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# Silver-Catalyzed Radical Tandem Cyclization for the Synthesis of 3,4-Disubstituted Dihydroquinolin-2(1*H*)-ones

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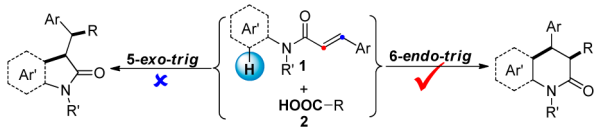
Supporting Information


**ABSTRACT:** A silver-catalyzed tandem decarboxylative radical addition/cyclization of *N*-arylcinnamamides with aliphatic carboxylic acids is reported. This method affords a novel and straightforward route to various 3,4-disubstituted dihydroquinolin-2(1*H*)-ones in aqueous solution.

Many substituted 3,4-dihydroquinolin-2(1*H*)-ones have attracted considerable attention in view of their cardiovascular, anti-inflammatory, and phosphodiesterase inhibitory activities.[1] Moreover, 3,4-dihydroquinolin-2(1*H*)-ones are a versatile structural unit for the preparation of other important pharmaceuticals and natural products such as 1,2,3,4-tetrahydroquinolines and quinolin-2(1*H*)-ones.[2] Thus, the development of a straightforward and highly efficient method for the construction of 3,4-dihydroquinolin-2(1*H*)-one is highly desirable. A general method for the preparation of a 3,4-dihydroquinolin-2(1*H*)-one system is the Friedel−Crafts cyclization requiring superacidity or stoichiometric amounts of metals.[3] Recently, several alternative methods such as Pd-catalyzed sequential Heck reduction−cyclization reactions, Pd-catalyzed cyclopropane ring expansions, Rh-mediated 1,4-additions of the boronic acid to enone, sequential Ugi/acrylanilide [6$\pi$]-photocyclizations, intermolecular tandem reactions, Mn-mediated intramolecular cyclizations, and Pd-catalyzed cyclocarbonylations reactions have been developed.[4] However, for 3,4-disubstituted dihydroquinolin-2(1*H*)-one, these processes still suffer from such drawbacks as the need for complicated starting materials[4g] or multistep synthesis.[4a,b,d,f] So far, radical reactions have widespread applications in organic synthesis, especially in cyclization reactions for the prepration of valuable heterocycles.[5] Recently, the synthesis of substituted oxindoles[6] and construction of multiple C−C/N bonds through radical cyclization to access 3,3-disubstituted oxindoles have attracted special attention.[7] These methods demonstrated that this kind of radical cyclization is a very powerful tool to construct oxindole's framework. Recent significant progress in radical cyclization inspired us to extend this highly efficient method to another prominent structural motif, 3,4-dihydroquinolin-2(1*H*)-one, because a general and tandem approach for the construction of its six-membered ring remains rare,[4f,g] in contrast to oxindole's synthesis. Herein we disclose the silver-catalyzed tandem decarboxylation and C−H functionalization in aqueous solution which provides a novel and simple protocol for valuable 3,4-disubstituted dihydroquinolin-2(1*H*)-ones (Scheme 1).

**Scheme 1. Ring Closure for 3,4-Dihydroquinolin-2(1*H*)-one**




We started our model reaction by investigating *N*-methyl-*N*-phenylcinnamamide **1a** and pivalic acid **2a** for the optimization of reaction conditions (Table 1). With 20 mol % $AgNO_3$ as the catalyst, the reaction of **1a** with pivalic acid (2.0 equiv) in aqueous solution ($CH_3CN/H_2O$) failed to provide the desired product at room temperature (Table 1, entry 1). To our delight, a 37% product yield was obtained when the temperature was increased to 70 °C (Table 1, entry 2). The expected product was obtained in 69% yield when the temperature was further improved to 100 °C with a 20% catalyst loading (Table 1, entry 4). The product yield decreased when the catalyst loading was reduced (Table 1, entries 5−7). However, no more conversion of **1a** to **3a** was observed with 30 mol % $AgNO_3$ as the catalyst at 110 °C (Table 1, entry 8). Other Ag(I) salts such as $AgBF_4$, $Ag_2O$, and $Ag_2CO_3$ showed similar catalytic activity under the same conditions (Table 1, entries 9−11). Investigation of other oxidants such as $(NH_4)_2S_2O_8$, TBHP, oxone, DTBP, and $O_2$ for this transformation showed that $K_2S_2O_8$ was the best choice (Table 1, entries 12−16). The transformation did not proceed in such aqueous solutions as DCE/$H_2O$, $CH_2Cl_2/H_2O$, EtOH/$H_2O$,

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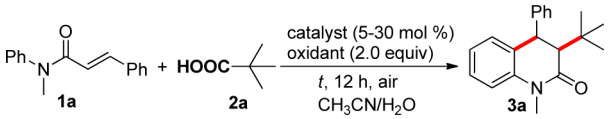
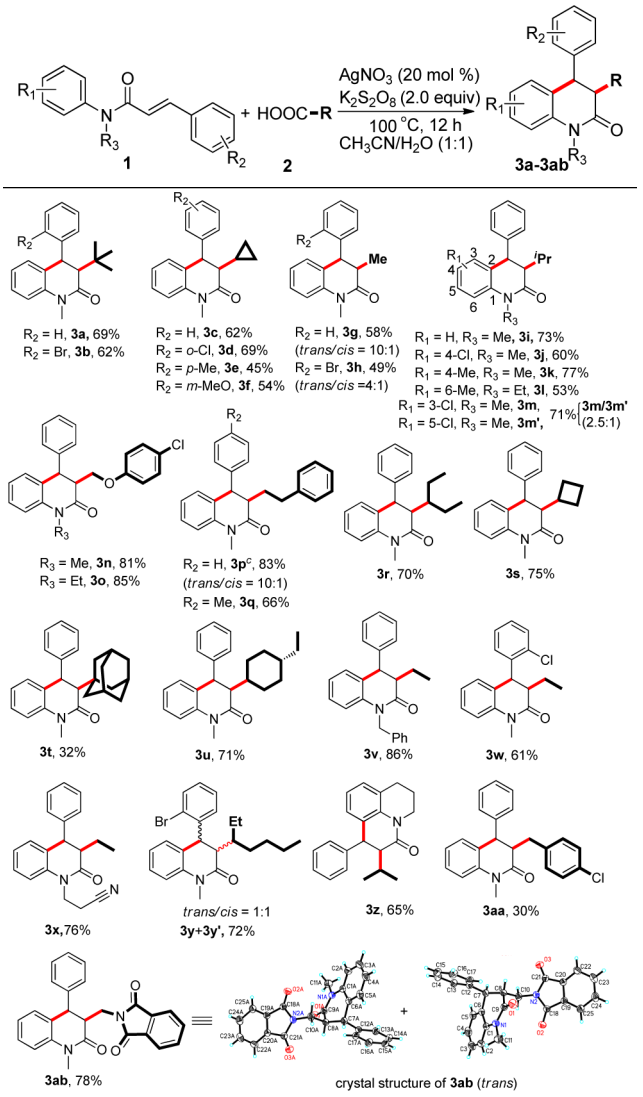
**Table 1. Screening of Reaction Conditions**[a]




| entry | catalyst (mol %) | oxidant | $t$ (°C) | yield[b] |
|---|---|---|---|---|
| 1 | $AgNO_3$ (20) | $K_2S_2O_8$ | rt (18) | N.R. |
| 2 | $AgNO_3$ (20) | $K_2S_2O_8$ | 70 | 37 |
| 3 | $AgNO_3$ (20) | $K_2S_2O_8$ | 90 | 65 |
| 4 | $AgNO_3$ (20) | $K_2S_2O_8$ | 100 | 69 (35[c]) |
| 5 | $AgNO_3$ (5) | $K_2S_2O_8$ | 100 | 22 |
| 6 | $AgNO_3$ (10) | $K_2S_2O_8$ | 100 | 29 |
| 7 | $AgNO_3$ (15) | $K_2S_2O_8$ | 100 | 47 |
| 8 | $AgNO_3$ (30) | $K_2S_2O_8$ | 110 | 66 |
| 9 | $AgBF_4$ (20) | $K_2S_2O_8$ | 100 | 57 |
| 10 | $Ag_2O$ (20) | $K_2S_2O_8$ | 100 | 48 |
| 11 | $Ag_2CO_3$ (20) | $K_2S_2O_8$ | 100 | 60 |
| 12 | $AgNO_3$ (20) | $(NH_4)_2S_2O_8$ | 100 | 55 (42[c]) |
| 13 | $AgNO_3$ (20) | oxone | 100 | N.R. |
| 14 | $AgNO_3$ (20) | TBHP | 100 | N.R. |
| 15 | $AgNO_3$ (20) | DTBP | 100 | N.R. |
| 16 | $AgNO_3$ (20) | $O_2$ | 100 | N.R. |
| 17[d] | $AgNO_3$ (20) | – | 100 | N.R. |
| 18[e] | – | $K_2S_2O_8$ | 100 | N.R. |
| 19[f] | $AgNO_3$ (20) | $K_2S_2O_8$ | 100 | N.R. (N.R[h]) |
| 20[g] | $AgNO_3$ (20) | $K_2S_2O_8$ | 100 | trace (N.R[i]) |
| 21[j] | $AgNO_3$ (20) | $K_2S_2O_8$ | 100 | trace |
| 22 | Cu (20) | $K_2S_2O_8$ | 100 | N.R. |
| 23 | CuCl (20) | $K_2S_2O_8$ | 100 | N.R. |
| 24 | $Cu(OAc)_2$ (20) | $K_2S_2O_8$ | 100 | N.R. |
| 25 | $FeSO_4$ (20) | $K_2S_2O_8$ | 100 | N.R. |
| 26 | $FeCl_3$ (20) | $K_2S_2O_8$ | 100 | N.R. |

[a]Reaction conditions: **1a** (1.0 mmol), **2a** (2.0 mmol), $CH_3CN/H_2O$ (3/3 mL), under air atmosphere, 12 h, 100 °C. [b]Isolated yield. [c]DMF/$H_2O$ as solvent. [d]Without catalyst. [e]Without oxidant. [f]$CH_2Cl_2/H_2O$ as solvent. [h]DCE/$H_2O$ as solvent. [g]Dioxane/$H_2O$ as solvent. [i]EtOH/$H_2O$ as solvent. [j]Acetone/$H_2O$ as solvent. N.R. = No Reaction.

**Table 2. Tandem Radical Addition/Cyclization of 1 and 2**[a,b]




[a]Reaction conditions: **1** (1.0 mmol), **2** (2.0 mmol), $AgNO_3$ (20 mol %), $K_2S_2O_8$ (2.0 mmol), $CH_3CN/H_2O$ (3/3 mL), 100 °C, 12 h. [b]Isolated yield. [c]Crystal structure in Supporting Information.

dioxane/$H_2O$, and acetone/$H_2O$ (Table 1, entries 19–21). We further explored other metals, such as copper powder, CuCl, $Cu(OAc)_2$, $FeSO_4$, and $FeCl_3$, as catalysts, but no reaction proceeded (Table 1, entries 22–26).

With the optimized conditions in hand, we then set out to explore the scope and limitations of the 6-*endo* radical cyclization, and the results are summarized in Table 2. In general, electron-donating or -withdrawing groups on aniline at the *ortho*, *meta*, and *para* positions did not affect the efficiency of the reaction, affording the desired products in moderate yields (Table 2, **3i–m**). Substituents such as Cl, Br, Me, and OMe at the *ortho*, *meta*, and *para* positions on the other aromatic ring of the substrate **1** were well tolerated in the process of ring closure under the optimal conditions and the corresponding products were obtained in moderate yields (Table 2, **3b–f**). The reaction still proceeded well when different *N*-protected groups (e.g., Me, Et, Bz, and $CH_3CH_2CN$) of substrate **1** were used (Table 2, **3m**, **3n–o**, **3v**, and **3x**). After investigating $R_1$, $R_2$, and $R_3$ of substrate **1**, aliphatic carboxylic acids were further tested under the optimal conditions. Various primary, secondary, and tertiary aliphatic carboxylic acids underwent efficient intermolecular ring closure to provide the expected products **3a–z** and **3aa–3ab** in moderate to good yields. With this method, methyl or ethyl could be introduced into the 3-position of 3,4-dihydroquinolin-2(1*H*)-one in one step only using cheap AcOH or propionic acid instead of MeLi or EtLi as substrates (Table 2, **3g–h** and **3v–x**).[8] Primary carboxylic acids containing aromatic ringlike 3-phenylpropanoic acid and 2-(4-chlorophenyl)-acetic acid showed different reactivity; products **3p** and **3aa** were obtained in 83% and 30% yields respectively. In addition, primary carboxylic acids containing heteroatoms (O, N) at adjacent positions of the carboxyl group also displayed high reactivity in this transformation (Table 2, **3n–o** and **3ab**). As for secondary alkyl acids, 4-ethylcyclohexanecarboxylic acid worked well in the same fashion and gave the corresponding product in 71% yield (Table 2, **3u**). Moreover, cyclopropanecarboxylic acid and cyclobutanecarboxylic acid were also successfully converted to the desired products without ring opening (Table 2, **3c–f** and **3s**). Interestingly, product **3y** was isolated in 72% yield as a 1:1 ratio of *cis*/*trans* isomers when 2-ethylhexanoic acid was explored as the substrate (Table 2, **3y** and **3y′**). However, in comparison with pivalic acid, 1-adamantanecarboxylic acid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which is more congested gave a low yield under the same conditions (Table 2, **3t**).

The structure of product **3b** was clearly confirmed by single-crystal X-ray crystallographic analysis (Figure 1). Obviously, product **3b** is a *trans*-form which contains a six-membered ring.

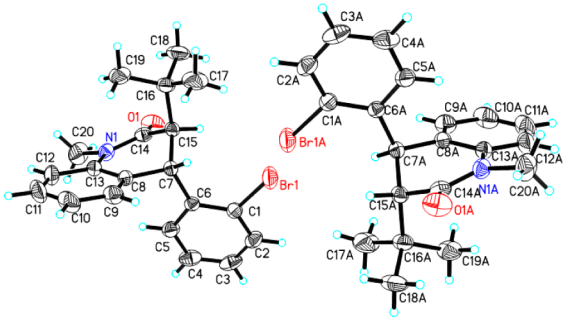
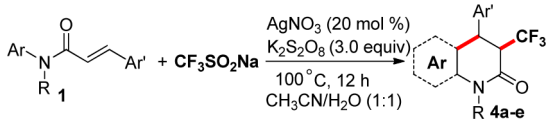
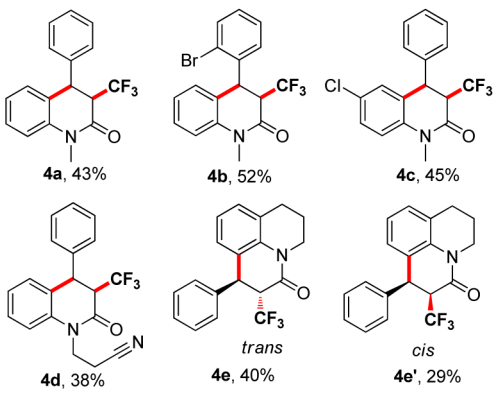



**Figure 1.** Molecular structure of **3b**.

Encouraged by this finding, we continued to explore the *trans*-selective 6-*endo* radical cyclization leading to 3,4-disubstituted dihydroquinolin-2(1*H*)-one derivatives. Trifluoromethylated heteroaryl compounds represent an important structural motif in pharmaceuticals and advanced organic materials.[9] Thus, two alkyl acids containing the $CF_3$ group were subjected to the reaction. Unfortunately, both TFA and 3,3,3-trifluoropropanoic acid failed to provide any of the desired products. The reason for this is still not clear at present. However to our delight, when $CF_3SO_2Na$ (Langlois reagent) was employed as the source of the $CF_3$ radical,[10] the reaction took place under the slightly modified conditions, in which the $CF_3$ group was introduced into the 3-position of 3,4-dihydroquinolin-2(1*H*)-one successfully (Table 3). An investigation into different substituents ($R_{1-3}$) of substrate **1** showed that the transformation still proceeded well, affording the desired trifluoromethylated products **4a**–**4e** without much difference in yields. Products **4e** and **4e′** were isolated as two stereoisomers in a 4:3 ratio under the same reaction conditions.

**Table 3. Study of Trifluoromethylation in the Cyclization**[a]

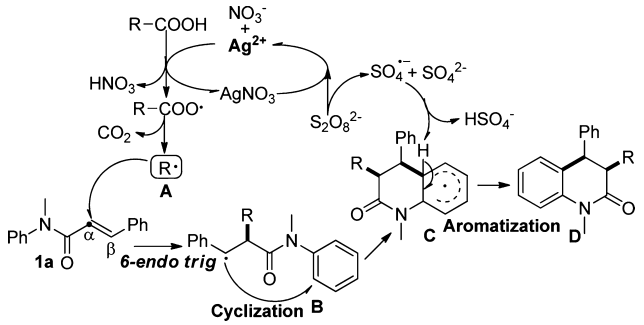



[a]Reaction conditions: **1** (1.0 mmol), $CF_3SO_2Na$ (3.0 mmol), $AgNO_3$ (20 mol %), $K_2S_2O_8$ (3.0 mmol), $CH_3CN/H_2O$ (3/3 mL), 100 °C, 12 h.

A postulated mechanism is described in Scheme 2. Initially, $Ag^+$ is oxidized by the persulfate anion ($S_2O_8^{2-}$) to generate the

**Scheme 2. Postulated Mechanism for the Radical Tandem Cyclization**




$Ag^{2+}$ cation and sulfate radical anion. Then, the $Ag^{2+}$ cation obtains a single electron from carboxylate to produce the carboxyl radical.[11] Quick decarboxylation of the carboxyl radical provides the corresponding alkyl radical **A**, followed by addition to the double bond of cinnamamide **1a**, thus leading to intermediate **B**. Intramolecular cyclization of **B** gives the intermediate **C**. Finally, intermediate **C** can be aromatized to the desired product **D** after hydrogen abstraction by the sulfate radical anion.

In summary, we have developed an unprecedented and highly efficient method for the preparation of biologically interesting 3,4-disubstituted dihydroquinolin-2(1*H*)-one derivatives by silver-catalyzed tandem decarboxylative radical addition/cyclization of *N*-arylcinnamamides with aliphatic carboxylic acids in aqueous solution. A significant feature of the novel protocol is the formation of a six-membered ring; meanwhile, it could be alkylated or trifluoromethylated. This approach represents one of the most straightforward routes to various functional 3,4-dihydroquinolin-2(1*H*)-one syntheses.

## ■ ASSOCIATED CONTENT

### Supporting Information

Experimental procedures, characterization of products, and copies of $^1H$ and $^{13}C$ NMR spectra are provided. This material is available free of charge via the Internet at http://pubs.acs.org.

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### Notes

The authors declare no competing financial interest.

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