Supporting Information

Copper-Mediated Trifluoromethylation of Propiolic Acids:
Facile Synthesis of \( \alpha \)-Trifluoromethyl Ketones

Zhengbiao He, Rui Zhang, Mingyou Hu, Lingchun Li, Chuanfa Ni, and Jinbo Hu*

Key Laboratory of Organofluorine Chemistry
Shanghai Institute of Organic Chemistry
Chinese Academy of Sciences
345 Ling-Ling Road, Shanghai, 200032 (China)
Fax: (+86) 21-64166128
E-mail: jinbohu@sioc.ac.cn

General Methods: ………………………………………………………………………………2
Survey of the additive for the trifluoromethyldecarboxylation reaction of 2a………2
Preparation of substituted propiolic acids: …………………………………………3
Charaterization data of substituted propiolic acids: ………………………………3
General procedure for trifluoromethylation of substituted propiolic acids: ………4
Charaterization data of compounds 4: …………………………………………………4
Screens for the trifluoromethyldecarboxylation reaction of 4a …………………...8
General procedure for the trifluoromethylative decarboxylation reaction of 4………..9
Charaterization data of compounds 5 ……………………………………………………9
The isotopic labeling experiment ……………………………………………………10
Charaterization data of compound 3a' ………………………………………………12
Reference ……………………………………………………………………………………14
\(^1\text{H}, \(^{19}\text{F}, \text{and } ^{13}\text{C} \text{NMR spectra of all new products:}…………………………15
MS spectra of compound 3a' ……………………………………………………………70
General Methods

Unless otherwise mentioned, solvents and reagents were purchased from commercial sources and used without further purification. The water was used after distillation. All the melting points were uncorrected. $^1$H, $^{13}$C and $^{19}$F NMR spectra were recorded on a 400 MHz or 300 MHz NMR spectrometer. $^1$H NMR chemical shifts were determined relative to internal (CH$_3$)$_4$Si (TMS) at $\delta$ 0.0 or to the signal of a residual protonated solvent: CDCl$_3$ $\delta$ 7.26. $^{13}$C NMR chemical shifts were determined relative to internal TMS at $\delta$ 0.0. $^{19}$F NMR chemical shifts were determined relative to CFCl$_3$ at $\delta$ 0.0. Data for $^1$H, $^{13}$C and $^{19}$F NMR are recorded as follows: chemical shift ($\delta$, ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet, q = quartet, br = broad). Mass spectra were obtained on a mass spectrometer. High-resolution mass data were recorded on a high-resolution 10 mass spectrometer in the EI, ESI or MALDL mode.

**Table S1. Survey of the additive for the trifluoromethyldecarboxylation reaction of 2a**

![Chemical Structure](image)

<table>
<thead>
<tr>
<th>Entry</th>
<th>Additive (1.0 equiv)</th>
<th>Yield [%]$^a$</th>
<th>Entry</th>
<th>Additive (1.0 equiv)</th>
<th>Yield [%]$^a$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td><img src="image" alt="Additive" /></td>
<td>70</td>
<td>5</td>
<td><img src="image" alt="Additive" /></td>
<td>37</td>
</tr>
<tr>
<td>2</td>
<td><img src="image" alt="Additive" /></td>
<td>53</td>
<td>6</td>
<td><img src="image" alt="Additive" /></td>
<td>51</td>
</tr>
<tr>
<td>3</td>
<td><img src="image" alt="Additive" /></td>
<td>45</td>
<td>7</td>
<td><img src="image" alt="Additive" /></td>
<td>40</td>
</tr>
<tr>
<td>4</td>
<td><img src="image" alt="Additive" /></td>
<td>10</td>
<td>8</td>
<td><img src="image" alt="Additive" /></td>
<td>61</td>
</tr>
</tbody>
</table>

$^a$ Determined by $^{19}$F NMR spectroscopy using PhCF$_3$ as an internal standard.
Preparation of substituted propionic acids

The substituted propionic acids were prepared according to the literature procedures. The compounds 2a–2m, 4a–4h, 4l are known compounds.

Characterization data of substituted propionic acids

3-(5-Methylthiophen-2-yl)propionic acid (2o): 65% yield, yellow solid. Mp = 127–129 °C. ¹H NMR (300 MHz, CDCl₃/TMS): δ 10.1 (br, 1H), 7.36 (d, J = 3.7 Hz, 1H), 6.73 (d, J = 3.7 Hz, 1H), 2.52 (s, 3H); ¹³C NMR (100 MHz, CDCl₃/TMS): δ 158.8, 147.9, 138.2, 126.3, 116.3, 84.3, 84.2, 15.7; IR (film): 2814, 2607, 2545, 2196, 1671, 1529, 1460, 1380, 1342, 1283, 1221, 1181, 1160, 1048, 915, 881, 801, 743, 695, 665, 610 cm⁻¹; MS (EI, m/z): 166 (M⁺, 100.00), 121 (97.96); HRMS (EI): exact mass calcd for C₈H₆O₂S (M⁺): 166.0089, found: 166.0091.

3-(5-Methylfuran-2-yl)propionic acid (2p): 60% yield, yellow solid. Mp = 101–103 °C. ¹H NMR (300 MHz, CDCl₃/TMS): δ 10.2 (br, 1H), 6.93 (d, J = 15.34 Hz, 1H), 6.10 (d, J = 3.4 Hz, 1H), 2.36 (s, 3H); ¹³C NMR (100 MHz, CDCl₃/TMS): δ 158.4, 157.9, 132.6, 124.0, 108.3, 85.9, 80.5, 14.1; IR (film): 2807, 2579, 1667, 1585, 1526, 1409, 1376, 1357, 1298, 1263, 1233, 1200, 1026, 952, 910, 881, 800, 740, 689, 639, 611 cm⁻¹; MS (EI, m/z): 150 (M⁺, 100.00); HRMS (EI): exact mass calcd for C₈H₆O₃ (M⁺): 150.0317, found: 150.0316.

3-(Bicyclo[2.2.1]heptan-2-yl)propionic acid (4i): 50% yield, yellow oil. ¹H NMR (300 MHz, CDCl₃/TMS): δ 10.8 (br, 1H), 2.75 (d, J = 5.1 Hz, 0.66 H), 2.40–2.27 (m, 2.23H), 2.00–1.80 (m, 1.55H), 1.66–1.18 (m, 6.63H); ¹³C NMR (100 MHz, CDCl₃/TMS): δ 159.0, 158.8, 97.1, 96.3, 72.9, 72.2, 43.1, 41.1, 39.2, 38.0, 37.1, 36.9, 36.8, 36.1, 32.7, 31.4, 29.4, 28.7, 28.5, 24.2; IR (film): 2959, 2874, 2647, 2231, 1683, 1455, 1412, 1328, 1281, 1145, 1125, 1085, 884, 855, 826, 777, 756, 732 cm⁻¹; MS (EI, m/z): 164 (M⁺, 2.93); HRMS (EI): exact mass calcd for C₁₀H₁₂O₂ (M⁺): 164.0837, found: 164.0835.

4-Cyclopentylbut-2-ynoic acid (4j): 65% yield, yellow oil. ¹H NMR (300 MHz, CDCl₃/TMS): δ 10.8 (br, 1H), 2.37 (d, J = 6.8 Hz, 2H), 2.22–2.03 (m, 1H), 1.92–1.75 (m, 2H), 1.72–1.48 (m, 4H), 1.37–1.18 (m, 2H); ¹³C NMR (100 MHz, CDCl₃/TMS): δ 158.7, 92.3, 72.7, 38.0, 32.0, 25.0, 24.4; IR (film): 2955, 2870,
2237, 1686, 1452, 1282, 1076, 912, 786, 756 cm⁻¹; MS (ESI, m/z): 151(M-H⁺); HRMS (ESI): exact mass calcd for C₉H₁₁O₂(M-H⁺): 151.07645, found: 151.07670

5-(4-Tert-butylphenyl)-4-methylpent-2-ynoic acid: 70% yield, colorless solid. Mp= 81–83°C. ¹H NMR (300 MHz, CDCl₃/TMS): δ 11.2 (br, 1H), 7.32 (d, J = 8.1Hz, 2 H), 7.13(d, J = 8.1Hz, 2 H), 2.92-2.65 (m, 3H), 1.30 (s, 9H), 1.22 (d, J = 6.5Hz, 3 H); ¹³C NMR (100 MHz, CDCl₃/TMS): δ 158.8, 149.5, 135.1, 128.8, 125.3, 96.0, 73.4, 41.3, 34.4, 31.4, 28.0, 19.2; IR (film):2965, 2653, 2523, 2228, 1686, 1516, 1459, 1408, 1377, 1364, 1277, 1127, 1107, 1020, 982, 911, 860, 835, 805, 768, 731, 604, 583 cm⁻¹; MS (EI, m/z): 244(M⁺, 4.61), 147 (100.00); HRMS (EI): exact mass calcd for C₁₆H₂₀O₂ (M⁺): 244.1463, found: 244.1464.

General procedure for trifluoromethylation of substituted propiolic acids (Table 2) A Schlenk test tube with a magnetic stirring bar was charged with 1 (0.8 mmol, 2.0 equiv), 2 (0.4mmol, 1.0 equiv), Cu(OAc)₂·H₂O (0.8 mmol), TMEDA (1.0 mmol), followed by DCM (3 mL) and H₂O (4.5 mL). The reaction mixture was stirred at room temperature. After stirring for 24 h, the reaction mixture was extracted with CH₂Cl₂ (15 mL × 3), dried over MgSO₄, filtered and concentrated. The residue was purified with silica gel chromatography to provide pure product 3.

Charaterization data of compounds 3

3,3,3-Trifluoro-1-phenylpropan-1-one (3a): 90% yield, colorless liquid. ¹H NMR (300 MHz, CDCl₃/TMS): δ 7.94 (d, J = 7.4 Hz, 2H), 7.67–7.61 (m, 1H), 7.54–7.49 (m, 2H), 3.80 (q, J = 10.0 Hz, 2H); ¹⁹F NMR (282 MHz , CDCl₃/CFCI₃): δ − 61.8 (t, J = 10.0 Hz, 3F); MS (EI, m/z): 188(M⁺, 23.05), 105 (100.00), 77 (66.29). The data are consistent with the previous report[2].

3,3,3-Trifluoro-1-p-tolylpropan-1-one (3b): 91% yield, colorless solid. ¹H NMR (300 MHz, CDCl₃/TMS): δ 7.84 (d, J = 7.8 Hz, 2H), 7.30 (d, J = 7.8 Hz, 2H), 3.77 (q, J = 10.0 Hz, 2H), 2.44 (s, 3H); ¹⁹F NMR (282 MHz , CDCl₃/CFCI₃): δ − 61.8 (t, J = 10.0 Hz, 3F); MS (EI, m/z): 202(M⁺, 23.47), 119 (100.00), 91(63.09); The data are consistent with the previous report[3].

3,3,3-Trifluoro-1-(4-methoxyphenyl)propan-1-one (3c): 90% yield, yellow liquid. ¹H NMR (300 MHz, CDCl₃/TMS): δ 7.92 (d, J = 8.9 Hz, 2H),
6.97 (d, J = 8.9 Hz, 2H), 3.89 (s, 3H), 3.74 (q, J = 10.1 Hz, 2H); 19F NMR (282 MHz, CDCl3/CFCl3): δ – 61.7 (t, J = 10.1 Hz, 3F); MS (EI, m/z): 218(M+, 24.57), 135 (100.00); The data are consistent with the previous report[2].

3,3,3-Trifluoro-1-(4-fluorophenyl)propan-1-one (3d): 89% yield, yellow liquid. 1H NMR (300 MHz, CDCl3/TMS): δ 7.98 (dd, J1 = 7.4 Hz, J2 = 5.5 Hz, 2H), 7.19 (t, J = 8.1 Hz, 2H), 3.77 (q, J = 9.9 Hz, 2H); 19F NMR (282 MHz, CDCl3/CFCl3): δ – 61.7 (t, J = 9.9 Hz, 3F), –102.6 (m, 1F); MS (EI, m/z): 206(M+, 14.50), 123 (100.00), 95 (59.39); The data are consistent with the previous report[2].

3,3,3-Trifluoro-1-(2-methoxyphenyl)propan-1-one (3e): 88% yield, colorless solid. 1H NMR (300 MHz, CDCl3/TMS): δ 7.82 (dd, J1 = 7.8 Hz, J2 = 1.6 Hz, 1H), 7.53 (td, J1 = 7.8 Hz, J2 = 1.8 Hz, 1H), 7.04 (t, J = 7.9 Hz, 1H), 6.99 (d, J = 8.4 Hz, 1H), 3.95 (s, 3H), 3.88 (q, J = 10.3 Hz, 2H); 19F NMR (282 MHz, CDCl3/CFCl3): δ – 61.8 (t, J = 10.3 Hz, 3F); MS (EI, m/z): 218(M+, 15.92), 135 (100.00); The data are consistent with the previous report[4].

1-(4-Chlorophenyl)-3,3,3-trifluoropropan-1-one (3f): 86% yield, white solid. 1H NMR (300 MHz, CDCl3/TMS): δ 7.88 (d, J = 8.5 Hz, 2H), 7.49 (d, J = 8.5 Hz, 2H), 3.77 (q, J = 9.9 Hz, 2H); 19F NMR (282 MHz, CDCl3/CFCl3): δ – 61.7 (t, J = 9.9 Hz, 3F); MS (EI, m/z): 222(M+, 22.36), 139 (100.00), 111 (52.26); The data are consistent with the previous report[2].

3,3,3-Trifluoro-1-m-tolylpropan-1-one (3g): 84% yield, yellow liquid. 1H NMR (300 MHz, CDCl3/TMS): δ 7.75–7.71 (m, 2H), 7.46–7.39 (m, 2H), 3.78 (q, J = 10.0 Hz, 2H), 2.43 (s, 3H); 13C NMR (100 MHz, CDCl3/TMS): δ 189.3, 158.2, 133.4, 128.4, 125.9, 124.2 (q, J = 277.1 Hz), 42.0 (q, J = 28.2 Hz), 35.2, 31.0; 19F NMR (282 MHz, CDCl3/CFCl3): δ – 62.0 (t, J = 10.0 Hz, 3F); MS (EI, m/z): 202(M+, 27.87), 119 (100.00), 91 (66.96); The data are consistent with the previous report[5].

1-(4-Tert-butylphenyl)-3,3,3-trifluoropropan-1-one (3h): 84% yield, yellow liquid. 1H NMR (300 MHz, CDCl3/TMS): δ 7.88 (d, J = 8.4 Hz, 2H), 7.52 (d, J = 8.4 Hz, 2H), 3.77 (q, J = 10.1 Hz, 2H), 1.35 (s, 9H); 13C NMR (100 MHz, CDCl3/TMS): δ 189.3, 158.2, 133.4, 128.4, 125.9, 124.2 (q, J = 277.1 Hz), 42.0 (q, J = 28.2 Hz), 35.2, 31.0; 19F NMR (282 MHz, CDCl3/CFCl3): δ – 61.8 (t, J = 10.1 Hz, 3F); IR (film): 2967, 2909, 2873, 1698, 1606, 1567, 1466, 1417, 1371, 1339, 1271, 1257, 1235, 1197,
3,3,3-Trifluoro-1-(2-fluorophenyl)propan-1-one (3i): 80% yield, white solid. 

\[
\text{MP} = 68-70°C. \quad \text{H} NMR (300 MHz, CDCl}_3/TMS): \delta 7.94 (t, J = 7.6 \text{ Hz}, 1H), 7.61 (dd, J = 13.8 \text{ Hz}, J = 6.7 \text{ Hz}, 1H), 7.28 (d, J = 7.9 \text{ Hz}, 1H), 7.18 (dd, J = 12.8 \text{ Hz}, 5J = 8.5 \text{ Hz}, 1H), 3.84 (qd, J = 9.8 \text{ Hz}, J = 2.0 \text{ Hz}, 2H); \quad \text{C} NMR (100 MHz, CDCl}_3/TMS): \delta 187.6, 162.1 (d, J = 254.2 \text{ Hz}), 162.1 (d, J = 9.2 \text{ Hz}), 135.9 (d, J = 1.9 \text{ Hz}), 124.9 (d, J = 3.2 \text{ Hz}), 124.4 (d, J = 11.4 \text{ Hz}, J = 2.0 \text{ Hz}, 2H), 123.8 (qd, J = 276.8 \text{ Hz}, J = 2.8 \text{ Hz}), 116.80 (d, J = 23.8 \text{ Hz}), 46.60 (qd, J = 28.4 \text{ Hz}, J = 9.7 \text{ Hz}); \quad \text{F} NMR (282 MHz, CDCl}_3/CFCl}_3): \delta -62.4 (td, J = 9.8 \text{ Hz}, J = 1.6 \text{ Hz}, 3F), -109.5 (m, 1F); \quad \text{IR (film): 3370, 2987, 2956, 2924, 1695, 1653, 1610, 1579, 1484, 1457, 1419, 1379, 1288, 1278, 1262, 1217, 1161, 1117, 1104, 1044, 1000, 966, 920, 860, 839, 797, 769, 674, 618, 592 cm}^{-1}; \quad \text{MS (EI, m/z): 206(M^+, 14.85), 123 (100.00); HRMS (EI): exact mass calcd for C}_9\text{H}_6\text{O}_2\text{F}_3 (M^+): 206.0355, found: 206.0354.}

3,3,3-Trifluoro-1-(3-methoxyphenyl)propan-1-one (3j): 80% yield, colorless liquid. 

\[
\text{H} NMR (300 MHz, CDCl}_3/TMS): \delta 7.54–7.35 (m, 3H), 7.18 (d, J = 8.7 \text{ Hz}, 1H), 3.87 (s, 3H), 3.78 (q, J = 10.0 \text{ Hz}, 2H); \quad \text{C} NMR (100 MHz, CDCl}_3/TMS): \delta 189.5, 160.1, 137.2, 129.9, 124.0 (q, J = 277.0 \text{ Hz}), 121.0, 120.7, 112.6, 55.5, 42.2 (q, J = 28.2 Hz); \quad \text{F} NMR (282 MHz, CDCl}_3/CFCl}_3): \delta -61.8 (t, J = 10.0 \text{ Hz}, 3F); \quad \text{IR (film): 3079, 2946, 2841, 1699, 1599, 1585, 1488, 1466, 1454, 1432, 1417, 1372, 1337, 1259, 1206, 1179, 1128, 1104, 1049, 1023, 927, 873, 853, 778, 735, 684, 619 cm}^{-1}; \quad \text{MS (EI, m/z): 218(M^+, 43.71), 135 (100.00); HRMS (EI): exact mass calcd for C}_10\text{H}_9\text{O}_2\text{F}_3 (M^+): 218.0555, found: 218.0556.}

1-(3,4-Dimethoxyphenyl)-3,3,3-trifluoropropan-1-one (3k): 83% yield, colorless solid. 

\[
\text{MP} = 100–102°C. \quad \text{H} NMR (300 MHz, CDCl}_3/TMS): \delta 7.70–7.41 (m, 2H), 6.92 (d, J = 8.1 Hz, 1H), 3.97 (s, 3H), 3.95 (s, 3H), 3.76 (q, J = 10.1 Hz, 2H); \quad \text{C} NMR (100 MHz, CDCl}_3/TMS): \delta 188.2, 154.3, 149.4, 129.1, 124.2 (q, J = 276.9 Hz), 123.4, 110.2, 110.1, 56.1, 56.0, 41.6 (q, J = 28.1 Hz); \quad \text{F} NMR (282 MHz, CDCl}_3/CFCl}_3): \delta –61.7 (t, J = 10.1 Hz, 3F); \quad \text{IR (film): 3082, 2975, 2946, 2849, 1686, 1587, 1519, 1470, 1456, 1443, 1424, 1362, 1284, 1262, 1208, 1172, 1157, 1103, 1020, 933, 912, 873, 853, 800, 784, 765, 659, 635, 625, 585 cm}^{-1}; \quad \text{MS (EI, m/z): 248(M^+, 28.65), 165 (100.00); HRMS (EI): exact mass calcd for C}_11\text{H}_11\text{O}_3\text{F}_3 (M^+): 248.0660, found: 248.0663.}
1-(4-Bromophenyl)-3,3,3-trifluoropropan-1-one (3l): 78% yield, colorless solid. $^1$H NMR (300 MHz, CDCl$_3$/TMS): $\delta$ 7.80 (d, $J = 8.2$ Hz, 2H), 7.66 (d, $J = 8.2$ Hz, 2H), 3.76 (q, $J = 9.9$ Hz, 2H); $^{19}$F NMR (282 MHz, CDCl$_3$/CFCl$_3$): $\delta$ – 61.8 (t, $J = 9.9$ Hz, 3F); MS (EI, m/z): 266(M$^+$, 17.80), 183 (100.00); The data are consistent with the previous report[2].

3,3,3-Trifluoro-1-(4-(trifluoromethyl)phenyl)propan-1-one (3m): 74% yield. colorless solid. Mp = 68–71$^\circ$C. $^1$H NMR (300 MHz, CDCl$_3$/TMS): $\delta$ 8.05 (d, $J = 7.7$ Hz, 2H), 7.79 (d, $J = 7.7$ Hz, 2H), 3.84 (q, $J = 9.8$ Hz, 2H); 13C NMR (100 MHz, CDCl$_3$/TMS): $\delta$ 188.9 (d, $J = 2.5$ Hz), 138.4, 135.5 (q, $J = 32.9$ Hz), 128.7, 126.0 (q, $J = 3.6$ Hz), 123.7 (q, $J = 277.1$ Hz), 123.3 (q, $J = 274.2$ Hz), 42.4 (q, $J = 28.7$ Hz); $^{19}$F NMR (282 MHz, CDCl$_3$/CFCl$_3$): $\delta$ – 61.8 (t, $J = 9.9$ Hz, 3F), – 63.1 (s, 3F); IR (film): 3068, 2956, 1705, 1584, 1515, 1480, 1278, 1229, 1167, 1137, 1101, 1065, 1001, 920, 858, 834, 770, 696, 631, 609 cm$^{-1}$; MS (EI, m/z): 256(M$^+$, 4.81), 173 (100.00), 145 (67.58); HRMS (EI): exact mass calcd for C$_{10}$H$_6$OF$_6$ (M$^+$): 256.0323, found: 256.0326.

1-(Biphenyl-4-yl)-3,3,3-trifluoropropan-1-one (3n): 68% yield, colorless solid. Mp = 135–137$^\circ$C. $^1$H NMR (300 MHz, CDCl$_3$/TMS): $\delta$ 8.01 (d, $J = 8.5$ Hz, 2H), 7.74–7.71 (m, 2H), 7.65–7.62 (m, 2H), 7.51–7.42 (m, 3H), 3.83 (q, $J = 10.1$ Hz, 2H); 13C NMR (100 MHz, CDCl$_3$/TMS): $\delta$ 189.3, 146.9, 139.4, 134.6, 129.1, 129.0, 128.6, 127.5, 127.3, 124.1 (q, $J = 277.0$ Hz), 42.2 (q, $J = 28.4$ Hz); $^{19}$F NMR (282 MHz, CDCl$_3$/CFCl$_3$): $\delta$ – 61.8 (t, $J = 10.0$ Hz, 3F); IR (film): 3067, 2966, 2938, 1686, 1651, 1604, 1582, 1559, 1451, 1416, 1371, 1277, 1228, 1198, 1138, 1102, 1023, 999, 968, 914, 853, 841, 822, 767, 749, 724, 698, 664, 618, 594 cm$^{-1}$; MS (EI, m/z): 264(M$^+$, 27.02), 181 (100.00), 152 (62.39); HRMS (EI): exact mass calcd for C$_{15}$H$_{11}$OF$_3$ (M$^+$): 264.0762, found: 264.0763.

3,3,3-Trifluoro-1-(5-methylthiophen-2-yl)propan-1-one (3o): 70% yield, colorless solid. Mp = 55–58$^\circ$C. $^1$H NMR (300 MHz, CDCl$_3$/TMS): $\delta$ 7.55 (d, $J = 25.3$ Hz, 1H), 6.85 (d, $J = 3.7$ Hz, 2H), 6.85 (d, $J = 3.7$ Hz, 1H), 3.65 (d, $J = 10.2$ Hz, 2H), 2.56 (s, 3H); 13C NMR (100 MHz, CDCl$_3$/TMS): $\delta$ 181.6, 152.2, 141.0, 134.2, 127.3, 123.8 (q, $J = 277.3$ Hz), 42.6 (q, $J = 28.6$ Hz), 16.1; $^{19}$F NMR (282 MHz, CDCl$_3$/CFCl$_3$): $\delta$ – 62.4 (t, $J = 10.2$ Hz, 3F); IR (film): 3099, 2918, 2849, 1667, 1538, 1456, 1372, 1343, 1242, 1130, 1068, 1008, 960, 944, 919, 851, 805, 783, 635, 618, 599 cm$^{-1}$; MS (EI, m/z): 208(M$^+$, 21.31), 125 (100.00); HRMS (EI): exact mass calcd for C$_8$H$_7$OSF$_3$ (M$^+$): 208.0170, found: 208.0167.
3,3,3-Trifluoro-1-(5-methylfuran-2-yl)propan-1-one (3p): 72% yield, colorless solid. Mp = 57−59°C. $^1$H NMR (300 MHz, CDCl$_3$/TMS): δ 7.23 (d, $J = 3.4$ Hz, 1H), 6.24 (d, $J = 3.4$ Hz, 1H), 3.61 (q, $J = 10.3$ Hz, 2H), 2.42 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$/TMS): 177.2, 159.3, 150.8, 123.9 (q, $J = 277.1$ Hz), 121.0, 109.9, 41.9 (q, $J = 28.7$ Hz), 14.0; $^{19}$F NMR (282 MHz, CDCl$_3$/CFCl$_3$): δ −62.4 (t, $J = 10.3$ Hz, 3F); IR (film): 3127, 2960, 1752, 1674, 1589, 1515, 1415, 1379, 1267, 1212, 1144, 1107, 1054, 1031, 963, 917, 851, 795, 736, 637, 623 cm$^{-1}$; MS (EI, $m/z$): 192(M$^+$, 29.85), 109 (100.00); HRMS (EI): exact mass calcd for C$_8$H$_7$O$_2$F$_3$(M$^+$): 192.0398, found: 192.0400.

**Screens for the Trifluoromethyldecarboxylation reaction of 4a.** A Schlenk test tube with a magnetic stirring bar and a reflux condensing tube was charged with 1, 4a, and catalyst followed by solvent. The reaction mixture was stirred and heated for 12 h. The reaction mixture was cooled to ambient temperature, extracted with CH$_2$Cl$_2$ (15 mL $\times$ 3) and PhOCF$_3$ was added. The yield was determined by $^{19}$F NMR. The results are summarized in Table S-1.

Table S2. Screens for the Trifluoromethyldecarboxylation reaction of 4a
### General procedure for the trifluoromethylative decarboxylation reaction of 4 (for Table 3 in manuscript)

General procedure for 4a-4g: Into a reaction flask equipped with a magnetic stirring bar and a reflux condenser, was added 1 (2.4 mmol, 3.0 equiv), 4a (0.8 mmol, 1.0 equiv), copper(II) gluconate (1.6 equiv) toluene (1.0 mL), and copper(II) gluconate (1.0 equiv) toluene (1.0 mL). The reaction mixture was heated at 80 °C for 24 h. After cooling to room temperature, the mixture was diluted with ethyl acetate and washed with water. The organic layer was dried over sodium sulfate and concentrated in vacuo. The crude product was purified by column chromatography on silica gel to afford the desired product.

### Supporting Information

<table>
<thead>
<tr>
<th>entry</th>
<th>1(equiv)</th>
<th>solvent&lt;sup&gt;a&lt;/sup&gt;</th>
<th>metal (equiv)</th>
<th>T (°C)</th>
<th>yield (%)&lt;sup&gt;b&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2.0</td>
<td>H&lt;sub&gt;2&lt;/sub&gt;O/CCl&lt;sub&gt;4&lt;/sub&gt; (3:2)</td>
<td>CuF&lt;sub&gt;2&lt;/sub&gt;·2H&lt;sub&gt;2&lt;/sub&gt;O (2.0)</td>
<td>80</td>
<td>29</td>
</tr>
<tr>
<td>2</td>
<td>2.0</td>
<td>H&lt;sub&gt;2&lt;/sub&gt;O/CCl&lt;sub&gt;4&lt;/sub&gt; (3:2)</td>
<td>Cu(OH)&lt;sub&gt;2&lt;/sub&gt; (2.0)</td>
<td>80</td>
<td>24</td>
</tr>
<tr>
<td>3</td>
<td>2.0</td>
<td>H&lt;sub&gt;2&lt;/sub&gt;O/CCl&lt;sub&gt;4&lt;/sub&gt; (3:2)</td>
<td>Cu(HCOO)&lt;sub&gt;2&lt;/sub&gt;·4H&lt;sub&gt;2&lt;/sub&gt;O (2.0)</td>
<td>80</td>
<td>30</td>
</tr>
<tr>
<td>4</td>
<td>2.0</td>
<td>H&lt;sub&gt;2&lt;/sub&gt;O/CCl&lt;sub&gt;4&lt;/sub&gt; (3:2)</td>
<td>Bis(2,4-pentanedionato)copper (II)(2.0)</td>
<td>80</td>
<td>44</td>
</tr>
<tr>
<td>5</td>
<td>2.0</td>
<td>H&lt;sub&gt;2&lt;/sub&gt;O/CCl&lt;sub&gt;4&lt;/sub&gt; (3:2)</td>
<td>copper(II) gluconate (2.0)</td>
<td>80</td>
<td>45</td>
</tr>
<tr>
<td>6</td>
<td>2.0</td>
<td>H&lt;sub&gt;2&lt;/sub&gt;O/DMF(3:2)</td>
<td>copper(II) gluconate (2.0)</td>
<td>80</td>
<td>22</td>
</tr>
<tr>
<td>7</td>
<td>2.0</td>
<td>H&lt;sub&gt;2&lt;/sub&gt;O/DCE(3:2)</td>
<td>copper(II) gluconate (2.0)</td>
<td>80</td>
<td>32</td>
</tr>
<tr>
<td>8</td>
<td>2.0</td>
<td>H&lt;sub&gt;2&lt;/sub&gt;O/Dioxane(3:2)</td>
<td>copper(II) gluconate (2.0)</td>
<td>80</td>
<td>43</td>
</tr>
<tr>
<td>9</td>
<td>2.0</td>
<td>H&lt;sub&gt;2&lt;/sub&gt;O/CH&lt;sub&gt;2&lt;/sub&gt;Br&lt;sub&gt;2&lt;/sub&gt;(3:2)</td>
<td>copper(II) gluconate (2.0)</td>
<td>80</td>
<td>40</td>
</tr>
<tr>
<td>10</td>
<td>2.0</td>
<td>H&lt;sub&gt;2&lt;/sub&gt;O/Dioxane(3:2)</td>
<td>copper(II) gluconate (2.0)</td>
<td>80</td>
<td>40</td>
</tr>
<tr>
<td>11</td>
<td>2.0</td>
<td>H&lt;sub&gt;2&lt;/sub&gt;O/CH&lt;sub&gt;2&lt;/sub&gt;CN(3:2)</td>
<td>copper(II) gluconate (2.0)</td>
<td>80</td>
<td>27</td>
</tr>
<tr>
<td>12</td>
<td>2.0</td>
<td>H&lt;sub&gt;2&lt;/sub&gt;O/DMSO(3:2)</td>
<td>copper(II) gluconate (2.0)</td>
<td>80</td>
<td>26</td>
</tr>
<tr>
<td>13</td>
<td>2.0</td>
<td>H&lt;sub&gt;2&lt;/sub&gt;O/DME(3:2)</td>
<td>copper(II) gluconate (2.0)</td>
<td>80</td>
<td>14</td>
</tr>
<tr>
<td>14</td>
<td>2.0</td>
<td>H&lt;sub&gt;2&lt;/sub&gt;O/CCl&lt;sub&gt;4&lt;/sub&gt; (3:2)</td>
<td>copper(II) gluconate (1.0)</td>
<td>80</td>
<td>38</td>
</tr>
<tr>
<td>15</td>
<td>2.0</td>
<td>H&lt;sub&gt;2&lt;/sub&gt;O/CCl&lt;sub&gt;4&lt;/sub&gt; (3:2)</td>
<td>copper(II) gluconate (0.5)</td>
<td>80</td>
<td>10</td>
</tr>
<tr>
<td>16</td>
<td>3.0</td>
<td>H&lt;sub&gt;2&lt;/sub&gt;O/CCl&lt;sub&gt;4&lt;/sub&gt; (3:2)</td>
<td>copper(II) gluconate (2.0)</td>
<td>80</td>
<td>58</td>
</tr>
<tr>
<td>17</td>
<td>3.0</td>
<td>H&lt;sub&gt;2&lt;/sub&gt;O/CCl&lt;sub&gt;4&lt;/sub&gt; (3:2)</td>
<td>copper(II) gluconate (2.0)</td>
<td>60</td>
<td>53</td>
</tr>
<tr>
<td>18</td>
<td>3.0</td>
<td>H&lt;sub&gt;2&lt;/sub&gt;O/CCl&lt;sub&gt;4&lt;/sub&gt; (2:1)</td>
<td>copper(II) gluconate (2.0)</td>
<td>80</td>
<td>56</td>
</tr>
<tr>
<td>19</td>
<td>3.0</td>
<td>H&lt;sub&gt;2&lt;/sub&gt;O/CCl&lt;sub&gt;4&lt;/sub&gt; (1:4)</td>
<td>copper(II) gluconate (2.0)</td>
<td>80</td>
<td>37</td>
</tr>
</tbody>
</table>

<sup>a</sup>The data in the parentheses refers to the volume ratio. <sup>b</sup>Determined by <sup>19</sup>F NMR spectroscopy using PhOCF<sub>3</sub> as an internal standard.
mmol, 2.0 equiv), CCl₄ (3 mL), and H₂O (4.5 mL). The reaction mixture was stirred and heated to 80 °C. After stirring at this temperature for 12 h, the reaction mixture was cooled to ambient temperature and extracted with CH₂Cl₂ (15 mL × 3), dried over MgSO₄, filtered and concentrated under vacuum. The residue was purified with silica gel chromatography to provide pure product.

General procedure for 4h-4j: Into a reaction flask equipped with a magnetic stirring bar and a reflux condenser, was added 1 (2.4 mmol, 3.0 equiv), 4h (0.8 mmol, 1.0 equiv), copper(II) gluconate (1.6 mmol, 2.0 equiv), DMSO (3 mL), and H₂O (4.5 mL). The reaction mixture was stirred and heated to 80 °C. After stirring at this temperature for 12 h, the reaction mixture was cooled to ambient temperature and extracted with ether (15 mL × 3), dried over MgSO₄, filtered and concentrated under vacuum. The residue was purified with silica gel chromatography to provide pure product.

Characterization data of compounds 5

1,1,1-Trifluoro-5-phenylpentan-3-one (5a): 54% yield, colorless liquid. $^1$H NMR (300 MHz, CDCl₃/TMS): δ 7.32-7.16 (m, 5H), 3.19 (q, $J = 10.4$Hz, 2H), 2.99-2.78 (m, 4H); $^{19}$F NMR (282 MHz, CDCl₃/CFCl₃): δ − 62.3 (t, $J = 10.3$Hz, 3F); MS (EI, $m/z$): 216(M⁺, 64.71), 105 (100.00); The data are consistent with the previous report[6]

4,4,4-Trifluoro-1-phenylbutan-2-one (5b): 50% yield, colorless liquid. $^1$H NMR (300 MHz, CDCl₃/TMS): δ 7.39-7.30 (m, 3H), 7.20 (d, $J = 7.0$Hz, 2H), 3.80 (s, 2H), 3.23 (q, $J = 10.3$Hz, 2H); $^{19}$F NMR (282 MHz, CDCl₃/CFCl₃): δ − 62.4 (t, $J = 10.3$Hz, 3F); MS (EI, $m/z$): 202(M⁺, 22.22), 91 (100.00); The data are consistent with the previous report[7]

1,1,1-Trifluoro-6-phenylhexan-3-one (5c): 40% yield, colorless liquid. $^1$H
NMR (300 MHz, CDCl3/TMS): δ 7.32-7.14 (m, 5H), 3.17 (q, J = 10.5Hz, 2H), 2.64 (t, J = 7.5Hz, 2H), 2.53 (t, J = 7.2Hz, 2H), 1.99-1.89 (m, 2H); 13C NMR (100 MHz, CDCl3/TMS): δ 199.9, 141.1, 128.5, 128.4, 126.1, 123.6 (q, J = 276.9Hz), 46.2 (q, J = 28.1Hz), 42.5 (q, J = 1.9Hz), 34.6, 24.5; 19F NMR (282 MHz, CDCl3/CFCl3): δ – 62.4 (t, J = 10.4 Hz, 3F); IR (film): 3443, 3087, 3064, 3029, 2940, 2864, 1732, 1604, 1497, 1455, 1418, 1375, 1271, 1153, 1099, 1016, 911, 735, 701, 649 cm⁻¹; MS (EI, m/z): 230(M⁺, 5.82), 104 (100.00); HRMS (EI): exact mass calcd for C12H13OF3 (M⁺): 230.0918, found: 230.0916.

1-Cyclohexyl-3,3,3-trifluoropropan-1-one (5d): 51% yield, colorless liquid. 1H NMR (300 MHz, CDCl3/TMS): δ 3.26 (q, J = 10.3Hz, 2H), 2.41 (t, J = 9.0Hz, 1H), 2.00-1.08 (m, 10H); 19F NMR (282 MHz, CDCl3/CFCl3): δ – 62.4 (t, J = 10.3Hz, 10F); MS (EI, m/z): 194(M⁺, 20.30), 83 (100.00); The data are consistent with the previous report[6]

2-(4,4,4-Trifluoro-2-oxobutyl)isoindoline-1,3-dione (5e): 50% yield, colorless solid. Mp = 166−168°C. 1H NMR (300 MHz, CDCl3/TMS): δ 7.93-7.85 (m, 2H), 7.81-7.72 (m, 2H), 4.59 (s, 2H), 3.39 (q, J = 10.2Hz, 2H); 13C NMR (100 MHz, CDCl3/TMS): δ 192.2, 167.3, 134.4, 131.8, 123.7, 123.2 (q, J = 277.2Hz), 46.7 (q, J = 2.6Hz), 44.2 (q, J = 29.6Hz); 19F NMR (282 MHz, CDCl3/CFCl3): δ – 61.9(t, J = 10.2Hz, 3F); IR (film): 3484, 2981, 2950, 1786, 1743, 1709, 1618, 1422, 1376, 1357, 1329, 1279, 1193, 1154, 1077, 1055, 1017, 960, 909, 874, 845, 799, 750, 716, 708, 661, 600 cm⁻¹; MS (EI, m/z): 271(M⁺, 1.66), 160 (100.00); HRMS (EI): exact mass calcd for C12H8NO3F3 (M⁺): 271.0456, found: 271.0451.

1,1,1-Trifluorooctan-3-one (5g): 45% yield, yellow oil. 1H NMR (300 MHz, CDCl3/TMS): δ 3.21 (q, J = 10.5Hz, 2H), 2.53 (t, J = 7.3Hz, 2H), 1.66-1.56 (m, 2H), 1.34-1.25 (m, 4H), 0.90 (t, J = 6.8Hz, 3H); 19F NMR (282 MHz, CDCl3/CFCl3): δ – 62.4(t, J = 10.6Hz, 3F); MS (EI, m/z): 182(M⁺, 3.20), 126(100.00);The data are consistent with the previous report[8]

1,1,1-Trifluoroundecan-3-one (5h): 49% yield, colorless oil. 1H NMR (300 MHz, CDCl3/TMS): δ 3.22 (q, J = 10.5Hz, 2H), 2.52 (t, J = 7.3Hz, 2H), 1.61-1.58 (m, 2H), 1.36-1.19 (m, 10H), 0.88 (t, J = 6.5 Hz, 3H); 19F NMR (282 MHz, CDCl3/CFCl3): δ – 62.4(t, J = 10.5Hz, 3F); MS (EI, m/z): 224(M⁺, 5.13), 126 (88.04), 110 (100.00); The data are consistent with the previous report[6]
1-(Bicyclo[2.2.1]heptan-2-yl)-3,3,3-trifluoropropan-1-one (5i): 50% yield, yellow oil. $^1$H NMR (300 MHz, CDCl$_3$/TMS): $\delta$ 3.31-3.15 (m, 2H), 2.93-2.88 (m, 0.68H), 2.63 (br, 0.68H), 2.52-2.46 (m, 0.68H), 2.33-2.28 (m, 1H), 1.83-1.77 (m, 1H), 1.61-1.10 (m, 7H); $^{13}$C NMR (100 MHz, CDCl$_3$/TMS): $\delta$ 201.22 (q, $J = 2.1$ Hz), 202.18 (q, $J = 2.0$ Hz), 123.8 (q, $J = 276.9$ Hz), 123.7 (q, $J = 276.9$ Hz), 54.7 (q, $J = 1.6$ Hz), 54.6 (q, $J = 1.6$ Hz), 45.2 (q, $J = 27.6$ Hz), 44.7 (q, $J = 27.8$ Hz), 40.6, 40.2, 39.7, 37.1, 36.0, 35.8, 32.1, 29.6, 29.5, 28.9, 28.6, 24.3; $^{19}$F NMR (282 MHz, CDCl$_3$/CFCl$_3$): $\delta$ – 62.30 (t, $J = 10.2$ Hz, 1.78F), 62.32 (t, $J = 10.1$ Hz, 1.22F); IR (film): 2959, 2876, 1726, 1455, 1415, 1371, 1265, 1159, 1125, 1098, 1056, 954, 911, 850, 805, 735 cm$^{-1}$; MS (EI, $m/z$): 206(M+, 3.01), 95 (100.00); HRMS (EI): exact mass calcd for C$_{10}$H$_{13}$OF$_3$ (M$^+$): 206.0918, found: 206.0914.

1-Cyclopentyl-4,4,4-trifluorobutan-2-one (5j): 46% yield, yellow oil. $^1$H NMR (300 MHz, CDCl$_3$/TMS): $\delta$ 3.21 (q, $J = 10.5$ Hz, 2H), 2.55 (d, $J = 7.1$ Hz, 2H), 2.39-2.16 (m, 1H), 1.89-1.79 (m, 2H), 1.68-1.49 (m, 4H), 1.14-1.02 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$/TMS): $\delta$ 200.0 (q, $J = 2.1$ Hz), 123.6 (q, $J = 276.9$ Hz), 49.7 (q, $J = 1.8$ Hz), 46.1 (q, $J = 28.0$ Hz), 34.9, 32.3, 24.8; $^{19}$F NMR (282 MHz, CDCl$_3$/CFCl$_3$): $\delta$ – 62.4 (t, $J = 10.6$ Hz, 3F); IR (film): 2956, 2872, 1731, 1454, 1420, 1366, 1263, 1148, 1114, 1039, 912, 848, 805, 645 cm$^{-1}$; MS (EI, $m/z$): 194(M$^+$, 0.63), 68 (100.00); HRMS (EI): exact mass calcd for C$_9$H$_{13}$OF$_3$ (M$^+$): 194.0918, found: 194.0914.

5-(4-Tert-butylphenyl)-1,1,1-trifluoro-4-methylpentan-3-one (5k): 63% yield, colorless oil. $^1$H NMR (300 MHz, CDCl$_3$/TMS): $\delta$ 7.31 (d, $J = 8.0$ Hz, 2H), 7.07 (d, $J = 8.0$ Hz, 2H), 3.27-2.80 (m, 4H), 2.62-2.56 (m, 1H), 1.30 (s, 9H), 1.13 (d, $J = 6.4$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$/TMS): $\delta$ 203.6, 149.5, 135.7, 128.6, 125.5, 123.8 (q, $J = 276.9$ Hz), 48.7 (q, $J = 1.5$ Hz), 44.9 (q, $J = 27.8$ Hz), 38.4, 34.4, 31.3, 15.8; $^{19}$F NMR (282 MHz, CDCl$_3$/CFCl$_3$): $\delta$ – 62.4 (t, $J = 10.2$ Hz, 3F); IR (film): 2966, 2872, 1730, 1510, 1460, 1412, 1367, 1268, 1159, 1118, 1024, 838, 805, 628, 567 cm$^{-1}$; MS (EI, $m/z$): 286(M$^+$, 19.38), 271(100.00); HRMS (EI): exact mass calcd for C$_{16}$H$_{21}$OF$_3$ (M$^+$): 286.1545, found: 286.1547.

1-(Adamantan-1-yl)-3,3,3-trifluoropropan-1-one (5l): 60% yield, colorless solid. Mp = 60–62°C. $^1$H NMR (300 MHz, CDCl$_3$/TMS): $\delta$ 3.28 (q, $J = 10.1$ Hz, 2H), 2.07 (br, 3H), 1.80-1.67 (m, 12H); $^{13}$C NMR (100 MHz, CDCl$_3$/TMS): $\delta$ 204.8 (q, $J = 1.6$ Hz), 124.2 (q, $J = 276.8$ Hz), 46.9 (q, $J = 1.5$ Hz), 39.3 (q, $J = 27.6$ Hz), 37.5, 36.2, 27.6; $^{19}$F NMR (282 MHz, CDCl$_3$/CFCl$_3$): $\delta$ – 62.2 (t, $J = 10.1$ Hz, 3F); IR (film): 2962, 2910, 2854, 1716, 1453,
1413, 1365, 1317, 1260, 1201, 1176, 1096, 1014, 937, 910, 871, 850, 802, 736, 620 cm\(^{-1}\); MS (EI, \(m/z\)): 246\((M^+, 1.77)\), 135(100.00); HRMS (EI): exact mass calcd for \(\text{C}_{13}\text{H}_{17}\text{OF}_{3}\) (\(M^+\)): 246.1232, found: 246.1237.

The isotopic labeling experiment: A Schlenk test tube with a magnetic stirring bar was charged with 1 (0.8 mmol, 2.0 equiv), 2a (0.4 mmol, 1.0 equiv), Cu(OAc)\(_2\) (0.8 mmol), TMEDA (1.0 mmol), followed by DCM (2 mL) and \(\text{H}_2^{18}\text{O}\) (3 mL, with 97% abundance of \(^{18}\text{O}\)). The reaction mixture was stirred at room temperature. After stirring for 24 h, the reaction mixture was extracted with \(\text{CH}_2\text{Cl}_2\) (15 mL × 3), dried over MgSO\(_4\), filtered and concentrated. The residue was purified with silica gel chromatography to provide pure product 3a’ in 86% yield. The abundance of \(^{18}\text{O}\) in 3a’ was determined by EI-MS, which was shown in page 73.

Charaterization data of compound 3a’

\[\text{3,3,3-Trifluoro-1-phenylpropan-1-one (3a’): 86\% yield, colorless solid. Mp = 31-33\,^\circ\text{C}. I\text{H NMR (300 MHz, CDCl}_3/\text{TMS): }\delta 7.94 (d, J = 7.3 \,\text{Hz}, 2\text{H}), 7.64 (t, J = 7.3 \,\text{Hz}, 1\text{H}), 7.51 (t, J = 7.8 \,\text{Hz}, 2\text{H}), 3.80 (q, J = 10.0 \,\text{Hz}, 2\text{H}); I^{13}\text{C NMR (100 MHz, CDCl}_3/\text{TMS): }\delta 189.7 (q, J = 2.4 \,\text{Hz}), 135.7 (q, J = 1.7 \,\text{Hz}), 134.2, 128.9, 128.3, 124.0 (q, J = 276.9 \,\text{Hz}), 42.0 (q, J = 28.2 \,\text{Hz}); I^{19}\text{F NMR (282 MHz, CDCl}_3/\text{CFCl}_3): }\delta -62.1 (t, J = 10.0 \,\text{Hz}, 3\text{F}); MS (EI, \(m/z\)): 190(M^+, 36.34), 188 (M^-2, 4.73), 107 (100.00), 105 (18.78), 77 (56.46). HRMS (EI): exact mass calcd for \(\text{C}_{9}\text{H}_{7}^{18}\text{OF}_3\) (\(M^+\)): 190.0491, found: 190.0488.\]
Reference:

1H, 13C NMR and 19F spectra of all new products:
\[ ^{13}\text{C NMR} \]

COOH

4j
$^1$H NMR

![Chemical Structure](image)

3h
$^{13}$C NMR

MeO

OMe

3k
$^{19}$F NMR

![Chemical Structure: MeO-\(\text{CF}_3\)-OME-3k](image)

Electronic Supplementary Material (ESI) for Chemical Science
This journal is © The Royal Society of Chemistry 2013
$^{19}$F NMR

3o
$^{1}H$ NMR

$$\text{5c}$$

Electronic Supplementary Material (ESI) for Chemical Science
This journal is © The Royal Society of Chemistry 2013
Supporting Information

$^{13}$C NMR

![Chemical Structure Image]

Electronic Supplementary Material (ESI) for Chemical Science
This journal is © The Royal Society of Chemistry 2013
$^{19}$F NMR

$5e$
$^{13}$C NMR

5j
$^1$H NMR

$^{18}$O

CF$_3$

3a'}