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Radical C-H Bond Trifluoromethylation of Alkenes by High-Valent Copper(III) Trifluoromethyl Compounds

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Abstract. A general and selective method is developed that allows direct vinylic C-H bond trifluoromethylation of 1,1-diarylalkenes by a high-valent copper(III) trifluoromethyl complex, producing biologically active trifluoromethylated alkenes (as well as trifluoromethylated carbocyclic compounds). This fundamental reactivity of Cu(III)-CF₃ compounds has thus far been unknown. The presence of a tertiary amine is crucial to this reaction, acting as both a weak base and a single electron transfer (SET) promoter to abstract the vinylic hydrogen. This method starts from bulk olefins under cost-effective conditions (without the need for external noble metal photocatalyst or stoichiometric amounts of oxidant), and thus is valuable for practical and sustainable applications.

Keywords: Trifluoromethylation; Cu(III)-CF₃ compounds; C-H activation; Trifluoromethylated alkenes; Tertiary amine

Trifluoromethylated alkenes are an important structural motif occurring in a range of biologically active compounds. Therefore, great efforts have been stimulated to develop efficient methods for constructing the vinyl-CF₃ bond. Despite the impressive progress achieved for trifluoromethylation of vinyl electrophiles^[1] (e.g. halides, tosylates) (Scheme 1a) and nucleophiles (e.g. vinyl boronic acids,^[2] carboxylic acids^[3]) (Scheme 1b), and hydrotrifluoromethylation of alkynes,^[4] direct alkenes C-H bond trifluoromethylation is much less developed. Considering the ready availability and bulkness of olefins, there is great demand for developing direct alkene trifluoromethylation methods which are more straightforward and sustainable without the need for any prefunctionalization.

Very recently, activated alkenes functionalized with strong electron-withdrawing or -donating groups, typically acrylamides and enamides, have been studied as substrates for C-H trifluoromethylation (Scheme 1c).^[5] However, general methods for simple unactivated alkenes remain very rare.^[6] Notably, Cho et al. reported a photocatalyst induced *E*-selective C-

H trifluoromethylation of simple aliphatic alkenes with gaseous CF₃I.^[6a] Qing et al. reported stereoselective orthogonal access to both E- and Ztrifluoromethylated styrenes exploiting a different combination of photocatalyst and CF₃ reagents (Togni's or Umemoto's reagent) for the trifluoromethylation of ortho-amino styrenes.[6b] Loh reported copper-catalyzed et al. а trifluoromethylation of styrenes with CF₃SO₂Na as the CF₃ reagent in the presence of excess peroxide as the oxidant.^[6c] Despite these elegant advances, either expensive noble metal photocatalyst assisted by photo-irradiation or excess external oxidants ar required wherein substrates containing redox sensitive groups are not compatible, thus limiting the generality and wide application of the methods. Therefore, alternative methods are desirable for C-H trifluoromethylation of simple alkenes.



(c) direct C-H trifluoromethylation of activated alkenes



(d) This study: direct C-H trifluoromethylation of unactivated



Scheme 1. Methods for vinyl-CF₃ bond formation.

In continuation of our recent interest in developing Cu(III)- CF_3 chemistry,^[7-9] we report herein the direct C-H bond trifluoromethylation of 1,1-diarylalkenes with a Cu(III)-CF3 compound under simple conditions (Scheme 1d), without the need for either photocatalyst/photoirradiation or external oxidant. The Cu(III)-CF₃ compound offers CF₃ radical to selectively add to the alkene double bond, giving a crucial carbon-centred tertiary radical that is sufficiently stabilized with two aryl and an alkyl groups (Scheme 1d). The presence of DBU facilitates single electron transfer (SET) and hydrogenabstraction events to occur with the tertiary radical to give the desired trifluoromethylated alkenes. Notably, such trifluoromethylated alkenes with two vicinal aryl groups are structural motif with important biological activity (e.g., anti-breast cancer drug panomifene), but are very challenging to prepare.^[8c,10]

Table 1. Optimization of reaction conditions.^{a)}

(p	ohen)Cu ^{III} (C	$(F_3)_3 + Ph$ Ph	$= \frac{\text{base, solve}}{\text{temp, N}_2, 1}$	$\stackrel{\text{ent}}{\longrightarrow} \stackrel{\text{Ph}}{\longrightarrow} \stackrel{\text{Ph}}{\longrightarrow}$	CF ₃ F	'n C	≻F3
	1 (0.2 mm	nol) 2a (0.2	mmol)	3a		4a	
	entry	base	solvent	temp	yield 3a	(%) ^b 4a	
	1	CsF	DMF	100	12	29	
	2	CsF	DCE	100	12	39	
	3	NaOtBu	DCE	100	7	5	
	4	Et ₃ N	DCE	90	49	49	
	5	DBU	DCE	90	56	18	
	6	DABCO	DCE	90	37	18	
	7	pyridine	DCE	90	33	35	
	8	DBN	DCE	90	32	6	
	9	DMAP	DCE	90	19	7	
	10		DCE	90	18	25	
_				N-	-	N	_
		DBU D	ABCO D	BN DN	IAP	1	

^{a)} Reaction conditions: **1** (0.2 mmol), **2a** (0.2 mmol), base (0.6 mmol), 4,4'-difluorobiphenyl (0.2 mmol, internal standard) and solvent (2 mL) stirred under N_2 . ^{b) 19}F NMR yield.

Our study began by reaction of (phen)Cu^{III}(CF₃)₃ (1) (phen denotes phenanthroline)^[7b] and 1,1diphenylethylene (2a) under N₂ condition. With CsF or NaOtBu as the additive, the conversion of 2a is low to give a mixture containing majorly the desired byproduct 4a arising from alkene **3a** and hydrotrifluoromethylation (Table 1, entries 1-3).^[6f,11,12] It was delighting to see that when an organic base Et_3N was used, the conversion of 2a was greatly improved to almost quantitative, albeit the selectivity was low to give a mixture of 3a and 4a in a 1:1 ratio (entry 4). To improve the selectivity, other organic bases were then examined including DBU (1,8-diazabicyclo[5.4.0]undec-7-ene), DABCO (1,4diazabicyclo[2.2.2]octane), pyridine, DBN (1,5diazabicyclo[4.3.0]non-5-ene) and dimethylaminopyridine (DMAP) (entries 5-9). This led to the finding that DBU is a suitable organic base compromising the efficiency and the selectivity. The organic base is crucial to this reaction; without it, the reaction could not give the desired **3a** or **4a** in significant amounts (entry 10).



Scheme 2. Substrate scope of this C-H trifluoromethylation reaction. Both ¹⁹F NMR and isolated yields (in parentheses) are provided if available. The E/Z ratios were determined by ¹⁹F NMR analysis of the crude reaction mixture.

With the optimized conditions in hand (Table 1, entry 5), a range of 1,1-diarylalkenes were studied to show the synthetic compatibility (Scheme 2).^[13] Functional groups such as alkyl, phenyl, methoxy, amino, fluoro, chloro, nitro and cyano are tolerated on the phenyl ring. Heteroarenes, such as pyridine and thiophene are also compatible to produce **30** and **3p**. It is interesting to see that **2q** with the two phenyl rings tethered by an ethylene bridge, was able to

produce the desired 3q in a 21% isolated yield. Generally, symmetric 1,1-diarylalkenes give a single dominant product (**3a-3f**) while unsymmetrical 1,1diarylalkenes give a mixture of E/Z isomers in a ratio ranging from 1:1 to 6:1. The **2r** with a phenyl and a methyl at the alkenes gave a complex mixture that was inseparable containing the desired **3r**. This result also highlights the importance of two aryl/heteroaryl groups for stabilizing the potential tertiary radical (Scheme 1d) and thus achieving high efficiency and selectivity.

Crucial electronic effects of aryl/heteroaryl groups have been observed in two aspects. First, electrondonating groups improve the reaction efficiency with higher yields while substrates with electronwithdrawing groups react sluggishly. This is obvious by the reactivity order for the preparation of **3b-d** > 3a > 3e-f, and the better reactivity toward 3p with an electron-rich thiophene than to 30 with an electronpoor pyridine. Second, the electronic property of the aryl/heteroaryl groups has a significant influence on the diastereoselectivity for unsymmetrical 1,1diarylalkenes. The isomeric products with the CF₃ trans to the more electron-rich aryl group are more favored than the isomers with *cis*-CF₃. The extent of this diastereo bias largely depends on the electronic difference between the two geminal aryl groups, and becomes more predominant for substrates with two electronically distinct aryl groups, such as 3m, 3o and 3p, as reflected by the higher ratios of diastereoisomers.



Scheme 3. Application to the synthesis of trifluoromethylated indene.

As a further demonstration of the synthetic potential, reaction of 1,3-diene 2s with Cu(III)-CF₃ compound 1' can efficiently give trifluoromethylated indene 3s in a high yield of 84% from a formal aryl-trifluoromethylation (Scheme 3). A radical species I is proposed, generated from trifluoromethylation of the right double bond, to participate in aromatic radical attack to produce radical species II. Aromatizing hydrogen abstraction from II would give favorably indene 3s. 2,2,2-Trifluoroethylated indenes are potentially an important motif for electric/optic materials design.

Radical trapping study was conducted in the presence of two equivalents of additional TEMPO

(2,2,6,6-tetramethylpiperidyloxy) (Scheme 4a). This led to a low NMR yield of only 9% for 3a, implying that radical species might be involved in the reaction course. To prove the intermediacy of radical species, radical clock experiment using α -cyclopropyl styrene 2t as the substrate was conducted under the optimized conditions with the expectation of the occurrence of ring opening of the cyclopropyl group. Indeed, a trifluoromethylated dihydronaphthalene 5 was obtained in 24% NMR yield as yellow oil (Scheme 4b). The formation of 5 is rationalized by the ring opening of cyclopropyl group in the initial intermediate III, giving a homoallylic radical IV which then proceeds via aromatic radical substitution to give 5. This result of radical clock experiment, the radical trapping study (Scheme 4a), the formation of indene 3s using 1,3-dienes in Scheme 3, and the observation of byproduct of type 4a in most of our studies, provide compelling evidence supporting the involvement of radical trifluoromethylation of the alkene double bond with the Cu(III)-CF₃ compound.



Scheme 4. Radical scavenger and radical clock experiments.



Scheme 5. Plausible mechanism.

As a result, a plausible mechanism involves the initial addition of a CF₃ radical to the double bond to form a key tertiary carbon radical A (Scheme 5).^[14] The fate of A depends largely on the reaction conditions. In the presence of DBU, this intermediate tends to undergo SET to give tertiary carbocation **B**,^[8,15] which is then deprotonated by DBU to produce the desired product 3.^[16] The released ammonium and the phenCu(I)(CF_3)₂ anion generated from SET process are proposed to participate in further exothermic decomposition, for example, pathways involving the generation of nitrogen ylide^[17] as well as R₃N•HF,^[8e] which provides strong thermodynamic driving force for the formation of product 3. Alternatively, without the efficient assistance of a tertiary amine, intermediate A might also take a hydrogen from the environment (e.g. the solvent) to produce the hydrotrifluoromethylation byproduct 4a. In the cases of using 2s and 2t as the substrates, the initial radical A from radical trifluoromethylation (i.e., intermediate I and III) can cyclize with neighbouring aryl ring (Scheme 3) or rearrange to another radical species driven by subsequent cyclization to a stable cyclic compounds (Scheme 4b).

In conclusion, this study describes a direct C-H bond trifluoromethylation of simple alkenes by Cu(III)-CF₃ compound in synergy of a tertiary amine. This reaction occurs under simple and cost-effective conditions, without the need for external oxidant or photocatalyst. It is attractive for preparing 1,1-diaryl trifluoromethylated alkenes and trifluoromethylated carbocycles from simple alkenes. This study consolidates one fundamental reactivity property of crucial Cu(III)-CF₃ compounds that is currently unknown.

Experimental Section

In an oven-dried 25-mL Schlenk tube equipped with a magnetic stir bar were charged with phenCu(CF₃)₃ (1) (90 mg, 0.2 mmol) and 1,1-disubstituted alkene (2) (0.2 mmol). The Schlenk tube was evacuated and refilled with dry nitrogen. A DCE (2 mL) solution of DBU (0.6 mmol) was then added by syringe. The contents in the tube were vigorously stirred for 18 hours at 90 °C (seated in an oil bath). The reaction mixture was then allowed to cool to room temperature, diluted with CH₂Cl₂ and then filtrated. The filtrate was concentrated by vacuum evaporation. The resulting residual was purified by preparative TLC (petroleum ether/ethyl acetate = 30:1 (v/v)) to provide trifluoromethylated alkene products **3**.

Acknowledgements

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C-H trifluoromethylation of alkenes by Cu(III)-CF₃ compound



crucial stabilized tertiary radical intermediate