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An Effective [Fe^{III}(TF$_4$DMAP)Cl] Catalyst for C−H Bond Amination with Aryl and Alkyl Azides

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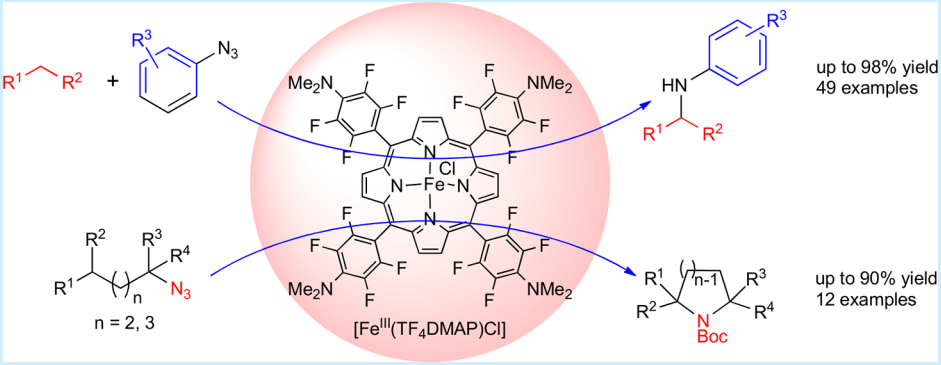
Supporting Information



ABSTRACT: [Fe^{III}(TF$_4$DMAP)Cl] can efficiently catalyze intermolecular sp^3 C−H amination using aryl azides and intramolecular sp^3 C−H amination of alkyl azides in moderate-to-high product yields. At catalyst loading down to 1 mol %, the reactions display high chemo- and regioselectivity with broad substrate scope and are effective for late-stage functionalization of complex natural/bioactive molecules.

Catalytic C−H amination with nitrene sources represents a general and efficient approach to the formation of C−N bonds, which are ubiquitous in molecules of pharmaceutical and/or biological interest.[1] The ubiquity of chemically similar C−H bonds in complex molecules presents major challenges for site-selective C−H amination, including direct functionalization of C−H bonds, via nitrene insertion, for late-stage modification of natural products.[2] Among generally used nitrene sources (e.g., *N*-arylsulfonylimino phenyliodinanes,[3] bromamine-T,[4] chloramine-T,[5] and organic azides[6]), organic azides offer a broad nitrene scope and high atom efficiency. Transition-metal catalysts are well documented to be effective for the decomposition of these nitrene sources to generate metal−nitrene intermediates.[7] Compared with other transition-metal catalysts, iron complexes are inexpensive and biocompatible and often exhibit unique catalytic activity in nitrene insertion reactions of organic azides.[8−10,11b,d−k] For example, iron−dipyrrinato complex[9] and N-heterocyclic carbene iron(III) porphyrin[10] were found to be effective for C−H amination of alkyl azides, which remain a formidable challenge for the commonly used Rh and Cu catalysts. In view of the importance of iron catalysts in C−H amination via nitrene transfer,[8−11] the development of a more efficient iron catalyst for C−H amination using organic azides as nitrene sources with high selectivity and broad substrate scope (covering complex molecules/natural products) is highly appealing. Our recent work revealed that [Fe^{III}(TF$_4$DMAP)Cl][12] (Figure 1, TF$_4$DMAP = *meso*-tetrakis(*o*,*o*,*m*,*m*-tetrafluoro-*p*-(dimethylamino)phenyl)porphyrinato dianion) is efficient in catalyzing anti-Markovnikov oxidation of terminal aryl alkenes to aldehydes with H_2O_2 as a terminal oxidant,[13] which involves a reactive iron−oxo porphyrin intermediate. This

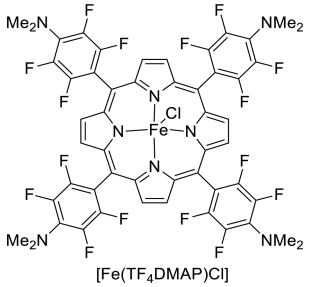


Figure 1. Structure of iron porphyrin [Fe(TF$_4$DMAP)Cl].

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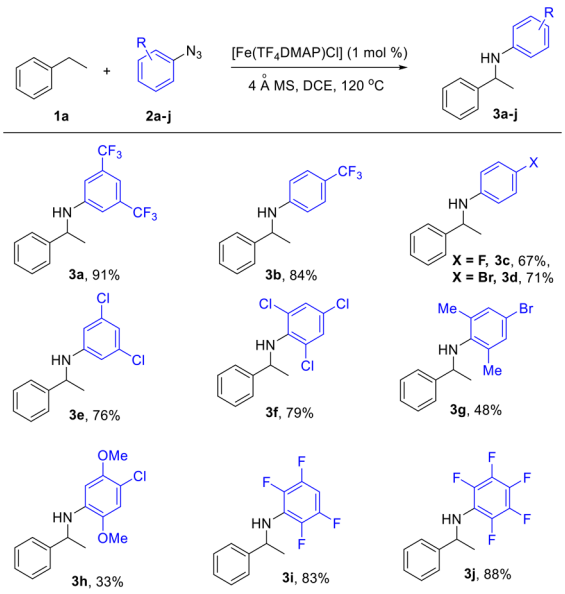
finding promoted us to examine the catalytic activity of this complex in isoelectronically related nitrene transfer/insertion reactions. Herein, we report an efficient C−H amination with aryl and alkyl azides catalyzed by [Fe^{III}(TF_4DMAP)Cl] under thermal conditions and its application in derivatization of natural products.

At the outset, we evaluated the catalytic activity of [Fe^{III}(TF_4DMAP)Cl] in the C−H amination of ethylbenzene (**1a**) with 3,5-bis(trifluoromethyl)phenyl azide (**2a**); the desired amine **3a** was obtained in 91% isolated yield when the reaction was performed in DCE at 120 °C for 12 h. Under the optimized reaction conditions, we investigated the scope of aryl azides as the nitrene source using **1a** as a model substrate (Scheme 1). High yields (67−84%) of C−H amination

Scheme 1. Scope of Aryl Azides.[a]

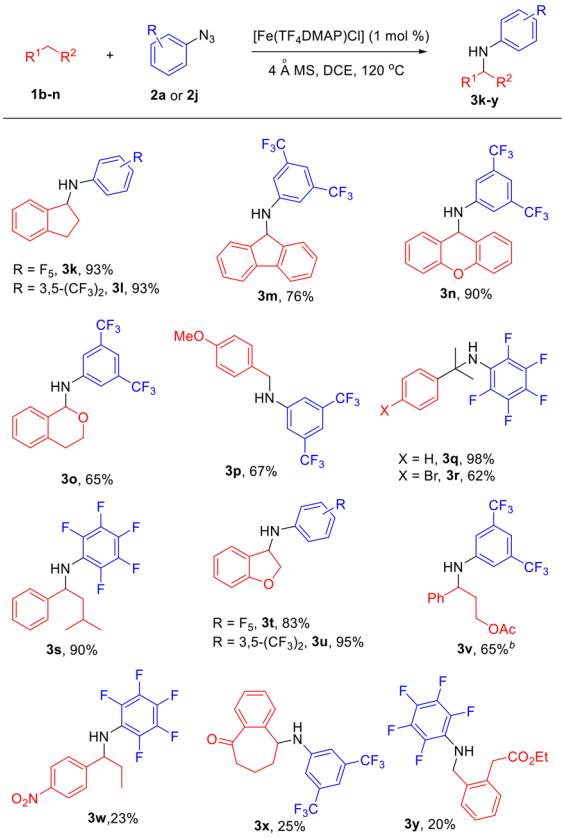


[a]Conditions: **1a** (5.0 mmol), **2** (0.5 mmol), [Fe(TF_4DMAP)Cl] (1 mol %), 4 Å MS (120 mg), DCE (2.0 mL), under argon, 120 °C; isolated yield.

products were obtained for mono-*para*-substituted aryl azides (**3b**−**d**). 3,5-Dichlorophenyl azide (**2e**) and 2,4,6-trichlorophenyl azide (**2f**) afforded the corresponding amines **3e** and **3f** in 76% and 79% yield, respectively. When aryl azides containing electron-donating substituent(s) (**2g** and **2h**) were used as the nitrene sources, the corresponding amines **3g** and **3h** were obtained in moderate yields. The beneficial effect of incorporating multi-electron-withdrawing substituents was also demonstrated in the reactions of 2,3,5,6-tetrafluoro- and pentafluorophenyl azides (**2i**, **2j**) which afforded the corresponding amines in high yields (**3i**, 83%; **3j**, 88%).

We further investigated the scope of C−H amination under the optimized conditions. As depicted in Scheme 2, when benzylic C−H bonds were subjected to the reaction with aryl azide **2a** or **2j** as nitrene source, the corresponding C−H amination products (**3k**−**y**) were obtained in 20−98% yields. It is noteworthy that for 4-methylanisole, containing a primary benzylic C−H bond, the C−H amination proceeded smoothly in 67% yield (**3p**). The reaction protocol also worked well for tertiary benzylic C−H bonds as shown in the cases of **3q** (98% yield) and **3r** (62% yield). When the substrates had both benzylic C−H bonds and tertiary C−H bonds or C−H bonds

Scheme 2. Amination of Benzylic C−H Bonds with Aryl Azides[a]



[a]Conditions: **1** (5.0 mmol), **2** (0.5 mmol), [Fe(TF_4DMAP)Cl] (1 mol %), 4 Å MS (120 mg), DCE (2.0 mL), under argon, 120 °C; isolated yield; [b]2.5 mmol of substrate was used.

adjacent to oxygen atom, benzylic C−H bonds were preferentially aminated in good-to-high yields (**3s**−**v**). The electron-withdrawing substituent on the phenyl ring was found to reduce the activity of benzylic C−H bonds, leading to low product yields (**3w**−**y**). This is in agreement with the electrophilic nature of the reactive iron−imido/nitrene intermediate.

Given the good results of benzylic C−H bond amination, we then investigated the application of the amination for unactivated C−H bonds (Scheme 3). Tetrahydrofuran was reactive toward the C−H amination, giving the corresponding products in 67−75% yields (**4a**, **4b**). Tetrahydropyran and 1,4-dioxane also worked well with **2a** to give **4c** and **4d** in 65% and 60% yield, respectively. Treatment of cyclooctane with **2j** in the presence of [Fe(TF_4DMAP)Cl] (3 mol %) gave C−H amination product **4e** in 60% yield. When 1°, 2°, or 3° aliphatic C−H bonds and C−H bonds adjacent to the oxygen atom were present in the substrates, the Fe(III)-catalyzed C−H amination occurred at 3 °C−H bond preferentially and in 40−72% yields (**4f**−**o**). The site selectivity of the C−H amination for the substrates bearing multiple 3° C−H bonds was also examined by using two derivatives of dihydrocitronellol, each containing two 3° C−H sites. In both cases, the amination was favored at the site distal to the electron-withdrawing group (bromo or acetate, **4p**, **4q**). This regioselectivity could be attributed to the electron deactivation of the C(3)−H bond by an electron-withdrawing group.

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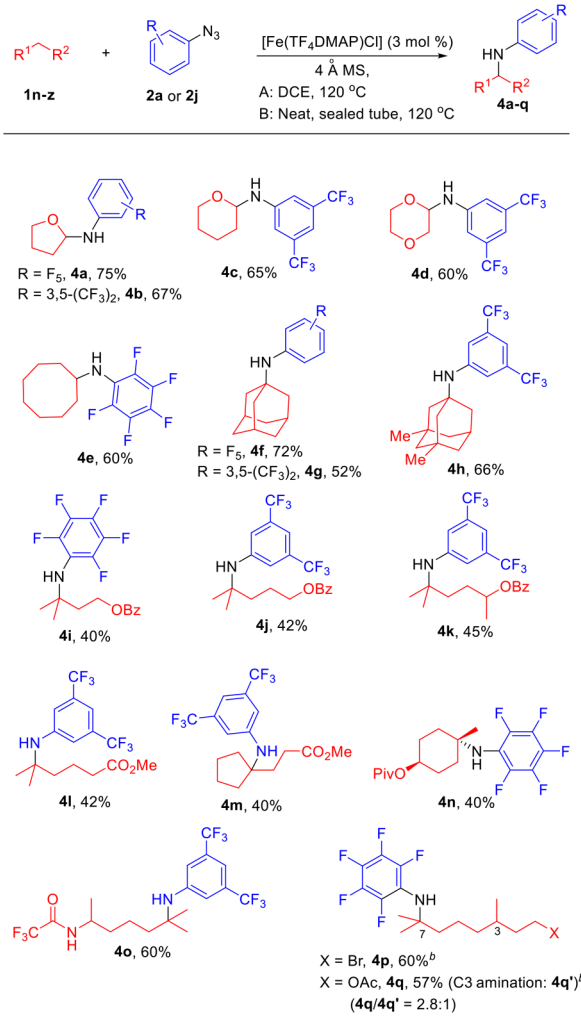
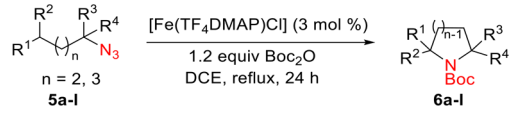
Scheme 3. Amination of Unactivated C−H Bonds with Aryl Azides[a,b]



[a]Conditions A: **1** (1.0 mmol), **2** (0.2 mmol), [Fe(TF$_4$DMAP)Cl] (3 mol %), 4 Å MS (120 mg), DCE (1.0 mL), under argon, 120 °C; isolated yield; [b]Conditions B: the same as A except using neat **1** (1.0 mL) without DCE.

Table 1. Intramolecular C−H Amination of Alkyl Azide[a]



entry	substrate	product	conv. (%)	yield (%)
1	**5a**	**6a**	>99	66
2	**5b**	**6b**	>99	60
3	**5c**	**6c**	>99	82
4	**5d**	**6d**	>99	60
5	**5e**	**6e**	>99	76
6	**5f**	**6f**	>99	62
7	**5g**	**6g**	>99	78
8	**5h**	**6h**	>99	90
9	**5i**	**6i**	>99	62
10	**5j**	**6j**	>99	86
11	**5k**	**6k**	>99	45[b]
12	**5l**	**6l**	>99	78

[a]Conditions: **2** (0.3 mmol), [Fe(TF$_4$DMAP)Cl] (3 mol %), Boc_2O (0.36 mmol), 4 Å MS (120 mg), DCE (2.0 mL), under argon, 120 °C; isolated yield. [b]dr = 4.2:1.

The intramolecular C−H amination of alkyl azides[9a,c,10,11g,i] is challenging because of the facile 1,2-hydride shift of the alkylnitrene intermediate. Our recent work[10] revealed that the N-heterocyclic carbene iron(III) porphyrin [Fe^{III}(TDCPP)(IMe)$_2$]I (TDCPP = *meso*-tetrakis(2,6-dichlorophenyl)porphyrinato dianion) can efficiently catalyze intramolecular C−H amination of a broad array of alkyl azides. However, 10 mol % catalyst loading was required to guarantee high substrate conversion and product yields for the reaction. This promoted us to test the catalytic activity of [Fe(TF$_4$DMAP)Cl] for the intramolecular C−H amination of alkyl azides with lower catalyst loading. As depicted in Table 1, a variety of alkyl azides underwent intramolecular C−H amination in the presence of 3 mol % of [Fe(TF$_4$DMAP)Cl] in 45−90% isolated yields. It is noteworthy that when alkyl azide **5f** was used, the six-membered piperidine product was selectively formed in 62% yield, showcasing the strong preference for amination at the 3° C−H bond (entry 6, Table 1). For secondary alkyl azides **5g** and **5h**, the corresponding pyrrolidines were obtained in 78−90% yields (entries 7 and 8, Table 1). Notably, when a cyclic secondary alkyl azide **5i** was subjected to the reaction, a tropane derivative with a bicyclic structure prevalent in a wide range of alkaloids was isolated in 62% yield (entry 9, Table 1). Similarly, the α-azido ketone **5j** gave a tropane analogue in 86% yield, which is an important intermediate for the synthesis

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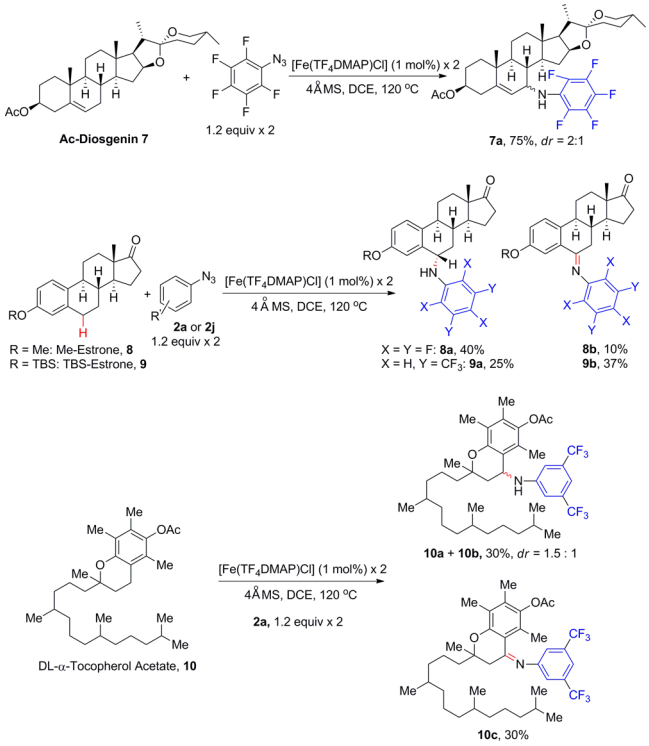
of cocaine. Interestingly, a secondary alkyl azide **5k** derived from L-menthol underwent intramolecular amination exclusively at a 1° C−H bond to give the *cis*-octahydroindole in 45% yield with a dr ratio of 4.2:1 (entry 11, Table 1), revealing that primary unactivated aliphtic C−H bonds are also amenable for amination in this protocol when competing secondary or tertiary C−H bonds are inaccessible. For substrate **5l** bearing a cyclopropyl ring at C4, the iron-porphyrin-catalyzed reaction gave the pyrrolidine **6l** in 78% yield, with no cyclopropyl ring-opening product(s) observed in the crude ^{1}H NMR spectra. This is indicative of a concerted or very fast radical rebound mechanism for the C−H amination.

The high efficiency of the [Fe^{III}(TF$_4$DMAP)Cl] catalyst was further demonstrated by its applicability to the late-stage functionalization of complex natural product derivatives, which possess various C−H bonds and functional groups (Scheme 4). With **2j** as the nitrene source, Ac-diosgenin (**7**) underwent

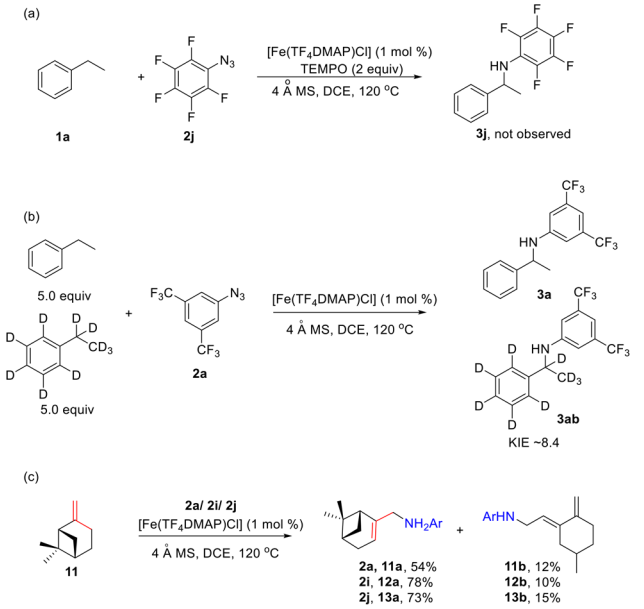
Scheme 4. Late-Stage C−H Functionalization of Natural Product Derivatives



amination exclusively at the allylic C−H bond remote from the −OAc group, resulting in 75% isolated yield with a dr ratio of 2:1; Me-estrone (**8**) reacted exclusively at the benzylic C−H bond with azide **2j**, giving the desired amine **8a** and its imine analogue **8b** in 40% and 10% yield, respectively, while amination of TBS-estrone with azide **2a** gave amine **9a** and the corresponding imine **9b** in 25% and 37% yield, respectively (no imine products were observed for the examples in Schemes 2 and 3 under the reaction conditions indicated therein). For the reaction of racemic DL-α-tocopherol acetate (**10**), the desired benzylic C−H amination products **10a** (*cis*) and **10b** (*trans*) were isolated in a combined 30% yield with a dr ratio of 1.5:1, along with 30% of imine product **10c**.

Several experiments were performed to shed light on the mechanism of the catalytic C−H amination process. First, addition of TEMPO to a standard reaction mixture of **1a** and **2j** completely shut down the reaction, with no amination product **3j** observed (Scheme 5a). Second, the amination of a

Scheme 5. Reaction Mechanism Study



mixture of ethylbenzene and ethylbenzene-d_{10} with **2a** gave a kinetic isotope effect (KIE, k_H/k_D) of ~8.4 (Scheme 5b), suggesting that C−H bond cleavage is the rate-determining step. The KIE value is in the range of k_H/k_D ~5−13 reported for the H atom abstraction by metal−nitrene/imido complexes.[14,15] Third, when (−)-β-pinene **11** was subjected to the reactions with three different azides **2a**, **2i**, and **2j** separately, two types of allylic C−H aminated products were observed in each case (Scheme 5c), with the major ones presumably arising from a radical rearrangement or an alkene aziridination and subsequent ring-opening process.[11b]

In summary, we have demonstrated that [Fe^{III}(TF$_4$DMAP)Cl] can efficiently catalyze C−H amination of aryl azides and alkyl azides with broad substrate scope and with high chemo- and regioselectivity. The catalytic system is highly effective for amination of a variety of sp^3 C−H bonds including unactivated aliphatic C−H bond, benzylic C−H bond, allylic C−H bond, and 1°−3° C−H bonds. This catalytic protocol is also applicable to late-stage amination of complex natural product derivatives.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.8b03765.

Detailed experimental procedures, characterization of new compounds, and copies of NMR spectra (PDF)

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Notes

The authors declare no competing financial interest.

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