

## Synthetic Methods

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Internationale Ausgabe: DOI: 10.1002/anie.201603809Aerobic Radical-Cascade Alkylation/Cyclization of  $\alpha,\beta$ -Unsaturated Amides: an Efficient Approach to Quaternary Oxindoles

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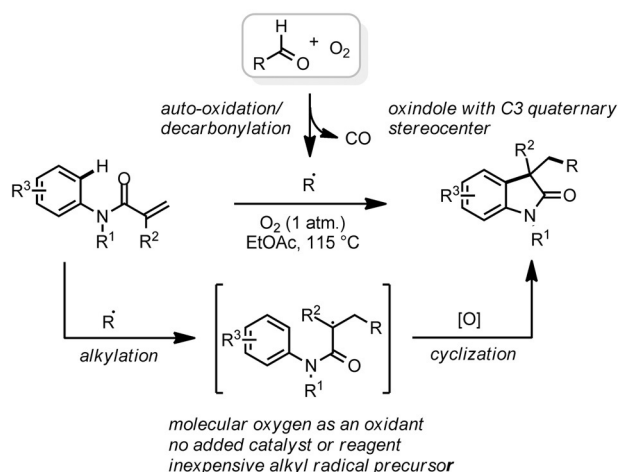
**Abstract:** An efficient method for the aerobic radical-cascade alkylation/cyclization of  $\alpha,\beta$ -unsaturated amides to afford functionalized oxindoles with a C3 quaternary stereocenter is described. The process is based on the generation of valuable alkyl radicals through sustainable aerobic C–H activation of aldehydes followed by decarbonylation using  $O_2$  as the sole oxidant. This method features a broad substrate scope, inexpensive alkyl radical precursors, and convenient reagents. Finally, the method was successfully applied to the synthesis of alkyl analogues of tetrahydrofuranoindoline and ( $\pm$ )-esermet-hole.

Cascade reactions initiated by intermolecular addition of a carbon-centered radical to an olefin enable the efficient construction of multiple carbon–carbon bonds in a single chemical operation with generally high functional-group compatibility.<sup>[1,2]</sup> The synthetic potential of this strategy has been well established in the synthesis of complex natural products.<sup>[3]</sup> However, a major disadvantage of this process is access to the free-radical intermediates, in particular alkyl radicals, which typically requires pre-functionalized substrates and undesirable reagents and/or reaction conditions.<sup>[2,3]</sup> Therefore, an environmentally friendly method for a free-radical-cascade reaction involving alkyl radicals generated from readily available precursors by a more convenient and efficient approach is still in great demand.

In our recent report on the C–H alkylation of heteroaromatic bases,<sup>[4]</sup> we demonstrated an efficient method for the generation of valuable alkyl radicals through aldehyde auto-oxidation. Despite this work, the scope of this simple alkylation technique needs to be further expanded with other radical acceptors to establish the synthetic power of the method. Hence, we became interested in developing an alkylation/cyclization cascade reaction with alkyl radicals generated through aerobic oxidation of an aldehyde, which may offer easy access to functionalized cyclic compounds under mild reaction conditions.

*N*-Arylacrylamides have recently received considerable attention as radical acceptors in developing free-radical-initiated addition/cyclization cascades to synthesize functionalized oxindole moieties,<sup>[5]</sup> since large numbers of bioactive natural products and pharmaceutical molecules contain an

oxindole scaffold.<sup>[6]</sup> Although, significant advances have been made in the construction of oxindoles with C3 quaternary stereocenters by intercepting alkyl radicals with *N*-arylacrylamides, the existing methods generally require a metal catalyst, stoichiometric oxidant, and/or toxic solvent.<sup>[7]</sup> As a consequence, the development of an efficient and economical method to such compounds under benign conditions would be highly desirable. It has been reported that the aerobic oxidation of aldehydes involves an acyl radical intermediate.<sup>[8]</sup> We thus hypothesized that an alkyl radical generated by aldehyde auto-oxidation under suitable reaction conditions.<sup>[9,10]</sup> The in situ generated alkyl radical is expected to react with *N*-alkyl-*N*-arylacrylamide to form a new carbon-centered radical intermediate that upon intramolecular cyclization with the aromatic ring would deliver the desired oxindole (Scheme 1). Herein, we report the successful



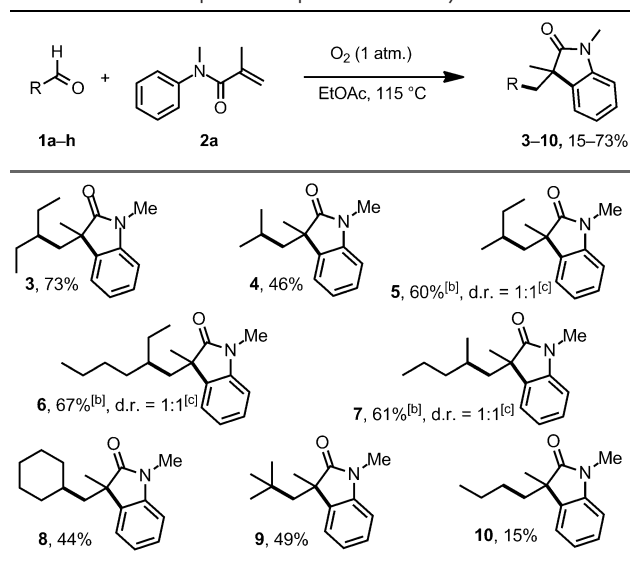
**Scheme 1.** An aerobic radical alkylation/cyclization cascade of *N*-alkyl-*N*-arylacrylamide.

realization of this approach to the preparation of various quaternary oxindoles with alkyl functionalities in good yields. The method employs inexpensive aldehydes as alkyl radical precursors,  $O_2$  as the sole oxidant, and ethyl acetate as a green solvent.

The concept presented in Scheme 1 was established from the results obtained from the reaction between 2-ethylbutanal **1a** and *N*-alkyl-*N*-arylacrylamide **2a** under various reaction conditions in the presence of  $O_2$  (1 atm.). The optimized reaction conditions revealed that the oxindole **3** could indeed be obtained in 73% yield of isolated product when using 8 equiv of **1a** and ethyl acetate as the solvent after 39 h (see

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**Table 1:** Reaction scope with respect to the aldehyde.<sup>[a]</sup>

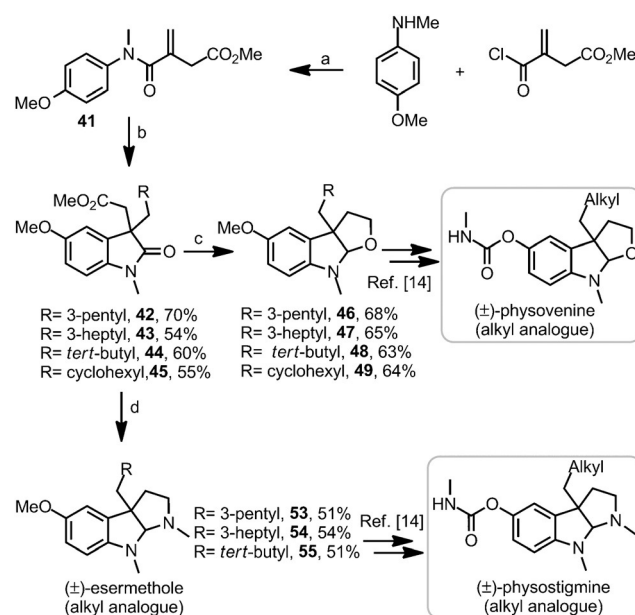
[a] All the reactions were carried out with amide **2a** (1.0 equiv), aldehydes **1a–h** (8–12 equiv), EtOAc (0.1–0.2 M) for 48 h. Yields of isolated products are given. [b] Combined yields are given. [c] The diastereoisomeric ratio (d.r.) was determined by <sup>1</sup>H NMR analysis of the sample.

the Supporting Information). With the optimized reaction conditions, the scope of the transformation was evaluated with different aliphatic aldehydes (Table 1). Symmetrically  $\alpha$ -substituted aldehydes furnished the corresponding 3,3'-dialkylated oxindoles **3** and **4** in good yields. Unsymmetrical  $\alpha$ -branched aldehydes reacted with equal efficiency, delivering the oxindoles **5–7** in a 1:1 mixture of two diastereoisomers. Cyclohexyl substituted oxindole **8** was obtained in moderate yield when the reaction was performed with cyclohexanecarboxaldehyde. Interestingly, the reaction enabled the synthesis of highly sterically demanding oxindole **9**, which has two alternative quaternary centers, in good yield when using pivaldehyde as the *tert*-butyl radical precursor. The reaction was found to be less efficient with a linear aldehyde, affording the desired product **10** in 15% yield.<sup>[11]</sup> The much lower reactivity of the linear aldehyde towards alkylation can be explained by the fact that decarbonylation of the primary acyl radical to form the corresponding alkyl radical is energetically less favorable.<sup>[9b]</sup>

We then directed our attention toward exploring the scope of this transformation with a broad range of *N*-alkyl-*N*-arylacrylamides with varying substituents on both the aromatic ring and the nitrogen center (Table 2). Substrates with a methyl substituent at any position of the aryl moiety exhibited good reactivity under the optimized reaction conditions (**11–16**). Varying the electronic properties of the substrate from electron-donating (methoxy) to electron-withdrawing (cyano, ester, trifluoromethyl) substituents made no considerable difference to the yields of the final products (**17–22**). Halide substituents at the *o*, *m*, and *p* positions of the aromatic ring were well tolerated in this transformation (**23–29**). Importantly, oxindole **30**, which bears a sensitive iodo functionality, could be prepared in 65% yield by this simple

method. For asymmetrical substrates with substituents at the *m* position of the aromatic ring, the formation of regioisomers was observed, with low selectivity. The reaction furnished the oxindole **31** in 65% yield when the benzene ring of the substrate was replaced with a naphthalene moiety. Importantly, various functionalized aza-oxindoles (**32–36**) were synthesized in moderate to good yields when using *N*-pyridine-substituted methacrylamide. Substrates with different *N* substituents, such as ethyl, benzyl, phenyl, and ester groups, could be converted into the desired products **37–40** in good yields. However, unprotected NH-methacrylamide was found to be completely ineffective for this transformation (see the Supporting Information).

The synthetic utility of this method was demonstrated in the preparation of alkyl-substituted tetrahydrofuranindoline and pyrrolidinoindoline moieties, which could be easily converted into alkyl analogues of naturally occurring alkaloids such as ( $\pm$ )-physoverine, and ( $\pm$ )-physostigmine. This family of alkaloids exhibit inhibitory activity against acetylcholinesterase and butyrylcholinesterase.<sup>[12]</sup> Furthermore, (–)-physostigmine has been used for the treatment of glaucoma and severe anticholinergic toxicity.<sup>[13]</sup> The synthesis was initiated by performing the key radical-cascade alkylation/cyclization of conjugated amide **41** (prepared as presented in Scheme 2) with various structurally diverse alde-



**Scheme 2.** Synthesis of alkylated tetrahydrofuranindolines and alkyl analogues of ( $\pm$ )-esermethole. Reaction conditions: a) Et<sub>3</sub>N, CH<sub>2</sub>Cl<sub>2</sub>, 12 h, RT, 67%; b) aldehydes (8.0 equiv), O<sub>2</sub> (1 atm.), EtOAc (1.0 mL, 0.2 M), 48 h, 115 °C; c) LiAlH<sub>4</sub> (4.0 equiv), THF, 1 h, RT; d) i. MeN-H<sub>2</sub>-EtOH (33%), 48 h, 70 °C, 79–92% (**50–52**); ii. LiAlH<sub>4</sub> (8.0 equiv), THF, 3 h, 70 °C. THF = tetrahydrofuran.

hydes, furnishing the desired products **42–45** in good yields (54–70%). Reduction of oxindoles **42–45** with LiAlH<sub>4</sub> delivered tetrahydrofuranindolines **46–49**, which can be easily converted into the ( $\pm$ )-physoverine analogues.<sup>[14]</sup> Furthermore, alkyl analogues of ( $\pm$ )-esermethole (**53–55**)

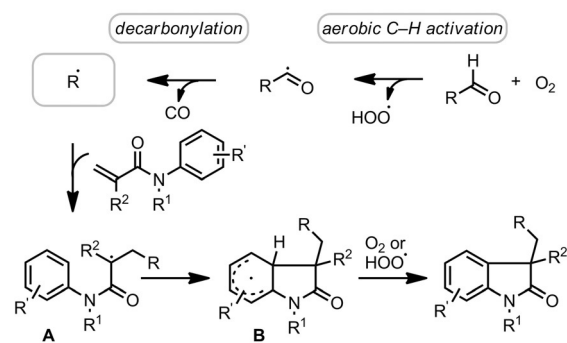
Table 2: Reaction scope with respect to the amide.

| $\text{R-CHO} + \text{R}^3\text{-C}_6\text{H}_4\text{-N(R}^1\text{)-C(=O)-CH(R}^2\text{)-CH}_3 \xrightarrow[\text{EtOAc, 115 } ^\circ\text{C}]{\text{O}_2 \text{ (1 atm.)}}$ <p>R = 3-pentyl, <b>1a</b>      <b>2b–2ae</b>      <b>11–40</b>, 32–81%</p> |  |  |  |  |  |
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[a] Unless otherwise indicated, all the reactions were carried out with *N*-alkyl-*N*-arylacrylamides **2b–2ae** (1.0 equiv), **1a** (8–10 equiv), EtOAc (0.2 M) for 40–90 h. Yields of isolated products are given. [b] The regioisomeric ratios refer to the isolated products and the structure of the major isomer is drawn. [c] Combined yields are given.

were synthesized from oxindoles **42–44** by amidation followed by  $\text{LiAlH}_4$  reduction. Compounds **53–55** may serve as precursors for the synthesis of ( $\pm$ )-physostigmine analogues.<sup>[14]</sup>

To shed light on the mechanism of the reaction, several control experiments were carried out (see the Supporting Information). The reaction gave only trace amounts of the desired product under argon atmosphere, thus suggesting that  $\text{O}_2$  is absolutely necessary for this transformation. However, a much lower yield of the desired product was observed when the reaction was performed using a  $\text{O}_2$  balloon. This result indicates that a higher concentration of  $\text{O}_2$  in the reaction mixture may be detrimental to the process. Furthermore, the reaction did not provide any desired product in the presence of 2,2,6,6-tetramethyl-1-piperidinoxyl (TEMPO), presumably owing to the involvement of various radical intermediates in this process. Based on the above observations and literature precedent, a possible reaction mechanism for the aerobic alkylation/cyclization cascade reaction is depicted in Scheme 3. The acyl radical generated from aldehyde auto-oxidation delivers the corresponding alkyl radical through decarbonylation. The alkyl radical thus formed reacts with electron-deficient *N*-alkyl-*N*-arylacrylamide to provide the



Scheme 3. A proposed reaction mechanism.

new carbon-centered radical intermediate **A**. Intramolecular cyclization of **A** with an aromatic ring gives cyclohexadienyl radical **B**, which furnishes the desired oxindole after rearomatization with  $\text{O}_2$  or a hydroperoxide radical.

In conclusion, we have developed an unprecedented and efficient method for the aerobic radical alkylation of electron-deficient amides through dual C–H bond functionalization under metal- and peroxide-free conditions. The

important aspects of the method include: 1) inexpensive alkyl radical precursors, 2) molecular oxygen as a green oxidant and ethyl acetate as the solvent, and 3) convenient operating conditions. This process represents a straightforward entry to different alkyl-functionalized oxindoles with a C3 quaternary stereocenter, as well as analogues of biologically important naturally occurring alkaloid such as ( $\pm$ )-esermethole.

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**Keywords:** alkylation · cascade reactions · cyclization · oxindoles · radical reactions

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