# Cyanohydrin-Mediated Cyanation of Remote Unactivated C(sp<sup>3</sup>)–H **Bonds**

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Supporting Information



ABSTRACT: A new approach for generation of alkoxy radical from the O-H bond of cyanohydrin promoted by visible-light photoredox catalysis is reported. The alkoxy radical triggers the successive remote HAT and intramolecular cyano migration, leading to the regioselective cyanation of remote  $C(sp^3)$ -H bonds. The reaction exhibits a broad functional group tolerance that allows many sensitive groups to remain intact under mild conditions. To demonstrate the utility of method, the ketonitrile products are converted to other synthetically valuable compounds.

**T**ransformation of inert  $C(sp^3)$ -H bonds to other chemical bonds represents an ideal synthetic strategy upon consideration of the atom- and step-economy of reaction. Nevertheless, it remains a long-term challenge in synthetic chemistry.<sup>1</sup> Despite the great progress made by transitionmetal catalysis, radical-mediated hydrogen atom transfer (HAT) provides an efficient approach for  $C(sp^3)$ -H activation.<sup>2</sup> This process is largely dependent on substrates where the tertiary C-H bonds are more preferred than the secondary and primary C-H bonds. In the presence of multiple reactive sites, the reaction usually loses regioselective control, leading to a mixture of regioisomers (Scheme 1a).

The radical translocation process mediated by alkoxy radicals has long been established to gain distinct chemo-/ regioselectivities.<sup>3</sup> However, this method is still underexplored. It is a formidable challenge to directly oxidize alcohol to alkoxy radical owing to the high bond dissociation energy (BDE) of O-H bond (ca. 105 kcal/mol). In this context, many alternative pathways are sought to transform alcohol to various precursors (e.g., metal alkoxides, nitrite esters, hypohalites, etc.) that could readily generate alkoxy radicals under mild conditions, but these surrogates are sometimes difficult to prepare or handle (Scheme 1b).<sup>4</sup> Photoredox catalysis proved to be a powerful tool for converting alcoholic O-H bonds to alkoxy radicals.<sup>5</sup> Recently, we reported the visible-lightpromoted heteroarylation of  $C(sp^3)$ -H bonds via alkoxy radical-triggered 1,5-HAT.<sup>6</sup> Later, Zuo disclosed the CeCl<sub>3</sub>catalyzed  $C(sp^3)$ -H amination of alcohols under visible-light irradiation.7 Considering that alcohols are abundant and commodity chemicals, the alcohol-mediated regioselective  $C(sp^3)$ -H functionalization via radical translocation is of particular synthetic value.



Scheme 1. Radical-Mediated HAT for C(sp<sup>3</sup>)-H Activation

The cyanohydrin-ketonitrile reaction was developed by Kalvoda half a century ago to functionalize unactivated C–H bonds in steroids. The early studies harnessed harsh conditions such as the combination of lead tetraacetate, iodine, and UV

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irradiation or strong metal oxidants (e.g., silver and cerium salts, etc.) that usually led to low yields and poor compatibility with susceptible groups.<sup>8</sup> To improve the reaction outcome, Watt employed the elaborate peracetoxynitrile instead of cyanohydrin as the precursor of alkoxy radical.9 However, these pioneering reports have not received much attention, and the synthetic value of this chemistry has been scarcely explored over the past few decades. Herein we disclose the visible-lightpromoted regioselective cyanation of remote  $C(sp^3)$ -H bonds (Scheme 1c). The alkoxy radical is directly generated from the O-H bond of cyanohydrin, which triggers the sequence of remote HAT and intramolecular cyano migration.<sup>10</sup> A broad range of functional groups are well tolerated under the mild reaction conditions. The versatile ketonitrile products can be conveniently converted to other synthetically valuable molecules, illustrating the utility of the protocol.

At the outset, cyanohydrin 1a was prepared as the model substrate to investigate reaction parameters. After a systematic survey of photocatalysts, oxidants, and solvents, the reaction was facilitated by visible-light irradiation to afford the desired ketonitrile 2a in 82% yield on a 0.3 mmol scale through the cyanation of unactivated  $\gamma$ -C(sp<sup>3</sup>)–H bond (Scheme 2; for

Scheme 2. Optimized Conditions for the Cyanohydrin-Ketonitrile Reaction



detailed reaction conditions survey, see the Supporting Information). Remarkably, the reaction was not interfered by the commonly occurring  $\beta$ -C–C bond fission of the alkoxyl radical or the Norrish type II fragmentation.<sup>11</sup> It was found that only persulfates proved to be efficient oxidants, and the reaction was halted while altering the photoredox catalyst. Among the solvents examined, a biphasic mixture consisting of PhCF<sub>3</sub>/H<sub>2</sub>O delivered the best yields. The addition of ammonium salt was indispensable to the transformation, presumably acting as a phase-transfer catalyst (PTC) in the reaction. Performing the reaction on a 1.0 mmol scale compromised the reaction outcome, probably due to the sharply decreased light penetration in the reaction at larger scale.

With the optimized reaction conditions in hand, we set about defining the substrate scope (Scheme 3). Initially, a variety of functional groups were tested. Both electron-rich and -deficient groups were well tolerated in the reaction to deliver good yields (2b-j). The examples of 2j and 2n were noteworthy, as the existence of aryl bromide reserved a platform for later modification of products by cross couplings. The change of substituents from the para- to the meta- or the ortho-position did not have much impact on reaction outcome (2k-n). Notably, some groups such as alkynyl and alkenyl susceptible to radical conditions remained intact in the reaction (20 and 2p). Apart from aryl cyanohydrins, aliphatic cyanohydrins were also suitable substrates (2q). The radical abstraction of tertiary C-H bonds is, in general, prior to other C-H bonds in accordance with BDEs, whereas the cases of 2s and 2t showed a reversed selectivity of secondary C-H bonds.





<sup>a</sup>Reaction conditions: cyanohydrin 1 (0.3 mmol),  $[Ir(dF(CF_3)-ppy)_2(dtbby)]PF_6$  (3 mol %),  $K_2S_2O_8$  (2.2 equiv), and TBAC (40 mol %) in PhCF<sub>3</sub>/  $H_2O$  (3 mL/0.3 mL) under 14W blue LED irradiation at rt. Yields of isolated products are given. TBAC = tetrabutylammonium chloride.

The intramolecular cyano migration underwent through a cyclic transition state that rendered a unique stereocontrol for cyanation of cyclic C-H bonds, affording the *cis*-product **2s**. Moreover, benzylic C-H bonds (**2u**-w) and the C-H bonds adjacent to the heteroatom (**2x** and **2y**) were also apt to furnish the corresponding cyanated products. In the case of **2u**-w, most of the unreacted cyanohydrins were recovered. In the presence of multiple reactive methylenes, the cyanation of  $\gamma$ -C-H bonds was superior to the  $\delta$ -ones (**2z**), ascribing to that 1,5-HAT via a six-membered cyclic transition state is kinetically more favorable in this reaction.

Several well-designed experiments were conducted to shed lights on the mechanistic pathways. To understand the reactivity of different types of  $C(sp^3)$ -H bonds in the reaction, a set of competition experiments were first performed (Scheme 4a). The observed trend, tertiary > benzylic > secondary > primary C-H, was identical to the routine on the basis of BDEs. The kinetic isotope effect value ( $k_H/k_D = 2.33$ ) gained from the intramolecular isotope competition experiment of 7 suggested that the C-H cleavage might be involved in the ratedetermining step of reaction (Scheme 4b). The free O-H bond of cyanohydrin was crucial to the direct formation of the key alkoxy radical intermediate from the O-H bond which

# Scheme 4. Mechanistic Investigation

a. Competition experiments



initiated the subsequent cascade. To provide support for the hypothesis, the TMS-protected cyanohydrin 9 was tested. As expected, only a trace amount of product 2a was obtained in the reaction (Scheme 4c). Additional experiments including the optical and electrochemical measurements were carried out to elucidate the oxidation process from cyanohydrin to alkoxy radical. The control experiments revealed that the reaction could also proceed in the absence of photocatalyst and visiblelight irradiation, albeit affording the desired product in a low yield (see the Supporting Information). Along with the detected quantum yield ( $\phi = 4.7$ ) of the reaction, these results suggested that the overall process of alkoxy radical formation might be the outcome of photocatalytic pathway and the persulfate-mediated radical- chain reaction. The Stern-Volmer analysis indicated that the excited state of  $\mathrm{Ir}^{\mathrm{III}}$ photocatalyst was readily oxidatively quenched by K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> to form the Ir<sup>IV</sup> species (see the Supporting Information). However, cyclic voltammograms displayed that neither Ir<sup>ív</sup>  $(E_{1/2}^{\text{IV/III}} = 1.69 \text{ V in MeCN vs SCE})$  nor persulfate  $(E^0 = 2.01)$ V) was capable of directly single-electron oxidizing 1a ( $E_{p/2}$  = 2.5 V in MeCN vs SCE; see the Supporting Information).

Based on the above results, we thus postulated a protoncoupled electron transfer (PCET) process for the generation of alkoxy radical (Scheme 5). The interaction of cyanohydrin with a conjugate base such as sulfate probably reduces the

## Scheme 5. Plausible Reaction Mechanistic Pathways



oxidation barrier of O–H bond and enables the formation of alkoxy radical via PCET.<sup>12</sup> Two pathways, the photocatalytic Ir<sup>III</sup>/Ir<sup>III</sup>\*/Ir<sup>IV</sup> cycle (path a) and persulfate-mediated chain reaction (path b), might be concurrently engaged in the PCET process. Furthermore, a competitive pathway, abstraction of the alcoholic hydrogen from cyanohydrin by sulfate radical anion to generate alkoxy radical, also could not be ruled out.

The synthetic utility of method was manifested by transformations of the product into other valuable molecules (Scheme 6). The product consists of ketone and nitrile; both

Scheme 6. Synthetic Value of Products



are versatile feedstocks in synthetic chemistry. First, the ketone could be readily converted to ester **10** by the Baeyer–Villiger oxidation. Then, the nitrile could be hydrolyzed to amide that underwent the intramolecular annulation with ketone to afford lactam **12**. Alternatively, reduction of **2a** resulted in 1,5-amino alcohol **13** which went through the intramolecular cyclization to furnish piperidine **14**. Notably, complete hydrolysis of nitrile led to a high yield of carboxylic acid **15**, which could be harnessed as precursor for incorporation of various functional groups through the radical decarboxylative functionalization according to Li's methods.<sup>13</sup>

In summary, we have described a synthetically useful cyanohydrin-mediated regioselective cyanation of remote  $C(sp^3)$ -H bonds. The alkoxy radical is readily generated from O-H bond under visible-light irradiation, which triggers the sequential HAT and distal cyano migration without the undesired  $\beta$ -C-C cleavage and the Norrish type II fragmentation. The generality and mechanism of the reaction have been investigated. A variety of functional groups including many susceptible groups such as alkenyl and alkynyl are well compatible with the mild conditions. The ketonitrile products are easily transformed into various valuable molecules, demonstrating the synthetic utility of method. Since cyanohydrins originate from ketones and lead to the cyanated ketones, this protocol can be regarded as a formal late-stage cyanation of aliphatic ketones.

# ASSOCIATED CONTENT

## **Supporting Information**

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Experimental details, compound characterization data, and NMR spectra (PDF)

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#### Notes

The authors declare no competing financial interest.

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