

Supporting Information

A facile process for the asymmetric synthesis of β -trifluoromethylated β -amino ketones via addition of ketone enolates to sulfinylimine

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Table of Contents

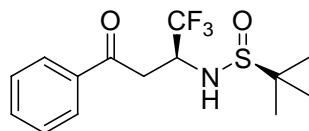
	Page
1. General information -----	2
2. Procedure for asymmetric addition of sulfinylimine -----	2
3. Reaction of large scale application study -----	9
4. X-ray crystallography for 3a -----	10
5. Conversion of 3a affording free β-amino ketone 4 -----	11
6. ¹H and ¹³C NMR spectra for compound 3 and 4 -----	12

1. General information

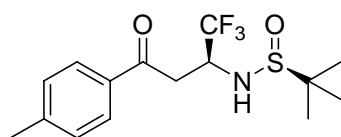
All imine addition reactions were performed in oven-dried vials under N₂ atmosphere. Solvent THF was dried and distilled prior to use. Sulfinylimine **1** was obtained from TOSOH F-TECH, INC.. LDA (2 M in THF) was from Aldrich. These and other chemicals were used as obtained from commercial sources without further purification. Flash chromatography was performed using silica gel 60 (200-300 mesh). Thin layer chromatography was carried out on silica gel 60 F-254 TLC plates of 20 cm × 20 cm. Melting points are uncorrected. IR spectra were collected on Bruker Vector 22 in KBr pellets. Values of optical rotation were measured on Rudolph Automatic Polarimeter A21101. ¹H and ¹³C NMR (TMS used as internal standard) spectra were recorded with a Bruker ARX 300 spectrometer and a Bruker ARX 500 spectrometer. ¹⁹F NMR spectra (referenced to external CF₃COOH) were recorded with a Bruker ARX 400 spectrometer. High resolution mass spectra for all the new compounds were done by Micromass Q-Tof instrument (ESI).

2. Typical procedure for asymmetric addition of sulfinylimine

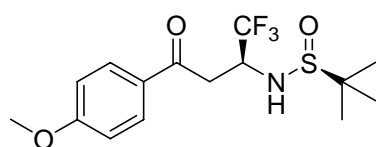
Into an oven-dried reaction vial flushed with N₂ were taken ketone (0.85 mmol) and anhydrous THF (3.0 mL). The reaction vial was cooled to -78 °C and LDA (2 M in THF, 0.47 mL) was added dropwise with stirring. After 40 min at -78 °C, sulfinylimine **1** (0.5 mmol) dissolved in anhydrous THF (2.0 mL) was added dropwise. Stirring was continued at -78 °C for 2 h, then the reaction was quenched with saturated NH₄Cl (3.0 mL), followed by H₂O (5.0 mL) and the mixture was brought to room temperature. The organic layer was taken and the aqueous layer was extracted with EtOAc (2 × 20 mL). The combined organic layers were dried with anhydrous Na₂SO₄, filtered and the solvent was removed to give the crude product, which was purified by TLC plate (hexane/EtOAc, 2:1).



(*S*)-2-methyl-*N*-((*S*)-1,1,1-trifluoro-4-oxo-4-phenylbutan-2-yl)propane-2-sulfinamide (**3a**): colorless solid, yield 84%, mp 146-148 °C, $[\alpha]_D^{25} +47.2$ ($c = 0.46$, CHCl₃). ¹H NMR (CDCl₃, 300 MHz): $\delta = 7.93$ -7.97 (m, 2 H), 7.56-7.62 (m, 1 H), 7.45-7.50 (m, 2 H), 4.49-4.63 (m, 1 H), 4.25 (d, $J = 8.7$ Hz, 1 H), 3.75 (dd, $J = 9.6, 17.7$ Hz, 1 H), 3.31 (dd, $J = 3.3, 17.7$ Hz, 1 H), 1.15 (s, 9 H). ¹³C NMR (CDCl₃, 75 MHz): $\delta = 194.9, 136.2, 133.8, 128.8, 128.2, 123.5$ (q, $J = 280.0$ Hz), 56.9, 53.8 (q, $J = 30.2$ Hz), 38.0, 22.3. ¹⁹F NMR (CDCl₃, 376 MHz): $\delta = -74.5$. IR (KBr): $\nu = 3277, 2966, 2925, 1685, 1292, 1269, 1168, 1121, 1060, 690, 753$ cm⁻¹. HRMS $[M+Na^+]$: calcd for C₁₄H₁₈O₂SNF₃Na: 344.0903, found: 344.0893.

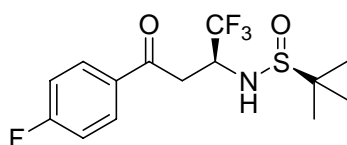


(*S*)-2-methyl-*N*-((*S*)-1,1,1-trifluoro-4-oxo-4-p-tolylbutan-2-yl)propane-2-sulfinamide (**3b**): colorless solid, yield 81%, mp 148-150 °C, $[\alpha]_D^{25} +36.1$ ($c = 0.17$, CHCl₃). ¹H NMR (CDCl₃, 300 MHz): $\delta = 7.87$ (d, $J = 6.9$ Hz, 2 H), 7.30 (d, $J = 5.7$ Hz, 2 H), 4.51-4.60 (m, 1 H), 4.07 (d, $J = 26.7$ Hz, 1 H), 3.71 (dd, $J = 9.3, 17.4$ Hz, 1 H), 3.29 (dd, $J = 2.7, 17.7$ Hz, 1 H), 2.43 (d, $J = 1.2$ Hz, 3 H), 1.16 (d, $J = 2.4$ Hz, 9 H). ¹³C NMR (CDCl₃, 75 MHz): $\delta = 194.4, 144.8, 133.8, 129.5, 128.3, 123.5$ (q, $J = 280.0$ Hz), 56.9, 53.9 (q, $J = 30.8$ Hz), 37.9, 22.3, 21.7. ¹⁹F NMR (CDCl₃, 376 MHz): $\delta = -74.5$. IR (KBr): $\nu = 3265, 2962, 2924, 1683, 1610, 1345, 1292, 1270, 1167, 1118, 1060, 804$ cm⁻¹. HRMS $[M+Na^+]$: calcd for C₁₅H₂₀O₂SNF₃Na: 358.1059, found: 358.1048.

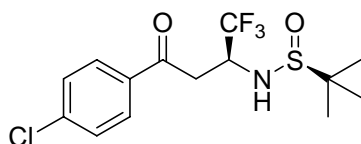


(*S*)-2-methyl-*N*-((*S*)-1,1,1-trifluoro-4-(4-methoxyphenyl)-4-oxobutan-2-yl)propane-2-

sulfinamide (**3c**): white solid, yield 84%, mp 99-100 °C, $[\alpha]_D^{25} +87.5$ ($c = 0.17$, CHCl₃). ¹H NMR (CDCl₃, 300 MHz): $\delta = 7.92$ -7.97 (m, 2 H), 6.94-6.98 (m, 2 H), 4.50-4.60 (m, 1 H), 4.12 (d, $J = 6.3$ Hz, 1 H), 3.88 (s, 3 H), 3.69 (dd, $J = 9.6, 17.4$ Hz, 1 H), 3.26 (dd, $J = 3.3, 17.4$ Hz, 1 H), 1.16 (s, 9 H). ¹³C NMR (CDCl₃, 75 MHz): $\delta = 193.3, 164.1, 130.5, 129.3, 123.5$ (q, $J = 279.6$ Hz), 114.0, 56.9, 55.5, 54.0 (q, $J = 30.9$ Hz), 37.6, 22.3. ¹⁹F NMR (CDCl₃, 376 MHz): $\delta = -74.5$. IR (KBr): $\nu = 3265, 2963, 2927, 1678, 1603, 1369, 1291, 1267, 1168, 1121, 1058, 820$ cm⁻¹. HRMS $[M+Na^+]$: calcd for C₁₅H₂₀O₃SNF₃Na: 374.1008, found: 374.1004.

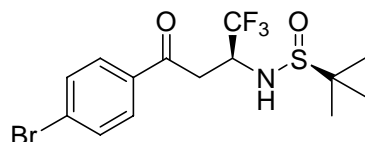


(*S*)-2-methyl-*N*-((*S*)-1,1,1-trifluoro-4-(4-fluorophenyl)-4-oxobutan-2-yl)propane-2-sulfinamide (**3d**): colorless solid, yield 74%, mp 149-151 °C, $[\alpha]_D^{25} +99.0$ ($c = 0.19$, CHCl₃). ¹H NMR (CDCl₃, 300 MHz): $\delta = 7.96$ -8.03 (m, 2 H), 7.11-7.19 (m, 2 H), 4.49-4.59 (m, 1 H), 4.15 (d, $J = 7.5$ Hz, 1 H), 3.76 (dd, $J = 9.3, 17.7$ Hz, 1 H), 3.29 (dd, $J = 3.3, 17.7$ Hz, 1 H), 1.16 (s, 9 H). ¹³C NMR (CDCl₃, 75 MHz): $\delta = 193.3, 167.8, 164.5, 131.0$ (d, $J = 9.5$ Hz), 123.4 (q, $J = 280.1$ Hz), 116.1 (d, $J = 21.9$ Hz), 56.9, 53.6 (q, $J = 30.5$ Hz), 37.9, 22.3. ¹⁹F NMR (CDCl₃, 376 MHz): $\delta = -74.4, -103.8$. IR (KBr): $\nu = 3271, 2966, 2926, 2871, 1687, 1599, 1293, 1271, 1233, 1169, 1121, 1061, 821$ cm⁻¹. HRMS $[M+Na^+]$: calcd for C₁₄H₁₇O₂SNF₄Na: 362.0808, found: 362.0810.

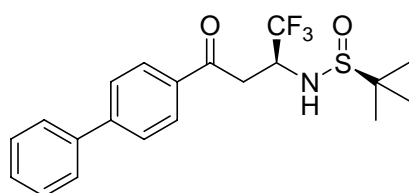


(*S*)-*N*-((*S*)-4-(4-chlorophenyl)-1,1,1-trifluoro-4-oxobutan-2-yl)-2-methylpropane-2-sulfinamide (**3e**): colorless solid, yield 70%, mp 147-148 °C, $[\alpha]_D^{25} +95.8$ ($c = 0.14$, CHCl₃). ¹H NMR (CDCl₃, 300 MHz): $\delta = 7.89$ -7.93 (m, 2 H), 7.44-7.49 (m, 2 H), 4.49-4.59 (m, 1 H), 4.06 (d, $J = 8.4$ Hz, 1 H), 3.77 (dd, $J = 9.9, 17.7$ Hz, 1 H), 3.30

(dd, $J = 3.0, 18$ Hz, 1 H), 1.17 (s, 9 H). ^{13}C NMR (CDCl_3 , 75 MHz): $\delta = 193.7, 140.4, 134.5, 129.6, 129.2, 123.4$ (q, $J = 279.0$ Hz), 56.9, 53.5 (q, $J = 30.8$ Hz), 38.0, 22.3. ^{19}F NMR (CDCl_3 , 376 MHz): $\delta = -74.3$. IR (KBr): $\nu = 3267, 2964, 2923, 1686, 1592, 1290, 1270, 1176, 1120, 1095, 1060, 813$ cm^{-1} . HRMS $[\text{M}+\text{Na}^+]$: calcd for $\text{C}_{14}\text{H}_{17}\text{O}_2\text{SNF}_3\text{ClNa}$: 378.0513, found: 378.0498.

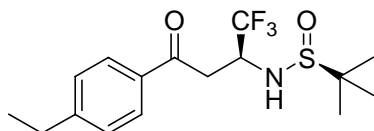


(*S*)-*N*-((*S*)-4-(4-bromophenyl)-1,1,1-trifluoro-4-oxobutan-2-yl)-2-methylpropane-2-sulfonamide (**3f**): white solid, yield 66%, mp 142-144 °C, $[\alpha]_{\text{D}}^{25} +61.1$ ($c = 0.55$, CHCl_3). ^1H NMR (CDCl_3 , 300 MHz): $\delta = 7.84$ (d, $J = 8.7$ Hz, 2 H), 7.65 (d, $J = 8.7$ Hz, 2 H), 4.49-4.59 (m, 1 H), 4.06 (d, $J = 8.1$ Hz, 1 H), 3.77 (dd, $J = 9.6, 17.4$ Hz, 1 H), 3.29 (dd, $J = 3.3, 17.7$ Hz, 1 H), 1.17 (s, 9 H). ^{13}C NMR (CDCl_3 , 75 MHz): $\delta = 193.9, 134.9, 132.2, 129.7, 129.2, 123.3$ (q, $J = 280.2$ Hz), 56.9, 53.5 (q, $J = 30.5$ Hz), 38.0, 22.3. ^{19}F NMR (CDCl_3 , 376 MHz): $\delta = -74.3$. IR (KBr): $\nu = 3202, 2964, 2923, 1688, 1586, 1397, 1348, 1287, 1175, 1112, 1056, 835$ cm^{-1} . HRMS $[\text{M}+\text{Na}^+]$: calcd for $\text{C}_{14}\text{H}_{17}\text{O}_2\text{SNF}_3\text{BrNa}$: 423.9988, found: 424.0010.

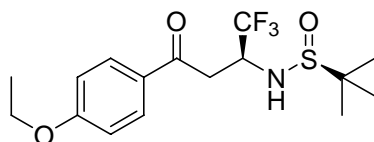


(*S*)-*N*-((*S*)-4-(biphenyl-4-yl)-1,1,1-trifluoro-4-oxobutan-2-yl)-2-methylpropane-2-sulfonamide (**3g**): white solid, yield 73%, mp 147-148 °C, $[\alpha]_{\text{D}}^{25} +91.2$ ($c = 0.19$, CHCl_3). ^1H NMR (CDCl_3 , 300 MHz): $\delta = 8.06$ (d, $J = 7.8$ Hz, 2 H), 7.70-7.73 (m, 2 H), 7.62-7.65 (m, 2 H), 7.40-7.52 (m, 3 H), 4.54-4.68 (m, 1 H), 4.29 (d, $J = 2.1$ Hz, 1 H), 3.83 (dd, $J = 9.6, 17.4$ Hz, 1 H), 3.37 (dd, $J = 2.7, 17.4$ Hz, 1 H), 1.19 (s, 9 H). ^{13}C NMR (CDCl_3 , 75 MHz): $\delta = 194.5, 146.5, 139.6, 135.0, 129.0, 128.8, 128.4, 127.5, 127.3, 123.5$ (d, $J = 278.4$ Hz), 56.9, 53.8 (q, $J = 31.3$ Hz), 38.1, 22.4. ^{19}F NMR (CDCl_3 , 376 MHz): $\delta = -74.4$. IR (KBr): $\nu = 3163, 2960, 2922, 1685, 1604, 1273,$

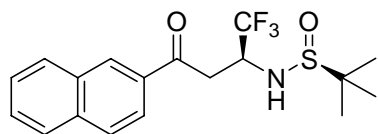
1158, 1122, 1061, 765 cm^{-1} . HRMS $[\text{M}+\text{Na}^+]$: calcd for $\text{C}_{20}\text{H}_{22}\text{O}_2\text{SNF}_3\text{Na}$: 420.1216, found: 420.1199.



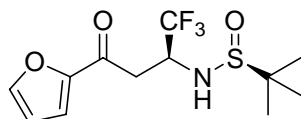
(*S*)-*N*-((*S*)-4-(4-ethylphenyl)-1,1,1-trifluoro-4-oxobutan-2-yl)-2-methylpropane-2-sulfonamide (**3h**): colorless solid, yield 68%, mp 97-98 °C, $[\alpha]_{\text{D}}^{25} +100.0$ ($c = 0.07$, CHCl_3). ^1H NMR (CDCl_3 , 300 MHz): $\delta = 7.88\text{-}7.91$ (m, 2 H), 7.28-7.33 (m, 2 H), 4.51-4.61 (m, 1 H), 3.95 (d, $J = 8.4$ Hz, 1 H), 3.73 (dd, $J = 9.6, 17.4$ Hz, 1 H), 3.31 (dd, $J = 3.0, 17.4$ Hz, 1 H), 2.76 (q, $J = 7.2$ Hz, 2 H), 1.30 (t, $J = 7.8$ Hz, 3 H), 1.17 (s, 9 H). ^{13}C NMR (CDCl_3 , 75 MHz): $\delta = 194.4, 151.0, 134.0, 128.4, 128.3, 123.5$ (q, $J = 279.8$ Hz), 56.9, 53.9 (q, $J = 30.5$ Hz), 37.9, 29.0, 22.3, 15.1. ^{19}F NMR (CDCl_3 , 376 MHz): $\delta = -74.4$. IR (KBr): $\nu = 3262, 2975, 2875, 1685, 1609, 1293, 1269, 1169, 1117, 1060, 819$ cm^{-1} . HRMS $[\text{M}+\text{Na}^+]$: calcd for $\text{C}_{16}\text{H}_{22}\text{O}_2\text{SNF}_3\text{Na}$: 372.1216, found: 372.1206.



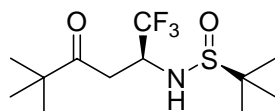
(*S*)-*N*-((*S*)-4-(4-ethoxyphenyl)-1,1,1-trifluoro-4-oxobutan-2-yl)-2-methylpropane-2-sulfonamide (**3i**): white solid, yield 87%, mp 114-115 °C, $[\alpha]_{\text{D}}^{25} +78.8$ ($c = 0.21$, CHCl_3). ^1H NMR (CDCl_3 , 300 MHz): $\delta = 7.90\text{-}7.95$ (m, 2 H), 6.90-6.96 (m, 2 H), 4.50-4.56 (m, 1 H), 4.15 (q, $J = 7.2$ Hz, 2 H), 4.03 (d, $J = 8.7$ Hz, 1 H), 3.68 (dd, $J = 9.3, 17.4$ Hz, 1 H), 3.26 (dd, $J = 3.0, 17.4$ Hz, 1 H), 1.47 (t, $J = 6.9$ Hz, 3 H), 1.16 (s, 9 H). ^{13}C NMR (CDCl_3 , 75 MHz): $\delta = 193.2, 163.5, 130.5, 129.1, 123.5$ (q, $J = 279.8$ Hz), 114.4, 63.9, 56.9, 54.0 (q, $J = 29.7$ Hz), 37.6, 22.3, 14.6. ^{19}F NMR (CDCl_3 , 376 MHz): $\delta = -74.5$. IR (KBr): $\nu = 3167, 2980, 1682, 1602, 1366, 1287, 1261, 1231, 1171, 1117, 1061, 833$ cm^{-1} . HRMS $[\text{M}+\text{Na}^+]$: calcd for $\text{C}_{16}\text{H}_{22}\text{O}_3\text{SNF}_3\text{Na}$: 388.1165, found: 388.1169.



(*S*)-2-methyl-*N*-((*S*)-1,1,1-trifluoro-4-(naphthalen-2-yl)-4-oxobutan-2-yl)propane-2-sulfonamide (**3j**): colorless solid, yield 76%, mp 140-143 °C, $[\alpha]_D^{25} +34.2$ ($c = 0.08$, CHCl₃). ¹H NMR (CDCl₃, 300 MHz): $\delta = 8.49$ (s, 1 H), 7.98-8.05 (m, 2 H), 7.94 (t, $J = 8.7$ Hz, 2 H), 7.56-7.67 (m, 2 H), 4.56-4.72 (m, 1 H), 4.01 (s, 1 H), 3.92 (dd, $J = 9.6$, 17.7 Hz, 1 H), 3.48 (dd, $J = 3.3$, 17.7 Hz, 1 H), 1.18 (s, 9 H). ¹³C NMR (CDCl₃, 75 MHz): $\delta = 194.7$, 135.9, 133.6, 132.5, 130.1, 129.7, 128.9, 128.8, 127.8, 127.0, 123.6, 123.5 (d, $J = 280.7$ Hz), 56.9, 53.9 (q, $J = 31.5$ Hz), 38.2, 22.3. ¹⁹F NMR (CDCl₃, 376 MHz): $\delta = -74.3$. IR (KBr): $\nu = 3260$, 2970, 2936, 1674, 1286, 1171, 1119, 1058, 1046, 822 cm⁻¹. HRMS $[M+Na^+]$: calcd for C₁₈H₂₀O₂SNF₃Na: 394.1059, found: 394.1050.



(*S*)-2-methyl-*N*-((*S*)-1,1,1-trifluoro-4-(furan-2-yl)-4-oxobutan-2-yl)propane-2-sulfonamide (**3k**): colorless solid, yield 42%, mp 110-111 °C, $[\alpha]_D^{25} +48.8$ ($c = 0.08$, CHCl₃). ¹H NMR (CDCl₃, 300 MHz): $\delta = 7.64$ (dd, $J = 0.6$, 1.5 Hz, 1 H), 7.31 (dd, $J = 0.6$, 3.3 Hz, 1 H), 6.61 (q, $J = 1.8$ Hz, 1 H), 4.45-4.55 (m, 1 H), 3.88 (d, $J = 9.0$ Hz, 1 H), 3.59 (dd, $J = 9.9$, 17.4 Hz, 1 H), 3.21 (dd, $J = 3.3$, 17.1 Hz, 1 H), 1.17 (s, 9 H). ¹³C NMR (CDCl₃, 75 MHz): $\delta = 183.8$, 152.2, 147.1, 123.2 (q, $J = 280.3$ Hz), 118.1, 112.8, 57.0, 53.8 (q, $J = 31.1$ Hz), 37.9 (d, $J = 1.3$ Hz), 22.3. ¹⁹F NMR (CDCl₃, 376 MHz): $\delta = -74.7$. IR (KBr): $\nu = 3283$, 3129, 3091, 2967, 1666, 1468, 1304, 1267, 1172, 1125, 1075, 767 cm⁻¹. HRMS $[M+Na^+]$: calcd for C₁₂H₁₆O₃SNF₃Na: 334.0695, found: 334.0699.



(*S*)-2-methyl-N-((*S*)-1,1,1-trifluoro-5,5-dimethyl-4-oxohexan-2-yl)propane-2-sulfonamide (**3I**): colorless solid, yield 61%, mp 85-86 °C, $[\alpha]_D^{25} +32.3$ ($c = 0.76$, CHCl₃). ¹H NMR (CDCl₃, 500 MHz): $\delta = 4.33$ -4.39 (m, 1 H), 3.83 (d, $J = 8.5$ Hz, 1 H), 3.25 (dd, $J = 9.5, 18.5$ Hz, 1 H), 2.88 (dd, $J = 3.5, 18.5$ Hz, 1 H), 1.20 (s, 18 H). ¹³C NMR (CDCl₃, 125 MHz): $\delta = 210.5, 124.2$ (d, $J = 279.9$ Hz), 56.8, 53.5 (q, $J = 30.8$ Hz), 44.2, 36.3, 26.3, 22.3. ¹⁹F NMR (CDCl₃, 376 MHz): $\delta = -74.2$. IR (KBr): $\nu = 3244, 2967, 2928, 1712, 1473, 1365, 1281, 1222, 1168, 1125, 1061$ cm⁻¹. HRMS [M+Na⁺]: calcd for C₁₂H₂₂O₂SNF₃Na: 324.1216, found: 324.1209.

3. Reaction of large scale application study

Into an oven-dried round-bottom flask flushed with N₂ were taken acetophenone (8.5 mmol) and anhydrous THF (20.0 mL). The reaction flask was cooled to -78 °C and LDA (2 M in THF, 4.7 mL) was added dropwise with stirring. After 45 min at -78 °C, sulfinylimine **1** (5 mmol) dissolved in anhydrous THF (10.0 mL) was added dropwise. Stirring was continued at -78 °C for 2.5 h, then the reaction was quenched with saturated NH₄Cl (10.0 mL), followed by H₂O (15.0 mL) and the mixture was brought to room temperature. The organic layer was taken and the aqueous layer was extracted with EtOAc (2 × 30 mL). The combined organic layers were dried with anhydrous Na₂SO₄, filtered and the solvent was removed to give the crude product, which was purified by column chromatography (hexane/EtOAc, 4:1).

4. X-ray crystallography for 3a

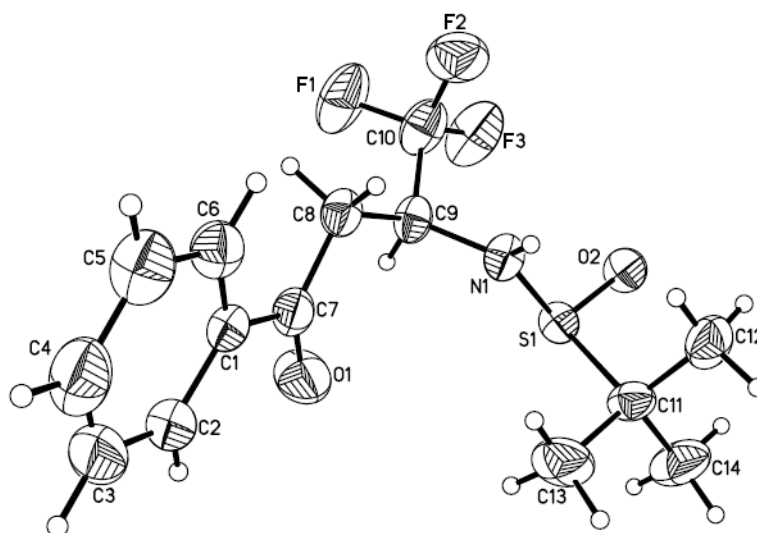
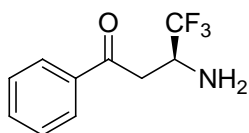


Figure 1 ORTEP structure of compound **3a**. (CCDC number 787957)

5. Conversion of 3a affording free β -amino ketone 4

3a (0.5 mmol) and MeOH (5.0 mL) were placed in a 25 mL round-bottom flask and aq HCl (36%, 1 mL) was added. The reaction was stirred at r.t. for 8 h, during which time the cleavage was monitored by TLC. Volatiles were removed under reduced pressure. The residue was dissolved in CH₂Cl₂ (10.0 mL) and Et₃N (15 mmol) was added. The reaction was stirred at r.t. for 1 h then H₂O (10.0 mL) was added. The organic layer was taken, washed with H₂O (2 \times 10 mL), dried with anhydrous Na₂SO₄, filtered and the solvent was removed to give the crude product, which was purified by TLC plate (hexane/EtOAc, 2:1).



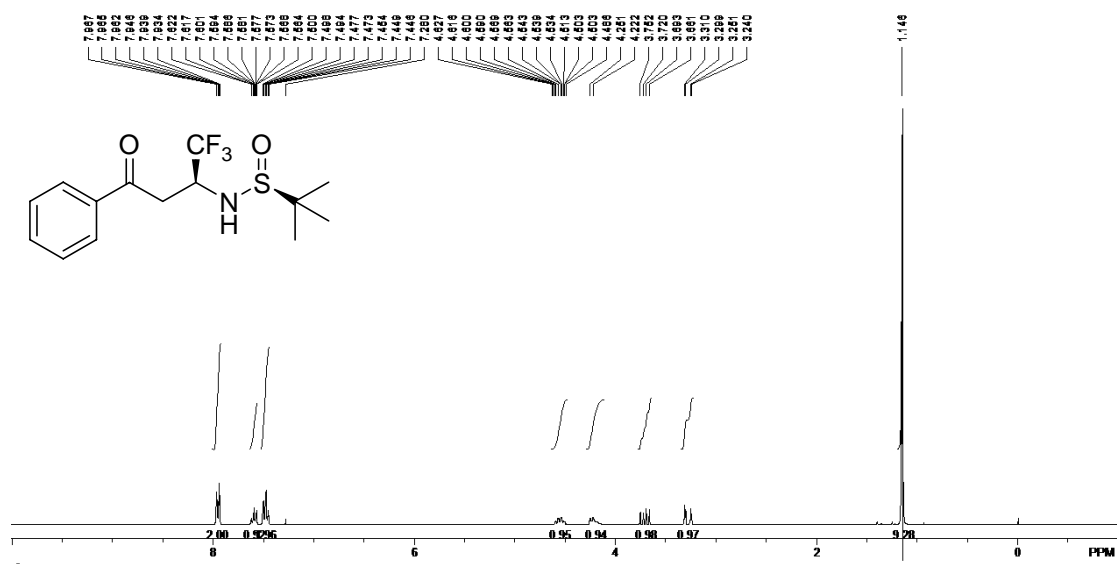
(*S*)-3-amino-4,4,4-trifluoro-1-phenylbutan-1-one (**4**)¹: white solid, yield 90%, mp 31-32 °C, [α]_D²⁵ -57.2 (*c* = 0.25, CHCl₃). ¹H NMR (CDCl₃, 300 MHz): δ = 7.97-8.00 (m, 2 H), 7.60-7.66 (m, 1 H), 7.49-7.54 (m, 2 H), 3.98-4.10 (m, 1 H), 3.38 (dd, *J* = 2.7, 17.7 Hz, 1 H), 3.25 (dd, *J* = 9.6, 17.1 Hz, 1 H), 1.66 (s, 2 H). ¹³C NMR (CDCl₃, 75 MHz): δ = 196.2, 136.3, 133.8, 128.8, 128.1, 124.6 (q, *J* = 278.8 Hz), 50.0 (q, *J* = 29.6 Hz), 39.3 (q, *J* = 1.4 Hz). ¹⁹F NMR (CDCl₃, 376 MHz): δ = -78.3. IR (KBr): ν = 3391, 3332, 2937, 1684, 1596, 1333, 1257, 1221, 1177, 1106, 754, 687 cm⁻¹. HRMS [*M*+H⁺]: calcd for C₁₀H₁₁ONF₃: 218.0787, found: 218.0796.

Reference

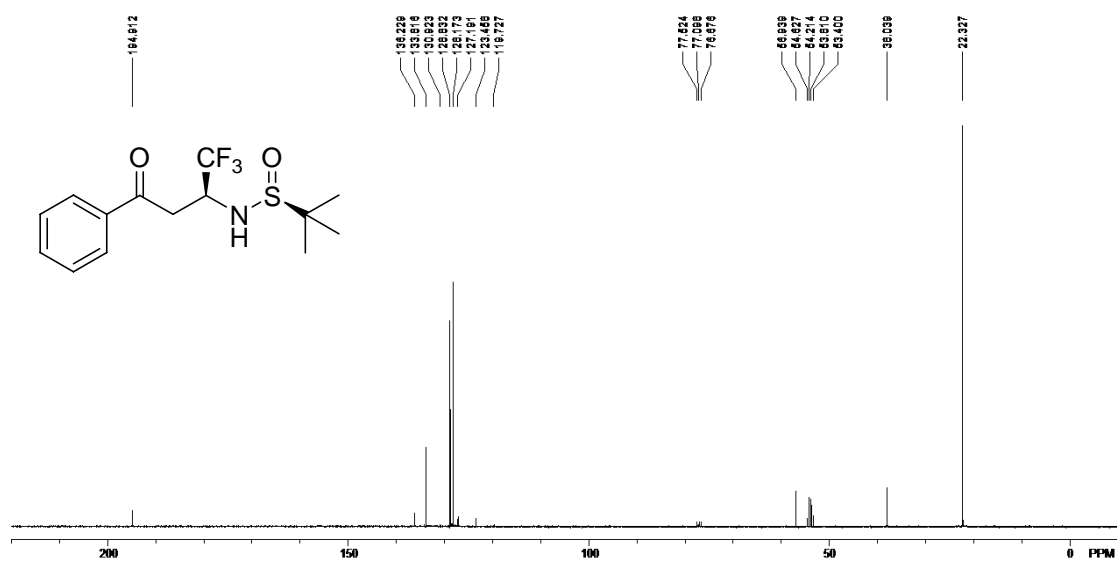
- 1 F. Huguenot and T. Brigaud, *J. Org. Chem.*, 2006, **71**, 2159–2162.

6. ^1H and ^{13}C NMR spectra for compound 3 and 4

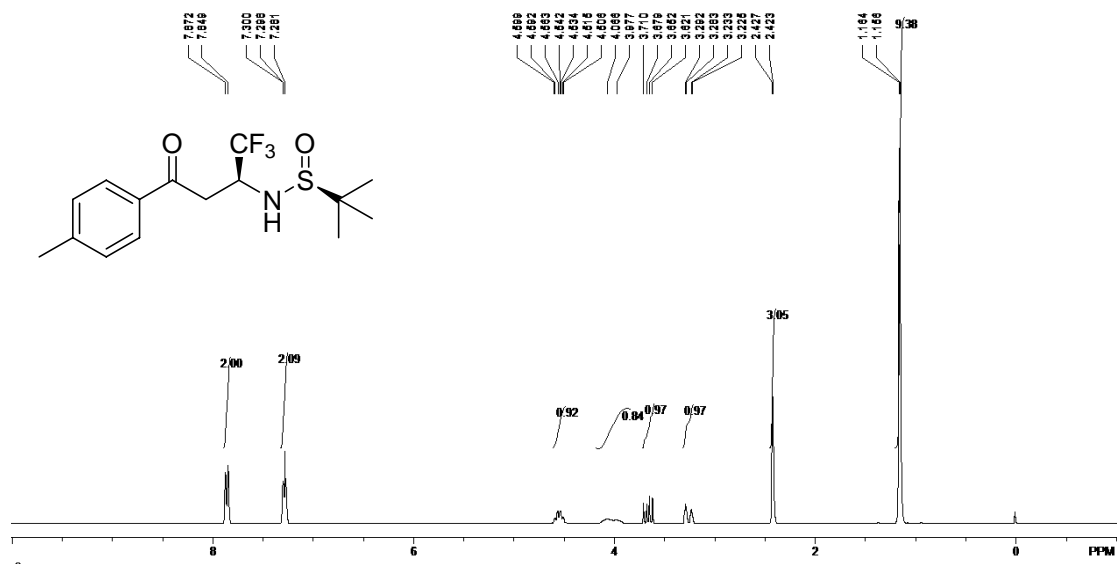
^1H NMR of 3a



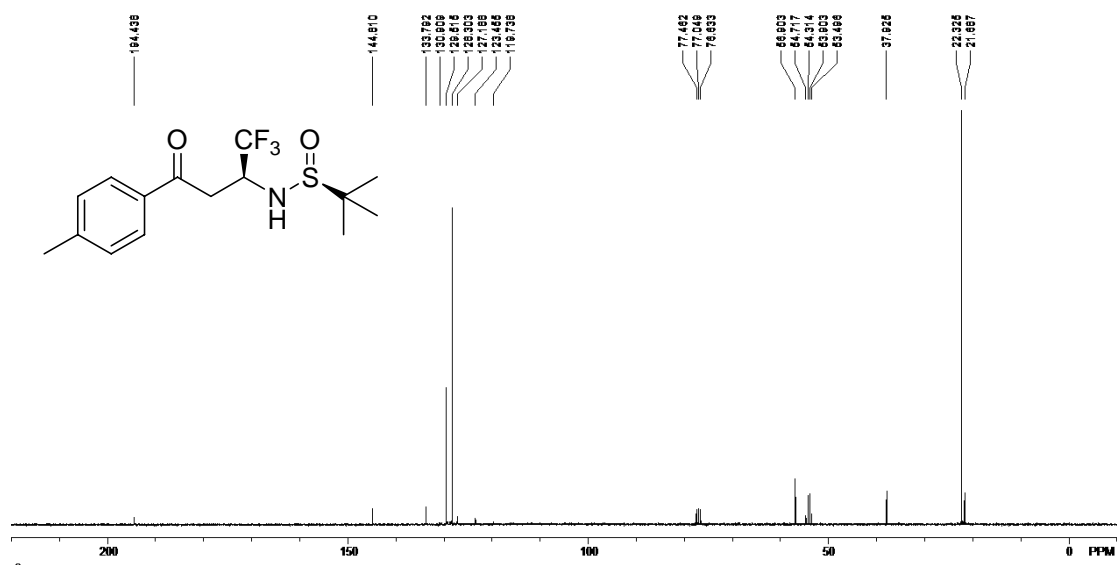
^{13}C NMR of 3a



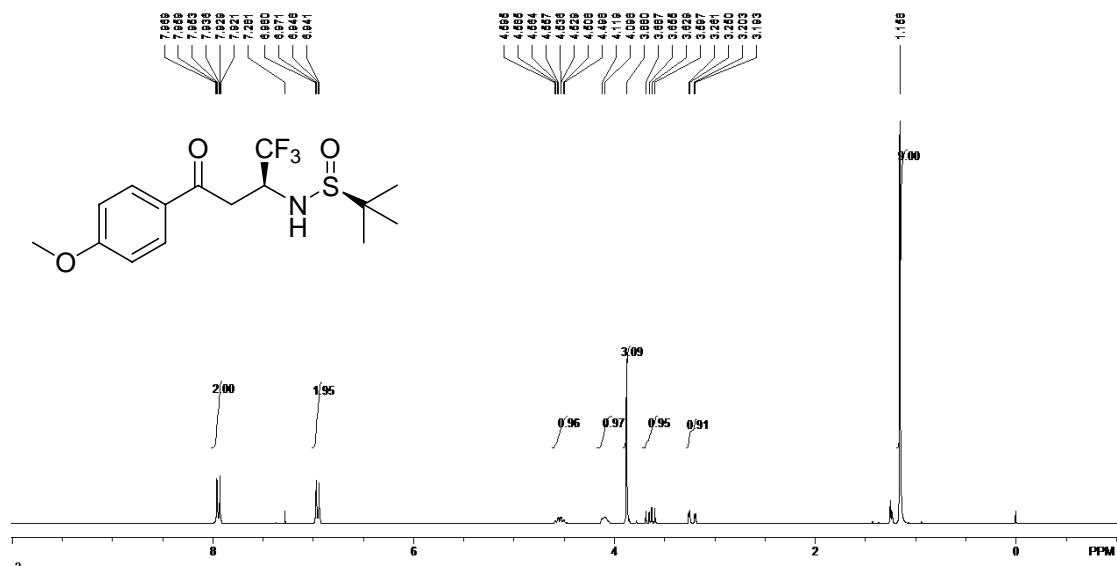
^1H NMR of **3b**



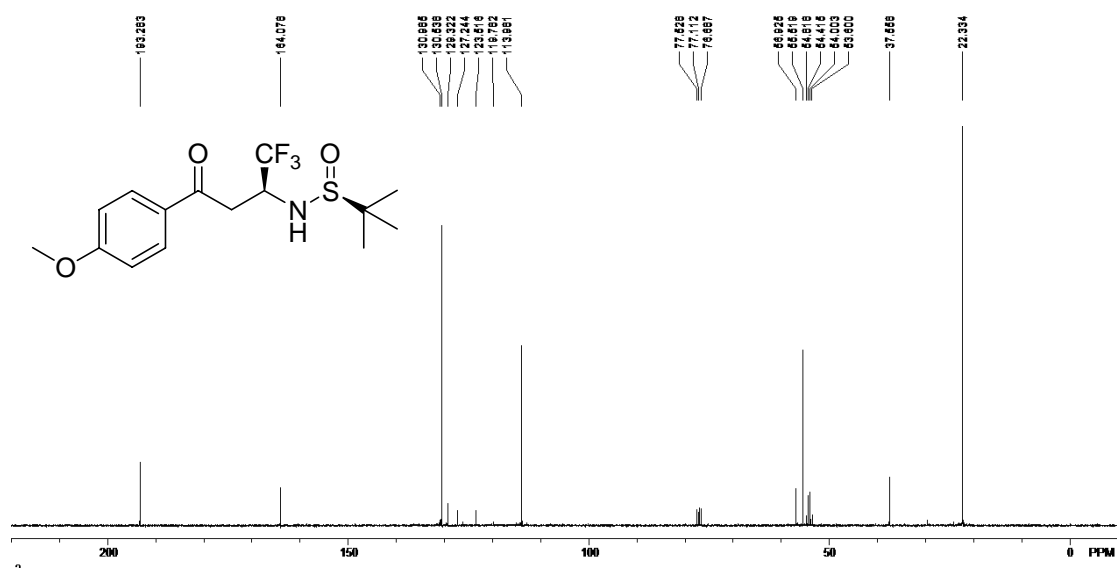
^{13}C NMR of **3b**



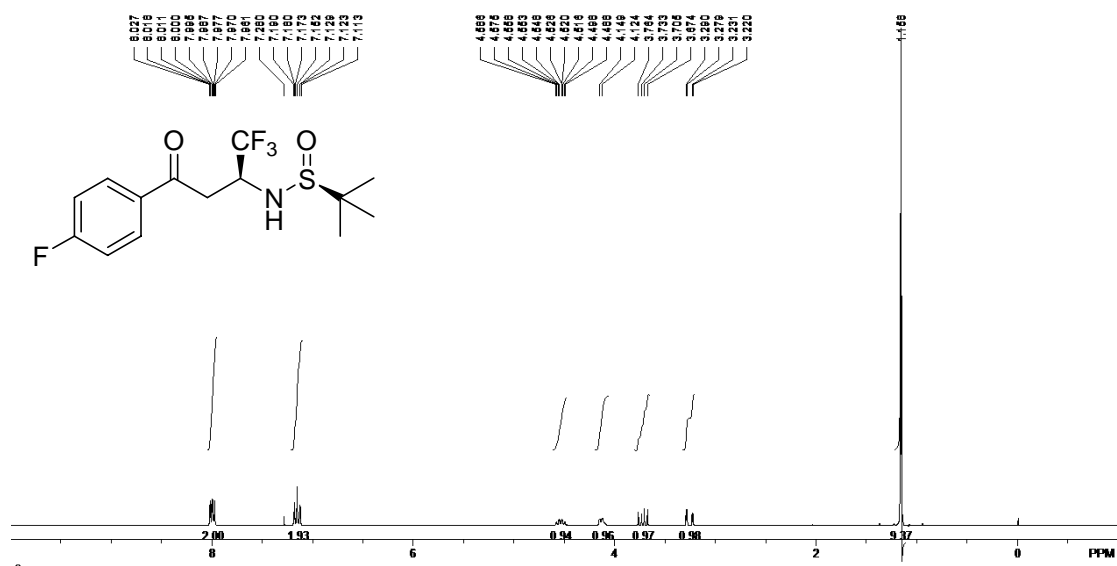
^1H NMR of **3c**



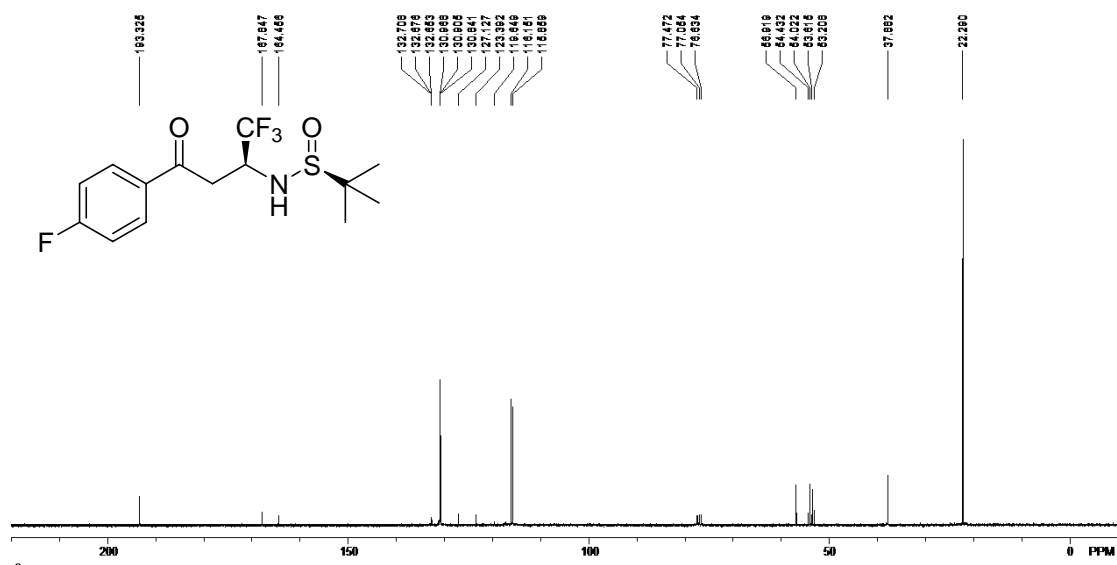
^{13}C NMR of **3c**



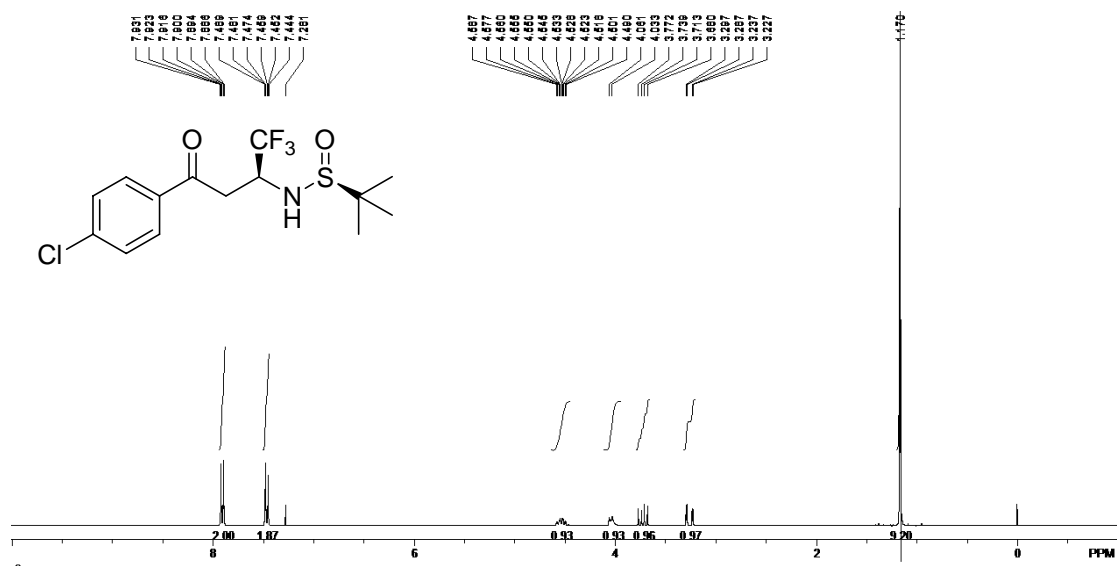
^1H NMR of **3d**



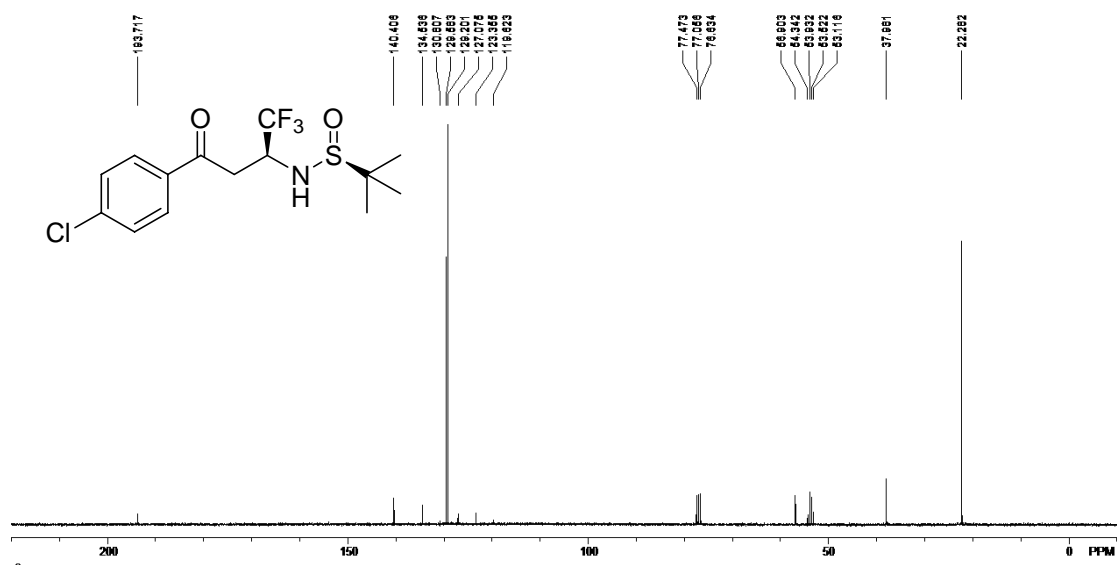
^{13}C NMR of **3d**



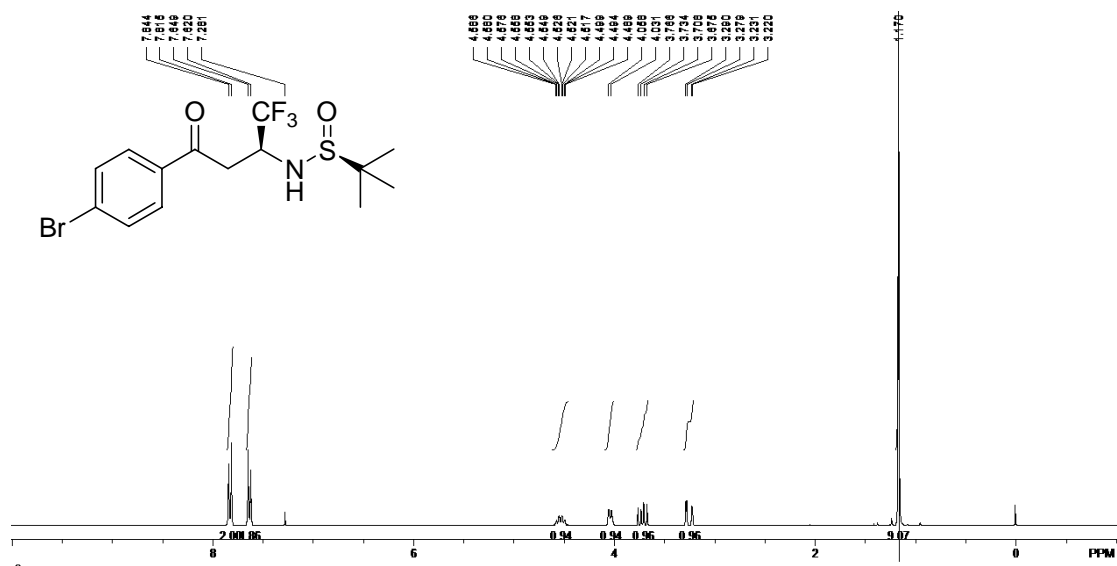
^1H NMR of **3e**



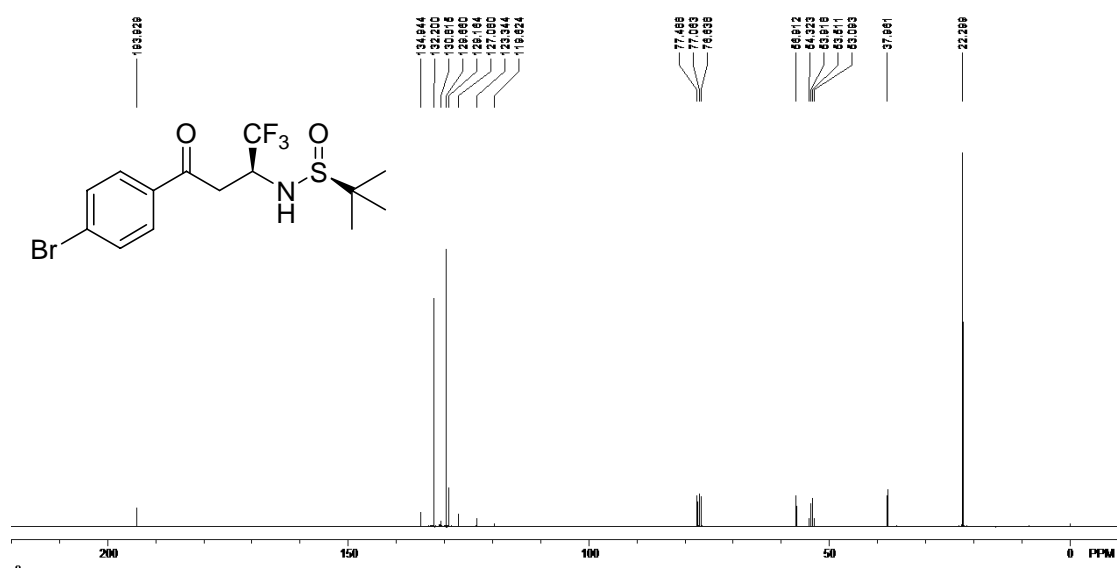
^{13}C NMR of **3e**

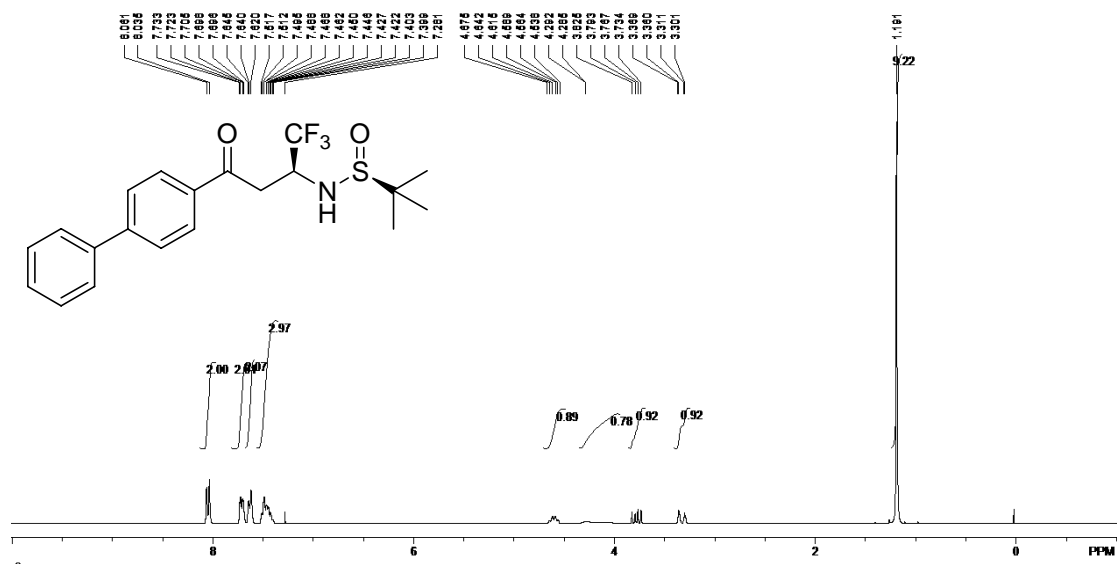
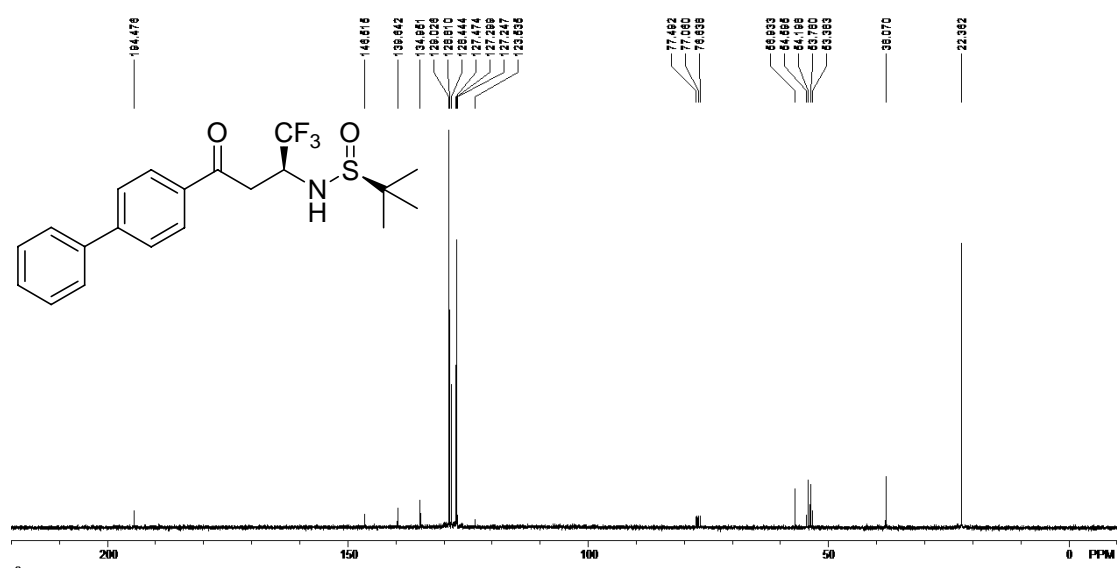


^1H NMR of **3f**

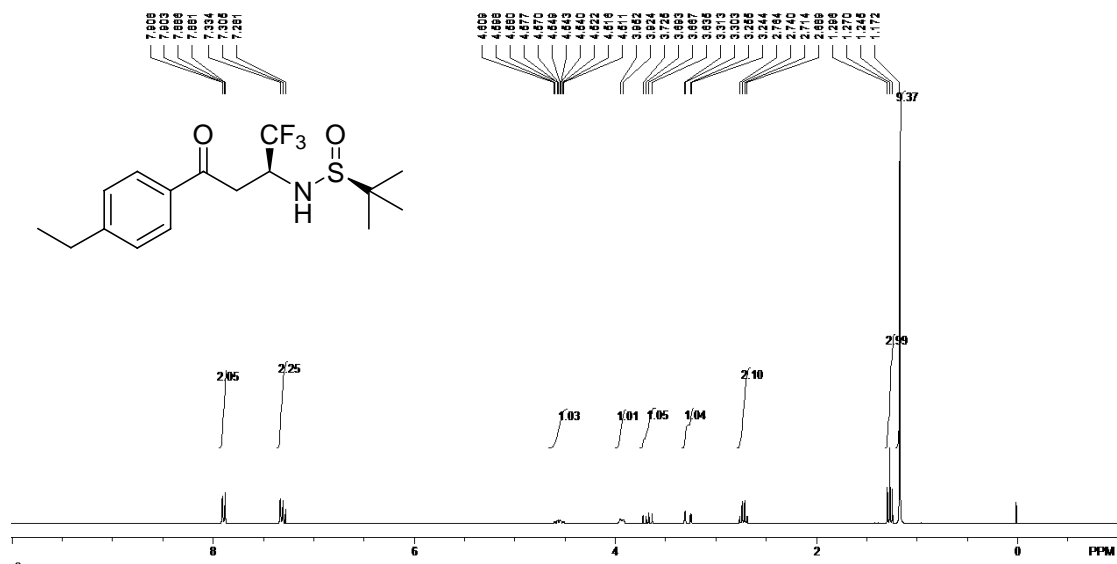


^{13}C NMR of **3f**

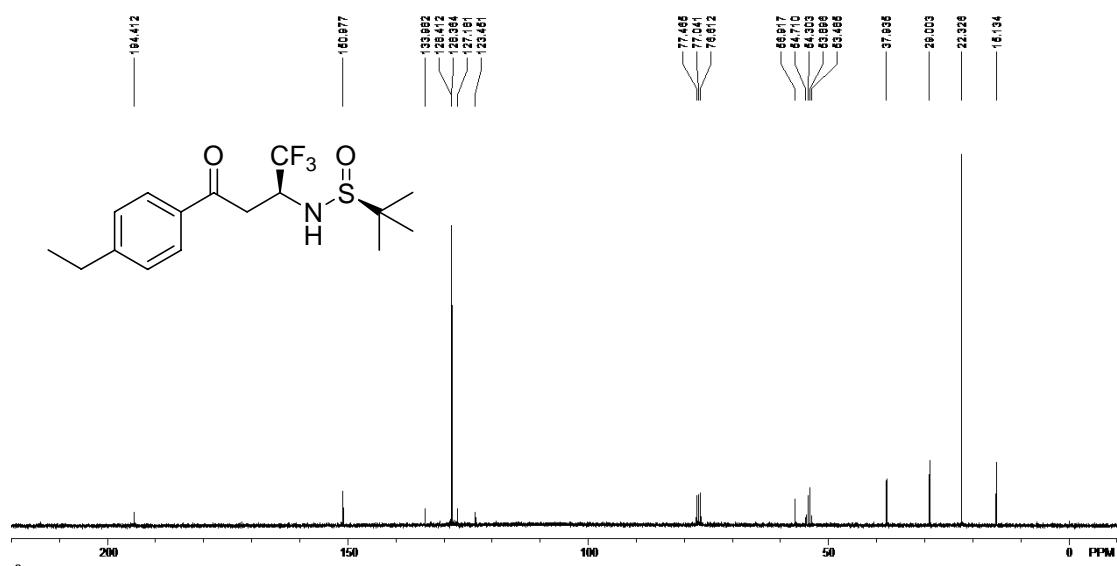


¹H NMR of **3g** ^{13}C NMR of **3g**

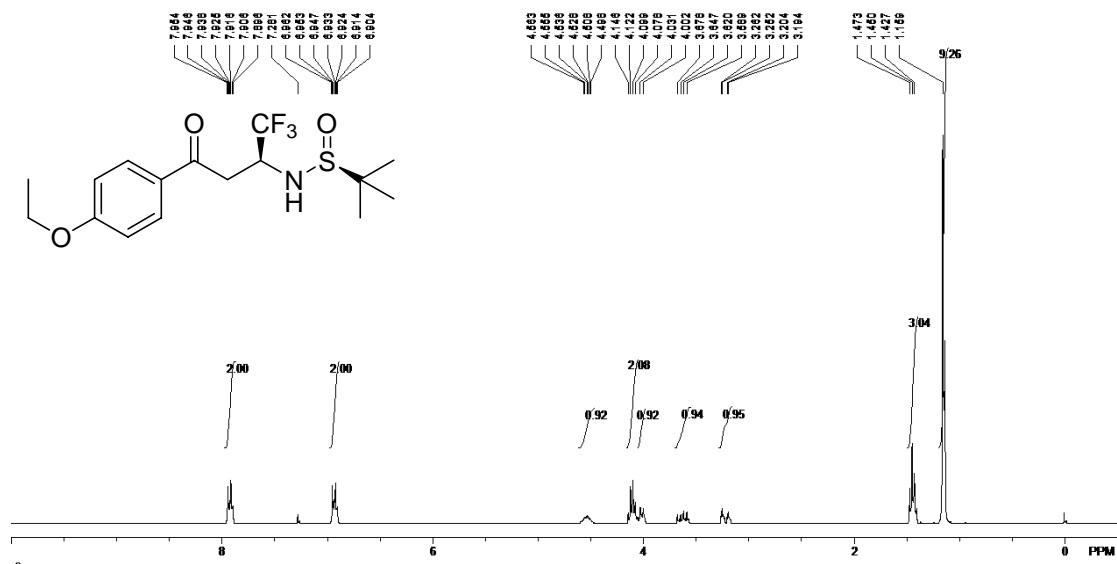
¹H NMR of **3h**



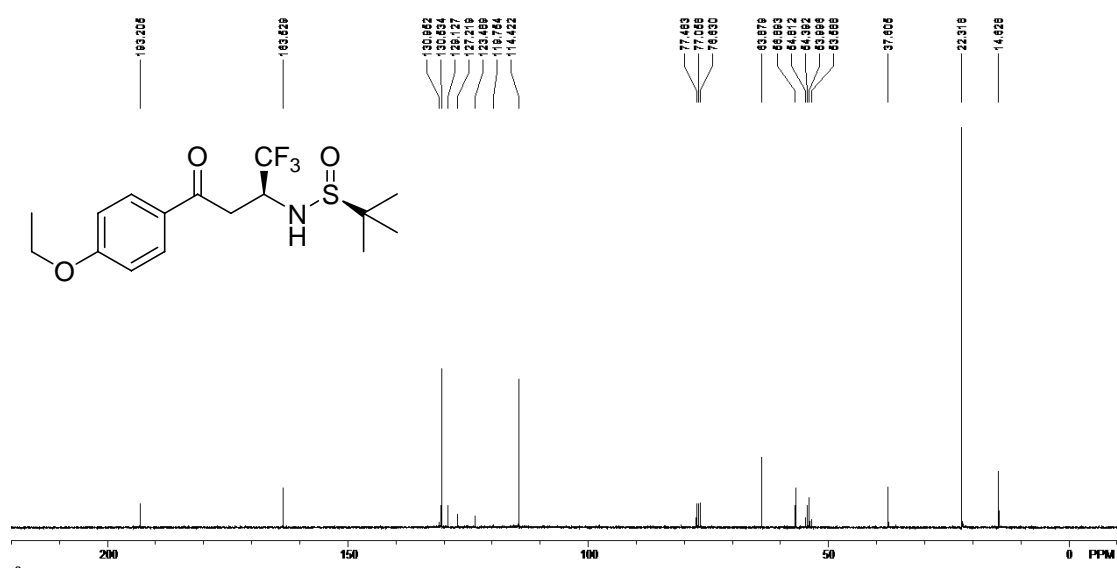
¹³C NMR of **3h**



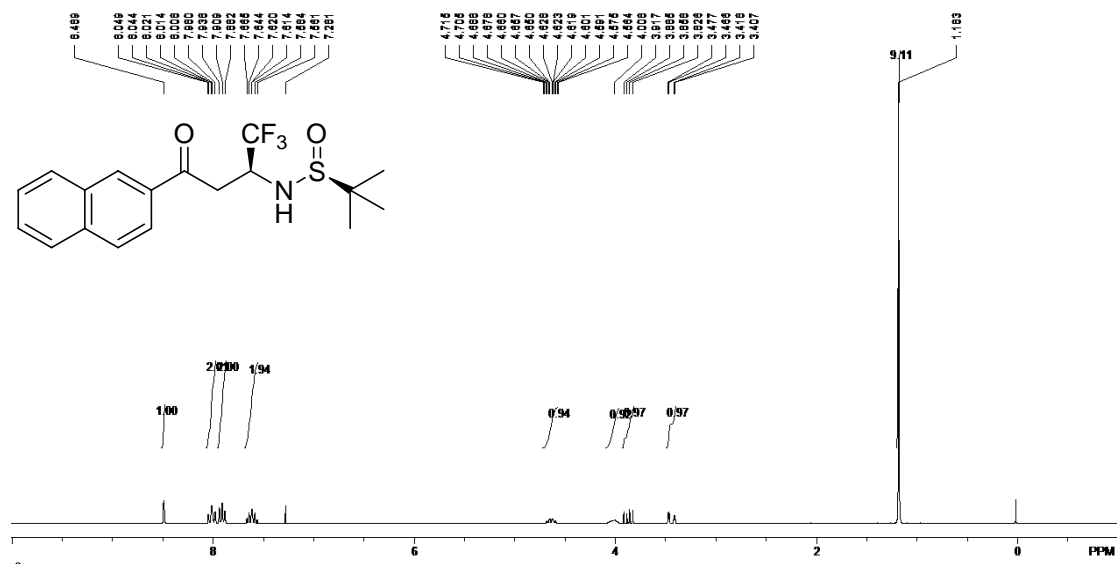
^1H NMR of **3i**



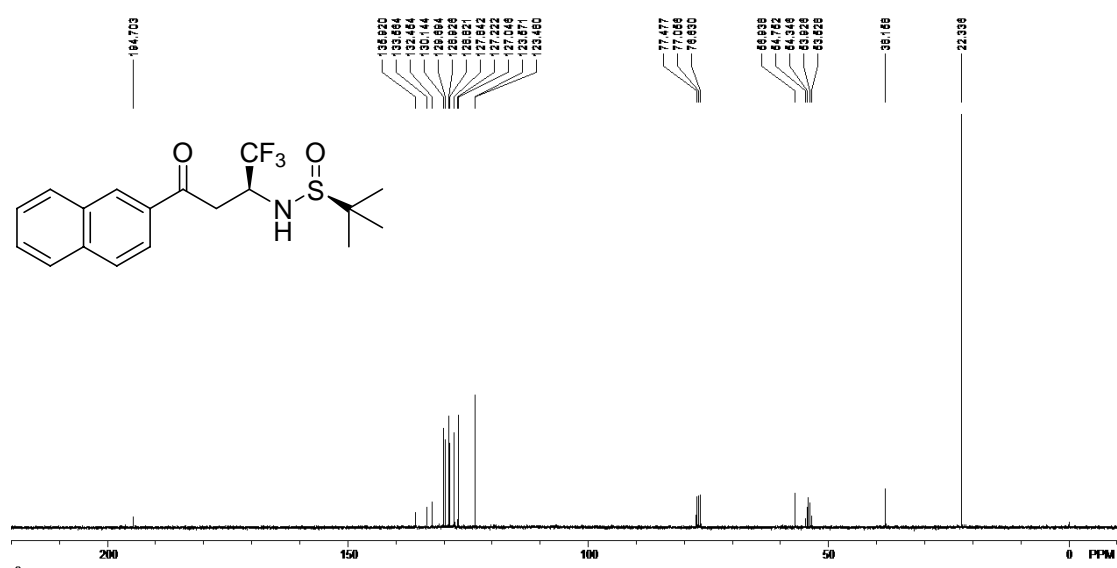
^{13}C NMR of **3i**

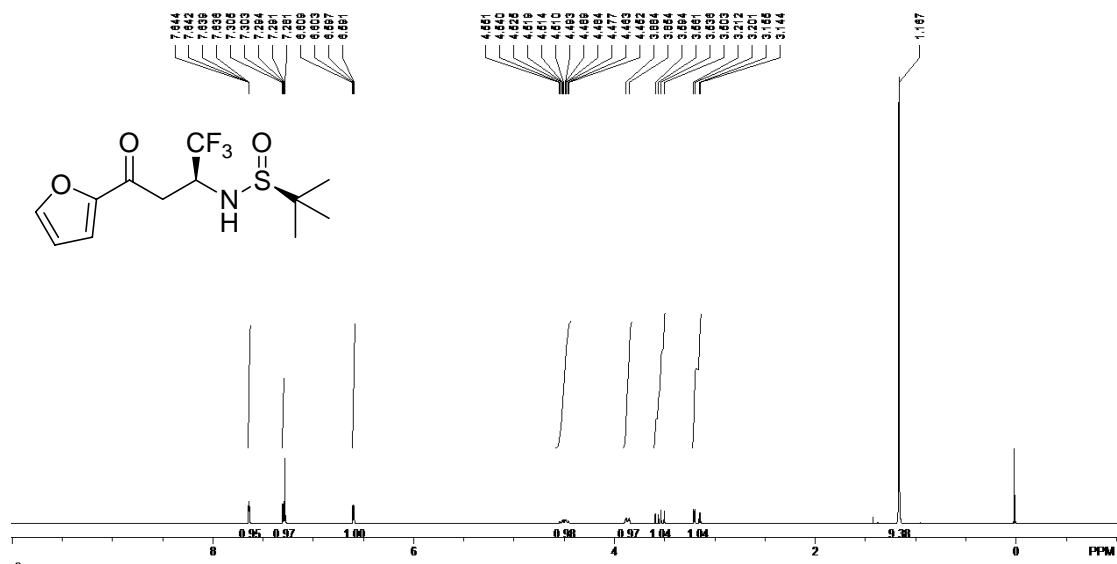
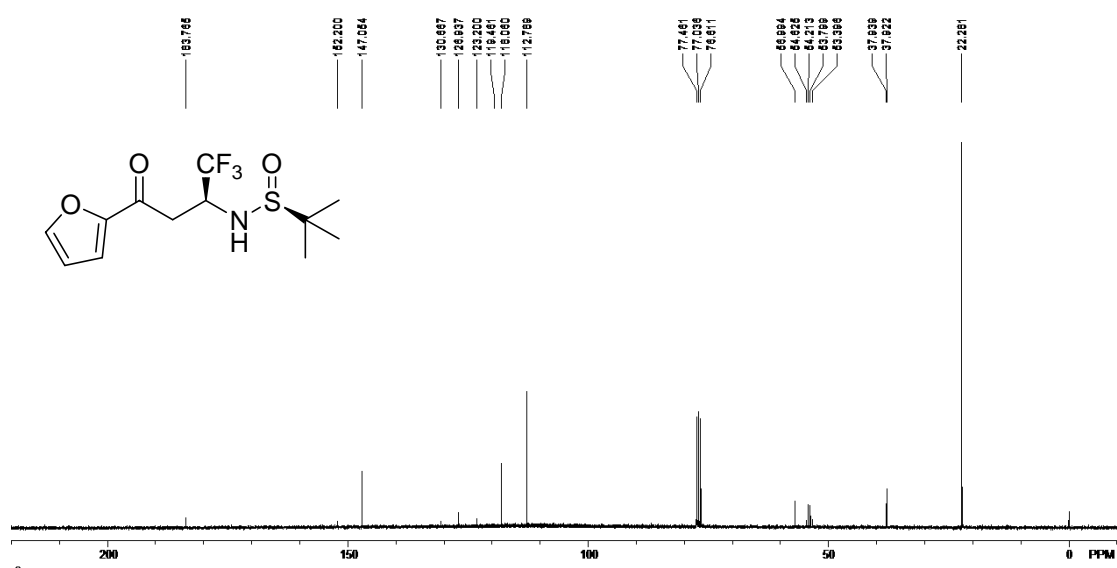


^1H NMR of **3j**

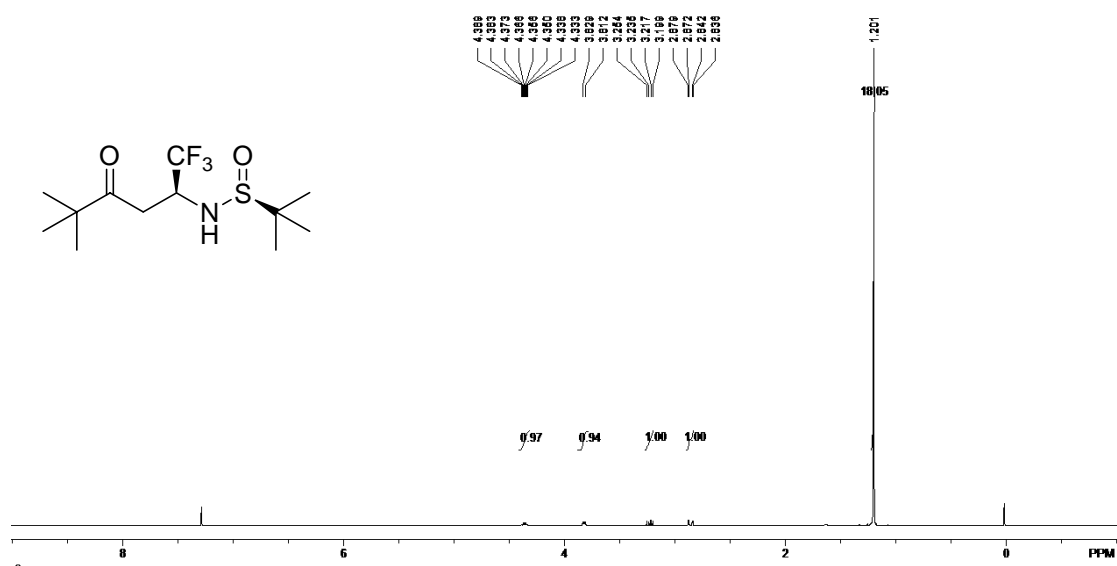


^{13}C NMR of **3j**

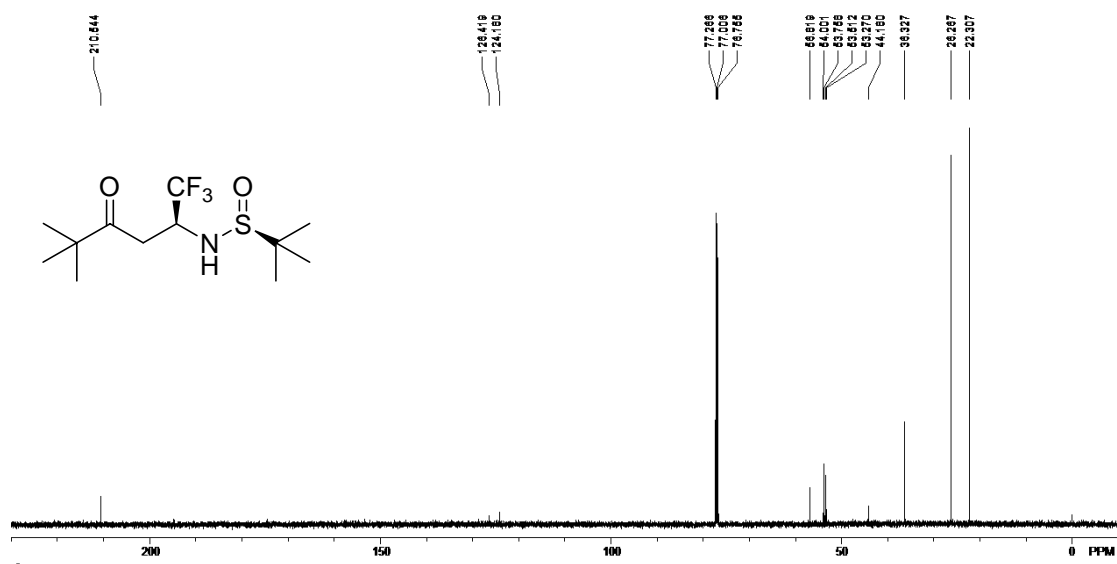


¹H NMR of **3k** ^{13}C NMR of **3k**

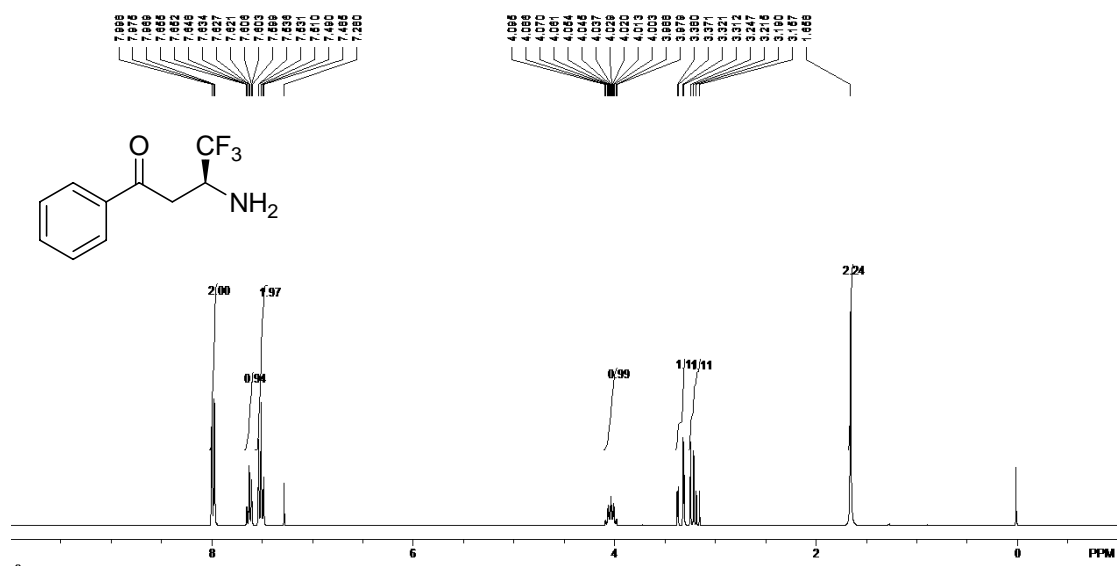
^1H NMR of **31**



^{13}C NMR of **31**



¹H NMR of 4



¹³C NMR of 4

