

Manganese(II)-catalyzed modular synthesis of isoquinolines from vinyl isocyanides and hydrazines

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Supporting Information

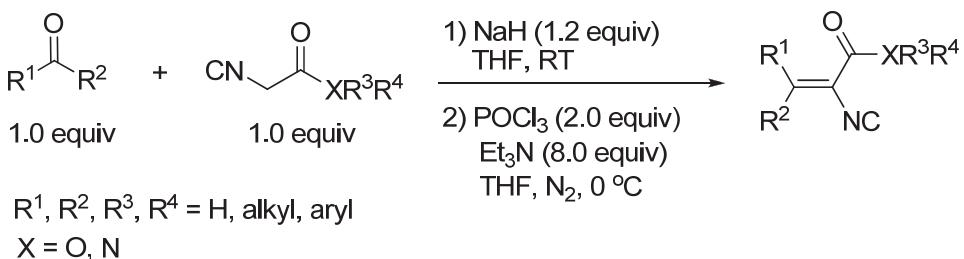
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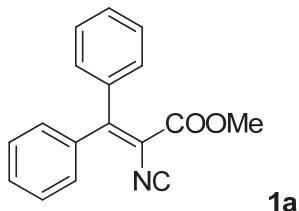
1. General Information

All reagents and metal catalysts were obtained from commercial sources without further purification, and commercially available solvents were purified before use. All new compounds were fully characterized. All melting points were taken on a WRS-1A or a WRS-1B Digital Melting Point Apparatus without correction. Infrared spectra were obtained using an AVATAR 370 FT-IR spectrometer. ^1H , ^{13}C and ^{19}F spectra were recorded with a Bruker AV-500 spectrometer operating at 500, 125 and 470 MHz, respectively, with chemical shift values being reported in ppm relative to chloroform ($\delta = 7.26$ ppm) or TMS ($\delta = 0.00$ ppm) for ^1H NMR, chloroform ($\delta = 77.16$ ppm) for ^{13}C NMR, and C_6F_6 ($\delta = -164.9$ ppm) for ^{19}F NMR. Mass spectra and high resolution mass spectra were recorded with an Agilent 5975N using an Electron impact (EI) or Electrospray ionization (ESI) techniques. Silica gel plate GF254 was used for thin layer chromatography (TLC) and silica gel H or 300-400 mesh was used for flash column chromatography. Yields refer to chromatographically and spectroscopically pure compounds, unless otherwise indicated.

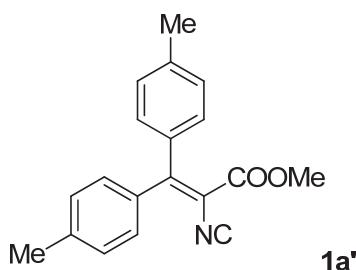
2. Synthesis and Characterization for vinyl isocyanides **1a-1m**:



General Procedure^[1]: A mixture of ketone or aldehyde (10.0 mmol) and isocyanide (10.0 mmol) in THF (10.0 mL) was added dropwise to a suspension of NaH (12.0 mmol, 60% in oil) in THF (10.0 mL) at room temperature. After stirring for 2 h at room temperature, 10% AcOH was added to the mixture at 0 °C until there was no hydrogen gas release. After removal of the solvent under reduced pressure, the residue was extracted with CH₂Cl₂ for three times and the organic layer was washed with H₂O, dried over Na₂SO₄ and concentrated under reduced pressure. Further recrystallization in MeOH afforded the corresponding formamide product as a white solid. To an ice cooled three necked flask containing prepared formamide (5.0 mmol), NEt₃ (40 mmol) and THF (10.0 mL) under N₂ atmosphere, POCl₃ (10.0 mmol) was added dropwise and the mixture was stirred for 1 h at 0 °C after the addition was completed. Then the mixture was quenched by saturated Na₂CO₃ aqueous and stirred for another 1 h. The mixture was extracted with CH₂Cl₂ for three times, dried over Na₂SO₄ and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel to give **1a-1m**.

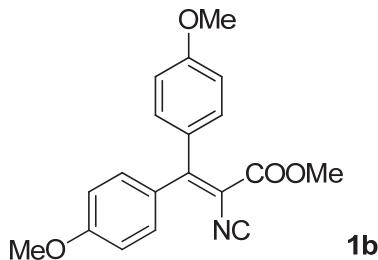


Methyl 2-isocyano-3,3-diphenylacrylate (1a)^[1]: White solid (62% yield for 2 steps). M.p. 104–105 °C. ¹H NMR (CDCl₃, 500 MHz): δ 7.44–7.38 (m, 4H), 7.38–7.34 (m, 4H), 7.16–7.12 (m, 2H), 3.69 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz): δ 169.78, 162.28, 154.60, 137.81, 137.39, 130.31, 129.88, 129.59, 129.09, 128.46, 128.25, 52.92.

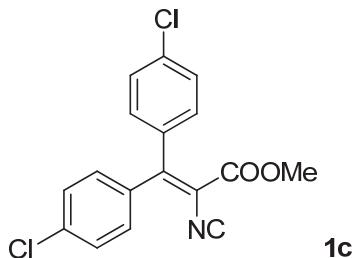


Methyl 2-isocyano-3,3-di-p-tolylacrylate (1a')^[1]: Pale yellow solid (64% yield for 2 steps). M.p. 97–99 °C. ¹H NMR (CDCl₃, 500 MHz): δ 7.25 (d, *J* = 7.5 Hz, 2H), 7.20

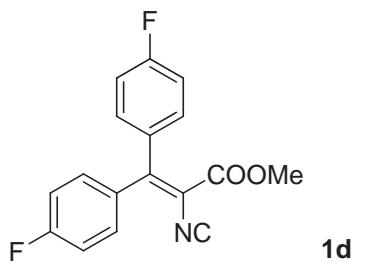
(d, $J = 8.0$ Hz, 2H), 7.16 (d, $J = 8.0$ Hz, 2H), 7.02 (d, $J = 8.0$ Hz, 2H), 3.70 (s, 3H), 2.38–2.39 (m, 6H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 169.14, 162.60, 155.10, 140.79, 139.89, 135.08, 134.75, 130.12, 129.32, 129.08, 128.92, 112.71, 52.83, 21.49, 21.45.



Methyl 2-isocyano-3,3-bis(4-methoxyphenyl)acrylate (1b)^[1]: Pale yellow solid (54% yield for 2 steps). M.p. 84–85 °C. ^1H NMR (CDCl_3 , 500 MHz): δ 7.31 (d, $J = 9.0$ Hz, 2H), 7.05 (d, $J = 8.5$ Hz, 2H), 6.90 (d, $J = 8.5$ Hz, 2H), 6.86 (d, $J = 9.0$ Hz, 2H), 3.83 (s, 3H), 3.82 (s, 3H), 3.69 (s, 3H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 168.57, 163.00, 161.36, 161.03, 154.68, 132.36, 131.40, 130.26, 129.88, 113.76, 113.63, 111.34, 55.43, 55.34, 52.76.

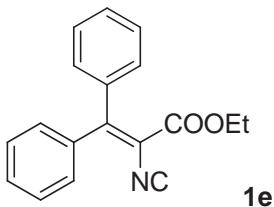


Methyl 3,3-bis(4-chlorophenyl)-2-isocyanoacrylate (1c)^[1]: Pale yellow solid (68% yield for 2 steps). M.p. 111–112 °C. IR (KBr, cm^{-1}): 2117.1, 1734.5, 1580.0, 1484.5, 1246.2, 1083.5, 827.4, 770.8. ^1H NMR (CDCl_3 , 500 MHz): δ 7.37 (td, $J = 9.5, 2.0$ Hz, 2H), 7.32 (td, $J = 9.5, 2.0$ Hz, 2H), 7.26 (td, $J = 9.5, 2.0$ Hz, 2H), 7.04 (td, $J = 9.0, 2.0$ Hz, 2H), 3.69 (s, 3H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 171.06, 161.73, 152.05, 136.80, 136.12, 135.70, 135.40, 131.30, 130.55, 128.96, 128.73, 114.24, 53.16. EI-MS m/z (%): 335 (11) [M^+ ($2 \times ^{37}\text{Cl}$)], 333 (60) [M^+ ($^{35}\text{Cl}, ^{37}\text{Cl}$)], 331 (90) [M^+ ($2 \times ^{35}\text{Cl}$)], 300 (47), 296 (44), 237 (100). HRMS (ESI) ($[\text{M}+\text{H}]^+$) Calcd. for $\text{C}_{17}\text{H}_{12}\text{Cl}_2\text{NO}_2$: 332.0245; found: 332.0237.

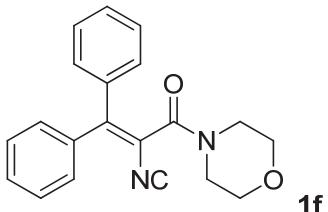


Methyl 3,3-bis(4-fluorophenyl)-2-isocyanoacrylate (1d)^[1]: Pale yellow solid (48% for 2 steps). M.p. 97–98 °C. ^1H NMR (CDCl_3 , 500 MHz): δ 7.38–7.33 (m, 2H), 7.15–7.03 (m, 6H), 3.71 (s, 3H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 170.38, 163.74 (d, $J = 251.2$ Hz), 163.58 (d, $J = 250.0$ Hz), 162.00, 152.42, 133.47 (d, $J = 3.5$ Hz), 133.21

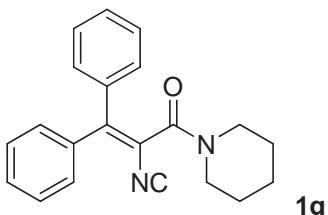
(d, $J = 3.5$ Hz), 132.23 (d, $J = 9.0$ Hz), 131.27 (d, $J = 8.5$ Hz), 115.82 (d, $J = 21.8$ Hz), 115.60 (d, $J = 21.8$ Hz), 113.80, 50.03; ^{19}F NMR (CDCl_3 , 470 MHz): δ -108.57 (m, Ar-F), -110.17 (m, Ar-F).



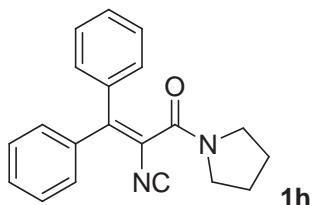
Ethyl 2-isocyano-3,3-diphenylacrylate (1e)^[1]: Pale yellow solid (72% yield for 2 steps). M.p. 104-105 °C. IR (KBr, cm^{-1}): 2112.4, 1721.4, 1444.8, 1323.7, 1260.4, 1116.0, 1009.1, 754.3, 697.5. ^1H NMR (CDCl_3 , 500 MHz): δ 7.44-7.38 (m, 4H), 7.38-7.33 (m, 4H), 7.16-7.12 (m, 2H), 4.11 (q, $J = 7.5$ Hz, 2H), 1.06 (t, $J = 7.5$ Hz, 3H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 169.60, 161.95, 153.90, 138.03, 137.37, 130.24, 129.88, 129.45, 129.10, 128.44, 128.22, 114.35, 62.24, 13.58. EI-MS m/z (%): 277 (25) [M^+], 204 (100), 203 (46), 177 (21), 176 (28). HRMS (ESI) ($[\text{M}+\text{H}]^+$) Calcd. for $\text{C}_{18}\text{H}_{16}\text{NO}_2$: 278.1181; found: 278.1173.



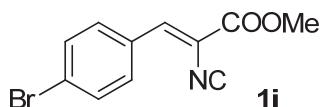
2-Isocyano-1-morpholino-3,3-diphenylprop-2-en-1-one (1f)^[1]: Pale yellow solid (54% yield for 2 steps). M.p. 131-132 °C. IR (KBr, cm^{-1}): 2111.5, 1633.0, 1433.2, 1257.7, 1105.4, 1015.6, 770.9, 697.3. ^1H NMR (CDCl_3 , 500 MHz): δ 7.45-7.30 (m, 8H), 7.21 (d, $J = 8.0$ Hz, 2H), 3.55-3.40 (m, 4H), 3.34-3.24 (m, 2H), 3.20-3.04 (m, 2H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 169.60, 161.82, 146.06, 136.58, 136.42, 130.05, 129.78, 129.73, 129.60, 128.65, 128.53, 114.79, 65.94, 65.91, 46.73, 42.13. EI-MS m/z (%): 318 (80) [M^+], 232 (71), 204 (100), 203 (85), 178 (69). HRMS (ESI) ($[\text{M}+\text{H}]^+$) Calcd. for $\text{C}_{20}\text{H}_{19}\text{N}_2\text{O}_2$: 319.1447; found: 319.1436.



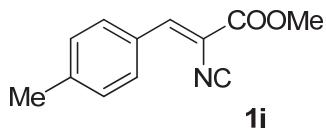
2-Isocyano-3,3-diphenyl-1-(piperidin-1-yl)prop-2-en-1-one (1g)^[1]: Pale yellow solid (69% yield for 2 steps). M.p. 143-144 °C. ^1H NMR (CDCl_3 , 500 MHz): δ 7.40-7.29 (m, 8H), 7.22 (d, $J = 7.5$ Hz, 2H), 3.45 (t, $J = 5.5$ Hz, 2H), 3.29 (t, $J = 5.5$ Hz, 2H), 1.50-1.43 (m, 2H), 1.43-1.35 (m, 2H), 1.30-1.00 (m, 2H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 169.23, 161.49, 145.06, 136.78, 136.75, 129.72, 129.65, 129.58, 129.48, 128.45, 115.87, 47.55, 42.73, 25.49, 24.83, 24.10.



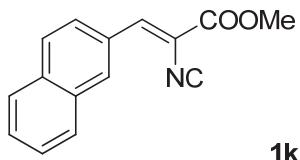
2-Isocyano-3,3-diphenyl-1-(pyrrolidin-1-yl)prop-2-en-1-one (1h)^[1]: Pale yellow solid (65% yield for 2 steps). M.p. 141-143 °C. IR (KBr, cm⁻¹): 2109.0, 1631.9, 1434.8, 1332.5, 1174.1, 771.2, 750.2, 702.8. ¹H NMR (CDCl₃, 500 MHz): δ 7.40-7.26 (m, 8H), 7.22-7.18 (m, 2H), 3.33 (t, J = 6.5 Hz, 2H), 3.20 (t, J = 6.5 Hz, 2H), 1.71-1.65 (m, 4H); ¹³C NMR (CDCl₃, 125 MHz): δ 169.37, 161.15, 145.48, 136.80, 136.74, 129.77, 129.75, 129.53, 129.46, 128.46, 128.40, 117.13, 47.12, 45.77, 25.58, 24.08. EI-MS m/z (%): 302 (100) [M⁺], 204 (70), 178 (68), 176 (59), 77 (96). HRMS (ESI) ([M+H]⁺) Calcd. for C₂₀H₁₉N₂O: 303.1497; found: 303.1488.



Methyl (Z)-3-(4-bromophenyl)-2-isocyanoacrylate (1i)^[1]: White solid (38% yield for 2 steps). M.p. 114-116 °C. IR (KBr, cm⁻¹): 2111.3, 1726.6, 1626.0, 1283.0, 1194.1, 1072.5, 813.4, 758.3. ¹H NMR (CDCl₃, 500 MHz): δ 7.71 (d, J = 9.0 Hz, 2H), 7.61 (s, 1H), 7.58 (d, J = 7.0 Hz, 2H), 3.92 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz): δ 174.71, 161.54, 137.03, 132.47, 132.17, 129.83, 126.72, 114.65, 53.63. EI-MS m/z (%): 267 (31) [M⁺ (⁸¹Br)], 265 (33) [M⁺ (⁷⁹Br)], 207 (32), 186 (100), 127 (94). HRMS (ESI) ([M+H]⁺) Calcd. For C₁₁H₉BrNO₂: 265.9817; found: 265.9805.

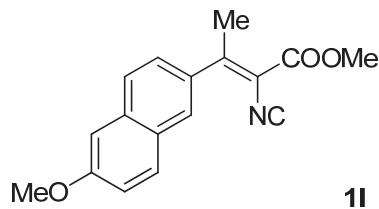


Methyl (Z)-2-isocyano-3-(p-tolyl)acrylate (1j)^[1]: White solid (32% yield for 2 steps). M.p. 80-81 °C. IR (KBr, cm⁻¹): 2115.6, 1727.2, 1613.7, 1282.7, 1201.5, 1088.3, 812.4. ¹H NMR (CDCl₃, 500 MHz): δ 7.77 (d, J = 8.0 Hz, 2H), 7.61 (s, 1H), 7.27 (d, J = 8.5 Hz, 2H), 3.91 (s, 3H), 2.40 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz): δ 173.58, 162.02, 143.03, 138.48, 131.10, 129.88, 128.35, 113.19, 53.40, 21.73. EI-MS m/z (%): 201 (32) [M⁺], 170 (25), 143 (100), 142 (40), 140 (24). HRMS (ESI) ([M+H]⁺) Calcd. for C₁₂H₁₂NO₂: 202.0868; found: 202.0857.

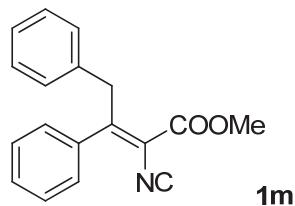


Methyl (Z)-2-isocyano-3-(naphthalen-2-yl)acrylate (1k)^[1]: White solid (45% yield for 2 steps). M.p. 120-121 °C. ¹H NMR (CDCl₃, 500 MHz): δ 8.31 (s, 1H), 8.01 (dd, J = 9.0, 2.0 Hz, 1H), 7.94-7.88 (m, 2H), 7.86 (d, J = 8.5 Hz, 1H), 7.80 (s, 1H), 7.59 (td,

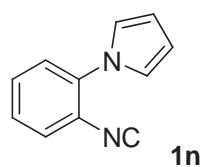
J = 7.0, 1.0 Hz, 1H), 7.55 (td, *J* = 7.0, 1.0 Hz, 1H), 3.95 (s, 3H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 174.03, 161.95, 138.50, 134.71, 132.97, 132.90, 129.14, 128.91, 128.56, 127.80, 127.05, 126.07, 114.07, 53.53.



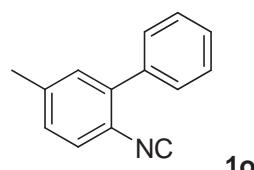
Methyl (Z)-2-isocyano-3-(6-methoxynaphthalen-2-yl)but-2-enoate (1l)^[1]: White solid (44% yield for 2 steps). M.p. 62-63 °C. ^1H NMR (CDCl_3 , 500 MHz): δ 7.80 (s, 1H), 7.77 (t, *J* = 8.0 Hz, 2H), 7.44 (dd, *J* = 8.5, 2.0 Hz, 1H), 7.18 (dd, *J* = 9.0, 2.5 Hz, 1H), 7.16-7.13 (m, 1H), 3.93 (s, 3H), 3.91 (s, 3H), 2.65 (s, 3H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 168.16, 162.10, 158.84, 156.13, 134.93, 134.35, 130.04, 128.23, 127.13, 127.09, 125.09, 119.66, 105.71, 55.42, 52.87, 21.79.



Methyl (Z)-2-isocyano-3,4-diphenylbut-2-enoate (1m)^[1]: Pale yellow solid (42% yield for 2 steps). M.p. 138-139 °C. ^1H NMR (CDCl_3 , 500 MHz): δ 7.38-7.33 (m, 3H), 7.23-7.14 (m, 5H), 7.05 (d, *J* = 6.5 Hz, 2H), 4.37 (s, 2H), 3.93 (s, 3H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 168.64, 161.70, 157.95, 137.81, 136.38, 129.32, 129.04, 128.53, 128.51, 127.48, 126.82, 115.36, 53.11, 39.47.



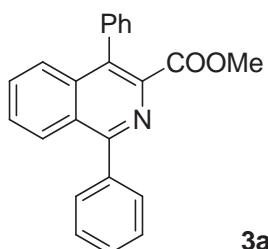
1-(2-isocyanophenyl)-1H-pyrrole (1n)^[2]: Yellow oil. ^1H NMR (CDCl_3 , 500 MHz): δ 7.54-7.45 (m, 2H), 7.40-7.32 (m, 2H), 7.06-7.02 (m, 2H), 6.44-6.39 (m, 2H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 169.76, 137.17, 130.42, 128.73, 127.29, 126.25, 121.40, 110.62.



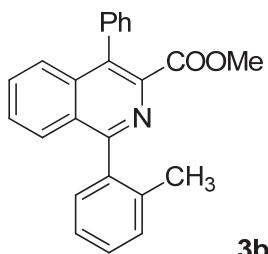
2-isocyano-5-methyl-1,1'-biphenyl (1o)^[3]: Yellow solid. M.p. 95-96 °C. ^1H NMR (CDCl_3 , 500 MHz): δ 7.55-7.48 (m, 4H), 7.46-7.42 (m, 1H), 7.40-7.36 (m, 1H), 7.27-7.23 (m, 1H), 7.19-7.16 (m, 1H), 2.43 (s, 3H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 165.93, 139.94, 138.59, 137.22, 131.20, 129.00, 128.85, 128.56, 128.31, 127.70, 21.41.

3. Synthesis and Characterization for Compounds 3-5.

General Procedure: A test tube equipped with a rubber septum and magnetic stir bar was charged with vinyl isocyanide **1** (0.4 mmol, 1.0 equiv), hydrazines **2** (1.6 mmol, 4.0 equiv), $\text{Mn}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$ (0.08 mmol, 20 mol%) and TBPB (0.448 mL, 6.0 equiv) in dry MeCN (3.0 mL). The tube was evacuated and backfilled with N_2 for three times. The reaction mixture was stirred in a pre-heated oil bath at 70 °C. The reaction was cooled to room temperature, washed with saturated sodium bicarbonate solution (50 mL) and then extracted with EtOAc (3×20 mL). The combined organic layer was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure after filtration. The crude reaction mixture was purified by column chromatography on silica gel to give the desired products **3-5**.

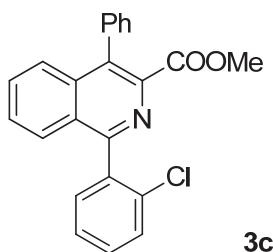


Methyl 1,4-diphenylisoquinoline-3-carboxylate (3a)^[1]: Following the general procedure, methyl 2-isocyano-3,3-diphenylacrylate **1a** (105.2 mg, 0.4 mmol), phenyl hydrazine **2a** (0.157 mL, 1.6 mmol), $\text{Mn}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$ (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 23 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 20:1) to give **3a** (111.2 mg, 82%) as a light yellow solid. M.p. 181–182 °C. IR (KBr, cm^{-1}): 1723.8, 1545.8, 1336.8, 1225.8, 1178.3, 996.3, 761.9, 698.7, 665.7. ^1H NMR (CDCl_3 , 500 MHz): δ 8.18 (d, J = 8.0 Hz, 1H), 7.78–7.75 (m, 2H), 7.71 (d, J = 8.0 Hz, 1H), 7.68–7.64 (m, 1H), 7.64–7.59 (m, 1H), 7.58–7.47 (m, 6H), 7.43–7.40 (m, 2H), 3.71 (s, 3H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 167.69, 160.35, 141.40, 138.88, 136.67, 136.15, 132.92, 130.75, 130.36, 129.97, 129.10, 128.58, 128.45, 128.42, 128.17, 127.91, 127.32, 126.87, 52.55. EI-MS m/z (%): 339 (44) [M^+], 281 (35), 280 (100), 279 (24), 278 (47).

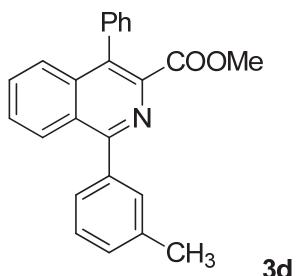


Methyl 4-phenyl-1-(o-tolyl)isoquinoline-3-carboxylate (3b)^[1]: Following the general procedure, methyl 2-isocyano-3,3-diphenylacrylate **1a** (105.2 mg, 0.4 mmol), *o*-tolylhydrazine **2b** (195.2 mg, 1.6 mmol), $\text{Mn}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$ (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 19 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 20:1 to 10:1) to give **3b**

(91.8 mg, 65%) as a yellow solid. M.p. 144-145 °C. IR (KBr, cm^{-1}): 1730.3, 1440.0, 1379.5, 1326.6, 1223.9, 1165.5, 996.8, 767.3, 692.9. ^1H NMR (CDCl_3 , 500 MHz): δ 7.76-7.71 (m, 2H), 7.67-7.63 (m, 1H), 7.58-7.49 (m, 4H), 7.48-7.45 (m, 1H), 7.43-7.39 (m, 3H), 7.38-7.32 (m, 2H), 3.71 (s, 3H), 2.16 (s, 3H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 167.58, 160.83, 141.24, 138.13, 136.76, 136.06, 135.99, 132.89, 130.78, 130.46, 129.92, 129.88, 129.85, 128.83, 128.43, 128.35, 128.29, 128.09, 127.89, 127.68, 126.68, 125.75, 52.50, 20.04. EI-MS m/z (%): 353 (51) [M^+], 352 (45), 293 (38), 292 (100), 291 (64).

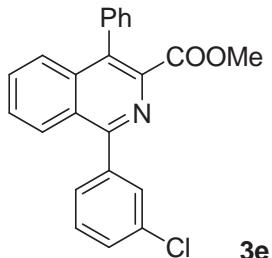


Methyl 1-(2-chlorophenyl)-4-phenylisoquinoline-3-carboxylate (3c): Following the general procedure, methyl 2-isocyano-3,3-diphenylacrylate **1a** (105.2 mg, 0.4 mmol), (2-chlorophenyl)-hydrazine **2c** (227.2 mg, 1.6 mmol), $\text{Mn(OAc)}_2 \cdot 4\text{H}_2\text{O}$ (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 18 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 20:1 to 10:1) to give **3c** (89.5 mg, 60%) as a yellow solid. M.p. 160-161 °C. IR (KBr, cm^{-1}): 1723.2, 1439.3, 1381.6, 1329.6, 1229.9, 1177.7, 998.1, 758.2, 690.5. ^1H NMR (CDCl_3 , 500 MHz): δ 7.72 (d, $J = 8.5$ Hz, 2H), 7.68-7.63 (m, 1H), 7.61-7.58 (m, 1H), 7.58-7.53 (m, 3H), 7.52-7.49 (m, 2H), 7.48-7.44 (m, 3H), 7.41-7.38 (m, 1H), 3.70 (s, 3H). ^{13}C NMR (CDCl_3 , 125 MHz): δ 167.41, 158.18, 141.24, 137.67, 135.99, 135.92, 133.80, 133.57, 131.69, 130.87, 130.24, 129.91, 129.77, 129.72, 128.62, 128.31, 128.14, 127.67, 127.46, 127.03, 126.76, 52.55. EI-MS m/z (%): 375 (11) [$\text{M}^+ ({}^{37}\text{Cl})$], 373 (23) [$\text{M}^+ ({}^{35}\text{Cl})$], 316 (39), 315 (42), 314 (100). HRMS (ESI) ($[\text{M}+\text{H}]^+$) Calcd. for $\text{C}_{23}\text{H}_{17}\text{ClNO}_2$: 374.0948; found: 374.0943.

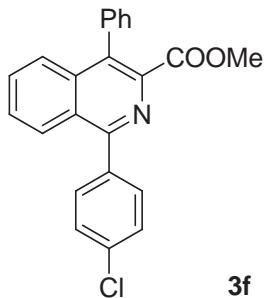


Methyl 4-phenyl-1-(*m*-tolyl)isoquinoline-3-carboxylate (3d)^[1]: Following the general procedure, methyl 2-isocyano-3,3-diphenylacrylate **1a** (105.2 mg, 0.4 mmol), *m*-tolylhydrazine **2d** (195.2 mg, 1.6 mmol), $\text{Mn(OAc)}_2 \cdot 4\text{H}_2\text{O}$ (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 23 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 20:1 to 10:1) to give **3d** (105.3 mg, 75%) as a yellow solid. M.p. 151-152 °C. IR (KBr, cm^{-1}): 1715.3, 1436.5, 1379.1, 1325.4, 1226.3, 1161.3, 1000.0, 767.1, 689.0, 599.6. ^1H NMR (CDCl_3 , 500 MHz): δ 8.20-8.17 (m, 1H), 7.72-7.69 (m, 1H), 7.67-7.63 (m, 1H), 7.63-7.58 (m, 2H),

7.55-7.47 (m, 4H), 7.45-7.40 (m, 3H), 7.33 (d, J = 7.5 Hz, 1H), 3.71 (s, 3H), 2.48 (s, 3H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 167.73, 160.58, 141.39, 138.81, 138.37, 136.58, 136.18, 132.76, 130.89, 130.66, 129.94, 129.79, 128.38, 128.34, 128.29, 128.11, 127.98, 127.42, 127.34, 126.78, 52.51, 21.62. EI-MS m/z (%): 353 (37) [M^+], 294 (100), 278 (39), 105 (77), 77 (72).

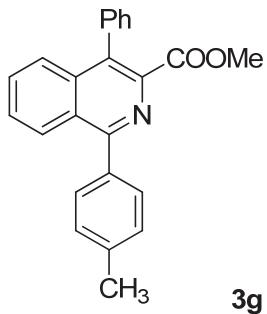


Methyl 1-(3-chlorophenyl)-4-phenylisoquinoline-3-carboxylate (3e)^[1]: Following the general procedure, methyl 2-isocyano-3,3-diphenylacrylate **1a** (105.2 mg, 0.4 mmol), (3-chlorophenyl)-hydrazine **2e** (227.2 mg, 1.6 mmol), $\text{Mn(OAc)}_2 \cdot 4\text{H}_2\text{O}$ (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 16 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 20:1 to 10:1) to give **3e** (111 mg, 74%) as a yellow solid. M.p. 130-131 °C. IR (KBr, cm^{-1}): 1724.4, 1329.3, 1227.3, 762.8, 698.4. ^1H NMR (CDCl_3 , 500 MHz): δ 8.14-8.11 (m, 1H), 7.78-7.76 (m, 1H), 7.74-7.71 (m, 1H), 7.70-7.62 (m, 3H), 7.55-7.47 (m, 5H), 7.42-7.38 (m, 2H), 3.70 (s, 3H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 167.56, 158.72, 141.44, 140.64, 136.68, 135.97, 134.67, 133.43, 130.91, 130.39, 129.90, 129.82, 129.22, 128.76, 128.53, 128.45, 128.26, 127.41, 127.15, 127.02, 52.61. EI-MS m/z (%): 375 (10) [$\text{M}^+ ({}^{37}\text{Cl})$], 373 (26) [$\text{M}^+ ({}^{35}\text{Cl})$], 314 (100), 278 (32), 57 (30).

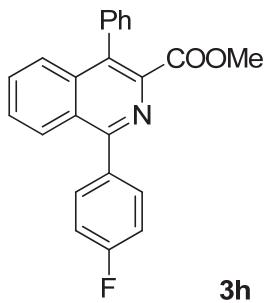


Methyl 1-(4-chlorophenyl)-4-phenylisoquinoline-3-carboxylate (3f)^[1]: Following the general procedure, methyl 2-isocyano-3,3-diphenylacrylate **1a** (105.2 mg, 0.4 mmol), (4-chlorophenyl)-hydrazine **2f** (227.2 mg, 1.6 mmol), $\text{Mn(OAc)}_2 \cdot 4\text{H}_2\text{O}$ (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 18 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 20:1 to 10:1) to give **3f** (103.4 mg, 69%) as a yellow solid. M.p. 174-175 °C. IR (KBr, cm^{-1}): 1715.6, 1597.4, 1491.0, 1437.7, 1326.2, 1225.7, 1170.7, 1086.1, 1003.5, 970.2, 841.7, 767.3, 694.9. ^1H NMR (CDCl_3 , 500 MHz): δ 8.14-8.11 (m, 1H), 7.73-7.71 (m, 3H), 7.69-7.65 (m, 1H), 7.65-7.61 (m, 1H), 7.56-7.48 (m, 5H), 7.42-7.39 (m, 2H), 3.70 (s, 3H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 167.55, 158.92, 141.41, 137.30, 136.58, 135.91, 135.19, 133.06, 131.60, 130.72, 129.80, 128.73, 128.55, 128.33, 128.12, 127.30, 127.04, 126.90, 52.46. EI-MS m/z (%): 375 (20) [$\text{M}^+ ({}^{37}\text{Cl})$], 373 (42) [$\text{M}^+ ({}^{35}\text{Cl})$],

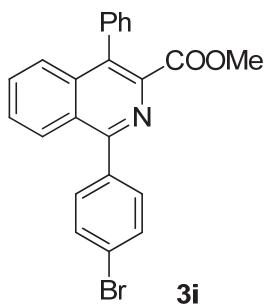
316 (39), 315 (51), 314 (100).



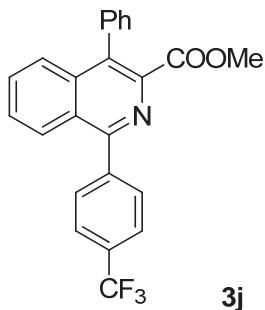
Methyl 4-phenyl-1-(*p*-tolyl)isoquinoline-3-carboxylate (3g)^[1]: Following the general procedure, methyl 2-isocyano-3,3-diphenylacrylate **1a** (105.2mg, 0.4 mmol), *p*-tolylhydrazine **2g** (195.2 mg, 1.6 mmol), Mn(OAc)₂·4H₂O (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 20 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 20:1 to 10:1) to give **3g** (84.8 mg, 60%) as a yellow solid. M.p. 144-145 °C. IR (KBr, cm⁻¹): 1732.1, 1437.0, 1227.0, 1176.3, 1111.6, 999.1, 769.7, 696.4. ¹H NMR (CDCl₃, 500 MHz): δ 8.22-8.19 (m, 1H), 7.71-7.58 (m, 5H), 7.55-7.46 (m, 3H), 7.42-7.39 (m, 2H), 7.36 (d, *J* = 8.0 Hz, 2H), 3.70 (s, 3H), 2.47 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz): δ 167.68, 160.32, 141.34, 138.92, 136.54, 136.13, 135.96, 132.52, 130.54, 130.19, 129.88, 129.15, 128.30, 128.23, 128.01, 127.86, 127.23, 126.71, 52.39, 21.42. EI-MS *m/z* (%): 353 (53) [M⁺], 294 (100), 293 (20).



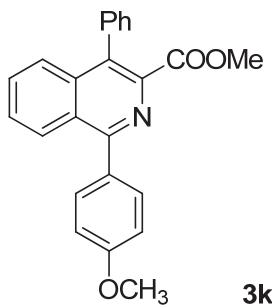
Methyl 1-(4-fluorophenyl)-4-phenylisoquinoline-3-carboxylate (3h)^[1]: Following the general procedure, methyl 2-isocyano-3,3-diphenylacrylate **1a** (105.2mg, 0.4 mmol), (4-fluorophenyl)-hydrazine **2h** (201.6 mg, 1.6 mmol), Mn(OAc)₂·4H₂O (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 16 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 20:1 to 10:1) to give **3h** (94.4 mg, 66%) as a yellow solid. M.p. 179-180 °C. IR (KBr, cm⁻¹): 1721.8, 1504.9, 1380.9, 1224.6, 1163.0, 1001.6, 845.1, 766.7, 693.0. ¹H NMR (CDCl₃, 500 MHz): δ 8.15-8.12 (m, 1H), 7.78-7.74 (m, 2H), 7.73-7.70 (m, 1H), 7.69-7.61 (m, 2H), 7.55-7.49 (m, 3H), 7.42-7.39 (m, 2H), 7.27-7.23 (m, 2H), 3.70 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz): δ 167.57, 163.36 (d, ¹J_{C-F} = 247.5 Hz), 159.11, 141.32, 136.58, 135.95, 134.92 (d, ⁴J_{C-F} = 3.75 Hz), 132.92, 132.10 (d, ³J_{C-F} = 8.75 Hz), 130.67, 129.81, 128.48, 128.32, 128.09, 127.44, 127.13, 126.87, 115.52 (d, ²J_{C-F} = 21.3 Hz), 52.45; ¹⁹F NMR (CDCl₃, 470 MHz): δ -112.5 (m, Ar-F). EI-MS *m/z* (%): 357 (45) [M⁺], 299 (31), 298 (100).



Methyl 1-(4-bromophenyl)-4-phenylisoquinoline-3-carboxylate (3i): Following the general procedure, methyl 2-isocyano-3,3-diphenylacrylate **1a** (105.2 mg, 0.4 mmol), (4-bromophenyl)-hydrazine **2i** (297.6 mg, 1.6 mmol), Mn(OAc)₂·4H₂O (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 23 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 20:1 to 10:1) to give **3i** (116.8 mg, 70%) as a yellow solid. M.p. 169-170 °C. IR (KBr, cm⁻¹): 1713.1, 1433.5, 1385.5, 1224.2, 1169.5, 1002.2, 967.9, 766.4, 693.8. ¹H NMR (CDCl₃, 500 MHz): δ 8.14-8.11 (m, 1H), 7.73-7.61 (m, 7H), 7.55-7.47 (m, 3H), 7.41-7.38 (m, 2H), 3.70 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz): δ 167.52, 158.92, 141.42, 137.73, 136.58, 135.90, 133.09, 131.88, 131.69, 130.77, 129.80, 128.59, 128.35, 128.14, 127.28, 126.98, 126.91, 123.49, 52.47. LC-MS (ESI) m/z: 420 [(M+H)⁺(⁸¹Br)], 418 [(M+H)⁺(⁷⁹Br)]. HRMS (ESI) ([M+H]⁺) Calcd. for C₂₃H₁₇BrNO₂: 418.0443; found: 418.0438.

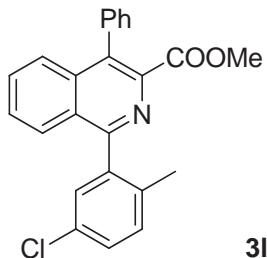


Methyl 4-phenyl-1-(4-(trifluoromethyl)phenyl)isoquinoline-3-carboxylate (3j)^[1]: Following the general procedure, methyl 2-isocyano-3,3-diphenylacrylate **1a** (105.2 mg, 0.4 mmol), (4-(trifluoromethyl)phenyl)hydrazine **2j** (281.6 mg, 1.6 mmol), Mn(OAc)₂·4H₂O (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 23 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 20:1 to 10:1) to give **3j** (113 mg, 69%) as a yellow solid. M.p. 164-165 °C. IR (KBr, cm⁻¹): 1719.9, 1439.9, 1325.8, 1228.9, 1165.4, 1117.0, 1064.6, 1009.4, 851.1, 772.8, 705.4. ¹H NMR (CDCl₃, 500 MHz): δ 8.11-8.07 (m, 1H), 7.90 (d, *J* = 8.0 Hz, 2H), 7.83 (d, *J* = 8.0 Hz, 2H), 7.76-7.73 (m, 1H), 7.71-7.67 (m, 1H), 7.66-7.62 (m, 1H), 7.56-7.50 (m, 3H), 7.44-7.39 (m, 2H), 3.70 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz): δ 167.46, 158.61, 142.40, 141.47, 136.58, 135.79, 133.46, 131.14, 130.88, 130.85, 130.61, 129.77, 128.75, 128.36, 128.19, 127.39, 127.09, 126.99, 125.48 (q, ²J_{C-F} = 3.5 Hz), 124.14 (q, ¹J_{C-F} = 270.0 Hz), 120.89, 52.49; ¹⁹F NMR (CDCl₃, 470 MHz): δ -62.58 (s, Ar-CF₃). EI-MS m/z: 407 (26) [M⁺], 349 (41), 348 (100), 346 (11), 278 (23).



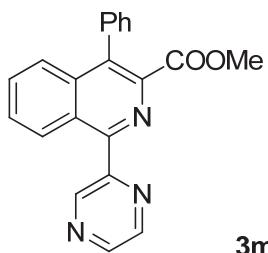
Methyl 1-(4-methoxyphenyl)-4-phenylisoquinoline-3-carboxylate (3k)^[1]:

Following the general procedure, methyl 2-isocyano-3,3-diphenylacrylate **1a** (105.2 mg, 0.4 mmol), (4-methoxy-phenyl)hydrazine **2k** (220.8 mg, 1.6 mmol), Mn(OAc)₂·4H₂O (19.6 mg, 20 mol%) and TBPPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 20 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 20:1 to 10:1) to give **3k** (78.2 mg, 53%) as a yellow solid. M.p. 173-174 °C. IR (KBr, cm⁻¹): 1730.7, 1604.9, 1507.0, 1386.5, 1334.2, 1244.1, 1170.2, 1111.8, 1028.7, 843.2, 769.6, 699.7, 522.0. ¹H NMR (CDCl₃, 500 MHz): δ 8.24-8.21 (m, 1H), 7.75-7.72 (m, 2H), 7.71-7.68 (m, 1H), 7.67-7.59 (m, 2H), 7.54-7.48 (m, 3H), 7.42-7.38 (m, 2H), 7.11-7.06 (m, 2H), 3.91 (s, 3H), 3.70 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz): δ 167.57, 160.42, 159.85, 141.14, 136.64, 136.07, 132.41, 131.71, 131.16, 130.59, 129.86, 128.30, 128.27, 128.01, 127.87, 127.18, 126.74, 113.95, 55.47, 52.42. EI-MS *m/z* (%): 369 (84) [M⁺], 310 (100), 308 (34), 265 (26).

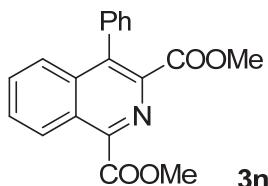


Methyl 1-(5-chloro-2-methylphenyl)-4-phenylisoquinoline-3-carboxylate (3l):

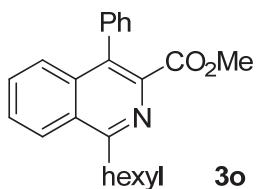
Following the general procedure, methyl 2-isocyano-3,3-diphenylacrylate **1a** (105.2 mg, 0.4 mmol), (5-chloro-2-methylphenyl)hydrazine **2l** (249.6 mg, 1.6 mmol), Mn(OAc)₂·4H₂O (19.6 mg, 20 mol%) and TBPPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 18 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 20:1 to 10:1) to give **3l** (107.8 mg, 70%) as a yellow solid. M.p. 119-120 °C. IR (KBr, cm⁻¹): 1731.9, 1440.9, 1323.5, 1229.3, 1178.2, 1111.5, 999.0, 772.1, 696.7. ¹H NMR (CDCl₃, 500 MHz): δ 7.74-7.70 (m, 2H), 7.69-7.64 (m, *J* = 7.5, 1H), 7.61-7.51 (m, 1H), 7.56-7.49 (m, 3H), 7.46-7.43 (m, 1H), 7.42-7.36 (m, 3H), 7.29 (d, *J* = 8.0 Hz, 1H), 3.71 (s, 3H), 2.11 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz): δ 167.57, 159.36, 141.46, 139.91, 136.18, 135.96, 135.45, 133.42, 131.86, 131.61, 130.99, 129.92, 129.90, 128.92, 128.79, 128.47, 128.38, 128.24, 127.71, 127.31, 126.92, 52.62, 19.57. EI-MS *m/z* (%): 389 (25) [M⁺(³⁷Cl)], 387 (65) [M⁺(³⁵Cl)], 326 (100), 292 (46), 291 (57). HRMS (ESI) ([M+H]⁺) Calcd. for C₂₄H₁₉ClNO₂: 388.1104; found: 388.1099.



Methyl 4-phenyl-1-(pyrazin-2-yl)isoquinoline-3-carboxylate (3m): Following the general procedure, methyl 2-isocyano-3,3-diphenylacrylate **1a** (105.2 mg, 0.4 mmol), 2-hydrazinyl-pyrazine **2m** (176 mg, 1.6 mmol), Mn(OAc)₂·4H₂O (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 23 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 20:1 to 10:1) to give **3m** (71 mg, 52%) as a yellow solid. M.p. 187-188 °C. IR (KBr, cm⁻¹): 1725.5, 1450.1, 1366.7, 1228.7, 1182.3, 1002.9, 762.7, 696.2, 406.7. ¹H NMR (CDCl₃, 500 MHz): δ 9.44 (s, 1H), 8.79-8.72 (m, 3H), 7.75-7.68 (m, 3H), 7.56-7.49 (m, 3H), 7.41-7.38 (m, 2H), 3.73 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz): δ 167.37, 153.82, 153.41, 146.96, 144.10, 143.00, 141.29, 137.19, 135.89, 135.13, 131.04, 129.83, 129.34, 128.46, 128.37, 127.36, 127.12, 52.64. EI-MS m/z (%): 341 (40) [M⁺], 282 (48), 281 (90), 280 (100). HRMS (ESI) ([M+H]⁺) Calcd. for C₂₁H₁₆N₃O₂: 342.1243; found: 342.1236.

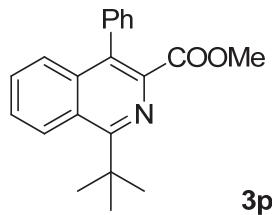


Dimethyl 4-phenylisoquinoline-1,3-dicarboxylate (3n): Following the general procedure, methyl 2-isocyano-3,3-diphenylacrylate **1a** (105.2 mg, 0.4 mmol), methyl hydrazinecarboxylate **2n** (144 mg, 1.6 mmol), Mn(OAc)₂·4H₂O (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 15 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 10:1 to 5:1) to give **3n** (38.9 mg, 30%) as a white solid. M.p. 118-119 °C. IR (KBr, cm⁻¹): 1731.4, 1434.8, 1368.8, 1236.5, 1198.0, 1154.5, 988.5, 762.7, 698.7. ¹H NMR (CDCl₃, 500 MHz): δ 8.85-8.82 (m, 1H), 7.79-7.75 (m, 1H), 7.71-7.68 (m, 2H), 7.52-7.50 (m, 3H), 7.35-7.31 (m, 2H), 4.12 (s, 3H), 3.72 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz): δ 166.56, 165.86, 148.17, 140.48, 137.37, 136.80, 135.48, 131.21, 129.87, 129.45, 128.36, 128.32, 127.01, 126.95, 126.42, 53.25, 52.62. EI-MS m/z (%): 321 (298) [M⁺], 231 (826), 203 (815), 202 (552). HRMS (ESI) ([M+H]⁺) Calcd. for C₁₉H₁₆NO₄: 322.1079; found: 322.1074.

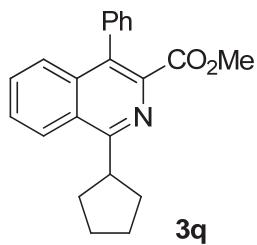


Methyl 1-hexyl-4-phenylisoquinoline-3-carboxylate (3o): Following the general

procedure, methyl 2-isocyano-3,3-diphenylacrylate **1a** (105.2 mg, 0.4 mmol), hexylhydrazine **2o** (186 mg, 1.6 mmol), Mn(OAc)₂·4H₂O (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 23 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 30:1) to give **3o** (79.8 mg, 57%) as a white solid. M.p. 57-58 °C. IR (KBr, cm⁻¹): 1719.1, 1446.6, 1433.4, 1384.8, 1228.9, 1196.2, 1161.8, 1007.8.5, 769.3, 701.5. ¹H NMR (CDCl₃, 500 MHz): δ 8.24 (d, *J* = 8.5 Hz, 1H), 7.68-7.57 (m, 3H), 7.50-7.42 (m, 3H), 7.35-7.31 (m, 2H), 3.68 (s, 3H), 3.41-3.36 (m, 2H), 1.95-1.86 (m, 2H), 1.57-1.49 (m, 2H), 1.41-1.30 (m, 4H), 0.93-0.88 (m, 3H); ¹³C NMR (CDCl₃, 125 MHz): δ 167.98, 162.27, 141.05, 136.35, 135.85, 132.08, 130.29, 129.83, 128.19, 128.16, 127.83, 127.27, 127.14, 125.44, 52.35, 35.91, 31.76, 30.11, 29.70, 22.65, 14.13. LC-MS (ESI) m/z: 348 [(M+H)⁺]. HRMS (ESI) ([M+H]⁺) Calcd. for C₂₃H₂₆NO₂: 348.1964; found: 348.1959.

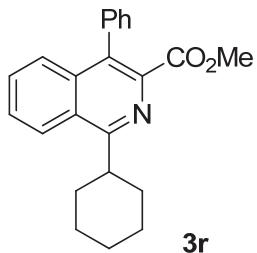


Methyl 1-(*tert*-butyl)-4-phenylisoquinoline-3-carboxylate (3p): Following the general procedure, methyl 2-isocyano-3,3-diphenylacrylate **1a** (105.2 mg, 0.4 mmol), *tert*-butylhydrazine hydrochloride **2p** (198.4 mg, 1.6 mmol), sodium bicarbonate (168 mg, 2.0 mmol), Mn(OAc)₂·4H₂O (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 18 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 60:1) to give **3p** (108.9 mg, 85%) as a white solid. M.p. 118-119 °C. IR (KBr, cm⁻¹): 1729.9, 1441.6, 1361.5, 1235.6, 1160.4, 987.4, 771.6, 697.3, 556.9. ¹H NMR (CDCl₃, 500 MHz): δ 8.63 (d, *J* = 8.5 Hz, 1H), 7.69-7.65 (m, 1H), 7.64-7.60 (m, 1H), 7.59-7.55 (m, 1H), 7.51-7.43 (m, 3H), 7.36-7.30 (m, 2H), 3.67 (s, 3H), 1.73 (s, 9H); ¹³C NMR (CDCl₃, 125 MHz): δ 168.28, 166.75, 139.95, 136.90, 136.50, 131.87, 129.89, 129.25, 128.17, 127.81, 127.78, 127.12, 126.80, 126.57, 52.12, 40.11, 31.19. EI-MS m/z (%): 319 (100) [M⁺], 318 (44), 304 (52), 277 (82), 258 (84). HRMS (ESI) ([M+H]⁺) Calcd. for C₂₁H₂₂NO₂: 320.1651; found: 320.1645.

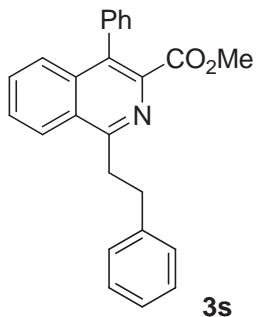


Methyl 1-cyclopentyl-4-phenylisoquinoline-3-carboxylate (3q): Following the general procedure, methyl 2-isocyano-3,3-diphenylacrylate **1a** (105.2 mg, 0.4 mmol), cyclopentyl-hydrazine hydrochloride **2q** (218 mg, 1.6 mmol), sodium bicarbonate (168 mg, 2.0 mmol), Mn(OAc)₂·4H₂O (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 22 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 30:1) to give **3q** (71 mg, 54%) as a light yellow

solid. M.p. 98-99 °C. IR (KBr, cm⁻¹): 1732.2, 1433.6, 1393.6, 1232.2, 1165.2, 991.6, 771.9, 705.6. ¹H NMR (CDCl₃, 500 MHz): δ 8.35-8.32 (m, 1H), 7.67-7.63 (m, 2H), 7.62-7.58 (m, 1H), 7.52-7.44 (m, 3H), 7.38-7.34 (m, 2H), 4.11-4.03 (m, 1H), 3.67 (s, 3H), 2.30-2.16 (m, 4H), 1.99-1.91 (m, 2H), 1.85-1.75 (m, 2H); ¹³C NMR (CDCl₃, 125 MHz): δ 168.36, 164.13, 141.16, 136.51, 135.79, 131.37, 130.01, 129.93, 128.20, 127.90, 127.79, 127.47, 127.04, 125.25, 52.14, 43.39, 32.58, 26.04. LC-MS (ESI) m/z: 332 [(M+H)⁺]. HRMS (ESI) ([M+H]⁺) Calcd. for C₂₂H₂₂NO₂: 332.1651; found: 332.1645.

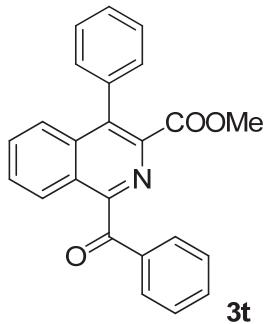


Methyl 1-cyclohexyl-4-phenylisoquinoline-3-carboxylate (3r): Following the general procedure, methyl 2-isocyano-3,3-diphenylacrylate **1a** (105.2 mg, 0.4 mmol), cyclohexylhydrazine **2r** (182 mg, 1.6 mmol), Mn(OAc)₂·4H₂O (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 18 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 30:1) to give **3r** (102 mg, 74%) as a white solid. M.p. 147-148 °C. IR (KBr, cm⁻¹): 1727.9, 1444.3, 1393.6, 1332.6, 1230.3, 1163.8, 996.4, 765.6, 698.9, 599.5, 539.3. ¹H NMR (CDCl₃, 500 MHz): δ 8.33-8.29 (m, 1H), 7.67-7.62 (m, 2H), 7.62-7.57 (m, 1H), 7.51-7.41 (m, 3H), 7.37-7.32 (m, 2H), 3.66 (s, 3H), 3.65-3.57 (m, 1H), 2.10-2.03 (m, 2H), 2.00-1.90 (m, 4H), 1.85-1.80 (m, 1H), 1.61-1.51 (m, 2H), 1.47-1.36 (m, 1H); ¹³C NMR (CDCl₃, 125 MHz): δ 168.42, 165.13, 141.46, 136.50, 135.82, 131.17, 129.94, 129.93, 128.18, 127.88, 127.77, 127.17, 126.61, 124.72, 52.15, 41.82, 32.35, 26.83, 26.16. LC-MS (ESI) m/z: 346 [(M+H)⁺]. HRMS (ESI) ([M+H]⁺) Calcd. for C₂₃H₂₄NO₂: 346.1807; found: 346.1802.

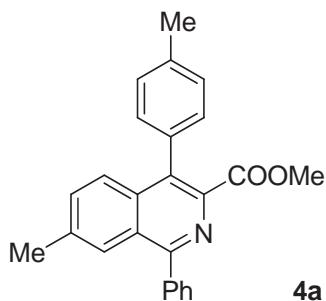


Methyl 1-phenethyl-4-phenylisoquinoline-3-carboxylate (3s): Following the general procedure, methyl 2-isocyano-3,3-diphenylacrylate **1a** (105.2 mg, 0.4 mmol), phenethylhydrazine **2s** (218 mg, 1.6 mmol), Mn(OAc)₂·4H₂O (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 22 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 20:1) to give **3s** (85 mg, 58%) as a white solid. M.p. 117-118 °C. IR (KBr, cm⁻¹): 1721.5, 1647.5, 1554.8, 1510.1, 1226.7, 1162.2, 1009.7, 763.3, 701.6, 427.7. ¹H NMR (CDCl₃, 500 MHz): δ

8.28-8.22 (m, 1H), 7.70-7.60 (m, 3H), 7.54-7.46 (m, 3H), 7.41-7.32 (m, 6H), 7.28-7.22 (m, 1H), 3.76-3.68 (m, 5H), 3.32-3.25 (m, 2H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 168.03, 160.78, 141.83, 141.17, 136.28, 135.86, 132.37, 130.42, 129.87, 128.56, 128.55, 128.37, 128.26, 127.94, 127.33, 127.23, 126.17, 125.14, 52.39, 37.31, 35.41. LC-MS (ESI) m/z: 368 $[(\text{M}+\text{H})^+]$. HRMS (ESI) $([\text{M}+\text{H}]^+)$ Calcd. for $\text{C}_{25}\text{H}_{22}\text{NO}_2$: 368.1651; found: 368.1639.

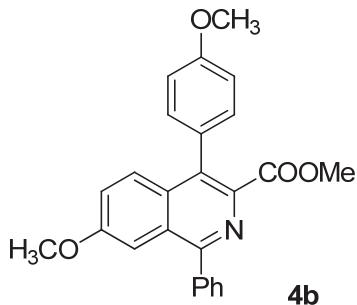


Methyl 1-benzoyl-4-phenylisoquinoline-3-carboxylate (3t): Following the general procedure, methyl 2-isocyano-3,3-diphenylacrylate **1a** (105.2 mg, 0.4 mmol), benzylhydrazine **2t** (195 mg, 1.6 mmol), $\text{Mn}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$ (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 23 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 20:1 to 10:1) to give **3t** (29.4 mg, 20%) as a yellow solid. M.p. 139-140 °C. IR (KBr, cm^{-1}): 1735.5, 1671.0, 1443.2, 1387.0, 1232.0, 1162.9, 995.0, 768.9, 699.9, 634.0. ^1H NMR (CDCl_3 , 500 MHz): δ 8.25-8.20 (m, 1H), 8.09-8.04 (m, 2H), 7.76-7.61 (m, 4H), 7.57-7.48 (m, 5H), 7.43-7.40 (m, 2H), 3.67 (s, 3H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 193.92, 167.14, 155.94, 140.56, 136.44, 136.15, 135.52, 135.29, 134.01, 131.29, 131.05, 129.65, 129.33, 128.57, 128.38, 128.36, 126.91, 126.61, 126.28, 52.52. LC-MS (ESI) m/z: 368 $[(\text{M}+\text{H})^+]$. HRMS (ESI) $([\text{M}+\text{H}]^+)$ Calcd. for $\text{C}_{24}\text{H}_{18}\text{NO}_3$: 368.1287; found: 368.1291.

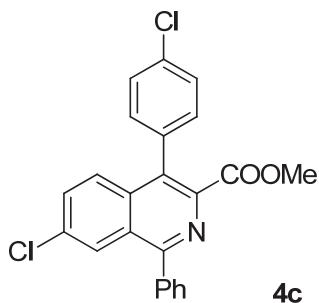


Methyl 7-methyl-1-phenyl-4-(p-tolyl)isoquinoline-3-carboxylate (4a)^[1]: Following the general procedure, methyl 2-isocyano-3,3-di-p-tolylacrylate **1a'** (116.4 mg, 0.4 mmol), phenylhydrazine **2a** (0.157 mL, 1.6 mmol), $\text{Mn}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$ (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 17 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 20:1 to 5:1) to give **4a** (100.3 mg, 68%) as a yellow solid. M.p. 143-144 °C. IR (KBr, cm^{-1}): 1724.9, 1313.7, 1224.7, 1164.0, 1012.2, 770.8, 700.8, 523.8. ^1H NMR (CDCl_3 , 500 MHz): δ 7.90 (s, 1H), 7.76-7.73 (m, 2H), 7.63 (d, $J = 8.5$ Hz, 1H), 7.58-7.46 (m, 4H), 7.35-7.26 (m, 4H), 3.72 (s, 3H), 2.48 (d, $J = 6.0$ Hz, 6H); ^{13}C NMR (CDCl_3 , 125

MHz): δ 167.57, 159.24, 140.28, 138.78, 138.69, 137.68, 134.98, 133.11, 133.06, 132.84, 130.21, 129.67, 129.03, 128.89, 128.45, 127.44, 126.73, 126.57, 52.47, 21.95, 21.43. EI-MS m/z (%): 367 (52) [M^+], 322 (14), 309 (30), 308 (100), 294 (69).

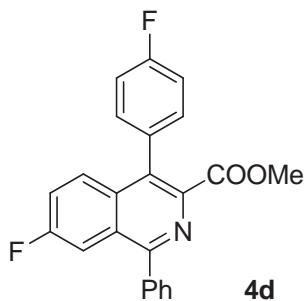


Methyl 7-methoxy-4-(4-methoxyphenyl)-1-phenylisoquinoline-3-carboxylate (4b)^[1]: Following the general procedure, methyl 2-isocyano-3,3-bis(4-methoxyphenyl)acrylate **1b** (129.2 mg, 0.4 mmol), phenylhydrazine **2a** (0.157 mL, 1.6 mmol), $Mn(OAc)_2 \cdot 4H_2O$ (19.6 mg, 20 mol%) and TBPPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 16 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 10:1 to 5:1) to give **4b** (100.8 mg, 63%) as a yellow solid. M.p. 179-180 °C. IR (KBr, cm^{-1}): 1719.9, 1612.6, 1508.7, 1407.4, 1218.0, 1175.7, 1025.6, 846.8, 700.4. 1H NMR ($CDCl_3$, 500 MHz): δ 7.79-7.75 (m, 2H), 7.65 (d, J = 9.0 Hz, 1H), 7.58-7.49 (m, 3H), 7.43 (d, J = 2.5 Hz, 1H), 7.32-7.28 (m, 3H), 7.06-7.03 (m, 2H), 3.90 (s, 3H), 3.81 (s, 3H), 3.73 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 167.83, 159.47, 159.36, 158.35, 139.79, 139.15, 133.00, 132.34, 131.04, 130.06, 128.97, 128.89, 128.67, 128.64, 128.37, 123.10, 113.88, 105.79, 55.59, 55.40, 52.52. EI-MS m/z (%): 399 (99) [M^+], 341 (47), 340 (100), 77 (38).



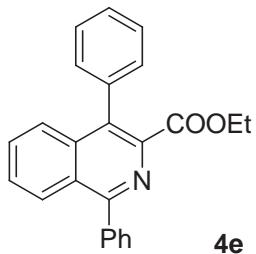
Methyl 7-chloro-4-(4-chlorophenyl)-1-phenylisoquinoline-3-carboxylate (4c)^[1]: Following the general procedure, methyl 3,3-bis(4-chlorophenyl)-2-isocyanoacrylate **1c** (132.8 mg, 0.4 mmol), phenylhydrazine **2a** (0.157 mL, 1.6 mmol), $Mn(OAc)_2 \cdot 4H_2O$ (19.6 mg, 20 mol%) and TBPPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 18 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 20:1 to 10:1) to give **4c** (122.1 mg, 75%) as a yellow solid. M.p. 211-212 °C. IR (KBr, cm^{-1}): 1727.0, 1488.6, 1399.4, 1292.7, 1219.8, 1172.4, 1008.9, 974.3, 837.0, 705.2, 527.8. 1H NMR ($CDCl_3$, 500 MHz): δ 8.15 (t, J = 1.5 Hz, 1H), 7.75-7.71 (m, 2H), 7.61-7.50 (m, 7H), 7.34-7.31 (m, 2H), 3.74 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 167.08, 159.75, 141.44, 138.13, 134.83, 134.78, 134.46, 134.06, 131.73, 131.55, 131.11, 130.08, 129.35, 128.76, 128.71, 128.29, 127.89, 126.64, 52.63; EI-MS m/z (%): 411 (6) [$M^+(2\times^{37}Cl)$], 409 (33) [$M^+(\text{one } ^{35}Cl, ^{37}Cl)$], 407 (47) [M^+

($2 \times ^{35}\text{Cl}$]), 350 (63), 348 (100), 314 (48).



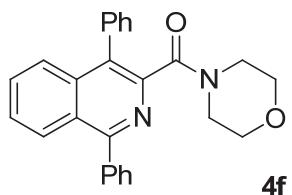
Methyl 7-fluoro-4-(4-fluorophenyl)-1-phenylisoquinoline-3-carboxylate (4d)^[1]:

Following the general procedure, methyl 3,3-bis(4-fluorophenyl)-2-isocyanoacrylate **1d** (119.6 mg, 0.4 mmol), phenylhydrazine **2a** (0.157 mL, 1.6 mmol), Mn(OAc)₂·4H₂O (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 18 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 20:1 to 10:1) to give **4d** (117.5 mg, 78%) as a yellow solid. M.p. 189-190 °C. IR (KBr, cm⁻¹): 1726.1, 1621.6, 1508.0, 1408.0, 1225.7, 1191.9, 1157.7, 848.1, 709.9. ¹H NMR (CDCl₃, 500 MHz): δ 7.80 (dd, *J* = 10.0, 2.5 Hz, 1H), 7.76-7.71 (m, 2H), 7.69 (q, *J* = 5.5 Hz, 1H), 7.60-7.51 (m, 3H), 7.47-7.42 (m, 1H), 7.39-7.34 (m, 2H), 7.27-7.20 (m, 2H), 3.73 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz): δ 167.31, 162.70 (d, ¹J_{C-F} = 246.2 Hz), 161.72 (d, ¹J_{C-F} = 250.0 Hz), 159.80 (d, ⁴J_{C-F} = 5.0 Hz), 141.09, 138.33, 133.70, 131.79, 131.64 (d, ⁴J_{C-F} = 3.8 Hz), 131.50 (d, ³J_{C-F} = 7.5 Hz) 129.97, 129.57 (d, ³J_{C-F} = 8.7 Hz), 129.26, 128.67, 128.41 (d, ³J_{C-F} = 8.7 Hz), 121.10 (d, ²J_{C-F} = 25.0 Hz), 115.56 (d, ²J_{C-F} = 21.2 Hz), 111.43 (d, ²J_{C-F} = 22.5 Hz), 52.55; ¹⁹F NMR (CDCl₃, 470 MHz): δ -108.8 (m, Ar-F), -113.4 (m, Ar-F). EI-MS *m/z* (%): 375 (31) [M⁺], 317 (33), 316 (100), 314 (44), 157 (15).

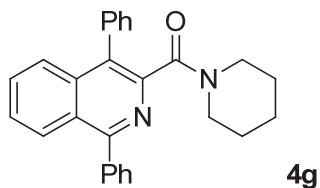


Ethyl 1,4-diphenylisoquinoline-3-carboxylate (4e)^[1]: Following the general procedure, ethyl 2-isocyano-3,3-diphenylacrylate **1e** (110.8 mg, 0.4 mmol), phenylhydrazine **2a** (0.157 mL, 1.6 mmol), Mn(OAc)₂·4H₂O (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 21 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 20:1 to 10:1) to give **4e** (95 mg, 67%) as a yellow solid. M.p. 104-105 °C. IR (KBr, cm⁻¹): 1729.6, 1391.4, 1223.5, 1178.2, 1107.0, 1008.6, 770.1, 701.0. ¹H NMR (CDCl₃, 500 MHz): δ 8.19-8.16 (m, 1H), 7.79-7.76 (m, 2H), 7.73 (d, *J* = 8.0 Hz, 1H), 7.68-7.63 (m, 1H), 7.62-7.58 (m, 1H), 7.58-7.46 (m, 6H), 7.44-7.42 (m, 2H), 4.12 (q, *J* = 7.0 Hz, 2H), 0.99 (t, *J* = 7.0 Hz, 3H); ¹³C NMR (CDCl₃, 125 MHz): δ 167.48, 160.27, 142.14, 138.78, 136.42, 136.09, 132.00, 130.60, 130.27, 130.05, 128.93, 128.41, 128.28, 128.18, 128.04, 127.80, 127.07, 126.58, 61.32, 13.70; EI-MS *m/z* (%): 353 (21) [M⁺], 281 (33), 280

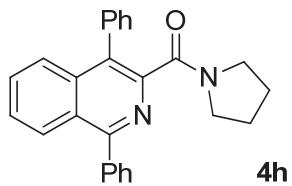
(100), 279 (15), 277 (18).



(1,4-Diphenylisoquinolin-3-yl)(morpholino)methanone (4f)^[1]: Following the general procedure, 2-isocyano-1-morpholino-3,3-diphenylprop-2-en-1-one **1f** (127.2 mg, 0.4 mmol), phenylhydrazine **2a** (0.157 mL, 1.6 mmol), Mn(OAc)₂·4H₂O (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 23 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 3:1 to 1:1) to give **4f** (115.8 mg, 73%) as a yellow solid after. M.p. 243-244 °C. IR (KBr, cm⁻¹): 1637.7, 1432.7, 1272.2, 1230.5, 1109.1, 988.0, 778.3, 701.1. ¹H NMR (CDCl₃, 500 MHz): δ 8.16 (d, *J* = 8.5 Hz, 1H), 7.81 (d, *J* = 8.5 Hz, 1H), 7.77-7.73 (m, 2H), 7.69-7.64 (m, 1H), 7.60-7.48 (m, 9H), 3.58 (t, *J* = 4.5 Hz, 2H), 3.44 (t, *J* = 4.5 Hz, 2H), 3.19-3.18 (m, 4H); ¹³C NMR (CDCl₃, 125 MHz): δ 167.64, 160.69, 145.03, 138.75, 135.96, 134.72, 130.72, 130.64, 130.19, 128.94, 128.86, 128.60, 128.54, 128.43, 127.87, 127.68, 126.56, 125.89, 66.44, 66.42, 46.80, 41.61; EI-MS *m/z* (%): 394 (9) [M⁺], 281 (54), 280 (100), 278 (12), 202 (12).

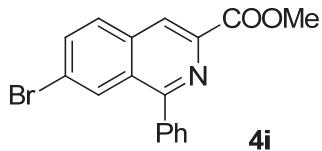


(1,4-Diphenylisoquinolin-3-yl)(piperidin-1-yl)methanone (4g)^[1]: Following the general procedure, 2-isocyano-3,3-diphenyl-1-(piperidin-1-yl)prop-2-en-1-one **1g** (126.4 mg, 0.4 mmol), phenylhydrazine **2a** (0.157 mL, 1.6 mmol), Mn(OAc)₂·4H₂O (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 19 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 5:1 to 3:1) to give **4g** (112 mg, 71%) as a yellow solid. M.p. 214-215 °C. IR (KBr, cm⁻¹): 1744.3, 1631.3, 1433.1, 1377.8, 1245.2, 1024.1, 985.9, 758.2, 698.0. ¹H NMR (CDCl₃, 500 MHz): δ 8.14 (d, *J* = 8.5 Hz, 1H), 7.79 (d, *J* = 8.5 Hz, 1H), 7.77-7.73 (m, 2H), 7.66-7.66 (m, 1H), 7.58-7.45 (m, 9H), 3.51 (t, *J* = 5.0 Hz, 2H), 3.11 (t, *J* = 5.0 Hz, 2H), 1.48-1.41 (m, 2H), 1.38-1.34 (m, 2H), 1.20-1.10 (m, 2H); ¹³C NMR (CDCl₃, 125 MHz): δ 167.35, 160.38, 146.11, 138.88, 136.11, 134.87, 130.77, 130.52, 130.24, 128.81, 128.50, 128.36, 128.24, 127.79, 127.37, 126.40, 125.93, 47.54, 42.10, 25.81, 25.13, 24.32. EI-MS *m/z* (%): 392 (9) [M⁺], 281 (51), 280 (100), 208 (53), 256 (40).

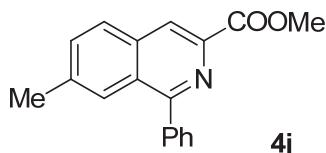


(1,4-diphenylisoquinolin-3-yl)(pyrrolidin-1-yl)methanone (4h)^[1]: Following the general procedure, 2-isocyano-3,3-diphenyl-1-(pyrrolidin-1-yl)prop-2-en-1-one **1h**

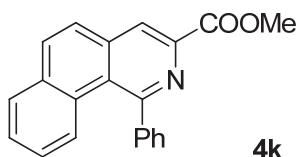
(120.8 mg, 0.4 mmol), phenylhydrazine **2a** (0.157 mL, 1.6 mmol), $\text{Mn}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$ (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 19 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 10:1 to 2:1) to give **4h** (98 mg, 65%) as a yellow solid. M.p. 232-233 °C. IR (KBr, cm^{-1}): 1741.0, 1634.1, 1458.4, 1377.4, 1177.0, 765.7, 705.5. ^1H NMR (CDCl_3 , 500 MHz): δ 8.13 (d, J = 8.5 Hz, 1H), 7.79 (d, J = 8.5 Hz, 1H), 7.76-7.72 (m, 2H), 7.64-7.59 (m, 1H), 7.56-7.42 (m, 9H), 3.38 (t, J = 6.5 Hz, 2H), 3.16 (t, J = 6.0 Hz, 2H), 1.73-1.61 (m, 4H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 167.40, 160.48, 147.01, 139.13, 136.17, 135.02, 130.46, 130.43, 130.14, 128.74, 128.52, 128.38, 128.36, 128.22, 127.76, 127.40, 126.52, 125.91, 47.44, 45.11, 25.75, 24.25. EI-MS m/z (%): 378 (4) [M^+], 309 (19), 281 (47), 280 (100), 277 (13).



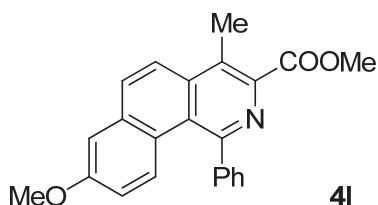
Methyl 7-bromo-1-phenylisoquinoline-3-carboxylate (4i)^[1]: Following the general procedure, methyl (Z)-3-(4-bromophenyl)-2-isocyanoacrylate **1i** (106.4 mg, 0.4 mmol), phenylhydrazine **2a** (0.157 mL, 1.6 mmol), $\text{Mn}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$ (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 14 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 20:1 to 10:1) to give **4i** (42.4 mg, 31%) as a yellow solid. M.p. 205-206 °C. IR (KBr, cm^{-1}): 1714.2, 1443.7, 1282.5, 1237.5, 1102.8, 1001.0, 810.1, 692.8. ^1H NMR (CDCl_3 , 500 MHz): δ 8.55 (s, 1H), 8.28 (s, 1H), 7.90 (d, J = 8.5 Hz, 1H), 7.85 (dd, J = 8.5, 2.0 Hz, 1H), 7.71-7.68 (m, 2H), 7.58-7.52 (m, 3H), 4.04 (s, 3H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 166.26, 160.34, 141.28, 138.25, 135.12, 134.38, 130.10, 130.06, 130.01, 129.26, 129.13, 128.67, 123.75, 122.90, 52.98. EI-MS m/z (%): 343 (28) [$\text{M}^+({}^{81}\text{Br})$], 341 (32) [$\text{M}^+({}^{79}\text{Br})$], 342 (20), 283 (100), 203 (62).



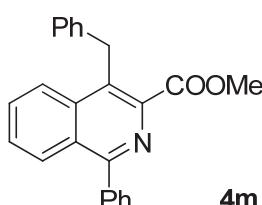
Methyl 7-methyl-1-phenylisoquinoline-3-carboxylate (4j)^[1]: Following the general procedure, methyl (Z)-2-isocyano-3-(*p*-tolyl)acrylate **1j** (80.4 mg, 0.4 mmol), phenylhydrazine **2a** (0.157 mL, 1.6 mmol), $\text{Mn}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$ (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 21 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 20:1 to 10:1) to give **4j** (45.7 mg, 41%) as a yellow solid. M.p. 179-180 °C. IR (KBr, cm^{-1}): 1712.8, 1445.7, 1322.5, 1298.6, 1239.4, 1002.7, 815.0, 758.7, 699.5. ^1H NMR (CDCl_3 , 500 MHz): δ 8.55 (s, 1H), 7.94 (d, J = 8.5 Hz, 1H), 7.88 (s, 1H), 7.72-7.70 (m, 2H), 7.62 (dd, J = 8.5, 1.5 Hz, 1H), 7.59-7.50 (m, 3H), 4.04 (s, 3H), 2.52 (s, 3H); ^{13}C NMR (CDCl_3 , 125 MHz): δ 166.72, 160.49, 140.15, 139.95, 139.13, 134.78, 132.93, 130.11, 128.79, 128.49, 128.41, 128.29, 126.66, 123.26, 52.82, 22.24. EI-MS m/z (%): 277 (28) [M^+], 219 (100), 218 (24), 217 (30), 216 (33).



Methyl 1-phenylbenzo[h]isoquinoline-3-carboxylate (4k)^[1]: Following the general procedure, methyl (Z)-2-isocyano-3-(naphthalen-2-yl)acrylate **1k** (94.8 mg, 0.4 mmol), phenylhydrazine **2a** (0.157 mL, 1.6 mmol), Mn(OAc)₂·4H₂O (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 21 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 20:1 to 10:1) to give **4k** (56.6 mg, 45%) as a yellow solid. M.p. 193-194 °C. IR (KBr, cm⁻¹): 1711.3, 1445.8, 1349.4, 1257.7, 1219.5, 992.4, 827.9, 756.9, 699.0. ¹H NMR (CDCl₃, 500 MHz): δ 8.55 (s, 1H), 8.0 (d, *J* = 8.5 Hz, 1H), 7.89 (d, *J* = 7.5 Hz, 1H), 7.82 (d, *J* = 9.0 Hz, 1H), 7.77 (d, *J* = 8.5 Hz, 1H), 7.60-7.58 (m, 2H), 7.55-7.52 (m, 1H), 7.49 (t, *J* = 3.0 Hz, 3H), 7.24-7.21 (m, 1H), 4.05 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz): δ 166.26, 159.40, 143.33, 141.61, 138.34, 134.34, 132.77, 129.30, 129.21, 129.11, 128.76, 128.74, 128.22, 127.70, 126.35, 126.21, 125.63, 123.03, 52.92. EI-MS *m/z* (%): 313 (41) [M⁺], 255 (100), 254 (43), 252 (71), 105 (23).

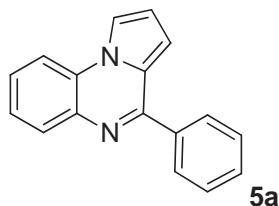


Methyl 8-methoxy-4-methyl-1-phenylbenzo[h]isoquinoline-3-carboxylate (4l)^[1]: Following the general procedure, methyl (Z)-2-isocyano-3-(6-methoxy-naphthalen-2-yl)but-2-enoate **1l** (112.4 mg, 0.4 mmol), phenylhydrazine **2a** (0.157 mL, 1.6 mmol), Mn(OAc)₂·4H₂O (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 23 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 20:1 to 10:1) to give **4l** (85.4 mg, 60%) as a yellow solid. M.p. 181-182 °C. IR (KBr, cm⁻¹): 1708.6, 1609.2, 1504.4, 1448.2, 1346.8, 1219.9, 1206.8, 1137.4, 1021.5, 847.6, 708.5, 623.9. ¹H NMR (CDCl₃, 500 MHz): δ 8.02 (d, *J* = 9.0 Hz, 1H), 7.93 (d, *J* = 9.0 Hz, 1H), 7.66 (d, *J* = 9.5 Hz, 1H), 7.57-7.54 (m, 2H), 7.47-7.45 (m, 3H), 7.23-7.21 (m, 1H), 6.82 (dd, *J* = 9.5, 2.5 Hz, 1H), 4.01 (s, 3H), 3.90 (s, 3H), 2.91 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz): δ 168.02, 158.50, 155.84, 143.63, 141.50, 136.66, 135.13, 131.71, 130.08, 129.21, 129.13, 128.45, 127.67, 125.01, 123.70, 122.02, 116.50, 108.34, 55.36, 52.66, 14.79. EI-MS *m/z* (%): 357 (90) [M⁺], 299 (100), 297 (73), 254 (36).

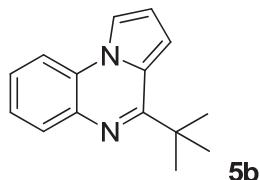


Methyl 4-benzyl-1-phenylisoquinoline-3-carboxylate (4m)^[1]: Following the general procedure, methyl (Z)-2-isocyano-3,4-diphenylbut-2-enoate **1m** (110.8 mg,

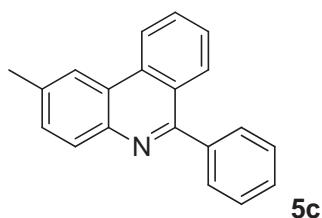
0.4 mmol), phenylhydrazine **2a** (0.157 mL, 1.6 mmol), Mn(OAc)₂·4H₂O (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 22 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 20:1 to 10:1) to give **4m** (75.7 mg, 54%) as a yellow solid. M.p. 96-97 °C. IR (KBr, cm⁻¹): 1722.7, 1441.5, 1380.9, 1248.0, 1213.6, 1070.9, 990.9, 771.4, 703.0. ¹H NMR (CDCl₃, 500 MHz): δ 8.12 (t, J = 8.5 Hz, 2H), 7.74-7.71 (m, 2H), 7.69-7.65 (m, 1H), 7.59-7.48 (m, 4H), 7.28-7.21 (m, 4H), 7.20-7.16 (m, 1H), 4.76 (s, 2H), 3.96 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz): δ 168.06, 159.77, 142.34, 139.91, 138.95, 136.50, 130.67, 130.23, 129.63, 128.87, 128.56, 128.44, 128.36, 128.15, 127.61, 126.18, 125.22, 52.83, 33.89. EI-MS m/z (%): 353 (67) [M⁺], 321 (100), 320 (57), 293 (24), 292 (72).



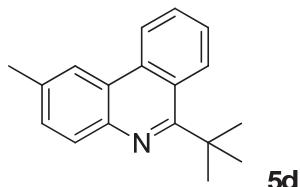
4-Phenylpyrrolo[1,2-a]quinoxaline (5a)^[4]: Following the general procedure, 1-(2-isocyano-phenyl)-1*H*-pyrrole **1n** (67.2 mg, 0.4 mmol), phenylhydrazine **2a** (0.157 mL, 1.6 mmol), Mn(OAc)₂·4H₂O (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 21 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 30:1) to give **5a** (43.1 mg, 44%) as a yellow solid. M.p. 96-97 °C. IR (KBr, cm⁻¹): 1524.3, 1462.9, 1363.7, 1317.3, 751.6, 698.9. ¹H NMR (CDCl₃, 500 MHz): δ 8.07-8.03 (m, 1H), 8.02-7.98 (m, 3H), 7.91-7.86 (m, 1H), 7.58-7.50 (m, 4H), 7.49-7.44 (m, 1H), 7.02-6.98 (m, 1H), 6.92-6.88 (m, 1H); ¹³C NMR (CDCl₃, 125 MHz): δ 154.44, 138.50, 136.30, 130.28, 129.81, 128.63, 128.60, 127.49, 127.19, 125.42, 125.29, 114.61, 113.99, 113.65, 108.71. LC-MS (ESI) m/z: 245 [(M+H)⁺].



4-(tert-Butyl)pyrrolo[1,2-a]quinoxaline (5b)^[2]: Following the general procedure, 1-(2-isocyano-phenyl)-1*H*-pyrrole **1n** (67.2 mg, 0.4 mmol), *tert*-butylhydrazine hydrochloride **2p** (198.4 mg, 1.6 mmol), sodium bicarbonate (168 mg, 2.0 mmol), Mn(OAc)₂·4H₂O (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 27 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 200:1) to give **5b** (32 mg, 36%) as a light yellow oil. IR (KBr, cm⁻¹): 1712.2, 1604.8, 1478.1, 1454.5, 1359.7, 1325.6, 1193.1, 1106.4, 1043.4, 943.5, 754.3, 712.4, 405.6. ¹H NMR (CDCl₃, 500 MHz): δ 7.97-7.93 (m, 1H), 7.90-7.86 (m, 1H), 7.84-7.80 (m, 1H), 7.49-7.38 (m, 2H), 7.07-7.03 (m, 1H), 6.86-6.83 (m, 1H), 1.60 (s, 9H); ¹³C NMR (CDCl₃, 125 MHz): δ 162.95, 135.38, 130.11, 127.12, 127.10, 124.83, 123.93, 113.34, 113.04, 112.72, 108.33, 39.32, 29.51. LC-MS (ESI) m/z: 225 [(M+H)⁺].

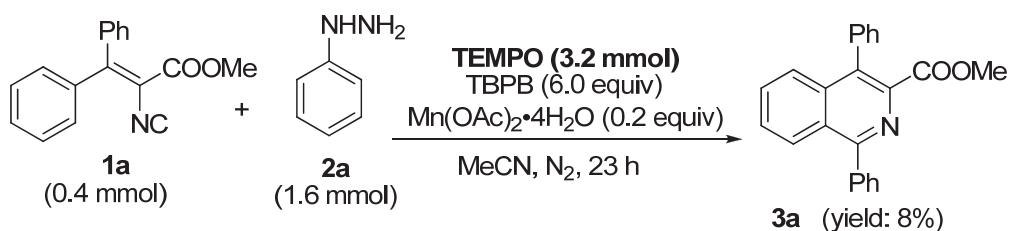


2-Methyl-6-phenylphenanthridine (5c)^[3]: Following the general procedure, 2-isocyano-5-methyl-1,1'-biphenyl **1o** (77.2 mg, 0.4 mmol), phenylhydrazine **2a** (0.157 mL, 1.6 mmol), Mn(OAc)₂·4H₂O (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 25 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 30:1) to give **5c** (60.6 mg, 56%) as a yellow solid. M.p. 86-87 °C. IR (KBr, cm⁻¹): 1551.3, 1359.1, 826.2, 767.9, 699.3, 578.6. ¹H NMR (CDCl₃, 500 MHz): δ 8.68 (d, *J* = 8.5 Hz, 1H), 8.39 (s, 1H), 8.15 (d, *J* = 8.5 Hz, 1H), 8.12-8.07 (m, 1H), 7.86-7.80 (m, 1H), 7.76-7.71 (m, 2H), 7.62-7.50 (m, 5H), 2.65 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz): δ 160.32, 142.13, 139.93, 136.83, 133.19, 130.59, 130.33, 130.09, 129.78, 128.84, 128.61, 128.43, 126.97, 125.31, 123.58, 122.17, 121.58, 22.05. LC-MS (ESI) m/z: 270 [(M+H)⁺].



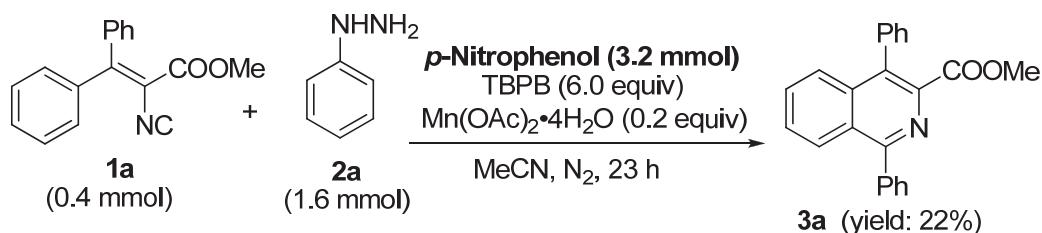
6-(*tert*-butyl)-2-methylphenanthridine (5d**)**: Following the general procedure, 2-isocyano-5-methyl-1,1'-biphenyl **1o** (77.2 mg, 0.4 mmol), *tert*-butylhydrazine hydrochloride **2p** (198.4 mg, 1.6 mmol), sodium bicarbonate (168 mg, 2.0 mmol), Mn(OAc)₂·4H₂O (19.6 mg, 20 mol%) and TBPB (0.448 mL) were stirred in dry MeCN (3.0 mL) at 70 °C for 27 h. Purification by chromatography on silica gel (eluent: hexane/EtOAc = 200:1) to give **5d** (77.5 mg, 78%) as a light yellow oil. IR (KBr, cm⁻¹): 1711.8, 1523.9, 1482.7, 1446.6, 1360.1, 1190.9, 1103.6, 1043.1, 945.4, 756.1, 711.5. ¹H NMR (CDCl₃, 500 MHz): δ 8.67 (q, 2H), 8.33 (s, 1H), 8.09 (d, *J* = 8.0 Hz, 1H), 7.74-7.80 (m, 1H), 7.69-7.62 (m, 1H), 7.58-7.54 (m, 1H), 2.64 (s, 3H), 1.80 (s, 9H); ¹³C NMR (CDCl₃, 125 MHz): δ 165.64, 141.34, 136.19, 133.86, 130.11, 130.10, 129.06, 128.24, 125.81, 124.44, 123.27, 122.99, 121.32, 40.14, 31.30, 22.05. LC-MS (ESI) m/z: 250 [(M+H)⁺].

4. Mechanistic Studies



The tube containing a mixture of methyl 2-isocyano-3,3-diphenylacrylate (**1a**) (105.2

mg, 0.4 mmol), phenyl hydrazine (**2a**) (0.157 mL, 1.6 mmol), Mn(OAc)₂·4H₂O (19.6 mg, 0.2 equiv) and 2,2,6,6-tetramethyl-piperidine-1-oxy (TEMPO) (500.0 mg, 3.2 mmol) in dry MeCN (3.0 mL) was evacuated and backfilled with N₂ for three times. The reaction mixture was stirred in a pre-heated oil bath at 70 °C for 23 h. The reaction was cooled to room temperature, washed with saturated sodium bicarbonate solution (50 mL) and then extracted with EtOAc (3×20 mL). The combined organic layers were dried over Na₂SO₄ and then purified by chromatography on silica gel (eluent: PE/EA = 20:1) to give the product **3a** (10.8 mg, 8%).



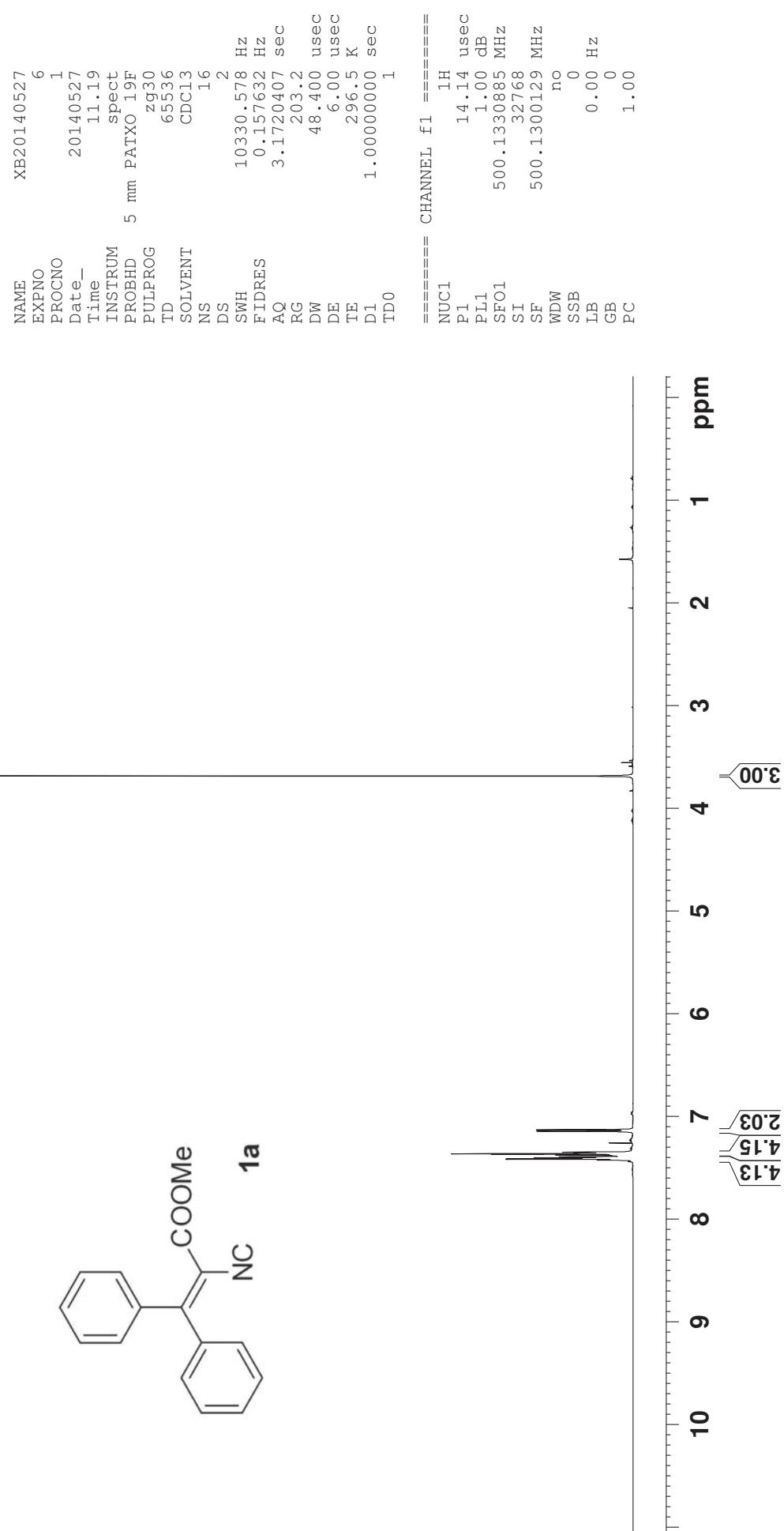
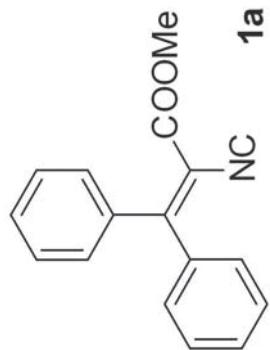
The tube containing a mixture of methyl 2-isocyano-3,3-diphenylacrylate (**1a**) (105.2 mg, 0.4 mmol), Phenyl hydrazine (**2a**) (0.157 mL, 1.6 mmol), Mn(OAc)₂·4H₂O (19.6 mg, 0.2 equiv.) and *p*-nitrophenol (445.2 mg, 3.2 mmol) in dry MeCN (3.0 mL) was evacuated and backfilled with N₂ for three times. The reaction mixture was stirred in a pre-heated oil bath at 70 °C for 23 h. The reaction was cooled to room temperature, washed with saturated sodium bicarbonate solution (50 mL) and then extracted with EtOAc (3×20 mL). The combined organic layers were dried over Na₂SO₄ and then purified by chromatography on silica gel (eluent: PE/EA = 10:1) to give the product **3a** (29.9 mg, 22%).

5. References:

- (1) H. Wang, Y. Yu, X. Hong, Y. Zhang and B. Xu, *Chem. Commun.*, 2014, **50**, 13485.
- (2) Z. He, M. Bae, J. Wu, and T. F. Jamison, *Angew. Chem., Int. Ed.*, 2014, **53**, 14451.
- (3) M. Tobisu, K. Koh, T. Furukawa and N. Chatani, *Angew. Chem. Int. Ed.*, 2012, **51**, 11363.
- (4) Z. Zhang, J. Li, G. Zhang, N. Ma, Q. Liu and T. Liu, *J. Org. Chem.*, 2015, **80**, 6875.
- (5) R. Haridharan, K. Muralirajan and C. H. Cheng, *Adv. Synth. Catal.*, 2015, **357**, 366.

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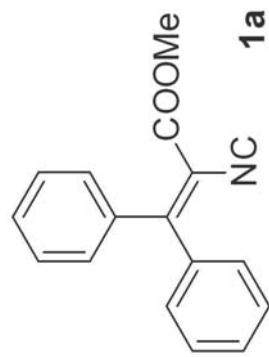
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Date_        20140529
Time       18.09
INSTRUM      spect
PROBHD      5 mm PATXO 19F
PULPROG     zgppg30
TD           65536
SOLVENT      CDCl3
NS            512
DS            4
SWH         30030.029 Hz
FIDRES      0.458222 Hz
AQ           1.0912410 sec
RG           181
DW           16.650 usec
DE           6.000 usec
TE           298.3 K
D1           2.0000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0           1

=====
CHANNEL f1 =====
NUC1          13C
P1             9.50 usec
PL1           -0.50 dB
SFO1        125.7703643 MHz

=====
CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           1.00 dB
PL12          16.05 dB
PL13          16.50 dB
SFO2        500.11320005 MHz
SI            32768
SF           125.7577890 MHz
WDW          no
SSB           0
LB            0.00 Hz
GB            0
PC           1.40

```

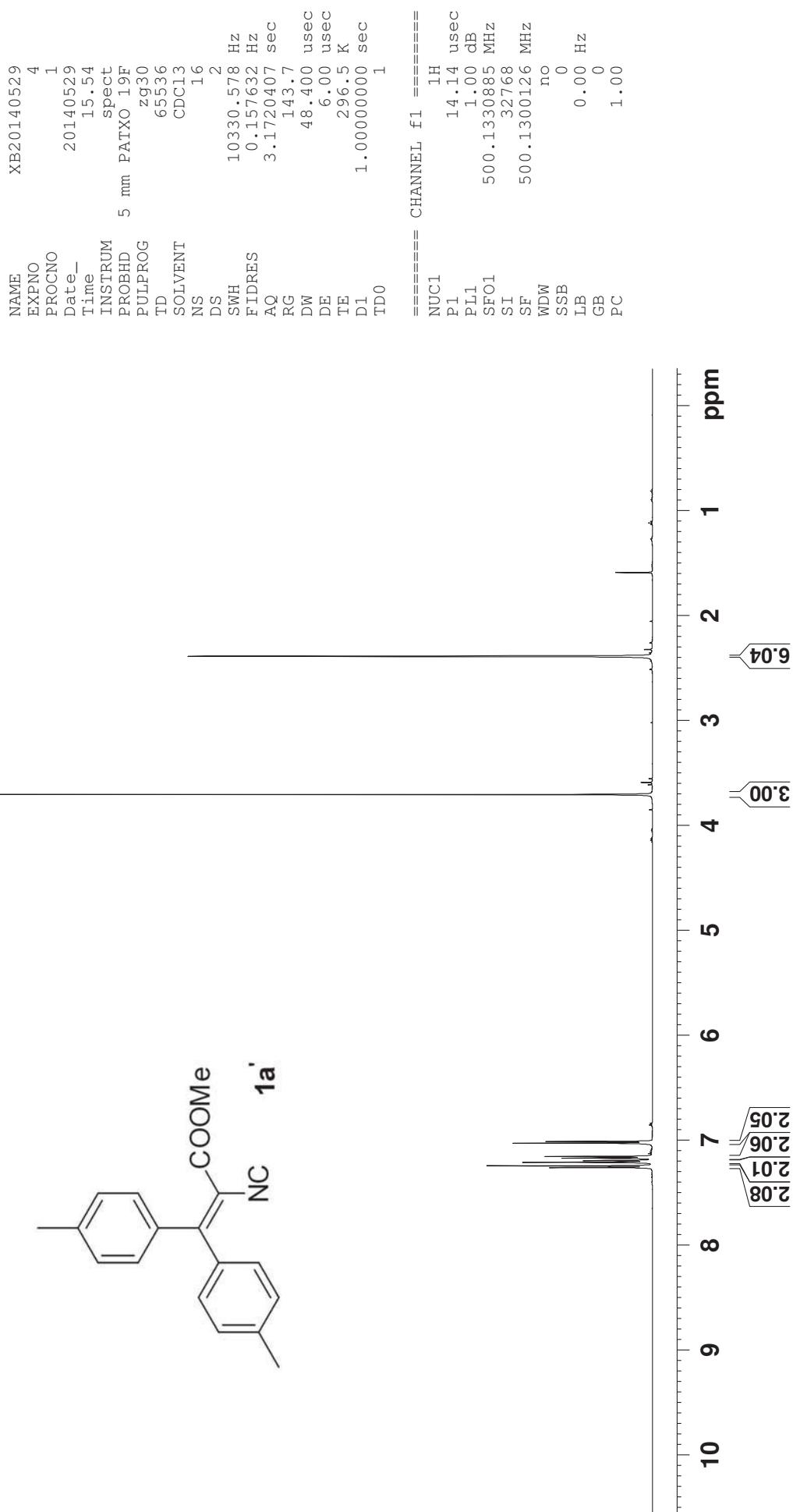
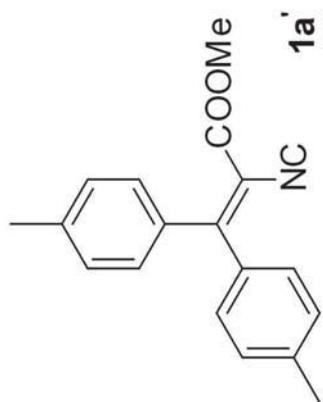


YY-1-90
PROTON CDCl₃

2.387
2.384

3.704

7.011
7.027
7.154
7.170
7.194
7.210
7.243
7.247
7.260

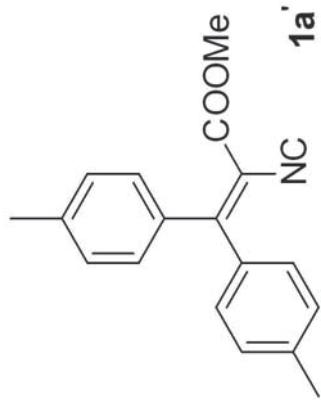


140.79
 135.08
 134.75
 130.12
 129.32
 129.08
 128.92

— 52.83 —

YY-1-90
C13CPD CDCL3

21.49
— V —



169.14
 162.60
 155.10

```

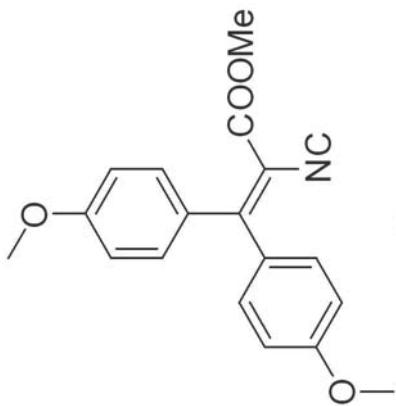
NAME          XB20140529
EXPNO         6
PROCNO        1
Date_         20140529
Time          16.25
INSTRUM      spect
PROPHD        5 mm PATXO 19F
PULPROG      zgpg30
TD            65536
SOLVENT       CDCl3
NS            512
DS            4
SWH           30030.029 Hz
SF             0.458222 Hz
FIDRES       1.0912410 sec
RG            322.5
DW            16.650 usec
DE            6.00 usec
TE            298.1 K
T1            2.0000000 sec
D1            0.0300000 sec
d1            1.89999998 sec
DELTA         1.89999998 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            9.50 usec
PL1           -0.50 dB
SFO1         125.7703643 MHz

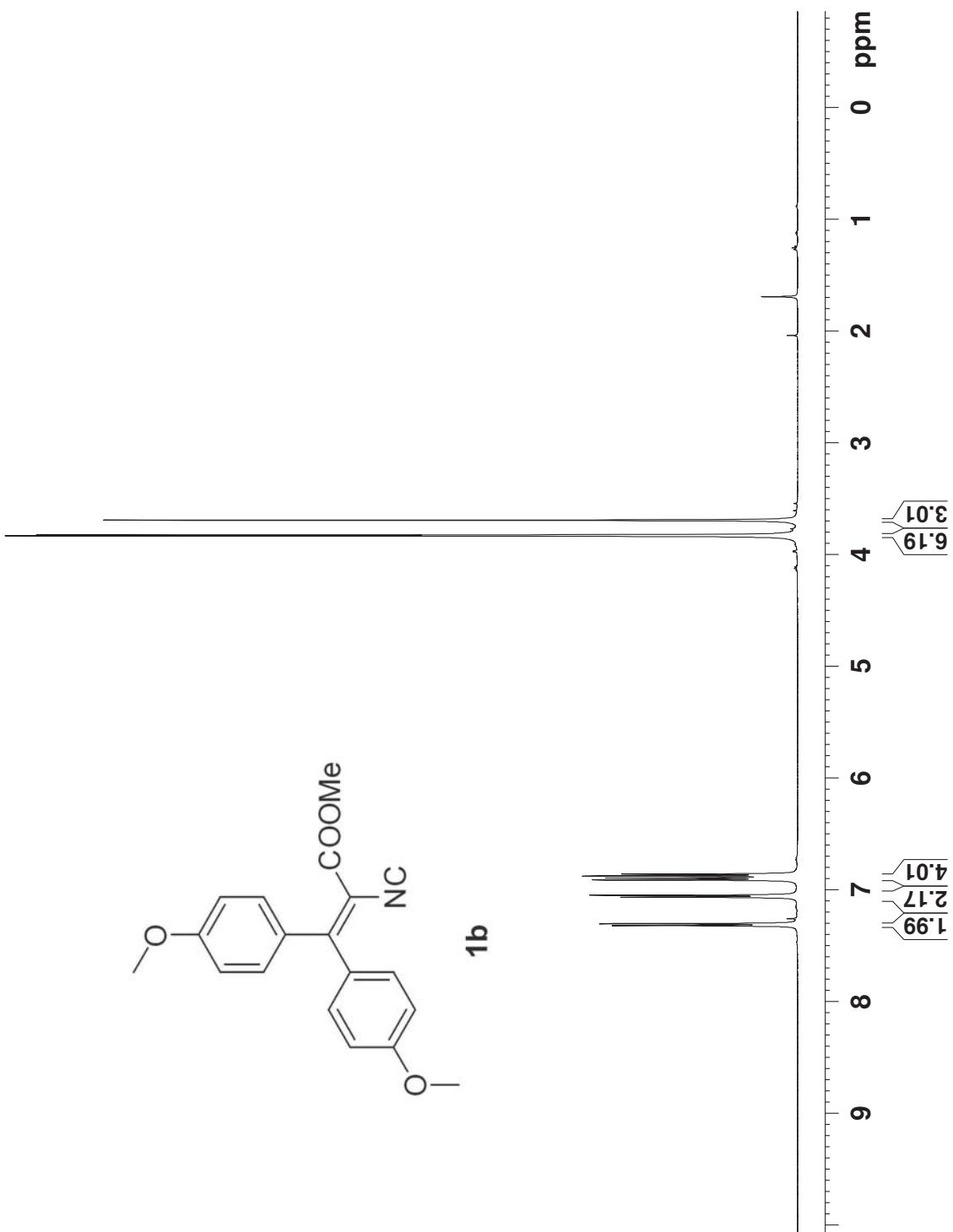
===== CHANNEL f2 =====
CPDRG2        waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           1.00 dB
PL12          16.05 dB
PL13          16.50 dB
SFO2         500.1320005 MHz
SI            32768
SF           125.7377890 MHz
WDW           0
SSB           1.00 Hz
LB            0
GB            0
PC           1.40
  
```



3.833 3.825 3.813 3.804 3.795 3.787 3.777 3.760



1b



MH-2-42-S
PROTON CDC13

NAME	XB20140707
EXPNO	18
PROCNO	1
Date—	20140707
Time—	15.05
INSTRUM	spect
PROBHD	5 mm
PULPROG	PATXO_19F
TD	zg30
SOLVENT	65536
NS	CDC13
DS	16
SWH	2
FIDRES	
AQ	10330.578 Hz
RG	0.157632 Hz
DW	3.1720407 sec
DE	64
TE	48.400 usec
D1	6.000 usec
TD0	297.7 K
	1.0000000 sec
	1

===== CHANNEL f1 =====	
NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	327.68
SF	500.1300128 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

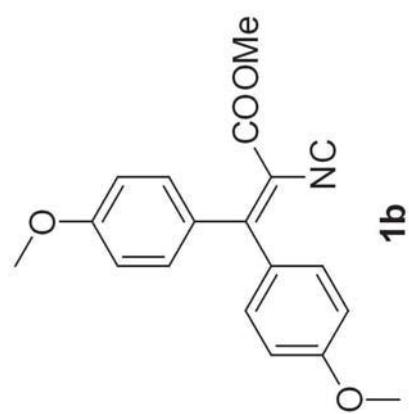
MH-2-42-S
C13CPD CDCl₃

55.43
55.34
52.76

111.34
113.63
113.00

129.88
130.26
131.40
132.36

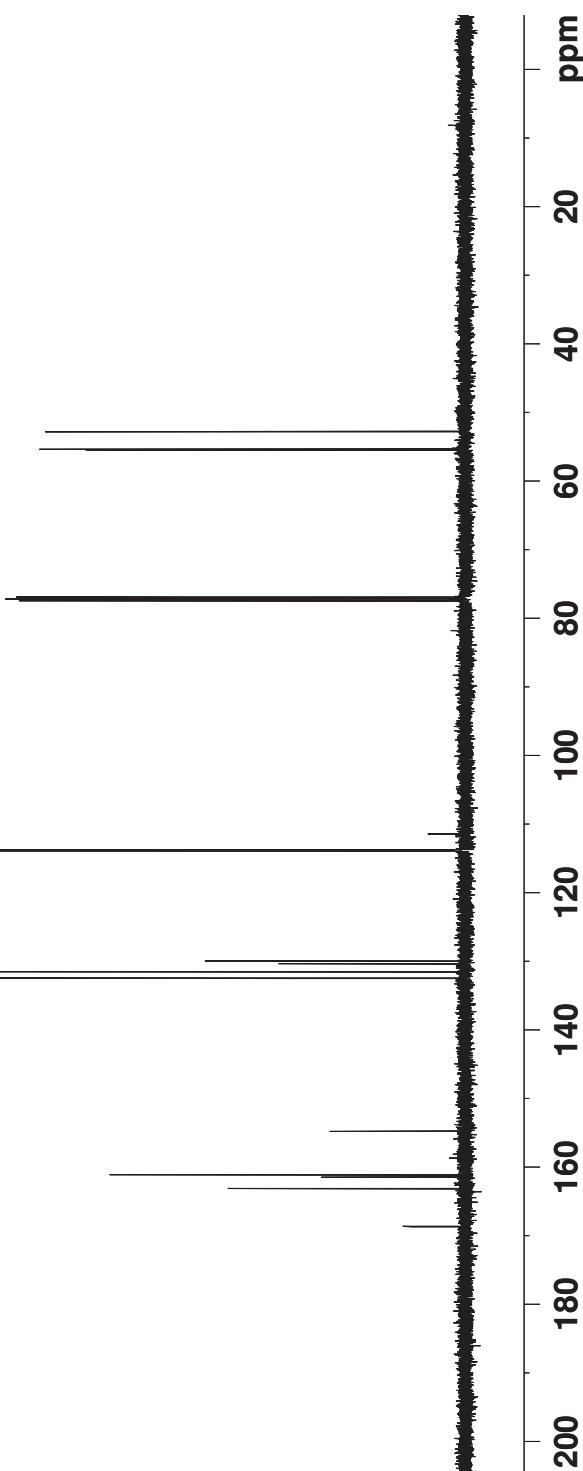
154.68
161.03
161.36
163.00
168.57



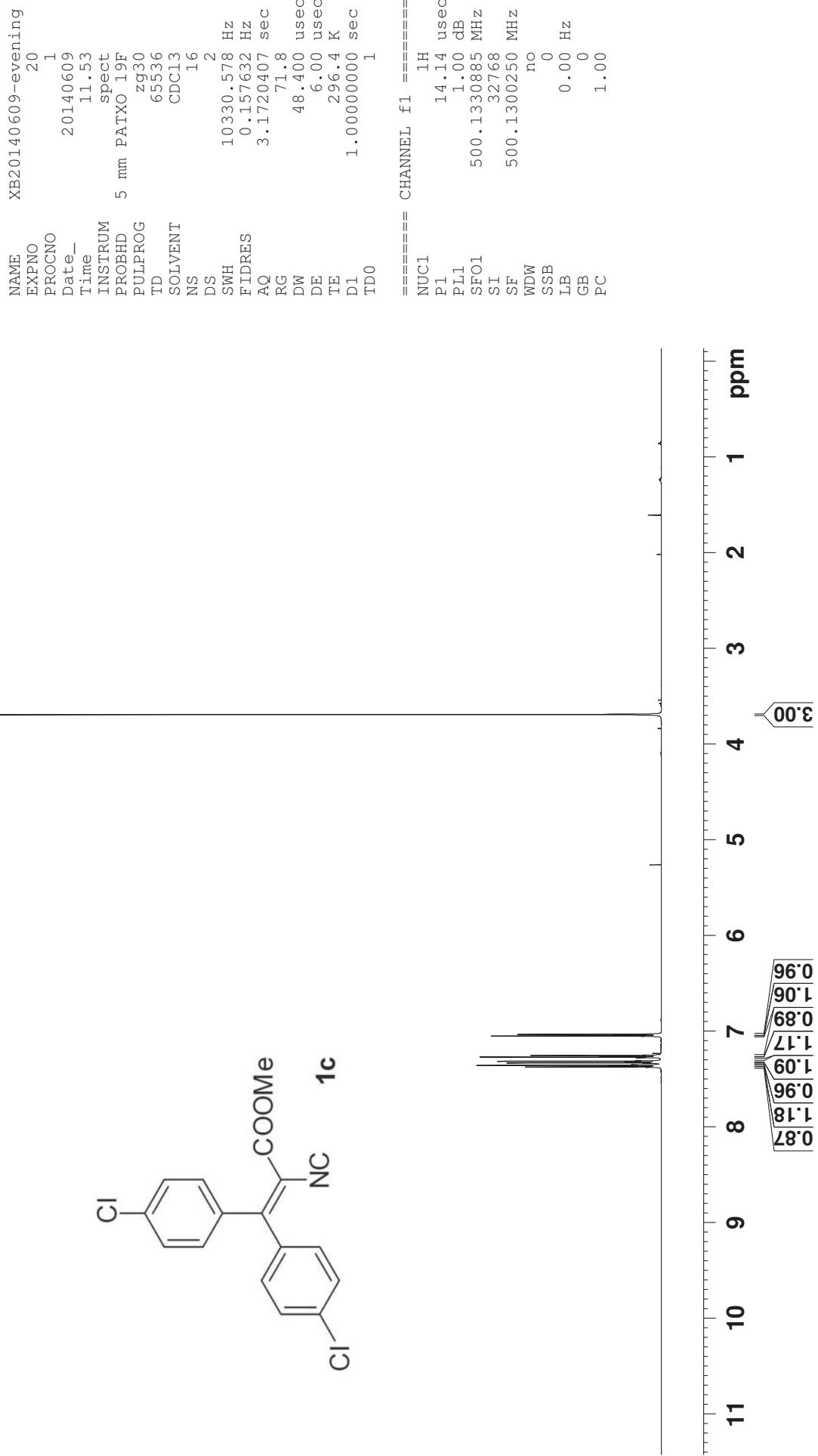
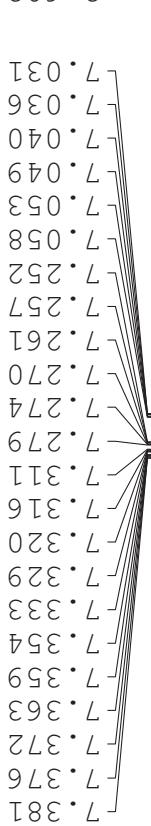
NAME XB20140707
EXPNO 20
PROCNO 1
Date 20140707
Time 15.22
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zgppg30
TD 65536
SOLVENT CDCl₃
NS 256
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 203.2
DW 16.650 usec
DE 6.00 usec
TE 299.4 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TDO 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.50 usec
PL1 -0.50 dB
SFO1 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.05 dB
PL13 16.50 dB
SFO2 500.1320005 MHz
SI 32768
SF 125.7577864 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40



YY-1-98
PROTON CDCl₃



YY-1-98
C13CPD CDC13

— 53.16 —

— 114.24 —

— 128.73 —

— 128.96 —

— 130.55 —

— 131.30 —

— 135.40 —

— 135.70 —

— 136.12 —

— 136.80 —

— 152.05 —

— 161.73 —

— 171.06 —

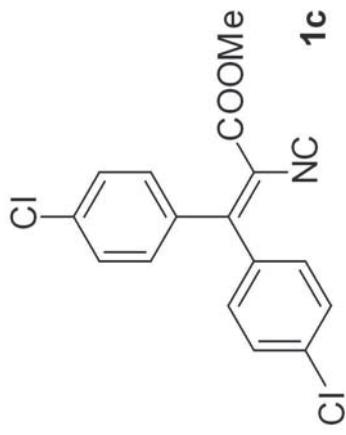
```

NAME          XB20140609-evening
EXPNO         21
PROCNO        1
Date_         20140610
Time          3.56
INSTRUM      spect
PROBHD       5 mm PATXO 19F
PULPROG      zgpg30
TD           65536
SOLVENT       CDC13
NS            256
DS            4
SWH          30030.029 Hz
FIDRES       0.458222 Hz
AQ            1.0912410 sec
RG            181
DW            16.650 usec
DE            6.00
TE            297.9 K
DI            2.0000000 sec
QSI           0.03000000 sec
DETA          1.89999998 sec
TDO          1

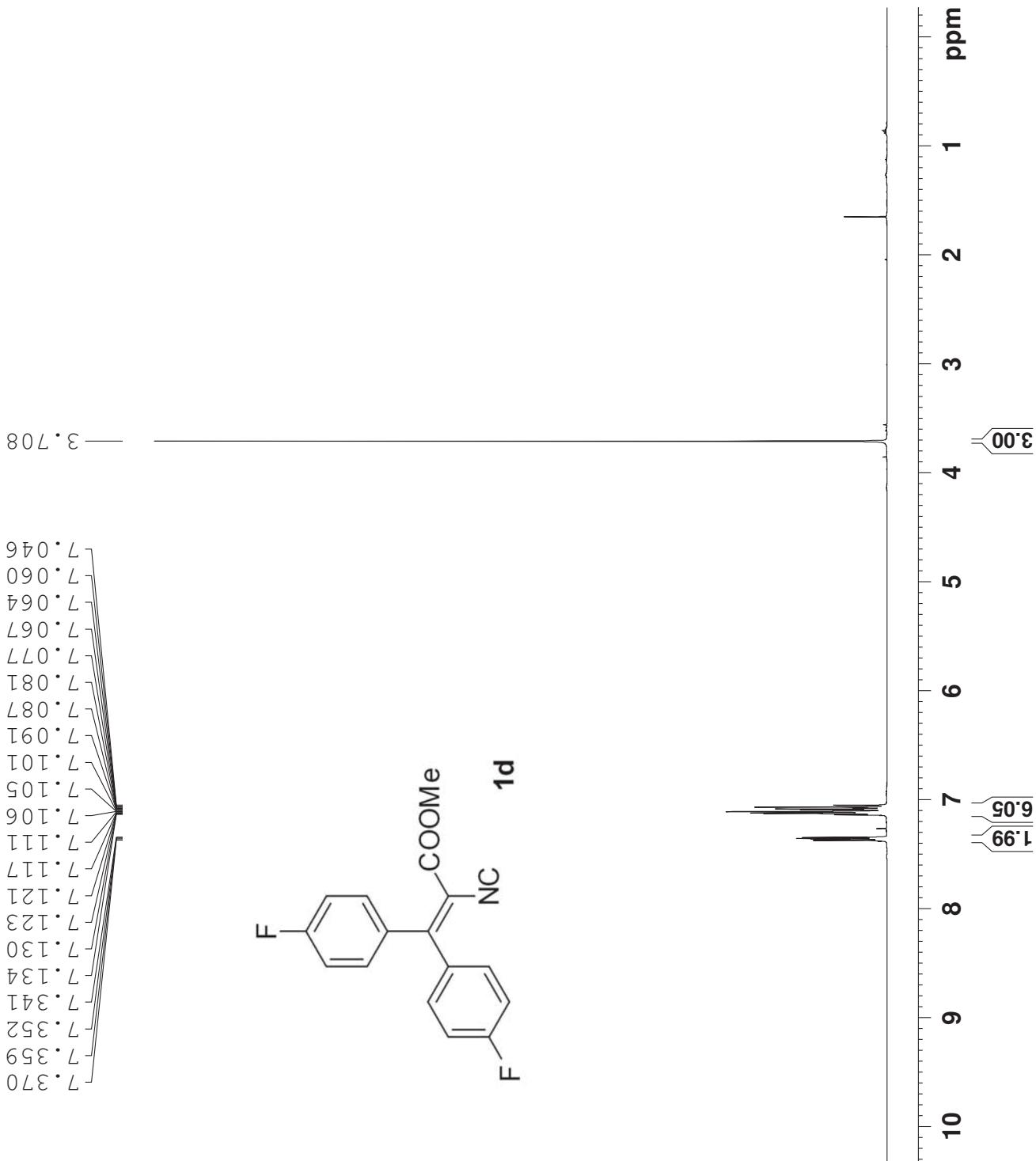
===== CHANNEL f1 =====
NUC1          13C
P1             9.50 usec
PL1           -0.50 dB
SF01          125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           1.00 dB
PL12          16.05 dB
PL13          16.50 dB
SF02          500.1320005 MHz
SI             327.68
SF             125.7577890 MHz
WDW           EM
SSB           0
LB             1.00 Hz
GB           0
PC           1.40

```



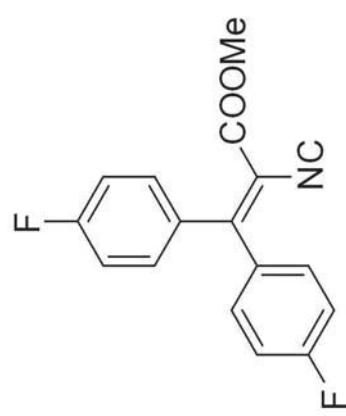
MH-2-44
PROTON CDCl₃



YY-1-94
C13CPD CDCL3

53.03

113.80
115.51
115.69
115.73
115.91
115.91
131.23
131.30
131.30
132.20
132.28
133.20
133.23
133.47
133.49
152.42
162.00
162.59
162.74
164.59
164.76
170.38



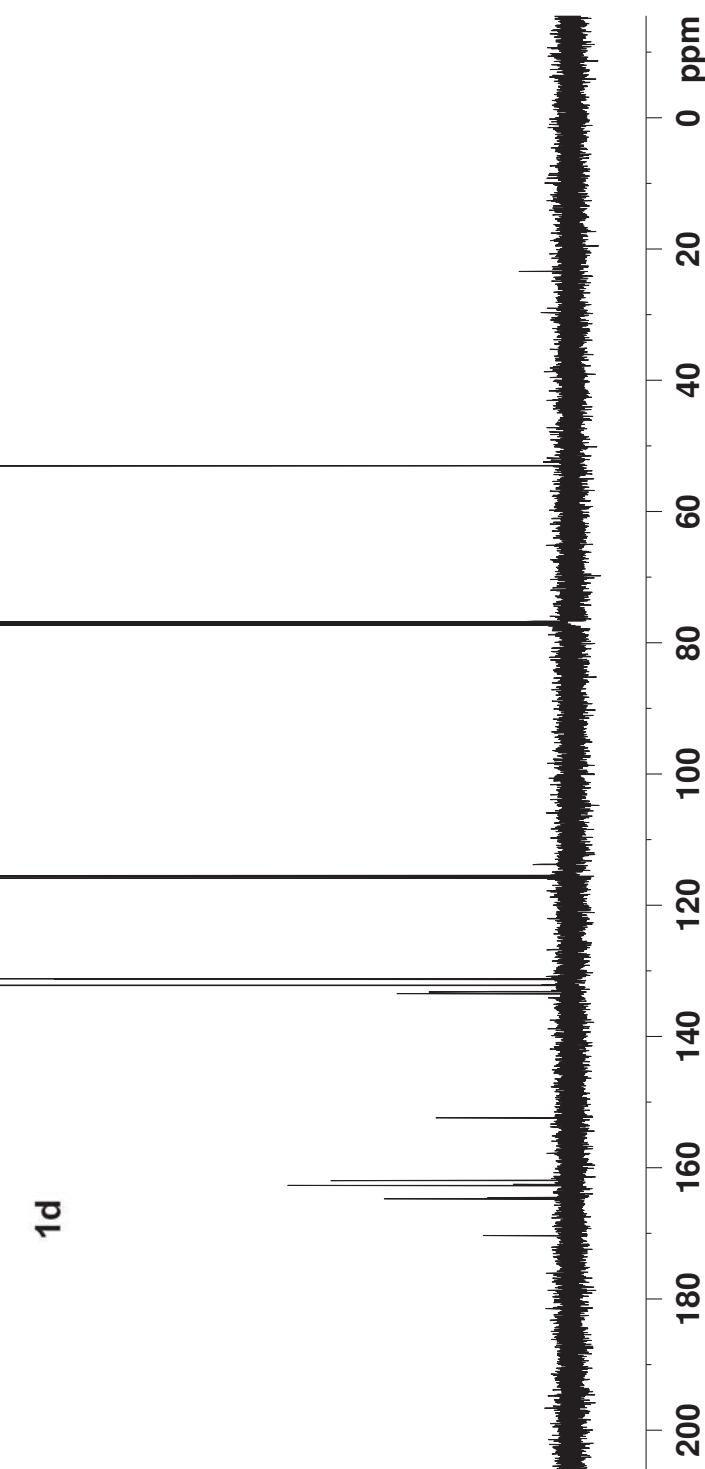
```

NAME          XB20140628
EXPNO         7
PROCNO        1
Date_         20140628
Time          15.10
INSTRUM      spect
PROBHD       5 mm PATXO 19F
PULPROG      zppg30
TD           65536
SOLVENT       CDC13
NS            1024
DS             4
SWH          30030.029 Hz
FIDRES       0.458222 Hz
AQ            1.0912410 sec
RG            128
DW           16.650 usec
DE            6.00 usec
TE            299.6 K
D1           2.0000000 sec
d11          0.0300000 sec
DELTA        1.8999998 sec
TD0            1

===== CHANNEL f1 =====
NUC1          13C
P1            9.50 usec
PL1          -0.50 dB
SFO1        125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2         80.00 usec
PL2            1.00 dB
PL12          16.05 dB
PL13          16.50 dB
SFO2        500.1320005 MHz
SI             32768
SF           125.7577890 MHz
WDDW          no
SSB            0 Hz
LB            0.00 Hz
GB            0
PC           1.40

```



YY-1-94
19Fdefft CDC13 D:\ \ deng 46

NAME	XB20140627
EXPNO	18
PROCNO	1
Date_	20140627
Time	11.05
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg
TD	131072
SOLVENT	CDCl3
NS	1
DS	4
SWH	1000000.000 Hz
FIDRES	0.762939 Hz
AQ	0.6554150 sec
RG	362
DW	5.000 usec
DE	6.000 usec
TE	296.9 K
D1	1.00000000 sec
TD0	1

===== CHANNEL f1 =====
NUC1 19F
P1 19.30 usec
PL1 4.00 dB
SFO1 470.5453180 MHz
SI 65536
SF 470.5923770 MHz

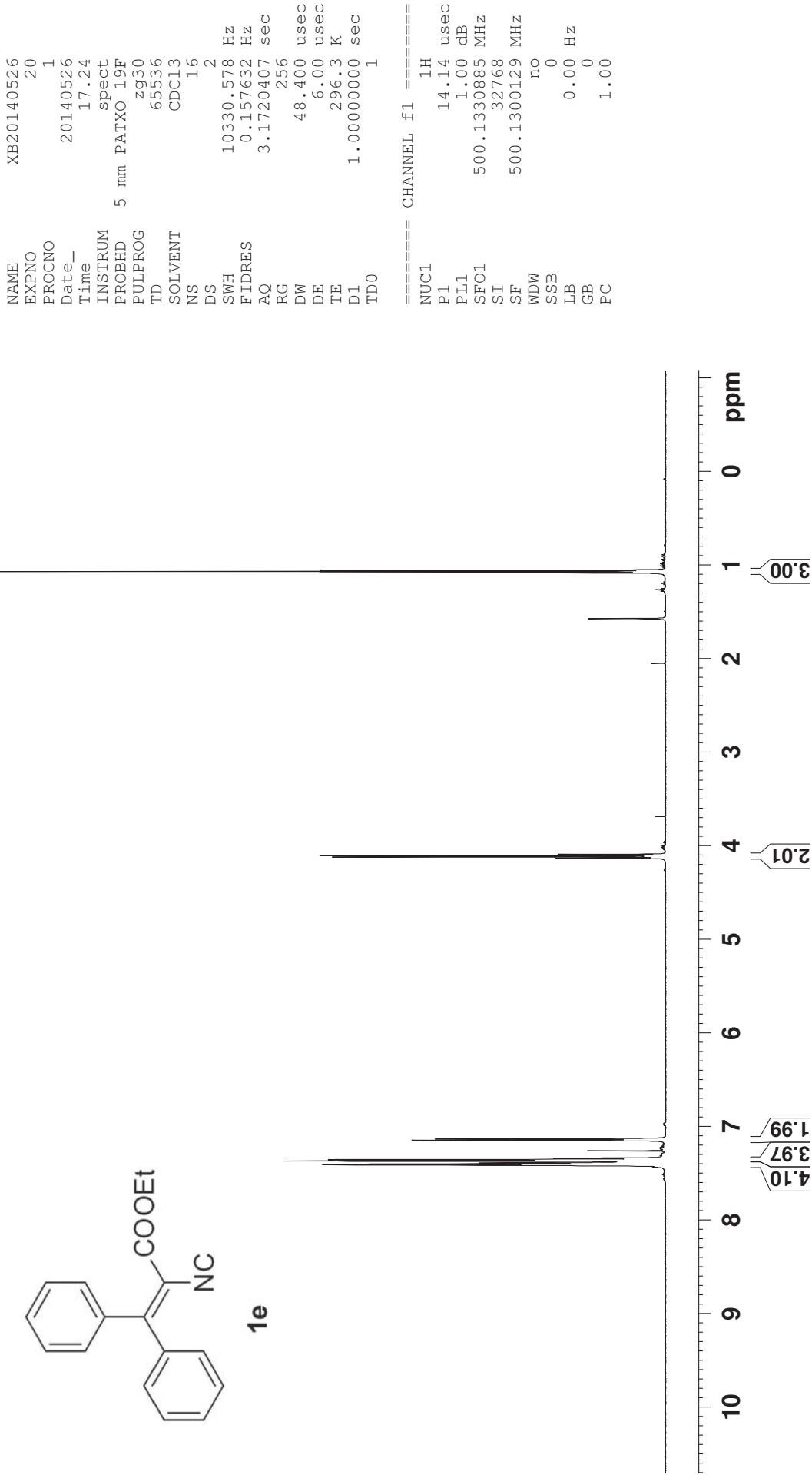
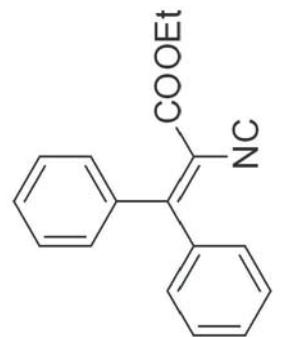


WH-6-29-S
PROTON CDCl₃

4.081
4.067
4.052

4.132
4.118
4.104
4.090

7.418
7.399
7.389
7.368
7.354
7.340
7.147
7.133
7.130



WH-6-29-S
C13CPD CDC13

13.58

62.24

114.35

128.22

128.44

129.10

129.45

129.88

130.24

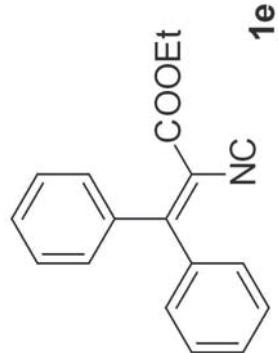
137.37

138.03

153.90

161.95

169.60



```

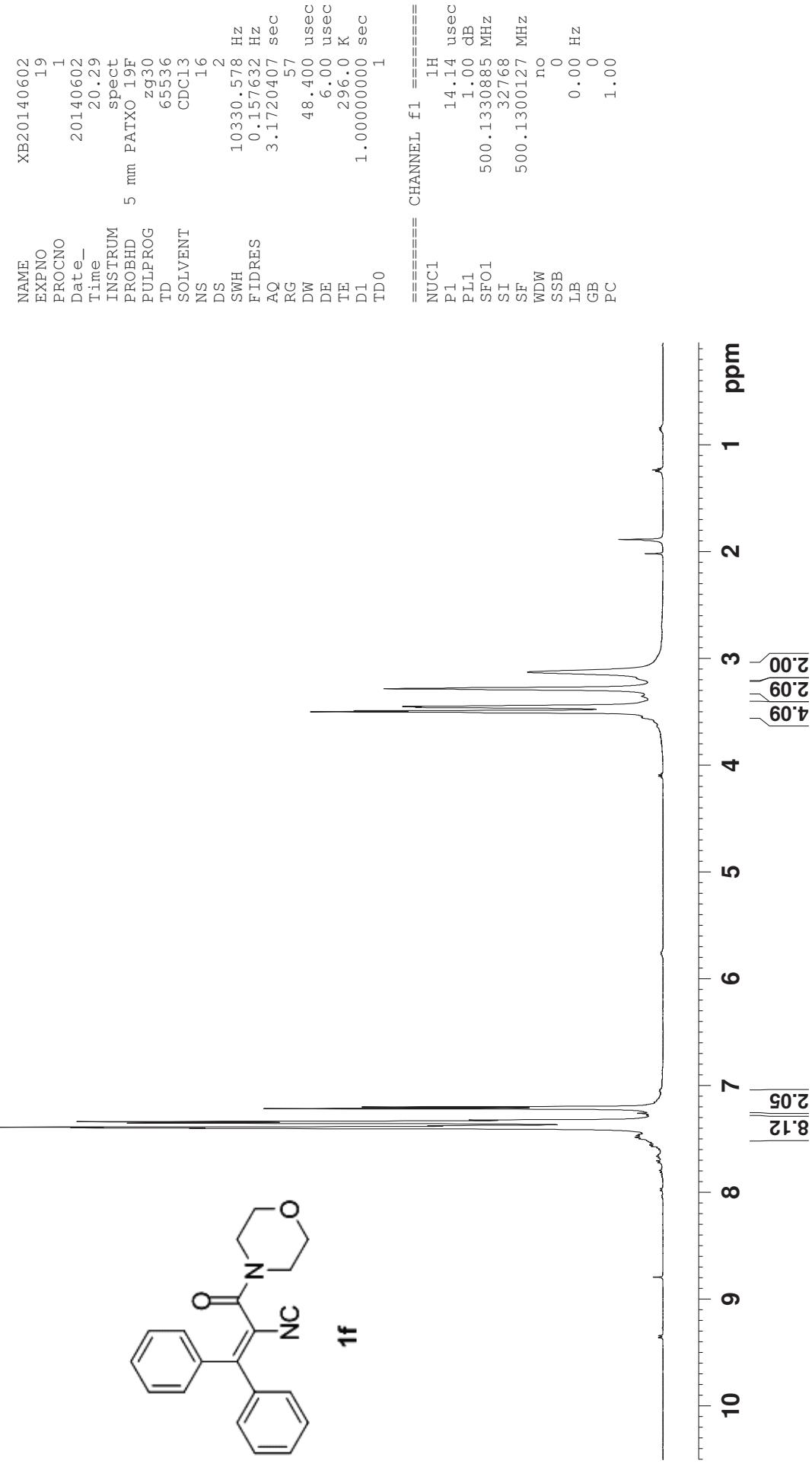
NAME          XB20140602
EXPNO         21
PROCNO        1
Date_         20140602
Time          21.57
INSTRUM      spect
PROBHD       5 mm PATXO 19F
PULPROG      zppg30
TD           65536
SOLVENT       CDCl3
NS            512
DS            4
SWH          30030.029 Hz
FIDRES       0.458222 Hz
AQ            1.0912410 sec
RG            181
DW            16.650 usec
DE            6.000 usec
TE            297.7 K
D1           2.0000000 sec
d1           0.03000000 sec
DELTA        1.89999998 sec
TD0           1
TDO           1

===== CHANNEL f1 =====
NUC1          13C
P1            9.50 usec
PL1           -0.50 dB
SFO1        125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           1.00 dB
PL12          16.05 dB
PL13          16.50 dB
SFO2        500.1320005 MHz
SI            32768
SF           125.7577890 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC           1.40

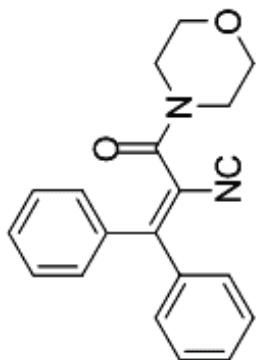
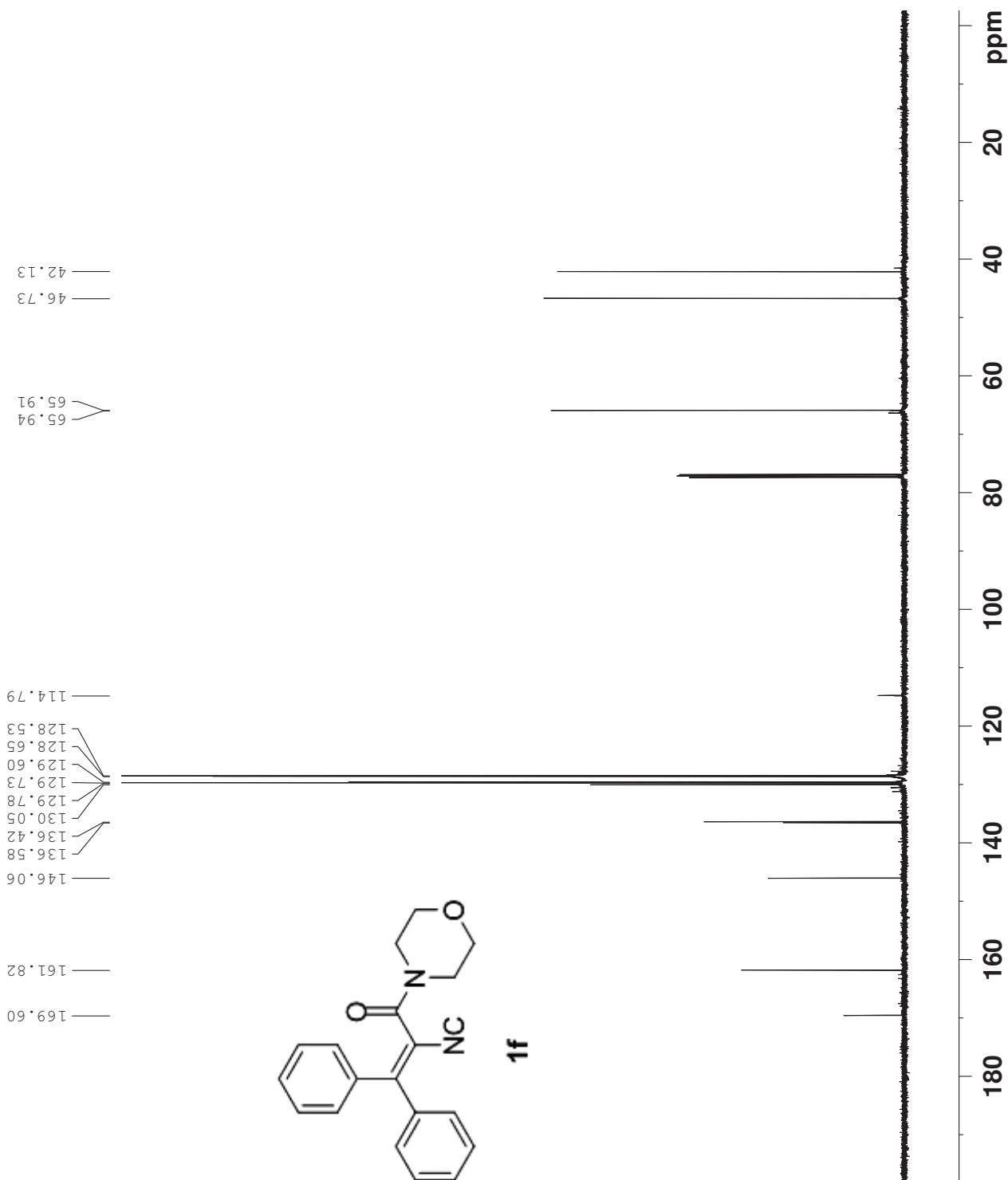
```

WH-7-5
PROTON CDCl₃

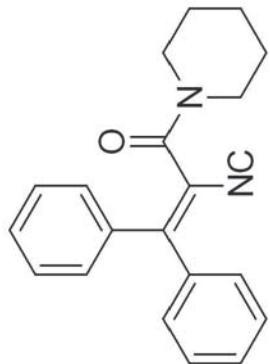


WH-7-5
C13CPD CDC13

NAME	XB20140602
EXPNO	20
PROCNO	1
Date—	20140602
Time—	21.25
INSTRUM	spect
PROBHD	5 mm
PULFRQG	PAIXO 19F
ID	zppg30
SOLVENT	65536
NS	CDDC13
DS	1024
SWH	300030.029 Hz
FIDRES	0.458222 Hz
AQ	1.091241 sec
RG	181
DW	16.650 usec
DE	6.00 usec
TE	297.7 K
D1	2.0000000 sec
D11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1
===== CHANNEL f1 =====	
NUC1.	13C
P1	9.50 usec
PL1	-0.50 dB
SFO1.	125.7703643 MHz
===== CHANNEL f2 =====	
CPDPFRG2	waltz16
NUC2	1H
PCPDP2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	327.68
SF	125.7577890 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.40



WH-7-7
PROTON CDC13

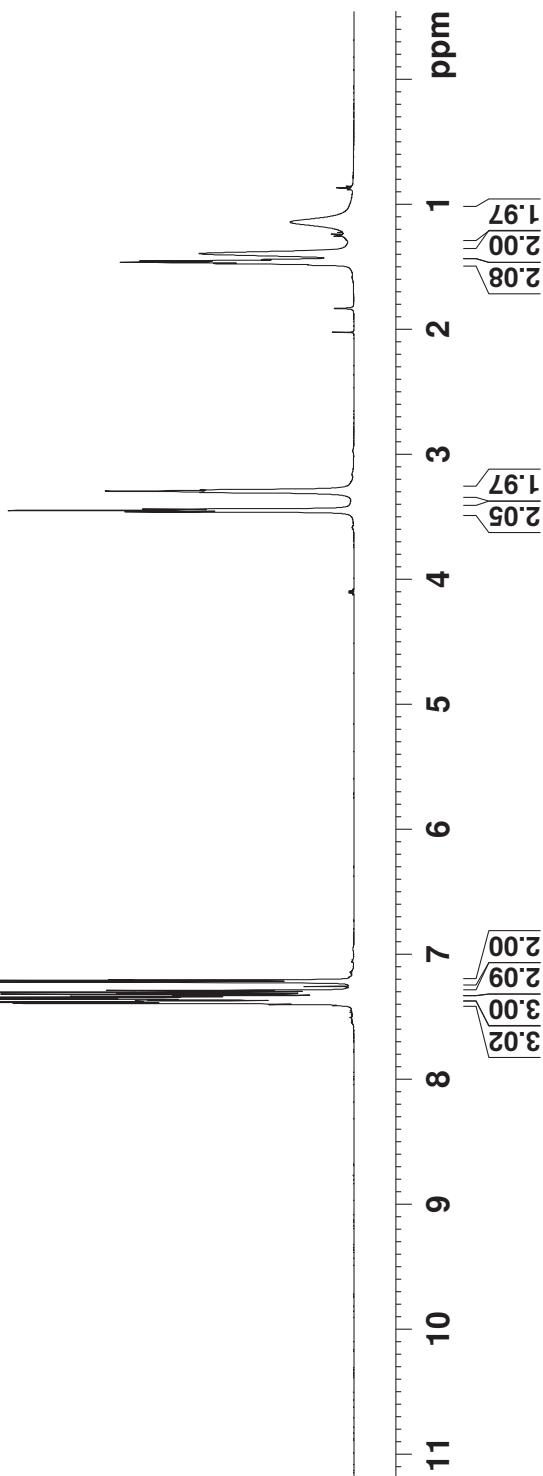


NAME	XBX20140603
EXPNO	5
PROCNO	1
Date_	20140603
Time	13.46
INSTRUM	spect
PROBHD	5 mm
PULPROG	PATXO 19F
TD	Zg30
SOLVENT	65536
NS	CDC13
DS	16
SWH	10330.578
FIDRES	0.157632
AQ	3.1720407
RG	64
DW	48 .400
DE	6 .00
TE	295.9
D1	1 .00000000
TD0	

```

===== CHANNEL f1 =====
NUC1          1H
P1           14.14 usec
PLI          1.00 dB
SFO1        500.1330885 MHz
SI            32.768
SF           500.1300126 MHz
WDW          no
SSB           0
LB           0.00 Hz
GB           0
PC          1.00

```



WH-7-7
C13CPD CDC13

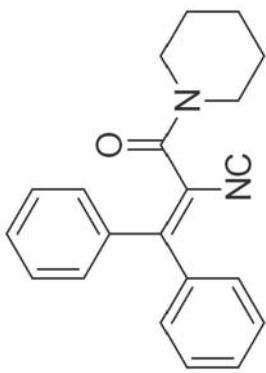
25.49
24.83
24.10

47.55
42.73

115.87

145.06
136.78
136.75
129.72
129.65
129.58
129.48
128.45

161.49
169.23



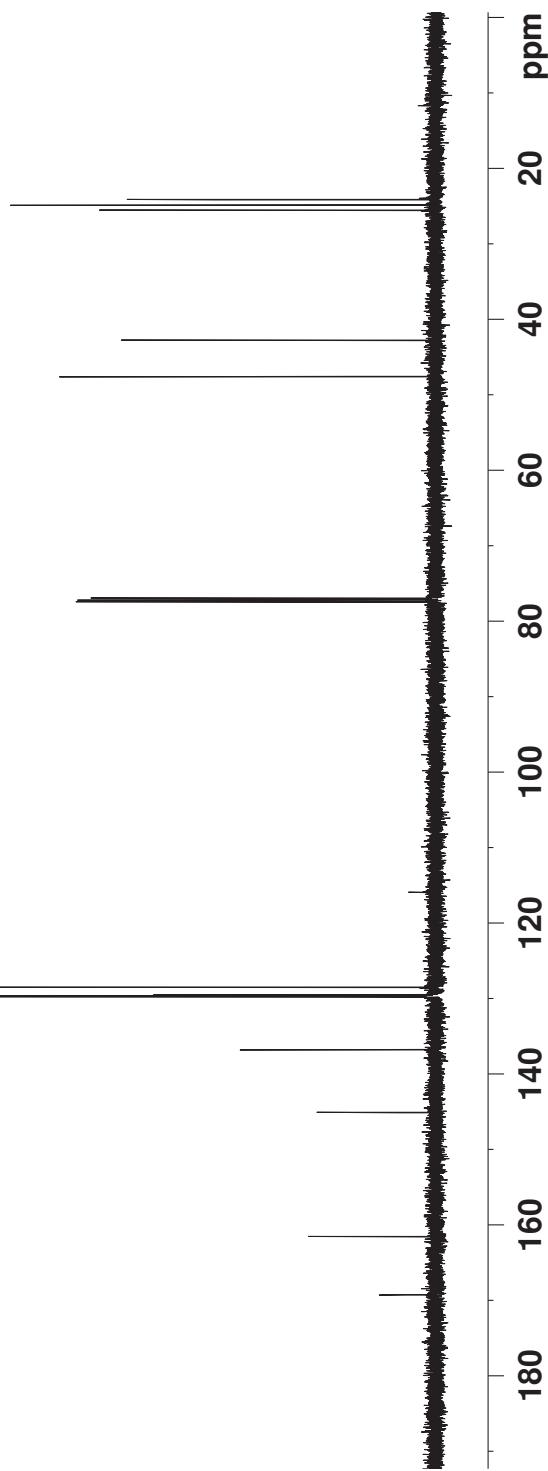
```

=====
NAME          XB20140603
EXPNO         6
PROCNO        1
Date_        20140603
Time         13.50
INSTRUM      spect
PROBHD      5 mm PATXO 19F
PULPROG     zppg30
TD           65536
SOLVENT      CDCl3
NS            145
DS            4
SWH          30030.029 Hz
FIDRES       0.458222 Hz
AQ           1.0912410 sec
RG           114
DW           16.650 usec
DE           6.00 usec
TE           296.8 K
D1           2.0000000 sec
d1.1         0.03000000 sec
DELTA        1.89999998 sec
TDO          1

===== CHANNEL f1 =====
NUC1          13C
P1             9.50 usec
PL1          -0.50 dB
SFO1        125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           1.00 dB
PL12          16.05 dB
PL13          16.50 dB
SFO2        500.1320005 MHz
SI            32768
SF           125.7577890 MHz
WDW          no
SSB          0
LB           0.00 Hz
GB           0
PC           1.40

```



WH-7-2³
PROTON CDCl₃

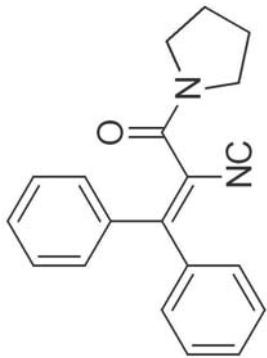
1.660
1.666
1.672
1.679
1.686
1.698
1.705
1.712
1.719
1.726
1.733
1.740
1.747
1.754
1.761
1.768
1.775
1.782
1.789
1.796
1.803
1.810
1.817
1.824
1.831
1.838
1.845
1.852
1.859
1.866
1.873
1.880
1.887
1.894
1.901
1.908
1.915
1.922
1.929
1.936
1.943
1.950
1.957
1.964
1.971
1.978
1.985
1.992
1.999
2.006
2.013
2.020
2.027
2.034
2.041
2.048
2.055
2.062
2.069
2.076
2.083
2.090
2.097
2.104
2.111
2.118
2.125
2.132
2.139
2.146
2.153
2.160
2.167
2.174
2.181
2.188
2.195
2.202
2.209
2.216
2.223
2.230
2.237
2.244
2.251
2.258
2.265
2.272
2.279
2.286
2.293
2.300
2.307
2.314
2.321
2.328
2.335
2.342
2.349
2.356
2.363
2.370
2.377
2.384
2.391
2.398
2.405
2.412
2.419
2.426
2.433
2.440
2.447
2.454
2.461
2.468
2.475
2.482
2.489
2.496
2.503
2.510
2.517
2.524
2.531
2.538
2.545
2.552
2.559
2.566
2.573
2.580
2.587
2.594
2.597
2.600
2.603
2.606
2.609
2.612
2.615
2.618
2.621
2.624
2.627
2.630
2.633
2.636
2.639
2.642
2.645
2.648
2.651
2.654
2.657
2.660
2.663
2.666
2.669
2.672
2.675
2.678
2.681
2.684
2.687
2.690
2.693
2.696
2.699
2.702
2.705
2.708
2.711
2.714
2.717
2.720
2.723
2.726
2.729
2.732
2.735
2.738
2.741
2.744
2.747
2.750
2.753
2.756
2.759
2.762
2.765
2.768
2.771
2.774
2.777
2.780
2.783
2.786
2.789
2.792
2.795
2.798
2.801
2.804
2.807
2.810
2.813
2.816
2.819
2.822
2.825
2.828
2.831
2.834
2.837
2.840
2.843
2.846
2.849
2.852
2.855
2.858
2.861
2.864
2.867
2.870
2.873
2.876
2.879
2.882
2.885
2.888
2.891
2.894
2.897
2.900
2.903
2.906
2.909
2.912
2.915
2.918
2.921
2.924
2.927
2.930
2.933
2.936
2.939
2.942
2.945
2.948
2.951
2.954
2.957
2.960
2.963
2.966
2.969
2.972
2.975
2.978
2.981
2.984
2.987
2.990
2.993
2.996
2.999
3.002
3.005
3.008
3.011
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WH-7-23
C13CPD CDCl₃

24.08
25.58

47.12
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169.37



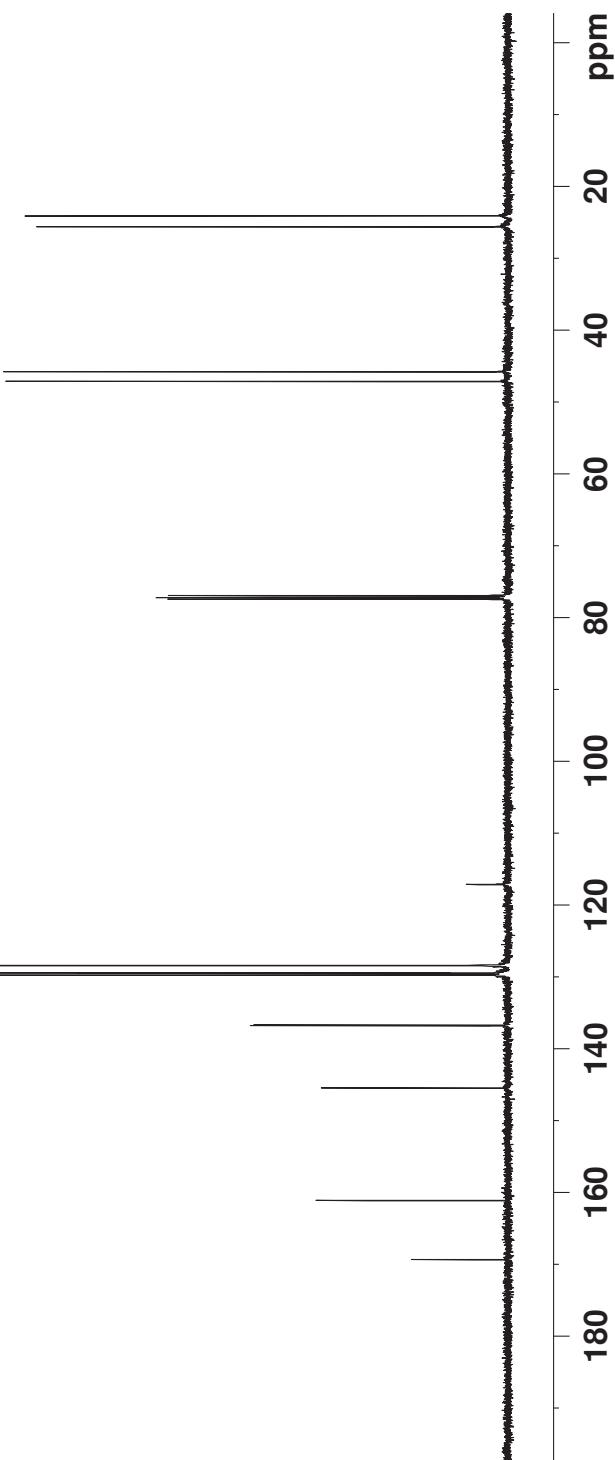
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EXPN0         23
PROCNO       1
Date_        20140610
Time_        4.18
INSTRUM     spect
PROBHD      5 mm PATXO 1.9F
PULPROG     zgpg30
TD          65536
SOLVENT      CDCl3
NS           256
DS            4
SWH         30030.029 Hz
ETDRES      0.458222 Hz
AQ          1.0912410 sec
RG           114
DW           16.650 usec
DE           6.00 usec
TE           297.8 K
D1          2.00000000 sec
DD1          0.03000000 sec
DELTA       1.89999998 sec
TD0          1

===== CHANNEL f1 =====
NUC1         13C
P1           9.50 usec
PL1          125.7703643 MHz
SF01

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2         1H
ECPD2       80.00 usec
PL2          1.00 dB
PL12         16.05 dB
PL13         16.50 dB
SF02       500.1320005 MHz
SI           32768
SF           125.7577890 MHz
WDW          EM
SSB           0
LB           1.00 Hz
GB           0
PC           1.40

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YY-1-100
PROTON CDCl₃

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7.708
7.611
7.594
7.579

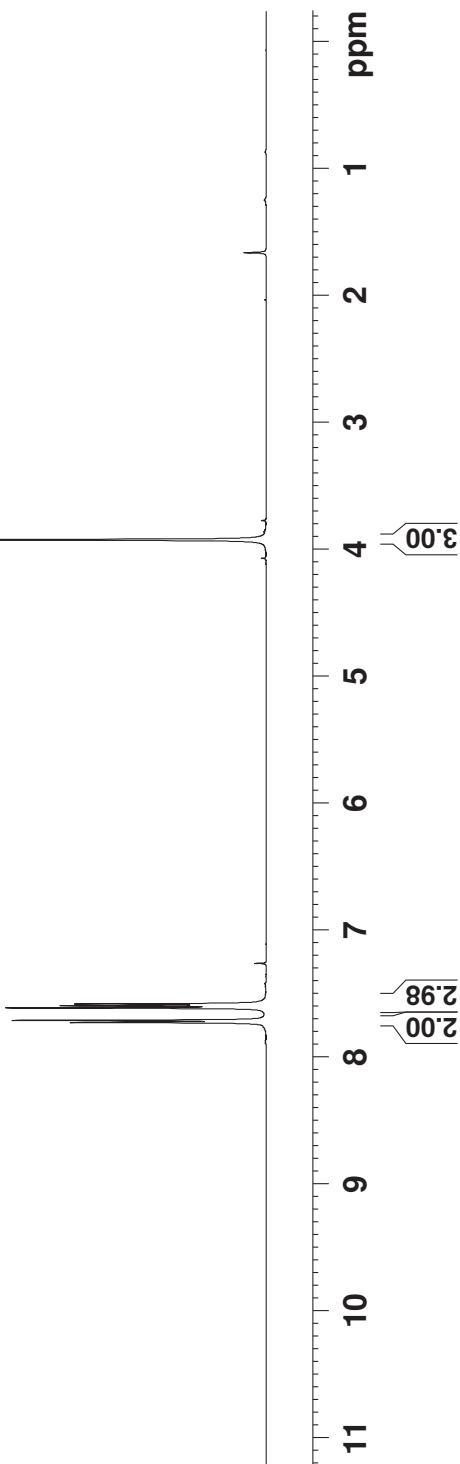
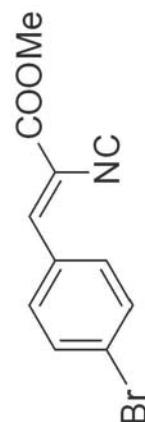
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NAME          XB20140617
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Time       15.34
INSTRUM      spect
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PULPROG     zg30
TD           65536
SOLVENT      CDCl3
NS            16
DS            2
SWH         10330.578 Hz
FIDRES      0.157632 Hz
AQ           3.1720407 sec
RG           143.7
DW           48.400 usec
DE           6.00 usec
TE           296.1 K
D1          1.00000000 sec
TDO          1

===== CHANNEL f1 =====
NUC1          1H
P1           14.14 usec
PL1          1.00 dB
SFO1        500.1330885 MHz
SI            32768
SF           500.1300126 MHz
WDW           no
SSB            0
LB            0.00 Hz
GB            0
PC            1.00

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YY-1-100
C13CPD CDC13

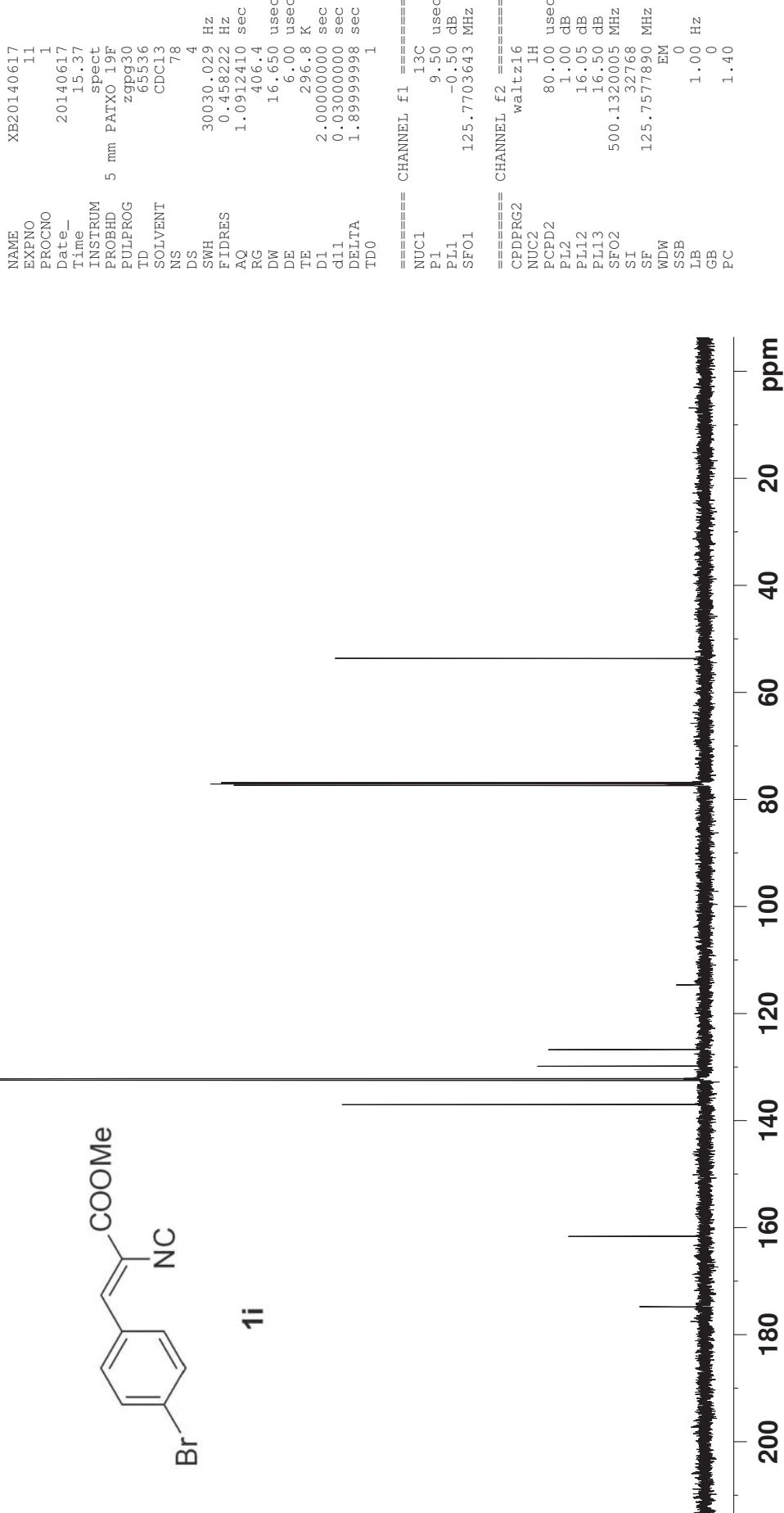
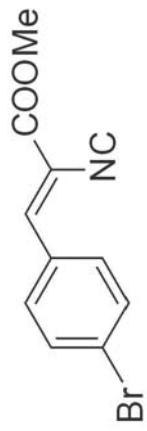
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— 114.65 —

— 126.72
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129.83
132.17
132.47
137.03

— 161.54 —

— 174.71 —



WH-7-35
PROTON CDCl₃

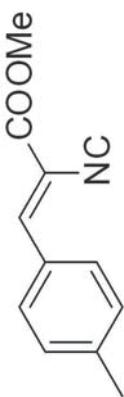
NAME	XB20140617
EXPNO	21
PROCNO	1
Date_	20140617
Time	19.02
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDCl ₃
NS	16
DS	2
SWH	103330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	101.6
DW	48.400 usec
DE	6.00 usec
TE	296.6 K
D1	1.0000000 sec
TD0	1

===== CHANNEL f1 =====	
NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300128 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

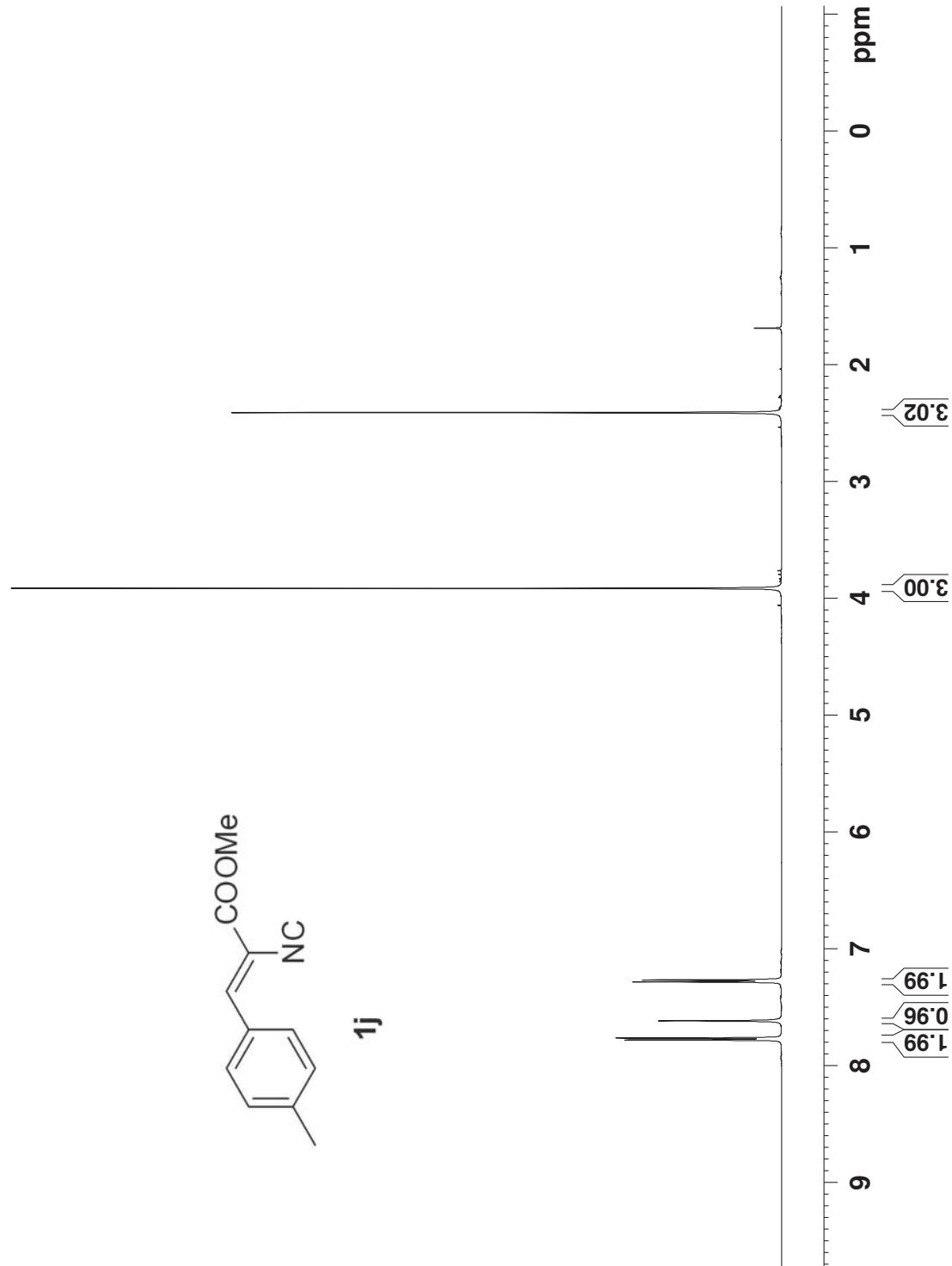
— 2.409 —

— 3.914 —

7.267
7.283
7.617
7.764
7.780



1j



WH-7-35
C13CPD CDC13

— 21.73

— 53.40

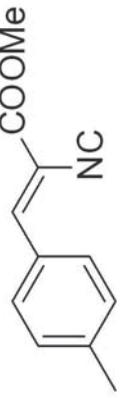
— 113.19

— 128.35
— 129.88
— 131.10
— 138.48
— 143.03

— 162.02

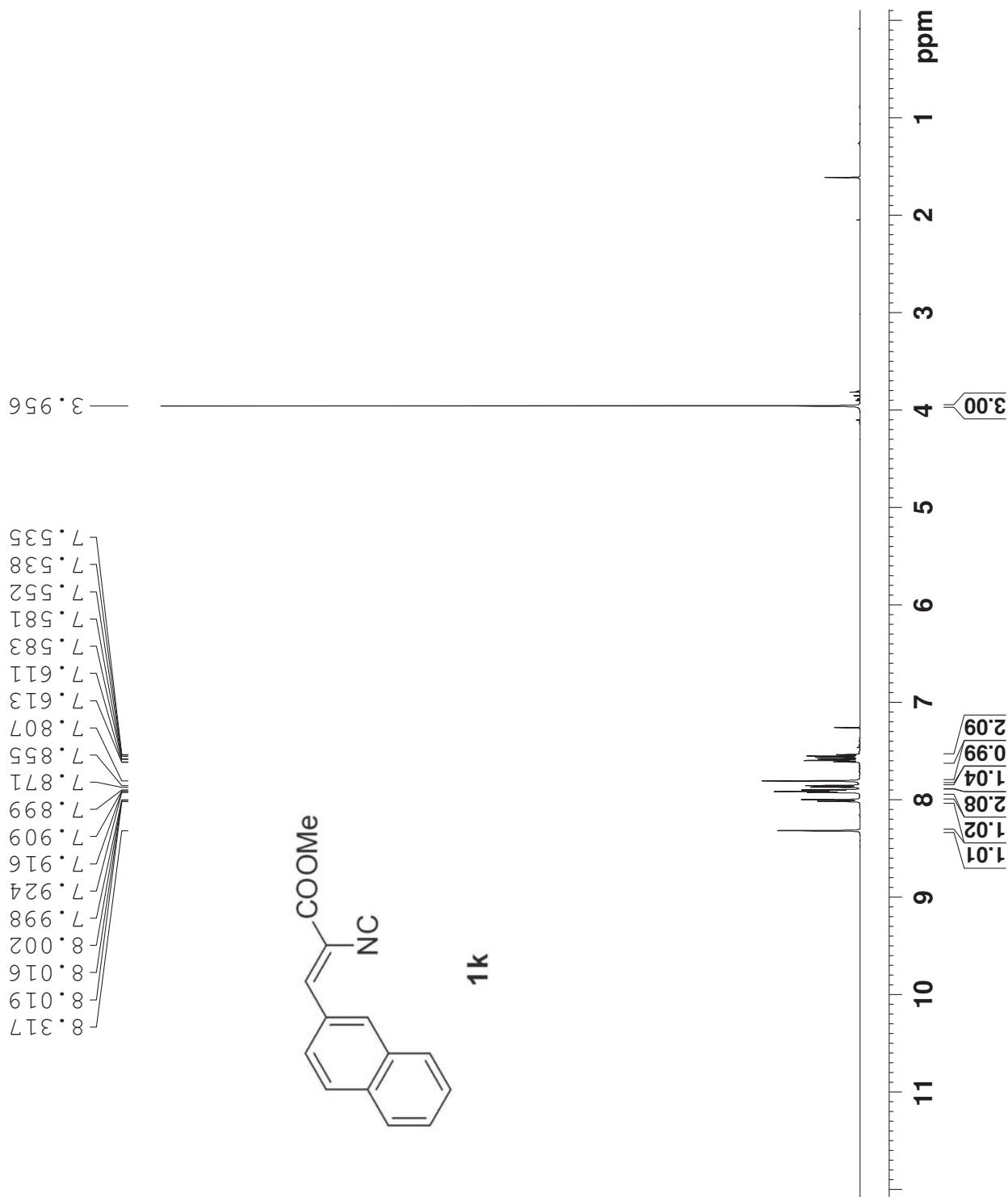
— 173.58

NAME XB20140617
EXPNO 22
PROCNO 1
Date 20140617
Time 19.19
INSTRUM spect
PROBHD 5 mm PAXTO 19F
PULPROG zgpp30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 228.1
DW 16.650 usec
DE 6.00 usec
TE 297.7 K
D1 2.0000000 sec
d11 0.0300000 sec
DELT1 1.8999998 sec
TDO 1
===== CHANNEL f1 =====
NUC1 13C
P1 9.50 usec
PL1 -0.50 dB
SFO1 125.7703643 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.05 dB
PLL3 16.50 dB
SFO2 500.1320005 MHz
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



1j

WH-7-29
PROTON CDCl₃



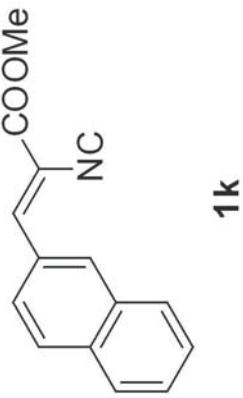
WH-7-29
C13CPD CDCl₃

53.53

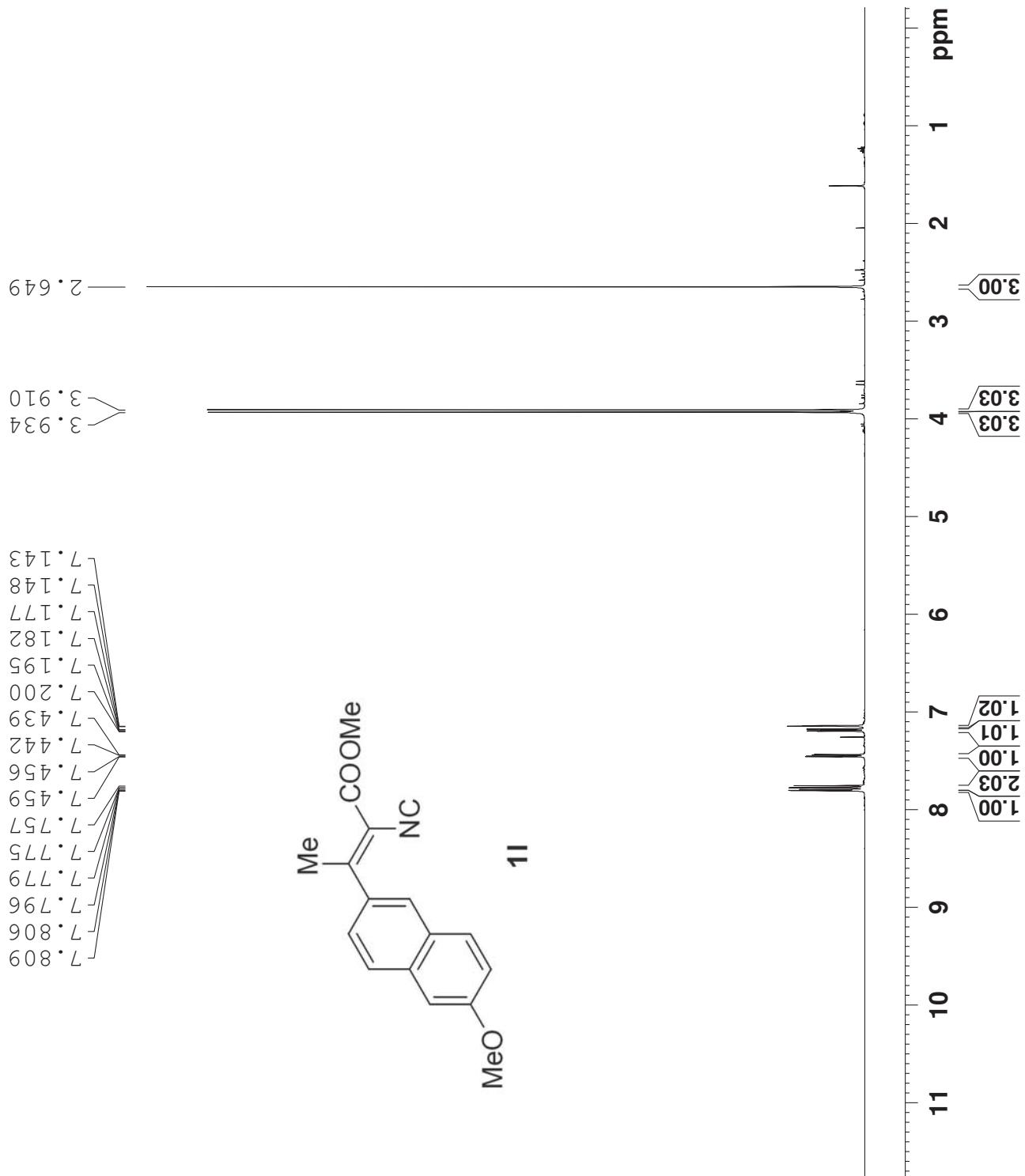
114.07
126.07
127.05
127.80
128.56
128.91
129.14
132.90
132.97
134.71
138.50

161.95
174.03

===== NAME XB20140613
EXPNO 7
PROCNO 1
Date_ 20140613
Time 11.42
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 128
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 322.5
DW 16.650 usec
DE 6.00 usec
TE 297.7 K
D1 2.0000000 sec
d11 0.0300000 sec
DELTA 1.8999998 sec
TD0 1
===== CHANNEL f1 13C
NUC1 1H
P1 9.50 usec
PL1 -0.50 dB
SF01 125.7703643 MHz
===== CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.05 dB
PL13 16.50 dB
SF02 500.1320005 MHz
SI 327.68 MHz
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



MH-2-57
PROTON CDCl₃



MH-2-57
C13CPD CDC13

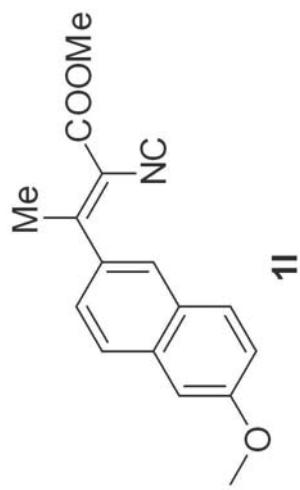
NAME XB20140714
EXPNO 18
PROCNO 1
Date_ 20140714
Time 13.12
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 300030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 114
DW 16.650 usec
DE 6.00 usec
TE 298.2 K
D1 2.00000000 sec
d1.1 0.0300000 sec
DELTA 1.899999998 sec
TD0 1
===== CHANNEL f1 =====
NUC1 13C
P1 9.50 usec
PL1 -0.50 dB
SFO1 125.7703643 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.05 dB
PL13 16.50 dB
SFO2 500.1320005 MHz
SI 32768
SF 125.7577890 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

21.79

55.42
52.87

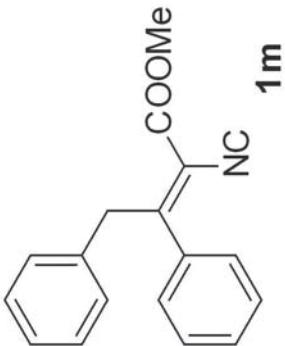
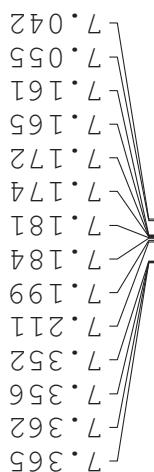
105.71

134.93
130.04
128.23
127.13
127.09
125.09
125.09
119.66



162.10
158.84
156.13
168.16

WH-7-40-S
PROTON CDC13



NAME XB20140617
 EXPNO 8
 PROCNO 1
 Date 20140617
 Time 12.44
 INSTRUM spect
 PROBHD 5 mm PATXO_19F
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1720407 sec
 RG 203.2
 DW 48.400 usec
 DE 6.000 usec
 TE 296.5 K
 D1 1.0000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 14.14 usec
 PL1 1.00 dB
 SFO1 500.1330885 MHz
 SI 32768
 SF 500.1300129 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

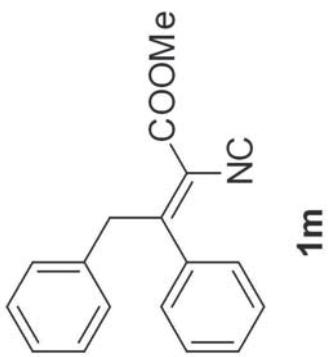
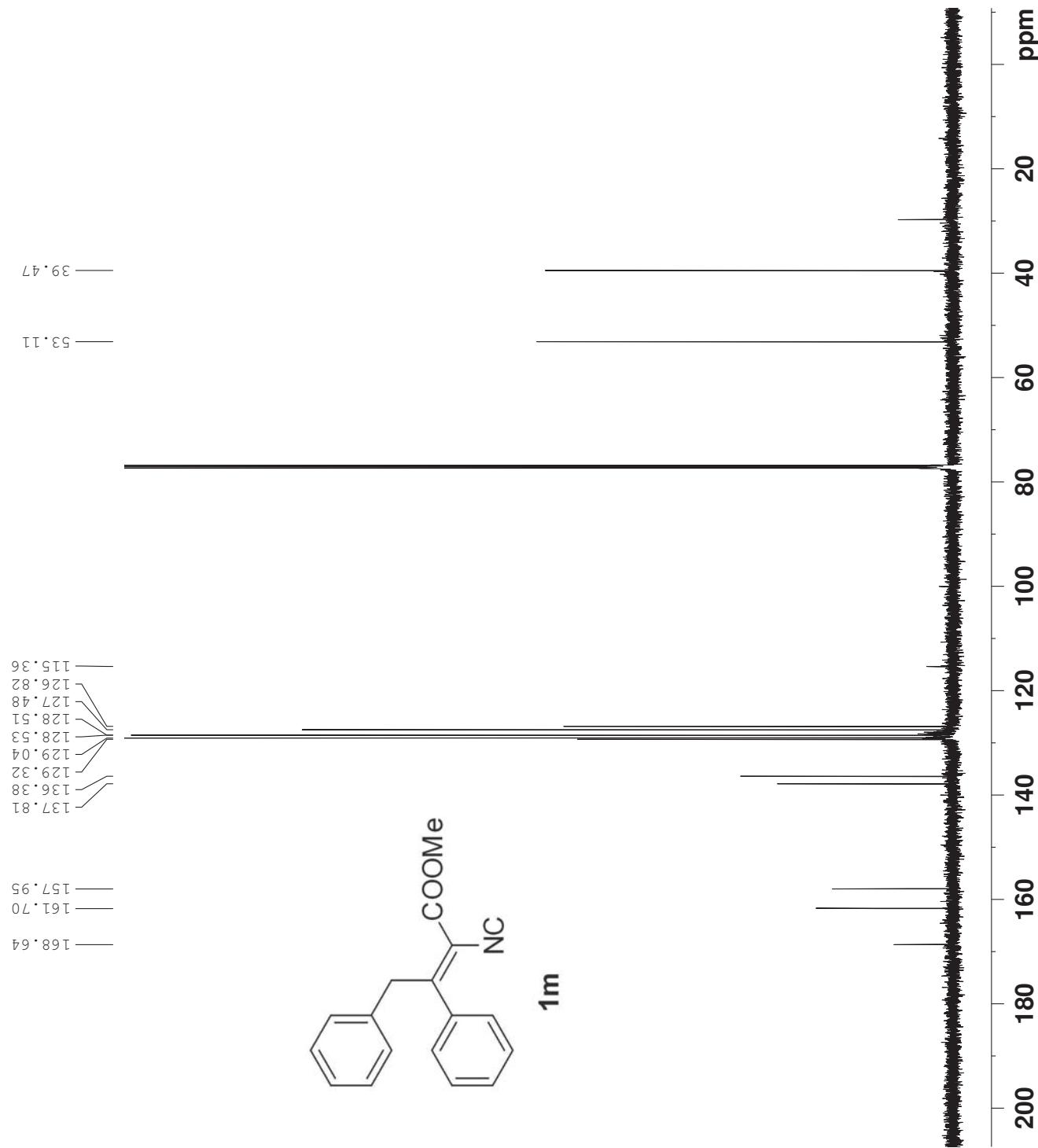
1m
C#Cc1ccc(cc1)CC(C(=O)OC)c2ccccc2

10 9 8 7 6 5 4 3 2 1 ppm

3.00 5.10 2.03 2.07

WH-7-40-S
C13CPD CDC13

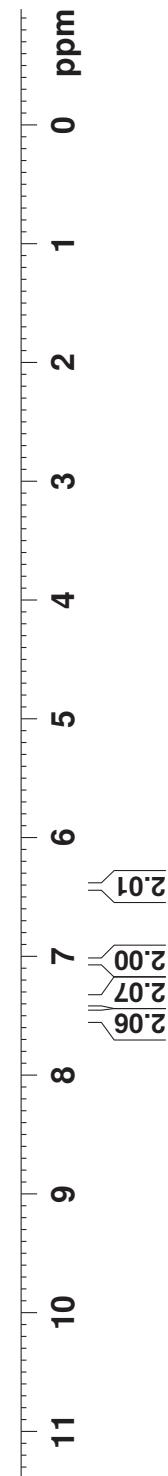
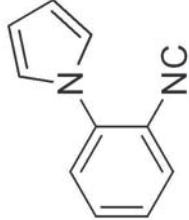
NAME	XB20140626
EXPNNO	9
PROCNO	1
Date_	20140626
Time_	10.49
INSTRUM	spect
PROBHDI	5 mm
PULPROG	PATXO 19F
TD	zppg30
SOLVENT	65536
NS	CDC13
DS	440
SWH	30030 0.029 Hz
FIDRES	0.458222 Hz
AQ	1.091240 sec
RG	228.1
DW	16.650 used
DE	6.00 used
TE	298.1 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TDO	1
===== CHANNEL f1 =====	
NUC1	13C
P1	9.50 used
PL1	-0.50 qB
SFO1	125.7703643 MHz
===== CHANNEL f2 =====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 used
PL2	1.00 qB
PL12	1.6.05 qB
PL13	1.6.50 qB
SFO2	500.1320005 MHz
SI	327.68
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



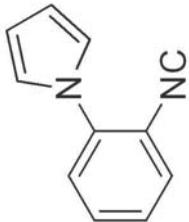
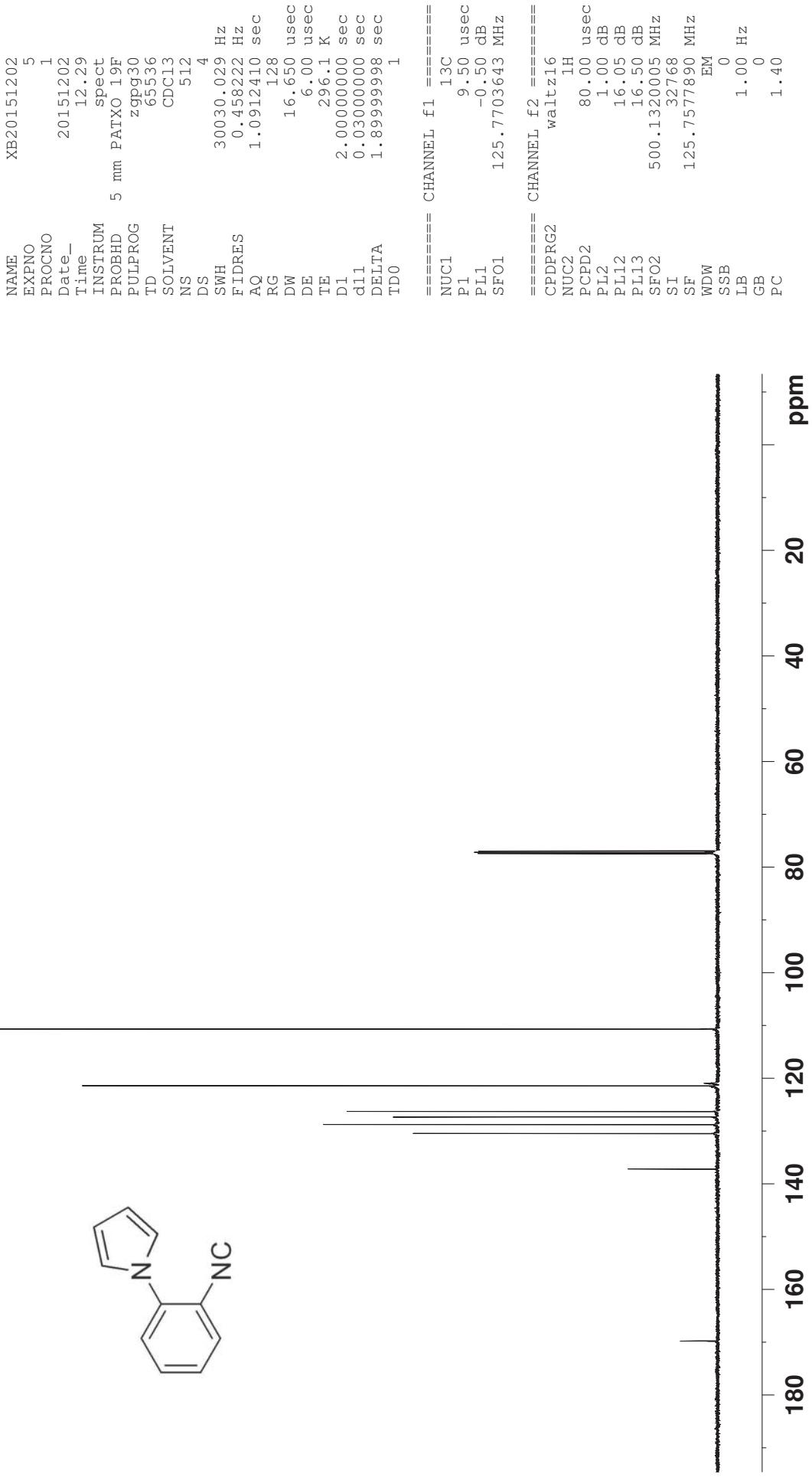
MH-3-123-S
PROTON CDCl₃

NAME XB20151202
EXPNO 3
PROCNO 1
Date 20151202
Time 11.58
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 90.5
DW 48.400 usec
DE 6.00 usec
TE 294.3 K
D1 1.0000000 sec
TD0 1
===== CHANNEL f1 ======
NUC1 1H
P1 14.14 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300129 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

6.405
6.409
6.413
7.035
7.040
7.044
7.333
7.336
7.348
7.351
7.364
7.367
7.377
7.380
7.394
7.396
7.466
7.469
7.482
7.485
7.485
7.498
7.501
7.513
7.529
7.532



MH-3-123-S
C13CPD CDC13

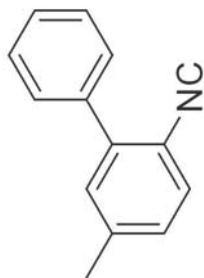


MH-3-124-S
PROTON CDC13

NAME XB20151202
 EXPNO 6
 PROCN0 1
 Date_ 20151202
 Time_ 12.35
 spect
 INSTRUM 5 mm PATXO 19F
 PROBHD Z930
 PULPROG 65536
 TD 16
 SOLVENT CDCl₃
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1720407 sec
 RG 45.3
 DW 48.400 usec
 DE 6.00 usec
 TE 295.1 K
 D1 1.00000000 sec
 TDD0 1

===== CHANNEL f1 ======
 NUC1 1H
 P1 14.14 usec
 PLL 1.00 dB
 SFO1 500.1330885 MHz
 SI 32768
 SF 500.1300129 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1

3.00
 0.96
 0.92
 0.91
 0.97
 11 10 9 8 7 6 5 4 3 2 1 0 ppm



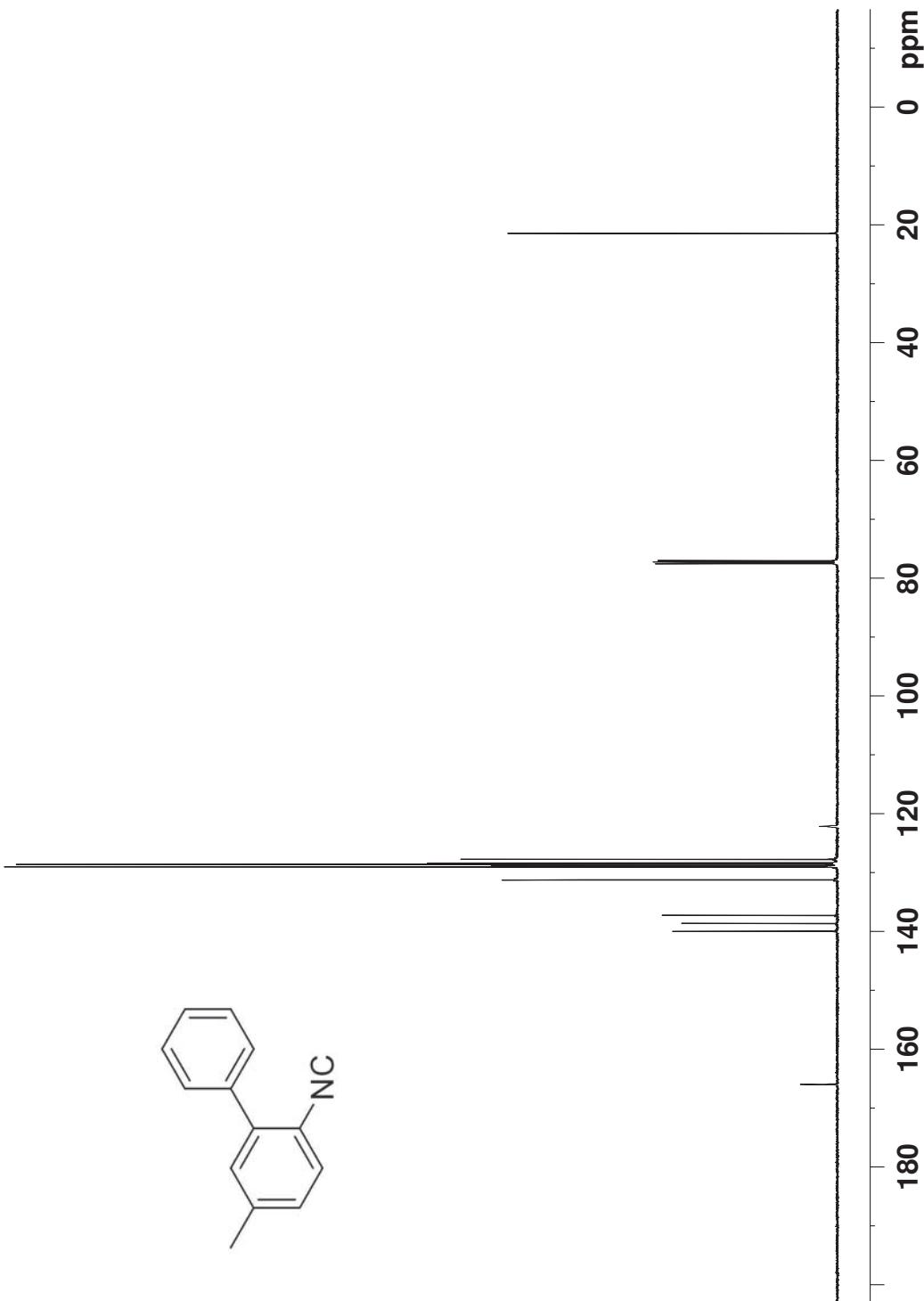
MH-3-124-S
C13CPD CDC13

— 21.41 —

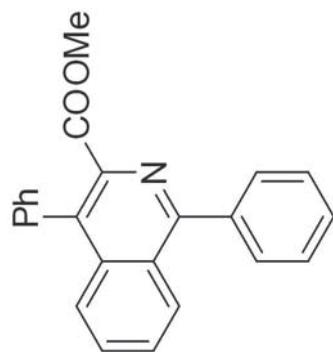
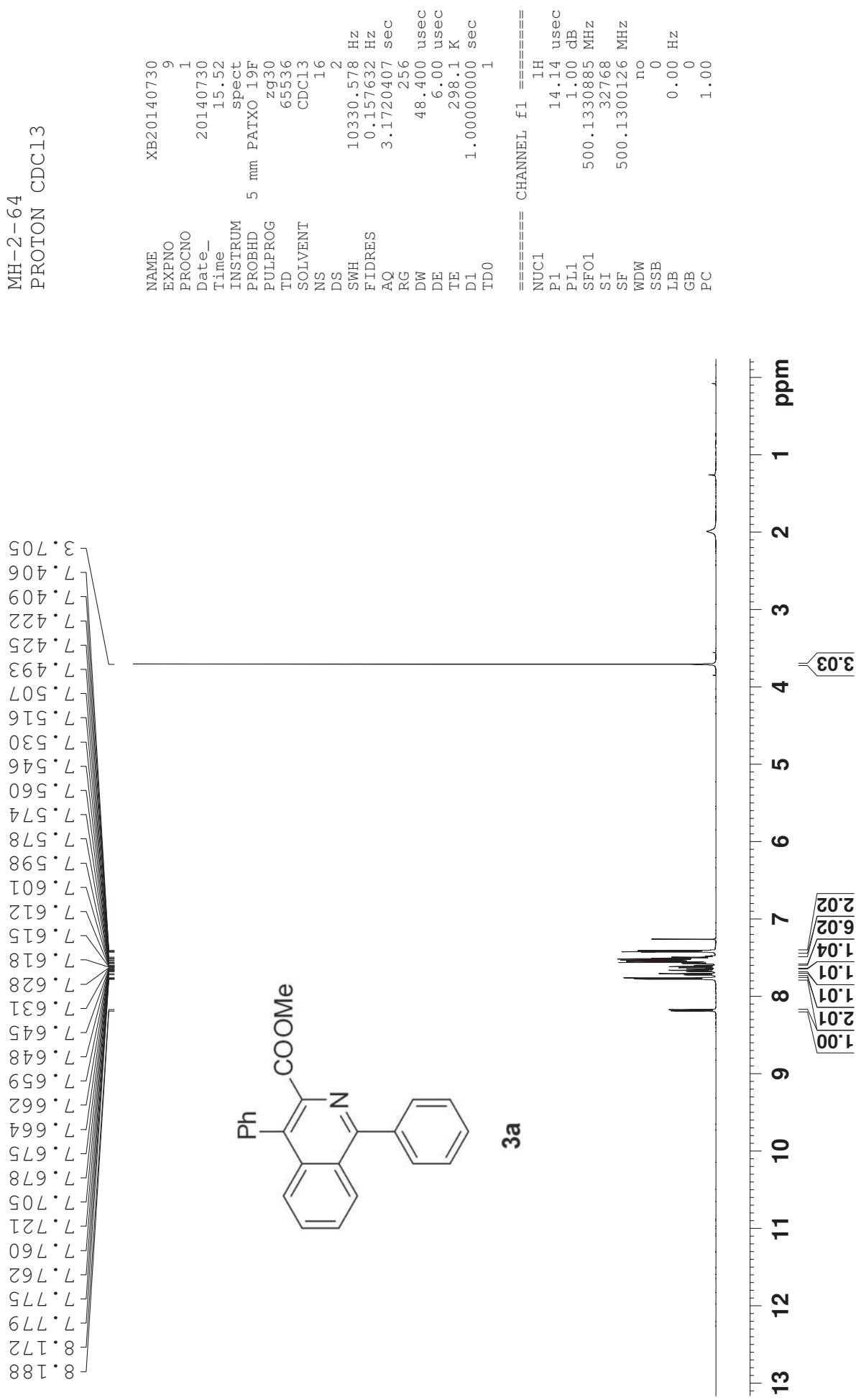
139.94
138.59
137.22
131.20
129.00
128.85
128.56
128.31
127.70

— 165.93 —

NAME XB20151202
EXPNO 7
PROCNO 1
Date 20151202
Time 13.04
INSTRUM spect
PROBHD 5 mm PAXTO 19F
PULPROG zppg30
TD 65536
SOLVENT CDCl₃
NS 512
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 128
DW 16.650 usec
DE 6.00 usec
TE 295.9 K
D1 2.0000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TDO 1
===== CHANNEL f1 =====
NUC1 13C
P1 9.50 usec
PL1 -0.50 dB
SFO1 125.7703643 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.05 dB
PL13 16.50 dB
SFO2 500.1320005 MHz
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



MH-2-64
PROTON CDC13

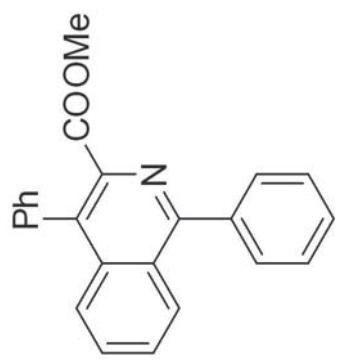


3a

MH-2-64
C13CPD CDCL3

160.35
141.40
136.67
138.88
141.40
136.15
132.92
130.75
130.36
129.97
129.10
128.58
128.45
128.42
128.17
127.91
127.32
126.87

— 52.55 —



```

NAME          XB20140730
EXPNO         10
PROCNO        1
Date_        20140730
Time       15.49
INSTRUM      spect
PROBHD      5 mm PAXTO 19F
PULPROG     zgpg30
TD           65536
SOLVENT      CDCl3
NS            428
DS             4
SWH         30030.029 Hz
FIDRES      0.458222 Hz
AQ           1.0912410 sec
RG            456.1
DW           16.650 usec
DE            6.00 usec
TE            298.9 K
D1           2.0000000 sec
D11           0.03000000 sec
d11           1.89999998 sec
DELTA        1
TDO           1

=====
CHANNEL f1 =====
NUC1          13C
P1            9.50 usec
PL1           -0.50 dB
SFO1        125.7703643 MHz

=====
CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           1.00 dB
PL12          16.05 dB
PL13          16.50 dB
SFO2        500.1320005 MHz
SI            32768
SF           125.7577735 MHz
WDW           EM
SSB            0
LB            1.00 Hz
GB            0
PC           1.40

```

200 180 160 140 120 100 80 60 40 20 0 ppm

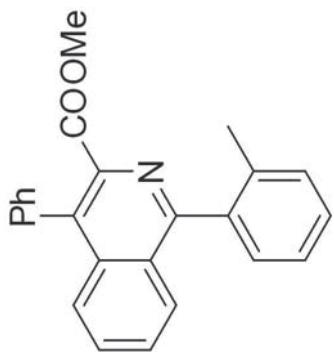
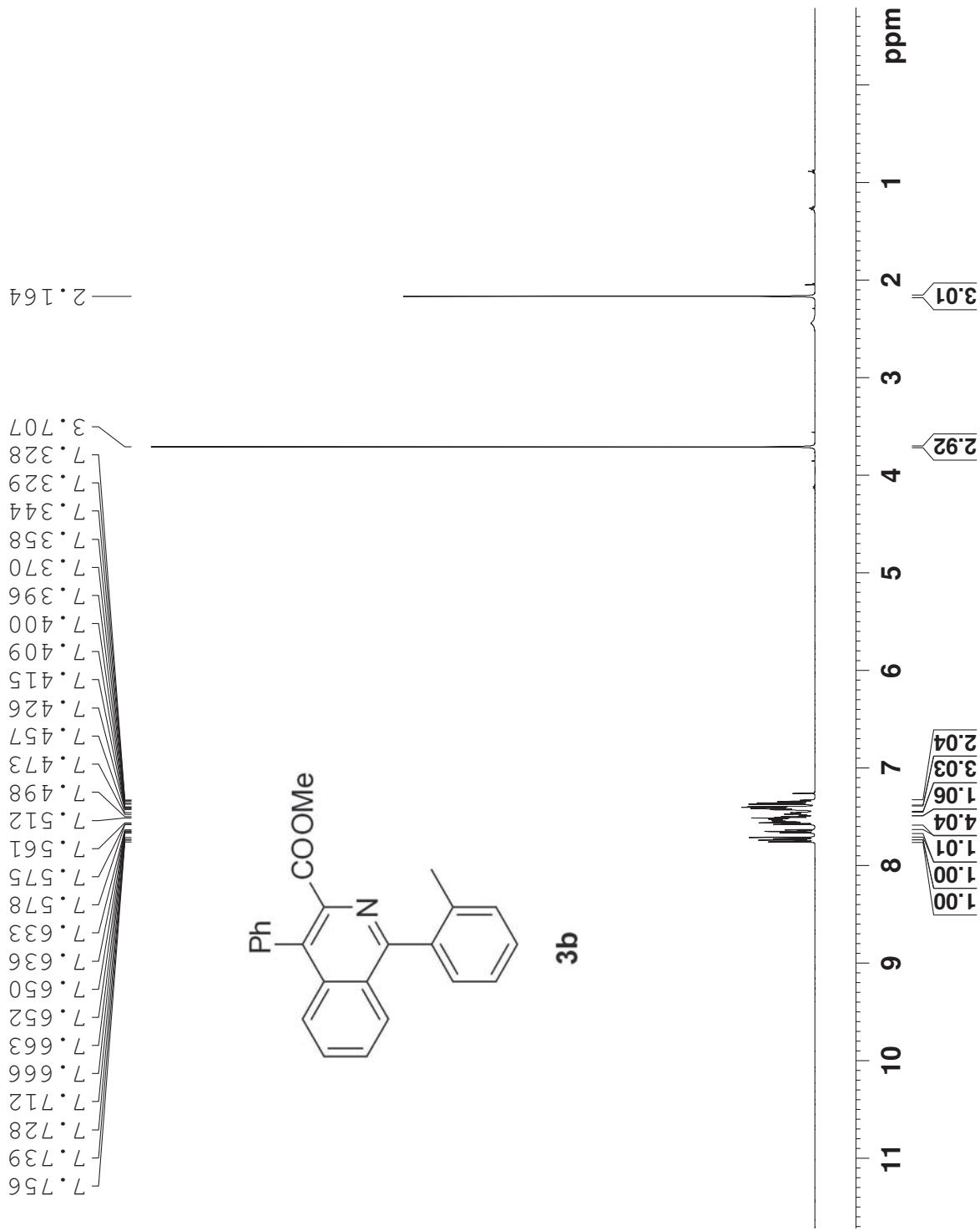
MH-2-4
PROTON CDC13

```

NAME          XB20140602
EXPNO         15
PROONO        1
Date_         20140602
Time_         19.59
INSTRUM      spect
PROBHD       5 mm
PULPROG      PATXO 1.9F
TD           zg30
SOLVENT      65536
SOLVENT      CDC13
NS           16
DS           2
SWH          10330.578 Hz
FIDRES      0.157632 Hz
AQ           3.172047 sec
RG           114
DW           48.400 usec
DE           6.00  usec
TE           295.9 K
D1           1.00000000 sec
TDO          1

===== CHANNEL f1 =====
NUC1L        1H
P1           14.14 usec
PL1          1.00 dB
SF01         500.1330885 MHz
SI           32768
SF           500.1300129 MHz
WDW          no
SSB          0
LB           0.00 Hz
GB           0
PC           1.00

```



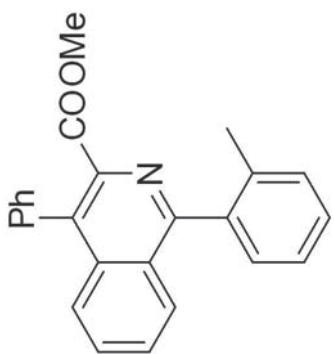
3b

MH-2-4
C13CPD CDC13

20.04

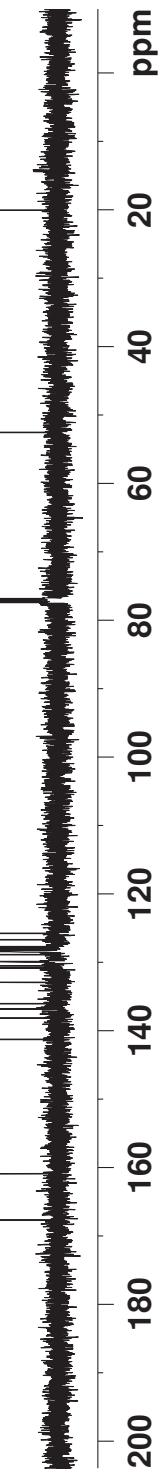
52.50

167.58
160.83
141.24
138.13
136.76
136.06
135.99
132.89
130.78
130.46
129.92
129.88
129.85
128.83
128.43
128.35
128.29
128.09
127.89
127.68
126.68
125.75

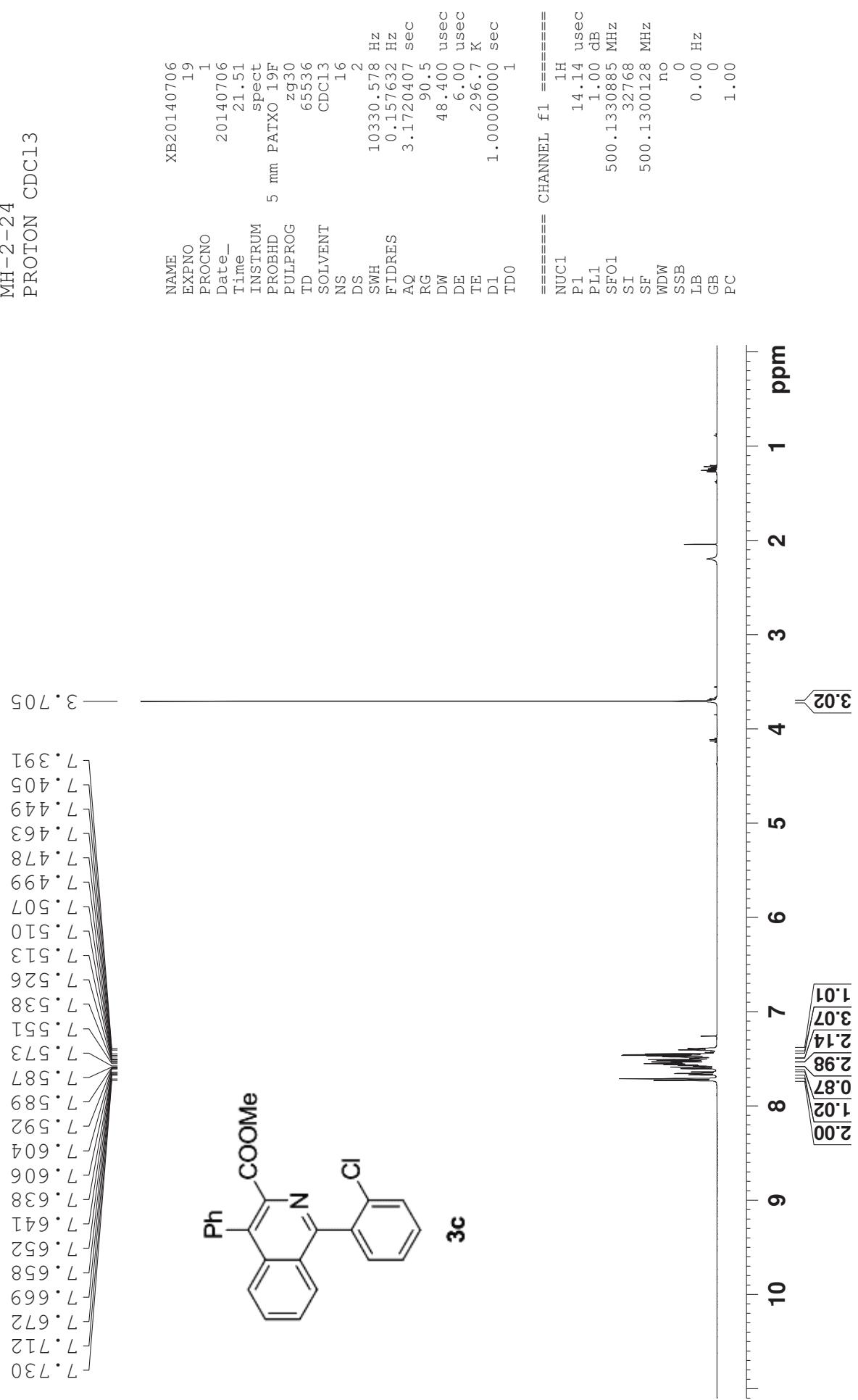


NAME	XB20140602
EXPNO	16
PROCNO	1
Date_	20140602
Time	20.08
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	Zppg30
TD	65536
SOLVENT	CDC13
NS	128
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	406.4
DW	16.650 usec
DE	6.00 usec
TE	297.0 K
D1	2.0000000 sec
d11	0.13000000 sec
DELTA	1.89999998 sec
TD0	1

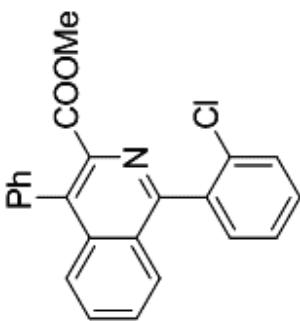
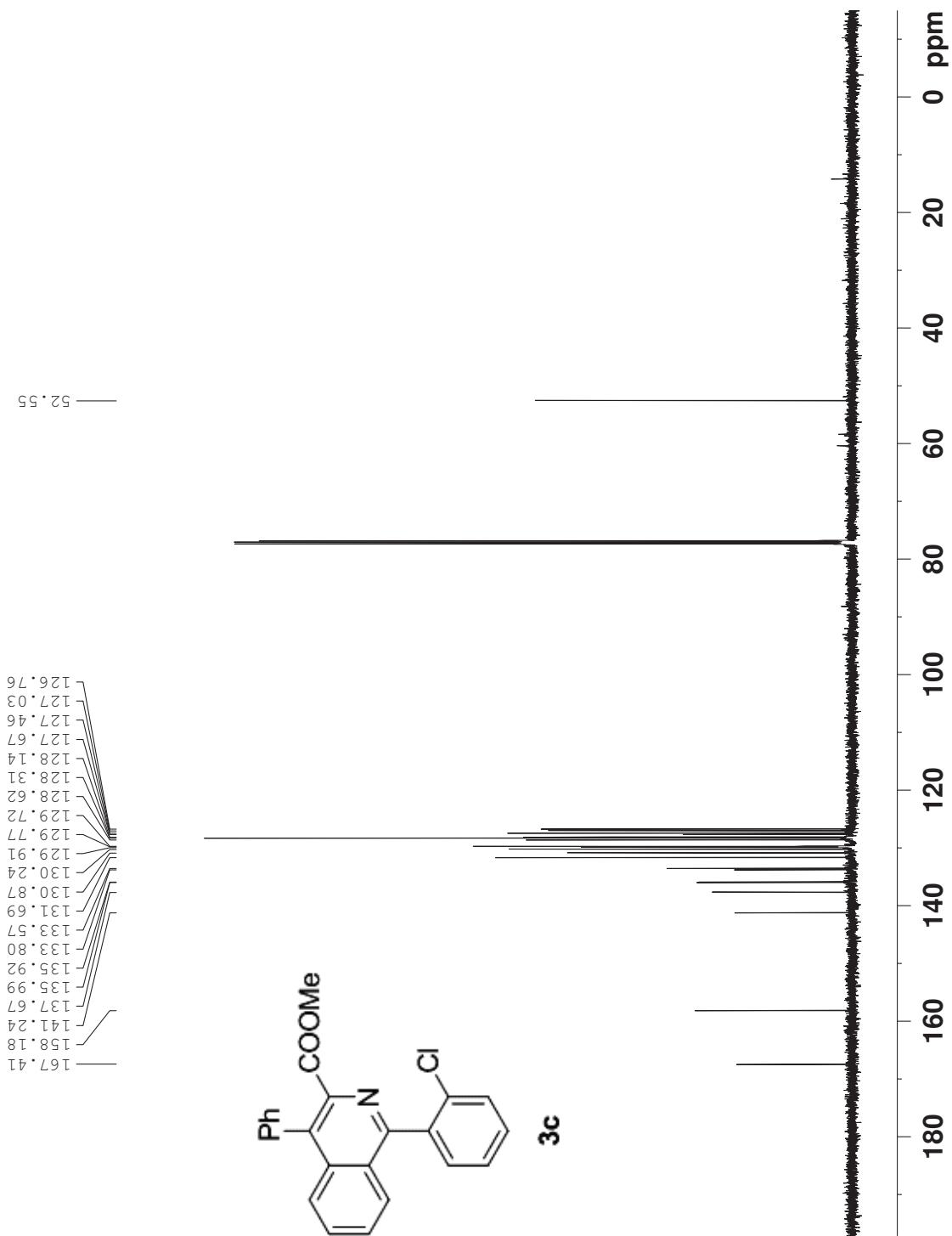
===== CHANNEL f1 =====	
NUC1	13C
p1	9.50 usec
PL1	-0.50 dB
SFO1	125.7703643 MHz
===== CHANNEL f2 =====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



MH-2-24
PROTON CDCl₃



MH-2-24
C13CPD CDC13



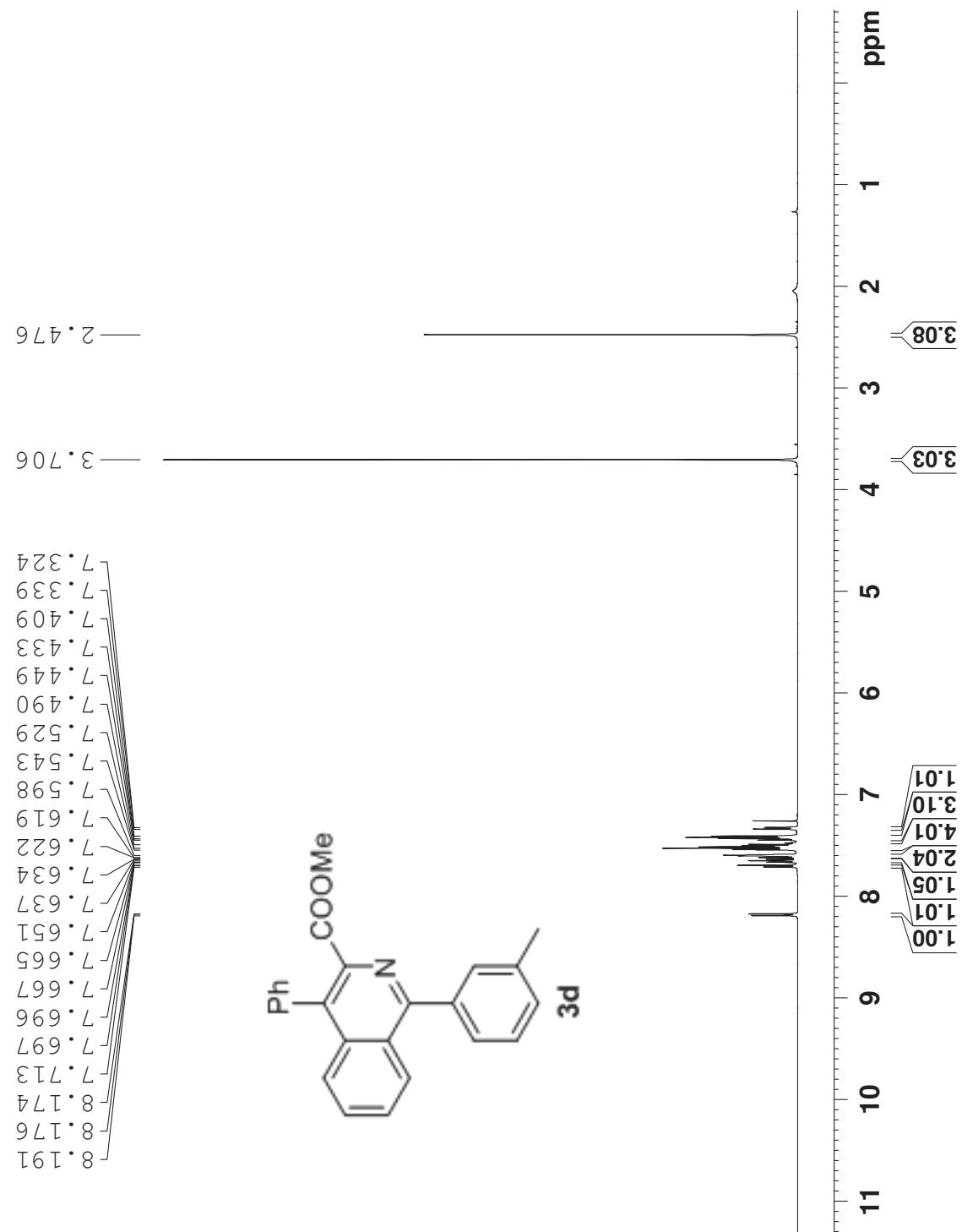
MH-2-67
PROTON CDCl₃

```

NAME          XB20140730
EXPNO         11
PROCNO        1
Date_         20140730
Time          15.57
INSTRUM      spect
PROBHD       5 mm PABXO 19F
PULPROG      zg30
TD           65536
SOLVENT       CDCl3
NS            16
DS            2
SWH          10330.578 Hz
FIDRES       0.1577632 Hz
AQ            3.1720407 sec
RG            128
DW            48.400 usec
DE            6.000 usec
TE            297.7 K
D1           1.00000000 sec
T1D0

===== CHANNEL f1 =====
NUC1          1H
P1           14.14 usec
PL1          1.00 dB
SF01         500.13330885 MHz
SI            32768
SF           500.13000129 MHz
WDW          no
SSB          0
LB          0.00 Hz
GB          0
PC          1.00

```



167.73
 160.58
 141.39
 138.81
 138.37
 136.58
 136.18
 132.76
 130.66
 130.69
 129.94
 129.79
 128.38
 128.34
 128.29
 128.11
 127.98
 127.42
 127.34
 126.78

MH-2-67
C13CPD CDC13

— 21.62 —
— 52.51 —

```

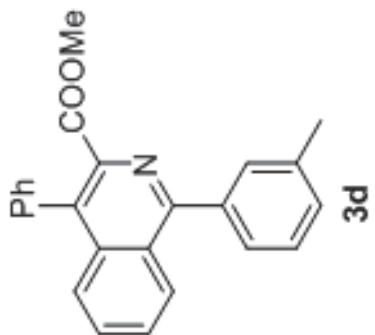
NAME          XB20140730
EXPNO         12
PROCNO        1
Date_        20140730
Time       21.09
INSTRUM      spect
PROBHD      5 mm PATXO 19F
PULPROG      zgpg36
TD           65536
SOLVENT      CDC13
NS            256
DS             4
SWH          30030.029 Hz
FIDRES     0.458222 Hz
AQ           1.0912410 sec
RG           1448.2
DW           16.650 usec
DE           6.00 usec
TE           298.3 K
D1          2.0000000 sec
d11          0.03000000 sec
DELTA        1.8999998 sec
TD0            1

```

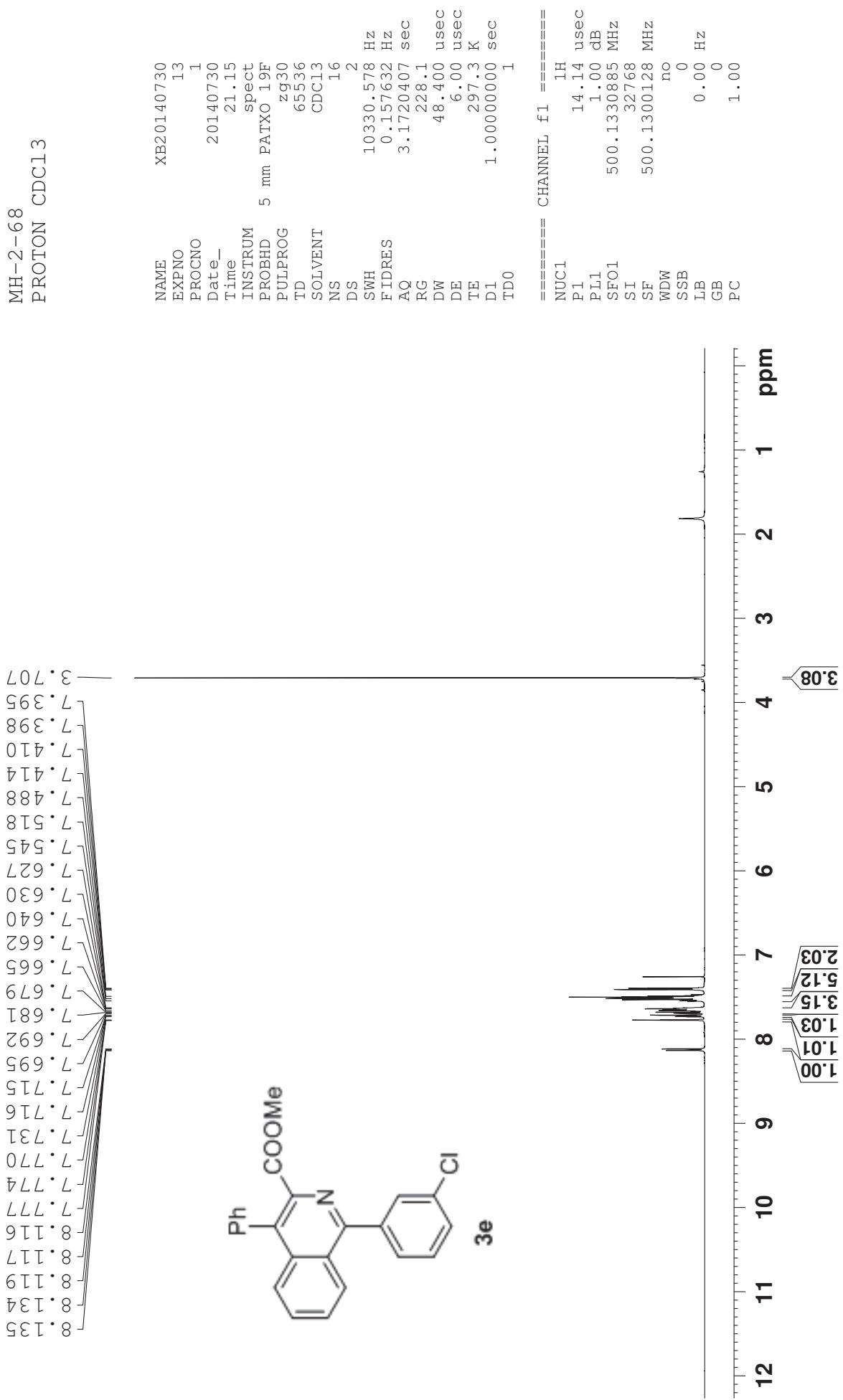
```

===== CHANNEL f1 =====
NUC1          13C
P1            9.50 usec
PL1          -0.50 dB
SFO1        125.7703643 MHz
===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           1.00 dB
PL12          16.05 dB
PL13          16.50 dB
SFO2        500.1320005 MHz
SI            32768
SF           EM
WDW          0
SSB          1.00 Hz
LB           0
GB           0
PC           1.40

```



MH-2-68
PROTON CDCl₃



MH-2-68
C13CPD CDC13

167.56
141.44
136.68
135.97
134.67
133.43
130.91
130.39
129.90
129.82
129.76
128.73
128.45
128.26
127.41
127.15
127.02

52.61

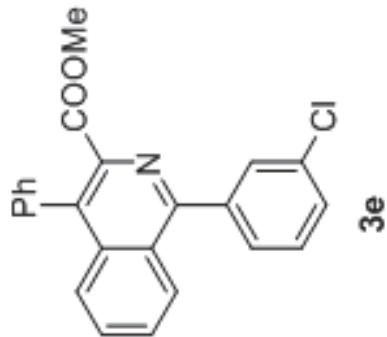
```

NAME          XB20140730
EXPNO         14
PROCNO        1
Date_         20140730
Time          21.31
INSTRUM      spect
PROBHD       5 mm PATXO 19F
PULPROG      zgp930
TD           65536
SOLVENT      CDCl3
NS            256
DS            4
SWH          30030.029 Hz
FIDRES       0.458222 Hz
AQ           1.0912410 sec
RG           228.1
DW           16.650 usec
DE           6.00 usec
TE           298.2 K
T1           2.00000000 sec
D1           0.03000000 sec
DELTA        1.899999998 sec
TD0          1
TDO          1

===== CHANNEL f1 =====
NUC1          13C
P1            9.50 usec
PL1           -0.50 dB
SFO1        125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           1.00 dB
PL12          16.05 dB
PL13          16.50 dB
SFO2        500.1320005 MHz
SI            32768
SF           125.7577744 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           1.40

```



MH-2-28
PROTON CDCl₃ D:\\ deng 47

```

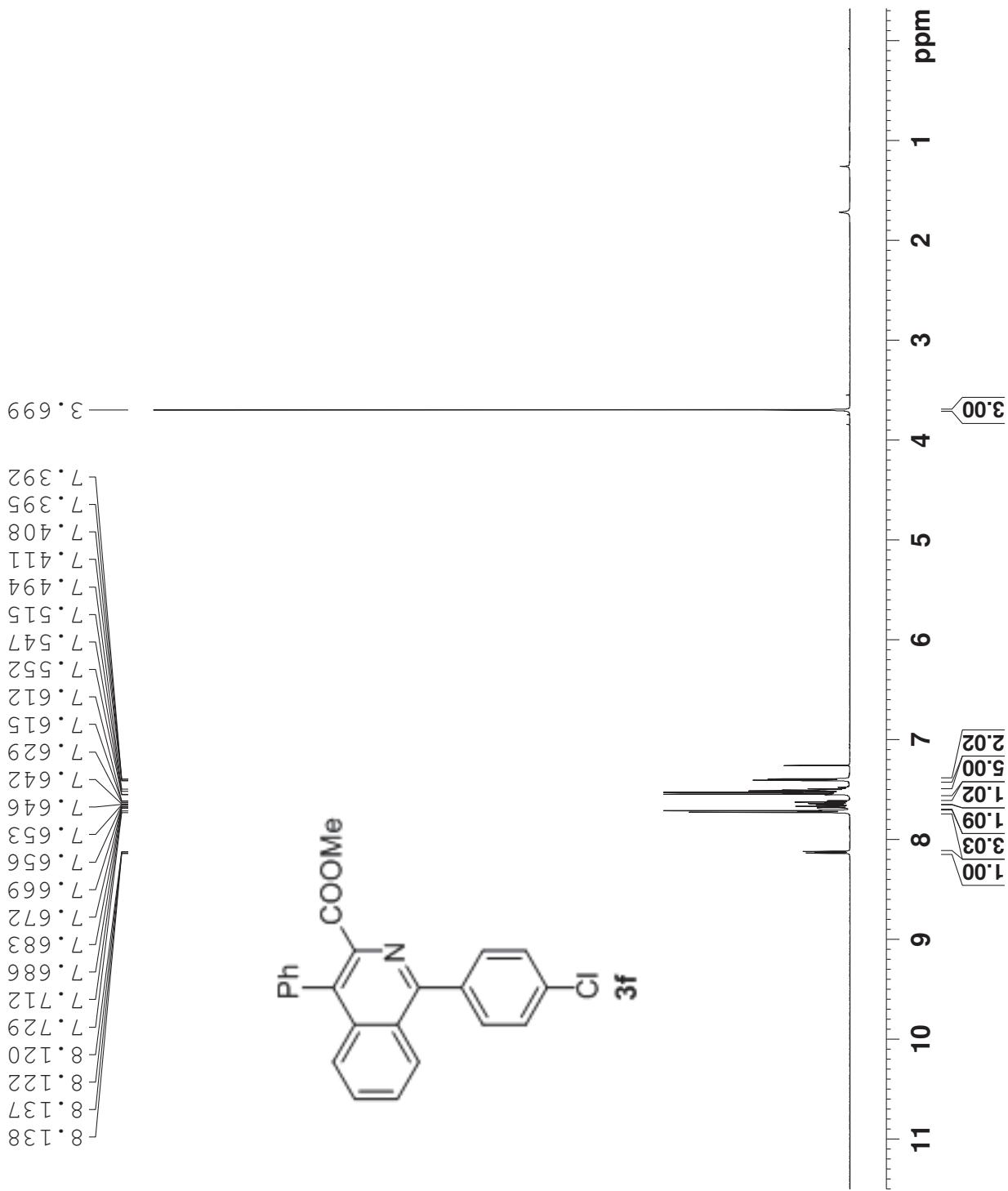
NAME          XB20141023
EXPNO         1
PROCNO        1
Date_        20141023
Time       11.21
INSTRUM     spect
PROBHD      5 mm PAXO 19F
PULPROG     zg30
TD           65536
SOLVENT      CDCl3
NS            16
DS            2
SWH         10330.578 Hz
FIDRES     0.157632 Hz
AQ          3.1720407 sec
RG           228.1
DW           48.400 usec
DE           6.00 usec
DE          296.6 K
D1        1.00000000 sec
TD0             1

```

```

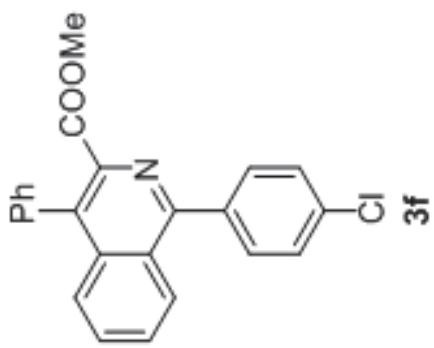
===== CHANNEL f1 =====
NUC1          1H
P1          14.14 usec
PL1           1.00 dB
SFO1      500.1330885 MHz
SI            32768
SF          500.1300129 MHz
WDW           no
SSB            0 Hz
LB            0 Hz
GB            0
PC           1.00

```



MH-2-28
C13CPD CDCl₃

— 52.46 —
— 158.92 —
— 167.55 —
— 141.41 —
— 137.30 —
— 136.58 —
— 135.91 —
— 135.06 —
— 131.60 —
— 130.72 —
— 129.80 —
— 128.73 —
— 128.55 —
— 128.33 —
— 128.12 —
— 127.30 —
— 127.04 —
— 126.90 —



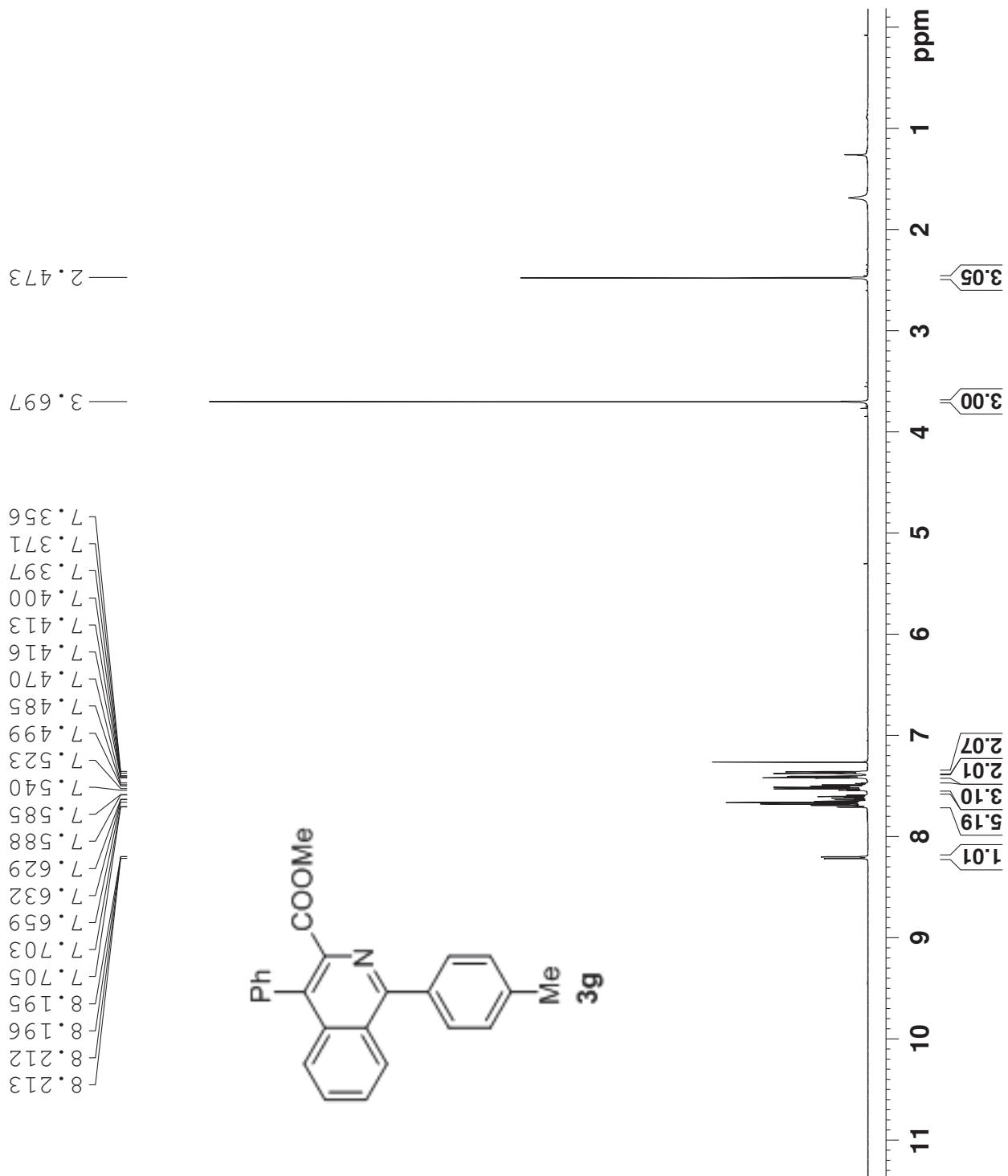
```

NAME          XB20140705
EXPNO         20
PROCNO        1
Date_         20140705
Time          22.53
INSTRUM       spect
PROBHD        5 mm PAXTO 19F
PULPROG       zgpg30
TD            65536
SOLVENT        CDCl3
NS             256
DS              4
SWH           30030.029 Hz
FIDRES       0.458222 Hz
AQ            1.0912410 sec
RG             256
DW             16.650 usec
DE             6.00 usec
TE             297.7 K
TEC            2.0000000 sec
D1             0.03000000 sec
d11            1.89999998 sec
DELTA          1.89999998 sec
TD0             1

=====
CHANNEL f1
NUC1          13C
PCPD2        80.00 usec
PL2          1.00 dB
PL12         16.05 dB
PL13         16.50 dB
SFO1        500.1320005 MHz
SI             32768
SF           125.7577890 MHz
WDW           no
SSB           0
LB            0.00 Hz
GB            0
PC           1.40

```

MH-2-31
PROTON CDCl₃

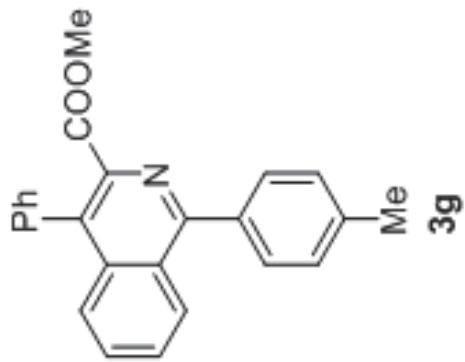


MH-2-31
C13CPD CDCl₃

— 21.42 —

— 52.39 —

— 167.68 —
160.32
138.92
136.13
135.96
132.52
130.54
130.19
129.88
129.15
128.30
128.23
128.01
127.86
127.23
126.71



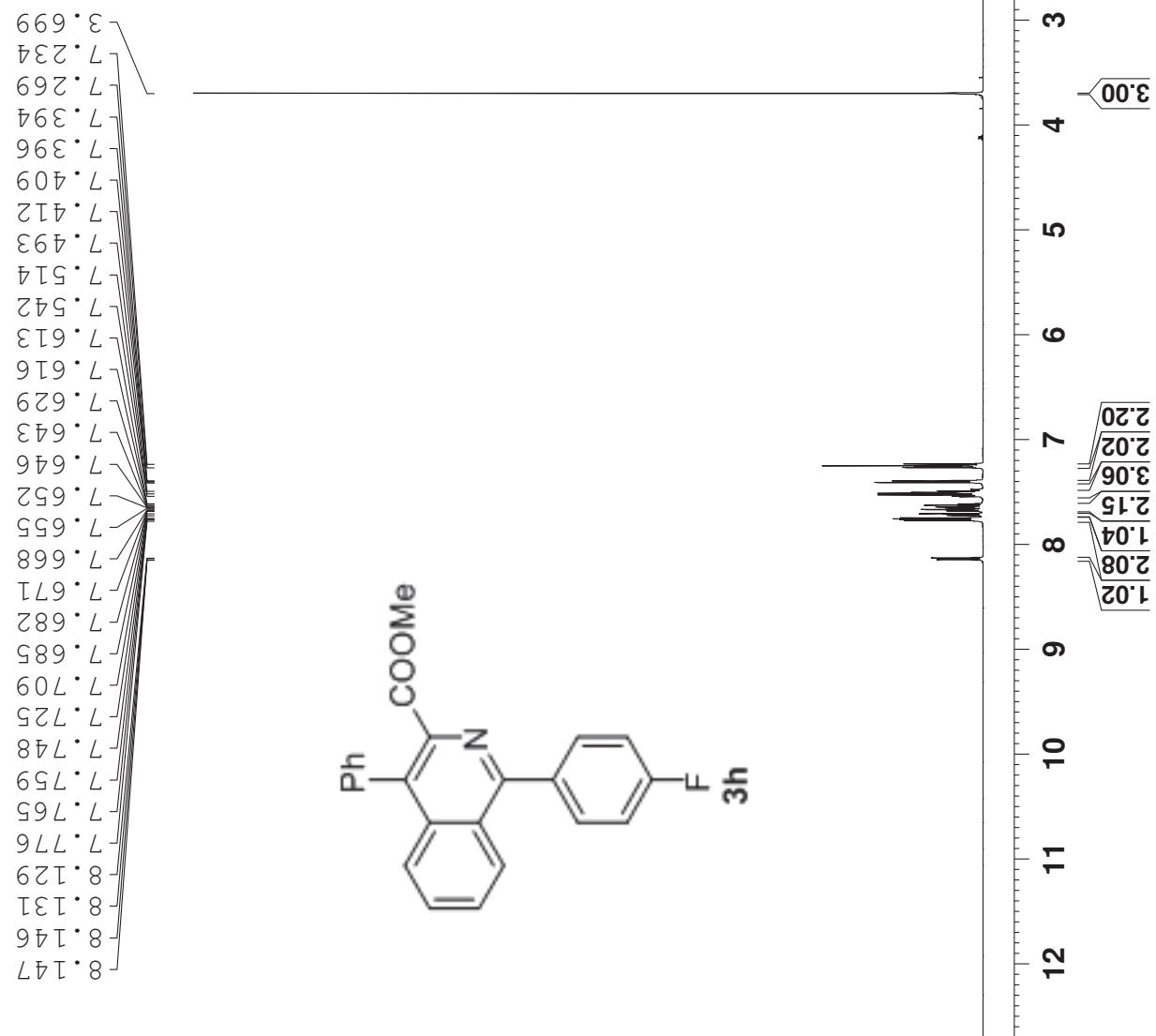
NAME	XB20140706
EXPNO	18
PROCNO	1
Date_	20140706
Time	21.45
INSTRUM	spect
PROBHD	5 mm PAXO 19F
PULPROG	zpgpg30
TD	65536
SOLVENT	CDCl ₃
NS	256
DS	4
SWH	300030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	203.2
DW	16.650 usec
DE	6.00 usec
TE	297.9 K
TM	2.0000000 sec
D1	0.03000000 sec
DELTA	1.89999998 sec
TDO	1
===== CHANNEL f1 =====	
NUC1	13C
P1	9.50 usec
PL1	-0.50 dB
SFO1	125.7703643 MHz
===== CHANNEL f2 =====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	322768
SF	125.7577890 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.40

MH-2-32 AGAIN
PROTON CDCl₃

NAME	XB20140705
EXPNO	14
PROCNO	1
Date—	20140705
Time	23.11
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	Zg30
TD	65536
SOLVENT	CDCl ₃
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	181
DW	48.400 usec
DE	6.00 usec
TE	296.5 K
D1	1.00000000 sec
TD0	1

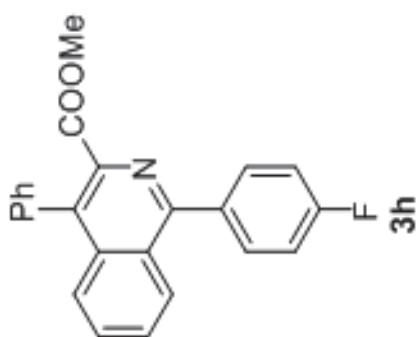
===== CHANNEL f1 =====

NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300129 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



MH-2-32
C13CPD CDC13

167.57
164.35
162.37
141.32
159.11
162.35
141.32
134.91
135.95
136.58
134.94
132.92
132.13
132.06
130.67
129.81
128.48
128.09
127.44
127.13
126.87
125.60
125.43



```

NAME          XB20140705
EXPNO         16
PROCNO        1
Date_         20140705
Time          22.08
INSTRUM       spect
PROBHD       5 mm PATXO 19F
PULPROG      zppg30
TD           256
SOLVENT       CDCl3
NS            4
DS            30030.029 Hz
SWH          0.458222 Hz
FIDRES       1.0912410 sec
AQ            128
RG            16.650 usec
DW            6.00 usec
DE            2.97.6 K
TE            2.0000000 sec
D1            0.0300000 sec
d11           1.8999999 sec
DETA          1
TD0           1

```

```

=====
CHANNEL f1 =====
NUC1          13C
P1            9.50 usec
PL1           -0.50 dB
SFO1         125.7703643 MHz

```

```

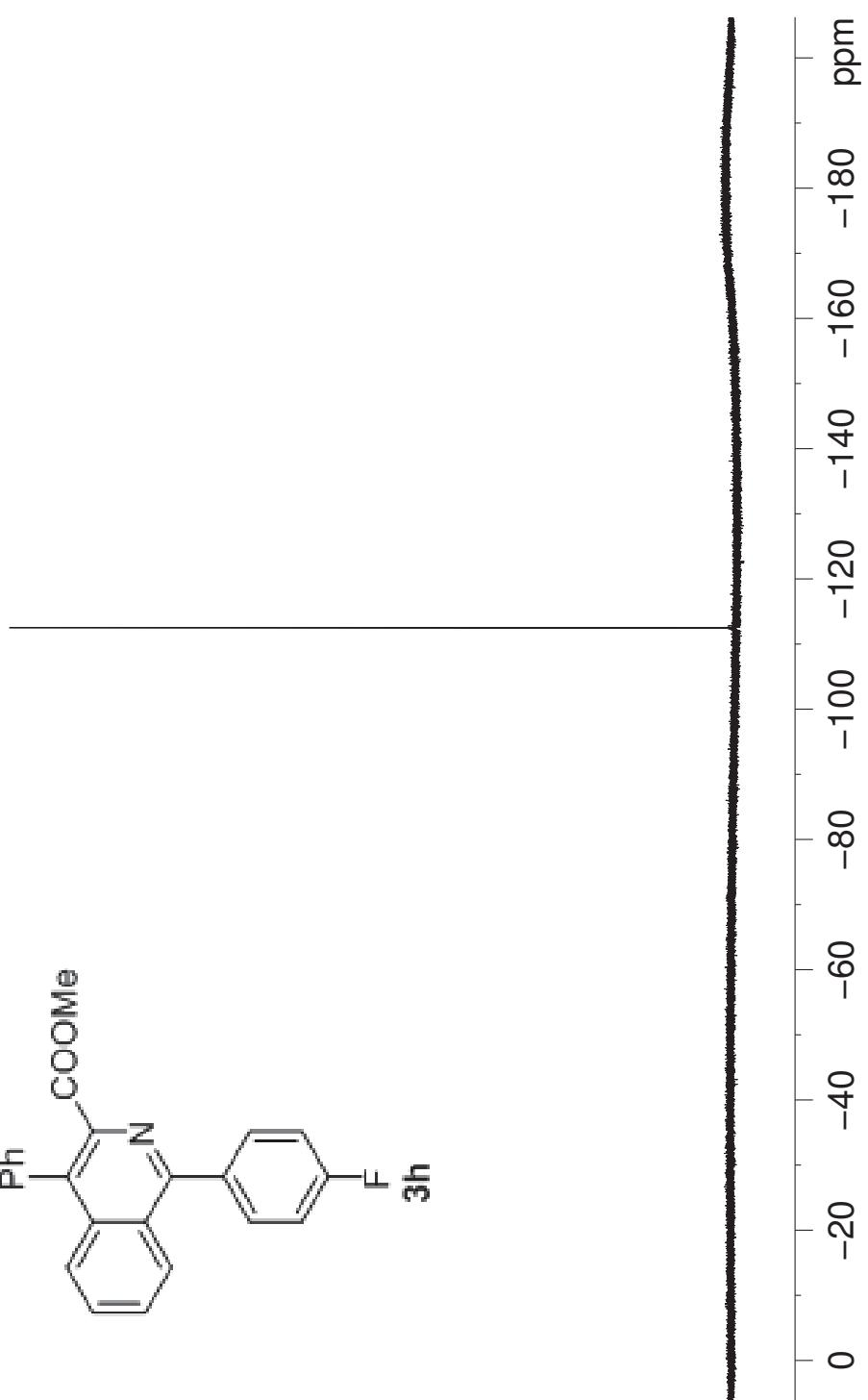
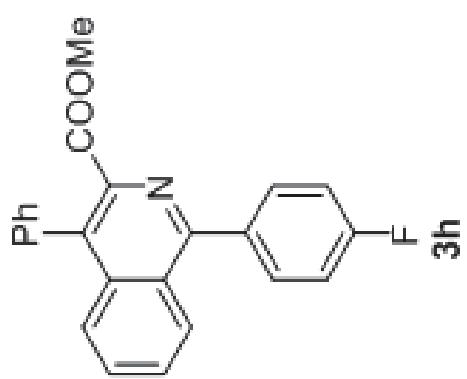
=====
CHANNEL f2 =====
waltz16
NUC2          1H
PCPD2        80.00 usec
PL2            1.00 dB
PL12          16.05 dB
PL13          16.50 dB
SFO2         500.1320005 MHz
SI             327.68
SF           125.7577890 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB           0
PC           1.40

```

— 52.45 —

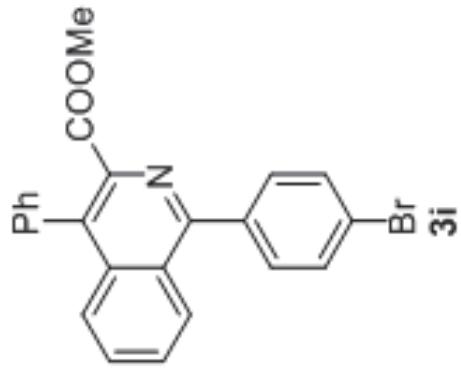
MH-2-32
19Fdeft CDCl₃ D:\\ deng 46

-112.517
-112.537
-112.546
-112.559
-112.565
-112.576



MH-2-34
PROTON CDCl₃

8.134
8.119
7.729
7.707
7.691
7.662
7.645
7.629
7.616
7.613
7.546
7.542
7.514
7.494
7.479
7.409
7.406
7.393
7.390

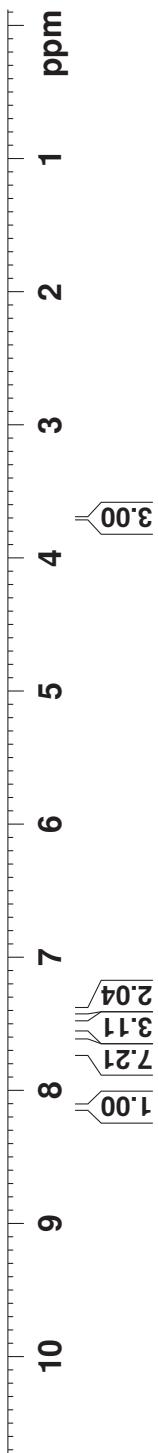


```

=====
NAME          XB20141023
EXPNO         7
PROCNO        1
Date_         20141023
Time          12:00
INSTRUM      spect
PROBHD       5 mm PATXO 19F
PULPROG      zg30
TD           65536
SOLVENT      CDCl3
NS            16
DS            2
SWH          10330.578 Hz
FIDRES       0.157632 Hz
AQ            3.1720407 sec
RG            287.4
DW            48.400 usec
DE            6.000 usec
TE            296.8 K
D1           1.00000000 sec
TD0           1

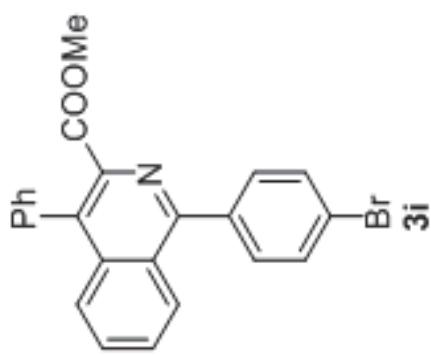
===== CHANNEL f1 =====
NUC1          1H
P1           14.14 usec
PL1          1.00 dB
SFO1        500.1330885 MHz
SI            32768
SF          500.1300129 MHz
WDW           no
SSB            0
LB            0.00 Hz
GB            0
PC           1.00

```



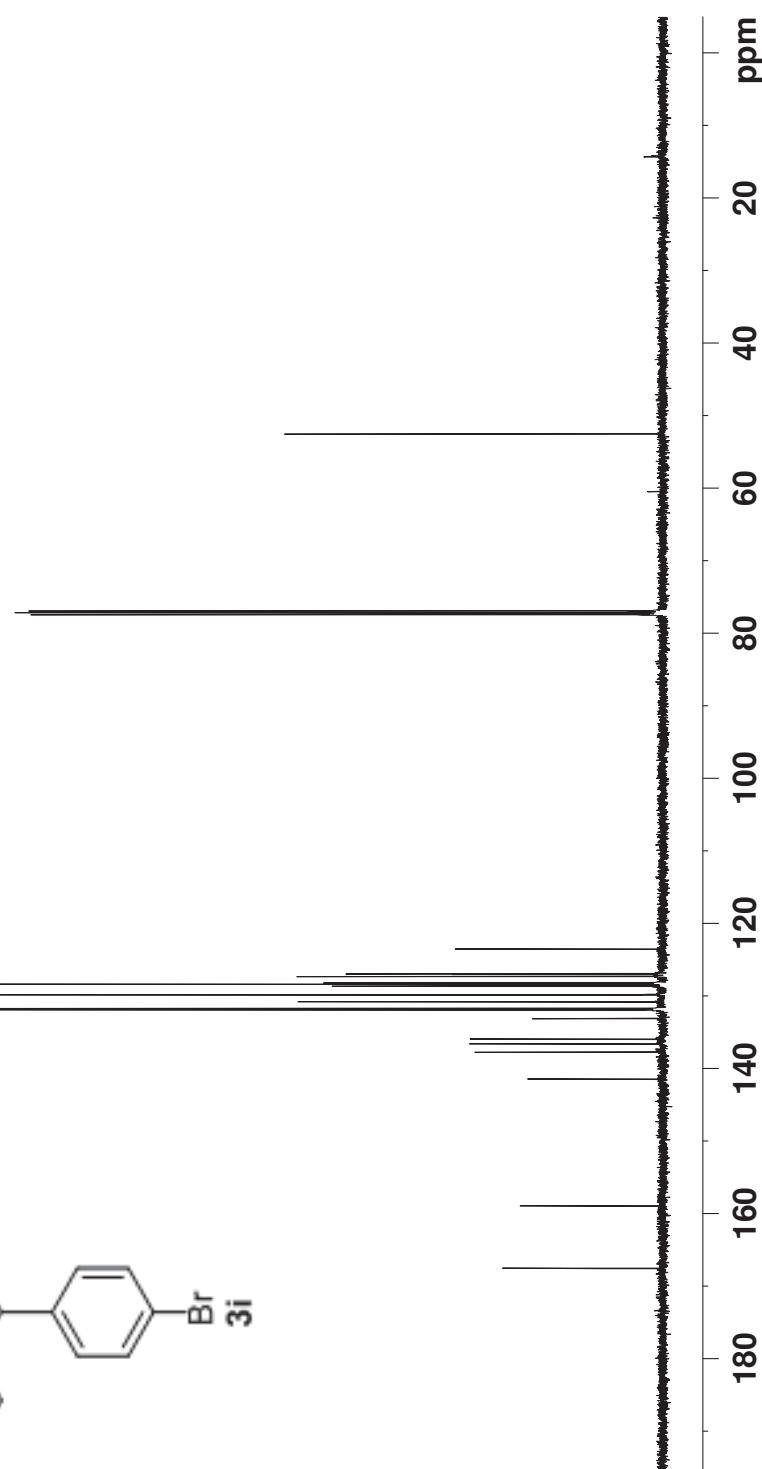
MH-2-34
C13CPD CDC13

158.92
167.52
141.42
137.73
136.58
135.90
133.09
131.88
130.69
130.77
129.80
128.59
129.77
128.35
128.14
127.28
126.98
126.91
123.49



NAME	XB20140705
EXPNO	18
PROCNO	1
Date_	20140705
Time	22.34
INSTRUM	spect
PROBHD	5 mm PAIXO 19F
PULPROG	29pg30
TD	65536
SOLVENT	CDC13
NS	256
DS	4
SWH	30030.029 Hz
FIDRES	0.45822 Hz
AQ	1.0912410 sec
RG	287.4
DW	16.650 usec
DE	6.00
TE	297.7 K
D1	2.0000000 sec
d11	0.0300000 sec
DELTA	1.8999998 sec
TDO	1
===== CHANNEL f1 =====	
NUC1	13C
P1	9.50 usec
PL1	-0.50 dB
SFO1	125.7703643 MHz
===== CHANNEL f2 =====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32.768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

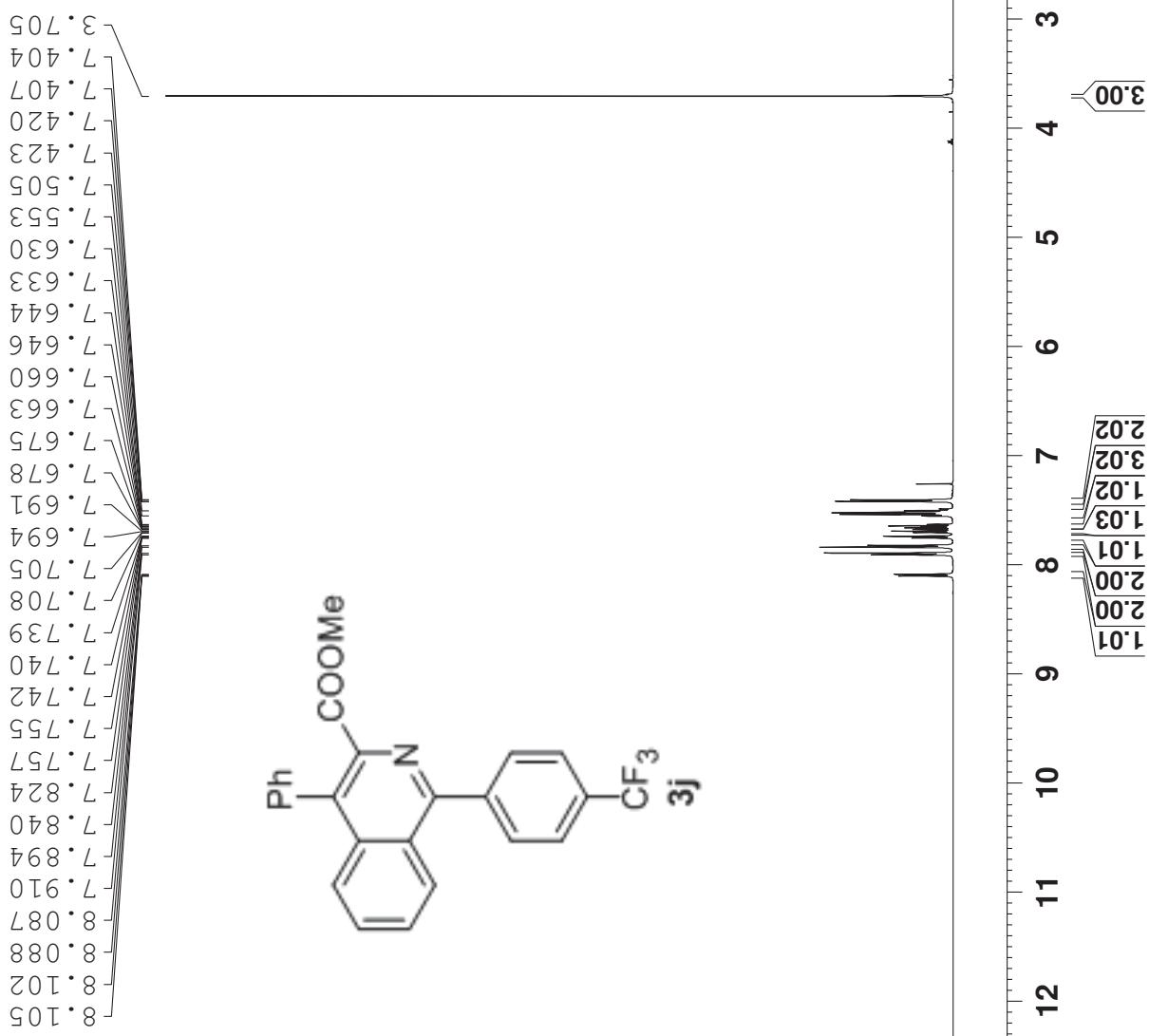
— 52.47 —



MH-2-35
PROTON CDCl₃

NAME	XB20140705
EXPNO	11
PROCNO	1
Date_	20140705
Time_	21.29
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	zg30
TD	65536
SOLVENT	CDCl ₃
NS	16
DS	2
SWH	1.03330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	161.3
DW	48.400 usec
DE	6.00 usec
TE	296.5 K
D1	1.00000000 sec
TD0	1

===== CHANNEL f1 =====	
NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	32768
SF	500.1300126 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



MH-2-35
C13CPD CDCl₃

52.49

120.89
123.06
125.22
125.43
125.46
125.49
125.52
125.99
126.99
127.09
127.39
128.19
128.36
128.73
129.77
130.61
130.85
130.88
131.14
133.46
135.79
136.58
141.47
142.40
158.61
167.46

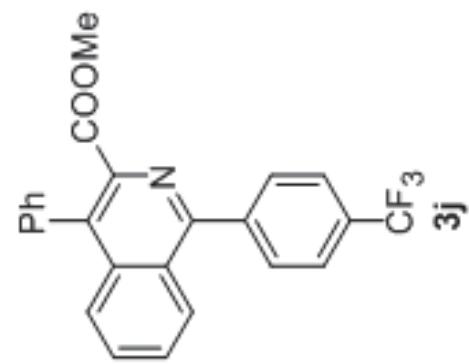
```

NAME          XB20140705
EXPNO         13
PROCNO        1
Date_         20140705
Time          21.46
INSTRUM      spect
PROBHD       5 mm PATXO 19F
PULPROG      29ppg30
TD           65536
SOLVENT       CDCl3
NS            256
DS             4
SWH          30030.029 Hz
FIDRES       0.458222 Hz
AQ            1.0912410 sec
RG            128
DW           16.650 usec
DE            6.00 usec
TE            297.6 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA         1.89999998 sec
TD0            1

===== CHANNEL f1 =====
NUC1          13C
P1            9.50 usec
PL1           -0.50 dB
SFO1        125.7703643 MHz

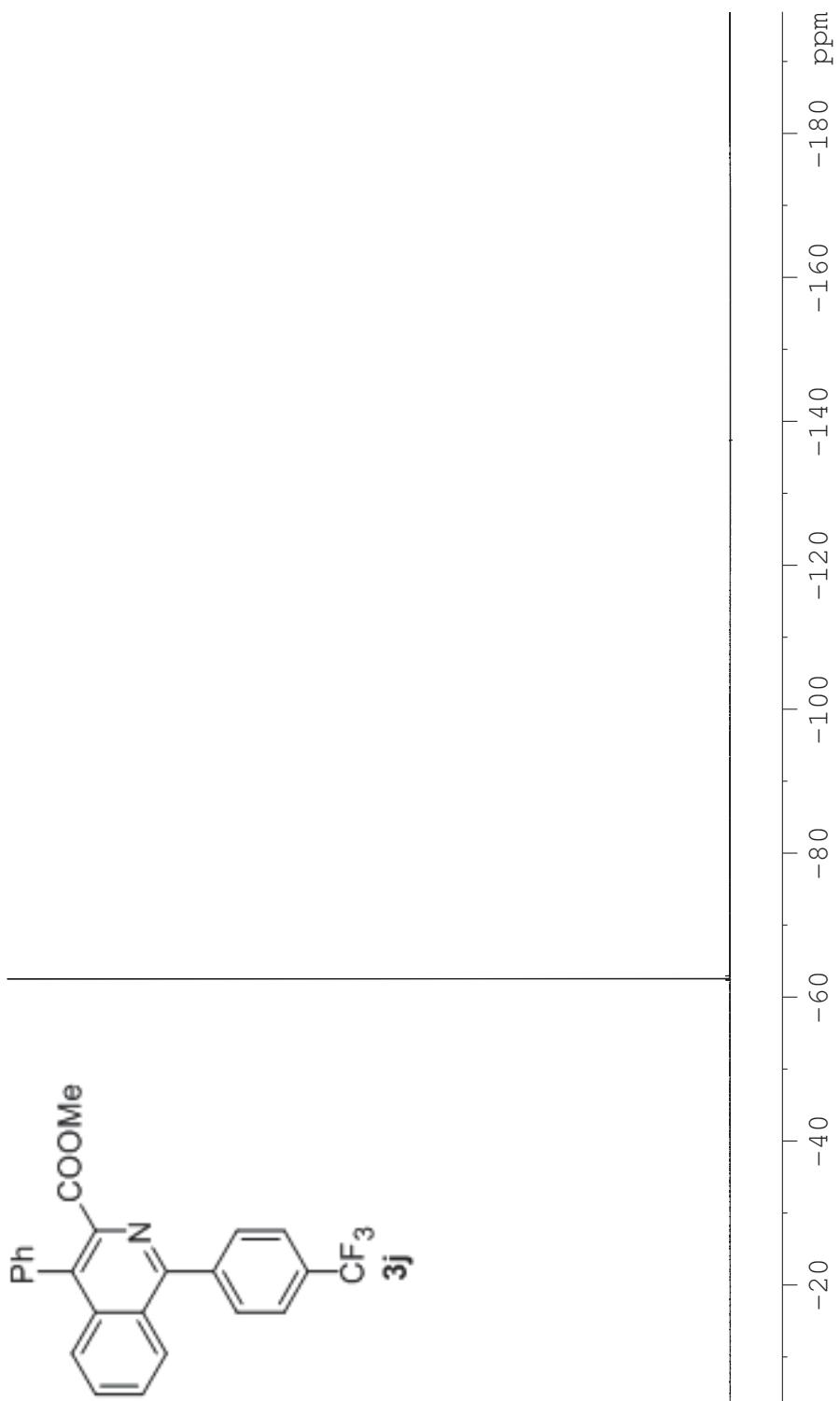
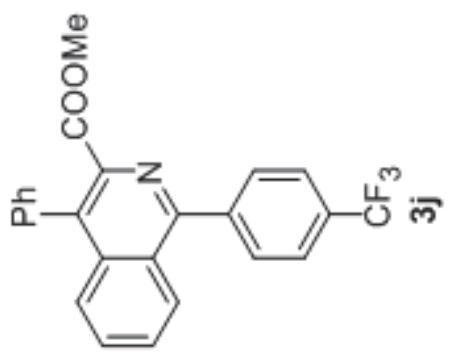
===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           1.00 dB
PL12          16.05 dB
PL13          16.50 dB
SFO2        500.1320005 MHz
SI             32768
SF           125.7577890 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC          0.20

```



19Fdeft MH-2-35
CDC13 D:\\ deng 45

-62.584



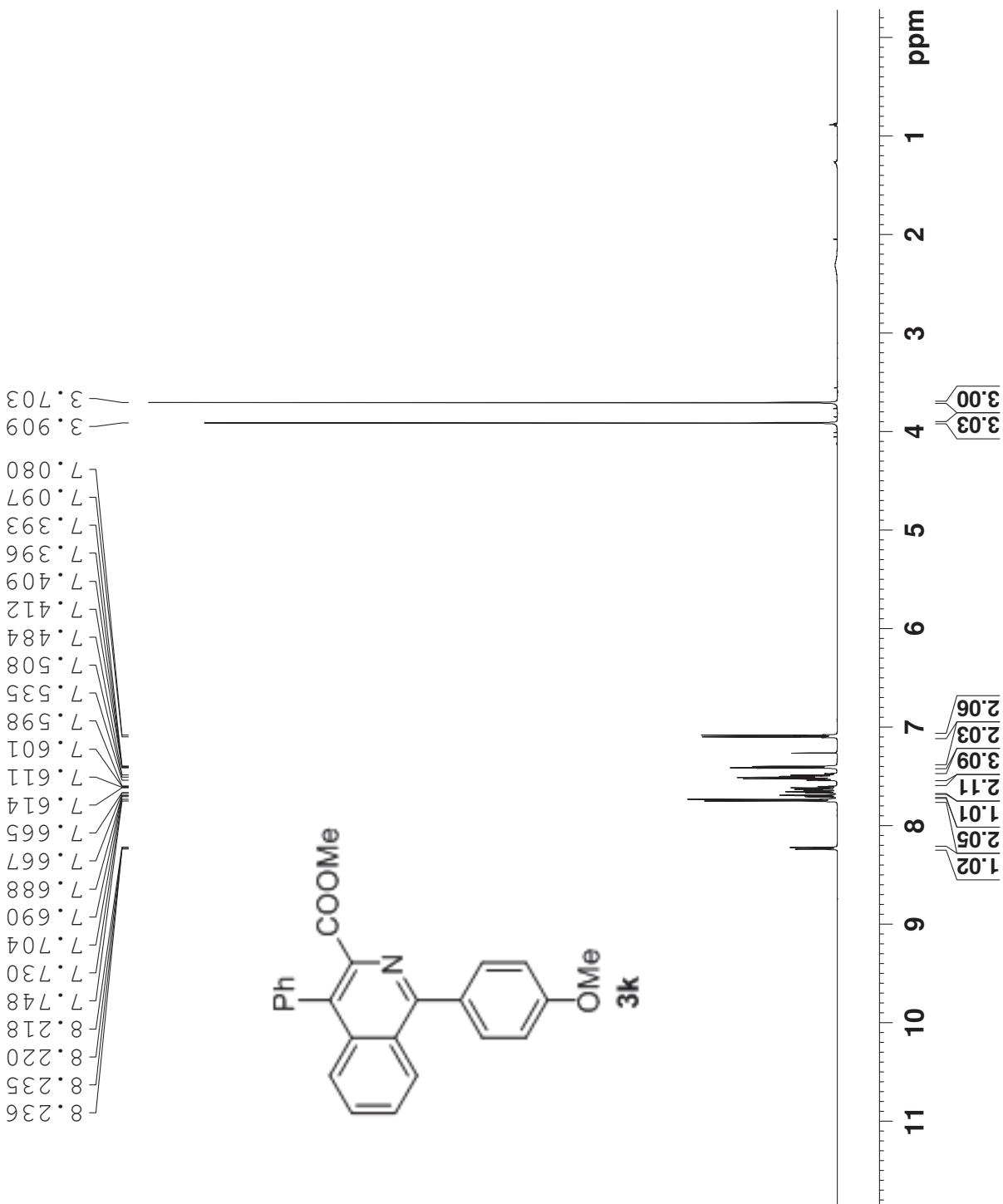
MH-2-2
PROTON CDC13

```

NAME          XIB20140602
EXPNO         12
PROCNO        1
Date_        20140602
Time         19.42
INSTRUM      spect
PROBHD      5 mm PATXO 19F
PULPROG     zg30
TD           65536
SOLVENT      CDCl3
NS            16
DS            2
SWH          10330.578 Hz
FIDRES       0.157632 Hz
AQ            3.1720407 sec
RG            203.2
DW           48.400 usec
DE            6.00 usec
TE            295.8 K
D1           1.00000000 sec
TDO          1

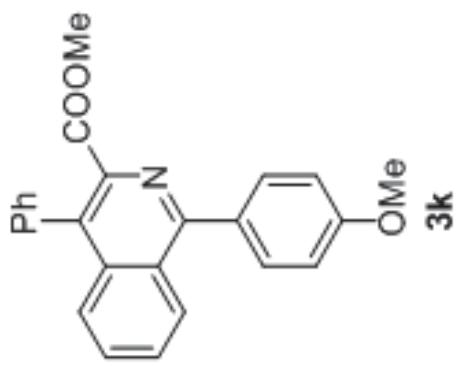
===== CHANNEL f1 =====
NUC1          1H
P1            14.14 usec
PL1           1.00 dB
SFO1         500.1330885 MHz
SI             32768
SF           500.13000129 MHz
WDW           no
SSB           0
LB            0.00 Hz
GB           0
PC           1.00

```



MH-2-2
C13CPD CDC13

— 167. 57
— 160. 42
— 159. 85
— 136. 64
— 132. 41
— 131. 71
— 130. 59
— 129. 86
— 128. 35
— 127. 27
— 127. 01
— 127. 18
— 126. 74
— 113. 95



NAME	EXPNO	PROCNO	Date—	Time	INSTRUM	PATROHD	PULPROG	SOLVENT	DS	SWH	FIDRES	AQ	RG	DW	DE	TE	D1	d1	DELTAL
XB20140602	14	1	20140602	19.53	spec	5 mm	PATXO 19F	zgfb30		300030	0.029	Hz		16.650	usec				
										0.45	0.222	Hz		6.00	usec				
										1.0912410	0.1	sec		2.970	K				
														2.000000000	sec				
														0.030000000	sec				
														1.899999998	sec				
														1.0	1				

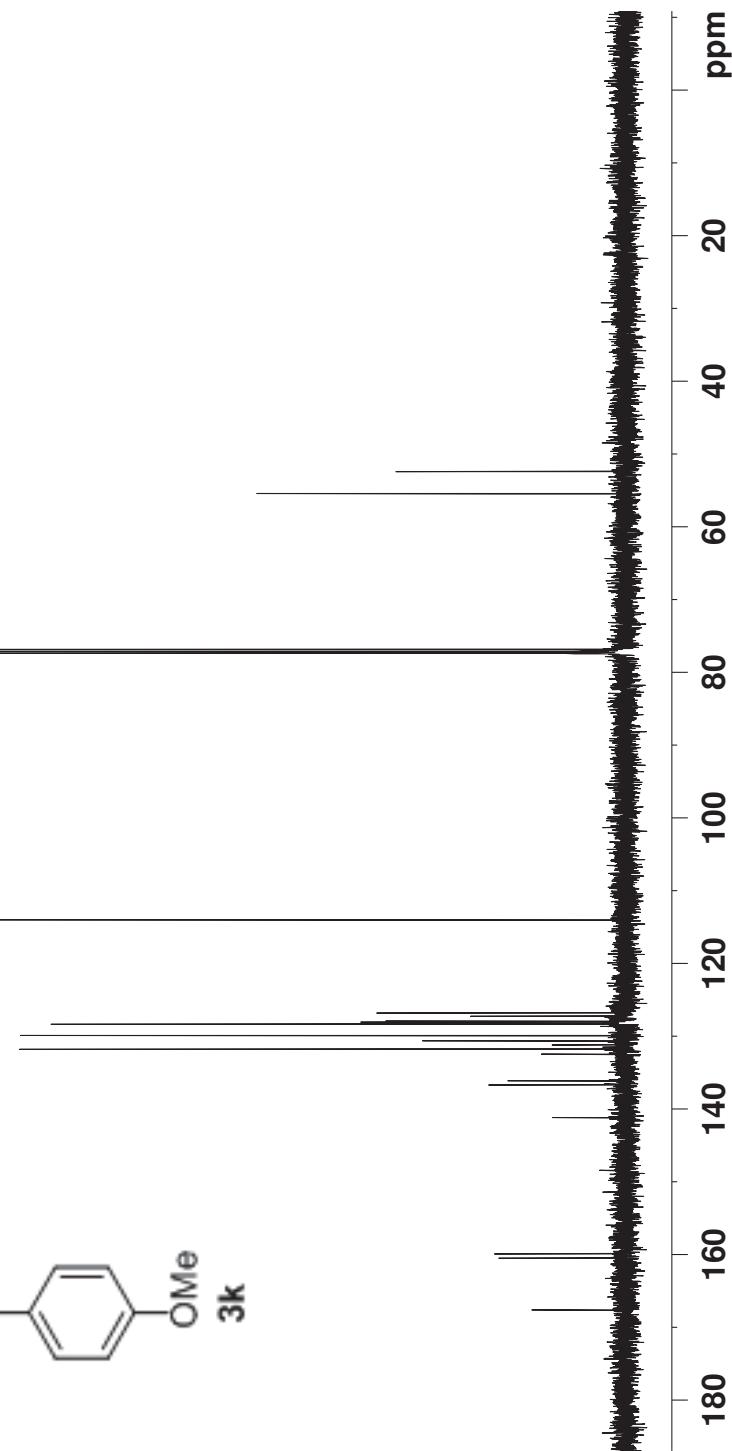
```

===== CHANNEL f1 =====
NUC1          13C
P1            9.50  usec
P2            0.50  dB
PPL1          -7.00  dB
SF01          125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00  usec
PL2           1.00   dB
PL1.2         16.05  dB
PL1.13        16.50  dB
SF02          500.1320005 MHz
SI             32768
SF             125.7577900 MHz
WDW           EM

===== CHANNEL f3 =====
SSB           0
LB            1.00  Hz
GB           0
PC           1.40

```



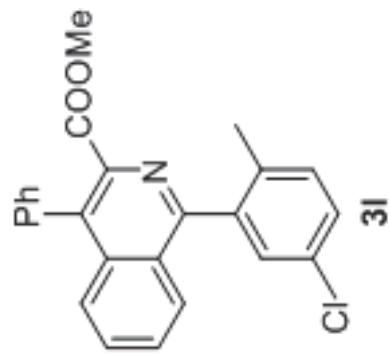
MH-2-6
PROTON CDCl₃

NAME XB20140926
EXPNO 15
PROCNO 1
Date 20140926
Time 15.32
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 143.7
DW 48.400 usec
DE 6.00 usec
TE 297.4 K
D1 1.00000000 sec
TDD0 1
===== CHANNEL f1 =====
NUC1 1H
P1 14.14 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300129 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

— 2.113 —

— 3.705 —

7.287
7.303
7.370
7.420
7.444
7.459
7.501
7.550
7.576
7.578
7.589
7.592
7.595
7.606
7.608
7.648
7.650
7.661
7.664
7.667
7.678
7.681
7.707
7.717
7.734

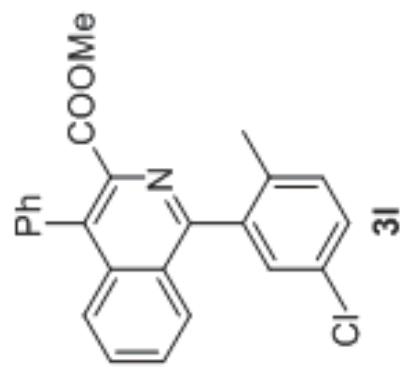


MH-2-6
C13CPD CDC13

19.57

52.62

126.92
127.31
127.71
128.24
128.38
128.47
128.79
128.92
128.93
129.90
129.92
130.99
131.61
131.86
133.42
135.45
135.96
136.18
139.91
141.46
159.36
167.57



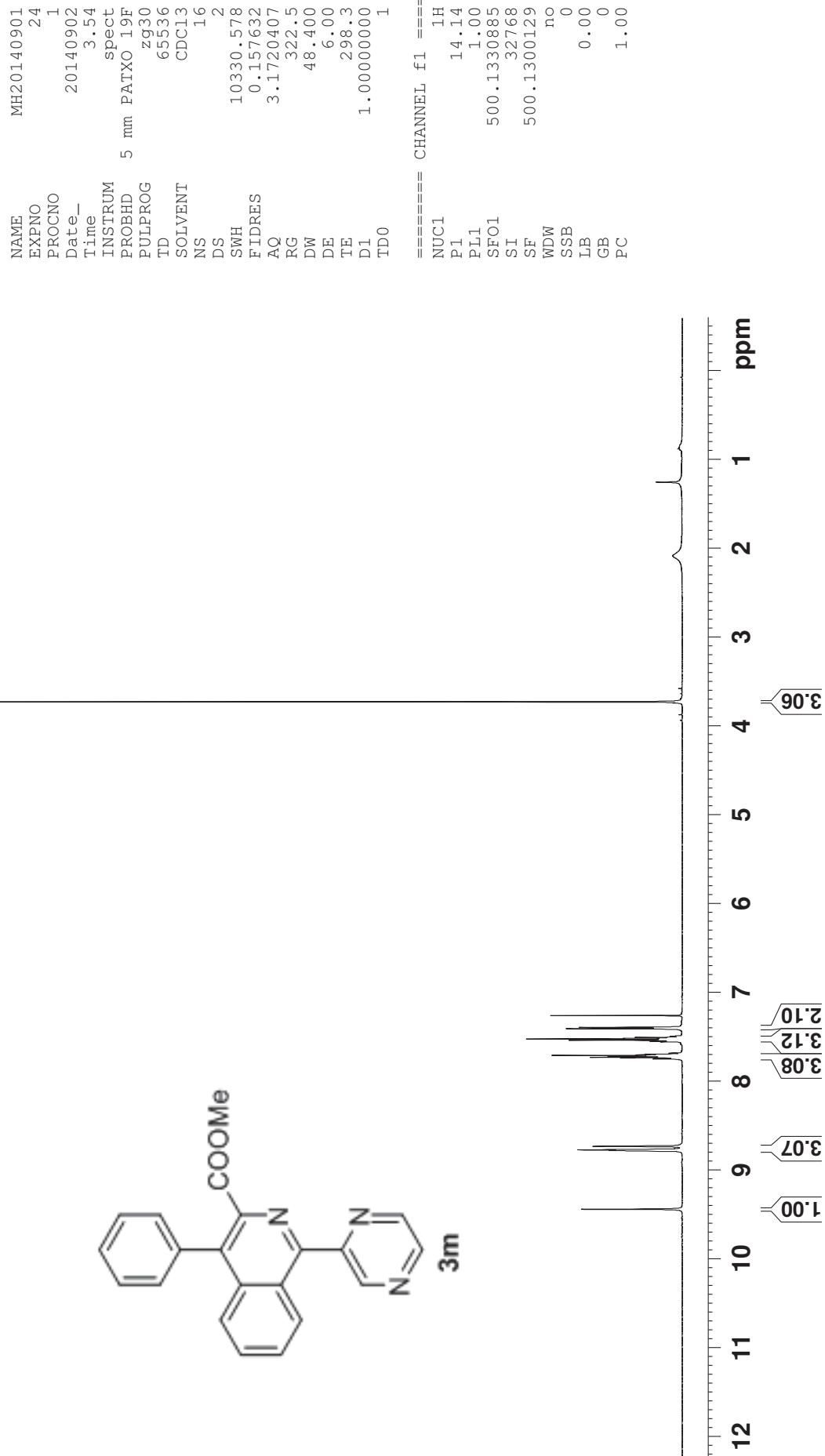
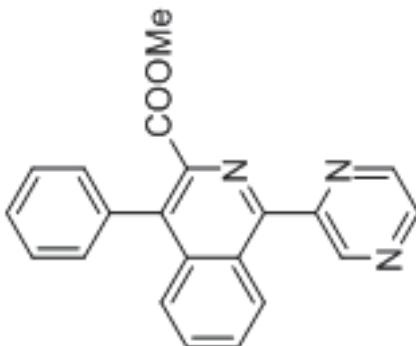
NAME XB20140926
EXPNO 16
PROCNO 1
Date_ 20140926
Time 15.47
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zppg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 300030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 228.1
DW 16.650 usec
DE 6.00 usec
TE 298.5 K
D1 2.0000000 sec
d11 0.0300000 sec
DELTA 1.89999998 sec
TD0 1
===== CHANNEL f1 =====
NUC1 13C
P1 9.50 usec
PL1 -0.50 dB
SFO1 125.7703643 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.05 dB
PL13 16.50 dB
SFO2 500.1320005 MHz
SI 32768
SF 125.7577890 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40



MH-2-78
PROTON CDCl₃

— 3.726 —

7.391
7.393
7.406
7.409
7.505
7.523
7.694
7.697
7.746
7.749
8.733
8.772
8.779
8.781
9.439



MH-2-78
C13CPD CDC13

— 52.64 —

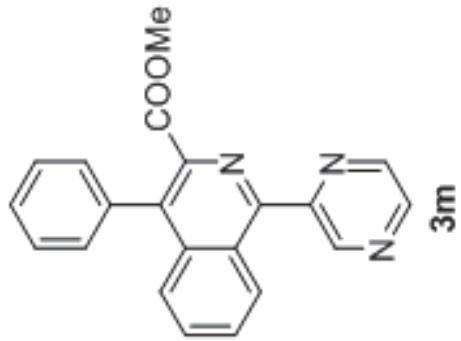
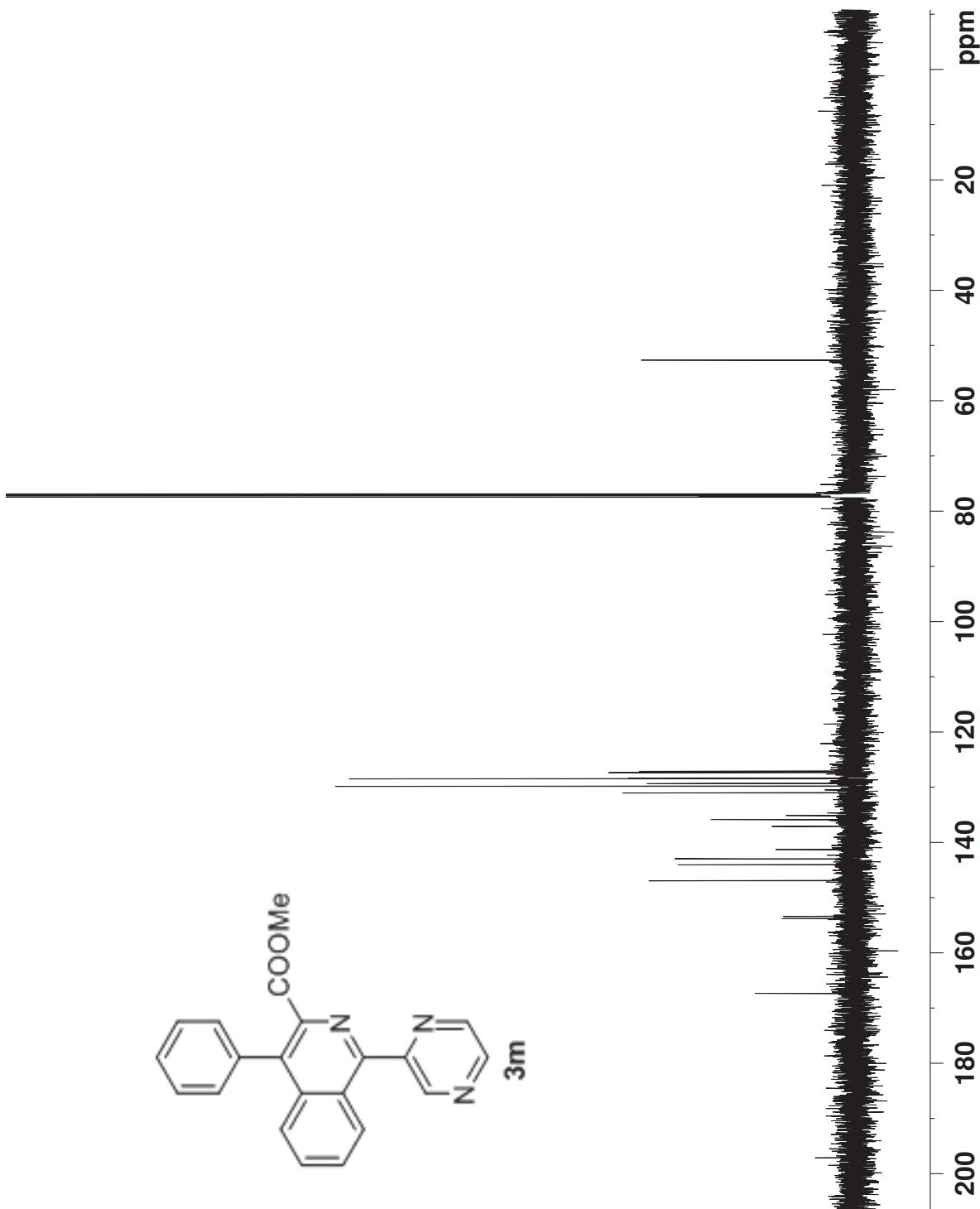
```

NAME          MH20140901
EXPNO         25
PROCNO        1
Date_         20140902
Time          4.10
INSTRUM      spect
PROBHD      5 mm PATXO 19F
PULPROG      zpg30
TD           65536
SOLVENT       CDC13
NS            256
DS             4
SWH          300030.029 Hz
FIDRES      0.458222 Hz
AQ            1.0912410 sec
RG           912.3
DW           16.650 usec
DE            6.00 usec
TE           299.5 K
D1           2.0000000 sec
d1           0.03000000 sec
DELTAB       1.89999998 sec
TD0          1

===== CHANNEL f1 =====
NUC1          13C
P1            9.50 usec
PL1          -0.50 dB
SFO1        125.7703643 MHz

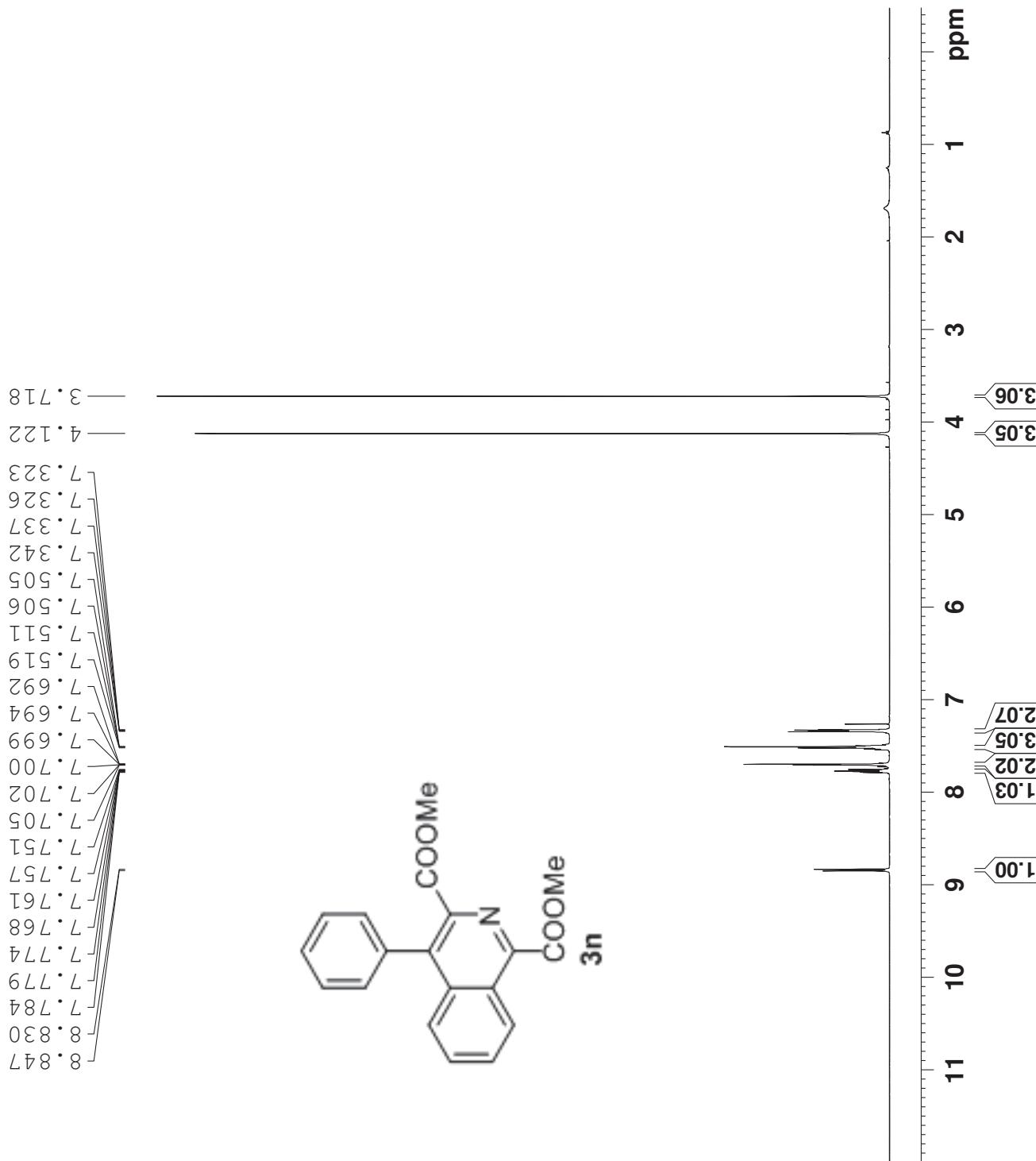
===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPFD2      80.00 usec
PL2           1.00 dB
PL12          16.05 dB
PL13          16.50 dB
SFO2        500.1320005 MHz
SI            327.68 MHz
SF           125.7577718 MHz
WDW          EM
SSB           0
LB            1.00 Hz
GB           0
PC           1.40

```



MH-1-124
PROTON CDCl₃

NAME XE20140527
EXPNO 1
PROCNO 1
Date 20140527
Time 10.48
INSTRUM spect
PROBHD 5 mm FIDXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 228.1
DW 48.400 usec
DE 6.00 usec
TE 296.1 K
D1 1.0000000 sec
TD0 1
===== CHANNEL f1 =====
NUC1 1H
P1 14.14 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300126 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



MH-1-124
C13CPD CDCL3

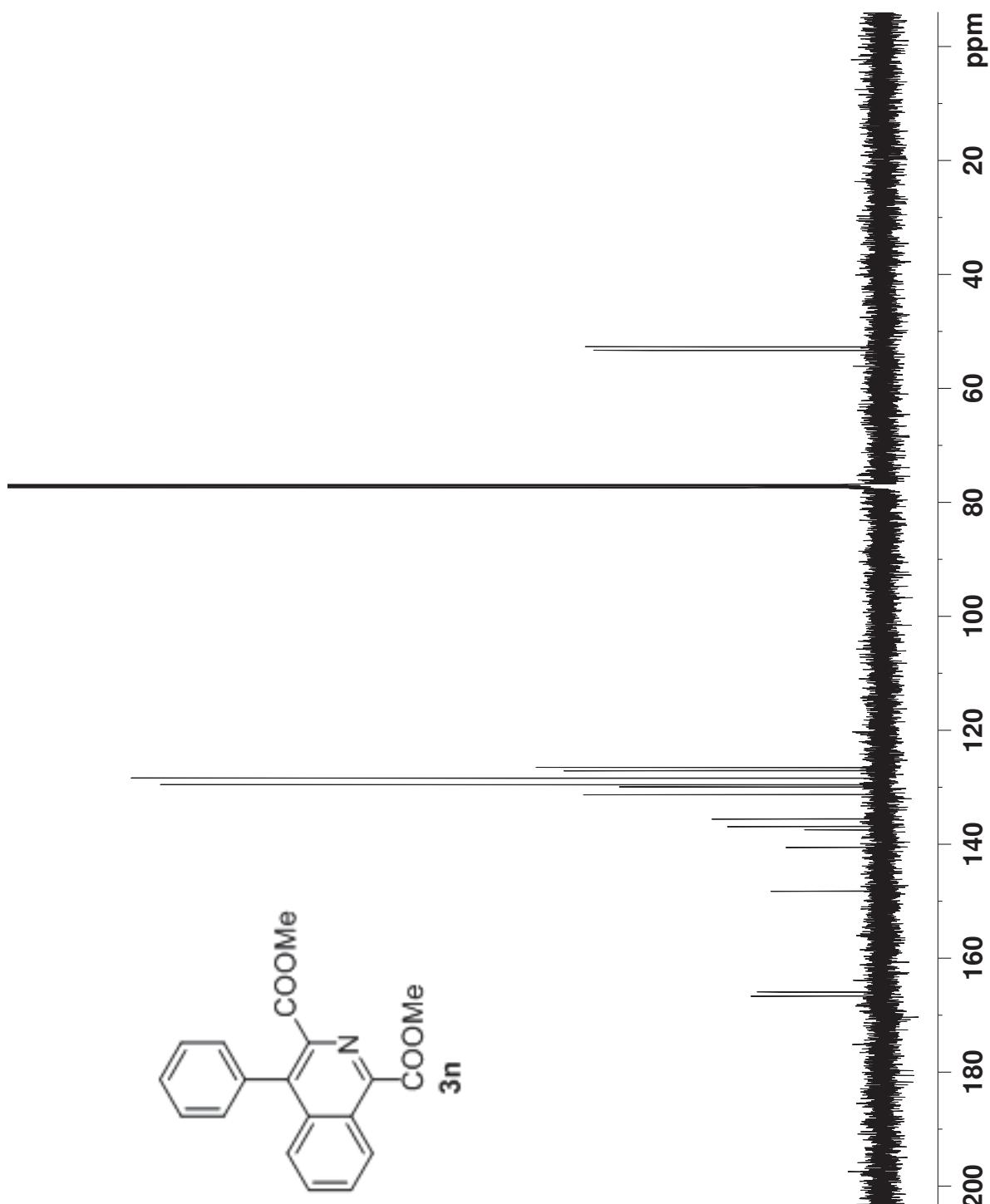
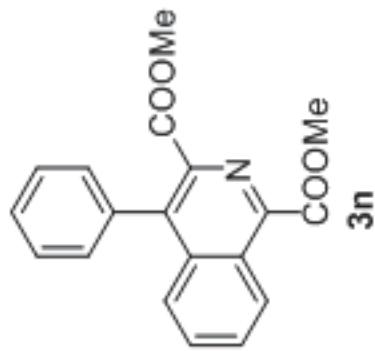
52.62
53.25

126.42
126.95
127.01
128.32
129.45
129.87
131.21
133.48
136.80
137.37
140.48
148.17

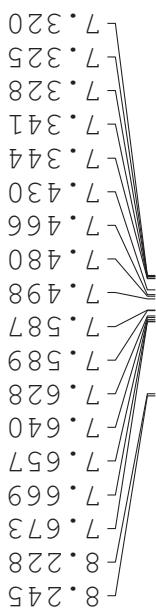
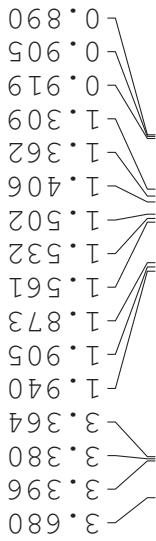
165.86
166.56

NAME	XB20140527
EXPNO	3
PROCNO	1
Date_	20140527
Time	10.58
INSTRUM	spect
PROBHD	5 mm PAXO 19F
PULPROG	zppg30
TD	65536
SOLVENT	CDC13
NS	128
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	161.3
DW	16.650 usec
DE	6.00 usec
TE	297.4 K
D1	2.0000000 sec
d1	0.0300000 sec
DELTA	1.8999999 sec
TD0	1

===== CHANNEL f1 =====	
NUC1	13C
P1	9.50 usec
PL1	-0.50 dB
SFO1	125.7703643 MHz
===== CHANNEL f2 =====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.00 dB
PL12	16.05 dB
PL13	16.50 dB
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



MH-3-106
PROTON CDCl₃

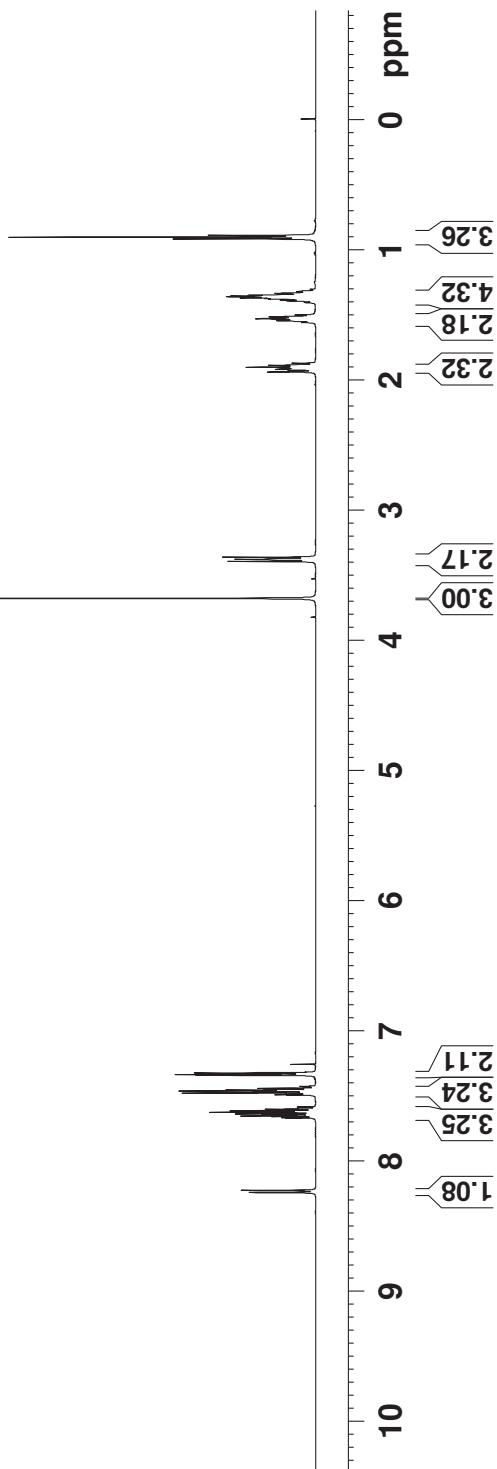


```

=====
NAME      xb20151105
EXPNO     20
PROCNO    1
Date_     20151105
Time      21.36
INSTRUM   spect
PROBHD   5 mm PATXO 19F
PULPROG  zg30
TD       65536
SOLVENT   CDCl3
NS        16
DS        2
SWH      10330.578 Hz
FIDRES   0.157632 Hz
AQ        3.1720407 sec
RG        57
DW        48.400 usec
DE        6.00 usec
TE        294.6 K
D1        1.0000000 sec
TD0      1

===== CHANNEL f1 =====
NUC1      1H
P1        14.14 usec
PL1      1.00 dB
SFO1     500.1330885 MHz
SI        32768
SF      500.1300126 MHz
WDW      no
SSB      0
LB       0.00 Hz
GB       0
PC      1.00

```



MH-3-106
C13CPD CDC13

141.05
136.35
132.08
135.85
130.29
129.83
128.19
127.83
127.27
127.14
125.44
52.35
35.91
31.76
30.11
29.70
22.65
14.13

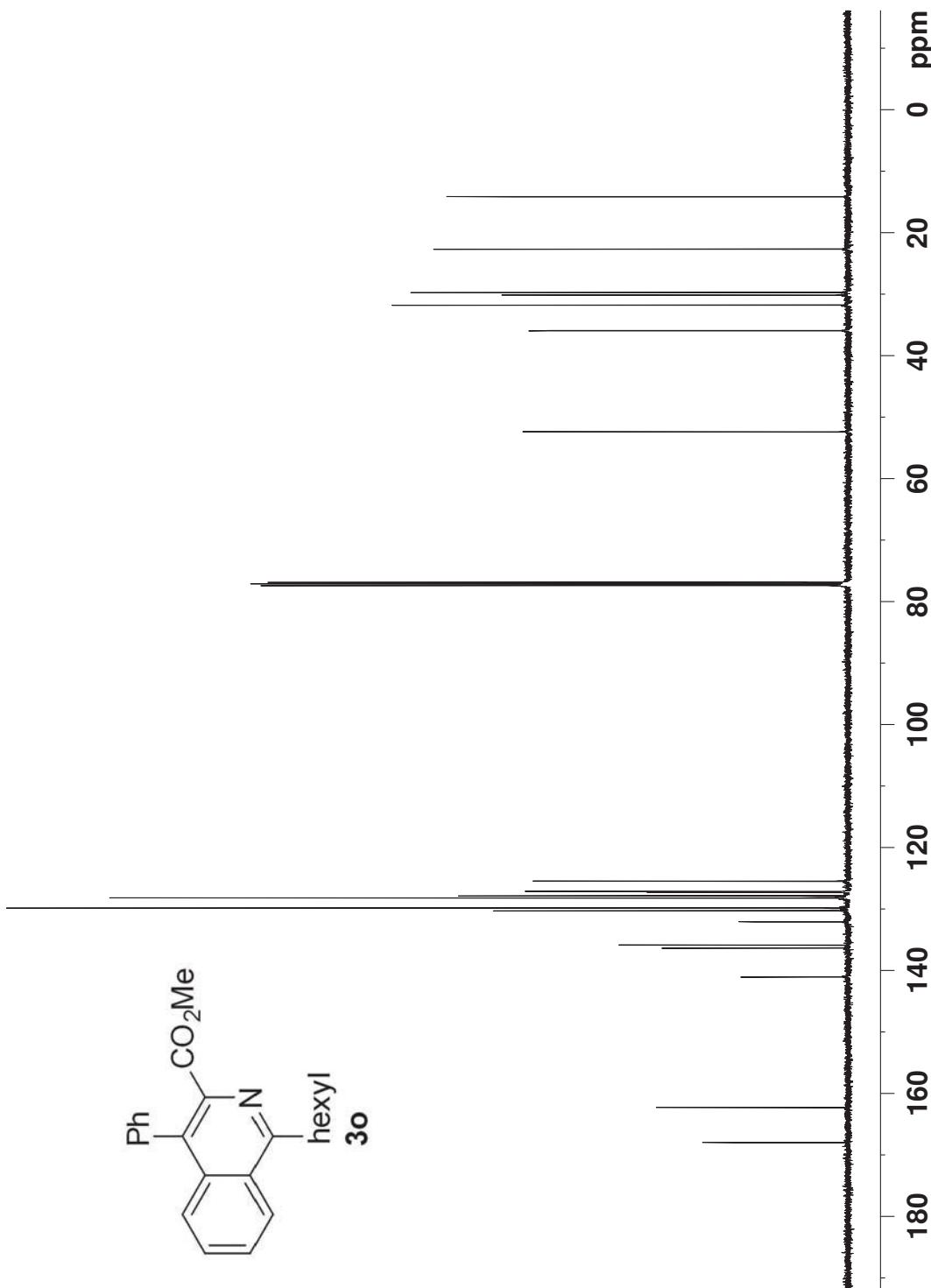
```

NAME          xb20151105
EXPNO        22
PROCNO       1
Date_        20151105
Time         21.42
INSTRUM     spect
PROBHD      5 mm PATXO 19F
PULPROG     zgpg30
TD           65536
SOLVENT      CDCl3
NS            512
DS            4
RG           128
DW           16.650 usec
DE           6.00  usec
TE           295.5 K
D1           2.0000000 sec
d11          0.03000000 sec
d12          1.89999998 sec
DELTA        1.0
TDO          1

===== CHANNEL f1 =====
NUC1         13C
P1           9.50 usec
PL1          -0.50 dB
SFO1        125.7703643 MHz

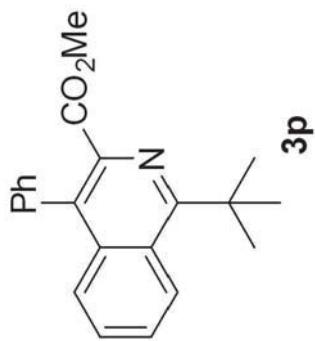
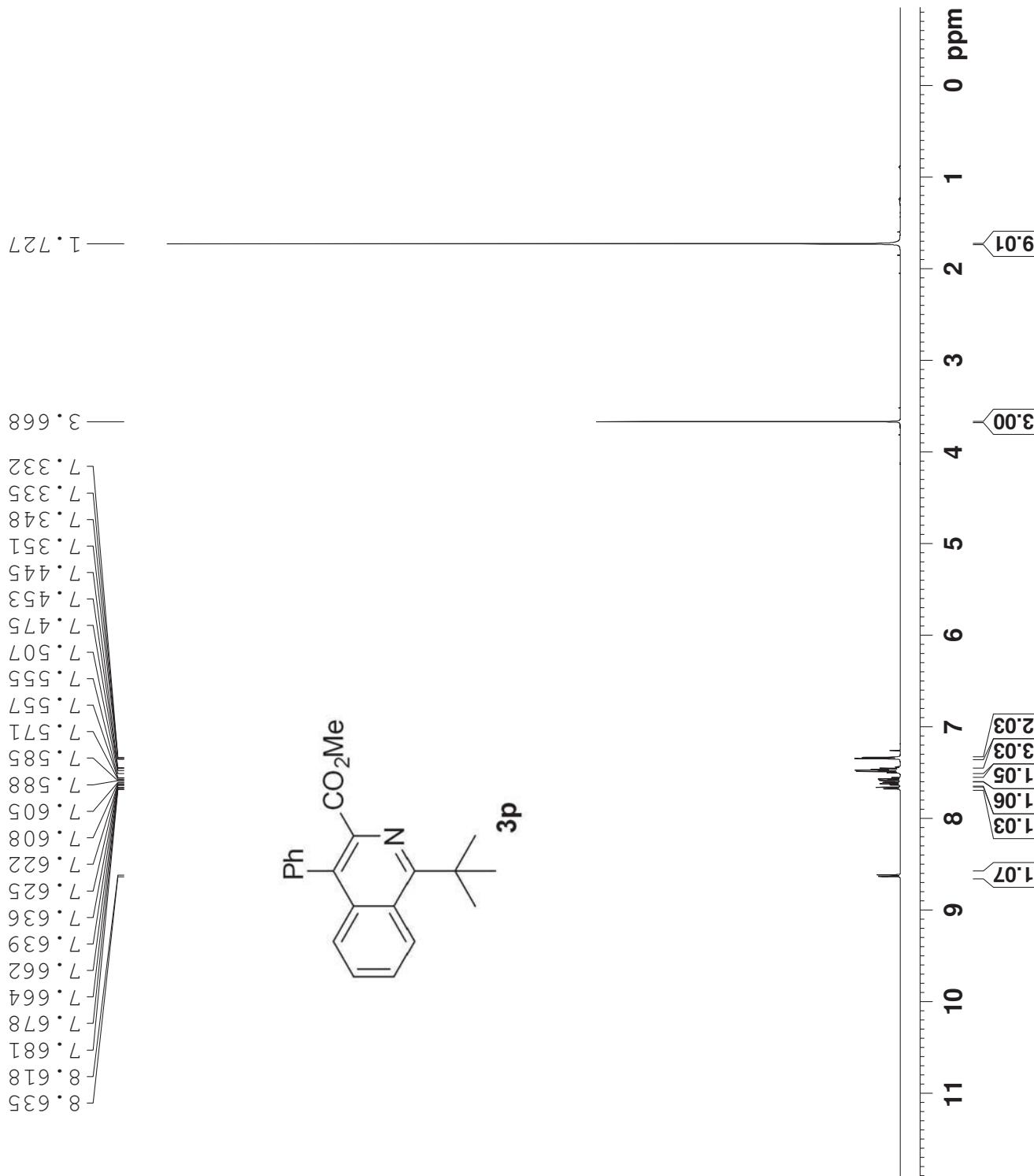
===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2         1H
PCPD2        80.00 usec
PL2          1.00 dB
PL12         16.05 dB
PL13         16.50 dB
SFO2        500.1320005 MHz
SI           32768
SF           125.7577890 MHz
WDW         EM
SSB          0
LB           1.00 Hz
GB           0
PC           1.40

```

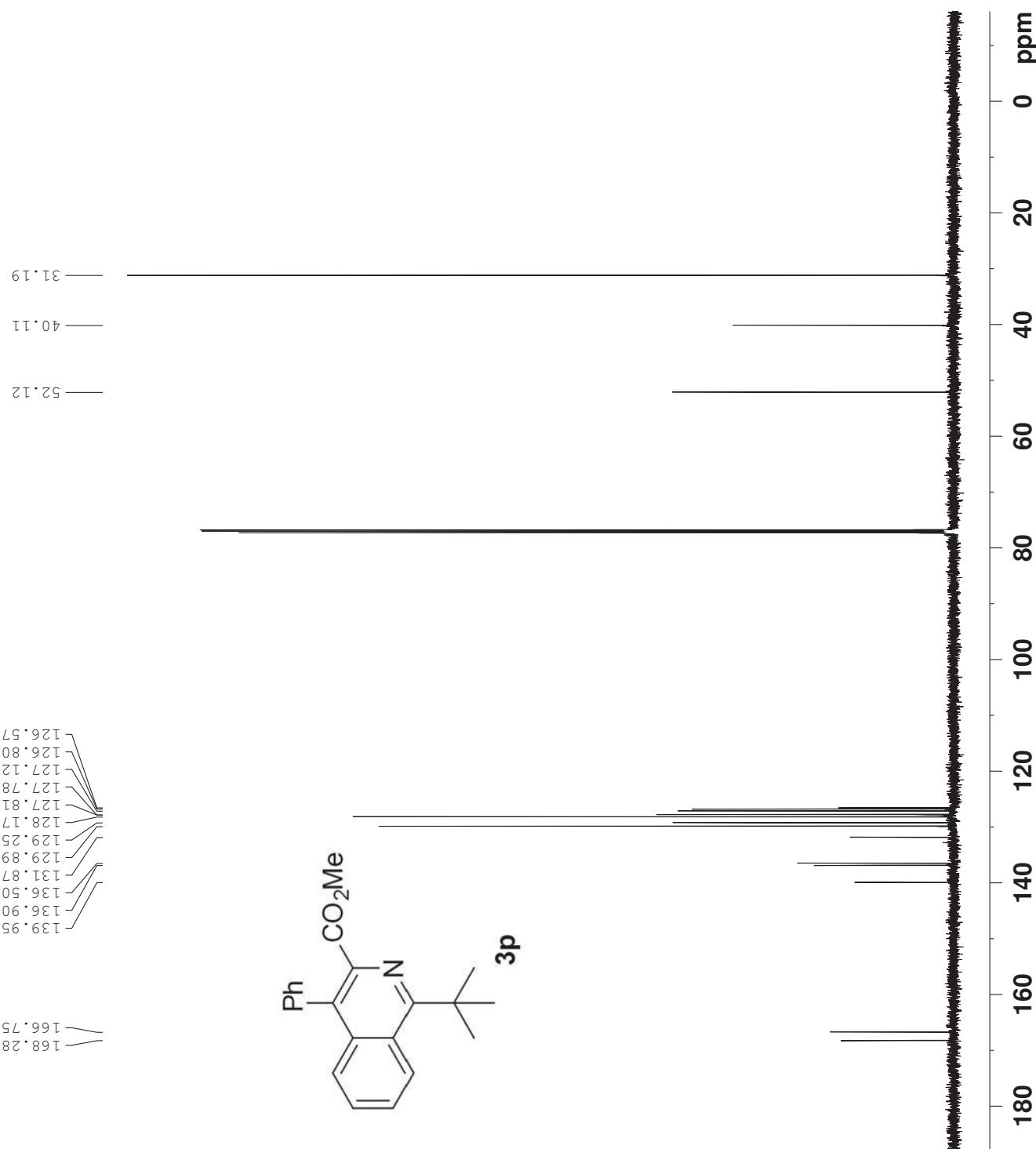


MH-2-14
PROTON CDCL3

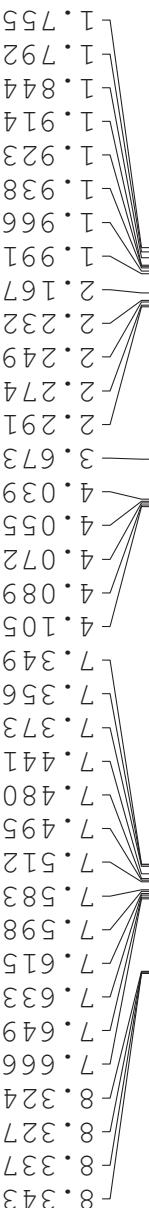
NAME	XB20140706
EXPNO	12
PROCNO	1
Date_	20140706
Time	19.03
INSTRUM	spect
PROBHD	5 mm
PULPROG	PATXO 19F
TD	zg30
SOLVENT	65536
NS	CDC13
DS	16
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	101.6
DW	48.400 usec
DE	6.00 usec
TE	296.5 K
DI	1.0000000 sec
TD0	1
===== CHANNEL f1 =====	
NUC1	1H
P1	14.14 usec
PPL1	1.00 dB
SFO1	500.11330885 MHz
SI	32768
SF	500.11300127 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



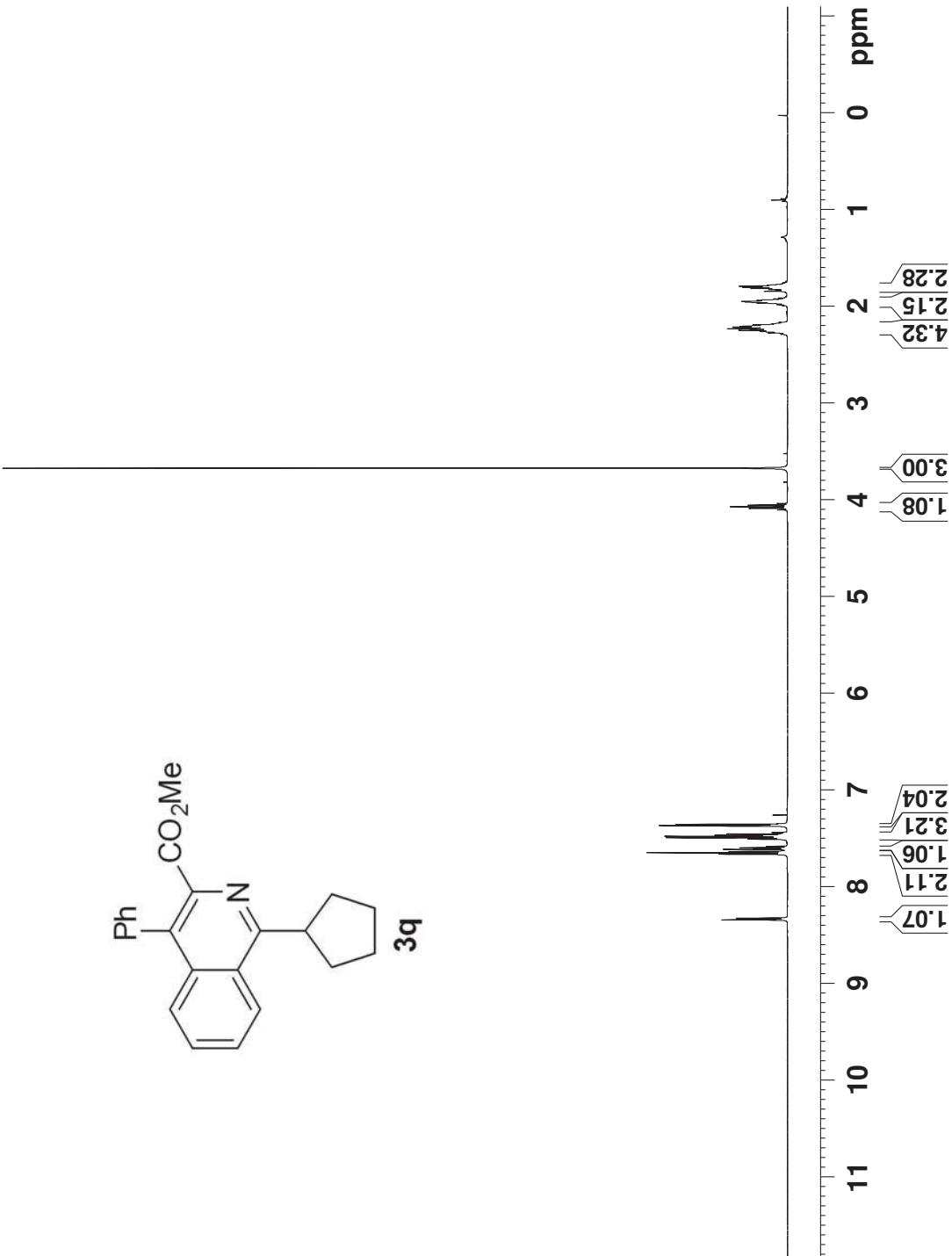
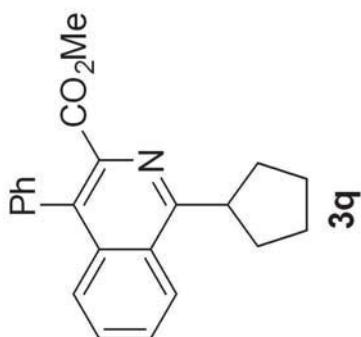
MH-2-14
C13CPD CDC13



MH-3-111
PROTON CDC13



NAME	xb20151105	CHANNEL	f1	=====
EXPNO	23			
PROCCNO	1			
Date_	20151105			
Time_	22:13			
INSTRUM	spect			
PROBHD	5 mm	PATXO	19F	
PULPROG		zg30		
TD	65536			
SOLVENT	CDC13			
NS	16			
DS	2			
SWH	10330 .578	Hz		
FIDRES	0 .157632	Hz		
AQ	3.1720407	sec		
RG	40 .3			
DW	48 .400	use		
DE	6 .00	use		
TE	295 .6	K		
D1	1 .00000000	sec		
TDO	1			
=====				
NUC1	1H			
P1	14 .14	use		
PL1	1 .00	dB		
SFO1	500 .1330885	MHz		
SI	32768			
SF	500 .1300126	MHz		
WDW	no			
SSB	0			
LB	0 .00	Hz		
GB	0			
PC	1 .00			



MH-3-111
C13CPD CDCl₃

```

NAME          XB20151106
EXPNO         3
PROCNO        1
Date_         20151106
Time          9.28
INSTRUM      spect
PROBHD       5 mm PATXO 19F
PULPROG      zgppg30
TD           65536
SOLVENT       CDCl3
NS            512
DS             4
SWH          30030.029 Hz
FIDRES       0.458222 Hz
AQ            1.0912410 sec
RG            406.4
DW           16.650 usec
DE            6.00 usec
TE            296.2 K
D1           2.00000000 sec
d1           0.03000000 sec
DELTA        1.89999998 sec
TD0            1

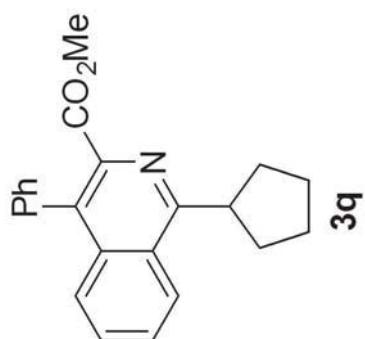
===== CHANNEL f1 =====
NUC1          13C
P1            9.50 usec
PL1           -0.50 dB
SFO1        125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           1.00 dB
PL12          16.05 dB
PL13          16.50 dB
SFO2        500.1320005 MHz
SI            32768
SF           125.7577890 MHz
WDW          EM
SSB           0
LB           1.00 Hz
GB           0
PC           1.40

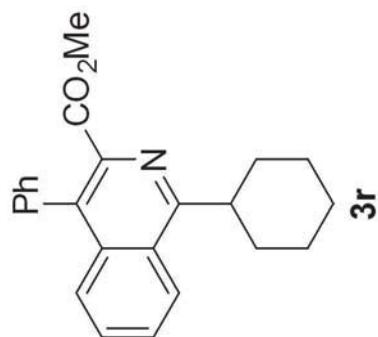
```

— 26.04
— 32.58
— 43.39
— 52.14

— 125.25
— 127.04
— 127.47
— 127.79
— 127.90
— 127.90
— 128.20
— 129.93
— 130.01
— 131.37
— 135.79
— 136.51
— 141.16
— 164.13
— 168.36



MH-3-117
PROTON CDC13

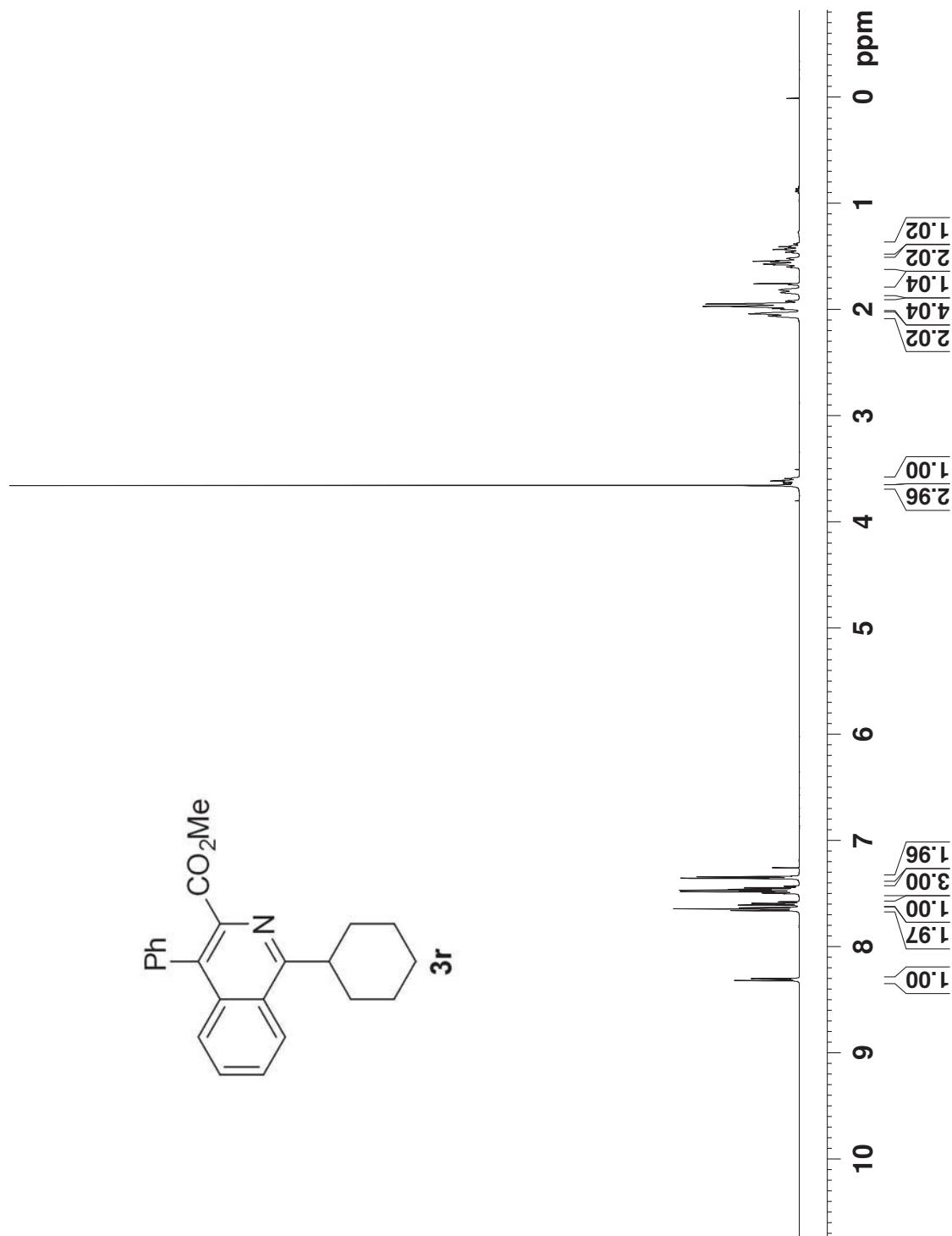


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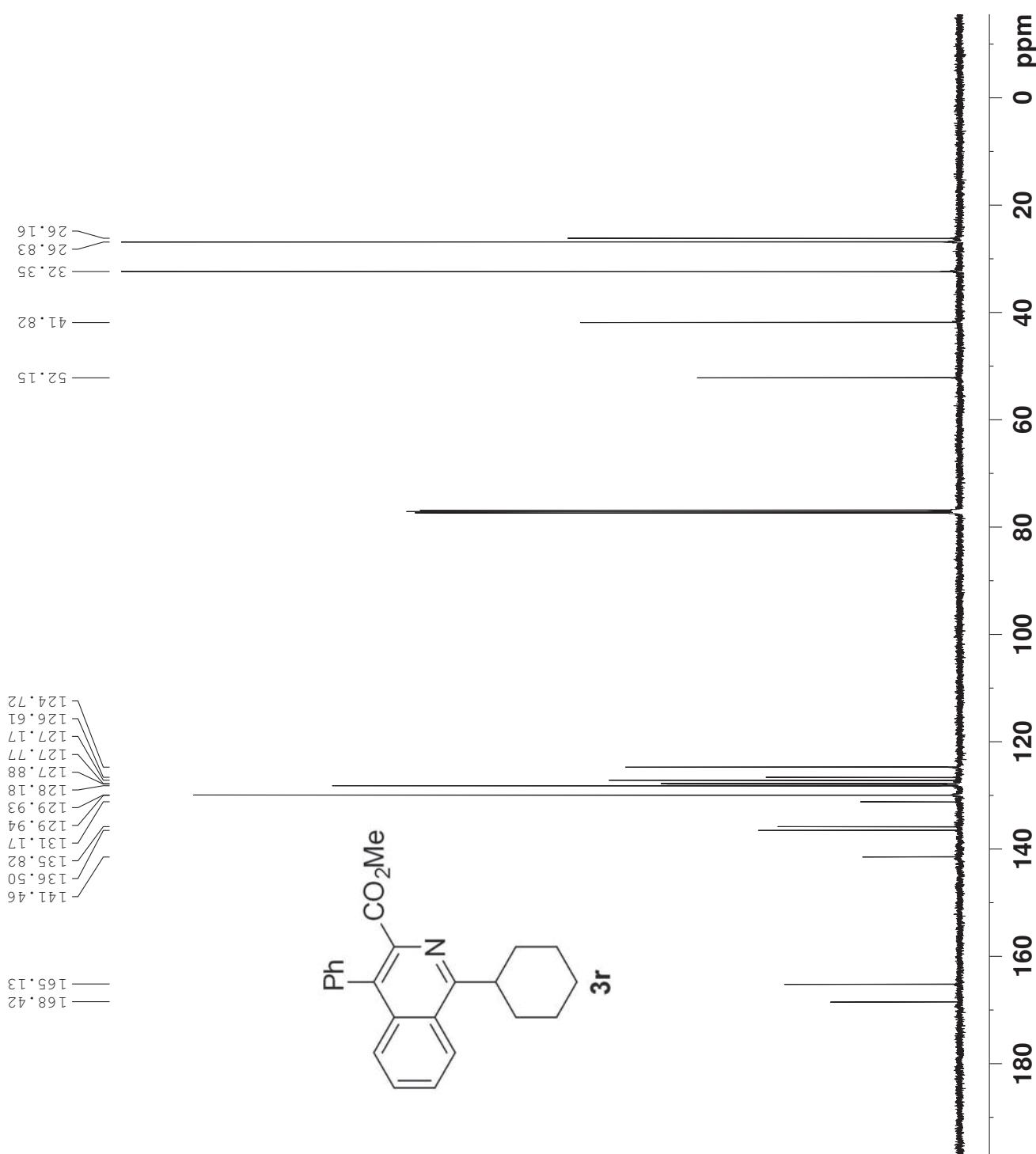
NAME XB20151106
EXPNO 4
PROCN0 1
Date_ 20151106
Time_ 9.39
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG Z930
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 90.5
DW 48.400 usec
DE 6.00 usec
TE 294.9 K
TE1 1.00000000 sec
DD1 1
TDD0

===== CHANNEL f1 =====
NUC1 1H
P1 14.14 usec
PPL1 1.00 dB
SF01 500.1330885 MHz
SI 32768
SF 500.1300129 MHz
WDW no
SSSB 0
LB 0.00 Hz
GB 0
PC 1.00

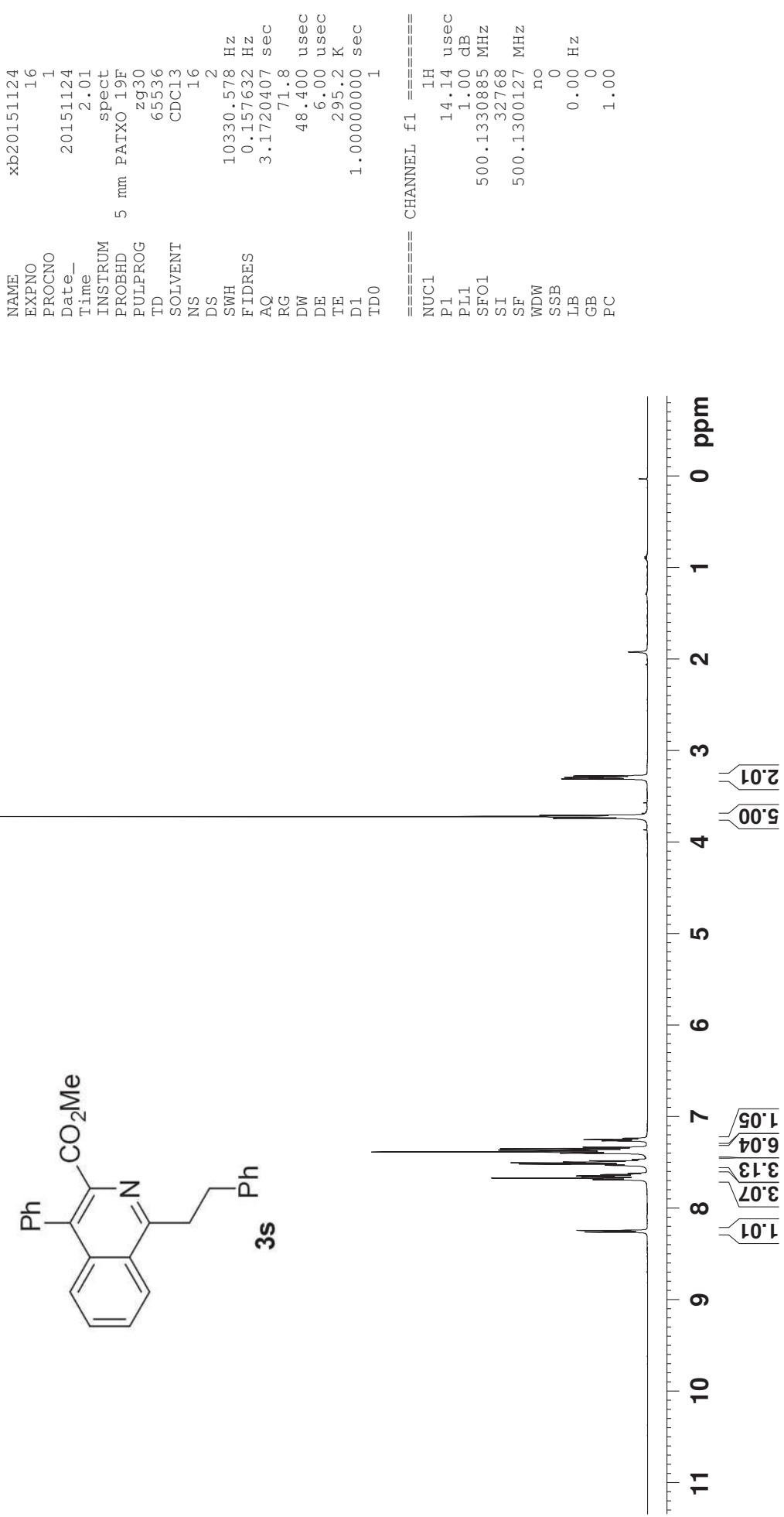
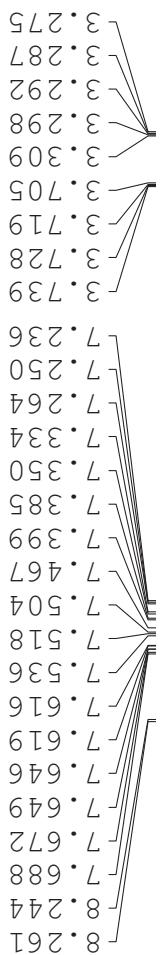
```



MH-3-117
C13CPD CDCl₃



MH-3-122-A
PROTON CDCl₃

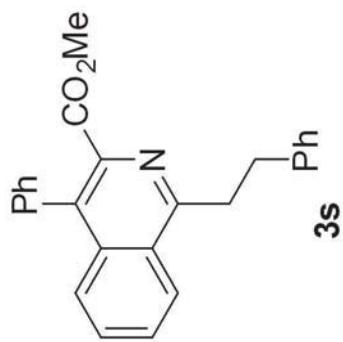


MH-3-122-A
C13CPD CDCL₃

35.41
37.31

52.39

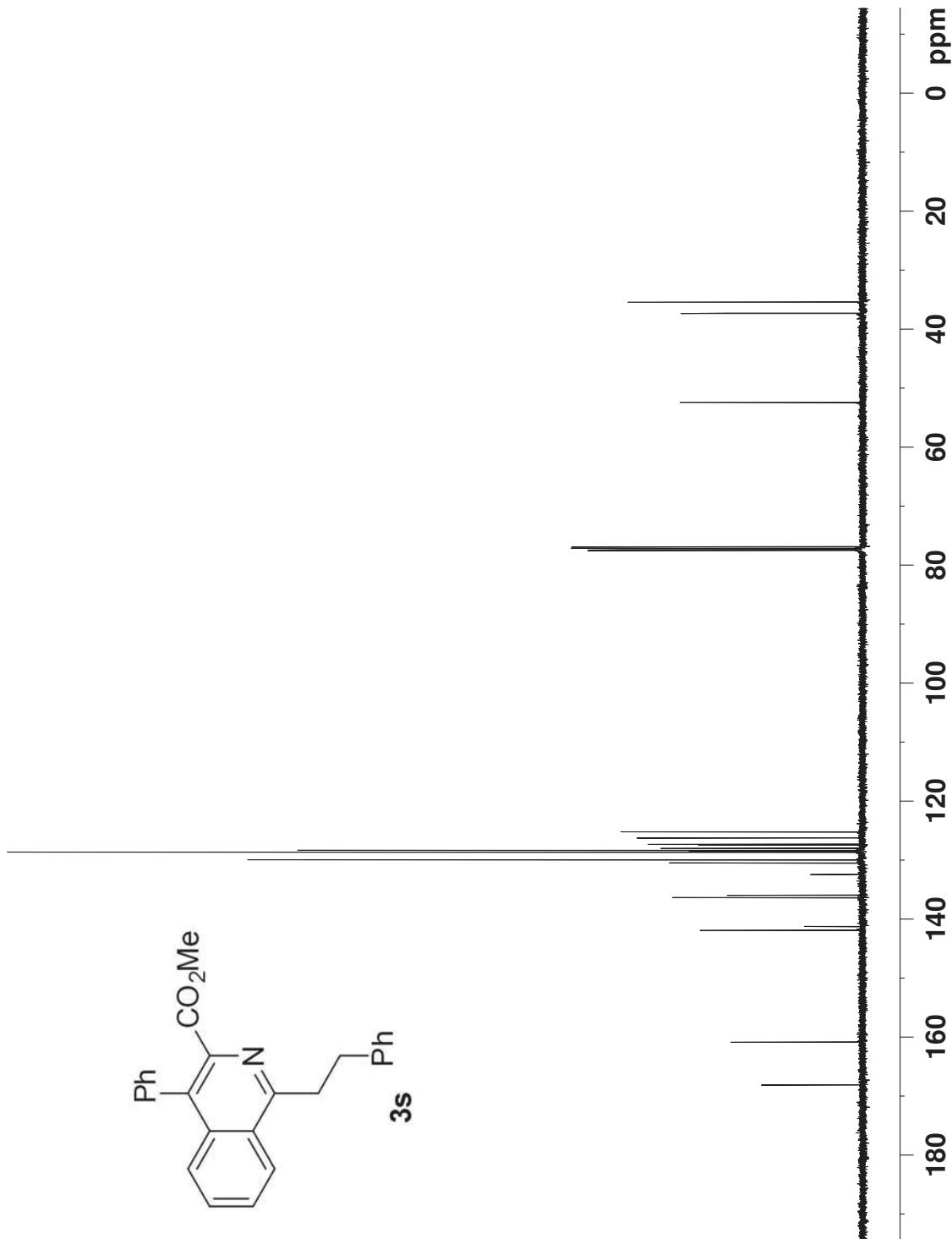
160.78
168.03
141.17
141.83
136.28
135.86
132.37
130.42
129.87
128.56
128.55
128.37
128.26
127.94
127.33
126.17
125.14



```

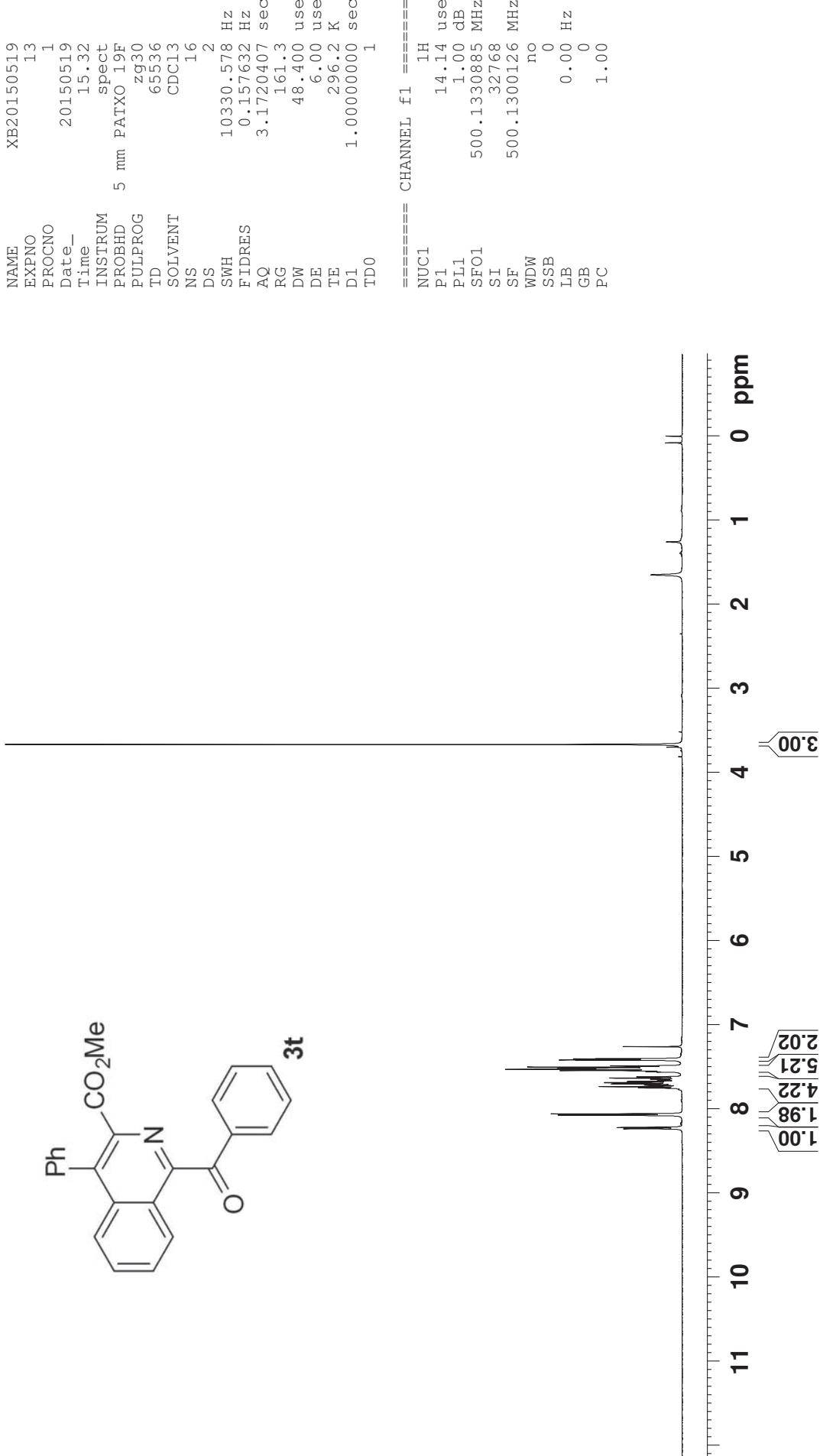
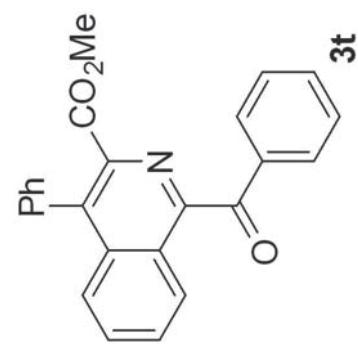
NAME          xb20151124
EXPNO        18
PROCNO       1
Date_        20151124
Time         2.31
INSTRUM     spect
PROBHD      5 mm PATXO 19F
PULPROG     zgppg30
TD           65536
SOLVENT      CDCl3
NS            512
DS            4
SWH          30030.029 Hz
FIDRES      0.458222 Hz
AQ           1.0912410 sec
RG           114
DW           16.650 usec
DE           6.00 usec
TE           296.6 K
D1           2.0000000 sec
D1           0.03000000 sec
D1           1.89999998 sec
DELTA        TDO
TDDF
        ===== CHANNEL f1 =====
NUC1          13C
P1            9.50 usec
PL1          -0.50 dB
SFO1        125.7703643 MHz
        ===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           1.00 dB
PL12          16.05 dB
PL13          16.50 dB
SFO2        500.1320005 MHz
SI            32768
SF           125.7577890 MHz
WDW          no
SSB          0.00 Hz
LB           0
GB           0
PC           1.40

```



MH-3-54-DOWN
PROTON CDCl₃

— 3.66 —
8.237 8.219 8.075 8.059 8.061 7.950 7.950 7.690 7.687 7.620 7.557 7.530 7.488 7.421 7.418 7.406



167.14
 155.94
 140.56
 136.44
 135.52
 135.29
 134.01
 131.29
 131.05
 129.65
 129.33
 128.57
 128.38
 128.36
 126.91
 126.61
 126.28

MH-3-54-DOWN
C13CPD CDC13

— 52.52 —

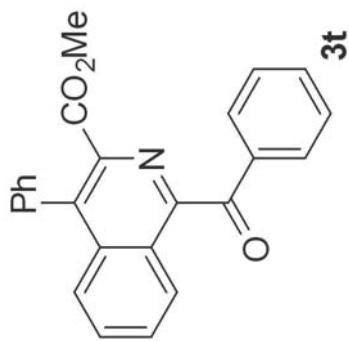
```

NAME          XB20150519
EXPNO         14
PROCNO        1
Date_         20150519
Time          15.47
INSTRUM      spect
PROBHD       5 mm PMTXO 19F
PULPROG      zpgpg30
TD           65536
SOLVENT      CDCl3
NS            256
DS            4
SWH          30030.029 Hz
FIDRES       0.458222 Hz
AQ            1.0912410 sec
RG            181
DW            16.650 usec
DE            6.00   usec
TE            297.1  K
D1           2.0000000 sec
d11          0.03000000 sec
DELTA         1.89999998 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            9.50  usec
PL1           -0.50  dB
SFO1         125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00  usec
PL2           1.00   dB
PL12          16.05  dB
PL13          16.50  dB
SFO2         500.1320005 MHz
SI            32768
SF           125.7577890 MHz
WDW
SSB
LB           0.00   Hz
GB           0
PC           1.40

```



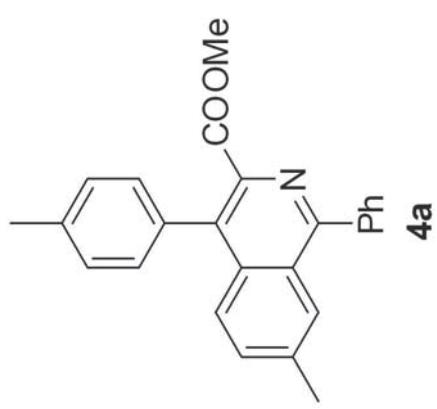
— 193.92 —

MH-2-39
PROTON CDCl₃

2.484
2.474

3.724

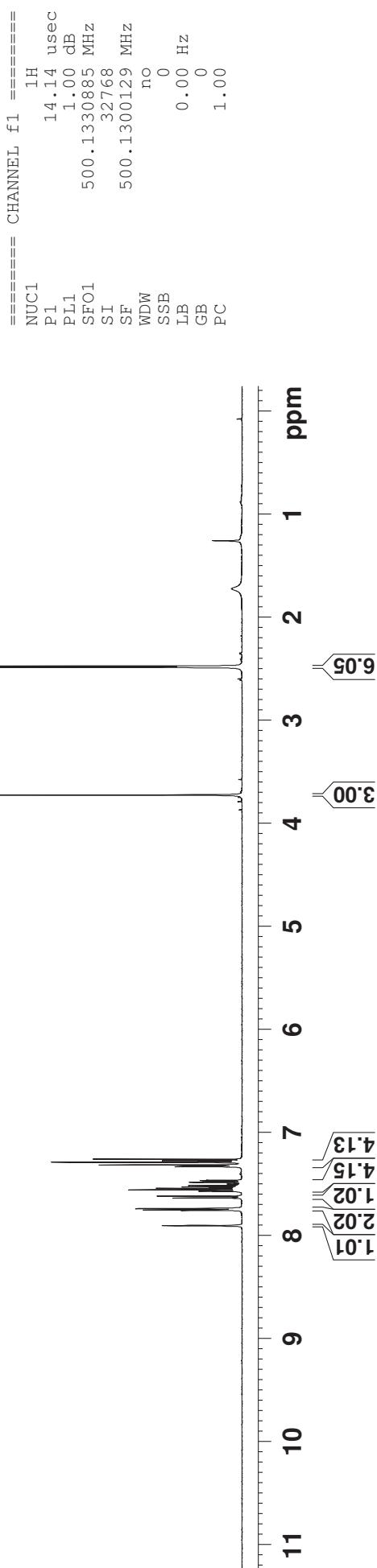
7.905
7.756
7.753
7.739
7.737
7.636
7.619
7.574
7.515
7.486
7.482
7.468
7.465
7.332
7.316
7.290
7.274



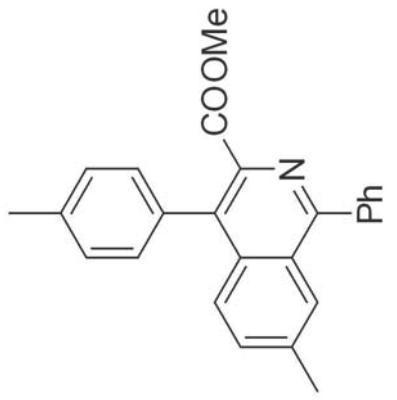
```

NAME          XB20141023
EXPNO         9
PROCNO        1
Date_        20141023
Time       12.12
INSTRUM      spect
PROBHD      5 mm PATXO 19F
PULPROG     zg30
TD        65536
SOLVENT      CDCl3
NS           16
DS            2
SWH        10330.578 Hz
FIDRES     0.157632 Hz
AQ        3.1720407 sec
RG          362
DW        48.400 usec
DE        6.000 usec
TE        296.7 K
D1    1.00000000 sec
TDO        1

```



167.57
 159.24
 140.28
 138.78
 138.69
 137.68
 134.98
 133.11
 133.06
 132.84
 130.21
 129.67
 129.03
 128.89
 128.45
 127.44
 126.73
 126.57



52.47
 21.95
 21.43

MH-2-39
C13CPD CDC13

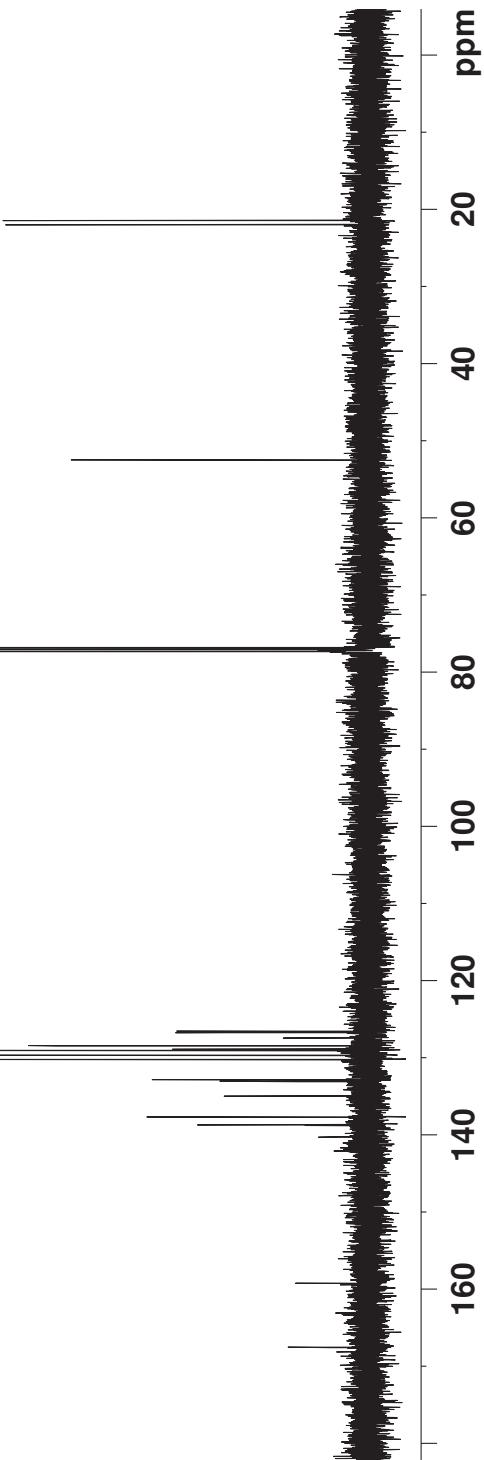
```

NAME          XB20140706
EXPNO         3
PROCNO        1
Date_         20140706
Time_         17.24
INSTRUM      spect
PROBHD       5 mm PATXO 19F
PULPROG      zgp930
TD           65536
SOLVENT       CDC13
NS            256
DS            4
SWH          30030.029 Hz
FIDRES       0.458222 Hz
AQ            1.0912410 sec
RG            128
DW            16.650 usec
DE            6.00 usec
TE            297.8 K
D1           2.0000000 sec
d11          0.03000000 sec
DELTA         1.89999998 sec
TDO           1

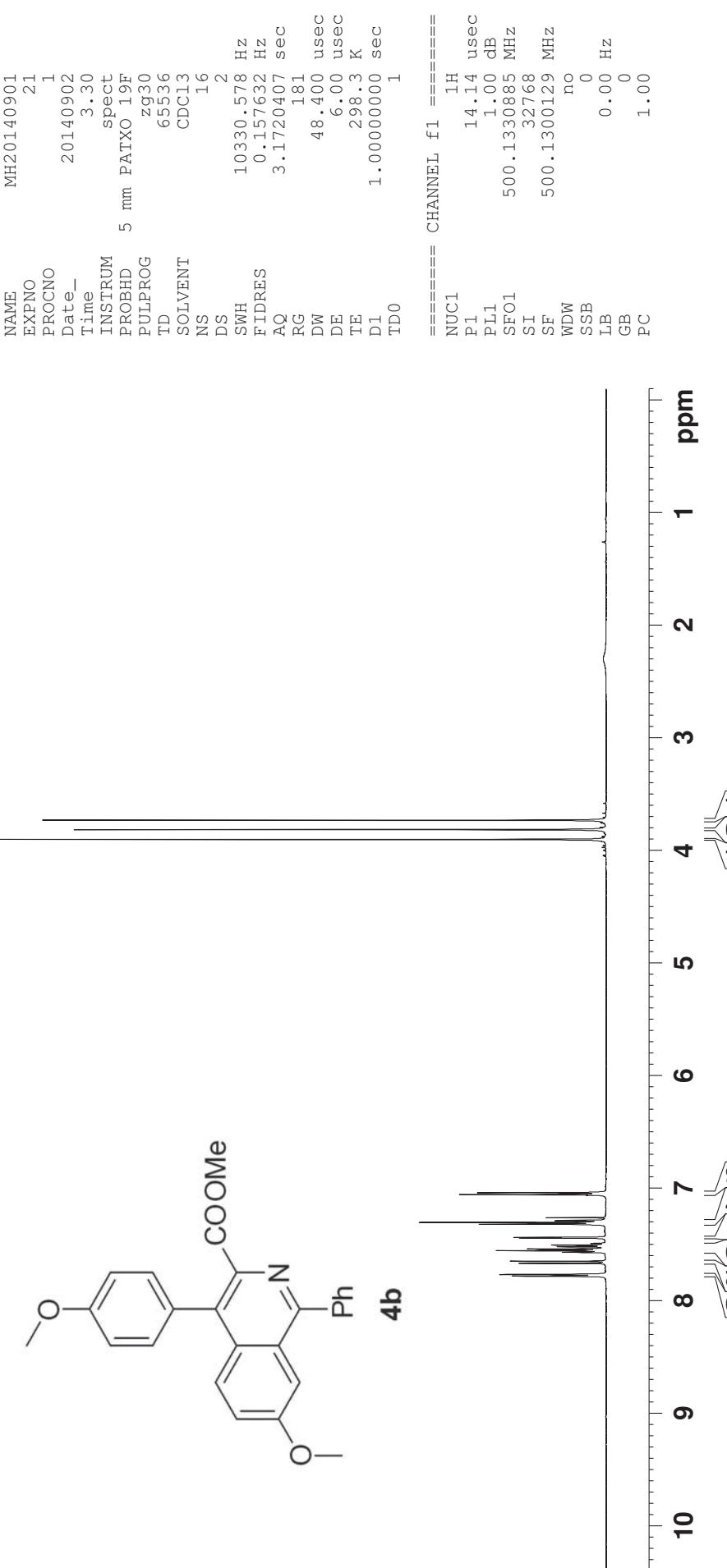
===== CHANNEL f1 =====
NUC1          13C
P1            9.50 usec
PL1           -0.50 dB
SF01         125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           1.00 dB
PL12          16.05 dB
PL13          16.50 dB
SFO2         500.1320005 MHz
SI             32768
SF           125.7577890 MHz
WDW           no
SSB           0
LB            0.00 Hz
GB           0
PC           1.40

```



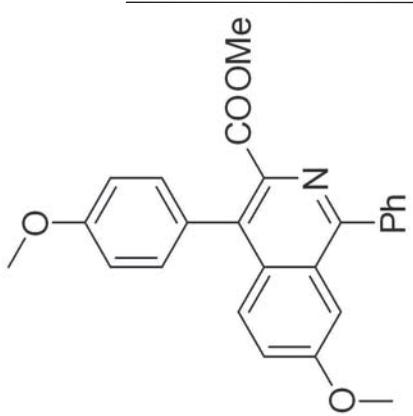
MH-2-42
PROTON CDCl₃



MH-2-42
C13CPD CDC13

167.83
139.79
139.15
133.00
132.34
131.04
130.06
128.97
128.89
128.67
128.64
128.37
123.10
113.88
105.79

159.47
158.36
139.15
133.00
132.34
131.04
130.06
128.97
128.89
128.67
128.64
128.37
123.10
113.88
105.79



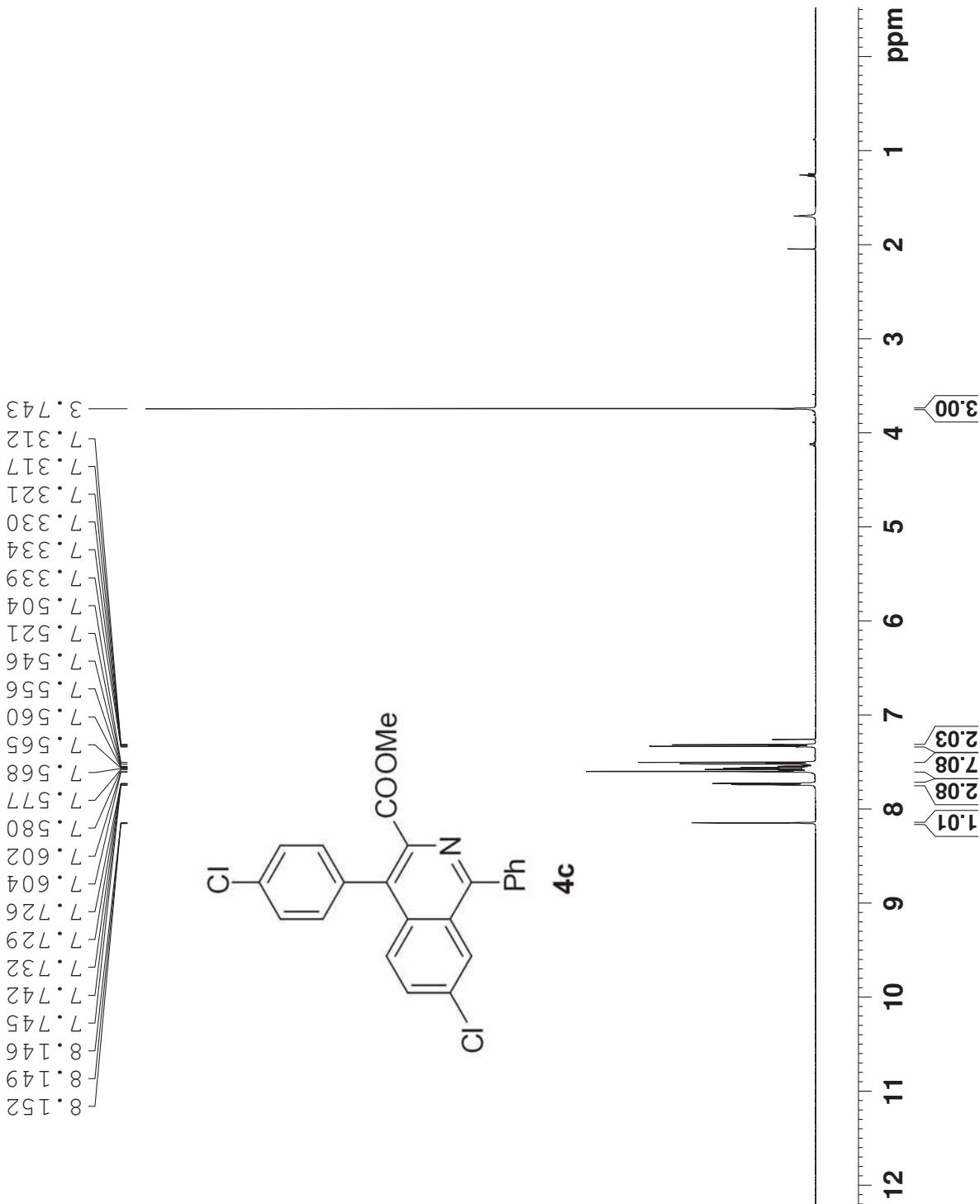
4b

```

=====
NAME          MH20140901
EXPNO        23
PROCNO       1
Date_        20140902
Time         3.48
INSTRUM      spect
PROBHD      5 mm PAXCO 19F
PULPROG     ZGPGR30
TD           65536
SOLVENT      CDCl3
NS            256
DS            4
SWH          30030.029 Hz
FIDRES      0.458222 Hz
AQ           1.0912410 sec
RG           912.3
DW           16.650 usec
DE           6.00 usec
TE           299.5 K
D1           2.0000000 sec
Q1           0.03000000 sec
DELTA        1.8999998 sec
TDO          1
        ===== CHANNEL f1 =====
NUC1         13C
P1           9.50 usec
PL1          125.7703643 MHz
SFO1        125.7703643 MHz
        ===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2         1H
BCPD2        80.00 usec
PL2          1.00 dB
PL12         16.05 dB
PL13         16.50 dB
SFO2        500.1320005 MHz
SI           32768
SF           125.7577755 MHz
WDW         EM
SSB          0
LB           1.00 Hz
GB           0
PC           1.40
  
```



MH-2-16
PROTON CDCl₃



MH-2-16
C13CPD CDCl₃

167.08
159.75
141.44
138.13
134.83
134.78
134.46
134.06
131.73
131.55
131.11
130.08
129.35
128.76
128.71
128.29
127.89
126.64

```

NAME          XB20140612
EXPNO         9
PROCNO        1
Date_         2014/06/12
Time          12:05
INSTRUM       spect
PROBHD        5 mm PAXCO 19F
PULPROG       zgpg30
TD            65536
SOLVENT        CDCl3
NS             256
DS              4
SWH           30030.029 Hz
FIDRES       0.45822 Hz
AQ            1.0912410 sec
RG            114
DW            16.150 usec
DE            6.00 usec
TE            298.1 K
D1           2.0000000 sec
d11          0.13000000 sec
DELT1A        1.89999998 sec
TD0           1

```

```

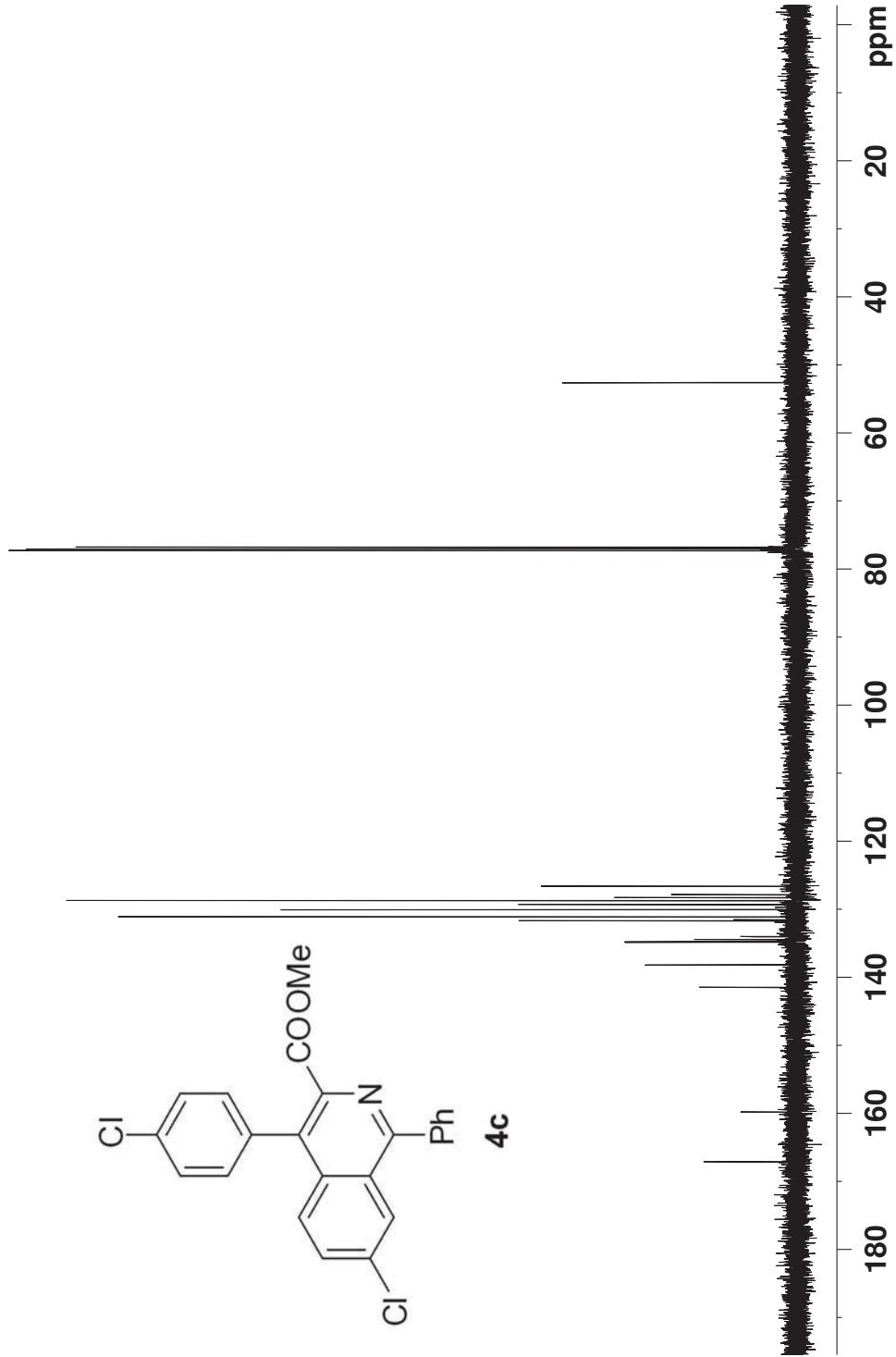
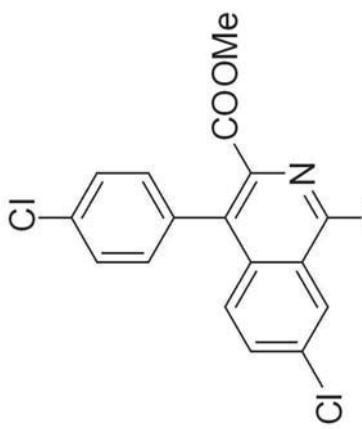
===== CHANNEL f1 =====
NUC1          13C
P1            9.50 usec
PL1           -0.50 GB
SFO1        125.7703643 MHz

```

```

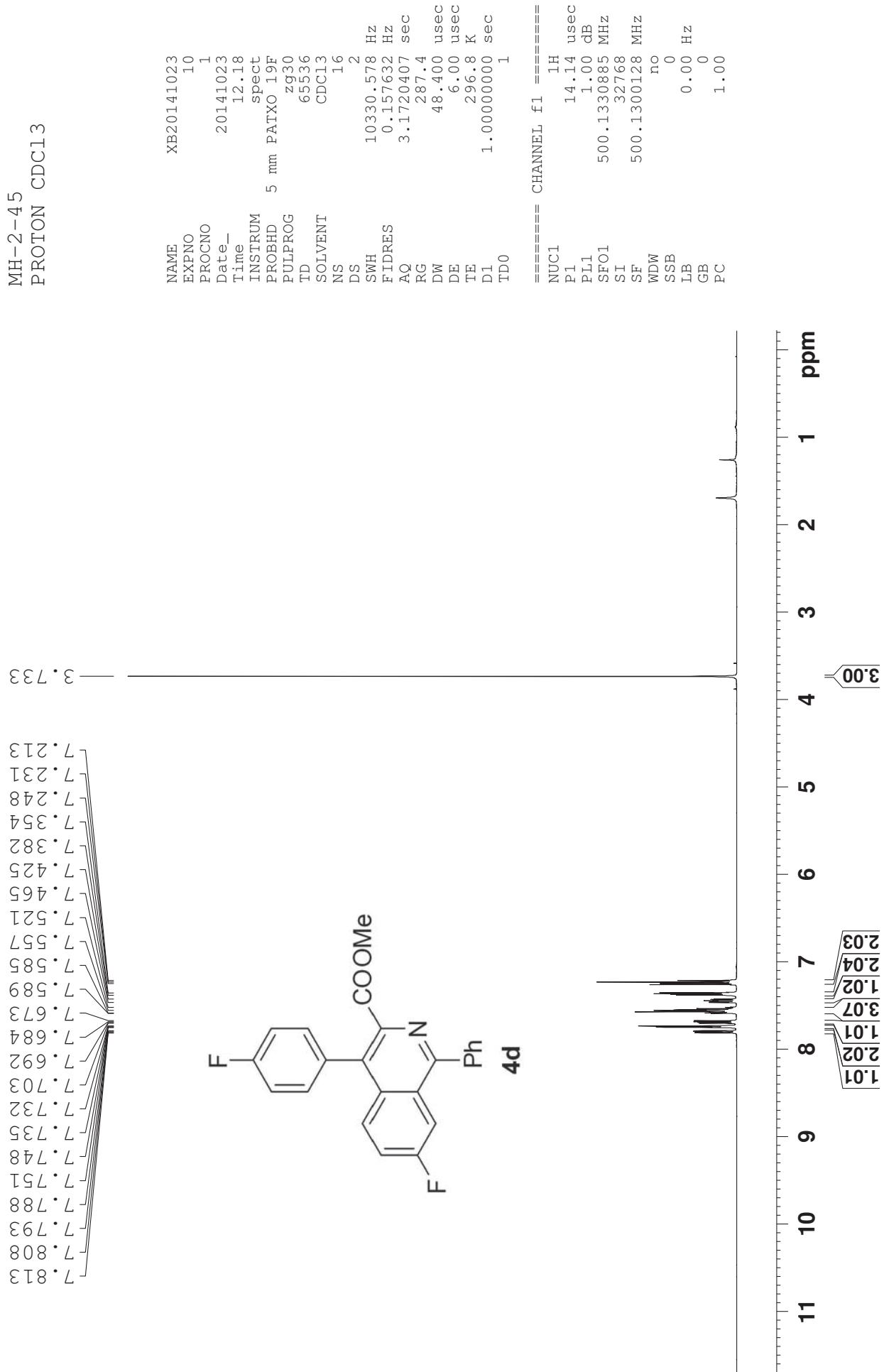
===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           1.00 dB
PL12          16.05 dB
PL13          16.50 dB
SFO2        500.1320005 MHz
SI            32768
SF           125.7577890 MHz
WDW           no
SSB            0
LB             0.00 Hz
GB             0
PC            1.40

```



— 52.63 —

MH-2-45
PROTON CDCl₃



MH-2-45
C13CPD CDCl₃

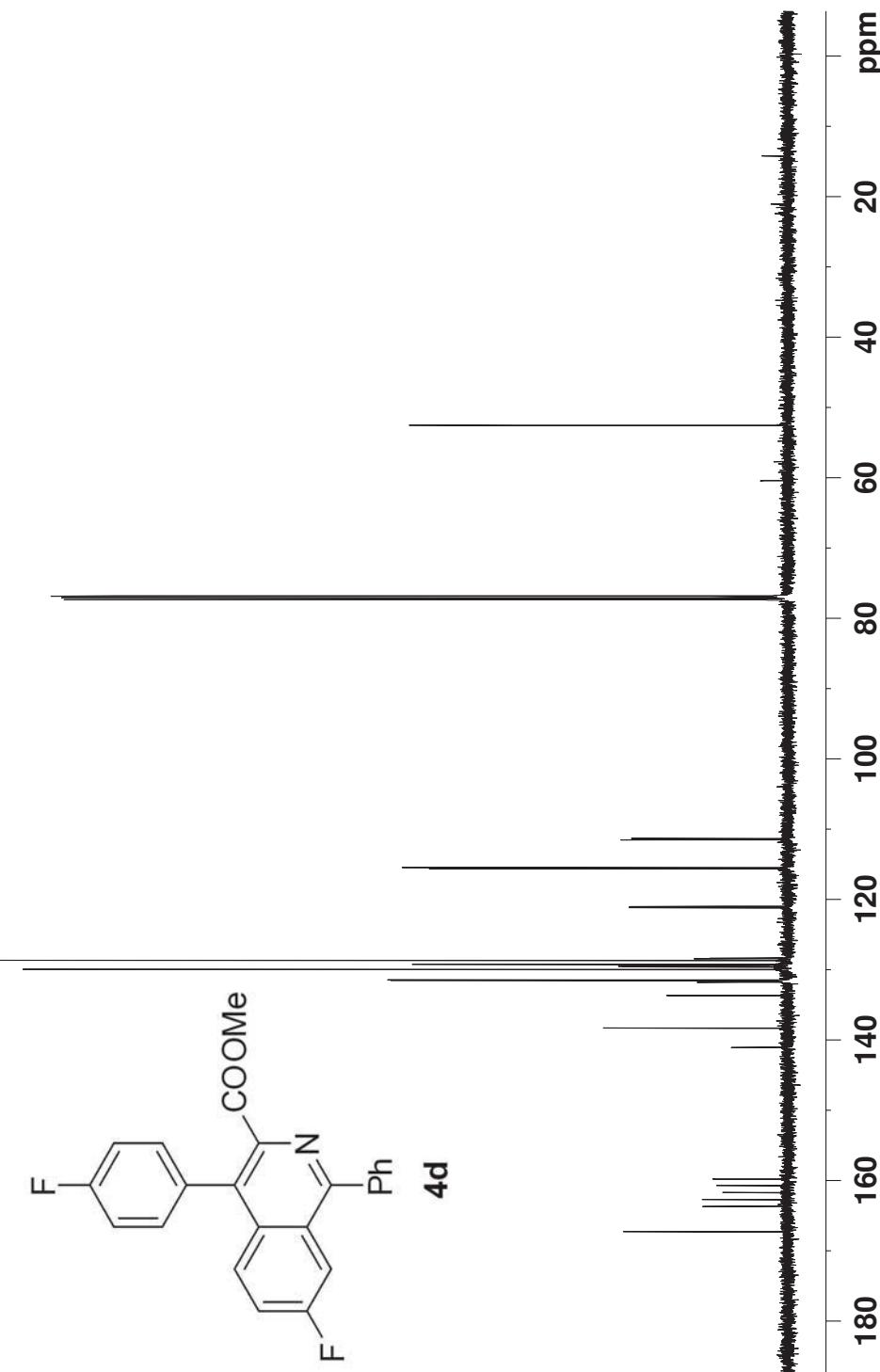
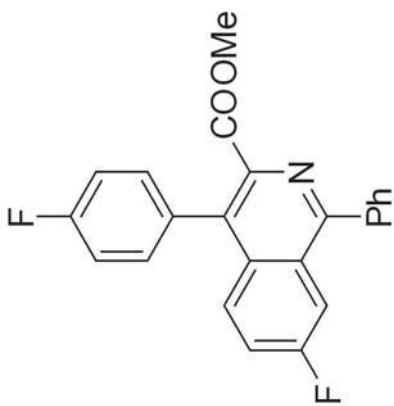
167.31
163.69
162.72
161.72
160.72
159.82
159.78
141.09
138.33
133.70
131.79
131.66
131.63
131.53
131.47
129.61
129.54
129.26
128.67
128.45
128.38
121.20
121.00
120.65
115.48
111.34
111.52

```

NAME          XB20140705
EXPNO         10
PROCNO        1
Date—       2014/07/05
Time         21.23
INSTRUM      spect
PROBHD      5 mm PATXO 19F
PULPROG     zgpg30
TD           65536
SOLVENT      CDCl3
NS            256
DS            4
SWH          30030.029 Hz
FIDRES      0.458222 Hz
AQ           1.0912410 sec
RG           322.5
DW           16.650 usec
DE           6.00
TE           297.6 K
D1           2.0000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TDO           1

===== CHANNEL f1 =====
NUC1          13C
P1            9.50 usec
PL1           -0.50 dB
SFO1        125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           1.00 dB
PL12          16.05 dB
PL13          16.50 dB
SFO2        500.1320005 MHz
SI             32768
SF           125.7577890 MHz
WDW          EM
SSB           0
LB            1.00 Hz
GB           0
PC           1.40
```



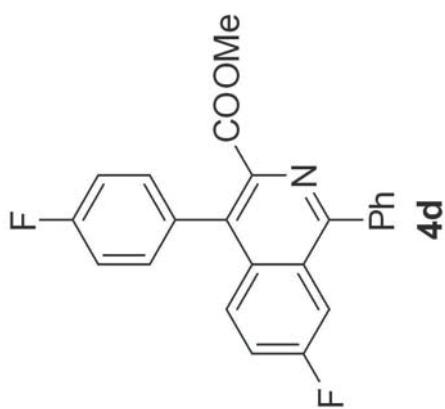
MH-2-45
19Fdeft CDCl₃ D:\deng 44

NAME	XB20140705
EXPNO	9
PROCNO	1
Date	20140705
Time	21.08
INSTRUM	spect
PROBHD	5 mm PATXO 19F
PULPROG	Zg
TD	131072
SOLVENT	CDCl ₃
NS	1
DS	4
SWH	100000.000 Hz
FIDRES	0.762939 Hz
AQ	0.6554150 sec
RG	362
DW	5.000 usec
DE	6.00 usec
TE	296.4 K
D1	1.0000000 sec
TD0	1

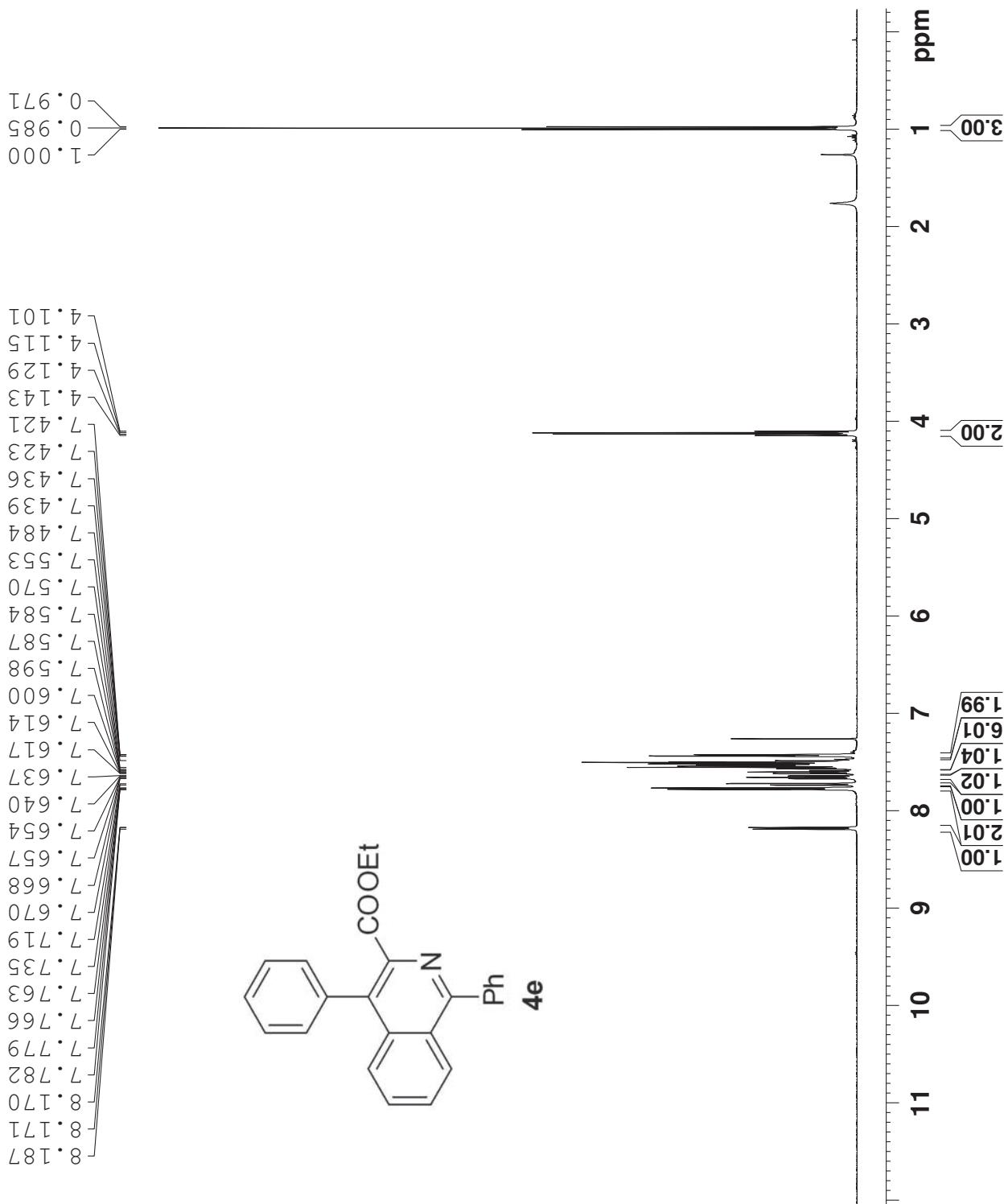
===== CHANNEL f1 =====

NUC1	19F
P1	19.30 usec
PL1	4.00 dB
SFO1	470.5453180 MHz
SI	65536
SF	470.5923770 MHz
WDW	no
SSB	0
LB	0.00 Hz

-113.384
-113.373
-113.366
-113.354
-113.344
-113.337
-113.324
-108.820
-108.806
-108.799
-108.790
-108.782
-108.772



MH-2-36
PROTON CDCl₃

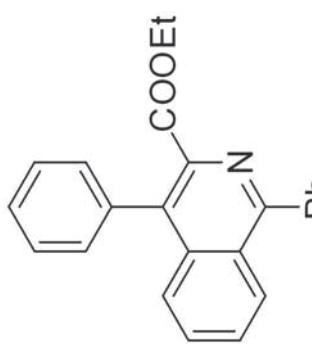


MH-2-36
C13CPD CDC13

— 13.70 —

— 61.32 —

142.14
138.78
136.09
132.00
130.60
130.27
130.05
128.93
128.41
128.28
128.18
128.04
127.80
127.07
126.58



4e

```

NAME          XB20140706
EXPNO         9
PROCNO        1
Date_         20140706
Time          18.36
INSTRUM      spect
PROSHD       5 mm PAIXQ 1.9F
PULPROG      zpgq30
TD           65536
SOLVENT      CDC13
NS            256
DS            4
SWH          30030.029 Hz
FIDRES       0.458222 Hz
AQ            1.0912410 sec
RG            16.128
DW            16.650 usec
DE            6.00  usec
TE            297.7 K
D1           2.0000000 sec
d1           0.0300000 sec
DELT1        1.89999998 sec
TD0           1

```

```

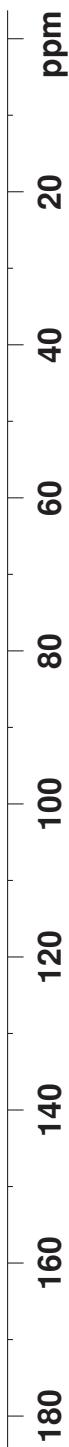
===== CHANNEL f1 =====
NUC1          13C
P1            9.50 usec
PL1           -0.50 dB
SF01        125.7703643 MHz

```

```

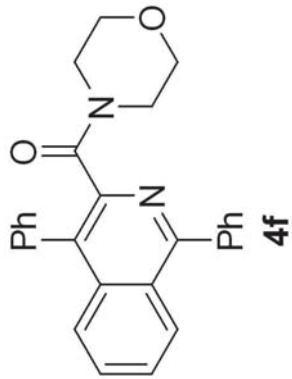
===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           1.00 dB
PL12          16.05 dB
PL13          16.50 dB
SF02        500.1320005 MHz
SI            32768
SF           125.7577890 MHz
WDW          EM
SSB           0
LB           1.00 Hz
GB           0
PC           1.40

```



MH-2-15
PROTON CDCl₃

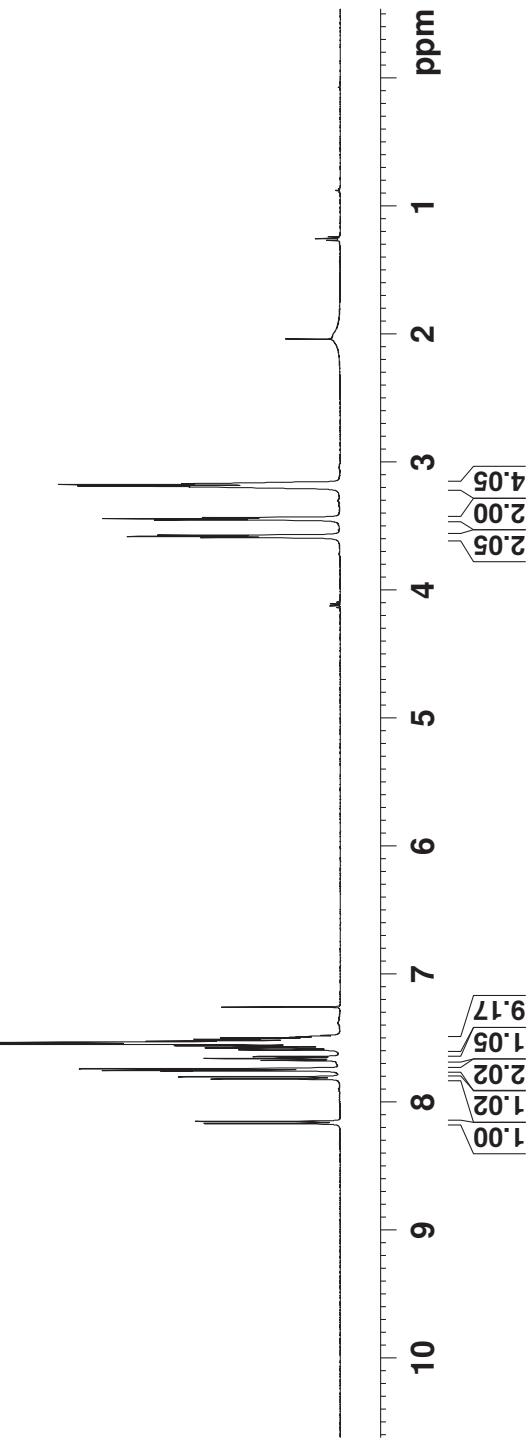
8.171
7.822
7.805
7.758
7.740
7.676
7.665
7.662
7.660
7.648
7.646
7.596
7.539
7.594
7.499
3.592
3.583
3.573
3.454
3.444
3.435
3.189
3.178



```

NAME          XB20140612
EXPNO         6
PROCNO        1
Date_        20140612
Time       11.28
INSTRUM spect
PROBHD   5 mm PABIXO 19F
PULPROG  zg30
TD      65536
SOLVENT    CDCl3
NS       16
DS            2
SWH     10330.578 Hz
FIDRES   0.157632 Hz
AQ      3.11720407 sec
RG      203.2
DW      48.400 usec
DE      6.000 usec
TE      296.8 K
D1   1.00000000 sec
TDO0             1

```

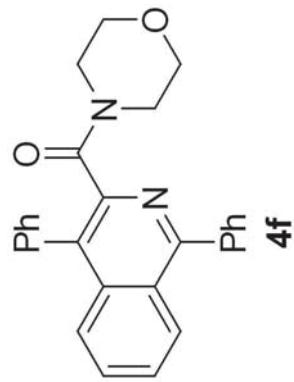


MH-2-15
C1 3CPD CDCl₃

— 41.61
— 46.80

— 66.42
V 66.44

— 145.03
— 145.69
— 138.75
— 135.96
— 134.72
— 130.64
— 130.19
— 128.94
— 128.86
— 128.60
— 128.54
— 128.43
— 127.87
— 127.68
— 126.56
— 125.89



```

=====
NAME          XB20140612
EXPNO         7
PROCNO        1
Date_         20140612
Time          11.44
INSTRUM      spect
PROBHD      5 mm PATXO 19F
PULPROG      zgppg30
TD           65536
SOLVENT      CDCl3
NS            256
DS             4
SWH          30030.029 Hz
FIDRES       0.458222 Hz
AQ            1.0912410 sec
RG            114
DW            16.650 usec
DE            6.00 usec
TE            298.0 K
T1             2.0000000 sec
D1            0.0300000 sec
d1            1.8999998 sec
DELTA         TD0
TD0           1
===== CHANNEL f1 =====
NUC1          13C
P1            9.50 usec
PL1          -0.50 dB
SFO1        125.703643 MHz
===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2            1.00 dB
PL12           16.05 dB
PL13           16.50 dB
SFO2        500.1320005 MHz
SI            32768
SF          125.7577890 MHz
WDW           no
SSB            0
LB            0.00 Hz
GB            0
PC            1.40

```

MH-2-46
PROTON CDCl₃

1.148
1.351
1.373
1.427
1.473

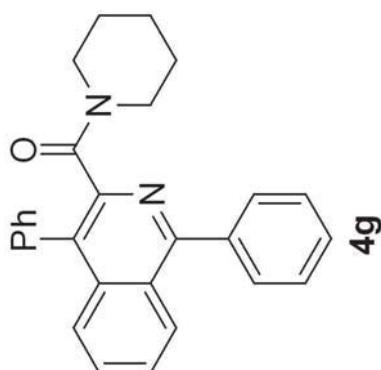
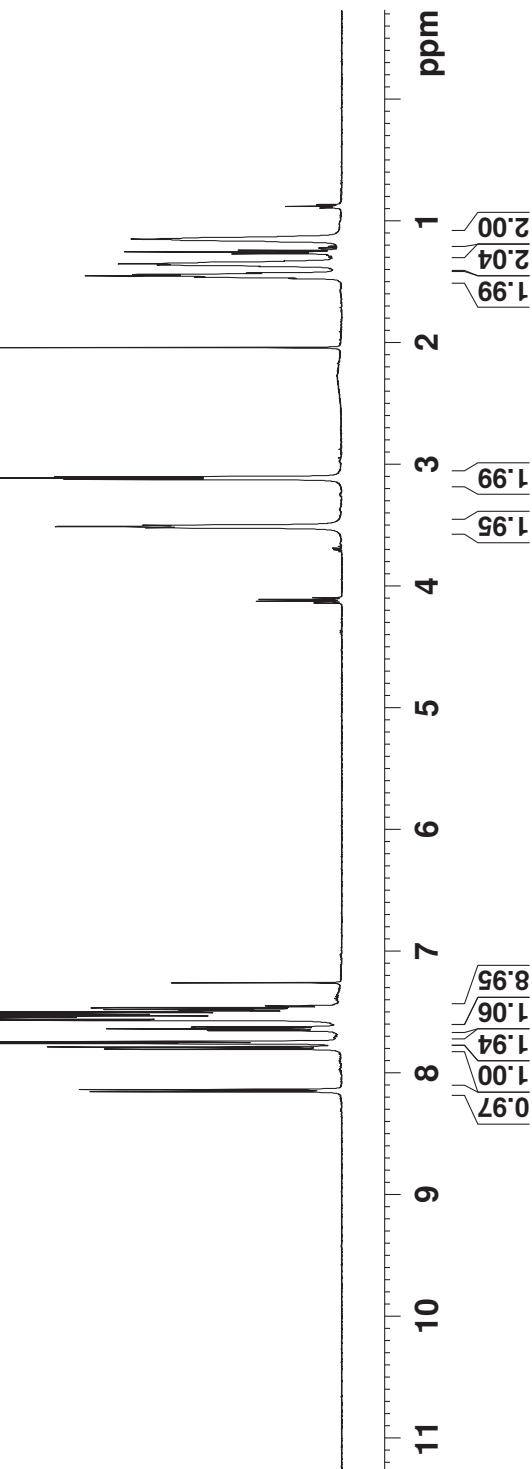
3.101
3.112
3.123
3.499
3.510
3.521

7.450
7.512
7.566
7.621
7.623
7.635
7.637
7.640
7.651
7.653
7.741
7.743
7.757
7.760
7.787
7.804
8.137
8.153

```

NAME          XB20140714
EXPNO         10
PROCNO        1
Date_-
Time_-
INSTRUM      FIDCO 19F
PROBHD       5 mm FIDCO 19F
PULPROG      zg30
TD           65536
SOLVENT       CDCl3
NS            16
DS            2
SWH          10330.578 Hz
FIDRES       0.157632 Hz
AQ            3.1720407 sec
RG            161.3
DW            48.400 usec
DE            6.00 usec
TE            296.4 K
D1           1.00000000 sec
TD0           1

```

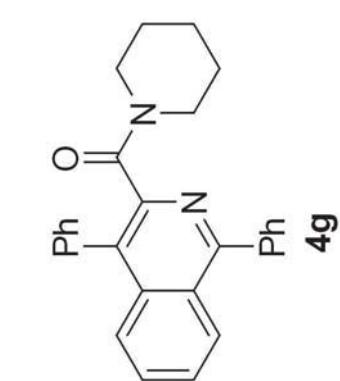


MH-2-46
C13CPD CDCL3

25.81
25.13
24.32

42.10
47.54

125.93
126.40
127.37
127.79
128.24
128.36
128.50
128.81
130.24
130.52
130.77
134.87
136.11
138.88
146.11
160.38
167.35



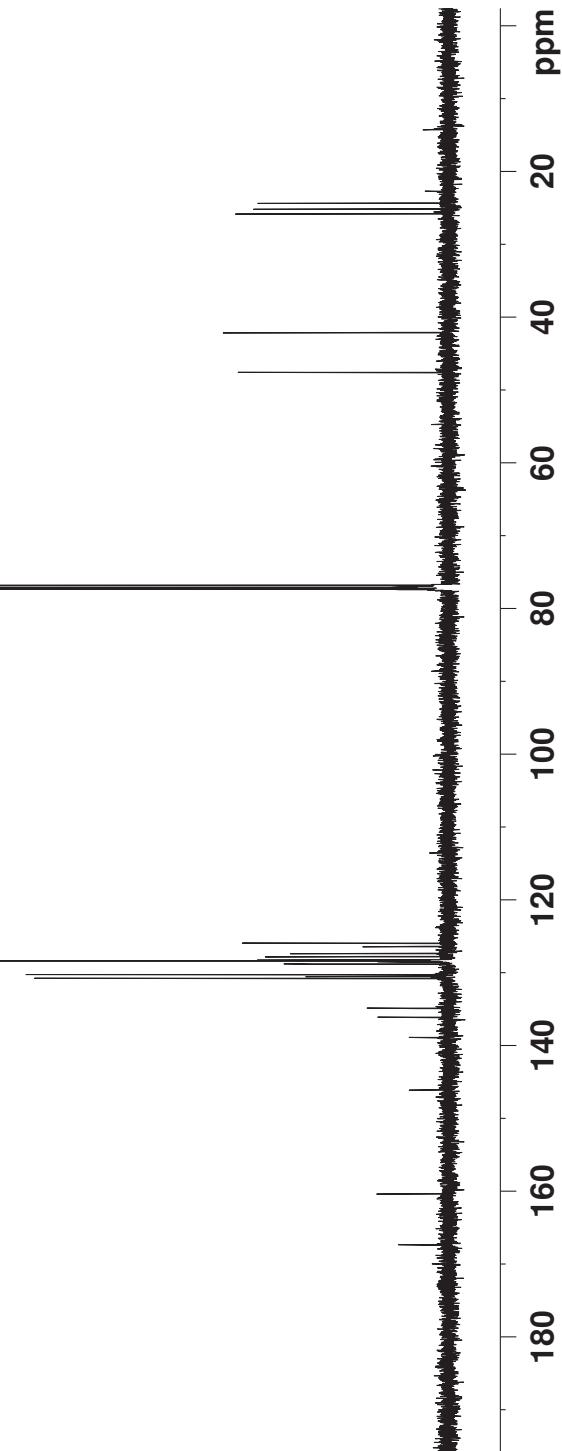
```

NAME: XB20140714
EXPNO: 12
PROCNO: 1
Date: 20140714
Time: 12.09
INSTRUM: spect
PROBHD: 5 mm PATXO 19F
PULPROG: zpg30
TD: 65536
SOLVENT: CDCl3
NS: 256
DS: 4
SWH: 300030.029 Hz
FIDRES: 0.458222 Hz
AQ: 1.0912410 sec
RG: 101.6
DW: 16.650 usec
DE: 6.00 usec
TE: 297.9 K
D1: 2.0000000 sec
d11: 0.03000000 sec
DELTA: 0.1.89999998 sec
TDO: 1

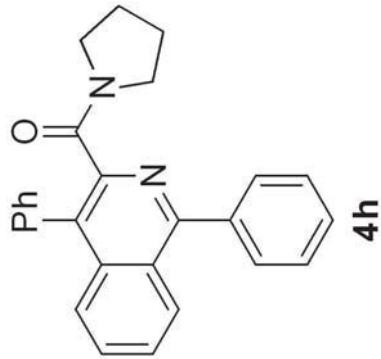
===== CHANNEL f1 =====
NUC1: 13C
P1: 9.50 usec
PL1: -0.50 dB
SFO1: 125.7703643 MHz

===== CHANNEL f2 =====
CPDPG2: waltz16
NUC2: 1H
PCPD2: 80.00 usec
PL2: 1.00 dB
PL12: 16.05 dB
PL13: 16.50 dB
SFO2: 500.1320005 MHz
SI: 32768
SF: 125.7577890 MHz
WDW: EM
SSB: 0
LB: 1.00 Hz
GB: 0
PC: 1.40

```



MH-2-47
PROTON CDC13

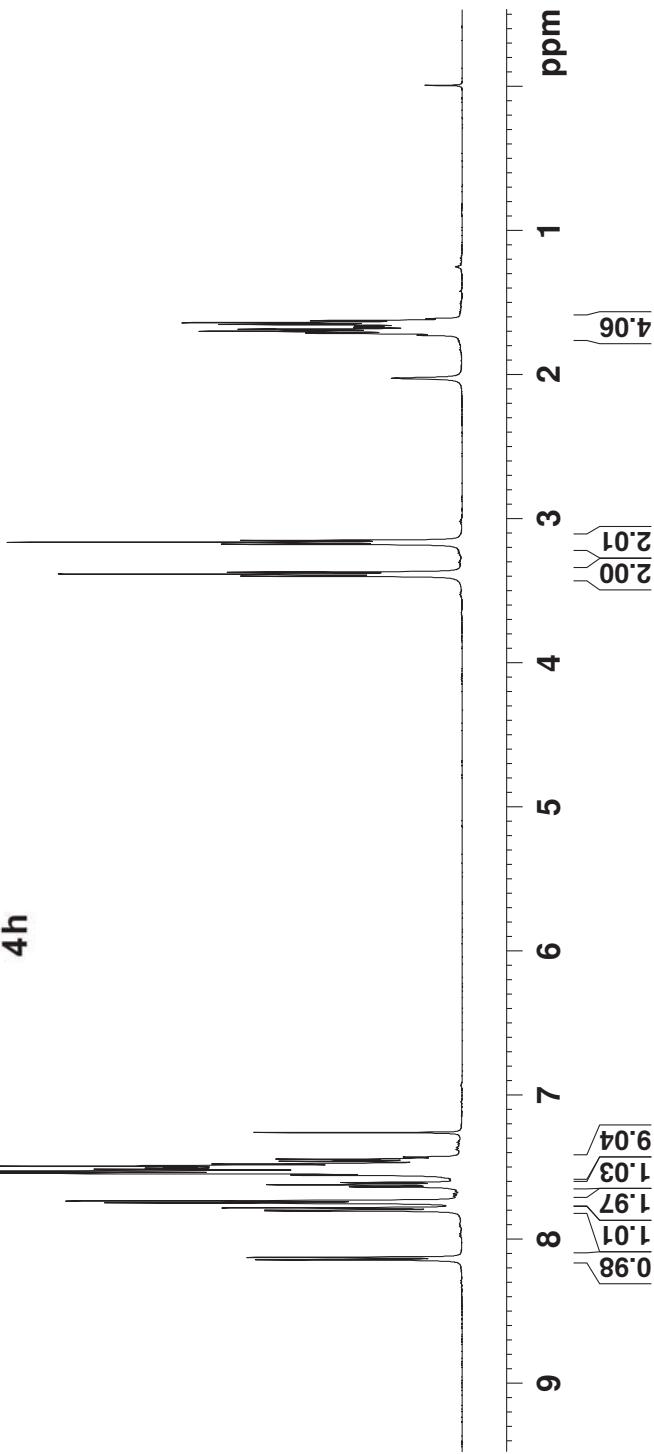


```

NAME          XB20150831
EXPNO         16
PROGNO        1
Date-        20150831
Time-        13.04
INSTRUM      PROBHD
PROBHD       5 mm
PULPROG      PTD
TD           16
SOLVENT      NS
DS           2
SWH          10330.578 Hz
FIDRES      0.157632 Hz
AQ           3.1720407 sec
RG           80.6
DW           48.400 use
DE           6.00 use
TE           295.4 K
TEDE        1.00000000 sec
D1           1
TD0          1

===== CHANNEL f1 =====
NUC1          1H
P1           14.14 use
PL1          1.00 dB
SFO1         500.1330885 MHz
SI           32768
SF           500.1300126 MHz
WDW          no
SSB           0
LB           0.00 Hz
GB           0
PC           1.00

```

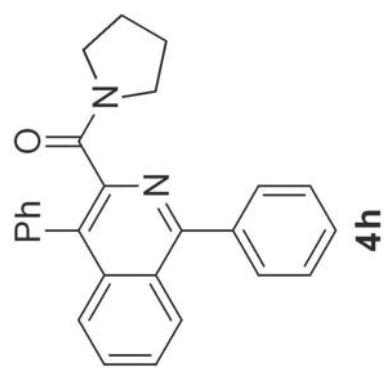


MH-2-47
C13CPD CDCl₃

25.75
24.25

47.44
45.11

125.91
126.52
127.40
127.76
128.22
128.36
128.38
128.52
128.74
128.74
130.14
130.43
130.46
130.50
130.52
136.17
139.13
147.01
160.48
167.40



NAME XB20150831-C4
EXPNO 20
PROCNO 1
Date_ 20150901
Time 0.44
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 512
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 181
DW 16.650 usec
DE 6.00 usec
TE 297.7 K
D1 2.0000000 sec
d1 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
===== CHANNEL f1 =====
NUC1 13C
P1 9.50 usec
PL1 -0.50 dB
SFO1 125.7703643 MHz
TD0 1
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL1.2 16.05 dB
PL1.3 16.50 dB
SFO2 500.1320005 MHz
SI 32768
SF 125.7577890 MHz
WDW 0
SSB LB 1.00 Hz
LB GB 0
PC PC 1.40

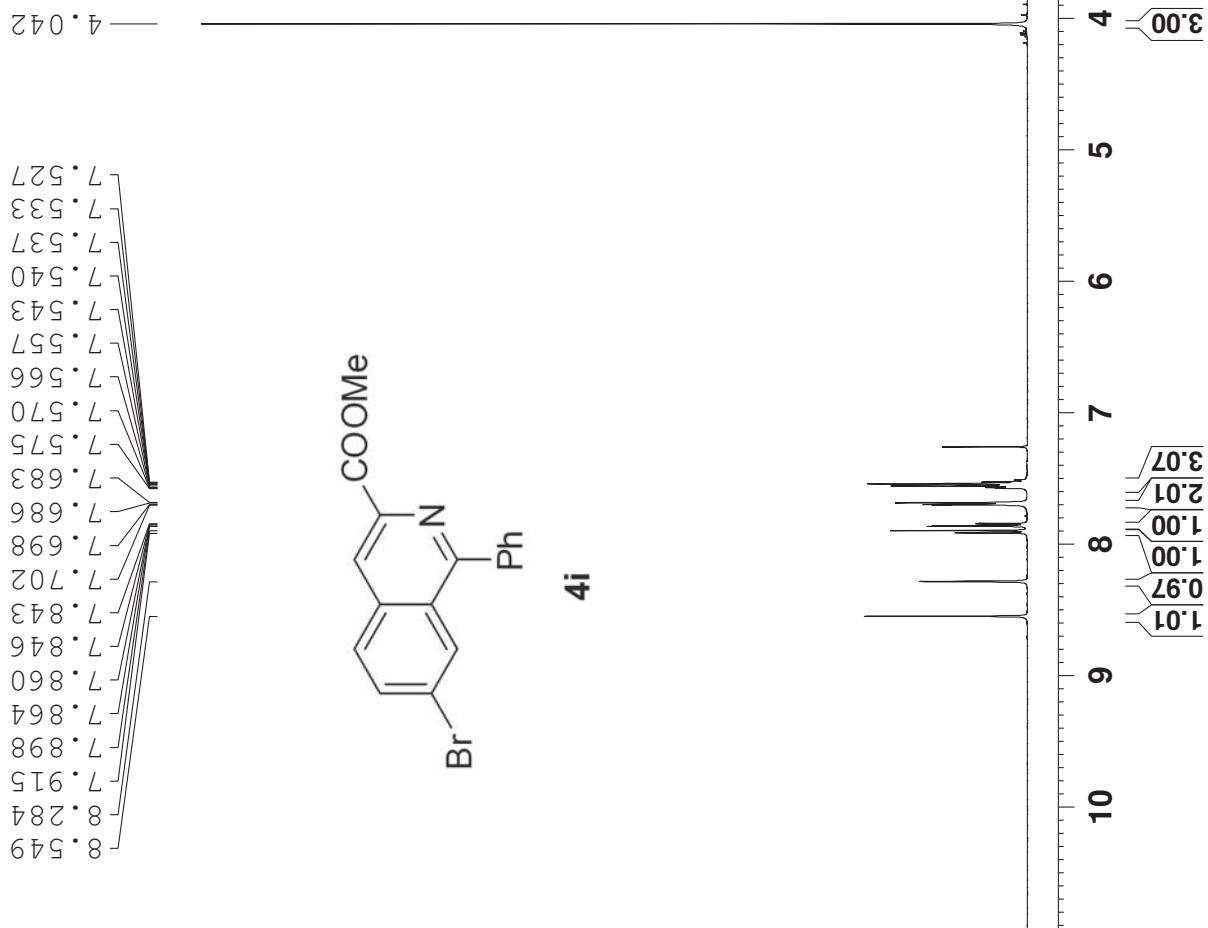
MH-2-51
PROTON CDCl₃

```

NAME          XB20140714
EXPNO         15
PROCNO        1
Date_         20140714
Time          12.36
INSTRUM       spect
PROBHD        5 mm PABXO 19F
PULPROG       zg30
TD             65536
SOLVENT        CDCl3
NS             16
DS             2
SWH            10330.578 Hz
FIDRES        0.157632 Hz
AQ             3.172047 sec
RG             256
DW             48.400 usec
DE             6.00 usec
TE             296.9 K
D1             1.00000000 sec
TDO             1

===== CHANNEL f1 =====
NUC1           1H
P1             14.14 usec
PL1            1.00 dB
SFO1          500.1330885 MHz
SI              32768
SF             500.1300129 MHz
WDW            no
SSB            0
LB             0.00 Hz
GB             0
PC             1.00

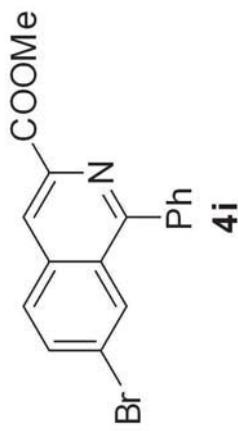
```



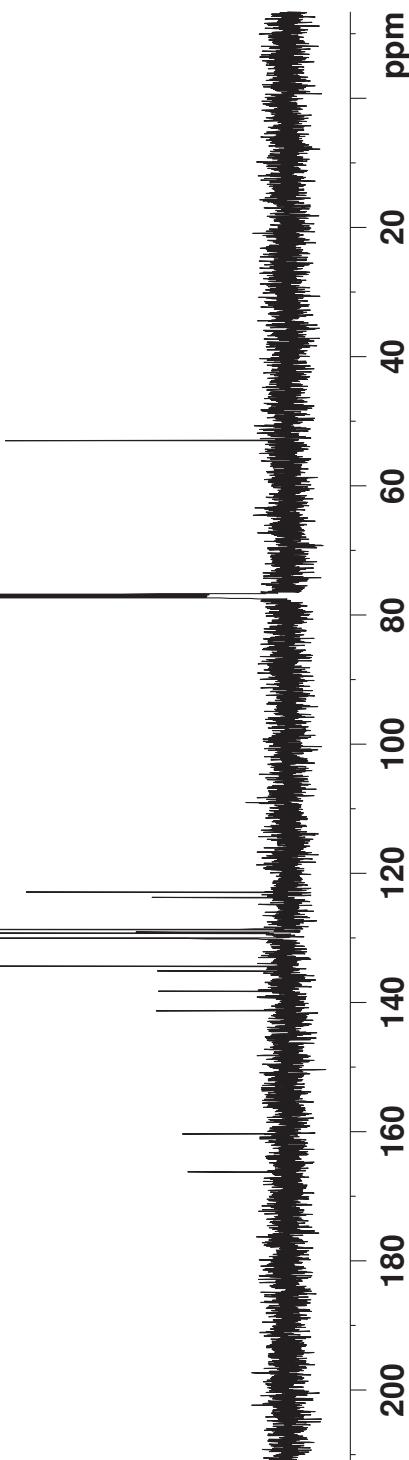
MH-2-51
C13CPD CDC13

— 52.98 —

166.26
160.34
141.28
135.12
138.25
130.10
134.38
130.06
130.01
129.26
129.13
128.67
123.75
122.90
—



NAME XB20140714
EXPNO 16
PROCNO 1
Date_ 20140714
Time 12.51
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 90.5
DW 16.650 usec
DE 6.00 usec
TE 298.1 K
D1 2.0000000 sec
d11 0.0300000 sec
DELTA 1.8999998 sec
TDO 1
===== CHANNEL f1 =====
NUC1 13C
P1 9.50 usec
PL1 -0.50 dB
SFO1 125.7703643 MHz
===== CHANNEL f2 =====
CPDPRG2 waitz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL1.2 16.05 dB
PL1.3 16.50 dB
SFO2 500.1320005 MHz
SI 322768
SF EM
WDW 0
SSB 2.00 Hz
LB 0
GB 1.40
PC

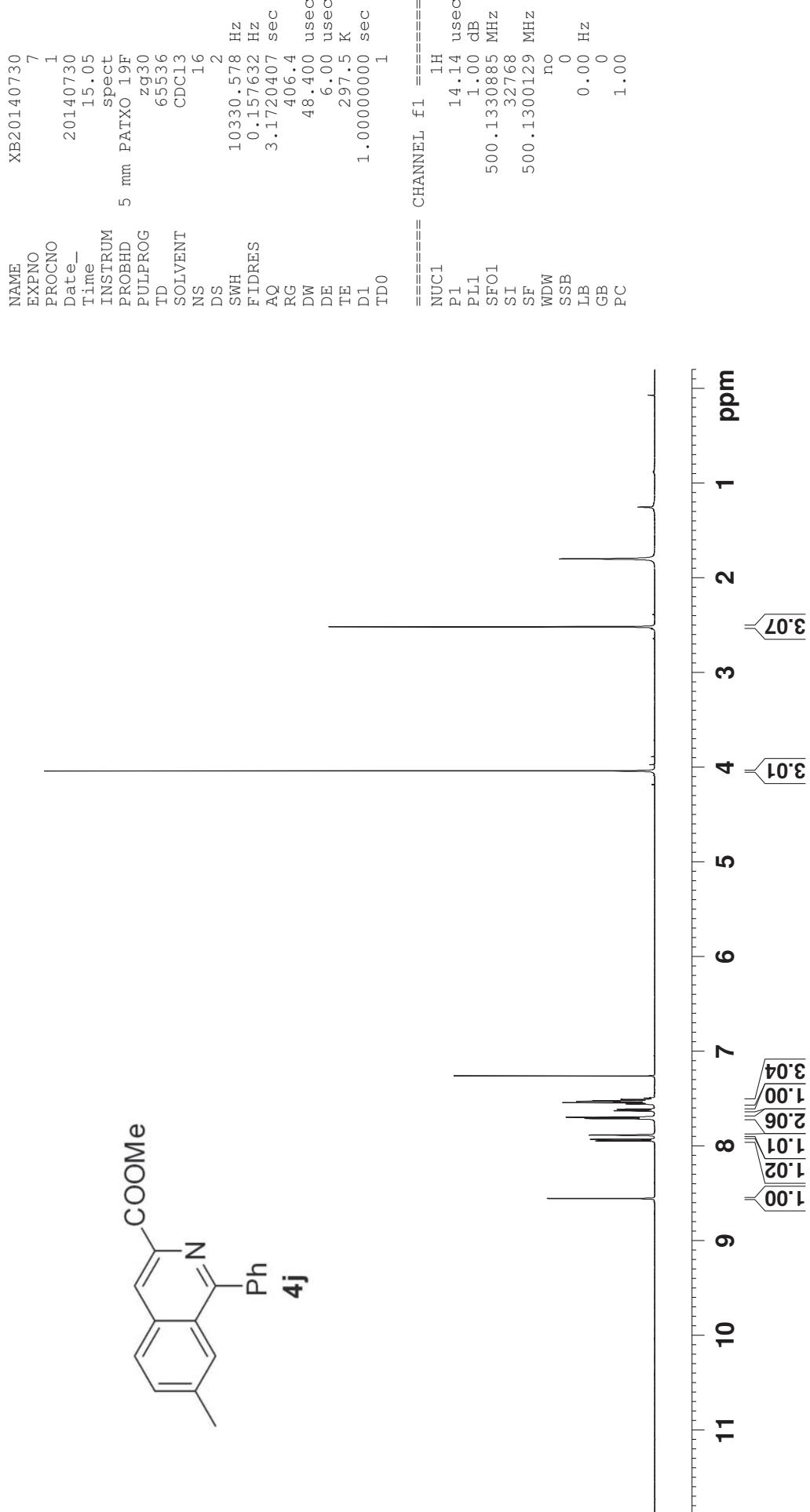
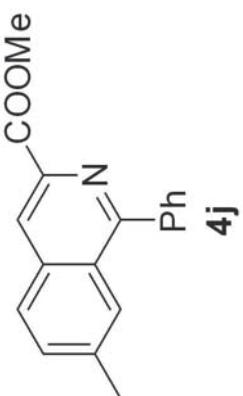


MH-2-62
PROTON CDCl₃

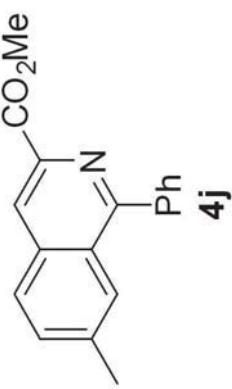
— 2.516 —

— 4.038 —

7.508
7.514
7.519
7.522
7.528
7.539
7.542
7.552
7.555
7.560
7.611
7.614
7.628
7.630
7.695
7.697
7.710
7.714
7.884
7.885
7.929
7.946
8.555



140.15
 139.95
 139.13
 134.78
 132.93
 130.11
 130.11
 128.79
 128.49
 128.41
 128.29
 126.66
 123.26

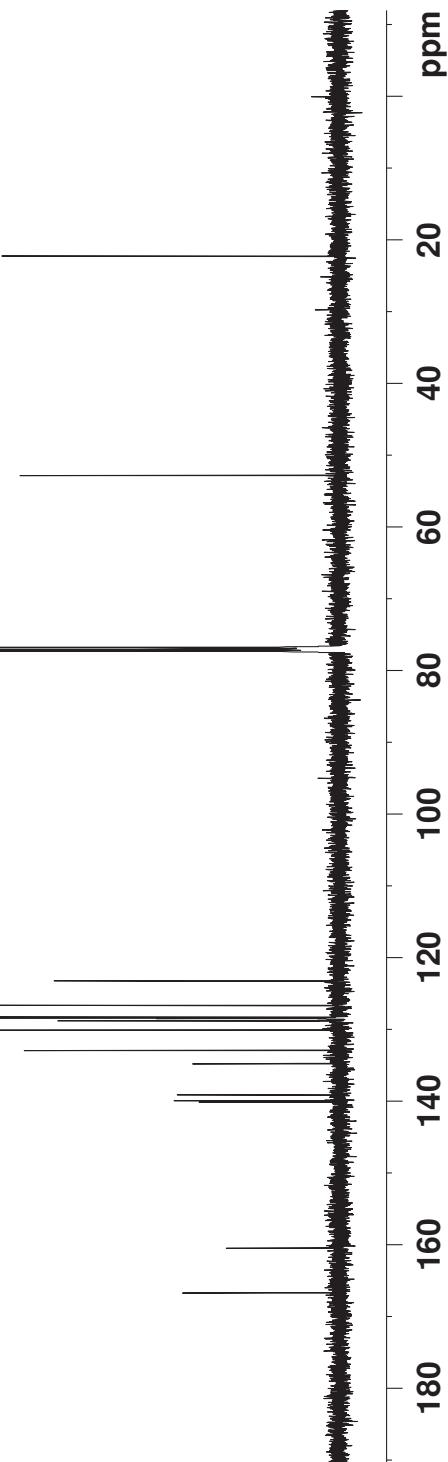


22.82
 22.4

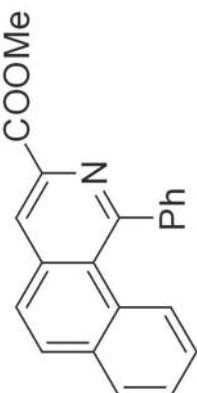
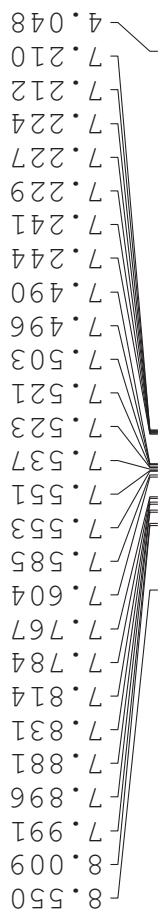
MH-2-62
C13CPD CDCl₃

```

NAME          XB20150831-C4
EXPNO         22
PROCNO        1
Date_        20150901
Time         2.16
INSTRUM      spect
PROBHD       5 mm PAXCO 19F
PULPROG      zpg30
TD           65536
SOLVENT      CDC13
NS            1024
DS            4
SWH          30030.029 Hz
FIDRES       0.458222 Hz
AQ           1.0912410 sec
RG           128
DW           16.650 usec
DE           6.00 usec
TE           297.8 K
D1           2.0000000 sec
d11          0.0300000 sec
DELTA        1.89999998 sec
TDO          1
        ===== CHANNEL f1 =====
NUC1          13C
P1            9.50 usec
PL1          -0.50 dB
SFO1        125.7703643 MHz
        ===== CHANNEL f2 =====
CPDPG2        waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           1.00 dB
PL12          16.05 dB
PL13          16.50 dB
SFQ2        500.1320005 MHz
SI            32768
SF           125.7577890 MHz
WDW          EM
SSB           0
LB           1.00 Hz
GB           0
PC           0.20
  
```

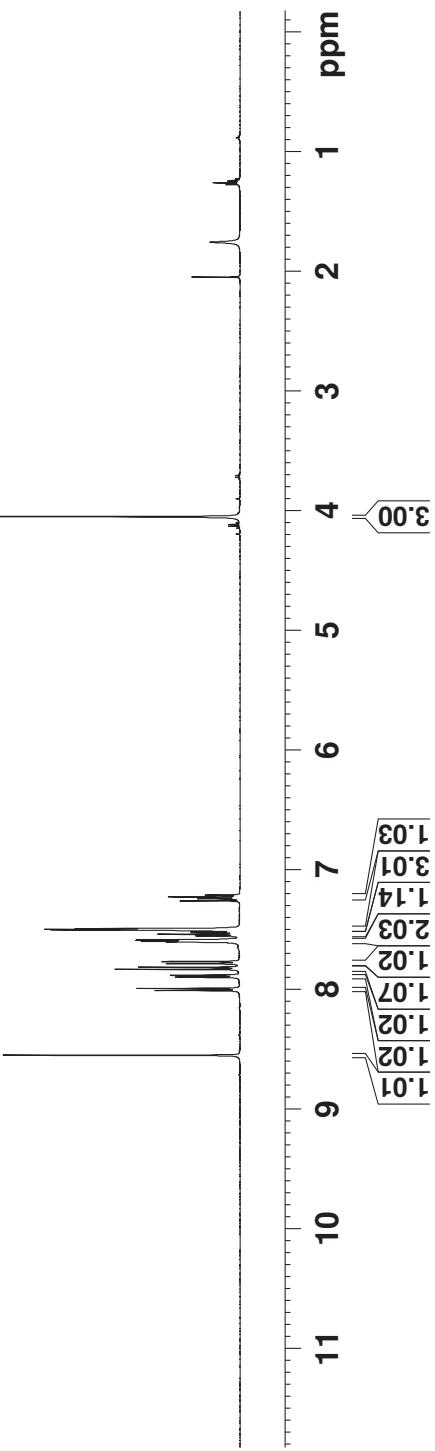


MH-2-50
PROTON CDC13



4k

NAME	EXPNO	PROCNNO	Date—	Time—	INSTRUM	PRBHD	PULPROG	TD	SOLVENT	NS	DS	SWH	FIDRES	AQ	RG	DW	DE	TE	D1	TDO	==== CHANNEL f1 =====	NUCI	P1	PL1	SFO1	SI	SF	WDW	SSB	LB	GB	PC
XB20140714	13		20140714	12.15	spec	5 mm	PATXO 19F						10.330	5.778 Hz	0.157632 Hz	3.1720407 sec	203.2	48.400 usec	6.00 usec	2.967 K	1.00000000 sec	1	1H	14.14 usec	1.00 dB	500.1330885 MHz	327.68	500.1330126 MHz	no	0	0.00 Hz	1.00



159.40
 166.26
 141.61
 134.33
 138.34
 132.77
 129.30
 129.21
 129.11
 128.76
 128.74
 128.22
 127.70
 126.35
 125.63
 125.21
 126.21
 127.44

MH-2-50
C13CPD CDCL3

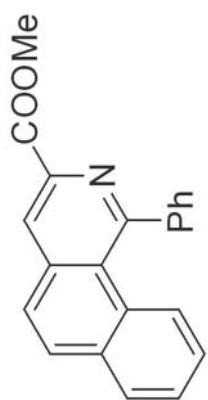
```

NAME          XB20140714
EXPNO         14
PROCNO        1
Date_         20140714
Time_         12.30
INSTRUM      spect
PROBHD       5 mm PATXO 19F
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3
NS            256
DS            4
SWH          30030.029 Hz
FIDRES       1.0912410 sec
AQ            128
RG            0.458222 Hz
DW            16.650 usec
DE            6.00 usec
TE            298.1 K
D1            2.0000000 sec
d11           0.0300000 sec
DELTA         1.89999998 sec
TD0           1

=====
CHANNEL f1
NUC1          13C
P1            9.50 usec
PL1           -0.50 dB
SFO1         125.7703643 MHz

=====
CHANNEL f2
CPDPRG2      waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           1.00 dB
PL12          16.05 dB
PL13          16.50 cB
SFO2         500.1320005 MHz
SI            32768
SF           125.7577890 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB           0
PC           1.40

```



4k

— 52.92 —

MH-2-59
PROTON CDCl₃

```

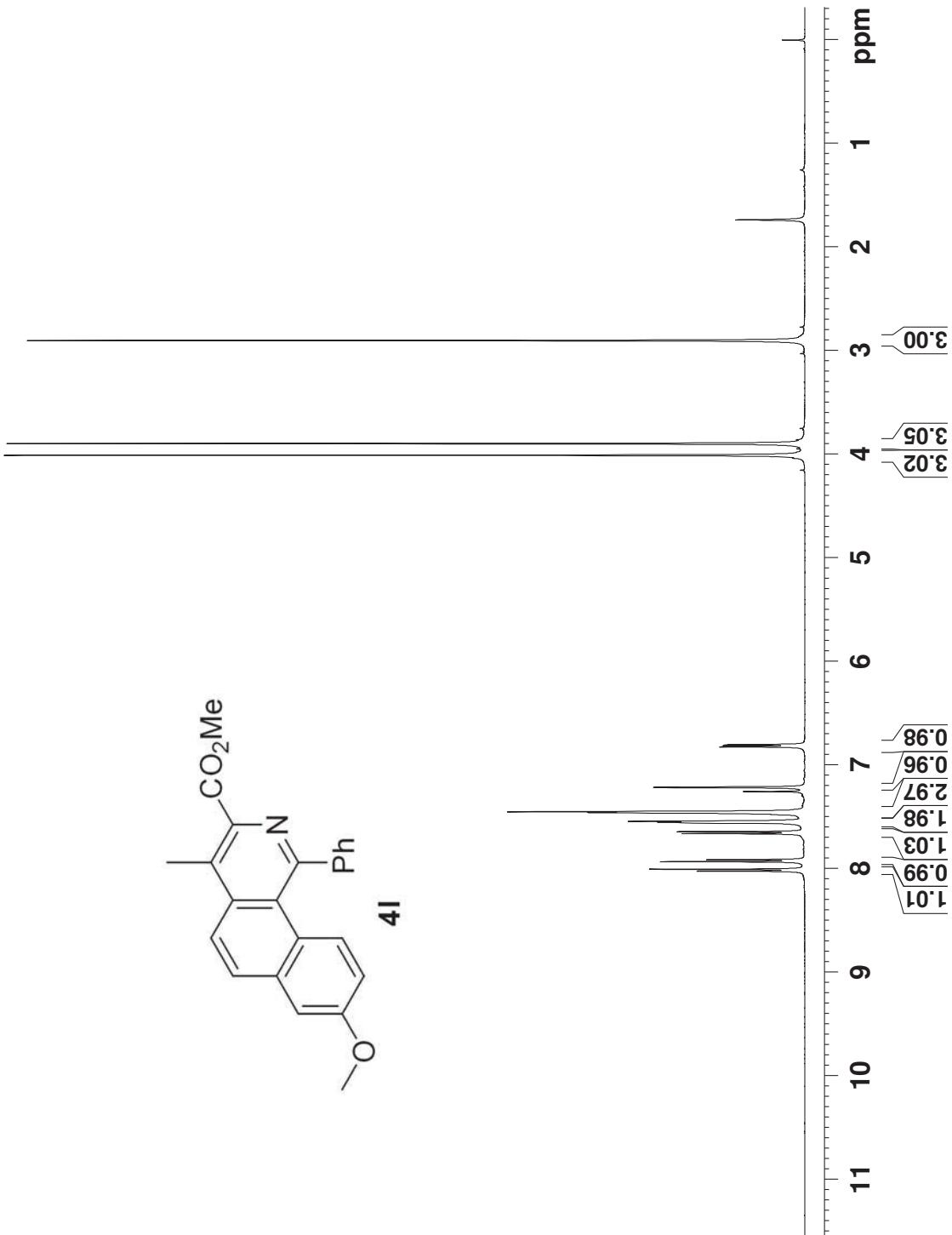
NAME          XB20150831
EXPNO         17
PROCNO        1
Date_         20150831
Time_         13.10
INSTRUM      spect
PROBHD       5 mm PATXO 19F
PULPROG      zg30
TD           65536
SOLVENT      CDCl3
NS            16
DS            2
SWH          103330.578 Hz
FIDRES       0.157632 Hz
AQ            3.1720407 sec
RG            114
DW            48.400 usec
DE            6.00 usec
TE            295.6 K
D1           1.0000000 sec
TDO          1

```

```

===== CHANNEL f1 =====
NUC1          1H
P1            14.14 usec
PL1           1.00 dB
SFO1         500.1330885 MHz
SI             32768
SF           500.1300123 MHz
WDW          no
SSB           0
LB            0.00 Hz
GB            0
PC            1.00

```



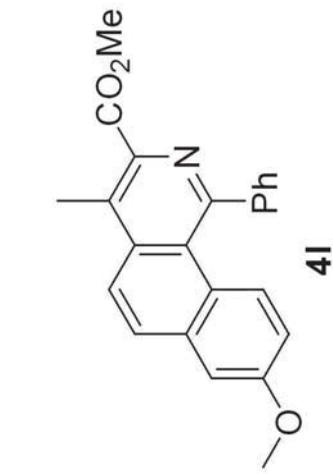
MH-2-59
C13CPD CDC13

14.79

168.02
158.50
143.63
141.50
136.66
135.13
131.71
130.08
129.21
128.45
127.67
125.01
123.70
122.02
116.50
111.50
108.34

55.36
52.66

XB20150831-C4
21
1
20150901
1
1.17
spect
5 mm PATXO 19F
zgppg30
65536
CDC13
51.2
4
30030.029 Hz
0.458222 Hz
1.0912410 sec
456.1
16.650 usec
6.00 usec
297.1 K
2.00000000 sec
0.03000000 sec
1.89999998 sec
1
1
===== CHANNEL f1 =====
NUC1 1.3C
P1 9.50 usec
PL1 -0.50 dB
SFO1 125.7703643 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.05 dB
PL13 16.50 dB
SF02 500.1320005 MHz
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

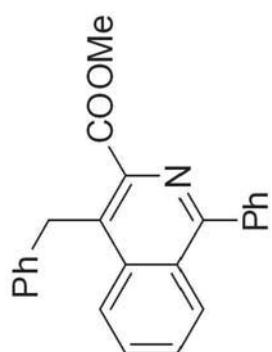
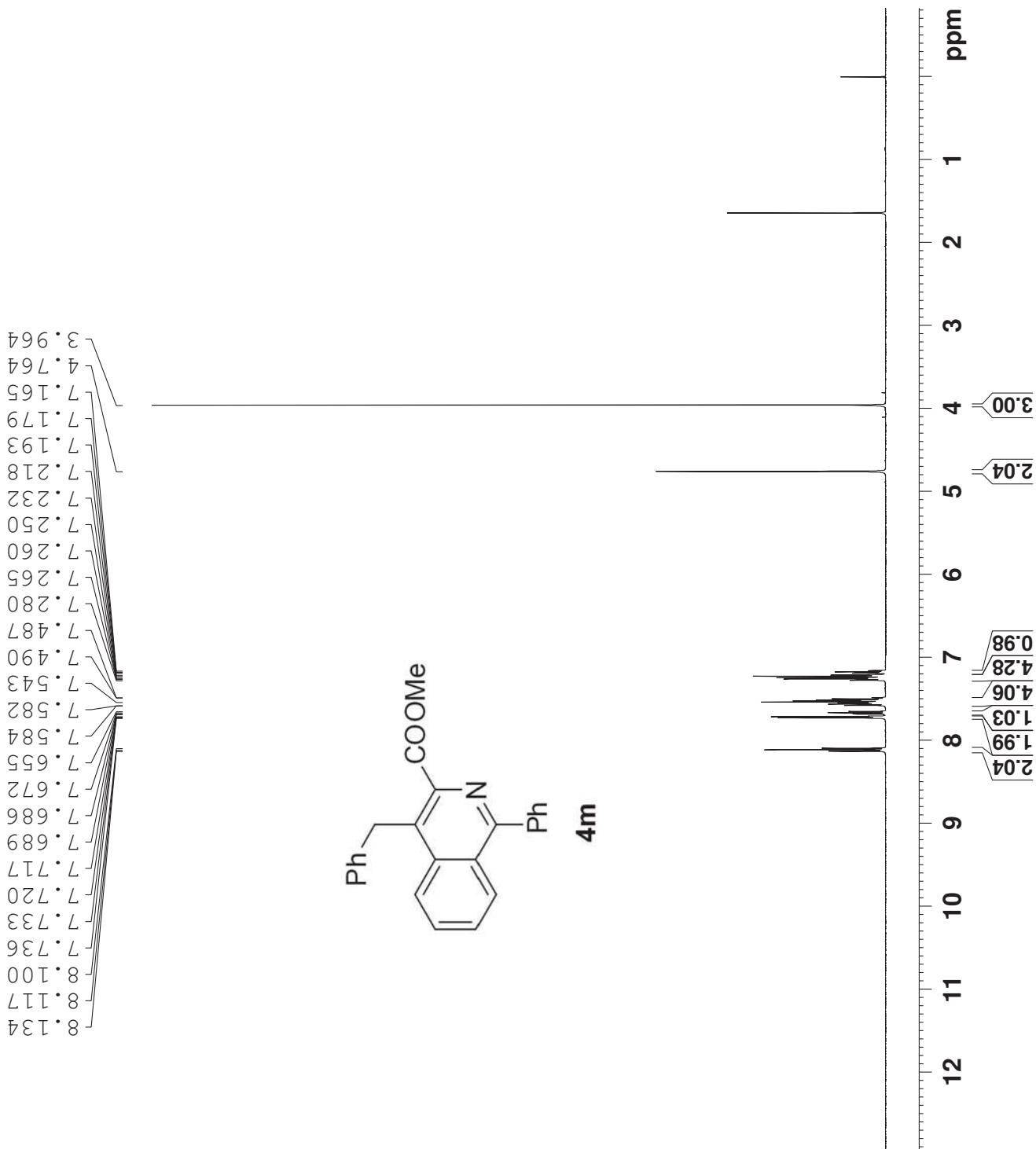


MH-3-97-A
PROTON CDC13

NAME	XB20150928
EXPNO	16
PROCNO	1
Date_	20150928
Time_	16.34
INSTRUM	spec
PROBHD	5 mm
PULPROG	PATXO_19F
TD	zg30 65536
SOLVENT	CDC13
NS	16
DS	2
SWH	10330.0.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	181
DW	48.400 usec
DE	6.00 usec
TE	295.3 K
D1	1.0000000 sec
TD0	1

===== CHANNEL f1 =====

NUC1	1H
P1	14.14 usec
PL1	1.00 dB
SFO1	500.1330885 MHz
SI	327.68
SF	500.1300129 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



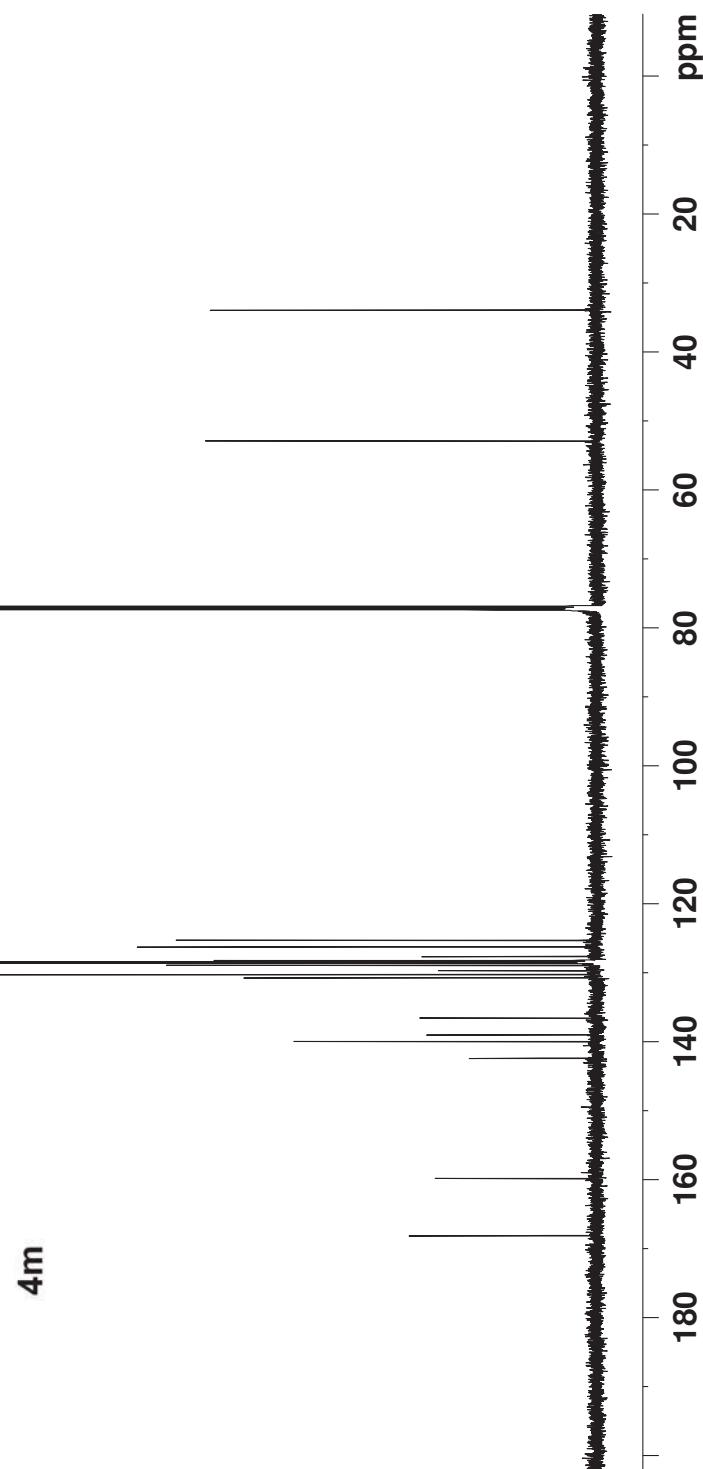
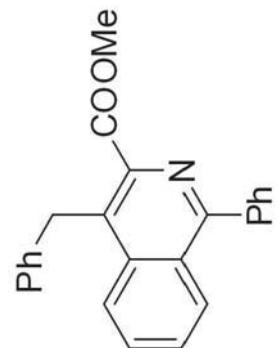
MH-3-97-A
C13CPD CDCl₃

NAME MH20150928-C
EXPNO 22
PROCNO 1
Date 20150929
Time 1.21
INSTRUM spect
PROBHD 5 mm PAXCO 1.9F
PULPROG zgppg30
TD 65536
SOLVENT CDCl₃
NS 1024
DS 4
SWH 30030.1029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 128
DW 16.650 usec
DE 6.00 usec
TE 297.1 K
D1 2.0000000 sec
d11 0.0300000 sec
DELTA 1.899999998 sec
TDD 1
===== CHANNEL f1 =====
NUC1 13C
P1 9.50 usec
PL1 -0.50 dB
SFO1 125.7703643 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.05 dB
PL13 16.50 dB
SFO2 500.1320005 MHz
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

33.89

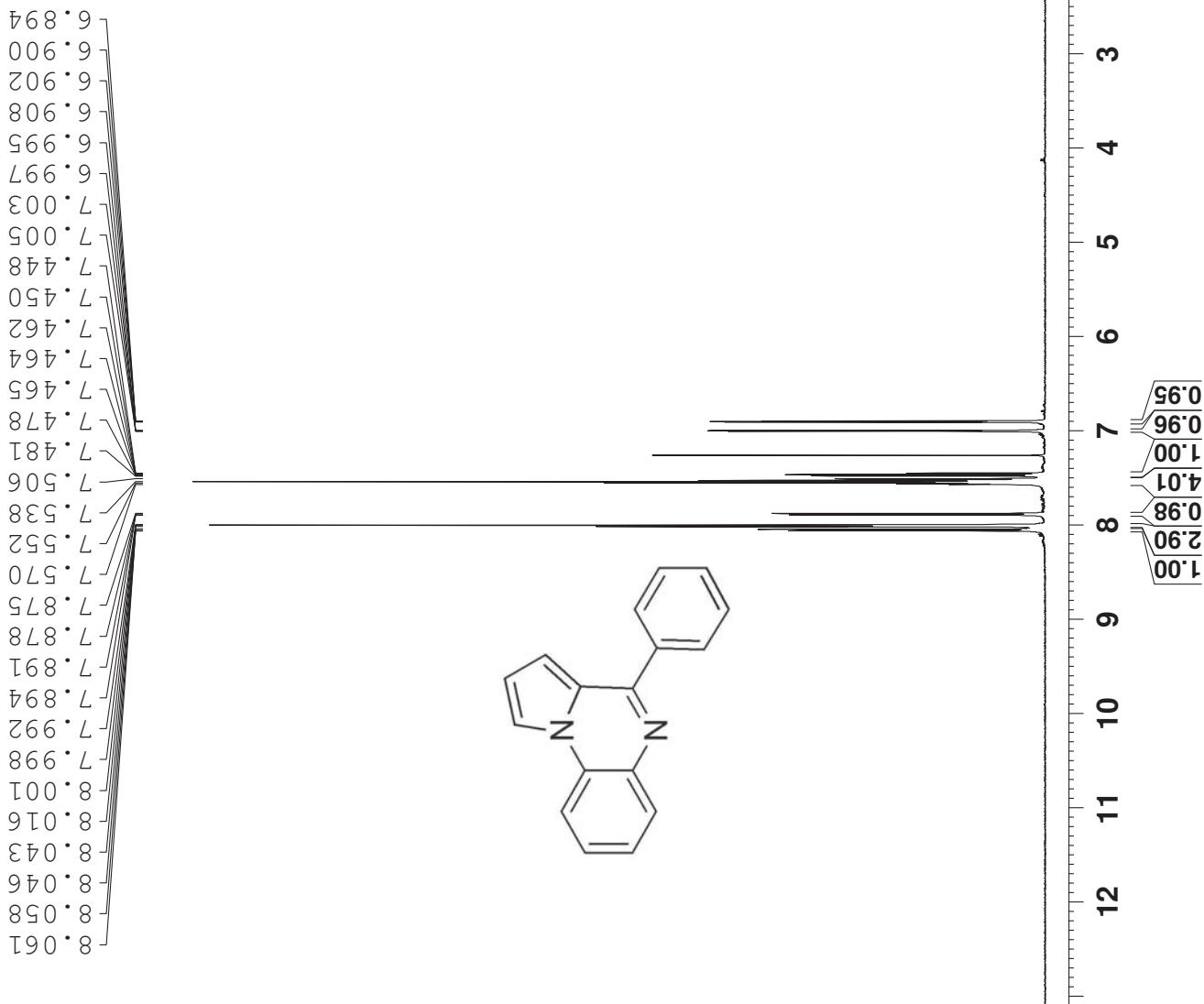
52.83

159.77
168.06
142.34
138.91
136.50
130.67
130.23
129.63
128.87
128.56
128.44
128.36
127.61
126.18
125.22



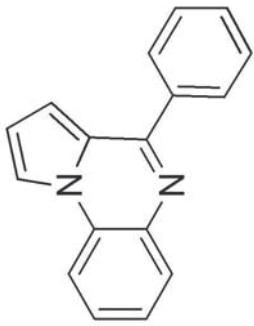
MH-3-123
PROTON CDCl₃

NAME XB20151128
EXPNO 1
PROCNO 1
Date_ 20151128
Time 23.11
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 256
DW 48.400 usec
DE 6.00 usec
TE 295.4 K
D1 1.0000000 sec
TDO 1
===== CHANNEL f1 ======
NUC1 1H
P1 14.14 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300129 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

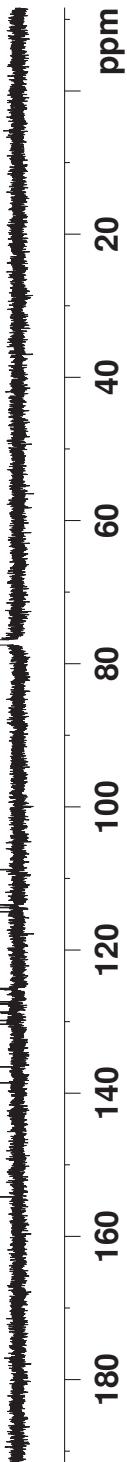


MH-3-123
C13CPD CDC13

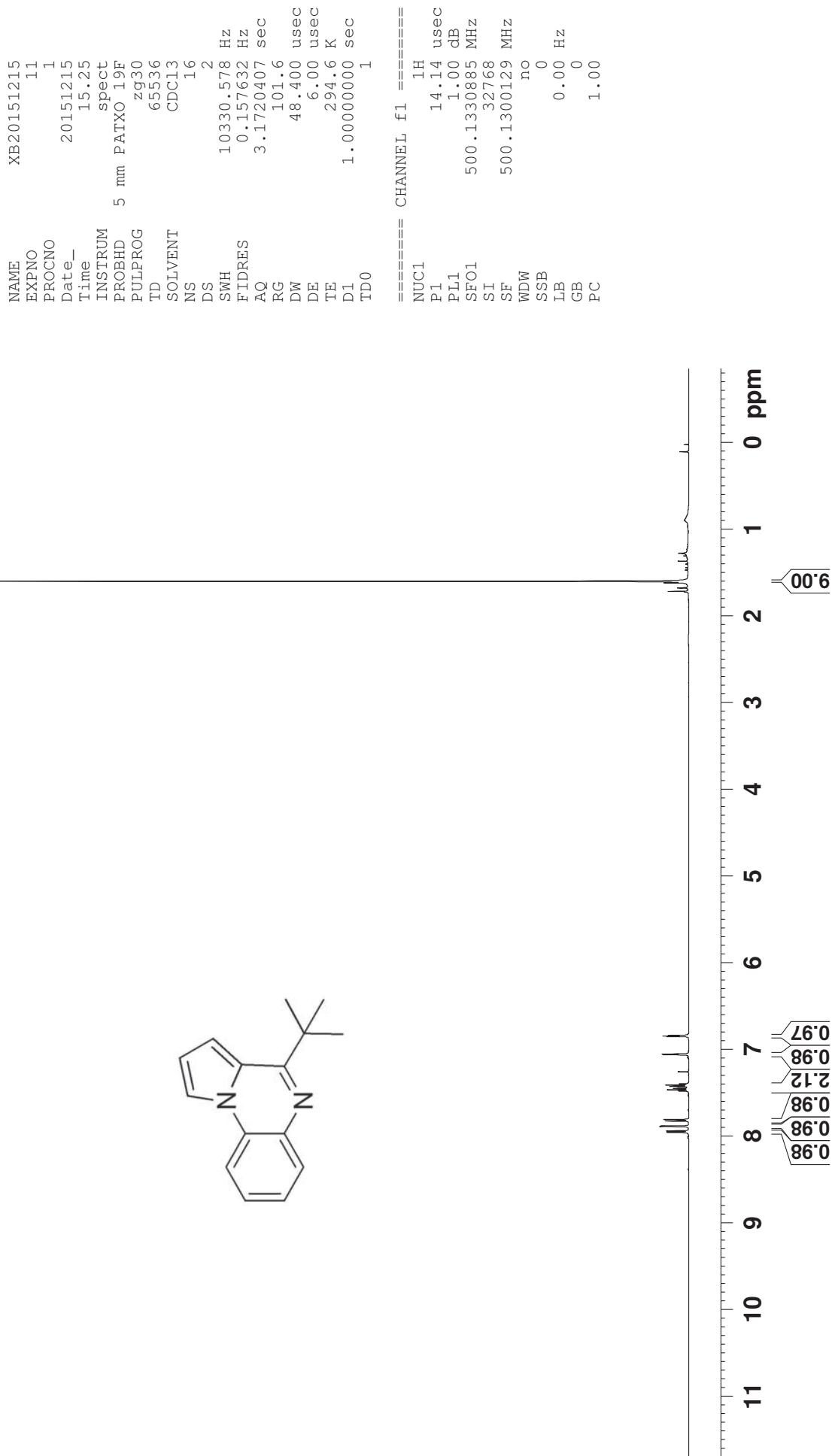
154.44
138.50
136.30
130.28
129.81
128.63
128.60
127.49
127.19
125.42
125.29
124.61
123.99
123.65
113.71



===== NAME XB20151128
EXPNO 2
PROCNO 1
Date_ 20151129
Time 0.07
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zppg30
TD 65536
SOLVENT CDC13
NS 1024
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 114
DW 16.650 usec
DE 6.00 usec
TE 297.5 K
D1 2.0000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
===== CHANNEL f1 ======
NUC1 13C
P1 9.50 usec
PL1 -0.50 dB
SFO1 125.7703643 MHz
===== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.05 dB
PL13 16.50 dB
SFO2 500.1320005 MHz
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



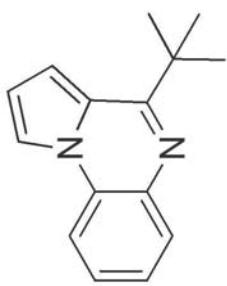
MH-5B
PROTON CDC13



MH-5B
C13CPD CDC13

162.95

135.38
130.11
127.12
127.10
124.83
123.93
113.34
113.04
112.72
108.33



39.32
29.51

===== NAME XB20151215
EXPNO 13
PROCNO 1
Date_ 20151215
Time 15.35
INSTRUM spect
PROBHD 5 mm PAXTO 19F
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 225
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 128
DW 16.650 usec
DE 6.00 usec
TE 295.3 K
D1 2.0000000 sec
d11 0.0300000 sec
DELTA 1.89999998 sec
TDO 1
===== CHANNEL f1
===== NUC1 13C
P1 9.50 usec
PL1 -0.50 dB
SFO1 125.7703643 MHz
===== CHANNEL f2
===== CPDPRG2 WALTZ16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.05 dB
PL13 16.50 dB
SFO2 500.1320005 MHz
SI 32768
SF 125.7577890 MHz
WDW 0
SSB 1.00 Hz
LB 0
GB 0
PC 1.40



MH-3-124
PROTON CDCl₃

— 2.653 —

```

NAME          xb20151124
EXPNO         21
PROCNO        1
Date_         20151124
Time_          3.12
INSTRUM      spect
PROBHD      5 mm PATXO 19F
PULPROG     zg30
TD           65536
SOLVENT      CDCl3
NS            16
DS            2
SWH          10330.578 Hz
FIDRES       0.157632 Hz
AQ            3.1720407 sec
RG            161.3
DW            48.400 usec
DE            6.00 usec
TE            295.6 K
D1           1.00000000 sec
TD0           1

```

```

===== CHANNEL f1 =====
NUC1          1H
P1           14.14 usec
PL1          1.00 dB
SFO1        500.1330885 MHz
SI            32768
SF          500.1300126 MHz
WDW           no
SSB            0
LB             0.00 Hz
GB             0
PC            1.00

```

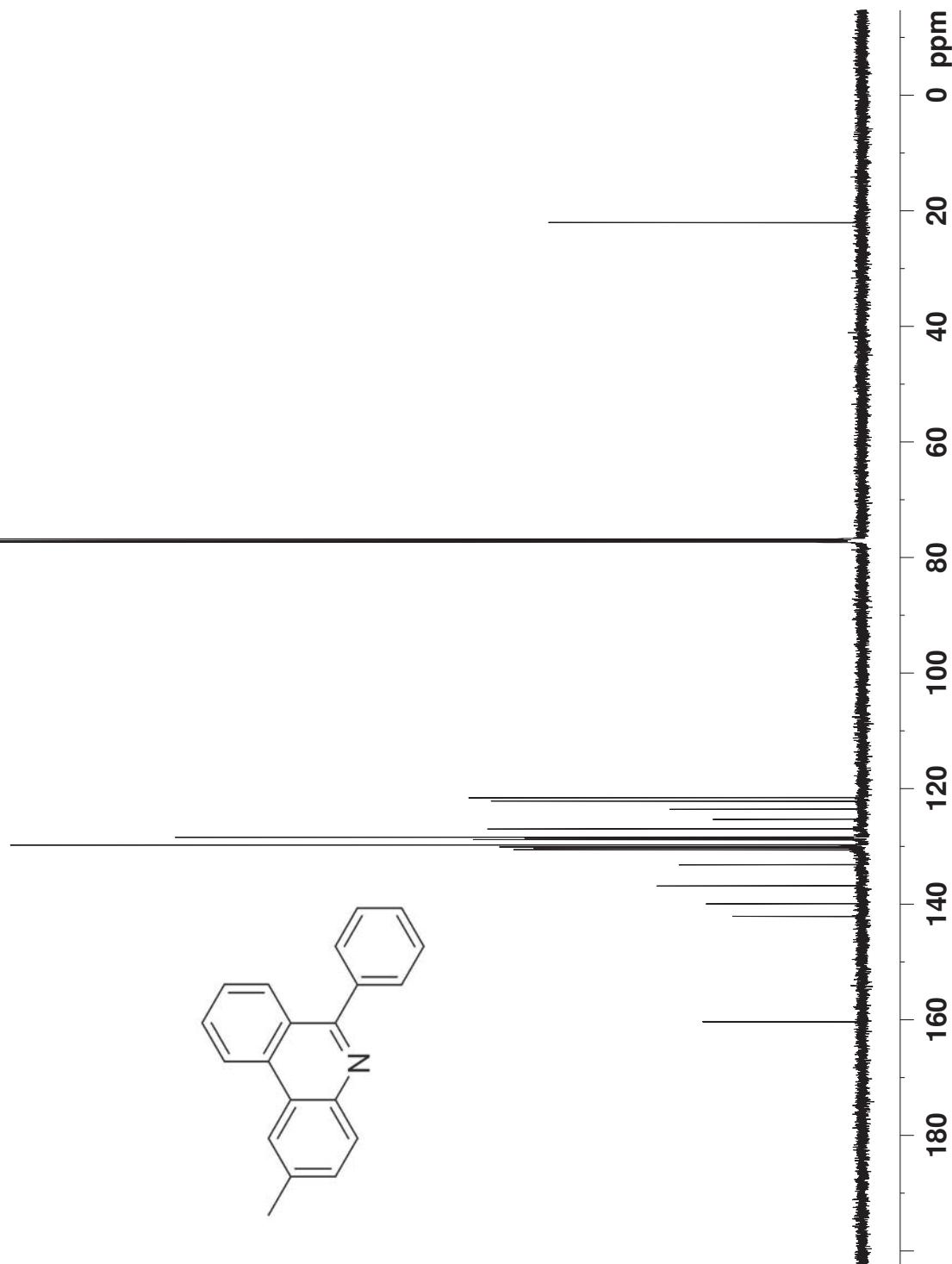
S132

MH-3-124
C13CPD CDC13

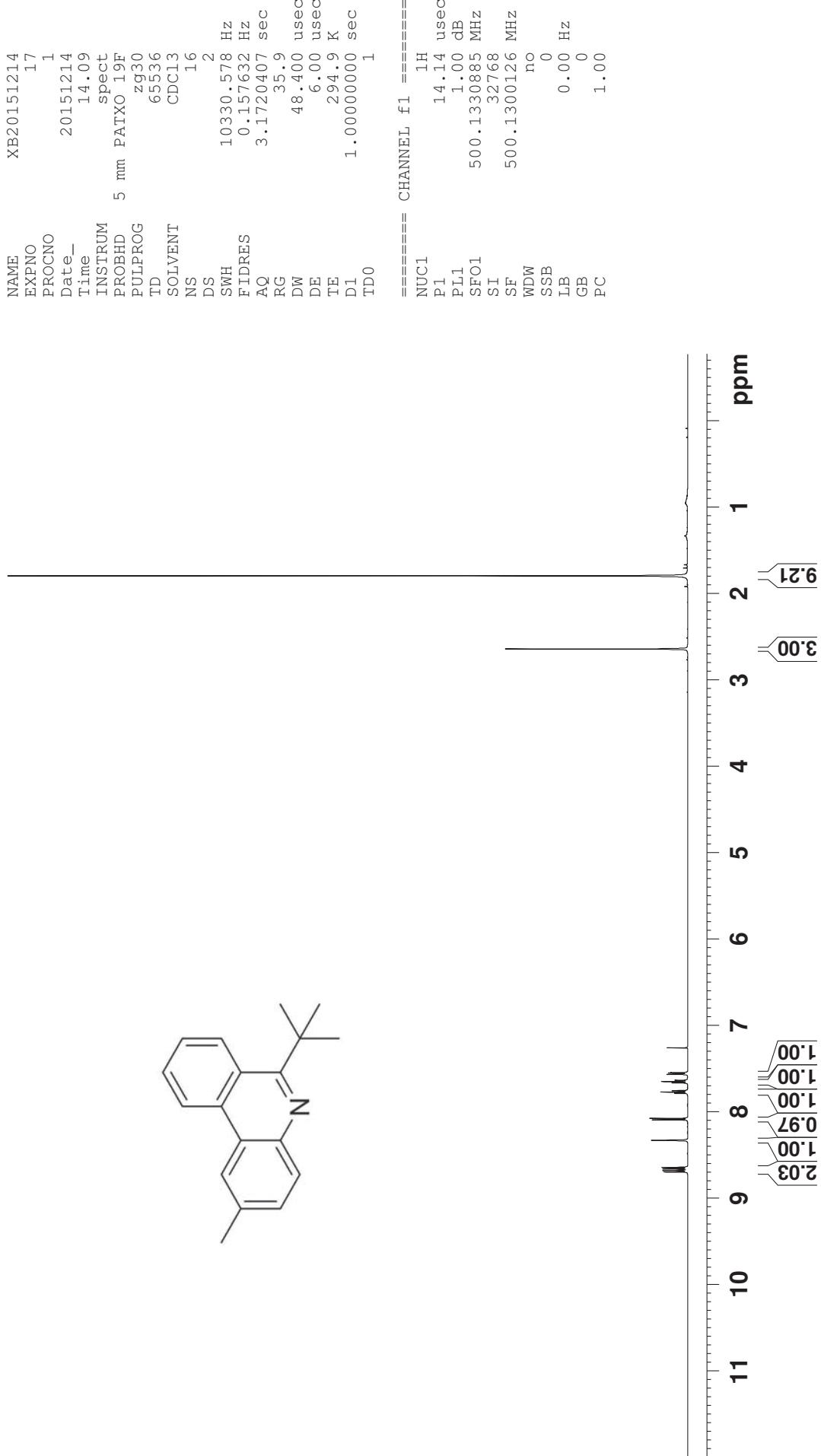
— 22.05 —

160.32
142.13
139.93
136.83
133.19
130.59
130.33
130.09
129.78
128.84
128.61
126.97
125.31
123.58
122.17
121.58

===== NAME xb20151124
EXPNO 22
PROCNO 1
Date 20151124
Time 3.41
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 114
DW 16.650 usec
DE 6.00 usec
TE 296.2 K
D1 2.0000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TDO 1
===== CHANNEL f1 ======
NUC1 13C
P1 9.50 usec
PL1 -0.50 QB
SFO1 125.7703643 MHz
===== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.05 dB
PL13 16.50 QB
SFO2 500.1320005 MHz
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



MH-5d
PROTON CDC13



MH-5d
C13CPD CDC13

NAME XB20151214
EXPNO 18
PROCNO 1
Date_ 20151214
Time 14.24
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zppg30
TD 65536
SOLVENT CDCl3
NS 276
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 161.3
DW 16.650 usec
DE 6.00 usec
TE 296.2 K
D1 2.0000000 sec
d1 0.03000000 sec
DELTA 1.89999998 sec
T0 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.50 usec
PL1 -0.50 dB
SF01 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.05 dB
PL13 16.50 dB
SF02 500.1320005 MHz
SI 327.68 MHz
SF 125.7577890 MHz
WDW EM
SSB 0 Hz
LB 1.00 Hz
GB 0
PC 1.40

22.05
31.30
40.14

121.32
122.99
123.27
124.44
125.81
128.24
129.06
130.10
130.11
133.86
136.19
141.34

165.64

