A facile trifluoromethylthiolation of 3-chloro-1H-inden-1-ones employing AgSCF₃ and KI

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Abstract
An efficient method for trifluoromethylthiolation of functionalized 3-chloro-1H-inden-1-ones was described. Within this method, AgSCF₃ was employed as a nucleophilic reagent and KI was functionalized as an activator. This reaction provided the trifluoromethylthiolated indenones with excellent yields under moderate conditions.

Keywords: 3-Chloro-1H-inden-1-ones, trifluoromethylthiolation, AgSCF₃, KI

Introduction
Fluorinated compounds have received increasing attention within organic synthesis and medicinal/pharmaceutical science because of special properties of fluorine atoms, which are the most electronegative elements with a small atomic radius. Among these fluorine-containing groups, the trifluoromethylthio group (SCF₃) is of particular interest. The incorporation of the SCF₃ moiety into drug candidates can lead to the increase of compound’s membrane permeability, bioavailability and metabolic stability because of its high lipophilicity and electron-withdrawing properties.¹⁻² Some SCF₃ group-containing pharmaceutical products or pesticides have been reported [shown in Figure 1  ],³ including toltrazuril,⁴ tiflorex,⁵ and fiptonil.⁶ And the increasing number of the SCF₃-containing bioactive lead compounds, such as amebiasis trifloromethionine⁷ and potential hypotensive agents of losartan and nifedipin analogues⁸ also prove the importance of introducing the SCF₃ moiety into drug candidates.
Many research groups have been managed to explore efficient methods introducing the SCF₃ group to small heterocyclic molecules. Earlier trifluoromethylthiolation strategies can be classified as indirect and direct methods. The indirect methods include halogen-fluorine exchange and trifluoromethylation of sulfur-containing compounds. However, both of the indirect methods require harsh conditions and have a narrow substrate scope. The direct trifluoromethylthiolation methods are based on either electrophilic or nucleophilic pathways. As for heterocyclic scaffolds, such as benzofurans/benzothiophenes, isocoumarin, indole and oxidine, they can be trifluoromethylthiolated by N-trifluoromethanesulfanylamides or hypervalent iodine trifluoromethanesulfenate reagent and N-trifluoromethylthiosaccharin. AgSCF₃, as the most common SCF₃-containing nucleophilic reagent, plays a key role in trifluoromethylthiolation of various bioactive structures in medicinal chemistry. And trifluoromethylthiolation of chromone derivatives using AgSCF₃ was achieved by our group.

The indenone moiety is one of the privileged scaffolds in medicinal chemistry owing to its various biological activities. Indenone-containing compounds were widely employed as agonists for estrogen receptor and peroxisome proliferator-activated receptor γ (PPARγ). They also has been used as cyclooxygenase-2 (COX-2) and topoisomerase I (Top I) inhibitors and so on. Therefore, incorporation of the SCF₃ group into the indenone moiety can lead to novel series of heterocyclic scaffolds and will bring about further advances in the pharmacological applications. Inspired by previous work, we proposed a simple synthetic route to generate substituted indenone analogues in this work.
Results and Discussion

Compound 1a, which was synthesized from phenylacetic acid, phthalic anhydride, and phosphorus oxychloride,\(^{30}\) was selected as the model substrate for optimal conditions’ screening (shown in Table 1). However, reactions did not occur when AgSCF\(_3\) was simply added even at different temperatures (entries 1-3). This may be caused by the low activity of AgSCF\(_3\) to proceed this reaction. Considering the application of KI for trifluoromethylthiolation as the addition,\(^{31-32}\) KI (2 Equiv.) was introduced to accelerate reaction. Then compound 2a was obtained at 60 °C in 51% yield (entry 4). Based on this, a series of solvents were tested. The results showed that the utilization of CH\(_3\)CN as solvent provided 2a with the highest yield (entries 4-6). This solvent was confirmed to have a significant impact on yields. Additionally, the replacement of KI to the NaI can lead to the decease of yields (entry 7). It was also found that reactions tend to have better yields under nitrogen environment compared with no inert gas protection (entry 8).

Table 1. Optimization of Reaction Conditions\(^a\)

<table>
<thead>
<tr>
<th>Entry</th>
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<th>Solvent</th>
<th>Temperature (°C)</th>
<th>Yield (%)(^c)</th>
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<td>DMSO</td>
<td>20</td>
<td>NR(^b)</td>
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<td>-</td>
<td>DMSO</td>
<td>40</td>
<td>NR</td>
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<td>DMSO</td>
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<td>NR</td>
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<td>DMSO</td>
<td>60</td>
<td>51</td>
</tr>
<tr>
<td>5</td>
<td>KI</td>
<td>DMF</td>
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<td>48</td>
</tr>
<tr>
<td>6</td>
<td>KI</td>
<td>CH(_3)CN</td>
<td>60</td>
<td>88</td>
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<tr>
<td>8</td>
<td>KI</td>
<td>CH(_3)CN</td>
<td>60</td>
<td>94(^d)</td>
</tr>
</tbody>
</table>

\(^{a}\)1a (0.5mmol), AgSCF\(_3\) (1mmol) and solvent (2mL) were used, \(^{b}\)NR= no reaction, \(^{c}\)Isolated yield, \(^{d}\)Under argon atmosphere.

Under the optimized reaction conditions (2 equiv. AgSCF\(_3\) and 2 equiv. KI in CH\(_3\)CN at 60 °C), a variety of indenone derivatives were applied as substrates. As shown in Figure 2, this reaction was compatible with both electron-withdrawing groups (fluoro, bromo, chloro, nitro, trifluoromethyl group) and electron-donating groups (methyl, methoxy, dimethoxy moities). The yields for different electron-withdrawing groups-containing substrates were similar. Besides, electron-withdrawing substituents at different positions of the aromatic ring provide desire
products with excellent yields (2d, 2e, 2f). However, substrates containing electron-donating groups were different (2j, 2k, 2l). Compound 2k has the lowest yield at 60 °C. At first we think this reaction was sensitive for steric hindrance (2e vs 2k). To our surprise, the yields are almost the same when the bulkier naphthalenyl group was employed instead of the phenyl group (2n, 2o vs 2a). This result suggests that trifluoromethylthiolation of 3-chloro-1H-inden-1-ones via AgSCF₃ and KI to functional 2-aryl-3-((trifluoromethyl)thio)-1H-inden-1-ones have broader application due to its wide substrates tolerance. Compared with previous work, the experimental conditions are milder and the synthetic method has broad scope of substrates and excellent compatibility of functional-groups for the presence of the activated chlorine atom on indenone core.

![Diagram of reaction scheme]

\(^{a}\)Reaction condition: 1(0.5mmol), AgSCF₃(1mmol) and KI(1mmol) in CH₃CN at 60 °C, 2-4 h.

\(^{b}\)Isolated yield. \(^{c}\)Raw material recycling is more than 80%.

**Figure 2.** Exploration of reaction scope.\(^{a,b}\)
Conclusions

A novel and facile method for synthesis of SCF$_3$-substituted indenones via AgSCF$_3$/KI was discovered. This novel method only requires mild conditions and short time without employment of Pd or Ni catalysts. Moreover, this novel synthetic method was followed by simple work up and provided with superior yields.

Experimental Section

**General.** All reactions were performed under an argon atmosphere. Solvents and reagents are commercially available and without pretreatment. Column chromatography was employed by silica gel (200—300 mesh). $^1$H NMR spectra were recorded on a Bruker Avance 300 or a Bruker Avance 400 spectrometer in CDCl$_3$ ($\delta$ 7.26 ppm), respectively. $^{13}$C NMR spectra were recorded on a Bruker Avance 400 or Bruker Avance 500 spectrometer in CDCl$_3$ ($\delta$ 77.16 ppm).

**General procedure for the synthesis of compounds 1**

![Chemical structure 1]

Compound 1 were prepared from corresponding unsubstituted/substituted phenylacetic acid, phthalic anhydride and phosphorus oxychloride.

**General procedure for the synthesis of compounds 2a-2o**

![Chemical structure 2]

To a reaction flask were added AgSCF$_3$ (1 mmol), KI (1 mmol), compound 1 (0.5 mmol), CH$_3$CN (2 mL). The mixture was stirred at 60 $^\circ$C for 2-4 h. Afterward, the reaction mixture was poured into water and extracted with EtOAc and dried with Na$_2$SO$_4$. The solution was concentrated in
vacuo to get a crude mixture, which was purified by flash column chromatography on silica gel (petroleum ether: acetate 100:1) to pure products.

2-Phenyl-3-((trifluoromethyl)thio)-1H-inden-1-one (2a). Yellow solid, 94% Yield; mp 107-109 °C. 1H NMR (400 MHz, CDCl3) δ 7.60 – 7.50 (m, 4H), 7.50 – 7.42 (m, 4H), 7.34 (t, J 7.4 Hz, 1H). 13C NMR (125 MHz, CDCl3) δ 193.72, 145.31, 144.42, 139.64, 135.01, 130.26, 129.71, 129.69, 129.47 (q, J 312.1 Hz), 129.28, 129.12, 128.41, 123.50, 121.86. 19F NMR (471 MHz, CDCl3), δ -37.37. HRMS-ESI(m/z) Calcd for (C16H10OF3NaS) ([M+Na]+): 329.0326; found: 329.0213.

2-(4-Chlorophenyl)-3-((trifluoromethyl)thio)-1H-inden-1-one (2b). Yellow solid, 87% Yield; mp 103-105 °C. 1H NMR (400 MHz, CDCl3) δ 7.58 (d, J 7.1 Hz, 1H), 7.56 – 7.50 (m, 3H), 7.48 – 7.43 (m, 3H), 7.36 (t, J 7.4 Hz, 1H). 13C NMR (125 MHz, CDCl3) δ 193.42, 144.27, 144.04, 139.94, 136.02, 135.15, 131.57, 129.90, 129.15, 128.81, 128.68 (d, J 314.3 Hz) 127.53, 123.63, 121.99. 19F NMR (471 MHz, CDCl3), δ -37.32. HRMS-ESI(m/z) Calcd for (C16H10OClF3S) ([M+H]+): 340.9936; found: 341.0002.

2-(4-Bromophenyl)-3-((trifluoromethyl)thio)-1H-inden-1-one (2c). Yellow solid, 93% Yield; mp 110-112 °C. 1H NMR (400 MHz, CDCl3) δ 7.61 (d, J 8.4 Hz, 2H), 7.58 (d, J 7.1 Hz, 1H), 7.53 (t, J 7.4 Hz, 1H), 7.44-7.47 (m, 3H), 7.35 (t, J 7.4 Hz, 1H). 13C NMR (125 MHz, CDCl3) δ 193.33, 144.25, 144.10, 140.01, 135.16, 131.77, 131.75, 129.92, 129.14, 128.60 (d, J 311.9 Hz), 127.97, 124.43, 123.64, 122.00. 19F NMR (471 MHz, CDCl3), δ -37.30. HRMS-ESI(m/z) Calcd for (C16H9OBrF3S) ([M+H]+): 384.9431; found: 384.9500.

2-(4-Fluorophenyl)-3-((trifluoromethyl)thio)-1H-inden-1-one (2d). Yellow solid, 94% Yield; mp 87-89 °C. 1H NMR (400 MHz, CDCl3) δ 7.62 – 7.56 (m, 3H), 7.53 (t, J 7.5 Hz, 1H), 7.46 (d, J 7.3 Hz, 1H), 7.35 (t, J 7.4 Hz, 1H), 7.17 (t, J 8.7 Hz, 2H). 13C NMR (125MHz, CDCl3) δ 193.68, 163.62 (d, J 251.0 Hz), 144.37, 144.18, 139.37, 135.13, 132.36, 132.29, 129.77, 129.13, 128.70 (d, J 312.1 Hz), 125.18 (d, J 3.3 Hz), 123.59, 121.89, 115.80, 115.63. 19F NMR (471 MHz, CDCl3), δ -37.40, -110.34. HRMS-ESI(m/z) Calcd for (C16H10OF3S) ([M+H]+): 325.0232; found: 325.0299.

2-(2-Fluorophenyl)-3-((trifluoromethyl)thio)-1H-inden-1-one (2e). Yellow solid, 96% Yield; mp 56-58 ºC. 1H NMR (400 MHz, CDCl3) δ 7.59 (d, J 7.1 Hz, 1H), 7.53 (t, J 7.8 Hz, 1H), 7.49 – 7.41 (m, 2H), 7.40 – 7.29 (m, 2H), 7.29 – 7.21 (m, 1H), 7.18 (t, J 9.1 Hz, 1H). 13C NMR (125 MHz, CDCl3) δ 192.45, 160.26 (d, J 249.6 Hz), 143.87, 143.81, 141.28, 134.90, 131.76 (d, J 2.1 Hz), 131.55 (d, J 8.4 Hz), 129.99, 129.56, 128.56 (q, J 311.5 Hz), 124.20 (d, J 3.5 Hz), 123.61, 122.00, 117.46 (d, J 15.3 Hz), 116.12 (d, J 21.9 Hz). 19F NMR (471 MHz, CDCl3), δ -37.16, -110.43. HRMS-ESI(m/z) Calcd for (C16H9OF4S) ([M+H]+): 325.0232; found: 325.0302.

2-(3-Fluorophenyl)-3-((trifluoromethyl)thio)-1H-inden-1-one (2f). Yellow solid, 95% Yield; mp 103-105 ºC. 1H NMR (400 MHz, CDCl3) δ 7.59 (d, J 7.1 Hz, 1H), 7.54 (t, J 7.5 Hz, 1H), 7.51 – 7.40 (m, 2H), 7.36 (t, J 7.7 Hz, 2H), 7.30 (d, J 9.8 Hz, 1H), 7.15 (t, J 8.3 Hz, 1H). 13C NMR (125 MHz, CDCl3) δ 193.16, 162.53 (d, J 246.3 Hz), 144.11, 143.89, 140.69, 135.13, 131.03 (d, J 8.4 Hz), 130.02, 129.97, 129.13, 128.64 (d, J 312.1 Hz), 126.07 (d, J 2.6 Hz), 123.66, 122.10, 117.23 (d, J 22.9 Hz), 116.70 (d, J 21.1 Hz). 19F NMR (471 MHz, CDCl3), δ -37.25, -112.37. HRMS-ESI(m/z) Calcd for (C16H9OF4S) ([M+H]+): 325.0232; found: 325.0301.
2-(4-Nitrophenyl)-3-((trifluoromethyl)thio)-1H-inden-1-one (2g). Yellow solid, 90% Yield; mp 155-157 °C. 1H NMR (400 MHz, CDCl₃) δ 8.33 (d, J 8.5 Hz, 2H), 7.75 (d, J 8.6 Hz, 2H), 7.67 – 7.55 (m, 2H), 7.51 (d, J 7.2 Hz, 1H), 7.41 (t, J 7.3 Hz, 1H). 13C NMR (125 MHz, CDCl₃) δ 192.53, 148.18, 143.73, 142.89, 142.67, 135.53, 135.35, 131.21, 130.60, 129.02, 128.42 (d, J 312.3 Hz), 123.92, 123.56, 122.48. 19F NMR (471 MHz, CDCl₃), δ -37.04. HRMS-ESI(m/z) Calcd for (C₁₆H₉O₃NF₃S) ([M+H]⁺): 352.0177; found: 352.0244.

2-(4-(Trifluoromethyl)phenyl)-3-((trifluoromethyl)thio)-1H-inden-1-one (2h). Yellow solid, 88% Yield; mp 80-82 °C. 1H NMR (400 MHz, CDCl₃) δ 7.73 (d, J 8.3 Hz, 2H), 7.67 (d, J 8.2 Hz, 2H), 7.60 (d, J 7.2 Hz, 1H), 7.55 (td, J 7.5, 1.1 Hz, 1H), 7.49 (d, J 7.3 Hz, 1H), 7.38 (t, J 7.6 Hz, 1H). 13C NMR (125 MHz, CDCl₃) δ 193.00, 143.97, 143.86, 141.54, 135.21, 132.66, 131.37 (q, J 32.7 Hz), 130.60, 130.23, 129.12, 128.57 (q, J 312.1 Hz), 125.39, 125.36, 124.05 (d, J 272.4 Hz), 123.75, 122.24. 19F NMR (471 MHz, CDCl₃), δ -37.20, -62.88. HRMS-ESI(m/z) Calcd for (C₁₇H₁₁O₃F₃S) ([M+H]⁺): 375.0200; found: 375.0265.

2-(p-Tolyl)-3-((trifluoromethyl)thio)-1H-inden-1-one (2i). Yellow solid, 90% Yield; mp 76-78 °C. 1H NMR (400 MHz, CDCl₃) δ 7.57 (d, J 7.1 Hz, 1H), 7.52 (td, J 7.5, 1.1 Hz, 1H), 7.48 (d, J 8.2 Hz, 2H), 7.45 (d, J 7.4 Hz, 1H), 7.35 – 7.31 (m, 1H), 7.29 (d, J 7.9 Hz, 2H), 2.42 (s, 3H). 13C NMR (125MHz, CDCl₃) δ 193.95, 145.30, 144.61, 140.03, 138.64, 134.98, 130.19, 129.49, 129.32, 129.20, 128.81 (d, J 312.0 Hz), 126.24, 124.43, 121.71, 21.65. 19F NMR (471 MHz, CDCl₃), δ -37.45. HRMS-ESI(m/z) Calcd for (C₁₇H₁₁O₃F₃SNa) ([M+23⁺]: 343.0483; found: 343.0370

2-(4-Methoxyphenyl)-3-((trifluoromethyl)thio)-1H-inden-1-one (2j). Yellow solid, 95% Yield; mp 117-119 °C. 1H NMR (400 MHz, CDCl₃) δ 7.60 (d, J 9.0 Hz, 2H), 7.56 (dd, J 7.1, 0.6 Hz, 1H), 7.51 (td, J 7.6, 1.1 Hz, 1H), 7.44 (d, J 0.9 Hz, 1H), 7.31 (td, J 7.4, 1.0 Hz, 1H), 7.00 (d, J 9.0 Hz, 2H), 3.87 (s, 3H). 13C NMR (125 MHz, CDCl₃) δ 194.29, 160.91, 144.90, 144.70, 137.13, 135.03, 131.92, 129.30, 129.27, 128.87 (d, J 312.1 Hz), 123.41, 121.61, 121.55, 114.03, 55.49. 19F NMR (471 MHz, CDCl₃), δ -37.60. HRMS-ESI(m/z) Calcd for (C₁₇H₁₁O₃F₃NaS) ([M+23⁺]: 359.0432; found: 359.0317.

2-(3-Methoxyphenyl)-3-((trifluoromethyl)thio)-1H-inden-1-one (2l). Yellow solid, 92% Yield; mp 60-62 °C. 1H NMR (400 MHz, CDCl₃) δ 7.58 (d, J 7.1 Hz, 1H), 7.53 (td, J 7.5, 1.1 Hz, 1H), 7.46 (d, J 7.3 Hz, 1H), 7.39 (t, J 8.0 Hz, 1H), 7.34 (td, J 7.4, 1.0 Hz, 1H), 7.13 (dt, J 7.7, 1.2 Hz, 1H), 7.11 – 7.08 (m, 1H), 7.00 (dd, J 8.7, 3.0 Hz, 1H), 3.85 (s, 3H). 13C NMR (125MHz, CDCl₃) δ 193.58, 159.41, 145.17, 144.36, 139.84, 135.01, 130.29, 129.73, 129.46, 128.76 (d, J 311.9 Hz), 123.50, 122.75, 121.91, 115.75, 115.43, 55.44. 19F NMR (471 MHz, CDCl₃), δ -37.30. HRMS-ESI(m/z) Calcd for (C₁₇H₁₂O₂F₃S) ([M+H⁺]: 337.0432; found: 337.0501.

2-(3,4-Dimethoxyphenyl)-3-((trifluoromethyl)thio)-1H-inden-1-one (2m). Red solid, 95% Yield; mp 85-87 °C. 1H NMR (400 MHz, CDCl₃) δ 7.56 (d, J 7.2 Hz, 1H), 7.52 (td, J 7.6, 1.2 Hz, 1H), 7.43 (d, J 7.4 Hz, 1H), 7.31 (t, J 7.4 Hz, 1H), 7.29 – 7.23 (m, 2H), 6.97 (d, J 8.2 Hz, 1H), 3.94 (s, 3H), 3.93 (s, 3H). 13C NMR (125 MHz, CDCl₃) δ 194.24, 150.56, 148.68, 144.85, 144.55, 137.08, 135.08, 129.35, 129.21, 128.89 (d, J 312.2 Hz), 123.94, 123.40, 121.78, 121.57, 113.06, 110.93,
56.05, 56.03. $^{19}$F NMR (471 MHz, CDCl$_3$), δ -37.60. HRMS-ESI(m/z) Calcd for (C$_{18}$H$_{14}$O$_3$F$_3$S) ([M+H]$^+$):367.0537; found: 367.0603.

2-(Naphthalen-1-yl)-3-((trifluoromethyl)thio)-1H-inden-1-one (2n). Yellow liquid, 93% Yield; $^1$H NMR (400 MHz, CDCl$_3$) δ 7.93 (d, $J$ 8.3 Hz, 1H), 7.89 (d, $J$ 8.1 Hz, 1H), 7.61 (d, $J$ 7.1 Hz, 1H), 7.59 – 7.47 (m, 5H), 7.47 – 7.41 (m, 1H), 7.37 (t, $J$ 7.5 Hz, 2H). $^{13}$C NMR (125MHz, CDCl$_3$) δ 193.42, 146.76, 144.09, 143.83, 134.95, 133.67, 131.99, 130.03, 129.94, 129.63, 128.69, 128.47 (d, $J$ 311.8 Hz), 128.37, 127.42, 126.56, 126.33, 125.60, 125.20, 123.67, 121.91. $^{19}$F NMR (471 MHz, CDCl$_3$), δ -37.96. HRMS-ESI(m/z) Calcd for (C$_{20}$H$_{12}$OF$_3$S) ([M+H]$^+$):357.0483; found: 357.0551.

2-(Naphthalen-2-yl)-3-((trifluoromethyl)thio)-1H-inden-1-one (2o). Yellow solid, 92% Yield; mp 86-88 °C. $^1$H NMR (400 MHz,CDCl$_3$) δ 8.07 (s, 1H), 7.92 (d, $J$ 8.0 Hz, 2H), 7.87 (d, $J$ 8.8 Hz, 1H), 7.69 (dd, $J$ 8.5, 1.6 Hz,1H), 7.60 (d, $J$ 7.1 Hz, 1H), 7.50-7.56 (m,3H), 7.48 (d, $J$ 7.4 Hz, 1H), 7.36 (d, $J$ 7.7 Hz, 1H). $^{13}$C NMR (125 MHz, CDCl$_3$) δ 193.85, 145.19, 144.55, 139.61, 135.07, 133.66, 132.98, 130.80, 129.71, 129.33, 128.89, 128.79(d, $J$ 312.1 Hz) 127.99, 127.85, 127.39, 126.91, 126.64, 126.56, 123.54, 121.84. $^{19}$F NMR (471 MHz, CDCl$_3$), δ -37.33. HRMS-ESI(m/z) Calcd for (C$_{20}$H$_{12}$OF$_3$S) ([M+H]$^+$): 357.0483; found: 357.0549.

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