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Redox-Divergent Synthesis of Fluoroalkylated Pyridines and 2-Pyridones through Cu-Catalyzed N–O Cleavage of Oxime Acetates

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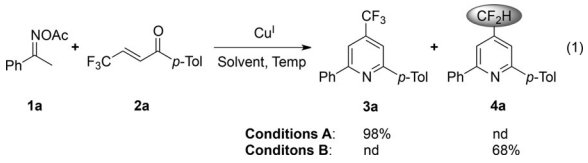
Abstract: *Cu-catalyzed redox-divergent [3+3] coupling of oxime esters with β-CF_3 enones and acrylates is described. This redox-neutral coupling with enones and acrylates affords trifluoromethylated pyridines and pyridones, respectively. Under reductive conditions, difluoromethylated pyridines, difluoromethlated pyridones, and trifluoromethylated dihydropyridones are obtained. The reactions occur under mild conditions with broad substrate scope and regio/redox selectivity.*

Organofluorine compounds are of vital significance in pharmaceutical, agrochemical, and materials studies, since the introduction of F atoms into organic compounds has a profound impact on their metabolic stability, solubility, and lipophilicity.[1] In particular, the CF_2H group serves as a bioisostere of hydroxy and thiol groups and a lipophilic hydrogen bond donor. Selective introduction of CF_2H into aromatic compounds can also remarkably improve these properties.[2] However, compared to the well-established trifluoromethylation systems, difluoromethylation methods remain limited.[3] On the other hand, pyridines and pyridones are prevalent structural motifs in natural products and bioactive molecules.[4] The introduction of fluoroalkyls into these heterocycles can modulate their basicity and binding properties.[1,2,5] Consequently, fluorinated heteroarenes possess beneficial properties at both units and are a promising family of pharmacores. For example, mefloquine is used to prevent or treat malaria, and fluazinam is a broad-spectrum fungicide.[1,6]

Recently, tremendous efforts have been made toward metal-catalyzed syntheses of pyridines.[7] The strategy of using readily available oximes to construct heterocycles is appealing.[8] Recent advancements in Cu-catalyzed [3+3]/[3+2] annulation of oxime esters with activated π bonds provided new routes for heterocycle synthesis, as has been reported by Guan, Yoshikai, Jiang, Wei, and others.[9,10] Recently, Yoshikai developed Cu-catalyzed synthesis of alkyl/aryl-substituted pyridines starting from oxime esters and α,β-unsaturated aldehydes/ketimines.[9c,i] However, methods for the synthesis of fluoroalkylated pyridines/pyridones have been rather limited. In addition, all these systems are limited to redox-neutral or oxidative conditions.[8f] Reductive coupling of oximes remains underexplored, which substantially limits the accessible patterns of heterocycle products. We now report the redox-divergent synthesis of five classes of heterocycles through [3+3] annulation of oxime acetates with β-CF_3-substituted enones/acrylates.

We commenced our investigation with optimization studies on the coupling of oxime acetate **1a** and β-CF_3 enone **2a** [11] (Eq. (1) and Table S1 in the Supporting Information). 4-Trifluoromethylpyridine **3a** was isolated in 96% yield when simply catalyzed by CuCl in DMSO at 60 °C (Conditions A). To our surprise, the CF_2H analogue **4a** was obtained in 68% yield when CuBr/PPh_3 was used as the catalyst in the presence of ^{t}BuOK with ^{i}PrOH as a reducing agent (Conditions B).



With the optimized conditions in hand, we next investigated the scope of the coupling systems (Scheme 1). Conditions A turned out to be broadly applicable for enones bearing diverse electron-donating groups (EDGs) and electron-withdrawing groups (EWGs) at the *para* position of the benzene ring (**3a**–**g**, 45–97%). The reaction also worked well for *meta*- and *ortho*-tolyl-substituted (**3h**, **3i**) and other (hetero)aryl-substituted (**3j**–**m**) enones. Replacement of the CF_3 group with C_2F_5 also led to smooth coupling, albeit at elevated temperature (**3n**). In several cases, CuBr exhibited higher activity than CuCl (**3c**,**f**). The scope with respect to the oxime ester was next examined in the coupling with a *p*-tolyl-substituted enone. A variety of electronically and sterically different acetophenone oximes reacted smoothly (**3o**–**3aa**), including those bearing an α-substituent (**3v**–**3aa**). This method is also applicable to the late-stage functionalization of a natural product (**3ab**). Besides enones, a β-CF_3 acrylonitrile also coupled smoothly to afford a 2-aminopyridine (**3ac**) in good yield.

We next investigated the scope of the reductive synthesis of 4-difluoromethylpyridines (Scheme 2). CF_3-substituted enones bearing various halogen, electron-donating, and electron-withdrawing substituents at the *para* position were

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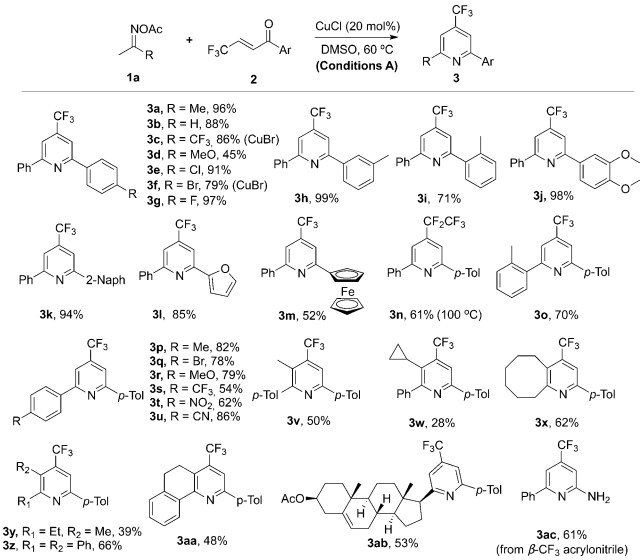
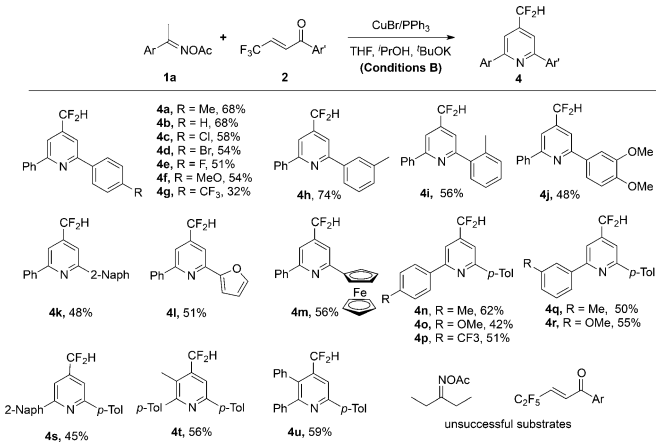


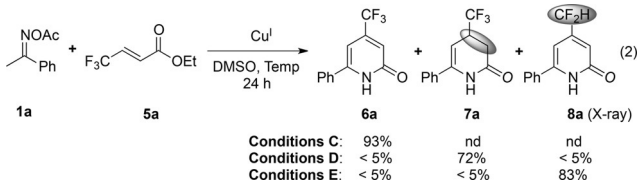
Scheme 1. Pyridine synthesis through condensation of oxime esters with β-fluoroalkylated enones (Conditions A). See the Supporting Information for details. DMSO = dimethyl sulfoxide.



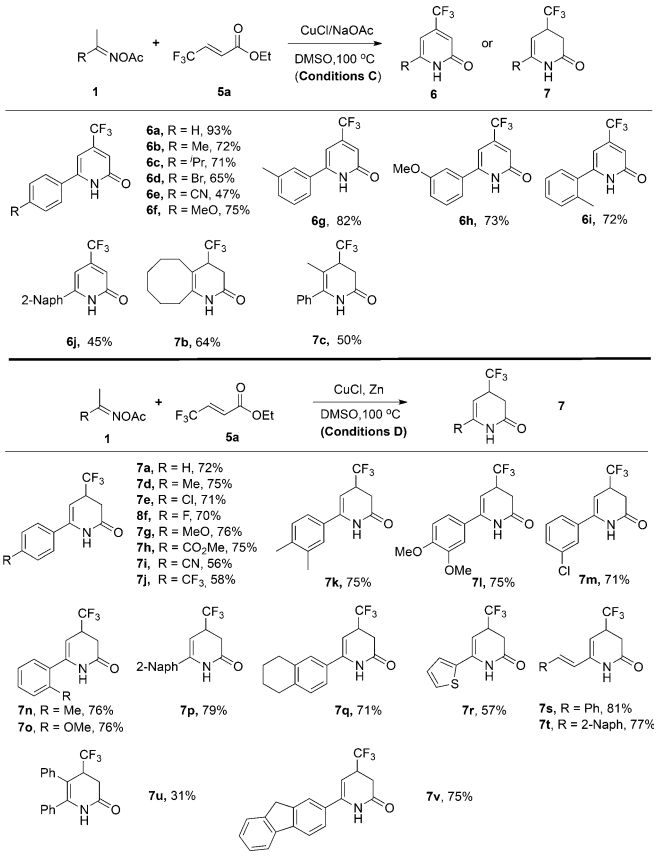
Scheme 2. Reductive coupling of oxime esters with β-CF_3 enones. Conditions B: **1a** (0.2 mmol), **2** (0.3 mmol), CuBr (0.04 mmol), PPh_3 (0.05 mmol), ^{i}PrOH (2.0 equiv), ^{t}BuOK (4.4 equiv) in THF (2.0 mL), yields of isolated product are given. THF = tetrahydrofuran.

fully amenable to this coupling (**4a**–**4g**, 32–68%). The reaction also tolerated enones bearing *o*-Me, *m*-Me, 2-Naph, and dimethoxy substituents (**4h**–**4k**). The coupling of 2-furyl- and ferrocenyl-substituted enones also proceeded smoothly to afford the corresponding products in good yield (**4l** and **4m**). Concerning the scope with regard oxime esters, introduction of EDGs and EWGs to different positions of the oxime ester is also well tolerated (**4n**–**4s**). Incorporation of a Me or Ph group to the α-position of the acetophenone oxime also allowed the synthesis of tetra-substituted pyridines (**4t**, **4u**). In contrast, for a β-C_2F_5 enone and the oxime ester derived from 3-pentanone (**3n** and **3y**; Scheme 1), no desired product was obtained under the current conditions.

To better define the scope of the present [3+3] annulation reaction, we next examined the annulation using β-trifluoromethylated acrylate (**5a**) as a less electrophilic coupling partner (Eq. (2) and Table S2). Extensive screening of the solvent, temperature, base, and reductant revealed that redox-neutral annulation occurred with NaOAc as an additive to give 4-CF_3 pyridone **6a** in high yield (Conditions C). Application of Zn as a reductant afforded CF_3-retentive reduction product **7a** (Conditions D). In addition, 4-CF_2H-substituted pyridone **8a** (confirmed by X-Ray analysis)[12] was isolated as the major product when DBU was employed as a base as well as a reductant (Conditions E).



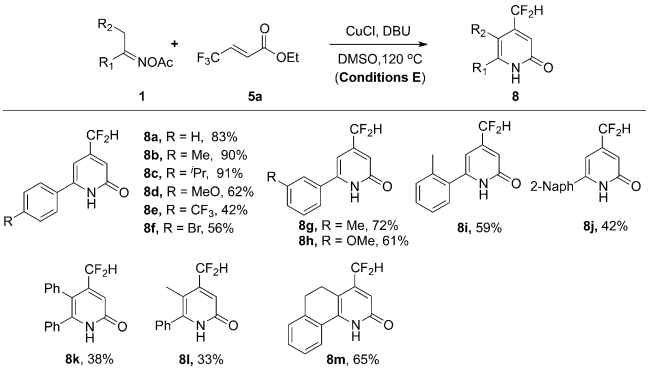
Synthesis of 4-trifluoromethylatedpyridones was then explored (Scheme 3). Various oximes derived from acetophenones exhibited good functional-group tolerance, regardless of the presence of halogens, EWGs, and EDGs in the benzene ring (**6a**–**6j**, 47–93%). Introduction of an alkyl group to the α-position of the oxime, surprisingly, shifted the selectivity to



Scheme 3. CF_3-retentive annulation of oxime esters with a β-CF_3 acrylate. Conditions C: oxime **1** (0.2 mmol), **5a** (0.4 mmol), CuCl (0.04 mmol), NaOAc (0.40 mmol), and DMSO (2.0 mL), 100 °C, 24 h. Conditions D: **1** (0.2 mmol), **5a** (0.24 mmol), CuCl (0.02 mmol), Zn (25 mol%), DMSO (3.5 mL), 80 °C, 24 h.

the reduction product (dihydropyridones **7b** and **7c**). It is likely that the steric effect of these alkyl groups inhibits oxidative aromatization. To highlight the synthesis of dihydropyridones, conditions D were adopted. This reaction was found to have a truly broad scope with respect to oxime esters, which coupled with **5a** in good to excellent yield, with full compatibility with EDGs and EWGs (**7a**–**7r, 7v**). The reaction was equally efficient for alkenyl-substituted oxime esters (**7s, 7t**). Introduction of a phenyl group to the α-position of oxime is also tolerated, albeit with lower yield (**7u**).

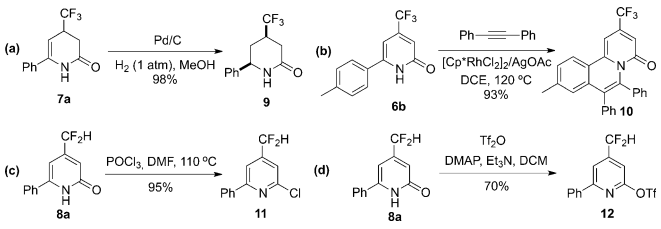
The scope for the fifth class of product, 4-difluomethylpyridone, was accordingly examined under conditions E (Scheme 4). In line with the scope of the other coupling systems, oxime esters derived from acetophenones and other ketones all underwent smooth coupling with acrylate **5a** to afford the desired products in moderate to excellent yield, although oxime esters bearing an α-alkyl/aryl group tend to react with lower efficiency (**8k**, **8l**).



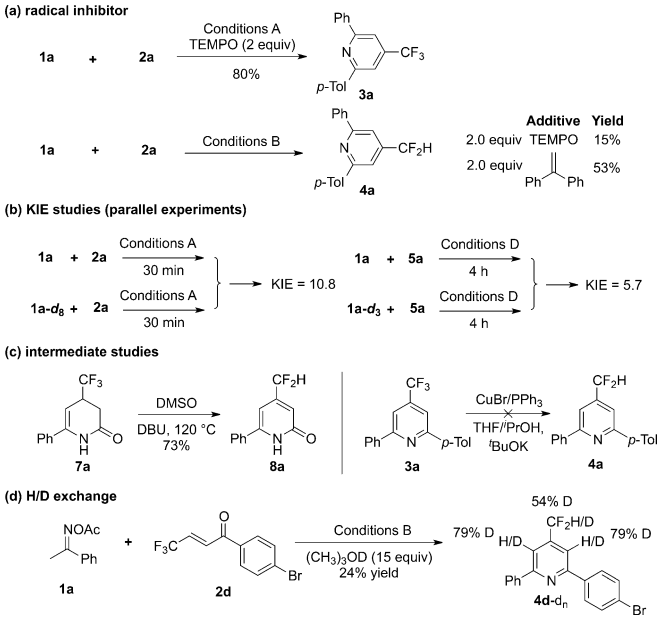
Scheme 4. Dehydrofluorinative coupling of oxime esters with β-CF_3 acrylate. Conditions E: **1** (0.2 mmol), **5a** (0.4 mmol), CuCl (0.04 mmol), DBU (0.40 mmol), DMSO (2.0 mL), 120 °C, yields of isolated product are given. DBU = 1,8-Diazabicyclo[5.4.0]undec-7-ene.

The synthetic utility of the (dihydro)pyridones was then briefly demonstrated (Scheme 5). Hydrogenation of dihydropyridone **7a** afforded lactam **9** as a single diastereomer in nearly quantitative yield. In addition, Rh^{III}-catalyzed oxidative [4+2] coupling between NH pyridone **6b** and diphenylacetylene afforded a fused heterocycle (**10**) in excellent yield.[13] 2-Chlorination of **8a** gave **11**, and O-triflation of **8a** yielded **12**.

Several experiments were performed to probe the mechanism of these coupling systems (Scheme 6). Addition of

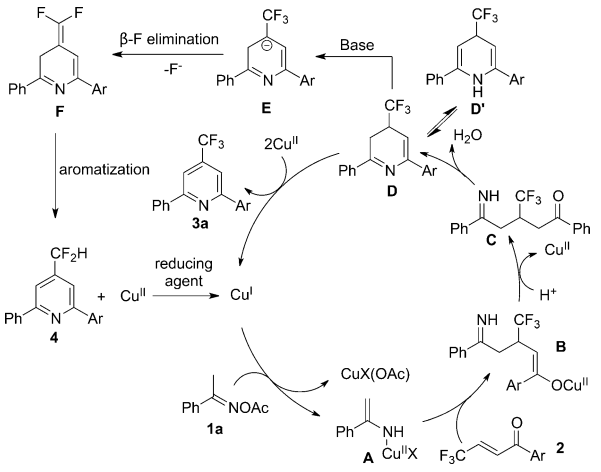


Scheme 5. Derivatization reactions. DCE = 1,2-dichloroethane, DMF = *N*,*N*-dimethylformamide, DMAP = 4-dimethylaminopyridine, DCM = dichloromethane.



Scheme 6. Mechanistic studies. TEMPO = 2,2,6,6-tetramethyl-1-piperidyloxy.

TEMPO to the coupling of **1a** and **2a** did not inhibit the formation of **3a** (conditions A), thus indicating an ionic pathway. In fact, this conclusion is consistent with the formation of product **3w** (Scheme 1), since a radical pathway would lead to ring scission.[10a] While formation of CF_2H-substituted pyridine **4a** was retarded by the addition of TEMPO (conditions B), the reaction was only slightly affected when 1,1-diphenylethylene was introduced (Scheme 6a). Parallel reactions were then conducted using **1a** and labeled **1a**-d_8 or **1a**-d_3 for kinetic isotope effect (KIE) studies. Significant kinetic isotope effects were observed for the coupling with an enone (KIE = 10.8, conditions A) or with an acrylate (KIE = 5.7, conditions D). These results suggest that cleavage of the α-C–H bond is involved in the turnover-limiting step (Scheme 6b). To identify intermediates during the formation of 4-CF_2H pyridone **8a**, 4-CF_3-functinalized dihydropyridone **7a** was treated with DBU in DMSO, which led to clean formation of **8a** (Scheme 6c), thus suggesting that this product is generated through elimination of HF followed by tautomerization. In contrast, **3a** proved not to be an intermediate for the formation of **4a** since no conversion of **3a** was observed under conditions B. Likewise, **4a** is likely generated through dehydrofluorination of a CF_3-functionalized 3,4-dihydropyridine. We also performed H/D exchange experiment for the coupling of **1a** and enone **2d**, with ^{t}BuOD as a deuterium source (modified conditions B). NMR analysis of the isolated product revealed that both the 3- and 5-positions are equally deuterated (79% D), and significant H/D exchange was also detected at the difluoromethyl position (Scheme 6d), which supports intermediacy of a CF_3-dihydropyridine (see Scheme 7) with subsequent tautomerization.[14] Note that no post-coupling H/D exchange was observed for **4d** under the standard conditions with CD_3OD or ^{t}BuOD, as confirmed by control experiments.



Scheme 7. Proposed mechanism.

On the basis of our studies and previous reports on Cu-catalyzed coupling of oximes, a plausible pathway is given for the formation of pyridines (Scheme 7).[9c,i] Oxidation of Cu^{I} by oxime ester **1** gives copper(II) enamide **A** together with a Cu^{II} acetate. Conjugate addition of **A** to enone **2a** then yields enolate species **B**, and subsequent protolonysis leads to ketone **C**. Dehydrative cyclization would produce dihydropyridine intermediate **D**, and further oxidation of **D** by two copper(II) species furnishes CF_3-pyridine product **3** with regeneration of the copper(I) catalyst. In the presence of a suitable base and reductant, **D** undergoes $E1_{cb}$ reaction to afford intermediate **F**, which then isomerizes to the 4-CF_2H-pyridine product **4**. Meanwhile, the Cu^{I} catalyst is regenerated by further reduction of Cu^{II}. The coupling with a β-CF_3 acrylate likely follows the same mechanism except that the cyclization process forms an amide bond. Following this cyclization, the dihydropyridone species is either released, oxidized by Cu^{II}, or undergoes elimination of HF under condition control.

In summary, we have demonstrated redox-divergent access to five classes of fluoroalkylated heterocycles through copper-catalyzed [3+3] coupling of oxime acetates with β-trifluoromethylated enones/acrylates. Under redox-neutral conditions, the reaction afforded 4-CF3-pyridines and 4-CF3-2-pyridones. Coupling using ^{i}PrOH, Zn, and DBU as a reductant led to selective formation of 4-CF_2H pyridines, 4-CF_3-dihydropyridones, and 4-CF_2H pyridones, respectively. The coupling systems cover a particularly broad range of oxime acetates derived from aryl-alkyl and dialkyl ketones in acyclic as well as cyclic settings. The redox diversity and elegant control of reaction selectivity may provide insight for future studies of other Cu-catalyzed systems, which are currently underway in our laboratories.

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Conflict of interest

The authors declare no conflict of interest.

Keywords: copper · fluoroalkylation · oximes · pyridines · pyridones

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[12] CCDC 1824515 (**8a)** contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre.

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