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Transition-Metal-Free α Csp^3–H Cyanation of Sulfonamides

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Abstract: This report describes the site-selective α-functionalization of sulfonylamide derivatives through the in-situ generation of imine intermediates. The N–F sulfonylamides, which could facilitate the elimination to generate imines, are coupled with TBACN to efficiently and mildly afford α-amino cyanides. Comparing with Strecker reaction, this transformation offers a complementary strategy to efficiently construct α-amino cyanides from direct α C–H functionalization of sulfonylamindes. The reaction is also characterized by broad substrate scope and flash chromatography column free workup. More importantly, the new two-electron pathway to generate imines through manipulation of the leaving group allows us to achieve excellent α site-selectivity.

Selective functionalization of aliphatic C–H bonds represents an important goal of modern synthetic chemistry.[1] Differentiating between such bonds in organic molecules with high levels of selectivity remains a considerable challenge.

C–H functionalization of amines has drawn increasing interest due to their presence in drugs and bioactive molecules.[2] Electron rich tertiary amines could be oxidized to the radical cation with subsequent deprotonation or directly hydrogen atom transfer to deliver an α-amino alkyl radical, therefore α C–H functionalization of amines have been well established.[3] In contrast, the development α C–H functionalization of sulfonylamides which are frequently found in drugs and biomolecules is challenging, because the nitrogen atom with an electron withdrawing protecting group has much lower electron density than amines.[4] In addition, the key intermediate of electrophilic N centered radical would prefer 1,5 HAT instead of 1,2 HAT (Figure 1).[5,7] For example, in 2016 Moriyama group had developed a nitroxyl-radicalcatalyzed oxidative coupling reaction of N-EWG-protected amines (amides) with silylated nucleophiles by the N chloro of N-EWG-protected amides with a halogenation reagent in situ.[6] Later, Muñiz and coworkers disclosed a bromide catalyzed α C–H oxygenation of sulfonylamide to afford N-sulfonyl oxaziridines.[7] However, substrates with a δ C–H are not compatible with both transformations due to favorable 1,5 HAT. In 2020, Montgomery and Martin group reported a nickel merged with photoredox catalyst catalyzed α-arylation and α-alkylation of benzamides which showed broad substrate scope in moderate to good yields.[8] On the other hand, the electron deficient nature of amide makes α C–H less nucleophilic and possess stronger bond strength which is harder to form carbon radical via directly hydrogen atom transfer. Relying on an electrostatic attraction to achieve α selectivity, Rovis group realized α-alkylation of primary aliphatic amines via in situ generating a negatively charged carbamate which would allow α C–H abstraction by the positively charged quinuclidinium radical.[9] Inspired by this work, they also developed α C–H alkylation of trifluoromethanesulfonamides by deprotonation of NH and then followed α C–H abstraction.[10]

We sought to develop an alternative strategy to realize highly efficient α C–H functionalization of amides which could avoid the problematic electrophilic N centered radical intermediate. On the other hand, α-aminonitriles are important and versatile intermediates in organic synthesis that can be readily converted into biologically active compounds and functional materials, such as α-amino acids, vicinal diamines, imidazoles, thiadiazoles, and others.[11] Inspired by Strecker reaction which

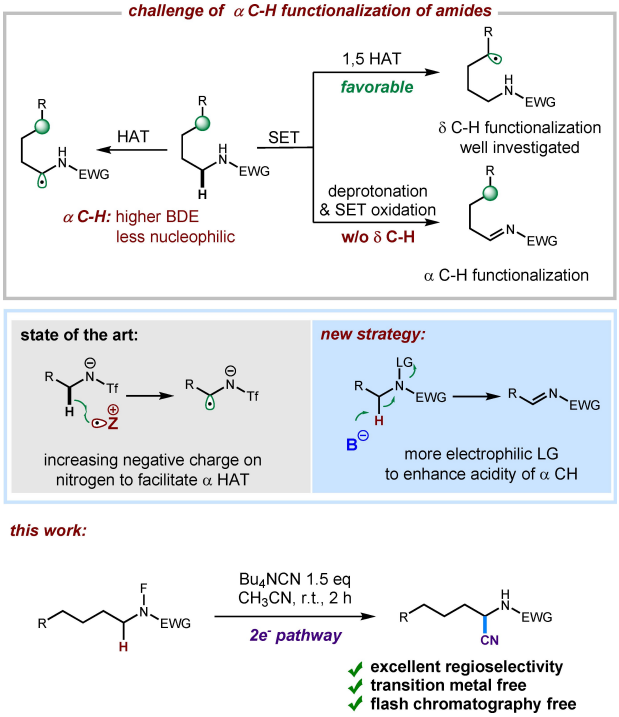

Figure 1. Selective α C–H cyanation of amides.

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has been studied over 150 years to synthesize α-amino nitrile via an imine intermediate.[12] We anticipated that a preinstalled leaving group on sulfonylamides would have more acidic α C–H to facilitate the formation of reactive imine in situ. Therefore, we can expect to access α-amino nitriles in presence of cyanide. Hence, we choose to investigate α cyanation of sulfonylamide as our model reaction. Recently, NF amides have been extensively studied in achieving δ C–H functionalization of amides via a 1,5 hydrogen atom transfer pathway.[13] However, to the best of our knowledge, an α C–H cyanation of N–F sulfonylamides is still unknown. Herein, we report a practical transition-metal-free α C–H cyanation of sulfonylamides which proceeds by cyanide reacting with in situ generated imine from NF substrates to afford the corresponding α-aminonitriles in excellent yield. The transformation is highlighted with mild conditions, excellent regioselectivity, a broad substrate scope, and flash chromatography column free workup.

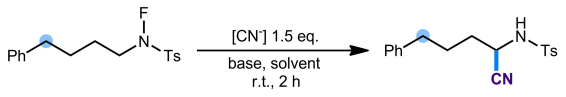
To test our hypothesis, we chose N–F sulfonylamide (**1a**) which possessed a benzylic C–H at δ position as our model substrate, TMSCN as cyanides source, CH_3CN as solvent to start our investigation of α C–H cyanation of sulfonylaminde (Table 1). Considering the importance of base which is needed to activate TMSCN to release free cyanide and deprotonate of substrate **1a** to form the imine in situ, a variety of bases were tested (entry 1–4). Fortunately, all the bases we tried could deliver us the desired product in very good yields (>90%) except Na_2CO_3, and in all cases there were no δ cyanation product observed. To further optimize the conditions to realize flash chromatography column free workup, we switched our cyanide source to TBACN (entry 5), and quantitative yield of α cyanation product was observed. More importantly, simply washing the reaction with water could remove the byproduct and afford pure product in quantitative yield. Other solvents such as acetone, DCM, DMF and DMSO could also furnish the desired product albeit in diminished yields (entry 6–9). Thus, the optimized reaction condition for α C–H cyanation of N–F sulfonylamide **1a** were as follows: **1a** (0.2 mmol) and TBACN (0.3 mmol) were stirred in acetonitrile at room temperature for 2 hours under nitrogen atmosphere.

Next we explored the substrate scope (Scheme 1). Satisfyingly, the scope proved quite general regarding both steric and electronic properties of the fluorosulfonamides. For example, both electron-deficient or electron-rich arylsulfonamides bearing benzylic C–H at δ position provided the corresponding α cyanation products **2a–h** in excellent yields and exclusive regioselectivity. Substrates bearing more steric groups on the aromatic ring also underwent α cyanation smoothly and furnished the expected products **2i–k** in excellent yields. Next, we explored the generality of the protocol with a wide range of linear amines using 4-toluenesulfonamide as the parent group.

The transformation provided excellent selectivity at the desired α-C–H bond irrespective of the substitution, including bearing several secondary C–H; tertiary C–H and benzylic C–H (**2l–p**). More importantly, the reaction also exhibits very good functional group tolerance. Such as heteroarene, alkyne, alkene and acetal could all survive under the reaction condition and afford the corresponding product in excellent yield (**2q–t**). Among them, the substrate bearing a alkene has no cyclization product at all which further indicate the advantage of this two electron pathway. Primary C–H could be efficiently cyanated as well (**2v**). Furthermore, a variety of tertiary C–H substrates were tested. Both cyclic and linear substrates are suitable for this reaction and afford the desired products in excellent yield (**2w–z**). N–F amide could also be converted into the α cyanation product in good yield under the standard condition (**2u**, **2aa**). More importantly, the α C–H cyanation could be implemented at a late-stage transformation. For example, complex natural product or drug derivatives were successfully converted into their α nitriles sulfonylamide analogues (**2ab–2ad**) site-selectively.

After investigated reaction substrate scope we turn our attention to explore the mechanism of the reaction. The N–Cl sulfonamide instead of **1a** was subjected to standard conditions, and only starting material was recovered which indicated the importance of leaving group to get proper acidity of α C–H (Figure 2a). To rule out radical pathway, radical inhibitors such as TEMPO were added into the reaction and showed no effect on the reactions (Figure 2b). Furthermore, a substrate containing cyclopropylmethyl moiety (**1ae**) could

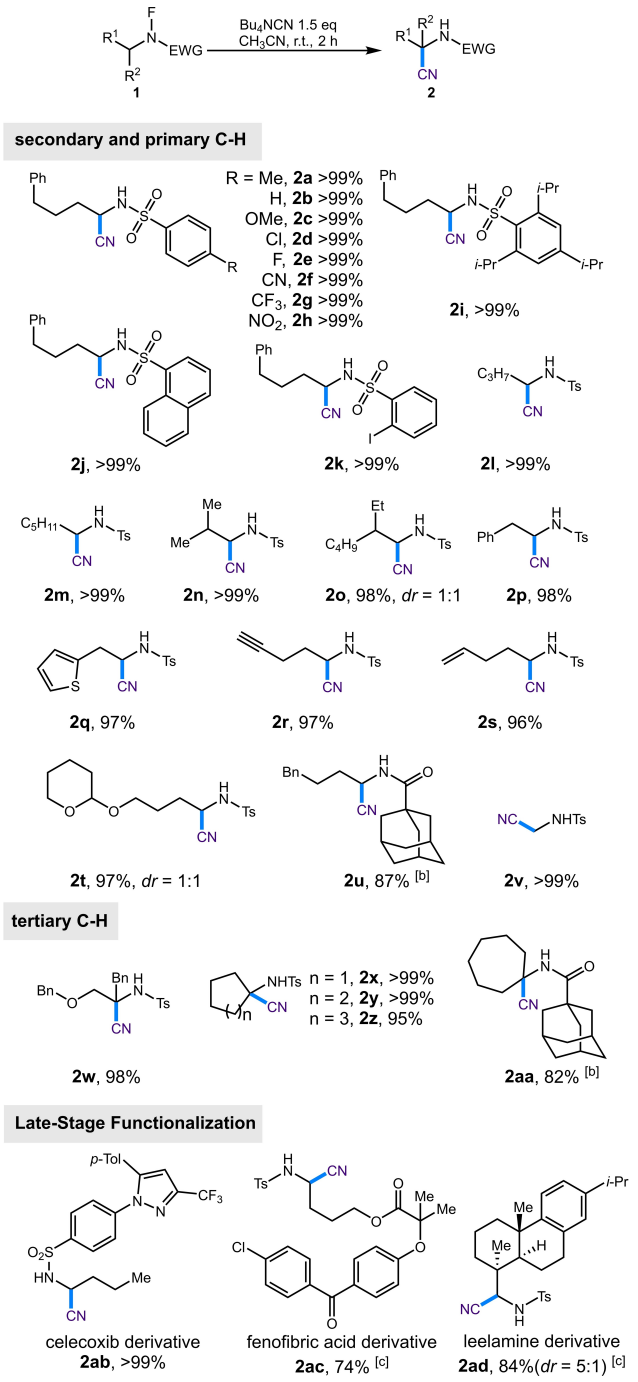
Table 1. Optimization of α cyanation of N–F sulfonylamide.[a]



1a **2a**

Entry	CN	Base	Solvent	**2a** Yield [%][b]
1	TMSCN	Na_2CO_3	CH_3CN	69
2	TMSCN	K_2CO_3	CH_3CN	93
3	TMSCN	TBAF	CH_3CN	94
4	TMSCN	Cs_2CO_3	CH_3CN	96
5	TBACN	none	CH_3CN	>99
6	TBACN	none	acetone	85
7	TBACN	none	DCM	88
8	TBACN	none	DMF	94
9	TBACN	none	DMSO	90

[a] General conditions (unless otherwise specified): **1a** (0.2 mmol), [CN^-] (0.30 mmol), base (0.20 mmol), solvent (2.0 mL), rt, 2 h. [b] Isolated yield of **2a**.



Scheme 1. Scope of **1a**.[a] [a] Reaction conditions as stated in Table 1, entry 5, Yields refer to flash chromatography column free workup unless otherwise noted. [b] The reaction time was extended to 12 h, yields are isolated after purification via column chromatography. [c] Yields are isolated after purification via column chromatography.

survive and afford α cyanation product in 99% yield without any ring opening (Figure 2c), which also suggested that the reaction did not invole an α carbon radical intermediate. Finally, when N–F amide (**1af**) was subjected to standard conditions, both the desired product (**2af**) and the enamine (**2af'**) from the

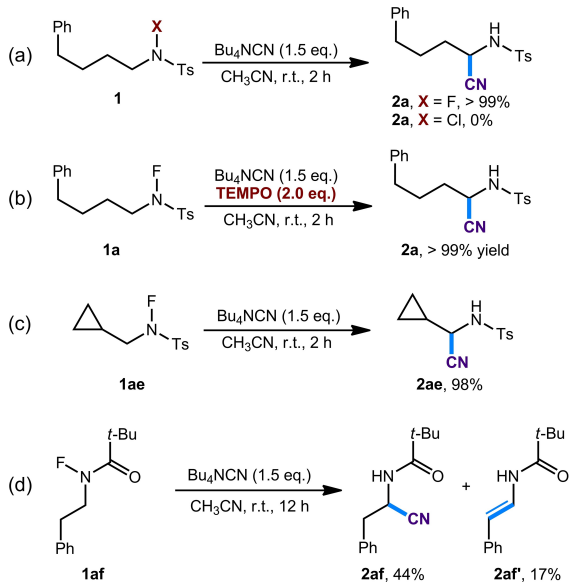


Figure 2. Mechanistic study.

corresponding imine were isolated which further indicated the imine intermediate mechanism.

To further explore the utility of this protocol (Figure 3), a large scale of α cyanation was conducted and the desired product was obtained in quantitative yield (e.g., **2a**, 1.64 gram). The product could be easily converted into valuable organic blocks. For example, the reaction of **2a** and H_2O_2 could produce the corresponding α amino amide **3** compound in 72% yield. The reduction of **2a** in the presence of BH_3·THF, then protected by Boc group, resulted in a 1,2 di-amine scaffold **4** with acceptable yield. Finally, the Ts group on the nitrogen atom could be replaced by benzoyl group after deprotection with methanesulfonic acid, and reprotection with benzoyl chloride to give **5** in 75% yield.

In conclusion, a flash chromatography column free and transition metal free cyanation of α C–H sulfonylamide was

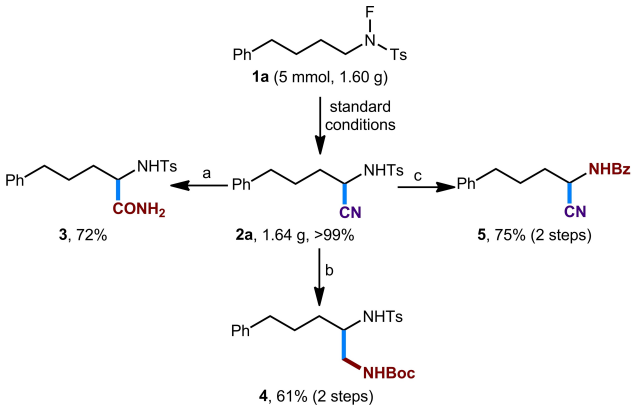


Figure 3. Gram scale synthesis and post functionalization. a. H_2O_2 (30%), MeOH, NaOH (aq.); b. 1.BH_3·THF; 2. Boc_2O; c. 1.$MeSO_3H$, TFA-thioanisole; 2. BzCl, K_2CO_3.

developed to access valuable α amino cyanation product from a variety of fluorine masked sulfonylamides. The transformation underwent a two-electron pathway, which avoided the problematic electrophilic N-centered radical to achieve exclusive regioselectivity. The reaction also featured broad substrate scope and functional group tolerance. Functionalization of complex molecules and gram scale experiments were also conducted to showcase the utility of this reaction. More importantly, it provides a complementary strategy to achieve regioselective α C–H functionalization of sulfonylamides by generating imines in situ. Considering the wide substrate scope and simple procedure, this reaction should be useful for the preparation of a host of potential medicinal and agrochemical agents.

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Conflict of Interest

The authors declare no conflict of interest.

Keywords: cyanation · C–H functionalization · N–F sulfonylamides · synthetic methods

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