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Synthesis of 2-(α-trifluoromethylamino)indoles *via* stepwise cascade transformation of imidoyl sulfoxonium ylides with *ortho*-chloromethyl anilines

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We have developed an efficient one-pot method for the synthesis of 2-(α-trifluoromethylamino)indole scaffolds from CF_3-imidoyl sulfoxonium ylides and *ortho*-chloromethyl anilines. This transformation exhibits broad functional group tolerance under ambient conditions, giving yields ranging from 49–83% across 23 examples. Mechanistic investigations suggest that the reaction pathway involves an initial cyclization, which is followed by a base-mediated isomerization and rearomatization sequence. This approach offers a novel strategy for introducing α-CF_3 amino groups into heterocycles and presents a valuable alternative for indole synthesis.

Introduction

The α-trifluoromethylamino group is a key structural moiety in medicinal chemistry which is widely found in a diverse range of biologically active molecules, including an agonist for LXRβ, the cathepsin K inhibitor Odanacatib, a selective inhibitor of PI3Kδ, anticancer agents, and an HCV NS4B inhibitor (Fig. 1).[1] In addition, the α-trifluoromethyl amine motif could serve as a viable amide bioisostere.[2] In contrast to amides, this motif features CF_3CH_2NH moieties, which could eliminate susceptibility to hydrolysis by amidases, esterases, and proteases.[1d] Moreover, the sp3-hybridized nature of α-carbon confers resistance to proteolytic cleavage, enhancing metabolic stability and reducing toxicity arising from the hydrolyzed amide.[1e,f] Therefore, the development of methods for the introduction of α-trifluoromethylamine is an important synthetic goal. Current strategies for accessing α-trifluoromethylamine primarily rely on the reductive or alkylative amination of trifluoromethyl ketones,[3,4] reduction of trifluoromethylated enamines,[5] nucleophilic addition to trifluoromethyl iminium species,[6,7] or trifluoromethyl anion addition to functionalized imines.[8] Furthermore, the direct α-trifluoromethylamination of unactivated alkenes and the reductive coupling of α-CF_3 amino acetates represent valuable complements to current methodologies.[9,10] However, these existing methods typically require multistep sequences and the use of metals. Consequently, the development of efficient and sustainable methodologies for the introduction of α-trifluoromethylamine remains highly desirable.

Recently, the synthesis of indole skeletons bearing an α-trifluoroethylamine moiety has garnered significant attention in medicinal chemistry, driven by the privileged status of indoles in pharmaceutical agents and the unique properties of trifluoroethylamino groups.[2,11] Although a diverse range of methodologies have been established for the synthesis of 3-substituted indoles,[12] the construction of 2-substituted indoles remains significantly less explored due to the inherently lower reactivity of the C2 position (Scheme 1a). Furman and coworkers developed a method to access 2-(α-trifluoromethylamino)indoles involving the reduction of

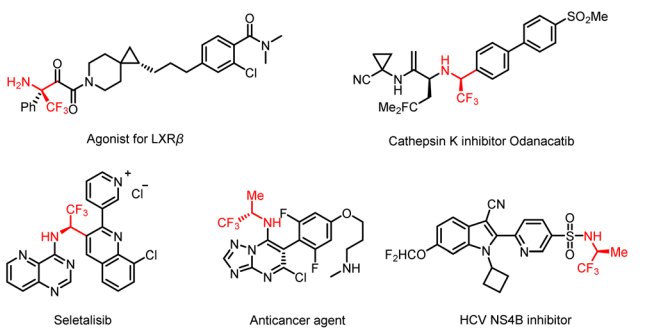

Fig. 1 The α-trifluoromethylamino group in biologically active molecules.

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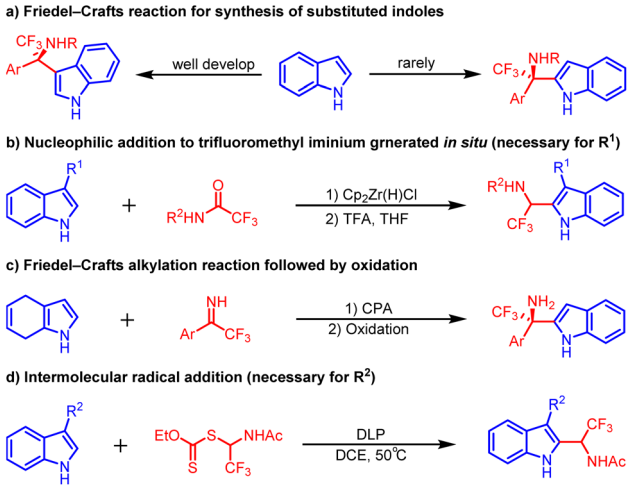


Scheme 1 Some strategies for the synthesis of 2-(α-trifluoromethyl amino)indole.

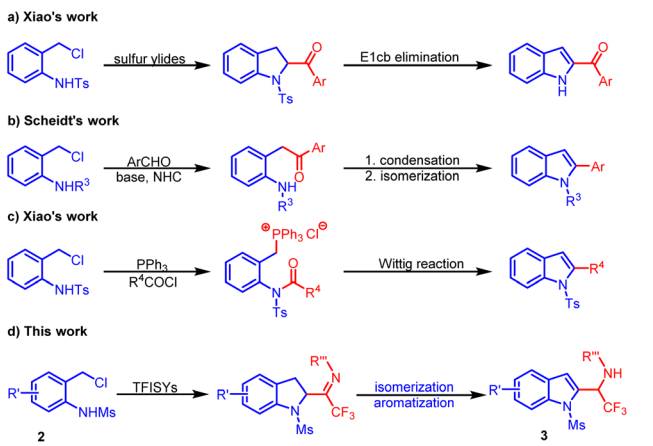
fluoroacetamides with Schwartz's reagent, followed by a one-pot addition to *in situ* generated imines (Scheme 1b).[12b] Additionally, 2-substituted indoles incorporating a trifluoromethyl moiety were obtained *via* a chiral phosphoric acid-catalyzed Friedel–Crafts alkylation of 4,7-dihydroindoles with trifluoromethyl ketimines, followed by oxidation with DDQ (Scheme 1c).[13] Furthermore, Zard's group established an intermolecular radical addition reaction between 3-substituted indoles and xanthates initiated by dilauroyl peroxide (DLP). This methodology provides an effective approach for the direct incorporation of the trifluoroethylamino moiety into such heterocyclic frameworks (Scheme 1d).[14] Despite these advances, there is still a strong demand for an efficient and eco-friendly methods to construct 2-(α-trifluoromethylamino)indole.

CF_3-Imidoyl sulfoxonium ylides (TFISYs) are versatile nitrogen-containing trifluoromethylated dipoles that have proven to be valuable building blocks in cascade annulation chemistry.[15] They react efficiently with various synthons to afford diverse trifluoromethyl-functionalized heterocycles, ranging from simple monocyclic to complex polycyclic systems.[16] In particular, cascade annulations involving TFISYs serve as a powerful strategy for the synthesis of trifluoromethylated indole derivatives. Liu's group reported a visible-light-promoted cascade annulation of TFISYs with azides for the construction of 2-trifluoromethyl indoles through thermal Curtius rearrangement of azides, intermolecular nucleophilic addition, and photoinduced intramolecular cyclization followed by a 1,3-hydrogen shift.[17] In addition, Chen and co-workers established an Rh(III)-catalyzed chemodivergent approach to provide CF_3-substituted indoles *via* the annulation of 2-arylpyridines with TFISYs, involving triple C–H activation.[18] Despite these progress, annulation strategies employing TFISYs for the construction of 2-(α-trifluoromethylamino)indoles are still underexplored.

The chemistry of aza-*ortho*-quinone methides (aza-*o*-QMs), which are generated from *N*-(*ortho*-chloromethyl)aryl amides under basic conditions, has garnered significant attention owing to their remarkable reactivity.[19] In recent years, substantial progress has been made in the development of cycloaddition reactions for the synthesis of diverse heterocyclic skeletons.[20] Notably, the cascade annulation of aza-*o*-QMs has emerged as a powerful strategy to access indole derivatives. For instance, Xiao and co-workers reported an efficient cascade reaction between sulfur ylides and *N*-(*ortho*-chloromethyl)aryl amides that furnished 2-acyl indoles *via* an E1cb elimination pathway (Scheme 2a).[21] In 2014, 2-aryl indoles were synthesized through the trapping of a transient aza-*ortho*-quinone methide intermediate by an NHC-generated acyl anion equivalent (Scheme 2b).[22] More recently, a tandem phospha-Michael addition/*N*-acylation/intramolecular Wittig reaction of aza-*o*-QMs was developed as an effective route for the construction of indole frameworks (Scheme 2c).[23] Despite these advances, the application of aza-*o*-QM cycloaddition to the synthesis of 2-(α-trifluoromethylamino)indoles remains unexplored. Furthermore, current methodologies for aza-*o*-QM-based indole synthesis are predominantly limited to pathways involving cycloaddition followed by E1cb elimination or dehydrative isomerization. Herein, we described a stepwise cascade transformation of imidoyl sulfoxonium ylides with *ortho*-chloromethyl anilines, providing an efficient method to 2-(α-trifluoromethylamino)indoles. Notably, the reaction proceeds without the formation of E1cb elimination byproducts.

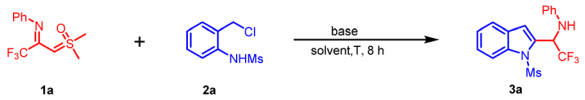
Results and discussion

To initiate the study, (*E*)-3-(dimethyl(oxo)-l6-sulfaneylidene)-1,1,1-trifluoro-*N*-phenylpropan-2-imine **1a** and *N*-(2-(chloromethyl)phenyl)methanesulfonamide **2a** were selected as model substrates and treated with a base in dichloromethane at 40 °C under air. To our delight, our hypothesis was validated, giving 2-(α-trifluoromethylamino)indole **3a** in 57% yield (Table 1, entry 1). The structure was established by NMR analysis and confirmed by X-ray crystallographic analysis of **3f**. Replacing the base from 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) with 1,5,7-triazabicyclo[4.4.0]dec-5-ene (TBD), 1,5-diazabicyclo[4.3.0]non-5-ene (DBN), 1,1,3,3-Tetramethylguanidine (TMG), or 1,4-



Scheme 2 The synthesis of indole skeletons *via* aza-*o*-QMs.

Table 1 Optimization of reaction conditions[a]



1a 2a 3a

Entry	Base	Solvent	Yield (%)[b]
1	DBU	CH_2Cl_2	57
2	TBD	CH_2Cl_2	31
3	DBN	CH_2Cl_2	36
4	TMG	CH_2Cl_2	14
5	DABCO	CH_2Cl_2	Trace
6	Na_2CO_3	CH_2Cl_2	Trace
7	Cs_2CO_3	CH_2Cl_2	Trace
8	KOH	CH_2Cl_2	Trace
9	—	CH_2Cl_2	Trace
10	DBU	DCE	22
11	DBU	THF	42
12	DBU	CH_3CN	30
13	DBU	Toluene	83
14	DBU	$CH_3CO_2C_2H_5$	71
15	DBU	Acetone	38
16	DBU	$CHCl_3$	49
17[c]	DBU	Toluene	36
18[d]	DBU	Toluene	63
19[e]	DBU	Toluene	60
20[f]	DBU	Toluene	77
21[g]	DBU	Toluene	52
22[h]	DBU	Toluene	79

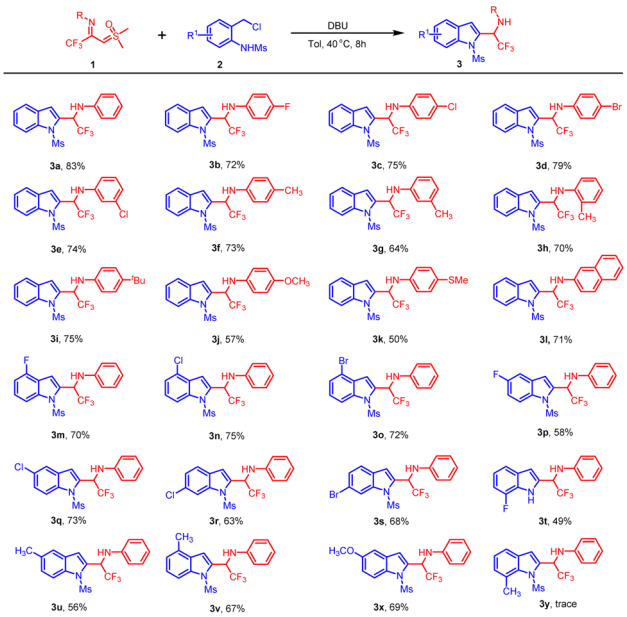
[a] Reaction conditions: **1a** (0.2 mmol), **2a** (0.1 mmol), base (0.15 mol), solvent (0.05 M), 40 °C, 8 h. [b] Yield of isolated product. [c] Reaction conducted at 25 °C. [d] Reaction conducted at 100 °C. [e] Reaction time was 4 h. [f] Reaction time was prolonged to 10 h. [g] Using Na_2CO_3 (0.1 mmol) as a base. [h] Using Na_2CO_3 (0.3 mmol) as a base.

diazabicyclo[2.2.2]octane (DABCO) resulted in decreased yields of **3a** (Table 1, entries 2–5). Notably, inorganic bases proved ineffective for this transformation (Table 1, entries 6–8). Additionally, the reaction failed to afford product **3a** in the absence of DBU (entry 9). A screening of various solvents (Table 1, entries 10–16) identified toluene as the most effective solvent for the generation of **3a**. Variation of the reaction temperature (Table 1, entries 17–18) did not lead to any improvement in the yield of **3a**. Further investigation disclosed neither prolonging nor shortening the reaction time increased the yield of desired product (Table 1, entries 19–20). In addition, further optimization of the base equivalents did not result in a higher yield (Table 1, entries 21–22).

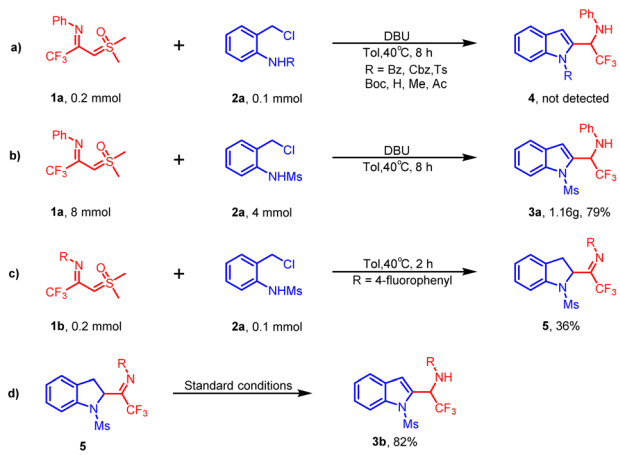
With the optimal conditions established (Table 1, entry 13), we investigated the substrate scope of CF_3-imidoyl sulfoxonium ylides **1**. Our investigation commenced with an evaluation of substituents on the phenyl moiety. As outlined in Table 2, substrates bearing electron-withdrawing groups (*e.g.*, F, Cl, Br) at the *para*-position of the benzene ring were well tolerated, delivering the expected products **3b–3d** in good yields (72–79%). Similarly, the *meta*-chlorophenyl substituted ylide proved to be a suitable substrate, providing **3e** in a moderate yield. Furthermore, the reaction scope was successfully extended to substrates with electron-donating groups at the *para*-, *meta*-, and *ortho*-positions, furnishing the corresponding products **3f–3h** in yields ranging from 64–73%. Intriguingly, the sterically hindered 4-*tert*-butyl-substituted ylide was also amenable to this transformation under standard conditions, delivering **3i** in 75%. Additionally, TFISYs featuring ether and thioether substituents at the 4-position of the arene moiety were well tolerated, providing the expected products **3j** and **3k** in acceptable yields. Notably, the sulfoxonium ylide bearing a naphthyl group was smoothly converted into the corresponding product **3l** by reacting with *N*-(*ortho*-chloromethyl)aryl amide **2**. Encouraged by these results, we proceeded to investigate the substrate scope of the reaction using *N*-(*ortho*-chloromethyl)aryl amides **2** bearing various R^1 groups. A variety of substrates were efficiently transformed into 2-(α-trifluoromethylamino)indoles **3** in good to excellent yields. The protocol was successfully applied to *N*-(*ortho*-chloromethyl)aryl amides **2** substituted with electron-withdrawing groups at the 6-position, affording products **3m–3o** in moderate yields. *N*-*ortho*-chloromethyl anilines **2** containing halogen substituents (*e.g.*, F, Cl) at the 5-position underwent the cyclization/isomerization/aromatization cascade to afford **3p** and **3q**. Furthermore, 6-substituted 2-(α-trifluoromethylamino)indoles **3r** and **3s** were successfully obtained utilizing the corresponding starting materials. Notably, the employment of *N*-(2-(chloromethyl)-6-fluorophenyl)meth-anesulfonamide **2** led to the formation of the deprotected product **3t** in 49% yield. Comparative analysis of **3t** with other derivatives suggested that the position of the substituent on the *N*-*ortho*-chloromethyl amides **2** significantly influences the efficiency of this cascade process. Furthermore, aza-*ortho*-quinone methide precursors **2** bearing electron-donating groups were well tolerated under the optimized conditions, delivering the corresponding products **3u–3x** in moderate yields. However, *N*-(*ortho*-chloromethyl)aryl amides **2** with a 3-methyl substituent failed to provide the desired product **3y** under the standard conditions.

To further investigate the effect of the *N*-substituent, the methanesulfonyl (Ms) group was replaced with benzoyl (Bz), benzyloxycarbonyl (Cbz), *p*-toluenesulfonyl (Ts), *tert*-butoxycarbonyl (Boc), H, methyl (Me), or acetyl groups (Ac). Unfortunately, the expected 2-(α-trifluoromethylamino)indoles **4** were not detected under the optimized conditions (Scheme 3a). The scalability of this transformation was further verified on a gram scale. Under standard conditions, the reaction of **1a** and **2a** in the presence of DBU proceeded smoothly to afford **3a** (1.16 g) in 79% yield (Scheme 3b). Furthermore, the control experiments were performed. Trifluoroacetimidoyl-substituted indoline **5** was obtained utilizing 4-fluorophenyl substituted **1b** and **2a** as substrate in the absence of DBU (Scheme 3c). Subsequently, treatment of **5** with DBU promoted the isomerization and aromatization cascade, affording 2-(α-trifluoromethylamino) indole **3b** (Scheme 3d). To further elucidate the reaction mechanism, the reaction of **1b** and **2a** was conducted under standard conditions in the absence of DBU, and no target product **3b** was observed. However, upon the addition of DBU, **3b** was obtained in 76% isolated yield after 8 hours.

Table 2 Substrate scope[a,b]

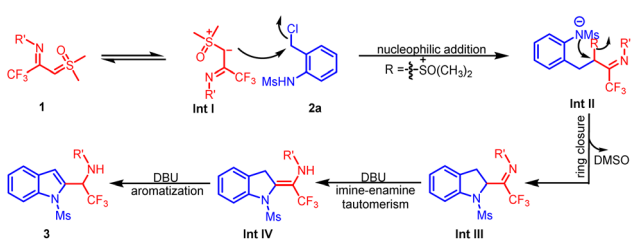


[a] Reaction conditions: **1** (0.2 mmol), **2** (0.1 mmol), DBU (0.15 mol), toluene (0.05 M), 40 °C, 8 h. [b] Yield of isolated product.



Scheme 3 Gram-scale experiment and control experiments.

Based on the results of control experiments and previous work,[16b,24] a plausible mechanism for the synthesis of 2-(α-trifluoromethylamino)indole was proposed. As depicted in Scheme 4, the zwitterionic intermediate **Int I**, derived from imidoyl sulfoxonium ylide **1**, reacts with the ambiphilic reagent **2a** to furnish the intermediate **Int II**. Subsequently, trifluoroacetimidoyl-substituted indoline **Int III** is formed *via* an S_N2 ring closure of **Int II**. This is followed by a DBU-mediated imine-enamine tautomerization of **Int III** to give **Int IV**, which finally undergoes base-promoted aromatization to afford the expected product **3**.



Scheme 4 Proposed mechanism.

Conclusions

In summary, we have discovered a DBU-promoted one-pot protocol to offer the 2-(α-trifluoromethylamino)indole core using CF_3-imidoyl sulfoxonium ylides and *N*-(*ortho*-chloromethyl)aryl amides as substances. This approach offers a novel strategy for installing α-CF_3 amino moieties into heterocycles under mild conditions, as well as a new route for indole construction. Mechanistic studies suggests that this reaction might proceed *via* a stepwise cascade to afford the corresponding indole analogs.

Author contributions

Conceptualization, Tao Jiang and Xiaoke Zhang; methodology, Yang Pan; investigation, Yang Pan, Yin Wang, Xiwei Zhou, Tengxiaoxiao Liu, and Xiaoke Zhang; writing—original draft preparation, Yang Pan and Xiaoke Zhang; writing—review and editing, Tao Jiang; supervision, Tao Jiang, Xiaoke Zhang; project administration, Tao Jiang.

Conflicts of interest

There are no conflicts to declare.

Data availability

CCDC 2504930 contains the supplementary crystallographic data for this paper.[25]

The data supporting this article have been included as part of the supplementary information (SI). Supplementary information: experimental procedures, characterization data, and copies of the ^{1}H NMR, ^{13}C NMR and HRMS spectra of all compounds are included. See DOI: https://doi.org/10.1039/d6ra01734g.

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