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Copper-catalysed direct difluoromethylselenolation of aryl boronic acids with Se-(difluoromethyl) 4-methylbenzenesulfonoselenoate

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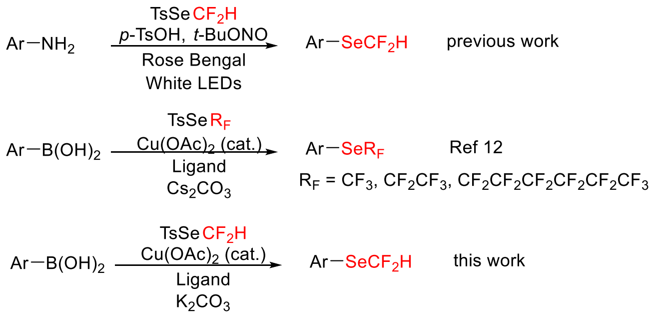
ABSTRACT

In this study, we developed the first copper-catalysed direct difluoromethylselenolation of aryl boronic acids using Se-(difluoromethyl) 4-methylbenzenesulfonoselenoate as a difluoromethylselenolation reagent. Owing to the cheap and readily accessible reagents, broad substrate scope, and mild reaction conditions, this is an alternative and practical strategy for the synthesis of aryl difluoromethylselenylether.

Introduction

Organofluorine compounds are widely used in academia and industry [1]. Incorporation of fluoroalkyl moieties into organic molecules can alter their physicochemical properties and biological activities significantly [2]. Over the past decade, a combination of fluoroalkyl groups and chalcogens, such as OCF_3 [3], OCF_2H [4], SCF_3 [5], and SCF_2H [6] has attracted significant research attention due to their high lipophilicity and good cell membrane permeability. Recently, great progress has been made on the introduction of trifluoromethylselanyl group ($SeCF_3$) into organic molecules [7]. However, the difluoromethylselenol group (HCF_2Se) has not been explored much [8], although it is a potential lipophilic isostere of OH, SH, and NH. The lack of studies on this group can be attributed to the easily oxidisable nature of selenium-containing molecules and their high toxicity and unpleasant odour [9]. The lack of efficient difluoromethylselenolation reagents could be another bottleneck in the synthesis of HCF_2Se-containing compounds.

Due to our interest in the direct incorporation of fluoroalkylchalcogen groups into organic molecules [10], we previously developed the metal-free, photocatalysed difluoromethylselenolation of arylamines under visible light using shelf-stable Se-(difluoromethyl) 4-methylbenzenesulfonoselenoate ($TsSeCF_2H$), which was first synthesised by our group as an $SeCF_2H$ source [11]. Besides arylamines, aryl boronic acids are readily accessible and widely used in many organic transformations as the key starting material. In 2017, Tlili and Billard reported a copper-catalyzed direct trifluoro- and perfluoroalkylselenolations of boronic acids with perfluoroalkyl tolueneselenosulfonates [12]. Inspired by this work, we report the copper-catalysed direct difluoroselenolation of aryl boronic acids with $TsSeCF_2H$ (Scheme 1).



Scheme 1. Synthesis of aryl difluoromethylselenylether or aryl difluoromethylselenylether with $TsSeCF_2H$ or $TsSeR_F$.

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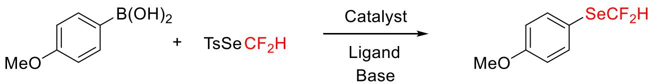
Results and discussion

To begin the investigation, (4-methoxyphenyl)boronic acid (**1a**) was treated with TsSeCF2H (**2**) and caesium carbonate in the presence of copper(II) acetate catalyst and 2,2′-bipyridine (**L1**) in 1,4-dioxane at 25 °C. Fortunately, desired difluoroselenolation product 3a was obtained in 62% yield (Table 1, entry 1). Various solvents such as tetrahydrofuran (THF), diglyme, acetonitrile (MeCN) 1,2-dichloroethane (DCE), and N,*N*-dimethylformamide (DMF) (Table 1, entries 2–6) were screened to improve the yield of this reaction, and the best yield was obtained using MeCN. Following this, various other catalysts such as copper(II) triflate, copper(II) bromide, copper(II) trifluoroacetate, copper(I) iodide, copper(I) iodide, cyanocopper, copper(I) triflate, copper(II) bromide, tetrakis(acetonitrile)copper(I) tetrafluoroborate, and silver triflate and bases including sodium carbonate, potassium carbonate, potassium phosphate, potassium fluoride, potassium bicarbonate, triethylamine, triethylenediamine (DABCO), and 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) (Entries 7–22) were examined. Both copper(I) and copper(II) catalysts with organic and inorganic bases were compatible in this reaction. The combination of copper (II) acetate with potassium carbonate resulted in the best yield. Ligand, catalyst loading, and the effects of reaction temperature and concentration of the starting material were also investigated. Ligands such as 4,4′-dimethyl-2,2′-bipyridine (**L2**), 1,10-phenanthroline (**L3**), *N1*, *N1*,*N2*,*N2*-tetramethylethane-1,2-diamine (**L4**), and 1,3-diphenylpropane-1,3-dione (**L5**) were examined for this reaction, and **L2** was found to give the best yield (Entries 23–26). Increasing the reaction temperature to 40 °C decreased the yield to 78% (Entry 27). Decreasing the catalyst and ligand loadings to 0.05 equiv. or increasing them to 0.15 equiv. also decreased the yield (Entries 28 and 29). When the concentration of **1a** was increased to 0.3 M or decreased to 0.1 M, the yield decreased to 67% and 75%, respectively (Entries 30 and 31). Increasing the loading of **2** to 1.2 equiv. increased the yield to 86% (Entry 32). Moreover, when the concentration of **1a** was increased to 1.2 equiv., the yield increased to 90% (Entry 33). Thus, the optimal reaction conditions for the difluoroselenolation of **1a** were as follows: 1**a**: 0.36 mmol, 2: 0.30 mmol, $Cu(OAc)_2$: 0.03 mmol, 4,4′-dimethyl-2,2′-bipyridine: 0.03 mmol, K_2CO_3: 0.30 mmol, and CH_3CN: 1.5 mL, temperature: 25 °C. Compared with Tlili and Billard's protocol about the copper-catalyzed direct trifluoro- and perfluoroalkylselenolations of boronic acids with perfluoroalkyl tolueneselenosulfonates, a slightly weaker base K_2CO_3 was used.

With the optimsed reaction conditions in hand, the generality of this reaction was examined using a series of aryl boronic acids (**2b**–**2z**). The substrate scope is summarised in Scheme 2. 2-Methoxyl, 3-methoxyl, di-methoxyl, and tri-methoxyl substituted phenylboronic acids (**1b**–**1f**) could be transformed to the corresponding difluoroselenolation products (**3b**–**3f**) in good yields. Moreover, phenylboronic acids bearing electron donating or electron withdrawing substituents in the *para*-, *meta*- or *ortho*-positions were well-tolerated in this reaction, giving the desired products (**3g**–**3q**) in moderate to excellent yields. To further

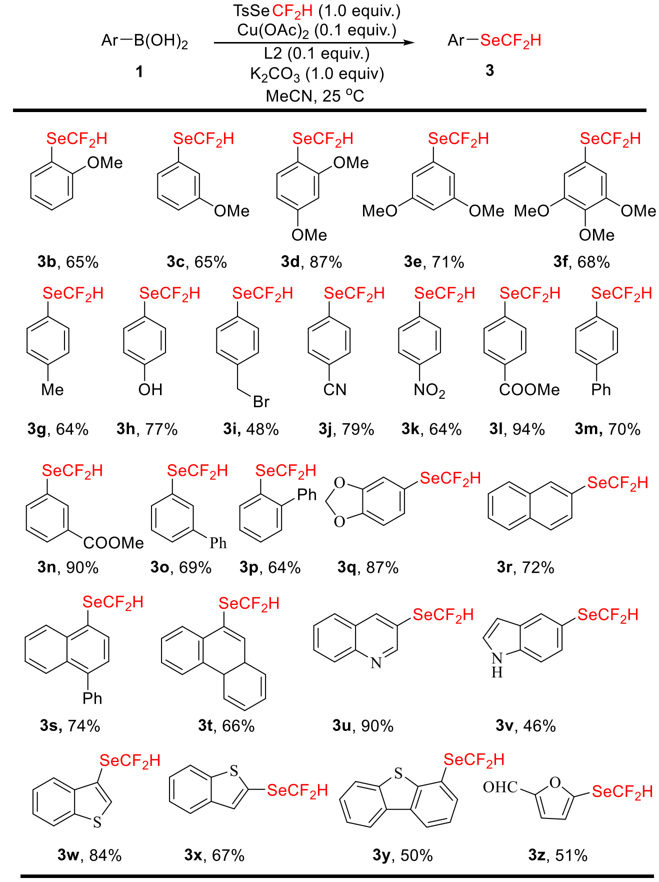
Table 1
Optimisation of difluoroselenolation of (4-methoxyphenyl)boronic.[a]



1a **2** **3a**

Entry	Catalyst/eq.	Ligand/eq.	Base	Solvent	Temperature (°C)	Yield (%)[b]
1	$Cu(OAc)_2$/0.1	L1/0.1	Cs_2CO_3	1,4-dioxane	25	62
2	$Cu(OAc)_2$/0.1	L1/0.1	Cs_2CO_3	THF	25	64
3	$Cu(OAc)_2$/0.1	L1/0.1	Cs_2CO_3	Diglyme	25	20
4	$Cu(OAc)_2$/0.1	L1/0.1	Cs_2CO_3	MeCN	25	67
5	$Cu(OAc)_2$/0.1	L1/0.1	Cs_2CO_3	DCE	25	63
6	$Cu(OAc)_2$/0.1	L1/0.1	Cs_2CO_3	DMF	25	28
7	$Cu(OTf)_2$/0.1	L1/0.1	Cs_2CO_3	MeCN	25	49
8	$CuBr_2$/0.1	L1/0.1	Cs_2CO_3	MeCN	25	62
9	$Cu(CF_3COO)_2$/0/1	L1/0.1	Cs_2CO_3	MeCN	25	60
10	CuI/0.1	L1/0.1	Cs_2CO_3	MeCN	25	62
11	CuCN/0.1	L1/0.1	Cs_2CO_3	MeCN	25	50
12	CuOTf/0.1	L1/0.1	Cs_2CO_3	MeCN	25	32
13	$Cu(CH_3CN)_4BF_4$/0.1	L1/0.1	Cs_2CO_3	MeCN	25	47
14	AgOAc/0.1	L1/0.1	Cs_2CO_3	MeCN	25	15
15	$Cu(OAc)_2$/0.1	L1/0.1	Na_2CO_3	MeCN	25	66
16	$Cu(OAc)_2$/0.1	L1/0.1	K_2CO_3	MeCN	25	73
17	$Cu(OAc)_2$/0.1	L1/0.1	K_3PO_4	MeCN	25	57
18	$Cu(OAc)_2$/0.1	L1/0.1	KF	MeCN	25	22
19	$Cu(OAc)_2$/0.1	L1/0.1	$KHCO_3$	MeCN	25	trace
20	$Cu(OAc)_2$/0.1	L1/0.1	Et_3N	MeCN	25	62
21	$Cu(OTf)_2$/0.1	L1/0.1	DABCO	MeCN	25	62
22	$Cu(OAc)_2$/0.1	L1/0.1	DBU	MeCN	25	trace
23	$Cu(OAc)_2$/0.1	L2/0.1	K_2CO_3	MeCN	25	80
24	$Cu(OAc)_2$/0.1	L3/0.1	K_2CO_3	MeCN	25	53
25	$Cu(OAc)_2$/0.1	L4/0.1	K_2CO_3	MeCN	25	0
26	$Cu(OAc)_2$/0.1	L5/0.1	K_2CO_3	MeCN	25	32
27	$Cu(OAc)_2$/0.1	L2/0.1	K_2CO_3	MeCN	40	78
28	$Cu(OTf)_2$/0.05	L2/0.05	K_2CO_3	MeCN	25	73
29	$Cu(OAc)_2$/0.15	L2/0.15	K_2CO_3	MeCN	25	77
30	$Cu(OAc)_2$/0.1	L2/0.1	K_2CO_3	MeCN	25	67[c]
31	$Cu(OAc)_2$/0.1	L2/0.1	K_2CO_3	MeCN	25	75[d]
32	$Cu(OAc)_2$/0.1	L2/0.1	K_2CO_3	MeCN	25	86[e]
33	$Cu(OAc)_2$/0.1	L2/0.1	K_2CO_3	MeCN	25	90[f]

[a] **1a** (0.30 mmol), **2** (0.30 mmol), catalyst (0.015–0.045 mmol), ligand (0.015–0.045 mmol) and base (0.30 mmol) in solvent (1.5 mL) for indicated reaction temperature. [b] Isolated yields. [c] CH_3CN (1.0 mL) was used. [d] CH_3CN (3.0 mL) was used. [e] **2** (0.36 mmol) was used. [f] **1a** (0.36 mmol) was used.

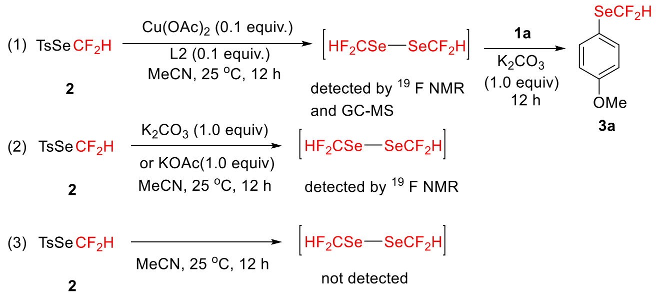


1 (0.36 mmol),TsSeCF$_2$H (0.30 mmol), Cu(OAc)$_2$ (0.03 mmol), 4,4'-dimethyl-2,2'-bipyridine (L2) (0.03 mmol) and K$_2$CO$_3$ (0.30 mmol) in CH$_3$CN (1.5 mL) at 25 °C

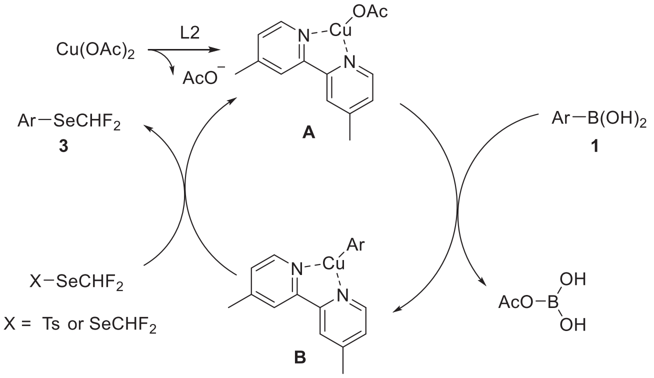
Scheme 2. Scope of difluoroselenolation of aryl boronic acids.

investigate the substrate scope of this reaction, a range of other aryl boronic acids (**1r–1z**) were examined. To our delight, these substrates were smoothly converted to the desired products (**3r–3z**) in moderate to good yields.

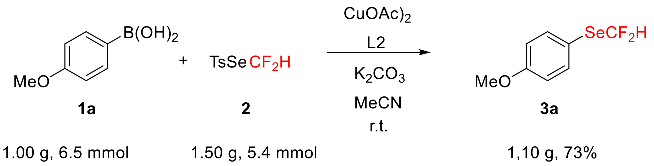
To investigate the reaction mechanism, the difluoroselenolation of **1a** by **2** under the optimised reaction conditions was closely monitored by ^{19}F NMR using (trifluoromethyl)benzene as the internal standard (δ = – 63.20 ppm). After 15 min of initiation of the reaction, three fluorine peaks were observed, which could be assigned to **2** (δ = – 91.06 ppm), product **3a** (δ = -90.47 ppm), and an unknown species (δ = – 88.59 ppm). After 30 min, the fluorine peak of compound **2** disappeared, and the fluorine peak intensities of **3a** and the unknown species increased. After 1 h, the fluorine peak intensity of compound **3a** increased continuously, while that of the unknown species decreased. Notably, a molecular ion peak corresponding to 1,2-bis(difluoromethyl)diselane could be identified from the reaction mixture by GC-MS. Moreover, in the absence of boronic acid and base, only the peak corresponding to the unknown species was detected after 12 h under the optimised reaction conditions. When **1a** and K$_2$CO$_3$ were added to the above mixture, compound **3a** was obtained after 12 h. Treatment of compound **2** with K$_2$CO$_3$ or KOAc in MeCN resulted in its degradation to the unknown species. When **2** was dissolved in MeCN and stirred for 12 h at room temperature, the fluorine peak of unknown species was not detected (Scheme 3) (See supporting information for detail.). These observations suggested that the unknown species was 1,2-bis(difluoromethyl)diselane, and it could be a key intermediate in this transformation. However, we cannot eliminate the possibility of **2** being the difluoroselenolation reagent.



Scheme 3. Additional reactions for investigating the mechanism.



Scheme 4. Possible reaction mechanism.



Scheme 5. Scale up of the difluoromethylselenolation reaction.

Based on literature [12] and the abovementioned results, a plausible reaction mechanism for this transformation was proposed (Scheme 4). First, 16-electron copper complex **A** was formed *in situ*, which was the active catalytic species. This was followed by transmetallation between complex **A** and the aryl boronic acid to form intermediate **B**. Finally, **B** reacted with trifluoromethylselenolated reagent **2** or 1,2-bis(difluoromethyl)diselane to give desired product **3**.

Finally, to illustrate the possible practical applications of this transformation, a gram-scale difluoromethylselenolation reaction of **1a** was conducted (Scheme 5). To our delight, the desired product **3a** was obtained in 73% yield.

Conclusion

In summary, we developed the first copper-catalysed direct difluoromethylselenolation of aryl boronic acids using TsSeCF$_2$H as a difluoromethylselenolation reagent. The cheap and readily accessible reagents, broad substrate scope, and mild reaction conditions render this reaction an alternative and practical strategy for the synthesis of aryl difluoromethylselenylether. Investigation of other difluoromethylselenolation reactions using TsSeCF$_2$H are underway in our lab.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.tetlet.2021.152897.

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