

## Accepted Manuscript

Copper(I)-Promoted Trifluoromethylthiolation of Arenediazonium salts with  $\text{AgSCF}_3$

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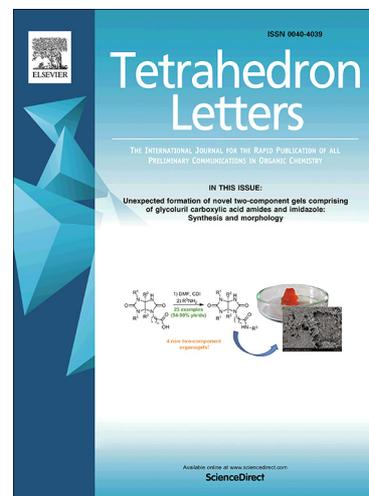
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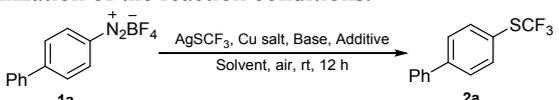
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storage for extended periods. Recently, Y. Yang and coworker have developed a low-cost method for the preparation of  $\text{CuSCF}_3$  via triphenylphosphine-mediated deoxygenative reduction of Langlois' reagent ( $\text{CF}_3\text{SO}_2\text{Na}$ ) in the presence of  $\text{CuCl}$ .<sup>7h</sup> However, the preparation procedure is somewhat complicated which must be carried out in high temperature under nitrogen atmosphere. Moreover, excessive amount of triphenylphosphine is also employed in this procedure, generating a large amount of environmentally harmful  $\text{Ph}_3\text{P}=\text{O}$  as by-product. As the relatively inexpensive, stable and easy-to-handle reagent,  $\text{AgSCF}_3$  could form a reactive source of  $^-\text{SCF}_3$  in the presence of  $n\text{-Bu}_4\text{NI}$  or  $\text{KI}$ .<sup>18</sup> Intrigued by the Sandmeyer trifluoromethylation of aromatic amines and trifluoromethylthiolation with  $\text{AgSCF}_3$ ,<sup>19, 20</sup> we have been trying to develop effective approaches for metal-mediated trifluoromethylthiolation of arenediazonium salts with  $\text{AgSCF}_3$ .

**Table 1**Optimization of the reaction conditions.<sup>a</sup>


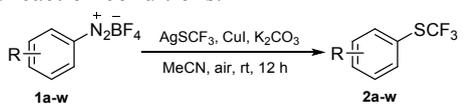
Entry	Cu salt	[Cu] (mmol)	Base	Additive	Solvent	2a Yield (%) <sup>b</sup>
1	CuCl	0.01	$\text{K}_2\text{CO}_3$	$n\text{-Bu}_4\text{NI}$	$\text{CH}_3\text{CN}$	13
2	CuBr	0.01	$\text{K}_2\text{CO}_3$	$n\text{-Bu}_4\text{NI}$	$\text{CH}_3\text{CN}$	10
3	CuI	0.01	$\text{K}_2\text{CO}_3$	$n\text{-Bu}_4\text{NI}$	$\text{CH}_3\text{CN}$	25
4	CuSCN	0.01	$\text{K}_2\text{CO}_3$	$n\text{-Bu}_4\text{NI}$	$\text{CH}_3\text{CN}$	21
5	$\text{CuBr}_2$	0.01	$\text{K}_2\text{CO}_3$	$n\text{-Bu}_4\text{NI}$	$\text{CH}_3\text{CN}$	14
6	$\text{Cu}(\text{OAc})_2$	0.01	$\text{K}_2\text{CO}_3$	$n\text{-Bu}_4\text{NI}$	$\text{CH}_3\text{CN}$	20
7	$\text{Cu}(\text{OTf})_2$	0.01	$\text{K}_2\text{CO}_3$	$n\text{-Bu}_4\text{NI}$	$\text{CH}_3\text{CN}$	19
8	$\text{Cu}(\text{MeCN})_4\text{BF}_4$	0.01	$\text{K}_2\text{CO}_3$	$n\text{-Bu}_4\text{NI}$	$\text{CH}_3\text{CN}$	23
9	CuI	0.02	$\text{K}_2\text{CO}_3$	$n\text{-Bu}_4\text{NI}$	$\text{CH}_3\text{CN}$	32
10	CuI	0.05	$\text{K}_2\text{CO}_3$	$n\text{-Bu}_4\text{NI}$	$\text{CH}_3\text{CN}$	53
11	CuI	0.1	$\text{K}_2\text{CO}_3$	$n\text{-Bu}_4\text{NI}$	$\text{CH}_3\text{CN}$	80
12	CuI	0.1	$\text{K}_2\text{CO}_3$	-	$\text{CH}_3\text{CN}$	82
13	CuI	0.1	$\text{Na}_2\text{CO}_3$	-	$\text{CH}_3\text{CN}$	46
14	CuI	0.1	$\text{NaHCO}_3$	-	$\text{CH}_3\text{CN}$	49
15	CuI	0.1	$\text{Cs}_2\text{CO}_3$	-	$\text{CH}_3\text{CN}$	52
16	CuI	0.1	$\text{K}_2\text{CO}_3$	-	DMSO	59
17	CuI	0.1	$\text{K}_2\text{CO}_3$	-	DMF	63
18	CuI	0.1	$\text{K}_2\text{CO}_3$	-	NMP	50
19 <sup>c</sup>	CuI	0.1	$\text{K}_2\text{CO}_3$	-	$\text{CH}_3\text{CN}$	78

<sup>a</sup> Reaction conditions: **1a** (0.1 mmol),  $\text{AgSCF}_3$  (0.15 mmol, 1.5 equiv.), copper salt, base (0.2 mmol, 2.0 equiv.), additive (0.2 mmol, 2.0 equiv.), solvent (1 mL). <sup>b</sup> Yields were determined by  $^{19}\text{F}$  NMR spectroscopy based on **1a** using 4,4'-difluorobiphenyl as an internal standard. <sup>c</sup> under  $\text{N}_2$ .

Initially, the model substrate **1a** of trifluoromethylthiolation reaction was screened to investigate the effects of different copper salts, bases, solvents, and additives (Table 1). There is only 13% yield of trifluoromethylthiolated product to be obtained, using 0.01 mmol  $\text{CuCl}$  as the catalyst. Investigating copper catalyst such as  $\text{CuCl}$ ,  $\text{CuBr}$ ,  $\text{CuI}$ ,  $\text{CuSCN}$ ,  $\text{CuBr}_2$ ,  $\text{Cu}(\text{OAc})_2$ ,  $\text{Cu}(\text{OTf})_2$  and  $\text{Cu}(\text{MeCN})_4\text{BF}_4$  (entries 1–8),  $\text{CuI}$  was suggested to be best choice for the reaction with the highest yield. With increase of the amount of  $\text{CuCl}$ , the improved significantly yield of **2a** show that catalytic amounts of copper salt were insufficient to achieve high yields. Surprisingly, a slightly higher yield of 82% was achieved in the absence of  $n\text{-Bu}_4\text{NI}$  (entry 12). Several bases such as  $\text{Na}_2\text{CO}_3$ ,  $\text{NaHCO}_3$ ,  $\text{Cs}_2\text{CO}_3$  and  $\text{K}_2\text{CO}_3$  were also examined to further optimize the reaction conditions. Obviously,  $\text{K}_2\text{CO}_3$  is most suitable for achievement of corresponding product **2a** in the highest yield (entries 12–15). The influence of solvents was also investigated in such solvents as DMSO, DMF and NMP (entries 16–18)

besides  $\text{CH}_3\text{CN}$ . Furthermore, the reaction was carried out under nitrogen to gain the expected product. A slightly higher yield in air shows that the reaction proceeds can be carried through in an easy-to-handle approach. (entry 19).

Based on the optimized reaction conditions (Table 1, Entry 12), the substrate scope of oxidative trifluoromethylthiolation for arenediazonium salts was investigated as summarized in Table 2. With the electron-donating substituent in aromatic ring as aryl, alkyl, alkoxy and methylthio, the arenediazonium salt derivatives reacted smoothly to give the corresponding trifluoromethylthiolated products (**2a–2i**) in reasonable yields of 58–72%. The 2-naphthyl diazonium salt was also suitable for the reaction system to give product **2j** in 60% yield. Examined the substrate with electron-withdrawing substituent in aromatic ring, such as halogen (**1k** and **1l**), acetyl (**1m** and **1n**), carbomethoxy (**1o** and **1p**) and cyano (**1q** and **1r**), the arenediazonium salt derivatives can tolerate this reaction system more efficiently with slightly higher yields (**2k–2u**, 73–92%, Table 2) than the electron-donating substituent. Comparing with aromatic substrates with in nitro substituent in ortho (**2t**, 89%) and meta- (**2u**, 90%) position, the pretty higher yield of the electron-withdrawing nitro in para (**2s**, 92%) position indicated that electronic effect rather than steric effects remains to be the most important influence to the reaction system. Contrary to other  $\text{SCF}_3$  or  $\text{CF}_3$  sources, the trifluoromethylthiolated yield of electron-deficient substrates is overall higher than the electron-rich substrates using  $\text{AgSCF}_3$ . In addition, some arenediazonium salt derivatives, bearing pyridine moiety (**1v**) and electron-withdrawing substituent, can be converted to the corresponding trifluoromethylthiolated products in higher yields to the reported.<sup>11–16</sup>

**Table 2.**Trifluoromethylthiolation of Arenediazonium salts under the optimized reaction conditions.<sup>a, b</sup>


Entry	Yield (%)
<b>2a</b>	70%
<b>2b</b>	65%
<b>2c</b>	60%
<b>2d</b>	63%
<b>2e</b>	58%
<b>2f</b>	67%
<b>2g</b>	63%
<b>2h</b>	72%
<b>2i</b>	68%
<b>2j</b>	60%
<b>2k</b>	78%
<b>2l</b>	86%
<b>2m</b>	81%
<b>2n</b>	73%
<b>2o</b>	76%
<b>2p</b>	79%
<b>2q</b>	89%
<b>2r</b>	83%
<b>2s</b>	92%
<b>2t</b>	89%
<b>2u</b>	90%
<b>2v</b>	76%
<b>2w</b>	55%

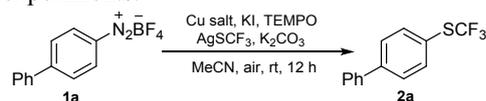
<sup>a</sup> Reaction conditions: **1a–1w** (0.5 mmol),  $\text{AgSCF}_3$  (0.75 mmol, 1.5 equiv.),  $\text{CuI}$  (0.5 mmol, 1.0 equiv.),  $\text{K}_2\text{CO}_3$  (1.0 mmol, 2.0 equiv.),  $\text{CH}_3\text{CN}$  (5 mL), air, rt, 12 h. <sup>b</sup> Isolated yields.

Secondarily, this reaction system is feasible particularly for large-scale applications due to its irreplaceable advantages as easily available raw material, air insensitivity and mild reaction conditions. To illustrate the practical application of this transformation, the reaction was scaled up using 5 mmol of the substrate **1s**. The corresponding trifluoromethylthiolated product

**2s** was also obtained in isolated yield of 85% through this reaction proceeds under the optimal conditions.

To elucidate a plausible reaction mechanism, some control experiments were carried as shown in Table 3. Without copper salt in the reaction system, only 10% desired product **2a** was detected. Obviously, the reactivity of metallic silver is low without copper salt. Using 1.0 equivalents of  $\text{Cu}(\text{MeCN})_4\text{BF}_4$  as catalyst instead of  $\text{CuI}$ , the yield of **2a** was apparently declined to 40%. Added 1.0 equivalents of  $\text{KI}$  to the above reaction system, the improved yield 78% of **2a** indicated that iodine anion ( $\text{I}^-$ ) play a significant role in this reaction system. As the previous reported,<sup>21</sup> the active species  $[\text{Ag}(\text{SCF}_3)\text{I}]^-$  can generate through this reaction system with the stronger nucleophilicity than  $\text{AgSCF}_3$ . The result of the above investigation show further that  $\text{CuSCF}_3$  species will generate on site in the presence of  $\text{AgSCF}_3$  or  $[\text{Ag}(\text{SCF}_3)\text{I}]^-$  with copper salt.<sup>22</sup> Added 4.0 equivalents of the radical-trapping reagent TEMPO (2,2,6,6-tetramethyl-1-oxy-piperidine), the reaction was obviously suppressed to give only 8% products under the optimal conditions. It indicates that the reaction may proceed *via* a radical pathway. The investigations of reaction temperature suggest that the yield of product **2a** decreases significantly by increase of the reaction temperature, along with the augment of the iodobenzene as main by-product (See the Supporting Information).

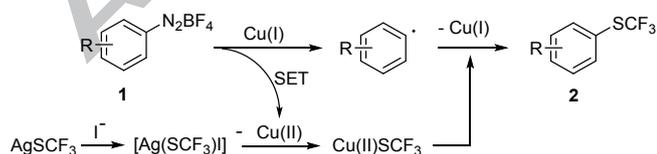
**Table 3**  
Control experiments.



Entry	Cu salt	KI	TEMPO	<b>2a</b> Yield (%) <sup>c</sup>
1 <sup>a</sup>	-	-	-	10%
2 <sup>a</sup>	$\text{Cu}(\text{MeCN})_4\text{BF}_4$ (1.0)	-	-	40%
3 <sup>a</sup>	$\text{Cu}(\text{MeCN})_4\text{BF}_4$ (1.0)	1.0	-	78%
4 <sup>b</sup>	$\text{CuI}$ (1.0)	-	2.0	21%
5 <sup>b</sup>	$\text{CuI}$ (1.0)	-	4.0	8%

Reaction conditions: **1a** (<sup>a</sup> 0.1 mmol, 1 equiv.; <sup>b</sup> 0.5 mmol, 1 equiv.),  $\text{AgSCF}_3$  (1.5 equiv.),  $\text{K}_2\text{CO}_3$  (2.0 equiv.),  $\text{CH}_3\text{CN}$  (5 mL), air, rt, 12 h, Cu salt, KI, and TEMPO (equiv.). <sup>c</sup>Yield was determined by <sup>19</sup>F NMR spectroscopy using 4,4'-difluorobiphenyl as an internal standard.

Based on the above experimental results, a plausible mechanism was proposed about the copper(I)-promoted trifluoromethylthiolation process *via*  $[\text{Ag}(\text{SCF}_3)\text{I}]^-$  as shown in Scheme 2. Firstly transferred a single electron (SET) to arenediazonium salts **1**,  $\text{Cu(I)}$  afford aryl radicals and  $\text{Cu(II)}$  species. Then,  $\text{Cu(II)SCF}_3$  species will generate on-site quickly through combination of  $\text{Cu(II)}$  and active species  $[\text{Ag}(\text{SCF}_3)\text{I}]^-$  from the reaction of  $\text{AgSCF}_3$  and iodine anion. The desired product **2a** can achieve by aryl radicals with  $\text{Cu(II)SCF}_3$ .



**Scheme 2.** Plausible mechanism.

In summary, we have developed an efficient preparation method through copper(I)-promoted trifluoromethylthiolation for the various arenediazonium salts, using a stable and inexpensive  $\text{AgSCF}_3$  in moderate to good yields and mild reaction conditions. A preliminary mechanistic investigation suggests that this reaction process *via* a radical pathway. With the extensive reaction tolerance, mild reaction condition and the easy-to-get raw material, this method achieve a useful and practical strategy in the synthesis of aryl trifluoromethyl sulfides, for great

potential application of synthetic, medicinal, and agrochemical research.

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### Supplementary Material

Supplementary material for this article is available online at <http://dx.doi.org/10.1016/j.tetlet>.

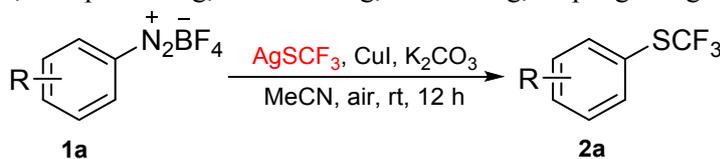
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An efficient method with a stable and convenient  $\text{AgSCF}_3$  provides an additional way for the synthesis of trifluoromethylthiolated compound with arenediazonium salts, in very mild conditions and good yield.

## Graphical Abstract

### Copper(I)-Promoted Trifluoromethylthiolation of Arenediazonium salts with $\text{AgSCF}_3$

Changge Zheng, Yang Liu, Jianquan Hong, Shuai Huang, Wei Zhang, Yupeng Yang and Ge Fang



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The mild reaction condition.

## Highlights

The first example of the stable  $\text{AgSCF}_3$  for trifluoromethylthiolation of arenediazonium salts.

An efficient method for trifluoromethylthiolation of arenediazonium salts was developed.

The trifluoromethylthiolated products with electron-withdrawing substituent were obtained in higher yields.