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Copper-catalyzed trifluoromethylation of arylsulfinate salts using an electrophilic trifluoromethylation reagent

Xiaoxi Lin [a,b], Guimei Wang [a], Huaifeng Li [c], Yuanyuan Huang [a], Weiming He [a], Dandan Ye [a], Kuo-Wei Huang [c,*], Yaofeng Yuan [a], Zhiqiang Weng [a,b,*]

[a] Department of Chemistry, Fuzhou University, Fujian 350108, PR China
[b] State Key Laboratory of Structural Chemistry, Fujian Institute of Research on the Structure of Matter, Chinese Academy of Sciences, Fuzhou, Fujian 350002, PR China
[c] King Abdullah University of Science and Technology (KAUST), Division of Physical Sciences and Engineering and KAUST Catalysis Center, Thuwal 23955-6900, Saudi Arabia

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ABSTRACT

A copper-catalyzed method for the trifluoromethylation of arylsulfinates with Togni's reagent has been developed, affording aryltrifluoromethylsulfones in moderate to good yields. A wide range of functional groups in arylsulfinates are compatible with the reaction conditions.

1. Introduction

Fluorinated aromatic molecules have important applications in the pharmaceutical, agricultural, and advanced materials industries.[1] In medicinal chemistry, the incorporations of fluorine containing substituents, such as F, CF_3, SCF_3, OCF_3, and SO_2CF_3 to a target molecule could dramatically improve their binding selectivity, metabolic stability, and lipophilicity.

Aryltrifluoromethylsulfones are frequently used as important structural motifs in bioactive compounds,[2] chiral catalysts,[3] and functional materials.[4] They can also serve as important precursors to many other chemicals, such as various trifluoromethylated compounds[5] or aryl sulfones.[6] However, relatively few synthetic approaches for the preparation of aryltrifluoromethylsulfones are currently available. The commonly known methods for the synthesis of aryltrifluoromethylsulfones are the oxidation of corresponding aryl trifluoromethylsulphides.[7] Notably, Yagupolski and co-workers have reported a one-pot procedure for the fluoride-catalyzed cross-coupling of arenesulfonyl fluorides with (trifluoromethyl) trimethylsilane and (trifluoromethyl)-trimethylstannane.[8] Despite these procedures being attractive for their simplicity, some limitations still remain, e.g., they are incompatible with some functional groups; the sulfides are not easily available;[8] and the yields and selectivity are poor.

A flourish of activity in the development of metal-mediated and catalyzed trifluoromethylation reactions has been recently witnessed in the literature,[9,10] using either nucleophilic or electrophilic trifluoromethylating reagents, such as the Ruppert–Prakash's reagent ($TMSCF_3$)[11] or its ethyl derivative,[12] fluoroform,[13] potassium (trifluoromethyl)trimethoxyborate,[14] Togni's reagent,[11a,15] Umemoto's reagent,[16] and the (trifluoromethyl)diphenylsulfonium salt as CF_3 sources.[17]

Our laboratories have initiated research programs aimed at the development of copper-catalyzed trifluoromethylation reactions.[11a,18] As part of the efforts of these programs, we sought to develop an effective route for the synthesis of aryltrifluoromethylsulfones through copper-catalyzed trifluoromethylation. Due to their stable nature, readily availability, and easiness to handle, sulfinic acid sodium salts have the potential to serve as an ideal arenesulfone source for the preparation of aryltrifluoromethylsulfones via trifluoromethylation. Herein, we report a copper-catalyzed cross-coupling reaction of arylsulfinate salts with Togni's reagent.

* Corresponding authors. Tel.: +966 28080328 (K.W.H.); tel./fax: +86 59122866121 (Z.W.); e-mail addresses: hkw@kaust.edu.sa (K.-W. Huang), zweng@fzu.edu.cn (Z. Weng).

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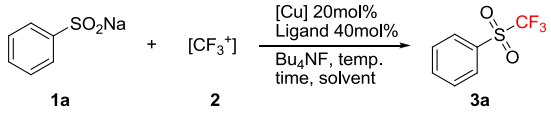
2. Results and discussion

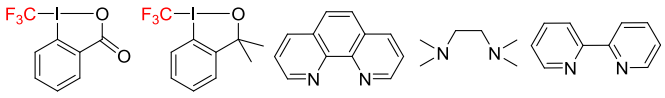
We chose to examine our strategy by screening the trifluoromethylation reactions of benzenesulfinate sodium salts (Table 1). Various copper precursors, bidentate ligands, solvents, reaction temperatures, and time were evaluated for the reaction of **1a** with Togni's reagent (1-trifluoromethyl-1,2-benziodoxol-3-(1*H*)-one (**2a**) and 1-trifluoromethyl-1,3-dihydro-3,3-dimethyl-1,2-benziodoxole (**2b**)).[1g,19] After considerable effort, we identified that formation of the (trifluoromethylsulfonyl)benzene product **3a** could be achieved in good yield (78%) when the reaction was conducted with a combination of $Cu(TFA)_2$ (TFA=trifluoroacetate), phenanthroline (**L1**), and tetrabutylammonium fluoride (Table 1, entry 1).

Table 1
Optimization of copper-catalyzed trifluoromethylation of sulfinate salts[a]



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

Entry	[Cu]	Ligand	CF_3^+ reagent	Solvent	Time (h)	Bu_4NF (equiv)	Temp (°C)	Yield[b] (%)
1	$Cu(TFA)_2$	**L1**	**2a**	DMSO	8.5	0.5	130	78
2	$Cu(TFA)_2$	**L1**	**2b**	DMSO	8.5	0.5	130	36
3	$Cu(TFA)_2$	**L1**	**2a**	DMSO	8.5	None	130	37
4	$Cu(TFA)_2$	**L1**	**2a**	DMSO	8.5	0.5	90	19
5	CuI	**L1**	**2a**	DMSO	8.5	0.5	130	32
6	$Cu(OTf)_2$	**L1**	**2a**	DMSO	8.5	0.5	130	45
7	$Cu(MeCN)_4PF_6$	**L1**	**2a**	DMSO	8.5	0.5	130	45
8	None	None	**2a**	DMSO	8.5	0.5	130	19
9	$Cu(TFA)_2$	**L2**	**2a**	DMSO	8.5	0.5	130	40
10	$Cu(TFA)_2$	**L3**	**2a**	DMSO	8.5	0.5	130	43
11	$Cu(TFA)_2$	**L1**	**2a**	DMSO	15	0.5	130	61
12	$Cu(TFA)_2$	**L1**	**2a**	THF	8.5	0.5	60	7
13	$Cu(TFA)_2$	**L1**	**2a**	CH_2Cl_2	8.5	0.5	40	6
14	$Cu(TFA)_2$	**L1**	**2a**	CH_3CN	8.5	0.5	80	3



2a **2b** **L1** **L2** **L3**

[a] Reaction conditions: **1a** (0.10 mmol), **2** (0.25 mmol), solvent (2.0 mL), under Ar atmosphere.
[b] Yields were determined by GC analysis of the crude reaction mixture with an internal standard.

Variation of the trifluoromethylation reagents showed that **2a** is superior to **2b** (Table 1, entry 2). Importantly, in the absence of either of the copper species/ligands, or Bu_4NF, the reaction efficiency was significantly decreased (Table 1, entries 3 and 8) as did lowering the reaction temperature (Table 1, entry 4). A subsequent screening of copper sources, i.e., CuI, $Cu(OTf)_2$, and $Cu(MeCN)_4PF_6$ with phen (**L1**) as the ligand, resulted in lower yields (Table 1, entries 5–7). The nature of the ligand also influences the yields of the trifluoromethylation products (Table 1, entries 9 and 10). A longer reaction time led to a significantly lower yield, presumably due to decomposition of the product (Table 1, entry 11). The choice of solvents proved crucial for the formation of **3a**. The use of THF, CH_2Cl_2, and CH_3CN at reflux retarded the formation of the product (Table 1, entries 12–14).

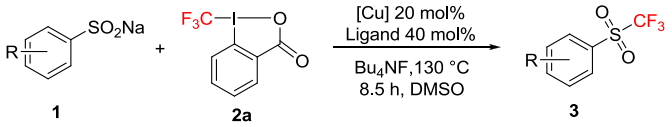
To explore the substrate scope, we examined a range of sulfinate sodium salts in the trifluoromethylation with **2a** under the optimized reaction conditions (Table 2). Reactions with **1a** as well as methyl- and *tert*-butyl-substituted phenyl sulfinate sodium salts proceeded well to give good yields of the desired products in 70–76% yield (Table 2, entries 1–3 and 5), although in the case of *p*-methyl phenyl sulfinate, $Cu(OTf)_2$ was applied instead of $Cu(TFA)_2$. The 4-*i*-propylphenyl and biphenyl sulfinate sodium give slightly lower yields (Table 2, entries 4 and 6), while both of the 1- and 2-naphthyl sulfinate sodium salts afforded the trifluoromethylate products in good yields (Table 2, entries 7 and 8). Notably, a wide range of functional groups, including methoxy, cyano, nitro, fluoro, and heteroaryl groups, are well tolerated under the present reaction conditions (Table 2, entries 9–13). Substrates bearing electron-donating groups, such as 4-methoxybenzenesulfinate, could also be employed to provide the corresponding product in acceptable yields (Table 2, entry 9). To achieve an effective trifluoromethylation of *m*-nitro benzenesulfinate, we optimized the reaction conditions again. Replacement of $Cu(OTf)_2$/**L3** with $Cu(TFA)_2$/**L1** led to a good yield of product (Table 2, entry 11). It should be mentioned that pharmaceutically interesting functional groups, such as heteroaryl groups were also completely compatible. Thereby, reaction of 8-quinoline sulfinate catalyzed by $CuPF_6$ $(CH_3CN)_4$/**L3** rather than $Cu(TFA)_2$/**L1**, afforded the trifluoromethylate product in good yields (Table 2, entry 13).

A plausible catalytic cycle for the present trifluoromethylation reaction is depicted in Scheme 1. First a bipyridine ligated (*N*,*N*) $Cu(TFA)_2$ complex (**A**), generated in situ, undergoes the transmetallation reaction with sodium arylsulfinate to give a copper(II) intermediate **B**. Subsequently, the nucleophilic attack of the $ArSO_2$ group of **B** to the CF_3^+moiety in the Togni's reagent affords the trifluoromethylation product $ArSO_2CF_3$ along with the copper–benzoate complex (**C**). Finally, **C** reacts with sodium arylsulfinate to regenerate **B** to complete the catalytic cycle.

3. Conclusions

In summary, we have developed an efficient copper-catalyzed protocol for the trifluoromethylation of arylsulfinates to synthesize aryltrifluoromethylsulfones. A wide variety of functional groups in arylsulfinates are compatible with the reaction conditions and the aryltrifluoromethylsulfone products have been obtained in good yields. This reaction expands the scope of Cu-catalyzed electrophilic trifluoromethylation reactions and should lead to future synthetic applications.

Table 2
Copper-catalyzed trifluoromethylation of sulfinate salts with Togni's reagent[a]



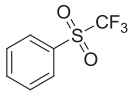
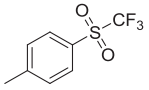
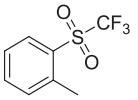
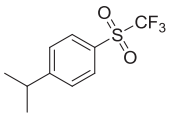
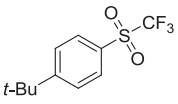
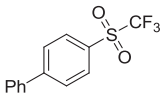
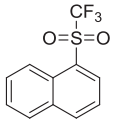
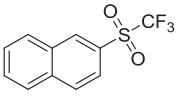
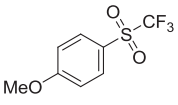
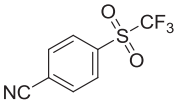
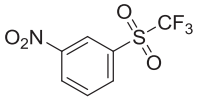
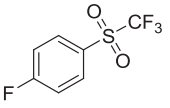
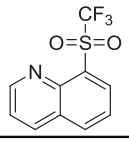
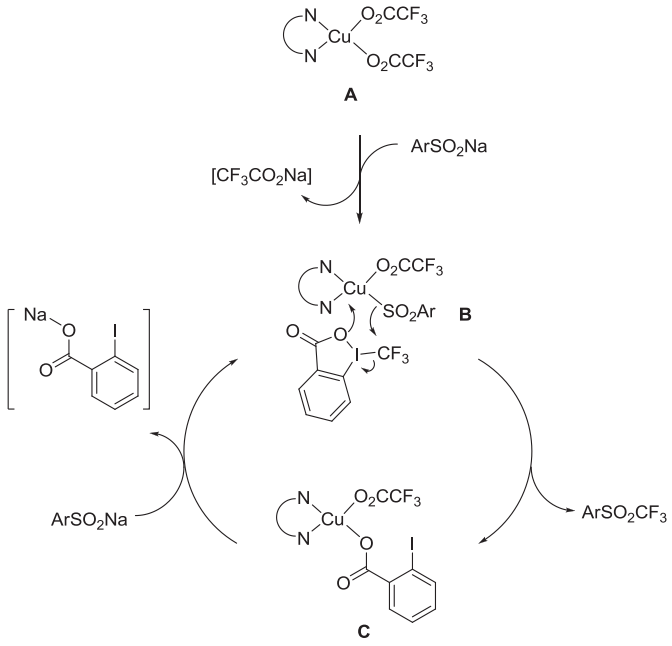
Entry	[Cu]	Ligand	Product	Yield (%)[b]
1	$Cu(TFA)_2$	**L1**		71
2	$Cu(OTF)_2$	**L1**		70
3	$Cu(TFA)_2$	**L1**		76
4	$Cu(TFA)_2$	**L1**		51
5	$Cu(TFA)_2$	**L1**		71
6	$Cu(TFA)_2$	**L1**		60
7	$Cu(TFA)_2$	**L1**		77
8	$Cu(TFA)_2$	**L1**		76
9	$Cu(TFA)_2$	**L1**		41
10	$Cu(TFA)_2$	**L1**		88
11	$Cu(OTF)_2$	**L3**		75
12	$Cu(TFA)_2$	**L1**		38

Table 2 (*continued*)

Entry	[Cu]	Ligand	Product	Yield (%)[b]
13	$CuPF_6(CH_3CN)_4$	**L1**		81[c]

[a] Reaction conditions: sulfinate salts (0.50 mmol), **2a** (1.25 mmol), [Cu] (0.10 mmol), phen (0.20 mmol), Bu_4NF (0.25 mmol), DMSO (10 mL), 130 °C, under Ar atmosphere.
[b] Isolation yield.
[c] CsF (1.0 mmol) was added.



Scheme 1. Plausible mechanism.

4. Experimental

4.1. General experimental

All solvents were purified by standard methods. ^1H, ^{19}F, and ^{13}C NMR spectra were recorded using BrukerA VIII 400 or AVIII 500 spectrometer. ^1H and ^{13}C NMR chemical shifts were reported in parts per million (ppm) downfield from tetramethylsilane and ^{19}F NMR chemical shifts were determined relative to $CFCl_3$ as external standard. Coupling constants (J) are reported in Hertz (Hz). The residual solvent peak was used as an internal reference: ^1H NMR (chloroform δ 7.26) and ^{13}C NMR (chloroform δ 77.0). The following abbreviations were used for the multiplicities: s: singlet, d: doublet, t: triplet, q: quartet, m: multiplet, and br: broad. Column chromatography purifications were performed by flash chromatography using Merck silica gel 60. The substrates of sodium benzenesulfinate and sodium *p*-toluenesulfinate were purchased from commercial sources and used without further purification. Other sodium sulfinates were prepared according to literature procedures.[20] Solvents were freshly dried and degassed according to the procedures in *Purification of Laboratory Chemicals* prior to use.

4.2. General procedure for the copper-catalyzed trifluoromethylation of arylsulfinate salts

In a glovebox, arylsulfinate salts (0.50 mmol), [Cu] (0.10 mmol), phen (0.20 mmol), Bu_4NF (0.25 mmol), and electrophilic trifluoromethylating reagent (1.25 mmol) were added to an oven-dried resealable Schlenk tube possessing a Teflon screw valve. Freshly distilled DMSO (10.0 mL) was added into this tube and the tube was sealed. The solution was stirred in a preheated (130 °C) oil bath for 8 h. The reaction mixture was then allowed to cool to room temperature and passed through a short silica gel pad to remove metal salts. Water (10.0 mL) was added to the mixture at 0 °C. The resulting mixture was extracted with diethyl ether (6.0 mL×2), and the combined organic layers were washed with water (6.0 mL×2), and then dried over magnesium sulfate. The solvent was removed by rotary evaporation in an ice bath and the resulting product was purified by column chromatography on silica gel with pentane.

4.2.1. Trifluoromethyl phenylsulfone[5d,7d] (Table 2, entry 1). Obtained as a colorless oil in 71% yield (74.6 mg). R_f (5% Et_2O/pentane): 0.33. ^1H NMR (500 MHz, $CDCl_3$): δ 8.08 (d, J=7.8 Hz, 2H), 7.88 (t, J=7.8 Hz, 1H), 7.72 (t, J=7.8 Hz, 2H). ^{19}F NMR (470 MHz, $CDCl_3$): δ −78.42 (s, 3F). ^{13}C NMR (126 MHz, $CDCl_3$): δ 131.8, 126.7, 126.0, 125.1, 115.0 (q, J=325 Hz). GC–MS m/z 210 (M^+). The spectroscopic data are consistent with those reported in the literature.

4.2.2. 1-Methyl-4-(trifluoromethylsulfonyl)benzene (Table 2, entry 2).[7e,21] Obtained as a colorless oil in 70% yield (78.9 mg). R_f (5% Et_2O/pentane): 0.33. ^1H NMR (500 MHz, $CDCl_3$): δ 7.94 (d, J=8.1 Hz, 2H), 7.49 (d, J=8.1 Hz, 2H), 2.54 (s, 3H). ^{19}F NMR (470 MHz, $CDCl_3$): δ −78.67(s, 3F). ^{13}C NMR (126 MHz, $CDCl_3$): δ 148.4, 130.8, 130.6, 128.2, 119.9 (q, J=323 Hz), 22.0. GC–MS m/z 224 (M^+). The spectroscopic data are consistent with those reported in the literature.

4.2.3. 1-Methyl-2-(trifluoromethylsulfonyl)benzene (Table 2, entry 3).[21,22] Obtained as a colorless oil in 76% yield (84.8 mg). R_f (5% Et_2O/pentane): 0.33. ^1H NMR (500 MHz, $CDCl_3$): δ 8.10 (d, J=8.0 Hz, 1H), 7.73–7.68 (m, 1H), 7.43–7.53 (m, 2H), 2.76 (s, 3H). ^{19}F NMR (470 MHz, $CDCl_3$): δ −78.08 (s, 3F). ^{13}C NMR (126 MHz, $CDCl_3$): δ 142.2, 136.4, 133.6, 133.4, 129.8, 127.2, 120.1 (q, J=323 Hz), 20.7. GC–MS m/z 224 (M^+). The spectroscopic data are consistent with those reported in the literature.

4.2.4. 1-Isopropyl-4-(trifluoromethylsulfonyl)benzene (Table 2, entry 4). Obtained as a colorless oil in 51% yield (63.6 mg). R_f (5% Et_2O/pentane): 0.36. ^1H NMR (500 MHz, $CDCl_3$): δ 7.98 (d, J=8.4 Hz, 2H), 7.54 (d, J=8.4 Hz, 2H), 3.13–3.02 (m, 1H), 1.33 (d, J=6.9 Hz, 6H). ^{19}F NMR (470 MHz, $CDCl_3$): δ −78.60 (s, 3F). ^{13}C NMR (126 MHz, $CDCl_3$): δ 158.8, 131.0, 128.6, 128.1, 119.8 (q, J=323 Hz), 34.6, 23.5. GC–MS m/z 252 (M^+). HRMS (EI) m/z: calcd for $[M]^+$: $C_{10}H_{11}F_3O_2S$: 252.0432; found: 252.0434. IR (KBr) ν 2966, 2920, 1595, 1369, 1219, 1196, 1142, 1078 cm^{-1}.

4.2.5. 1-tert-Butyl-4-(trifluoromethylsulfonyl)benzene (Table 2, entry 5). Obtained as a white solid in 71% yield (94.5 mg); mp 61–62 °C. R_f (5% Et_2O/pentane): 0.43. ^1H NMR (500 MHz, $CDCl_3$): δ 7.98 (d, J=8.5 Hz, 2H), 7.70 (d, J=8.5 Hz, 2H), 1.40 (s, 9H). ^{19}F NMR (470 MHz, $CDCl_3$): δ −78.57 (s, 3F). ^{13}C NMR (126 MHz, $CDCl_3$): δ 161.1, 130.7, 128.1, 127.0, 119.9 (q, J=323 Hz), 35.7, 30.9. GC–MS m/z 266 (M^+). HRMS (EI) m/z: calcd for $[M]^+$: $C_{11}H_{13}F_3O_2S$: 266.0588; found: 266.0596. IR (KBr) ν 2965, 2924, 1593, 1362, 1218, 1144, 637, 591 cm^{-1}.

4.2.6. 4-(Trifluoromethylsulfonyl)biphenyl (Table 2, entry 6). Obtained as a white solid in 60% yield (86.1 mg); mp 71–73 °C. R_f (5% Et_2O/pentane): 0.43. ^1H NMR (500 MHz, $CDCl_3$): δ 8.13 (d, J=8.4 Hz, 2H), 7.89 (d, J=8.4 Hz, 2H), 7.67–7.65 (m, 2H), 7.57–7.50 (m, 3H). ^{19}F NMR (470 MHz, $CDCl_3$): δ −78.60 (s, 3F). ^{13}C NMR (126 MHz, $CDCl_3$): δ 149.6, 138.4, 131.3, 129.6, 129.4, 129.3, 128.4, 127.6, 119.9 (q, J=323 Hz). GC–MS m/z 286 (M^+). HRMS (EI) m/z: calcd for $[M]^+$: $C_{13}H_9F_3O_2S$: 286.0275; found: 286.0274. IR (KBr) ν 3034, 2919, 1723, 1591, 1361, 1213, 1138, 1071, 764, 675 cm^{-1}.

4.2.7. 1-(Trifluoromethylsulfonyl)naphthalene[23] (Table 2, entry 7). Obtained as a yellow oil in 76% yield (99.4 mg). R_f (5% Et_2O/pentane): 0.43. ^1H NMR (500 MHz, $CDCl_3$): δ 8.84 (d, J=8.7 Hz, 1H), 8.50 (dd, J=7.5, 1.2 Hz, 1H), 8.33 (d, J=8.2 Hz, 1H), 8.04 (d, J=8.2 Hz, 1H), 7.82–7.76 (m, 1H), 7.74–7.68 (m, 2H). ^{19}F NMR (470 MHz, $CDCl_3$): δ −77.80 (s, 3F). ^{13}C NMR (126 MHz, $CDCl_3$): δ 138.4, 135.2, 134.3, 130.2, 129.7, 129.3, 127.7, 126.9, 124.5, 124.4, 120.3 (q, J=323 Hz). GC–MS m/z 260 (M^+).

4.2.8. 2-(Trifluoromethylsulfonyl)naphthalene (Table 2, entry 8). Obtained as a white solid in 76% yield (98.5 mg); mp 55–57 °C. R_f (5% Et_2O/pentane): 0.43. ^1H NMR (500 MHz, $CDCl_3$): δ 8.69 (s, 1H), 8.16–8.07 (m, 2H), 8.03 (d, J=8.2 Hz, 1H), 7.98 (d, J=9.8 Hz, 1H), 7.81 (t, J=7.6 Hz, 1H), 7.74 (t, J=7.6 Hz, 1H). ^{19}F NMR (470 MHz, $CDCl_3$): δ −78.18 (s, 3F). ^{13}C NMR (126 MHz, $CDCl_3$): δ 136.6, 134.1, 132.1, 130.9, 130.2, 129.9, 128.4, 128.2, 128.1, 123.8, 120.0 (q, J=323 Hz). GC–MS m/z 260 (M^+). HRMS (EI) m/z: calcd for $[M]^+$: $C_{11}H_7F_3O_2S$: 260.0119; found: 260.0121. IR (KBr) ν 3057, 2919, 2849, 1624, 1364, 1064, 1141, 1124 , 664, 578 cm^{-1}.

4.2.9. 1-Methoxy-4-(trifluoromethylsulfonyl)benzene[7e,24] (Table 2, entry 9). Obtained as a yellow oil in 41% yield (49.6 mg). R_f (5% Et_2O/pentane): 0.19. ^1H NMR (500 MHz, $CDCl_3$): δ 7.98 (d, J=9.0 Hz, 2H), 7.14–7.11 (m, 2H), 3.96 (s, 3H). ^{19}F NMR (470 MHz, $CDCl_3$): δ −78.88 (s, 3F). ^{13}C NMR (126 MHz, $CDCl_3$): δ 166.4, 133.2, 122.0, 119.9 (q, J=323 Hz), 115.3, 55.7. GC–MS m/z 240 (M^+). The spectroscopic data are consistent with those reported in the literature.

4.2.10. 4-(Trifluoromethylsulfonyl)benzonitrile[7c] (Table 2, entry 10). Obtained as a white solid in 88% yield (103.6 mg). R_f (10% Et_2O/pentane): 0.26. ^1H NMR (500 MHz, $CDCl_3$): δ 8.21 (d, J=8.3 Hz, 2H), 8.02 (d, J=8.3 Hz, 2H). ^{19}F NMR (470 MHz, $CDCl_3$): δ −77.67 (s, 3F). ^{13}C NMR (126 MHz, $CDCl_3$): δ 135.6, 133.5, 131.4, 120.8, 119.3 (q, J=323 Hz), 116.4. GC–MS m/z 235 (M^+).

4.2.11. 1-Nitro-3-(trifluoromethylsulfonyl)benzene[25] (Table 2, entry 11). Obtained as a yellow oil in 75% yield (95.6 mg). R_f (10% Et_2O/pentane): 0.13. ^1H NMR (500 MHz, $CDCl_3$): δ 8.91 (s, 1H), 8.75–8.72 (m, 1H), 8.41 (d, J=8.0 Hz, 1H), 7.99 (t, J=8.0 Hz, 1H). ^{19}F NMR (470 MHz, $CDCl_3$): δ −77.53 (s, 3F). ^{13}C NMR (126 MHz, $CDCl_3$): δ 148.8, 136.0, 133.8, 131.5, 130.9, 126.0, 119.5 (q, J=323 Hz). GC–MS m/z 255 (M^+). The spectroscopic data are consistent with those reported in the literature.

4.2.12. 1-Fluoro-4-(trifluoromethylsulfonyl)benzene[26] (Table 2, entry 12). Obtained as a yellow oil in 38% yield (43.9 mg). R_f (5% Et_2O/pentane): 0.52. ^1H NMR (500 MHz, $CDCl_3$): δ 8.14–8.09 (m, 2H), 7.42–7.36 (m, 2H). ^{19}F NMR (470 MHz, $CDCl_3$): δ −78.16 (s, 3F), −97.40 (s, 1F). ^{13}C NMR (126 MHz, $CDCl_3$): δ 167.7 (d, J=260.3 Hz), 134.0 (d, J=10.3 Hz), 127.2, 119.7 (q, J=323 Hz), 117.5 (d, J=22.7 Hz). GC–MS m/z 228 (M^+). The spectroscopic data are consistent with those reported in the literature.

4.2.13. 8-(Trifluoromethylsulfonyl)quinoline (Table 2, entry 13). Obtained as a white solid in 81% yield (105.3 mg); mp 106–108 °C. R_f (5% Et_2O/pentane): 0.17. ^1H NMR (500 MHz, $CDCl_3$): δ 9.21–9.18 (m, 1H), 8.70–8.66 (m, 1H), 8.35 (dd, J=8.3, 1.5 Hz, 1H), 8.31 (dd, J=8.3, 1.5 Hz, 1H), 7.81 (t, J=7.8 Hz, 1H), 7.66–7.64 (m). ^{19}F NMR (470 MHz, $CDCl_3$): δ −74.35 (s, 3F). ^{13}C NMR (126 MHz, $CDCl_3$):

δ 152.5, 145.1, 137.2, 136.5, 136.1, 130.7, 129.1, 125.7, 123.0, 120.2 (q, J=323 Hz). GC–MS m/z 261 (M^+). Anal. Calcd for $C_{10}H_6F_3NO_2S$: C, 45.98; H, 2.32; N, 5.36. Found: C, 45.88; H, 2.60. N, 5.35. IR (KBr) ν 3056, 1610, 1596, 1560, 1495, 1354, 1200, 1106, 833, 790, 673 cm^{-1}.

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Supplementary data

Copies of NMR spectra for all products. Supplementary data related to this article. Supplementary data related to this article can be found online at http://dx.doi.org/10.1016/j.tet.2013.01.041.

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