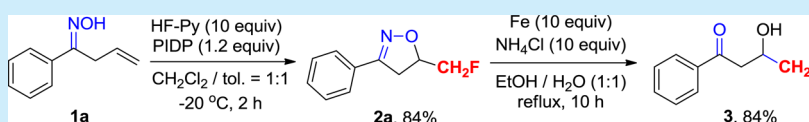


Iodine(III)-Mediated Oxy-fluorination of Alkenyl Oximes: An Easy Path to Monofluoromethyl-Substituted Isoxazolines

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Supporting Information



ABSTRACT: A highly regioselective intramolecular oxy-fluorination of alkenyl oximes was achieved. This new transformation represents an efficient method for the preparation of monofluoromethyl-substituted isoxazolines. The synthetic application of the oxy-fluorination product was demonstrated by a one-step synthesis of monofluoromethyl-substituted β -hydroxyl ketone derivatives.

Research in the field of organofluorine chemistry has increased dramatically in recent years due to the unique electronic properties of fluorine and its ability to induce conformational and electronic changes in organic molecules.¹ In drug discovery, the introduction of fluorine for drug candidate modification has become an important strategy.² Compounds containing C–F bonds also play important roles in material³ and agrochemical sciences.⁴ Therefore, it is not surprising that studies focused on the synthesis of fluorine-containing organic compounds are attracting more and more attention. Recently, the introduction of monofluoromethyl groups (–CH₂F) into drug candidates had been utilized as an important strategy in drug discovery. Indeed, –CH₂F motifs play important roles in many biologically active compounds, such as afloqualone, fluticasone propionate, and the anesthetic sevoflurane (Figure 1).⁵ Compared with the numerous reports

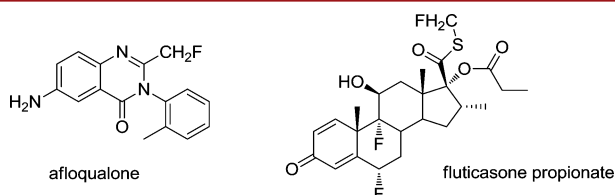


Figure 1. Representative bioactive molecules containing monofluoromethyl groups.

that exist on the preparation of trifluoromethyl⁶ and difluoromethyl-containing⁷ molecules, research on the synthesis of monofluoromethyl substituted molecules is very limited.⁸

The isoxazolines skeleton is found in several biologically active agents and also plays a versatile role in organic synthesis.⁹ As part of our research on the synthesis of isoxazolines,¹⁰ we recently became interested in the preparation of monofluor-

omethyl-substituted isoxazolines. Difunctionalization of an alkene through an oxy-fluorination reaction is the most straightforward way to construct a vicinal oxygen fluorine moiety.¹¹ We envisioned that by using an intramolecular oxy-fluorination reaction, an alkenyl oxime might be cyclized to form monofluoromethyl-substituted isoxazolines.¹² However, despite the significant attention received by the synthetic community, there are still some limitations in alkene oxy-fluorination reactions:¹³ (i) the oxy parts in previous studies were mainly restricted to carbons connected to an –OH group, such as simple alcohols and carbonate acids; (ii) electrophilic fluorination reagents, such as Selectfluor (1-chloromethyl-4-fluoro-1,4-diazoniabicyclo[2.2.2]octane bis(tetrafluoroborate)) or NFSI (*N*-fluorobenzenesulfonamide) are mostly used, whereas the nucleophilic fluorine resources were seldom studied.

Hypervalent iodine complexes are well-known as versatile oxidizing reagents for mediating organic reactions involving the formation of new C–C and C–heteroatom bonds.¹⁴ Because of their low toxicity, high stability, ease of handling, and now commercial availability, they have served as useful synthetic oxidants in organic synthesis and have become highly welcome both in the chemical industry and academic laboratories. In the last three decades, hypervalent iodine reagents have been employed to mediate the difunctionalization of alkenes without the use of metal catalysts.¹⁵ In 2000, Hara and Yoneda reported the oxy-fluorination of unsaturated alcohols or carboxylic acids by using difluoroiodotoluene (4-CH₃-PhIF₂) as the promoter, and the yields were between 40% to 60%.^{13d} However, the preparation of ArIF₂ usually requires relatively harsh conditions,

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such as the use of F_2 ,^{16a} XeF_2 ,^{16b} or toxic HgO and Cl_2 ,^{13d} which restricted its further application. Very recently, Li^{17a} and Nevado^{17b} described the intramolecular aminofluorination of alkenes by employing iodine(III) as a mediator, respectively.

In this paper, we report the first example of hypervalent iodine-promoted intramolecular oxy-fluorination of unsaturated oximes. By this method, the biologically interesting monofluoromethyl-substituted isoxazolines were obtained in good yield through fluorocyclization. In the reaction, benign PIDP ($PhI(OPiv)_2$) was used as the oxidant and the cost-effective HF-Py (HF-pyridine) was employed as the F-source. Furthermore, a synthetically useful building block, namely, monofluoromethyl-substituted β -hydroxyl ketone derivatives, can now be synthesized via a simple ring-opening reaction from the isoxazoline product.

We initiated our study with oxime **1a** as a substrate, Et_3N -3HF (10 equiv) as a F-source, and 1.2 equiv of PhIO as an oxidant. The reaction was carried out in dry CH_2Cl_2 under an argon atmosphere at room temperature. However, only 15% of the desired oxy-fluorination product **2a** was obtained (Table 1, entry 1). Reaction temperature screening revealed that $-20^\circ C$ was the most efficient for this reaction (entries 2–5). In all of the above-mentioned cases, some oxy-hydroxylation byproduct **2a'** was also detected in the system. Various fluorination reagents were also tested, and we found that AgF or KF were

completely ineffective for the reaction (entries 6 and 7). Fortunately, when HF-Py was used, no **2a'** was observed in the reaction and the yield increased to 69% (entry 8). Replacement of PhIO by other iodine(III) reagents, namely, PIDA ($PhI(OAc)_2$, entry 9) and PIFA ($PhI(O_2CCF_3)_2$, entry 10), led to the formation of byproducts **2a''** and **2a'''**, respectively. We envisioned that a large substituent, with weak nucleophilicity, on the iodine atom might be able to suppress the generation of this byproduct. To our delight, the bulky I(III) reagent, PIDP, promoted a clean transformation without any side product, and the yield increased to 77% (entry 11). Finally, a solvent survey indicated that the mixed solvent (CH_2Cl_2 /tol = 1:1) was optimal, whereafter an 84% yield of **2a** was obtained (entries 12–20).¹⁸

With the optimal reaction conditions in hand, we next investigated reaction generality (Scheme 1). Aromatic oximes bearing an $-CH_3$ or $-OCH_3$ substituent at the *o*-, *m*-, or *p*-

Scheme 1. Investigation of Substrate Scope^{a,b}

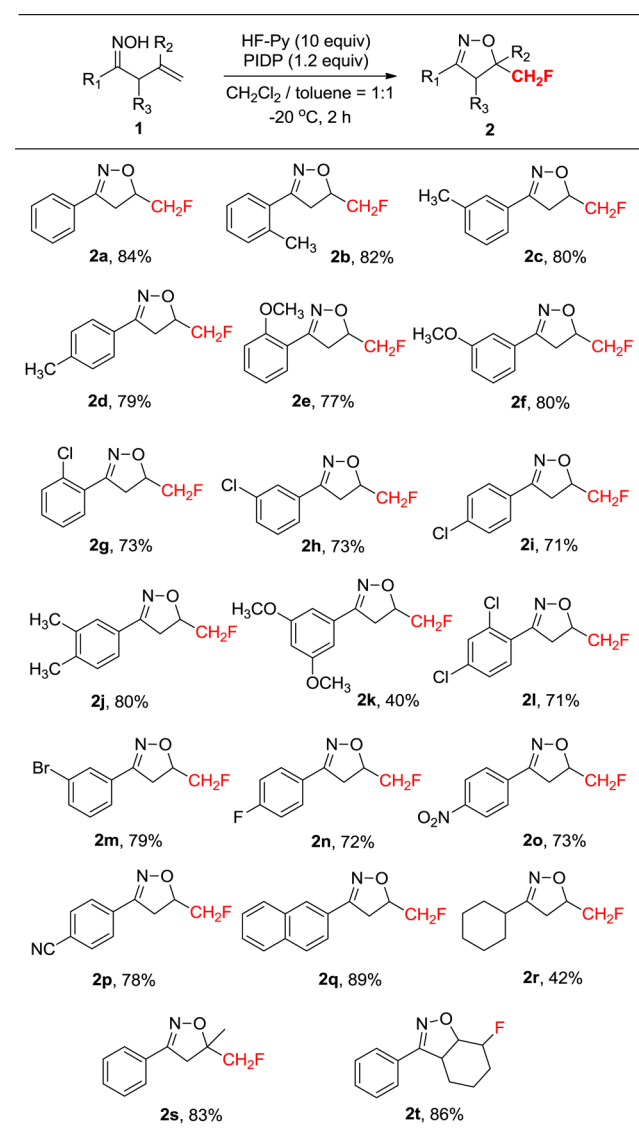


Table 1. Optimization of Reaction Conditions^a

entry	iodine(III)	F source	solvent	T (°C)	yield ^b (%)
1	PhIO	Et_3N -3HF	CH_2Cl_2	rt	15
2	PhIO	Et_3N -3HF	CH_2Cl_2	0	30
3	PhIO	Et_3N -3HF	CH_2Cl_2	-20	56
4	PhIO	Et_3N -3HF	CH_2Cl_2	-40	55
5	PhIO	Et_3N -3HF	CH_2Cl_2	-78	50
6	PhIO	AgF	CH_2Cl_2	-20	0
7	PhIO	KF	CH_2Cl_2	-20	0
8	PhIO	HF-Py	CH_2Cl_2	-20	69
9	PIDA	HF-Py	CH_2Cl_2	-20	58
10	PIFA	HF-Py	CH_2Cl_2	-20	32
11	PIDP	HF-Py	CH_2Cl_2	-20	77
12	PIDP	HF-Py	tol.	-20	76
13	PIDP	HF-Py	Et_2O	-20	68
14	PIDP	HF-Py	THF	-20	trace
15	PIDP	HF-Py	$CHCl_3$	-20	71
16	PIDP	HF-Py	$ClCH_2CH_2Cl$	-20	72
17	PIDP	HF-Py	CH_3CN	-20	trace
18	PIDP	HF-Py	Cl-Ph	-20	55
19	PIDP	HF-Py	xylene	-20	66
20	PIDP	HF-Py	CH_2Cl_2 /tol ^c	-20	84

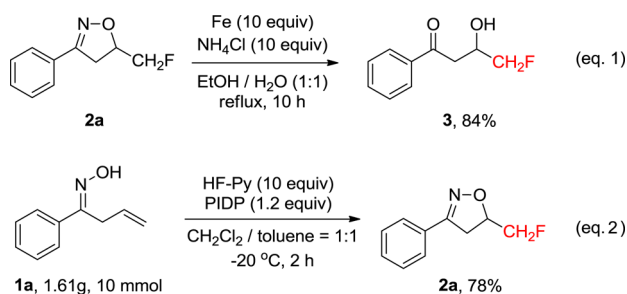
^aAll reactions were carried out by using 0.2 mmol of **1a**, 2 mmol of F source, 0.24 mmol of iodine(III), and 2 mL of solvent with stirring for 2 h. ^bIsolated yields. ^cThe ratio of CH_2Cl_2 and toluene is 1:1.

^aAll reactions were carried out by using 0.4 mmol of **1**, 10 equiv of HF-Py, 1.2 equiv of PIDP, and 4 mL of solvent (CH_2Cl_2 /toluene = 1/1) at $-20^\circ C$ for 2 h. ^bYields refer to isolated yields.

position on the aryl ring performed well under the standard conditions (**2b–f**). Substrates with a 2-, 3-, or 4-chlorine-substituted phenyl also reacted smoothly in good yield (**2g–i**). When disubstituted aromatic oximes were employed, different reactivities were observed. Oximes with a 3,4-dimethylphenyl group or a 2,4-dichlorophenyl showed good results (**2j** and **2l**), whereas a 3,5-dimethoxyphenyl-substituted oxime only led to a 40% yield (**2k**). Substrates with other types of substituents, such as *p*-F-phenyl, *p*-Br-phenyl, *p*-NO₂-phenyl, *p*-CN-phenyl, or 2-naphthalenyl, all worked well and gave the desired products in good yields (**2m–q**). Under the stated conditions, aliphatic oximes provided moderate conversions to the desired products (**2r**, 42%). We were pleased to find that the present method could be successfully applied to construct a monofluoromethyl-substituted isoxazoline containing a quaternary carbon center (**2s**, 83%). Finally, an oxime with an internal alkene was tested and gave the corresponding cyclization product **2t** in 86% isolated yield.

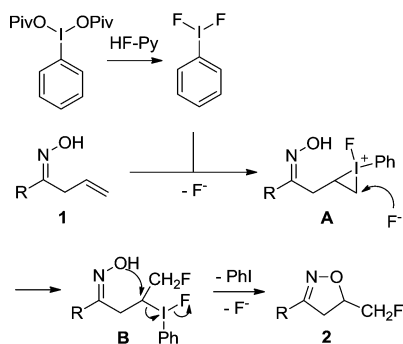
To further illustrate the synthetic utility of the oxy-fluorination product, isoxazoline **2a** was applied to a reductive ring-opening reaction. The reaction proceeded smoothly and gave the monofluoromethyl substituted β -hydroxyl ketone **3** in 84% yield (Scheme 2, eq 1). Moreover, the oxy-fluorination reaction could be conducted on a 10 mmol scale without affecting the reactivity of the process (eq 2).

Scheme 2. Synthetic Utility of Current Method



On the basis of previous reports and our own considerations,^{14,17} the pathway below can be proposed for the oxy-fluorination reaction (Scheme 3). At the beginning, PIDP

Scheme 3. Proposed Mechanism



reacted with HF-Py in situ and forms PhIF₂ through ligand exchange. The alkene then reacted with PhIF₂ to generate an iodonium cation **A**, which was attacked by a F⁻ to form intermediate **B**. A subsequent intramolecular nucleophilic attack of -OH and reductive elimination of I(III) led to the desired product **2**.

In conclusion, we have developed a highly regioselective metal-free method for the intramolecular oxidative oxy-fluorination of unactivated terminal alkenes, in which HF-Py is employed as the fluorine source in the presence of PIDP. This new transformation represents an efficient method for the preparation of monofluoromethyl-substituted isoxazolines. Moreover, the synthetic utility of the product was demonstrated by a one-step synthesis of monofluoromethyl-substituted β -hydroxyl ketone derivatives in good yield.

■ ASSOCIATED CONTENT

§ Supporting Information

Experimental details, characterization data, and NMR spectra. The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs-orglett.5b01646.

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Notes

The authors declare no competing financial interest.

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