a photocatalyst.²² In 2021, Yadav and co-workers achieved the oxidative functionalization of substituted tetrahydrocarbazoles and cyclohepta[*b*]indoles to fused quinolones (7 examples, 16–53% yields) in the presence of a crude chlorophyll extract as a photocatalyst and SDS as a surfactant.²³

Electrochemical synthesis ex-cell or in-cell proceeds under relatively mild and safe reaction conditions.^{24,25} Electricity, preferably from renewable sources,^{26,27} can be employed as a controllable and inherently clean redox tool. Periodate, a high-

performance platform oxidizer,²⁸ has been electrochemically generated^{29,30} and recycled³¹ by our group at a boron-doped diamond anode,^{32–35} with successful applications in lignin degradation³⁶ and in the final oxidative step of levetiracetam and sulfoximine synthesis.^{37,38} Motivated by these advances, we proposed the use of the electrochemically accessible “green” oxidizer sodium *para*-periodate (Na₃H₂IO₆) for the Witkop–Winterfeldt oxidation (Scheme 1b). While its direct use in organic synthesis is rare in the literature, owing to limited solubility³⁹ and the common practice of generating NaIO₄ *in situ* under acidic conditions, we envisioned that this oxidation might proceed efficiently in aqueous Na₃H₂IO₆ solution, obviating the need for additional base, surfactants, or a phase-transfer reagent such as 18-crown-6.

Results and discussion

To test our hypothesis, we selected 2,3,4,9-tetrahydrocarbazole (**1a**) as a model substrate to investigate the Witkop–Winterfeldt oxidation with sodium *para*-periodate. Initial experiments with **1a** revealed that the reaction proceeds in water as the sole solvent, and quinolone **2a** was obtained in 62% NMR yield after 240 h (see the SI). Using a design-of-experiments (DoE) study (2⁴ design plan, see the SI), the concentration of **1a** (*c*, 0.01–0.03 M), the equivalents of Na₃H₂IO₆ (1–3 equiv.), the temperature (*T*, 50–80 °C), and the reaction time (*t*, 2–15 h) were investigated. Within the studied design space, the highest yield obtained for **2a** was 47% NMR yield (Table 1, entry 1).

Table 1 Optimization of reaction conditions^a

Entry	Solvent	<i>T</i> (°C)	<i>t</i> (h)	Yield ^b (%)
1 ^c	H ₂ O	50	15	47
2	H ₂ O	40	24	31
3	H ₂ O	40	48	62
4	H ₂ O/MTBE (4 : 1)	40	72	78
5 ^d	H ₂ O/MTBE (4 : 1)	40	72	91 (85)
6 ^e	H ₂ O/MTBE (4 : 1)	40	72	69
7	H ₂ O/MTBE (4 : 1)	45	48	87
8 ^f	H ₂ O/MTBE (4 : 1)	45	48	81
9 ^{f,g}	H ₂ O/MTBE (4 : 1)	50	24	49
10	H ₂ O/MTBE (5 : 1)	45	48	87 (83)
11 ^h	H ₂ O/MTBE (5 : 1)	45	48	n.d. (78)
12 ⁱ	H ₂ O/MTBE (5 : 1)	45	48	n.d. (69)

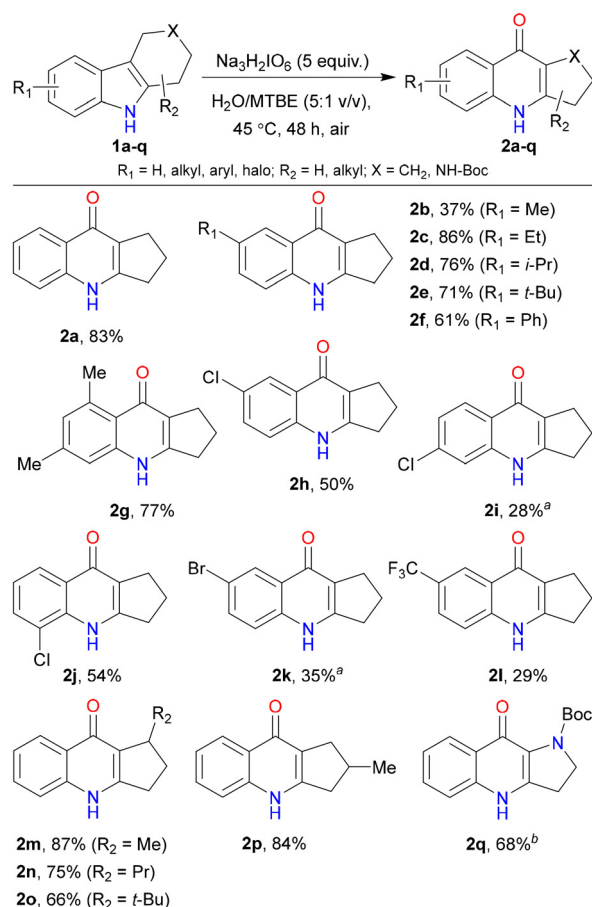
^a Reaction conditions: **1a** (0.4 mmol, 1 equiv., 0.01 M), Na₃H₂IO₆ (5 equiv.), solvent (40 mL), air. ^b Determined by ¹H NMR with 1,3,5-trimethoxybenzene as the internal standard. ^c 3 equiv. of Na₃H₂IO₆.

^d Fresh electrochemically synthesized Na₃H₂IO₆. ^e Commercially available Na₃H₂IO₆. ^f Under an oxygen atmosphere. ^g Reaction was conducted using a condenser to minimize MTBE loss. ^h At 0.02 M of **1a**. ⁱ At 0.03 M of **1a**. The isolated yields are given in parentheses. n.d.: not determined.

Analysis of the DoE results revealed reaction time (t) as the most influential factor, and optimization was pursued *via* a linear screening approach at a fixed substrate concentration (0.01 M), varying the amount of $\text{Na}_3\text{H}_2\text{IO}_6$ (3–5 equiv.) and temperature (40–50 °C). Performing the reaction in H_2O at 40 °C with 5 equivalents of $\text{Na}_3\text{H}_2\text{IO}_6$ gave a lower NMR yield of **2a** (31%) after 24 h (entry 2), which increased to an acceptable 62% after 48 h (entry 3). Reaction in an $\text{H}_2\text{O}/\text{MTBE}$ (4 : 1, v/v) solvent system with an extended reaction time of 72 h afforded **2a** in 78% NMR yield (entry 4). Notably, fresh electrochemically synthesized $\text{Na}_3\text{H}_2\text{IO}_6$ furnished **2a** in 91% NMR yield, with an 85% isolated yield (entry 5). In contrast, the commercially available $\text{Na}_3\text{H}_2\text{IO}_6$ provided **2a** in a 69% NMR yield under identical conditions (entry 6). At 45 °C, **2a** was produced in 87% NMR yield after 48 h (entry 7), while a decreased yield of 81% was observed under an oxygen atmosphere (entry 8). Conducting the reaction at 50 °C under oxygen furnished **2a** in 49% yield after 24 h (entry 9). We then evaluated the effect of different $\text{H}_2\text{O}/\text{MTBE}$ solvent ratios on the yield of **2a** at 45 °C (see the SI). Reducing the MTBE content clearly favored the reaction, and $\text{H}_2\text{O}/\text{MTBE}$ (5 : 1, v/v) gave **2a** in 87% NMR yield and 83% isolated yield after 48 h (entry 10). Monitoring the reaction kinetics revealed that the yield reached 93% at 60 h (see the SI), a modest increase over the 87% obtained at 48 h. Finally, the effect of substrate concentration was investigated. At an intermediate concentration of 0.02 M, the reaction furnished an isolated yield of 78% for quinolone **2a** (entry 11), while increasing the concentration to 0.03 M led to a lower isolated yield of 69% (entry 12), consistent with the DoE results (see the SI), which showed a decrease in yield at higher concentrations.

Scope and limitations

With the optimized reaction conditions in hand, we explored the reaction scope using various tetrahydrocarbazole derivatives, as well as N-protected tetrahydro- β - and - γ -carbolines (Scheme 2, and SI). Unsubstituted (**2a**) and 2,3,4,9-tetrahydrocarbazoles bearing 6-alkyl (**2c–2e**), 6-aryl (**2f**), or 5,7-dialkyl (**2g**) substituents on the phenyl ring furnished good isolated yields (61–86%), whereas the 6-methyl analogue (**2b**) gave only 37% yield, likely due to undesired oxidation of the 6-methyl group by periodate. Substrates bearing chloro (**2h–j**) or bromo substituents (**2k**) on the phenyl ring were well tolerated under the reaction conditions and provided the corresponding quinolones in 28–54% yields. Interestingly, a trifluoromethyl derivative (**2l**) was successfully engaged under the same conditions and afforded the product in 29% yield. As expected, substrates bearing strongly electron-withdrawing groups,⁴⁰ such as nitro and methyl ester, remained unreactive. 3-Alkyl- (**2m–2o**) and 2-alkyl-substituted (**2p**) tetrahydrocarbazoles were also compatible with our protocol and resulted in the desired products in good yields (66–87%). Regarding the C3-substituted tetrahydrocarbazoles (Me, Pr, *t*-Bu), the yields decreased with increasing steric bulk (87, 75, and 66%, respectively). Since the sequence involves both Witkop oxidation and Camps cyclization, any potential Thorpe–Ingold acceleration in the cycliza-



Scheme 2 Substrate scope of the Witkop–Winterfeldt oxidation with $\text{Na}_3\text{H}_2\text{IO}_6$. Reaction conditions: **1a–1q** (0.4 mmol, 1 equiv., 0.01 M), $\text{Na}_3\text{H}_2\text{IO}_6$ (5 equiv.), $\text{H}_2\text{O}/\text{MTBE}$ (5 : 1 v/v, 40 mL), 45 °C, 48 h, air. ^aReaction conducted for 72 h. ^b8 equiv. of NaOH added after 48 h; stirred 1 h further.

tion step may be masked by the overall reaction efficiency. Unfortunately, *N*-Boc-protected 1,2,3,4-tetrahydro- β -carboline was unreactive under our optimized reaction conditions. In contrast, the γ -carboline congener (**2q**) reacted smoothly and gave the product in 68% yield, indicating that our method is applicable to other derivatives. *N*-Alkylated substrates, such as 9-methyl- and 9-ethyl-2,3,4,9-tetrahydro-1*H*-carbazole, showed limited compatibility. The desired quinolones were obtained in 12% NMR yield and only trace amounts, respectively (see the SI). Further optimization of the reaction conditions would therefore be required for these *N*-alkylated substrates. Interestingly, our protocol also worked with a stoichiometric amount of $\text{Na}_3\text{H}_2\text{IO}_6$ in the presence of 8 additional equivalents of NaOH. These new reaction conditions were tested on selected substrates from the scope. The 6-bromo (**2k**, 35% vs. 31%) and 6-trifluoromethyl (**2l**, 29% vs. 27%) tetrahydrocarbazole derivatives provided yields comparable to those under the original conditions (5 equiv. of $\text{Na}_3\text{H}_2\text{IO}_6$), with no improvement in reaction time or overall yield. In contrast, the 6-methyl tetrahydrocarbazole derivative (**2b**, 37% vs. 20%) and the

N-Boc-protected tetrahydro- γ -carboline derivative (**2q**, 62% vs. 38%) afforded lower yields under the new conditions (see the SI, Table S16).

Gram-scale synthesis

To demonstrate the applicability of our protocol, quinolone **2a** was synthesized on a gram scale at 0.02 M concentration, with a 20-fold scale-up under the original conditions (5 equiv. of $\text{Na}_3\text{H}_2\text{IO}_6$) and a 40-fold scale-up under the new conditions (1 equiv. of $\text{Na}_3\text{H}_2\text{IO}_6$ with 8 equiv. of NaOH) (see the SI). Herein, a minor decline in yield was observed compared to the millimolar scale: 64% vs. 78% for the original, and 63% (72% brsm) vs. 78% for the new conditions. Importantly, the periodate employed in both the small-scale and scale-up reactions under the original conditions was recovered by simple filtration, likely as the disodium salt ($\text{Na}_2\text{H}_3\text{IO}_6$),³⁹ and reused in place of fresh trisodium salt by adding the necessary equivalents of NaOH, without any decrease in yield (see the SI). During the scale-up experiments, the known intermediate keto-lactam **3** and a spiroindolinone side product **4**, both previously observed at the micromolar scale, were successfully isolated and characterized (see the SI).

Mechanistic investigations

To gain insight into the possible reaction mechanism, a series of control experiments was conducted using the model substrate (see the SI). Only trace amounts of **2a** were detected in the absence of $\text{Na}_3\text{H}_2\text{IO}_6$, even with 10 equivalents of NaOH. Quinolone **2a** was obtained in comparable yields under air (87%) and argon (86%), indicating that molecular oxygen is not required. Using NaIO_4 instead of $\text{Na}_3\text{H}_2\text{IO}_6$ gave only the keto-lactam **3** (73% NMR yield) and no **2a** (entry 4), consistent with NaIO_4 promoting the initial cleavage (Witkop oxidation), while a base is required for product formation (Camps cyclization). Indeed, 1 equivalent of NaIO_4 with 10 equivalents of NaOH afforded **2a** in 90% NMR yield. With 8 equivalents of base, 20 mol% $\text{Na}_3\text{H}_2\text{IO}_6$ was sufficient to afford **2a** in 80% yield, demonstrating that the reaction can proceed with a sub-stoichiometric amount of oxidant. Interestingly, when conducted in a degassed solvent (30 min, Ar) under a pure Ar atmosphere, the yield of **2a** decreased to 37–49%, suggesting that oxygen may play a role under these sub-stoichiometric conditions. These conditions under Ar were also employed in control experiments with radical scavengers. Addition of *t*-BuOH or NaN_3 had little effect, affording **2a** in 89% and 74% yields, respectively, although NaN_3 also produced 15% of an unidentified side product. In contrast, the presence of *p*-benzoquinone completely suppressed the formation of **2a**, and the tetrahydrocarbazole was recovered in 98%. Given the known reactivity of *p*-benzoquinone under strongly alkaline conditions, this result alone does not establish the involvement of superoxide. Based on these results and the previous literature,^{14,19} including reports demonstrating superoxide anion formation in KIO_4/KOH solutions by the spontaneous reaction of IO_4^- with OH^- under both aerobic and anaerobic conditions,⁴¹ we tentatively propose a superoxide-mediated

pathway in our one-pot Winterfeldt protocol. However, the possible involvement of other reactive oxygen species, such as singlet oxygen, as well as other periodate-derived oxidizing species, cannot be excluded (see the SI for the proposed mechanism).

Conclusions

In summary, we have developed a simple protocol for the synthesis of quinolones and a dihydropyrrolo[3,2-*b*]quinolone *via* the direct use of electrochemically generated *para*-periodate. The synthetic utility of this transformation was demonstrated in a substrate scope of 17 examples with yields up to 87%, as well as in gram-scale synthesis and periodate recovery/reuse. These findings underscore the potential of this method as a practical approach for accessing these scaffolds, while also allowing us to rethink molecular connections using periodate.

Author contributions

Dr Aloisio de A. Bartolomeu: investigation (lead), methodology (lead), project administration (supporting), and writing—original draft (lead). Dr Thomas Weyhermüller: investigation (supporting; crystallographic measurements and interpretation of X-ray data). Prof. Dr Siegfried R. Waldvogel: conceptualization (lead), supervision (lead), project administration (lead), funding acquisition (lead), and writing—review & editing (lead).

Conflicts of interest

There are no conflicts to declare.

Data availability

The data underlying this study are available in the published article and its supplementary information (SI). Supplementary information: detailed experimental procedures; characterization data; ^1H , ^{13}C , and ^{19}F NMR spectra for all new compounds; HPLC calibration curves; Design of Experiments (DoE) study; crystallographic data; and additional supporting tables and figures related to reaction optimization and control experiments. See DOI: <https://doi.org/10.1039/d6ob01157h>.

CCDC 2487306 and 2487307 contain the supplementary crystallographic data for this paper.^{42a,b}

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