A convenient synthesis of CF_3-containing alkynyl imines by a copper-free Sonogashira coupling of imidoyl chlorides with terminal alkynes

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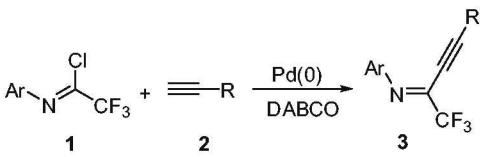
A series of *N*-aryltrifluoroacetimidoyl chlorides and 1-alkynes were converted into α-imino alkynes by a Sonogashira reaction using $Pd(PPh_3)_4$/DABCO as the catalyst. The reaction proceeded smoothly to give the coupled products in good to excellent yields.

Keywords: *N*-aryltrifluoroacetimidoyl chlorides, α-imino alkynes, Sonogashira reaction

The introduction of a trifluoromethyl group into organic compounds is attractive due to their unique chemical,[1] physical, and biological properties.[2] The trifluoromethylated organic, inorganic, and organometallic compounds have been used in the fields of pharmaceutical, agrochemical, and polymer chemistry.[3,4] Accordingly, the development of new reagents and improved techniques for the synthesis of the trifluoromethyl-containing organic compounds continues to be an important area of research in organofluorine chemistry.[5] Extensive studies have been made to develop a cheaper and more efficient synthetic methodology for the introduction of the trifluoromethyl group into organic compounds.[6]

Trifluoromethyl alkynyl imines can serve as important building blocks for the synthesis of different heterocycles, such as quinolines,[7] pyrazoles and pyrimidines.[8] There are many approaches to the preparation of alkynyl imines.[9-11] Among them, the most straightforward and powerful is the Sonogashira coupling reaction of imidoyl halides with terminal alkynes using $Pd(PPh)_2Cl_2$ and CuI as the catalyst.[12-14] However, the presence of CuI can result in the formation of some Cu(I) acetylides *in situ* which can readily produce an oxidative homocoupling reaction of the alkynes.[15] This side reaction is problematic when the terminal acetylenes are difficult to obtain. To overcome this drawback, examples have been reported concerning palladium-catalysed Sonogashira coupling reactiong using copper-free reaction conditions.[16,17]

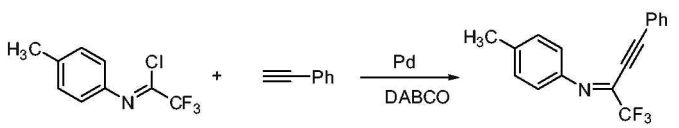
It is well known that the sp^2 carbon-iodine bond is more reactive than carbon-chlorine bond in coupling reaction. Uneyama *et al.*[18] reported the preparation of trifluoromethyl alkynyl imines from the Sonogashira reaction of *N*-aryl-trifluoroacetimidoyl iodides with 1-alkynes catalysed by $PdCl_2$/CuI.[18] They also pointed that the corresponding chlorides were recovered intact in the same conditions. Herein, we would like to report our research result of copper-free Sonogashira reaction of *N*-aryltrifluoroacetimidoyl chlorides with 1-alkynes in the catalyst system of $Pd(PPh_3)_4$/DABCO.



Scheme 1

The cross-coupling of *N*-(*p*-tolyl)-2,2,2-trifluoroacetimidoyl chloride(**1a**) with phenyl acetylene was chosen as the model reaction to optimise reaction conditions. DABCO was used as the base because triethylamine would evaporate at 110°C. In screening the catalyst, $Pd(OAc)_2$, $Pd(OOCCF_3)_2$, $Pd_2(dba)_3$ and $Pd(PPh_3)_4$ were tested at room temperature or 110°C in toluene. As shown in Table 1, $Pd(PPh_3)_4$ displayed higher catalytic activity than others. More importantly, increasing the reaction temperature accelerated the reaction to give 93% yield (entry 8). Note that 10 mol % catalyst was necessary and the yield of **3aa** decreased to 70% in the presence of only 5 mol % $Pd(PPh_3)_4$ (entry 9). In addition, the use of 1, 4-dioxane, acetonitrile or THF to replace toluene as solvent

Table 1 Optimisation of Sonogashira reaction of *N*-(*p*-tolyl)-2, 2, 2-trifluoroacetimidoyl chloride with phenylacetylene



Entry	Catalyst/mol%[a]	Solvent	Temperature/°C	**3aa** Yields/%[b]
1	$Pd(OAc)_2$(10)	Toluene	25	41
2	$Pd(OOCCF_3)_2$ (10)	Toluene	25	36
3	$Pd_2(dba)_3$(10)	Toluene	25	30
4	$Pd(PPh_3)_4$(10)	Toluene	25	48
5	$Pd(OAc)_2$(10)	Toluene	110	65
6	$Pd(OOCCF_3)_2$ (10)	Toluene	110	54
7	$Pd_2(dba)_3$(10)	Toluene	110	59
8	$Pd(PPh_3)_4$(10)	Toluene	110	93
9	$Pd(PPh_3)_4$(5)	Toluene	110	70
10	$Pd(PPh_3)_4$(10)	1,4-dioxane	100	43
11	$Pd(PPh_3)_4$(10)	MeCN	80	41
12	$Pd(PPh_3)_4$(10)	THF	65	53

[a]Reaction conditions were: **1a** (1.0 mmol), **2a** (1.2 mmol), DABCO(3.0 mmol) in solvent (3 ml).
[b]Isolated yield.

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afforded lower yield (entries 10–12). Therefore, the catalytic system of $Pd(PPh_3)_4$/DABCO in toluene was considered to be an efficient catalytic system for the Sonogashira cross-coupling of *N*-aryltrifluoroacetimidoyl chlorides. The results are summarised in Table 1.

With the optimised reaction conditions in hand, a series of *N*-aryltrifluoroacetimidoyl chlorides were converted to corresponding trifluoromethyl-containing alkynyl imines. The results are summarised in Table 2. All *N*-aryltrifluoroacetimidoyl chlorides reacted smoothly in the procedure and the products were isolated in excellent yields. Substituent effects of *N*-aryl have significant influence on yields. Electron–donating group in *N*-aryl (*e.g.* methyl or methoxy groups) raised the yields slightly (entries 1–7). On the contrary, electron-withdraw group in *N*-aryl reduced yields (entries 8–12). The strong electron-withdrawing group on the *N*-aryl (*e.g.* NO_2) reduced the stability of the coupling products. This resulted in the decomposition of the products and reduction of the yields[19] (entry 15). In addition, *o*-substituents on the aryl ring reduced the yields (entries 13–15), due to the steric hindrance in the substrate.

In conclusion, we have successfully developed a method for the synthesis of trifluoromethylated α-imino alkynes by the cross-coupling reaction of *N*-aryl-trifluoroacetimidoyl chlorides with 1-alkyenes catalysed by $Pd(PPh_3)_4$/DABCO system.

Experimental

^1H NMR were determined in a $CDCl_3$ solution with a Bruker Avance300 (300 M Hz) spectrometer using tetramethylsilane as the internal standard. ^{19}F NMR spectra were also obtained in a $CDCl_3$ solution on Bruker Avance300 (282.2 MHz) spectrometer using $CFCl_3$ as external standard. All chemical shifts (δ) were expressed in parts per million, and coupling constants (*J*) were given in Hertz. Column chromatography was performed using EM Silica gel 60 (300–400 mesh)

General procedure for the palladium-catalysed coupling reaction of phenylacetylene **2a** *with N-(p-Tolyl)-2, 2, 2-trifluoroacetimidoyl chloride* **1a**

N-(p-Tolyl)-2, 2, 2-trifluoroacetimidoyl chloride **1a** (221 mg, 1 mmol) and phenylacetylene **2a**(122 mg, 1.2 mmol) were added successively. To a mixture of $Pd(PPh_3)_4$(115 mg, 0.1 mmol), 1, 4-diazabicyclo[2.2.2] octane (DABCO) (336 mg, 3 mmol) in toluene (5 ml), The reaction mixture was stirred at 110 °C for 8 hours under N_2 atmosphere, then the solvent was evaporated under reduced pressure. The residue was purified by flash column chromatography on a silica gel using EtOAc/petroleum ether(1:10) as eluant to give the product.

The physical and spectra data of all compounds are as follows.

4-Methyl-N-(1,1,1-trifluoro-4-phenylbut-3-yn-2-ylidene)aniline (**3aa**): Yellowish oil. ^1H NMR: δ 7.42–7.47 (m, 3H), 7.32–7.39 (m, 4H), 7.24–7.27 (m, 2H), 2.41 (s, 3H); ^{19}F NMR: δ 71.36(s); IR (neat, cm^{-1}): 3041, 2928, 2199, 1605, 1499, 1445, 1342, 1151; MS (ESI) *m/z* 288 ($M + H^+$); Anal. Calcd for $C_{17}H_{12}F_3N$: C, 71.07; H, 4.21; N, 4.88. Found: C, 71.03; H, 4.19; N, 4.89%.

Methyl-N-(1,1,1-trifluoro-5-phenylpent-3-yn-2-ylidene)aniline (**3ab**): Yellowish solid, m.p. 95–96 °C ^1H NMR: δ 8.15 (d, *J* = 8.3 Hz, 1H), 8.11 (s, 1H), 7.64 (d, *J* = 8.3 Hz, 1H), 7.53 (s, 1H), 7.13–7.42 (m, 5H), 4.48 (s, 2H), 2.56 (s, 3H); ^{19}F NMR: δ 71.64(s); IR (KBr, cm^{-1}): 3043, 2928, 2211, 1592, 1505, 1460, 1373, 1151; MS (ESI) *m/z* 302 ($M + H^+$); Anal. Calcd for $C_{18}H_{14}F_3N$: C, 71.75; H, 4.68; N, 4.65. Found: C, 71.72; H, 4.65; N, 4.68%.

4-Methyl-N-(1,1,1-trifluorooct-3-yn-2-ylidene)aniline (**3ac**): Yellowish solid, m.p. 90–92 °C ^1H NMR: δ 8.11 (d, *J* = 8.6 Hz, 1H), 7.83 (s, 1H), 7.63 (d, *J* = 8.6 Hz, 1H),7.53 (s, 1H), 3.11 (t, *J* = 7.9 Hz 2H), 2.60 (s, 3H), 1.72–1.82 (m, 2H), 1.45–1.53 (m, 2H), 1.01 (t, *J* = 7.4 Hz, 3H); ^{19}F NMR: δ 67.39(s); IR (KBr, cm^{-1}): 3043, 2928, 2864, 2211, 1593, 1505, 1460, 1373, 1154; MS (ESI) *m/z* 268 ($M + H^+$); Anal. Calcd for $C_{15}H_{16}F_3N$: C, 67.40; H, 6.03; N, 5.24. Found: C, 67.36; H, 6.05; N, 5.22%.

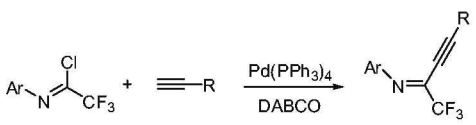
4-Methyl-N-(1,1,1-trifluorooct-3-yn-2-ylidene)aniline (**3ad**): Yellowish solid, m.p. 84–86 °C ^1H NMR: δ 8.11 (d, *J* = 8.6 Hz, 1H), 7.83 (s, 1H), 7.61 (d, *J* = 8.6 Hz, 1H), 7.53 (s, 1H), 3.11 (t, *J* = 7.9 Hz 2H), 2.60 (s, 3H), 1.76–1.81 (m, 2H), 1.50–1.54 (m, 2H), 1.38–1.44 (m, 2H), 0.97 (t, *J* = 7.4 Hz, 3H); ^{19}F NMR: δ 67.38(s); . IR (KBr, cm^{-1}): 3043, 2928, 2864, 2211, 1593, 1505, 1460, 1373, 1154; MS (ESI) *m/z* 282 ($M + H^+$); Anal. Calcd for $C_{16}H_{18}F_3N$: C, 68.31; H, 6.45; N, 4.98. Found: C, 68.28; H, 6.42; N, 4.97%.

4-Methoxy-N-(1,1,1-trifluoro-4-phenylbut-3-yn-2-ylidene)aniline (**3ba**): Yellowish oil. ^1H NMR: 7.48–7.56 (m, 4H), 7.43–7.49 (m, 3H), 6.97 (d, *J* = 8.9 Hz, 2H), 3.86 (s, 3H); ^{19}F NMR: δ 71.05(s); IR (neat, cm^{-1}): 2942, 2365, 1612, 1587, 1449, 1259, 1154; MS (ESI) *m/z* 304 ($M + H^+$); Anal. Calcd for $C_{17}H_{12}F_3NO$: C, 67.32; H, 3.99; N, 4.62. Found: C, 67.36; H, 3.97; N, 4.61%.

4-Methoxy-N-(1,1,1-trifluoro-5-phenylpent-3-yn-2-ylidene)aniline (**3bb**): Yellowish solid m.p. 88–90 °C ^1H NMR: δ 8.12 (d, *J* = 9.3 Hz, 1H), 7.47 (s, 1H), 7.33–7.42 (m, 4H), 7.20–7.31 (m, 3H), 4.41 (s, 2H), 3.85 (s, 3H); ^{19}F NMR: δ 71.18(s); IR (KBr, cm^{-1}): 3043, 2921, 2201, 1605, 1515, 1461, 1363, 1154; MS (ESI) *m/z* 318 ($M + H^+$); Anal. Calcd for $C_{18}H_{14}F_3NO$: C, 68.13; H, 4.45; N, 4.41. Found: C, 68.09; H, 4.46; N, 4.39%.

4-Methoxy-N-(1,1,1-trifluoronon -3-yn-2-ylidene)aniline (**3bc**): Yellowish solid, m.p. 76–78 °C ^1H NMR: δ 7.42 (d, *J* = 8.9 Hz, 2H), 6.92 (d, *J* = 8.9 Hz, 2H), 3.83 (s, 3H), 2.41 (t, *J* = 7.0 Hz, 2H), 1.54–1.59 (m, 2H), 1.33–1.36 (m, 2H), 1.30–1.32 (m, 2H), 0.96 (t, *J* = 6.8 Hz, 3H); ^{19}F NMR: δ 67.37(s); IR (KBr, cm^{-1}): 3043, 2958, 2863, 2231,

Table 2 The Sonogashira reaction of *N*-aryltrifluoroacetimidoyl chlorides and1-alkyne



1 2 3

Entry	Ar(**1a–e**)	R(**2a–d**)	Product **3**	Yield/%[a]
1	4-CH_3-C_6H_4(**1a**)	Ph(**2a**)	**3aa**	93
2	4-CH_3-C_6H_4(**1a**)	$C_6H_5CH_2$(**2b**)	**3ab**	95
3	4-CH_3-C_6H_4(**1a**)	n-C_4H_9(**2c**)	**3ac**	94
4	4-CH_3-C_6H_4(**1a**)	n-C_5H_{11}(**2d**)	**3ad**	96
5	4-CH_3O-C_6H_4(**1b**)	Ph(**2a**)	**3ba**	95
6	4-CH_3O-C_6H_4(**1b**)	$C_6H_5CH_2$(**2b**)	**3bb**	94
7	4-CH_3O-C_6H_4(**1b**)	n-C_5H_{11}(**2c**)	**3bc**	87
8	4-Cl-C_6H_4(**1c**)	Ph(**2a**)	**3ca**	88
9	4-Cl-C_6H_4(**1c**)	$C_6H_5CH_2$(**2b**)	**3cb**	73
10	4-Cl-C_6H_4(**1c**)	n-C_4H_9(**2c**)	**3cc**	83
11	4-Cl-C_6H_4(**1c**)	n-C_5H_{11}(**2d**)	**3cd**	82
12	4-Cl-C_6H_4(**1c**)	n-C_3H_7(**2e**)	**3ce**	83
13	2-CH_3-C_6H_4(**1d**)	Ph(**2a**)	**3da**	88
14	2-CH_3-C_6H_4(**1d**)	$C_6H_5CH_2$(**2b**)	**3db**	81
15	2-NO_2-C_6H_4(**1e**)	Ph(**2a**)	**3ea**	60

[a]Isolated yield.

1593, 1505, 1470, 1373, 1152; MS (ESI) *m/z* 298 (M + H^+); Anal. Calcd for $C_{16}H_{18}F_3NO$: C, 64.64; H, 6.10; N, 4.71. Found: C, 64.69; H, 6.13; N, 4.69%.

4-Chloro-N-(1,1,1-trifluoro-4-phenylbut-3-yn-2-ylidene)aniline (**3ca**): Yellowish oil. ^1H NMR: δ 7.42–7.47 (m, 3H), 7.32–7.39 (m, 4H), 7.24–7.29 (m, 2H); ^{19}F NMR: δ 71.66(s); IR (neat, cm^{-1}): 3052, 2926, 2199, 1603, 1507, 1442, 1154; MS (ESI) *m/z* 308 (M + H^+); Anal. Calcd for $C_{16}H_9ClF_3N$: C, 62.45; H, 2.95; N, 4.55. Found: C, 62.41; H, 2.96; N, 4.57%.

4-Chloro-N-(1,1,1-trifluoro-5-phenylpent-3-yn-2-ylidene)aniline (**3cb**): Yellowish solid, m.p. 106–108 °C ^1H NMR: δ 8.14 (d, *J* = 9.0 Hz, 1H), 8.05 (s, 1H), 7.70 (d, *J* = 9.0 Hz, 1H), 7.45 (s, 1H), 7.15–7.32 (m, 5H), 4.41 (s, 2H); ^{19}F NMR: δ 71.83(s); IR (KBr, cm^{-1}): 3054, 2918, 2196, 1605, 1516, 1443, 1154; MS (ESI) *m/z* 322 (M + H^+); Anal. Calcd for $C_{17}H_{11}ClF_3N$: C, 63.46; H, 3.45; N, 4.35. Found: C, 63.42; H, 3.43; N, 4.36%.

4-Chloro-N-(1,1,1-trifluorooct -3-yn-2-ylidene)aniline (**3cc**): Yellowish solid, m.p. 95–97 °C ^1H NMR: δ 8.16 (d, *J* = 9.0 Hz, 1H), 8.06 (s, 1H), 7.75 (d, *J* = 9.0 Hz, 1H), 7.58 (s, 1H), 3.10 (t, *J* = 8.0 Hz, 2H), 1.74–1.82 (m, 2H), 1.43–1.53 (m, 2H), 1.01 (t, *J* = 7.3 Hz, 3H); ^{19}F NMR: δ 67.63(s); IR (KBr, cm^{-1}): 3042, 2961, 2219, 1621, 1599, 1495, 1376, 1154; MS (ESI) *m/z* 288 (M + H^+); Anal. Calcd for $C_{14}H_{13}ClF_3N$: C, 58.44; H, 4.55; N, 4.87. Found: C, 58.48; H, 4.53; N, 4.85%.

4-Chloro-N-(1,1,1-trifluoronon-3-yn-2-ylidene)aniline (**3cd**): Yellowish solid, m.p. 96–98 °C ^1H NMR: δ 8.15 (d, *J* = 9.0 Hz, 1H), 8.05 (s, 1H), 7.73 (d, *J* = 9.0 Hz, 1H), 7.58 (s, 1H), 3.08 (t, *J* = 8.0 Hz, 2H), 1.76–1.83 (m, 2H), 1.47–1.60 (m, 2H), 1.37–1.46 (m, 2H), 0.93 (t, *J* = 7.1 Hz, 3H); ^{19}F NMR: δ 67.65(s); IR (KBr, cm^{-1}): 3042, 2931, 2217, 1599, 1509, 1495, 1375, 1154; MS (ESI) *m/z* 302 (M + H^+); Anal. Calcd for $C_{15}H_{15}ClF_3N$: C, 59.71; H, 5.01; N, 4.64. Found: C, 59.68; H, 5.02; N, 4.62%.

4-Chloro-N-(1,1,1-trifluorohept-3-yn-2-ylidene)aniline (**3ce**): Yellowish solid, m.p. 131–133 °C ^1H NMR: δ 8.15 (d, *J* = 9.0 Hz, 1H), 8.05 (s, 1H), 7.73 (d, *J* = 9.0 Hz, 1H), 7.58 (s, 1H), 3.07 (t, *J* = 7.6 Hz, 2H), 1.79–1.87 (m, 2H), 1.07 (t, *J* = 7.4 Hz, 3H); ^{19}F NMR: δ 67.67(s); IR (KBr, cm^{-1}): 3042, 2961, 2299, 1691, 1599, 1495, 1376, 1154; MS (ESI) *m/z* 274 (M + H^+); Anal. Calcd for $C_{13}H_{11}ClF_3N$: C, 57.05; H, 4.05; N, 5.12. Found: C, 57.09; H, 4.07; N, 5.09%.

2-Methyl-N-(1,1,1-trifluoro-4-phenylbut-3-yn-2-ylidene)aniline (**3da**): Yellowish oil. ^1H NMR: δ 7.42–7.55 (m, 3H), 7.24–7.36 (m, 4H), 7.16–7.19 (m, 2H), 2.83 (s, 3H); ^{19}F NMR: δ 71.17(s); IR (neat, cm^{-1}): 3062, 2934, 2200, 1607, 1479, 1347, 1150; MS (ESI) *m/z* 288 (M + H^+); Anal. Calcd for $C_{17}H_{12}F_3N$: C, 71.07; H, 4.21; N, 4.88. Found: C, 71.04; H, 4.18; N, 4.87%.

4-Methyl-N-(1,1,1-trifluoro-5-phenylpent-3-yn-2-ylidene)aniline (**3db**): Yellowish oil ^1H NMR: δ 7.88 (d, *J* = 8.2 Hz, 1H),7.57 (d, *J* = 8.2 Hz, 1H), 7.45 (t, *J* = 6.4 Hz, 2H),7.12–7.30 (m, 5H), 4.43 (s, 2H), 2.82 (s, 3H); ^{19}F NMR: δ71.26(s); IR (neat, cm^{-1}):3065, 2962, 2214, 1619, 1467, 1336, 1150; MS (ESI) *m/z* 302 (M + H^+); Anal. Calcd for $C_{18}H_{14}F_3N$: C, 71.75; H, 4.68; N, 4.65. Found: C, 71.72; H, 4.70; N, 4.66%.

2-Nitro-N-(1,1,1-trifluoro-4-phenylbut-3-yn-2-ylidene)aniline (**3ea**): Yellowish oil. ^1H NMR: δ 7.53–7.54 (m, 4H), 7.26–7.36 (m, 5H).); ^{19}F NMR: 71.70(s); IR (neat, cm^{-1}): 3065, 2922, 2236, 1591, 1479, 1152; MS (ESI) *m/z* 319 (M + H^+). Anal. Calcd for $C_{16}H_9F_3N_2O_2$: C, 60.38; H, 2.85; N, 8.80. Found: C, 60.35; H, 2.83; N, 8.83.

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