

Supporting Information

Rhodium-Catalyzed Borylative Cyclization of 2-Alkynylaryl Isocyanates with Bis(pinacolato)diboron

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General. All reactions were carried out with standard Schlenk techniques under an argon atmosphere. ^1H and ^{13}C NMR spectra were recorded on a Varian Gemini 2000 (^1H at 300.07 MHz and ^{13}C at 75.46 MHz) spectrometer. All NMR data were obtained in CDCl_3 . Proton chemical shifts were referenced to the residual proton signal of the solvent at 7.26 ppm. Carbon chemical shifts were referenced to the carbon signal of the solvent at 77.0 ppm. High-resolution mass spectra were recorded on a JEOL JMS-SX102A spectrometer. Infrared spectra were recorded on a Shimadzu FTIR-8100 spectrometer. Column chromatography was performed with Florisil[®] (60-100 mesh) (Wako). Preparative thin-layer chromatography was performed with silica 60 PF_{254} (Merck).

Materials. Anhydrous 1,2-dichloroethane was freshly distilled from calcium hydride. Bis(pinacolato)diboron (**2**) was purchased from Wako Pure Chemical Industries, Ltd and used as received. All other commercially available resources were used without further purification. $[\text{Rh}(\text{OH})(\text{cod})]_2$ ¹ and $[\text{Rh}(\text{cod})_2]\text{SbF}_6$ ² were prepared according to the reported procedure. 2-(Alkynyl)anilines were prepared by Sonogashira reaction³ of the corresponding 2-iodoaniline derivatives⁴ with alkyne. 2-(Alkynyl)aryl isocyanates were synthesized from the corresponding 2-(alkynyl)aniline according to the reported procedure.⁵ The analytical data of compounds **1a**, **1b**, **1c**, **1d**, **1e**, **1f**, **1j**, **1k**, **1l**, and **5** have been already reported.⁶

1g: IR (KBr): 2267, 1607, 1514, 1250, 1030 cm^{-1} ; ^1H NMR: δ = 3.83 (s, 3H), 6.87–6.94 (m, 2H), 7.06 (dd, J = 8.4, 1.5 Hz, 1H), 7.16 (td, J = 7.8, 1.5 Hz, 1H), 7.25 (td, J = 7.5, 1.8 Hz, 1H), 7.51 (dd, J = 7.5, 1.8 Hz, 1H), 7.54–7.61 (m, 2H); ^{13}C NMR: δ = 55.1, 83.6, 97.6, 113.9, 114.3, 121.2, 123.3, 125.2, 127.2, 128.7, 131.8, 132.8, 134.3, 159.9; HRMS (EI^+): Calcd for $\text{C}_{16}\text{H}_{11}\text{NO}_2$, M^+ 249.0790. Found m/z 249.0789.

1h: IR (KBr): 2251, 1595, 1509, 1487, 1071 cm^{-1} ; ^1H NMR: δ = 7.07 (dd, J = 7.8, 1.2 Hz, 1H), 7.18 (td, J = 7.5, 1.2 Hz, 1H), 7.29 (td, J = 7.5, 1.5 Hz, 1H), 7.44–7.54 (m, 5H); ^{13}C NMR: δ = 85.8, 96.3, 120.6, 121.3, 123.2, 123.6, 125.5, 127.1, 129.5, 131.6, 132.2, 132.8, 134.7; HRMS (EI^+): Calcd for $\text{C}_{15}\text{H}_8\text{BrNO}$, M^+ 296.9789. Found m/z 296.9785.

1i: IR (KBr): 2276, 1595, 1503, 1418, 1080 cm^{-1} ; ^1H NMR: δ = 7.06 (dd, J = 8.1, 1.2 Hz, 1H), 7.17 (td, J = 7.7, 0.9 Hz, 1H), 7.22–7.36 (m, 3H), 7.50 (dd, J = 7.5, 1.8 Hz, 1H), 7.66 (dd, J = 3.0, 1.2 Hz, 1H); ^{13}C NMR: δ = 84.4, 92.9, 120.7, 121.2, 123.3, 125.2, 125.3, 127.2, 128.9, 129.3, 129.4, 131.7, 134.5; HRMS (EI^+): Calcd for $\text{C}_{13}\text{H}_7\text{NOS}$, M^+ 225.0248. Found m/z 225.0247.

¹ Uson, R.; Oro, L. A.; Cabeza, J. A. *Inorg. Synth.* **1985**, 23, 126.

² RajanBabu, T. V.; Ayers, T. A.; Halliday, G. A.; You, K. K.; Calabrese, J. C. *J. Org. Chem.* **1997**, 62, 6012.

³ Kamijo, S.; Yamamoto, Y. *Angew. Chem. Int. Ed.* **2002**, 41, 3230.

⁴ Trost, B. M.; McClory, A. *Angew. Chem. Int. Ed.* **2007**, 46, 2074.

⁵ Li, H.; Yang, H.; Petersen, J. L.; Wang, K. K. *J. Org. Chem.* **2004**, 69, 4500.

⁶ Miura, T.; Takahashi, Y.; Murakami, M. *Org. Lett.* **2007**, 9, 5075.

Typical procedure for the rhodium-catalyzed borylative cyclization with B₂pin₂: To an oven-dried, Ar-purged flask was added [Rh(cod)₂]SbF₆ (3.6 mg, 6.5 μmol), **2** (49.2 mg, 0.193 mmol), and a solution of **1a** (25.8 mg, 0.129 mmol) in DCE (1.5 mL). The reaction mixture was stirred at 80 °C for 6 h, and then quenched with NaOH aq. (0.1 M, 5 mL). The aqueous layer was extracted with diethyl ether (4 x 15 mL). The combined extracts were washed with NaOH aq. (0.1 M, 2 x 10 mL), H₂O (3 x 10 mL), and brine (1 x 10 mL) and dried over MgSO₄. The solvent was removed under reduced pressure and the residue was quickly purified by Florisil[®] column chromatography (dichloromethane to diethyl ether) to give **3a** (30.1 mg, 0.092 mmol, 71%) as a yellow solid: Recrystallization from hexane; IR (KBr): 3204, 1698, 1615, 1466, 1347, 1221 cm⁻¹; ¹H NMR: δ = 0.97 (t, *J* = 7.4 Hz, 3H), 1.39–1.56 (m, 2H), 1.43 (s, 12H), 1.59–1.72 (m, 2H), 2.69–2.79 (m, 2H), 6.81 (d, *J* = 7.8 Hz, 1H), 6.99 (t, *J* = 7.4 Hz, 1H), 7.17 (td, *J* = 7.7, 0.6 Hz, 1H), 7.54 (d, *J* = 7.8 Hz, 1H), 9.10 (br s, 1H); ¹³C NMR: δ = 13.9, 23.1, 24.9, 29.9, 31.8, 83.9, 110.0, 121.7, 122.7, 123.7, 128.4, 132.4, 142.2, 151.9 (br), 170.9; HRMS (CI⁺): Calcd for C₁₉H₂₆BNO₃, M⁺ 327.2006. Found m/z 327.2006.

3b: Recrystallization from hexane; IR (KBr): 3179, 1698, 1615, 1464, 1348, 1221 cm⁻¹; ¹H NMR: δ = 1.28 (t, *J* = 7.7 Hz, 3H), 1.43 (s, 12H), 2.78 (q, *J* = 7.6 Hz, 2H), 6.81 (d, *J* = 7.5 Hz, 1H), 6.99 (td, *J* = 7.7, 1.2 Hz, 1H), 7.17 (td, *J* = 7.8, 1.2 Hz, 1H), 7.54 (d, *J* = 7.8 Hz, 1H), 8.86 (br s, 1H); ¹³C NMR: δ = 12.1, 25.0, 25.3, 84.0, 109.9, 121.9, 122.8, 123.9, 128.5, 132.0, 142.0, 152.8 (br), 170.6; HRMS (EI⁺): Calcd for C₁₇H₂₂BNO₃, M⁺ 299.1693. Found m/z 299.1691.

3c: Recrystallization from hexane; IR (KBr): 3187, 1698, 1613, 1464, 1344, 1223 cm⁻¹; ¹H NMR: δ = 1.07 (t, *J* = 7.2 Hz, 3H), 1.43 (s, 12H), 1.63–1.79 (m, 2H), 2.68–2.76 (m, 2H), 6.81 (d, *J* = 8.1 Hz, 1H), 6.98 (td, *J* = 7.7, 1.2 Hz, 1H), 7.17 (td, *J* = 7.8, 1.2 Hz, 1H), 7.54 (d, *J* = 7.8 Hz, 1H), 9.21 (br s, 1H); ¹³C NMR: δ = 14.7, 21.3, 25.0, 34.3, 84.0, 109.9, 121.9, 122.9, 123.9, 128.5, 132.4, 142.0, 170.6 (The boron-bound carbon was not detected due to the quadrupolar relaxation); HRMS (EI⁺): Calcd for C₁₈H₂₄BNO₃, M⁺ 313.1849. Found m/z 313.1852.

3d: Recrystallization from hexane; IR (KBr): 3179, 1698, 1615, 1464, 1354, 1223 cm⁻¹; ¹H NMR: δ = 1.28 (d, *J* = 7.2 Hz, 6H), 1.45 (s, 12H), 3.52 (septet, *J* = 6.9 Hz, 1H), 6.81 (d, *J* = 7.8 Hz, 1H), 6.98 (td, *J* = 7.7, 1.2 Hz, 1H), 7.16 (td, *J* = 7.8, 1.2 Hz, 1H), 7.57 (d, *J* = 7.5 Hz, 1H), 9.03 (br s, 1H); ¹³C NMR: δ = 21.4, 25.5, 31.0, 84.1, 109.9, 121.8, 122.6, 124.2, 128.5, 131.1, 142.0, 158.2 (br), 170.9; HRMS (EI⁺): Calcd for C₁₈H₂₄BNO₃, M⁺ 313.1849. Found m/z 313.1847.

3e: Recrystallization from hexane; IR (KBr): 3216, 1700, 1593, 1464, 1312, 1142 cm⁻¹; ¹H NMR: δ = 1.39 (s, 6H), 1.46 (s, 9H), 1.50 (s, 6H), 6.84 (dd, *J* = 7.5, 0.6 Hz, 1H), 7.01 (td, *J* = 7.7, 1.1 Hz, 1H), 7.16 (td, *J* = 7.7, 1.2 Hz, 1H), 7.72 (d, *J* = 7.8 Hz, 1H), 8.74 (br s, 1H); ¹³C NMR: δ = 25.7, 26.5, 28.9, 35.4, 83.7, 110.1, 121.2, 121.6, 127.8, 128.4, 132.0, 142.6, 164.9 (br), 171.7; HRMS (EI⁺): Calcd for C₁₉H₂₆BNO₃, M⁺ 327.2006. Found m/z 327.2006.

3f: Recrystallization from toluene; IR (KBr): 3204, 1709, 1613, 1466, 1343, 1215 cm⁻¹; ¹H NMR: δ = 1.38 (s, 12H), 6.72 (t, *J* = 7.7 Hz, 1H), 6.80 (d, *J* = 7.8 Hz, 1H), 7.01 (d, *J* = 7.5 Hz, 1H), 7.12 (t, *J* = 7.7 Hz, 1H), 7.34–7.54 (m, 5H), 9.05 (br s, 1H); ¹³C NMR: δ = 24.8, 84.3, 110.0, 121.7, 121.9, 123.4, 127.3, 128.3, 128.7, 129.4, 132.7, 137.7, 142.4, 147.9 (br), 170.8; HRMS (EI⁺): Calcd for C₂₁H₂₂BNO₃, M⁺ 347.1693. Found m/z 347.1692.

3g: Recrystallization from toluene; IR (KBr): 3169, 1690, 1577, 1464, 1345, 1250 cm⁻¹; ¹H NMR: δ = 1.39 (s, 12H), 3.87 (s, 3H), 6.75 (td, *J* = 7.8, 0.9 Hz, 1H), 6.80 (d, *J* = 7.5 Hz, 1H), 6.94–7.01 (m, 2H), 7.13 (td, *J* = 7.5, 1.2 Hz, 1H), 7.21 (d, *J* = 7.5 Hz, 1H), 7.44–7.52 (m, 2H), 8.76 (br s, 1H); ¹³C NMR: δ = 24.9, 55.3, 84.2, 110.0, 114.0, 121.5, 122.0, 123.0, 129.1, 129.2, 129.8, 131.8, 142.4, 147.9 (br), 159.8, 171.2; HRMS (EI⁺): Calcd for C₂₂H₂₄BNO₄, M⁺ 377.1798. Found m/z 377.1799.

3h: Recrystallization from toluene; IR (KBr): 3179, 1694, 1607, 1464, 1343, 1142 cm^{-1} ; ^1H NMR: δ = 1.37 (s, 12H), 6.76 (td, J = 7.8, 0.9 Hz, 1H), 6.80 (d, J = 7.8 Hz, 1H), 7.01 (d, J = 7.5 Hz, 1H), 7.15 (td, J = 7.8, 1.2 Hz, 1H), 7.34–7.41 (m, 2H), 7.55–7.61 (m, 2H), 8.58 (br s, 1H); ^{13}C NMR: δ = 24.8, 84.4, 110.2, 121.6, 121.8, 122.5, 123.4, 129.1, 129.8, 131.9, 133.0, 136.6, 142.5, 146.2 (br), 170.7; HRMS (EI^+): Calcd for $\text{C}_{21}\text{H}_{21}\text{BBrNO}_3$, M^+ 425.0798. Found m/z 425.0798.

3i: Recrystallization from toluene; IR (KBr): 3187, 1698, 1605, 1464, 1323, 1140 cm^{-1} ; ^1H NMR: δ = 1.41 (s, 12H), 6.77–6.85 (m, 2H), 7.15 (td, J = 7.8, 0.9 Hz, 1H), 7.28 (dd, J = 5.4, 1.4 Hz, 1H), 7.37 (d, J = 8.1 Hz, 1H), 7.43 (dd, J = 4.8, 3.0 Hz, 1H), 7.51 (dd, J = 3.0, 1.2 Hz, 1H), 9.12 (br s, 1H); ^{13}C NMR: δ = 24.9, 84.2, 110.1, 121.7, 121.9, 123.2, 124.5, 126.1, 127.5, 129.4, 132.4, 137.7, 142.0 (br), 142.4, 171.2; HRMS (EI^+): Calcd for $\text{C}_{19}\text{H}_{20}\text{BNO}_3\text{S}$, M^+ 353.1257. Found m/z 353.1262.

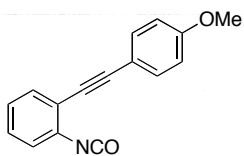
3j: Recrystallization from hexane/dichloromethane; IR (KBr): 3173, 1703, 1617, 1474, 1345, 1129 cm^{-1} ; ^1H NMR: δ = 0.98 (t, J = 7.4 Hz, 3H), 1.42 (s, 12H), 1.42–1.56 (m, 2H), 1.59–1.72 (m, 2H), 2.67–2.75 (m, 2H), 6.73 (d, J = 8.1 Hz, 1H), 7.15 (dd, J = 8.1, 2.1 Hz, 1H), 7.49 (d, J = 1.8 Hz, 1H), 9.19 (br s, 1H); ^{13}C NMR: δ = 14.0, 23.1, 25.0, 29.9, 32.0, 84.2, 110.8, 124.0, 124.1, 127.1, 128.3, 131.5, 140.4, 154.4 (br), 170.5; HRMS (EI^+): Calcd for $\text{C}_{19}\text{H}_{25}\text{BClNO}_3$, M^+ 361.1616. Found m/z 361.1623.

3k: Recrystallization from hexane/dichloromethane; IR (KBr): 3179, 1700, 1628, 1480, 1345, 1206 cm^{-1} ; ^1H NMR: δ = 0.97 (t, J = 7.4 Hz, 3H), 1.41 (s, 12H), 1.41–1.56 (m, 2H), 1.59–1.71 (m, 2H), 2.67–2.75 (m, 2H), 3.79 (s, 3H), 6.68–6.77 (m, 2H), 7.13–7.17 (m, 1H), 8.02 (br s, 1H); ^{13}C NMR: δ = 14.0, 23.2, 25.0, 29.9, 31.8, 55.8, 83.9, 109.9, 111.4, 112.8, 123.7, 132.6, 136.0, 152.4 (br), 155.0, 170.8; HRMS (EI^+): Calcd for $\text{C}_{20}\text{H}_{28}\text{BNO}_4$, M^+ 357.2111. Found m/z 357.2108.

3l: Recrystallization from hexane/dichloromethane; IR (KBr): 3158, 1701, 1615, 1478, 1341, 1254 cm^{-1} ; ^1H NMR: δ = 1.00 (t, J = 7.4 Hz, 3H), 1.39 (t, J = 7.1 Hz, 3H), 1.43 (s, 12H), 1.46–1.59 (m, 2H), 1.61–1.74 (m, 2H), 2.74–2.85 (m, 2H), 4.36 (q, J = 7.2 Hz, 2H), 6.85 (d, J = 8.1 Hz, 1H), 7.95 (dd, J = 8.4, 1.5 Hz, 1H), 8.24 (s, 1H), 9.30 (br s, 1H); ^{13}C NMR: δ = 13.9, 14.4, 23.1, 25.0, 29.8, 32.0, 60.9, 84.2, 109.4, 122.6, 124.3, 125.2, 130.9, 131.3, 145.7, 154.3 (br), 166.3, 171.0; HRMS (EI^+): Calcd for $\text{C}_{22}\text{H}_{30}\text{BNO}_5$, M^+ 399.2217. Found m/z 399.2212.

The Suzuki-Miyaura cross-coupling reaction with iodobenzene: To an oven-dried, N_2 -purged flask was added **3a** (18.0 mg, 0.0550 mmol), K_2CO_3 (22.8 mg, 0.165 mmol), **4** (33.7 mg, 0.165 mmol), H_2O (0.1 mL), and a solution of $\text{Pd}[\text{P}(t\text{-Bu})_3]_2$ (1.4 mg, 2.7 μmol) in 1,4-dioxane (1.0 mL). The reaction mixture was stirred at 100 $^\circ\text{C}$ for 2 h, and then quenched with H_2O (2 mL). The aqueous layer was extracted with ethyl acetate (4 x 15 mL). The combined extracts were washed with brine and dried over MgSO_4 . The solvent was removed under reduced pressure and the residue was purified by preparative thin-layer chromatography (chloroform/ethyl acetate = 10:1) to give **5** (13.0 mg, 0.0469 mmol, 85%) as a yellow solid.

The bromination reaction with copper(II) bromide: To an oven-dried, N_2 -purged flask was added **3a** (16.1 mg, 0.0492 mmol), EtOH (1.0 mL), and a solution of CuBr_2 (**6**, 55.0 mg, 0.246 mmol) in H_2O (1.0 mL). The reaction mixture was stirred at 80 $^\circ\text{C}$ for 12 h, and then quenched with H_2O (2 mL). The aqueous layer was extracted with ethyl acetate (4 x 15 mL). The combined extracts were washed with brine and dried over MgSO_4 . The solvent was removed under reduced pressure and the residue was purified by preparative thin-layer chromatography (chloroform/ethyl acetate = 10:1) to give **7** (13.2 mg, 0.0471 mmol, 96%, E/Z = 1:20) as a yellow solid: IR (KBr): 3335, 2959, 1709, 1684, 1605, 1470, 1192 cm^{-1} ; ^1H NMR: δ = 0.99 (t, J = 7.2 Hz, 3H), 1.44–1.60 (m, 2H), 1.70–1.85 (m, 2H), 3.14–3.23 (m, 2H), 6.89–6.95 (m, 1H), 7.01 (td, J = 7.8, 1.1 Hz, 1H), 7.25 (td, J = 7.8, 1.1 Hz, 1H), 7.45 (d, J = 7.8 Hz, 1H), 8.76 (br s, 1H); ^{13}C NMR: δ = 14.0, 22.4, 29.7, 42.3, 109.9, 122.0, 122.4, 123.3, 125.2, 129.1, 138.8, 141.1, 167.2; HRMS (EI^+): Calcd for $\text{C}_{13}\text{H}_{14}\text{BrNO}$, M^+ 279.0259. Found m/z 279.0251.



1g

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient Temperature

GBMIM-300BB "varian2"

Relax. delay 1.502 sec

Pulse 45.0 degrees

Acq. time 3.200 sec

Width 5000.0 Hz

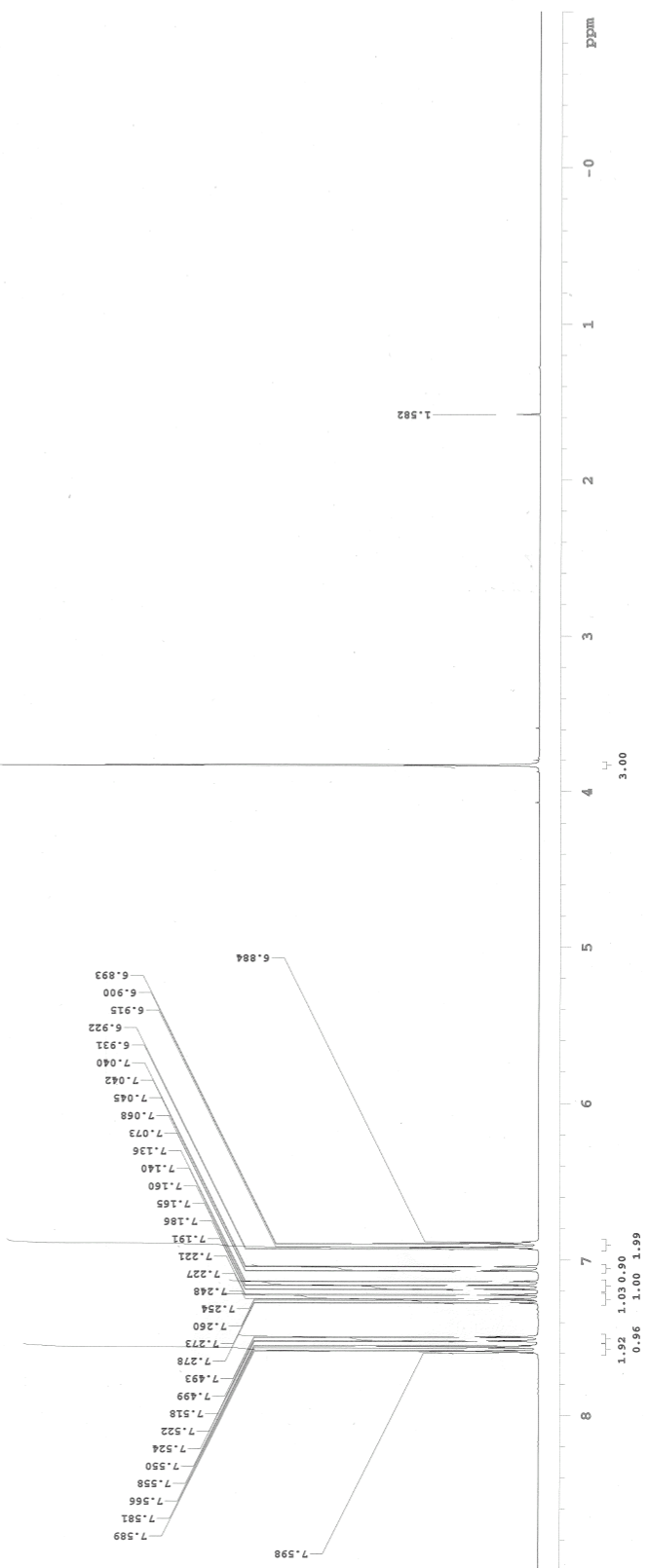
16 Repetitions

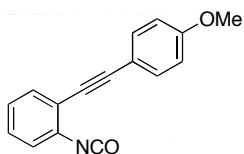
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DATA PROCESSING

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Total time 1 min, 24 sec

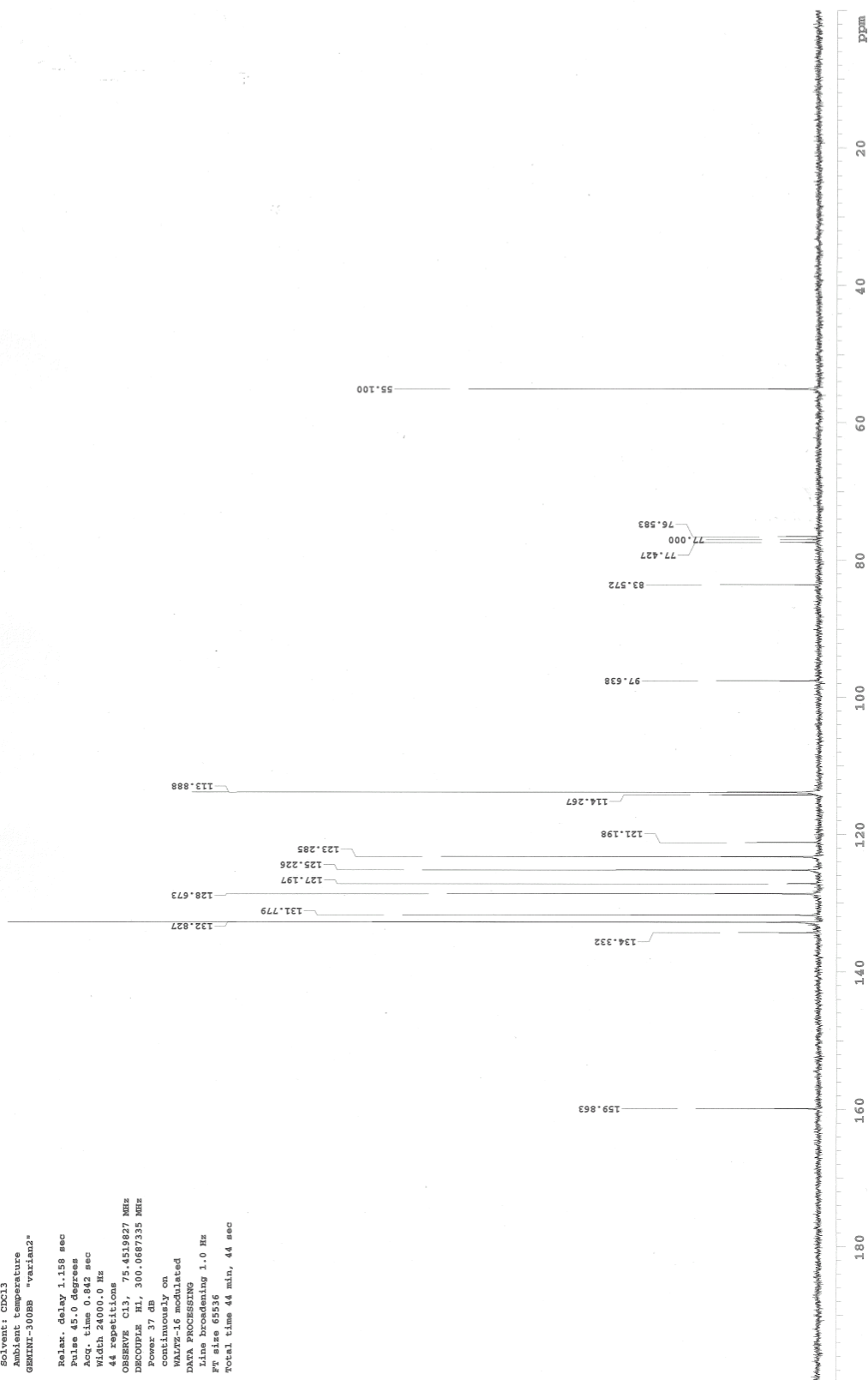


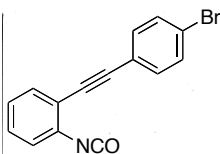


1g

13C OBSERVE

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 Ambient temperature
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 Acq. time 0.842 sec
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 44 Repetitions
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 DECOUPLE H1, 300.0697335 MHz
 Power 37 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 FINE DECODING 1.0 Hz
 F2 size 6528
 F2 time 44 min, 44 sec
 Total time 44 min, 44 sec





1h

STANDARD 1H OBSERVE

Pulse sequence: s2pul

Solvent: CDCl3

Ambient temperature

QNP1H-300MHz "varian2"

Relax. delay 1.502 sec

Pulse 45.0 degrees

Acq. time 3.200 sec

Width 5000 Hz

487.483

16 repetitions

OBSERVE H1, 300.10672328 MHz

DATA PROCESSING

FT size 32768

Total time 1 min, 24 sec

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7.179
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7.085
7.081
7.059
7.056

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7.270
7.291
7.296
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7.475
7.483

1.544

ppm

-0

1

2

3

4

5

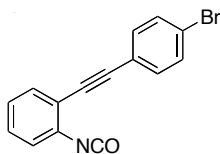
6

7

8

5.26 1.12

1.08 1.00



1h

13C OBSERVE

Pulse Sequence: zgpg30

Solvent: CDCl3

Ambient temperature

QNP-1300B8 "vian2"

Relax. delay 1.158 sec

Pulse 45.0 degrees

Acq. time 0.842 sec

Width 24000.0 Hz

12200 repetitions

OBSERVE C13, 75.4519696 MHz

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Power 37 dB

NUC13C13

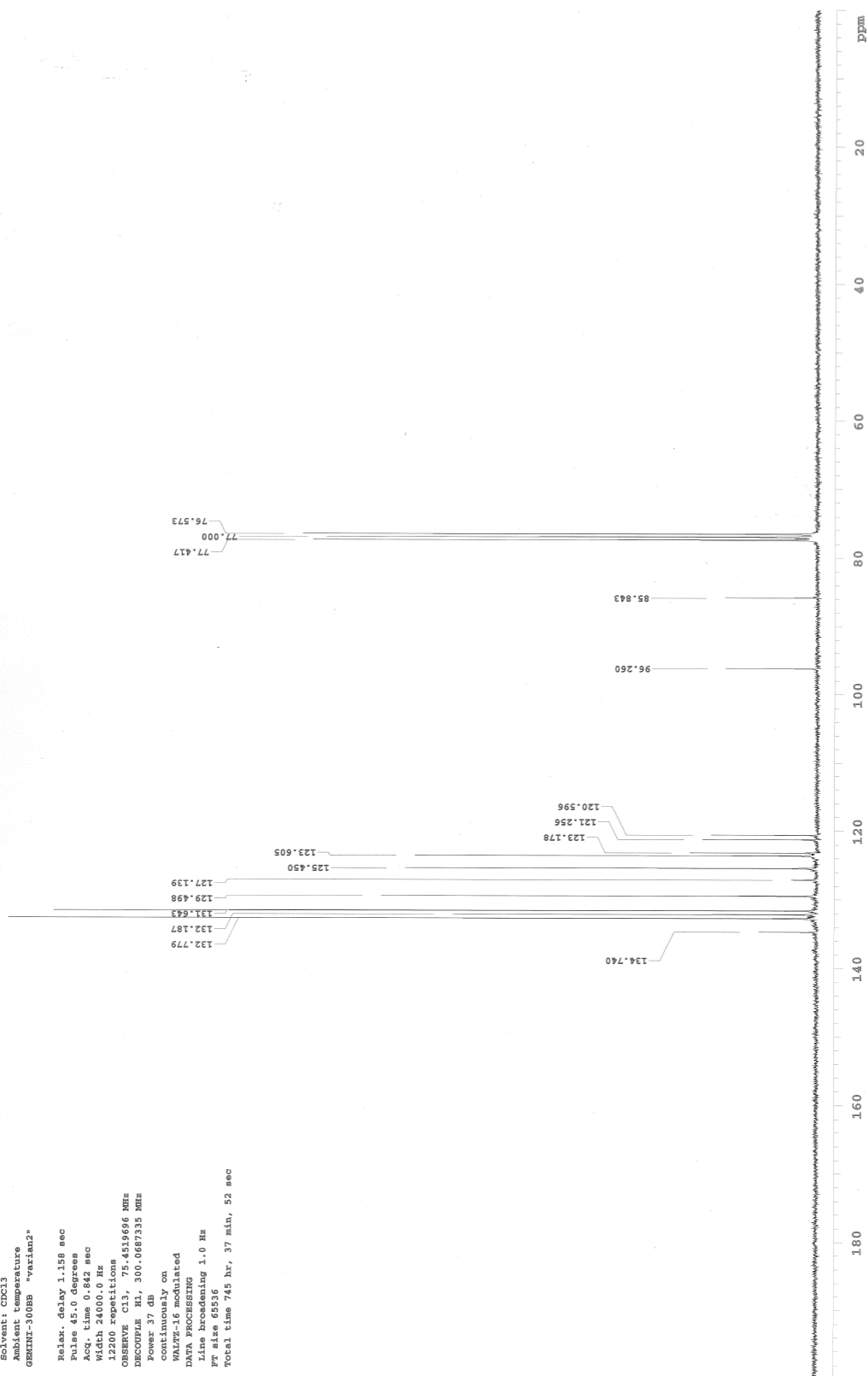
NUC13C13

DATA PROCESSING

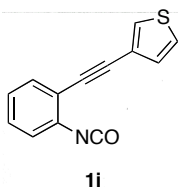
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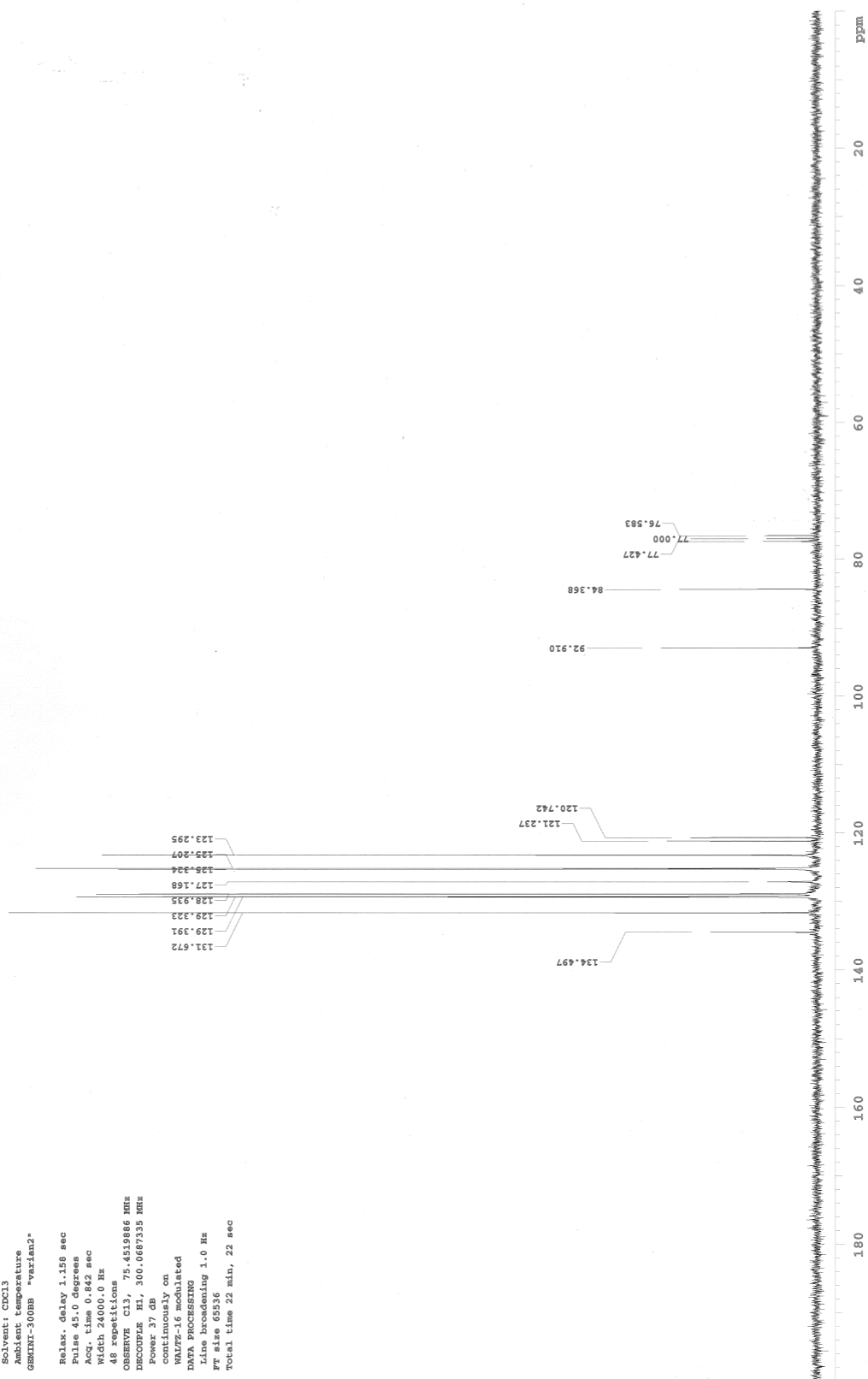


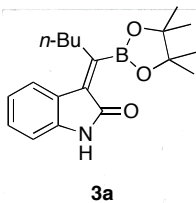




13C OBSERVE

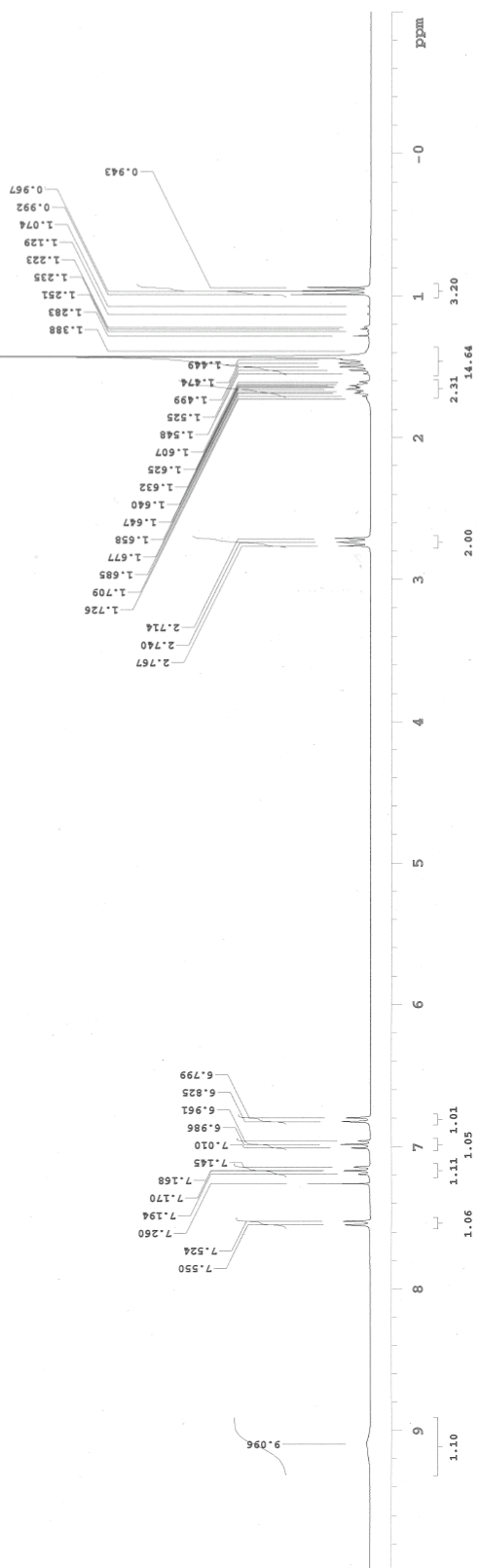
Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 GEMINI-300DB "varian2"
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 Pulse 45.0 degrees
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 Power 37 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
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 Total time 22 min, 22 sec

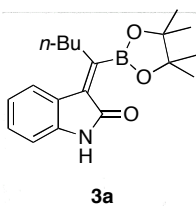




STANDARD IN OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 GEMINI-300DB "variant2"
 Relax. delay 1.502 sec
 Pulse 45.0 degrees
 Acq. time 3.200 sec
 Width 5000.0 Hz
 16 repetitions
 OBSERVE M1, 300.0672331 MHz
 DATA PROCESSING
 F1 size 32768
 Total time 1 min, 24 sec





13C OBSERVE

Pulse sequence: s2pul

Solvent: CDCl3

Ambient temperature

QNP1H-300BB "variant2"

Relax. delay 1.158 sec

Pulse 45.0 degrees

Acq. time 0.842 sec

Width 24000.0 Hz

84 Repetitions

OBSERVE C13, 75.4519769 MHz

DECOUPLE H1, 300.0687335 MHz

Power 37 dB

continuously on

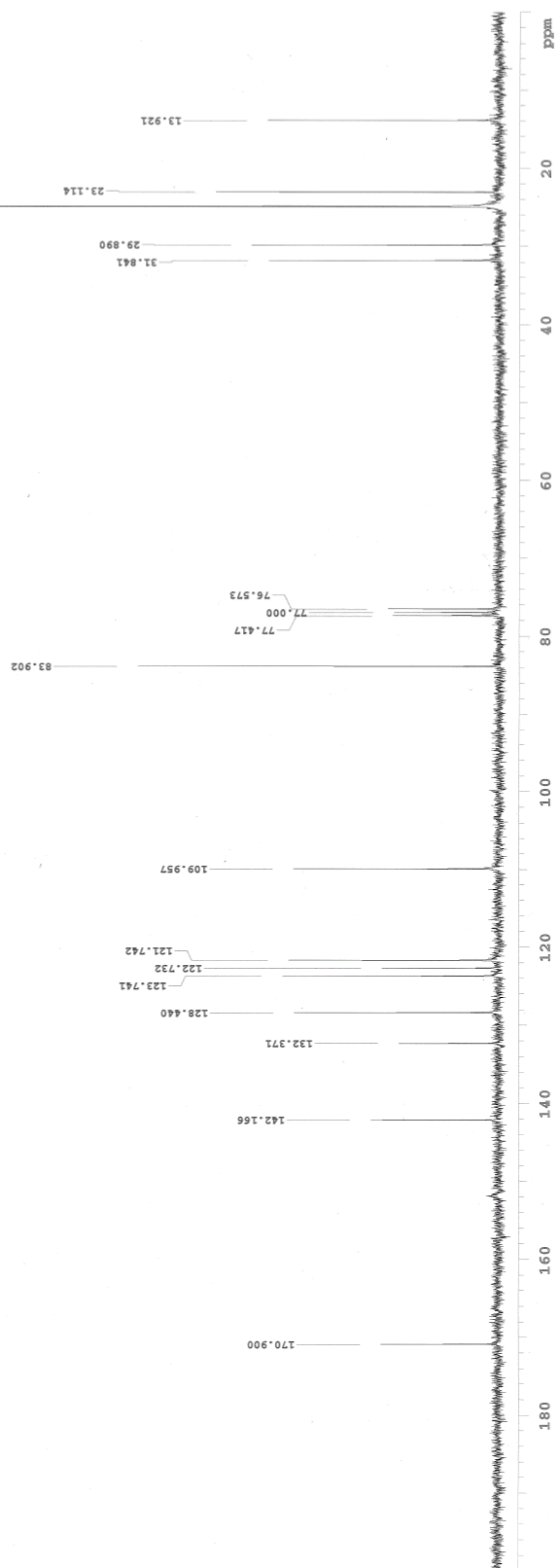
WALTZ-16 modulated

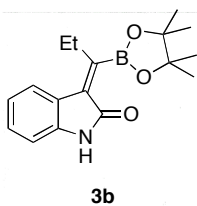
DATA PROCESSING

Acquisition 1.0 Hz

FT size 65536

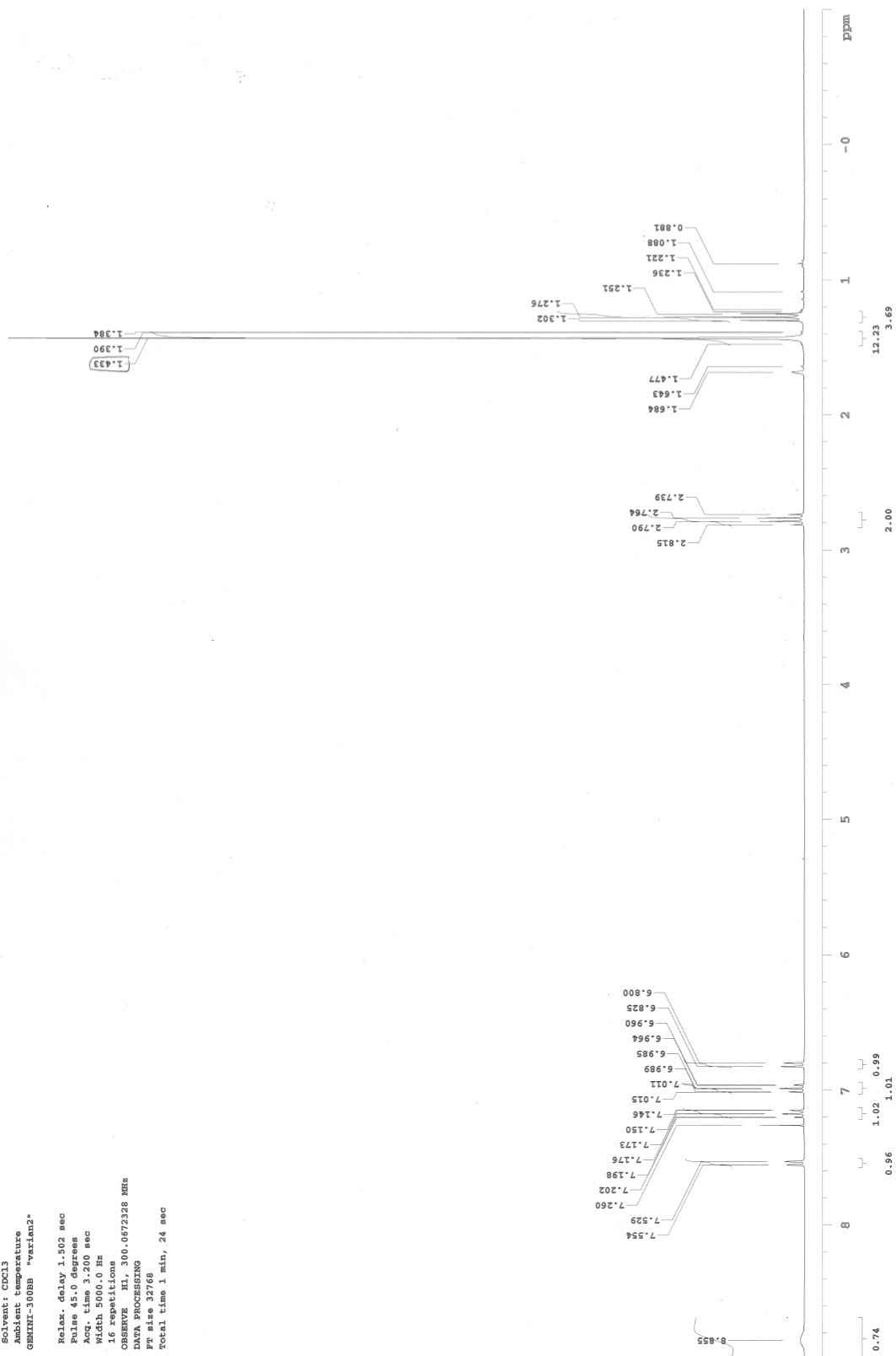
Total time 44 min, 44 sec

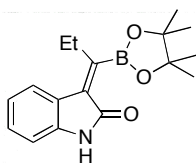




STANDARD IN OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 GEMINI-300BB "varian2"
 Relax. delay 1.502 sec
 Pulse 45.0 degrees
 Acq. time 3.200 sec
 Width 5000.0 Hz
 16 repetitions
 OBSERVE XL 300.0672328 MHz
 DATA PROCESSING
 FT size 32768
 Total time 1 min, 24 sec





3b

13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

GEMINI-300BB "variant2"

Relax. delay 1.158 sec

Pulse 45.0 degrees

Acq. time 0.842 sec

Width 24000.0 Hz

1964 repetitions

OBSERVE C13, 75.4519703 MHz

DECOUPLE H1, 300.0687335 MHz

Power 37 dB

continuously on

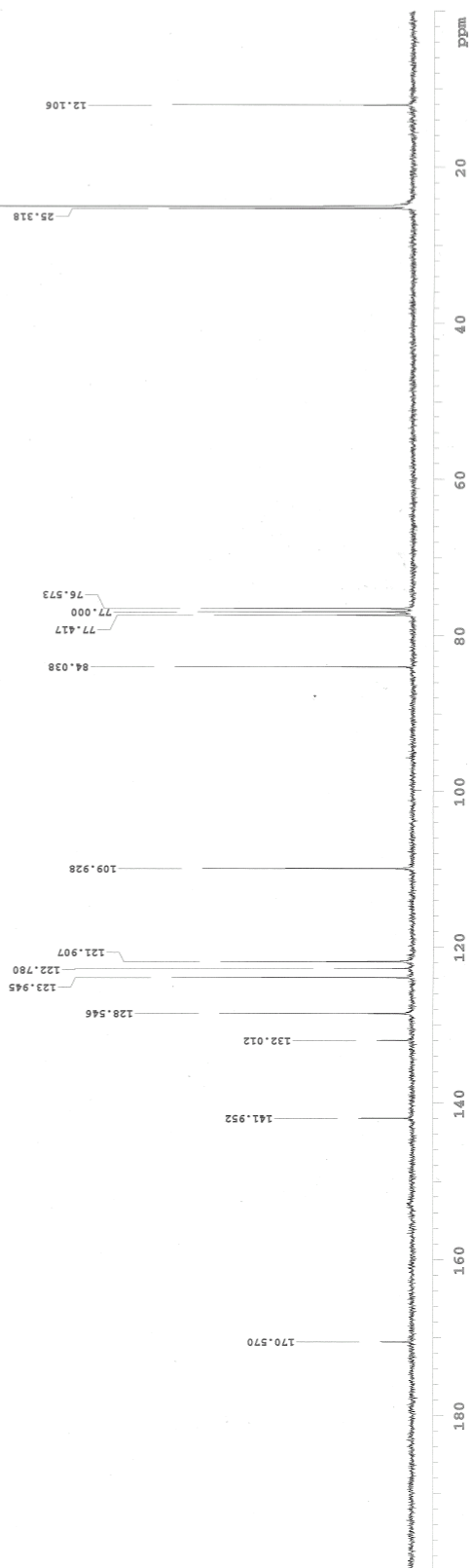
WALTZ-16 modulated

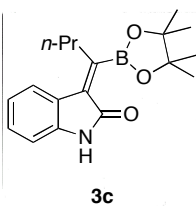
DATA PROCESSING

Acquisition 1.0 Hz

FT date 68536

Total time 37 hr, 16 min, 53 sec





STANDARD IN OBSERVE

Pulse Sequence: #2pul

Solvent: CDCl3

Substance: 3c

Chemical Shift: 300.0672328 MHz

Relax. delay: 1.502 sec

Pulse: 45.0 degrees

Acq. time: 3.200 sec

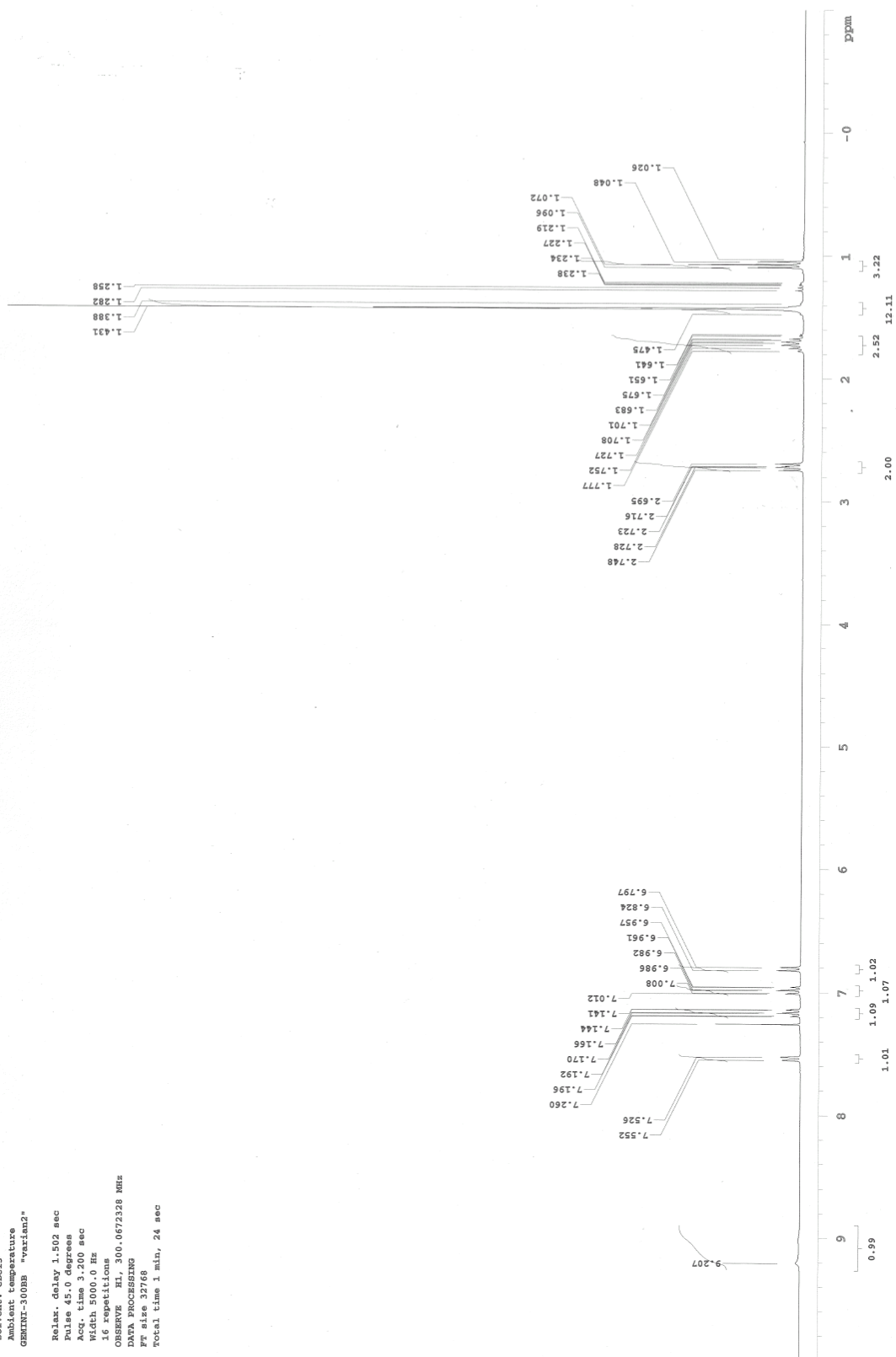
Width: 5000.0 Hz

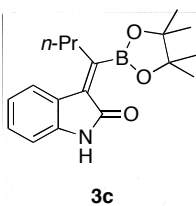
6 repetitions

DATA PROCESSING

PR size: 32768

Total time: 1 min, 24 sec





13C OBSERVE

Pulse Sequence: zgpg30

Solvent: CDCl3

Acquisition Temperature

CEM-300WB "Varian2"

Relax. delay 1.158 sec

Pulse 45.0 degrees

Acq. time 0.842 sec

Width 24000.0 Hz

1288 repetitions

Observed 13C, 75.4519710 MHz

Observed 1H, 300.087335 MHz

Decouple 1H

Power 37 dB

continuously on

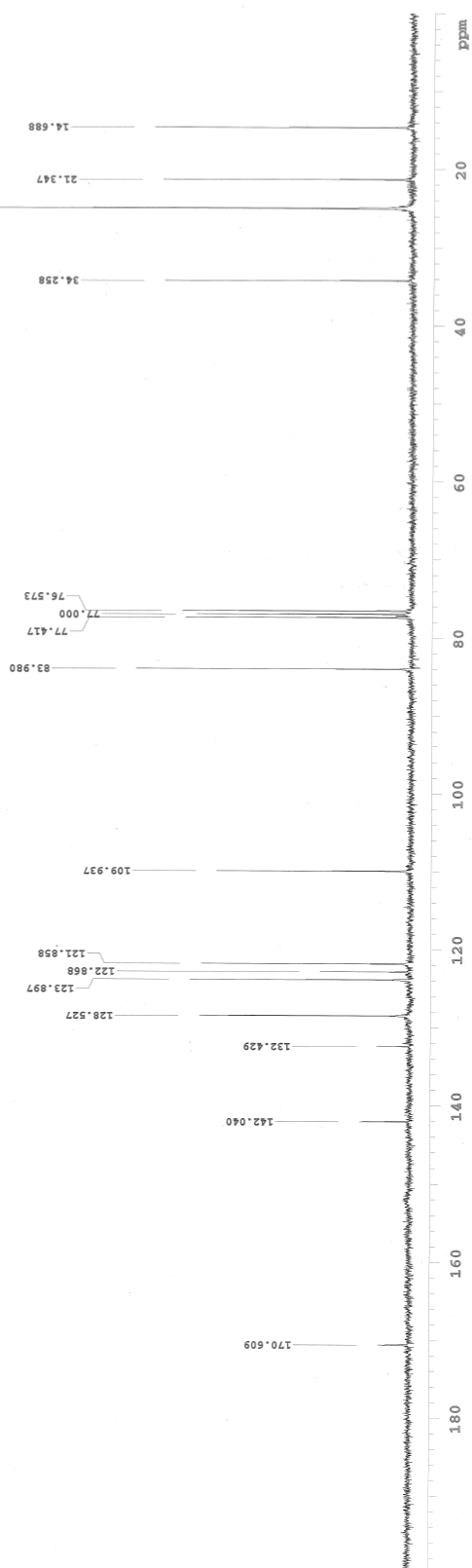
WALTZ-16 modulated

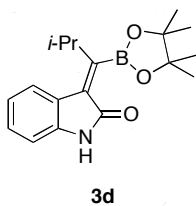
DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

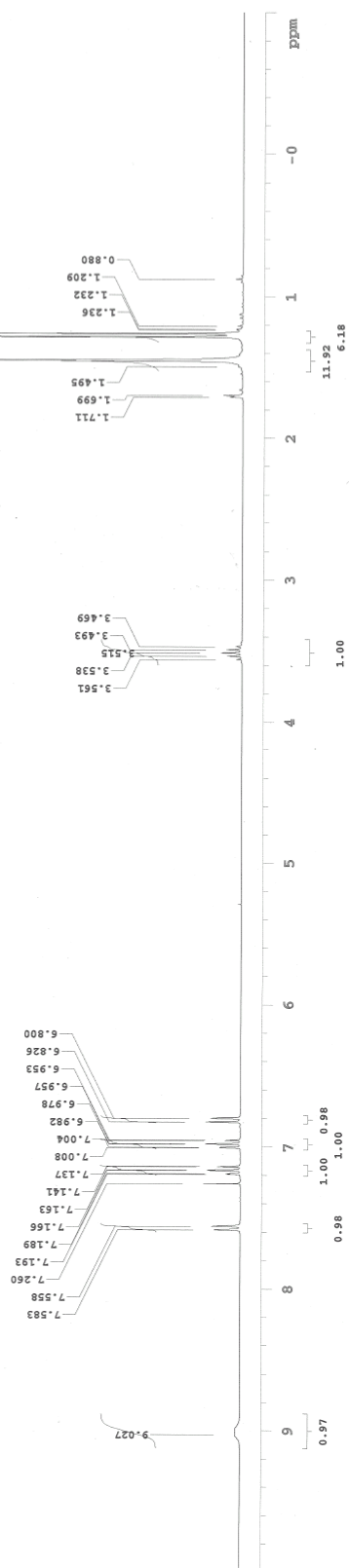
Total time 745 hr, 37 min, 52 sec

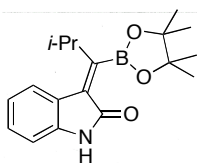




STANDARD IN OBSERVE

Pulse Sequence: a2gn1
 Solvent: CDCl3
 Ambient temperature
 GMSIN-300B5 "varian2"
 Relax. delay 1.502 sec
 Pulse 45.0 degrees
 Acq. time 3.200 sec
 Width 5000.0 Hz
 16 repetitions
 OBSERVE HL 300.0672331 MHz
 DATA PROCESSING
 File 32706
 Total time 1 min, 24 sec

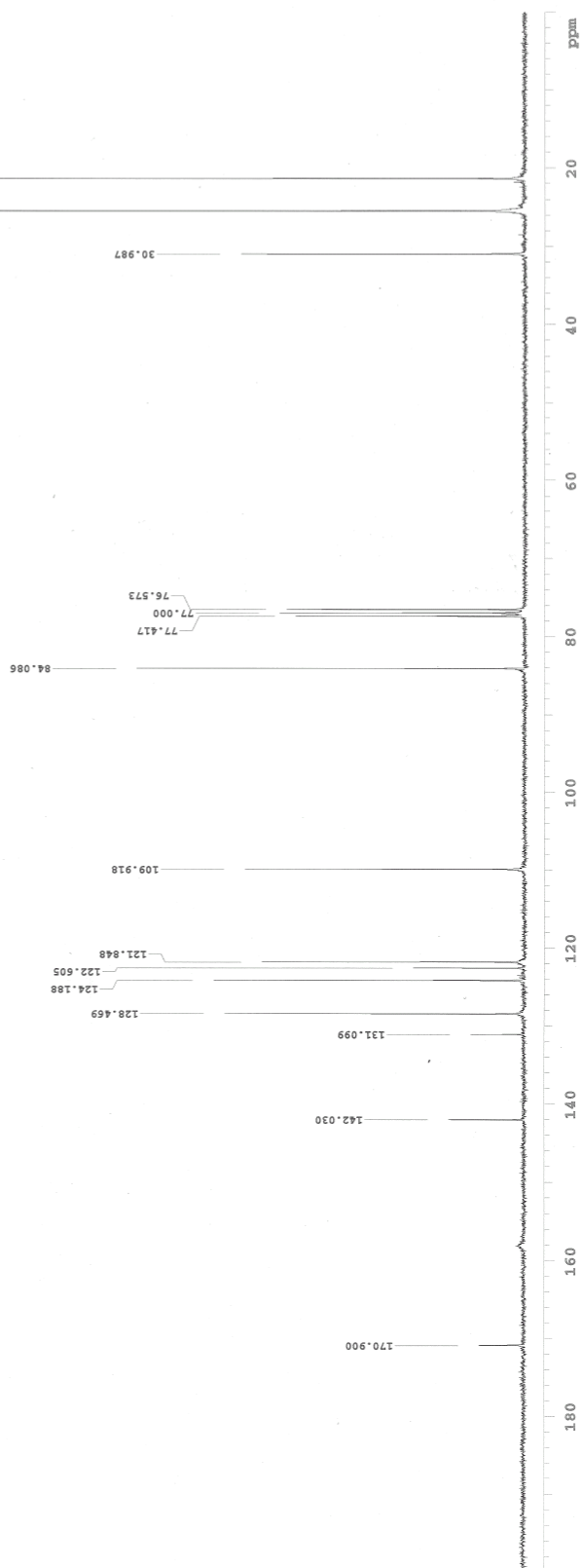


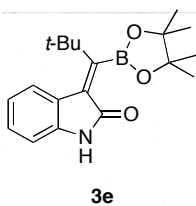


3d

13C OBSERVE

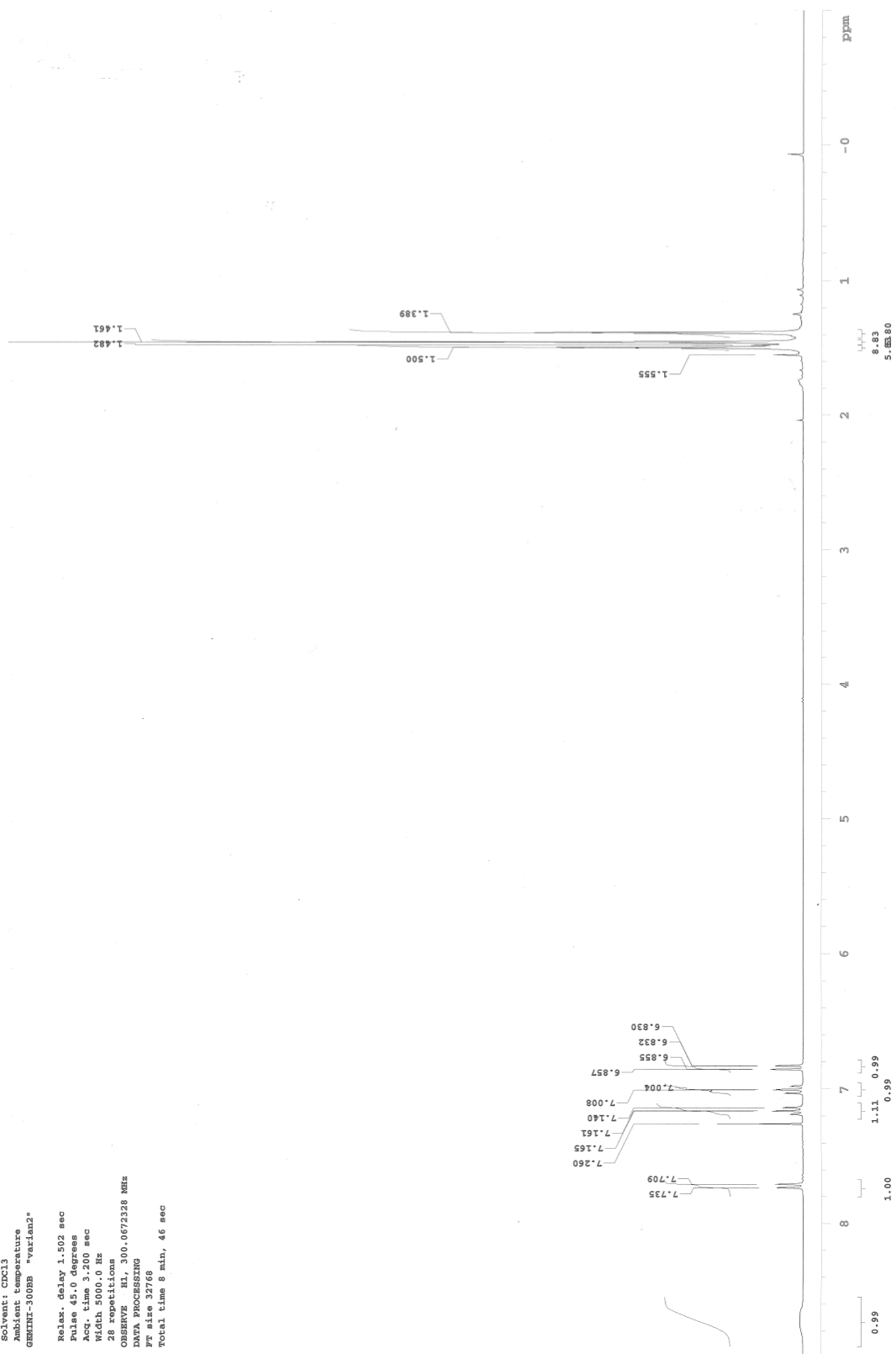
Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 GEMINI-300DB "variant2"
 Relax. delay 1.158 sec
 Pulse 45.0 degrees
 Acq. time 0.842 sec
 Width 24000.0 Hz
 2828 repetitions
 OBSERVE c13, 75.4519710 MHz
 DECOUPLE H1, 300.0667335 MHz
 Power 37 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line processing 1.0 Hz
 FT resolution 0.32 Hz
 Total time 74 hr, 33 min, 47 sec

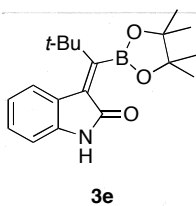




STANDARD 1H OBSERVE

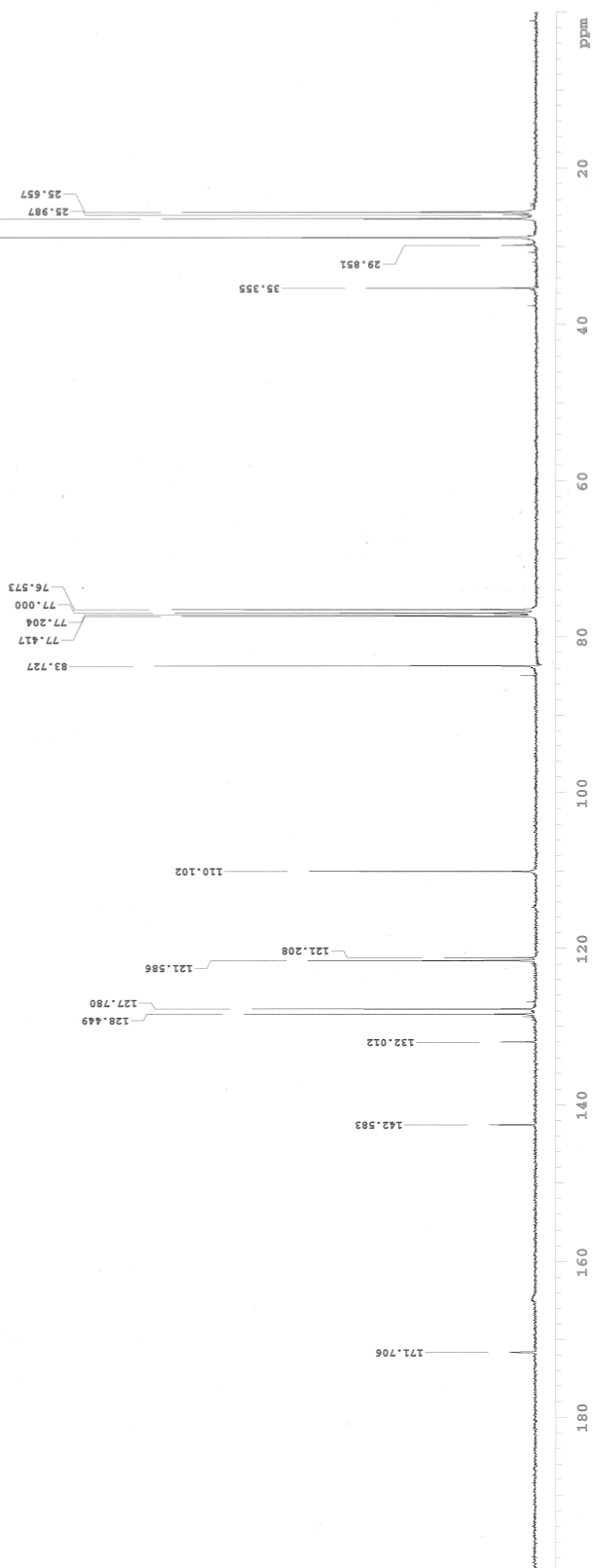
Pulse sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 GEMINI-300B8 "variant2"
 Relax. delay 1.502 sec
 Pulse 45.0 degrees
 Acq. time 3.200 sec
 Width 5000.0 Hz
 28 repetitions
 OBSERVE M1, 300.0672328 MHz
 DATA PROCESSING
 FT size 32768
 Total time 8 min, 46 sec

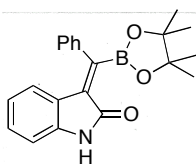




13C OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient Temperature
 GEMIN-300DB "variant2"
 Relax. delay 1.158 sec
 Pulse 45.0 degrees
 Acq. time 0.842 sec
 Width 24000.0 Hz
 13016 repetitions
 OBSERVE C13, 75.4519703 MHz
 DECOUPLE H1, 300.0687335 MHz
 Power 37 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 65536
 Total time 7456 hr, 18 min, 44 sec





3f

STANDARD 1H OBSERVE

Pulse sequence: #2pul

Solvent: CDCl3

Ambient temperature

GEMINI-300BB "variant2"

Relax. delay 1.502 sec

Pulse 45.0 degrees

Acq. time 3.200 sec

Width 5000.0 Hz

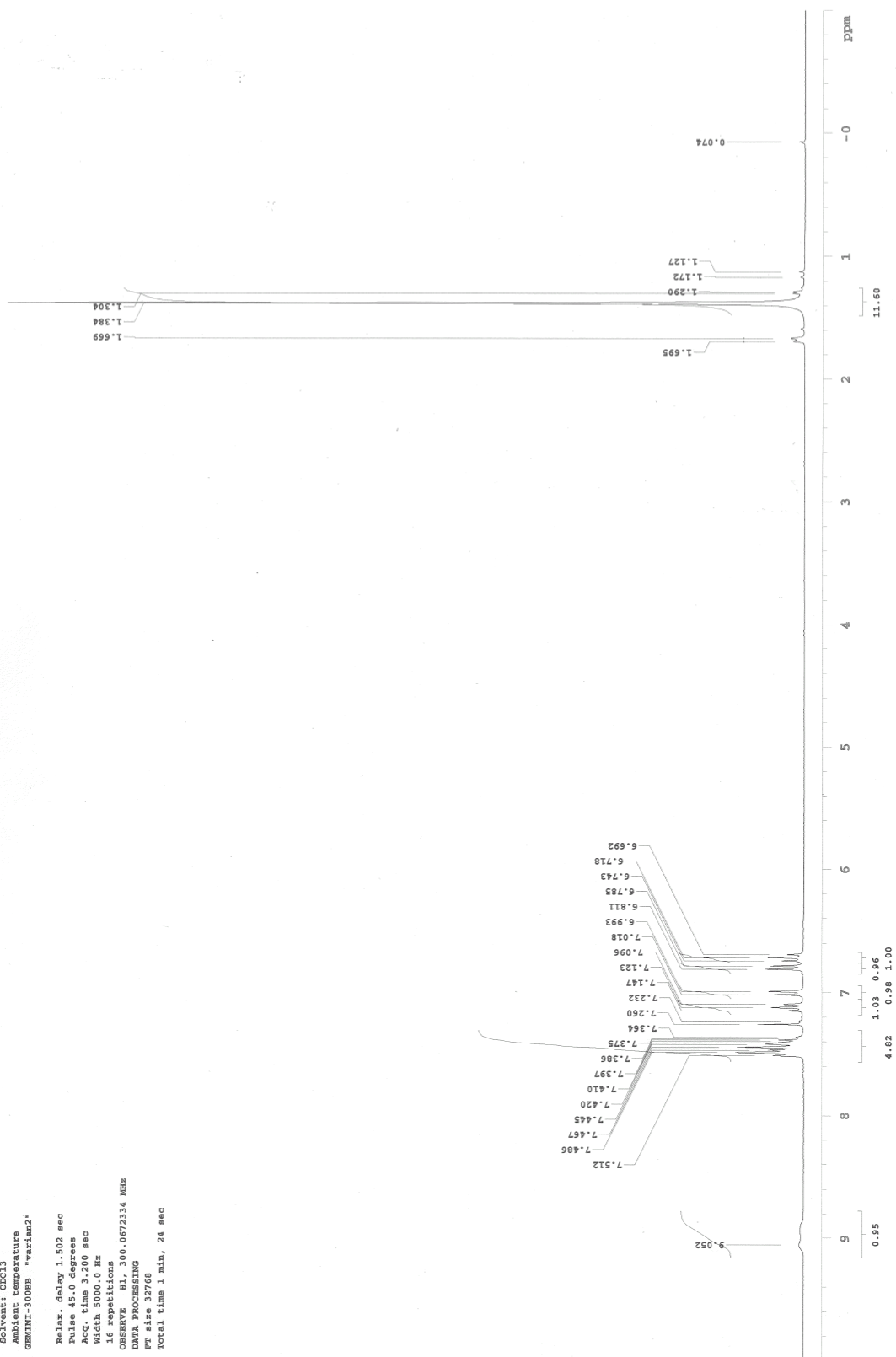
16 Repetitions

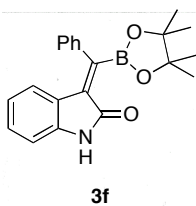
OBSERVE H1, 300.0672334 MHz

DATA PROCESSING

Ft size 32768

Total time 1 min, 24 sec





13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

gemin-300DB "variant2"

Relax. delay 1.158 sec

Pulse 45.0 degrees

Acq. time 0.842 sec

Width 24000.0 Hz

10628 repetitions

OBSERVE C13, 75.4519710 MHz

DECOUPLE H1, 300.0687335 MHz

Power 37 dB

continuously on

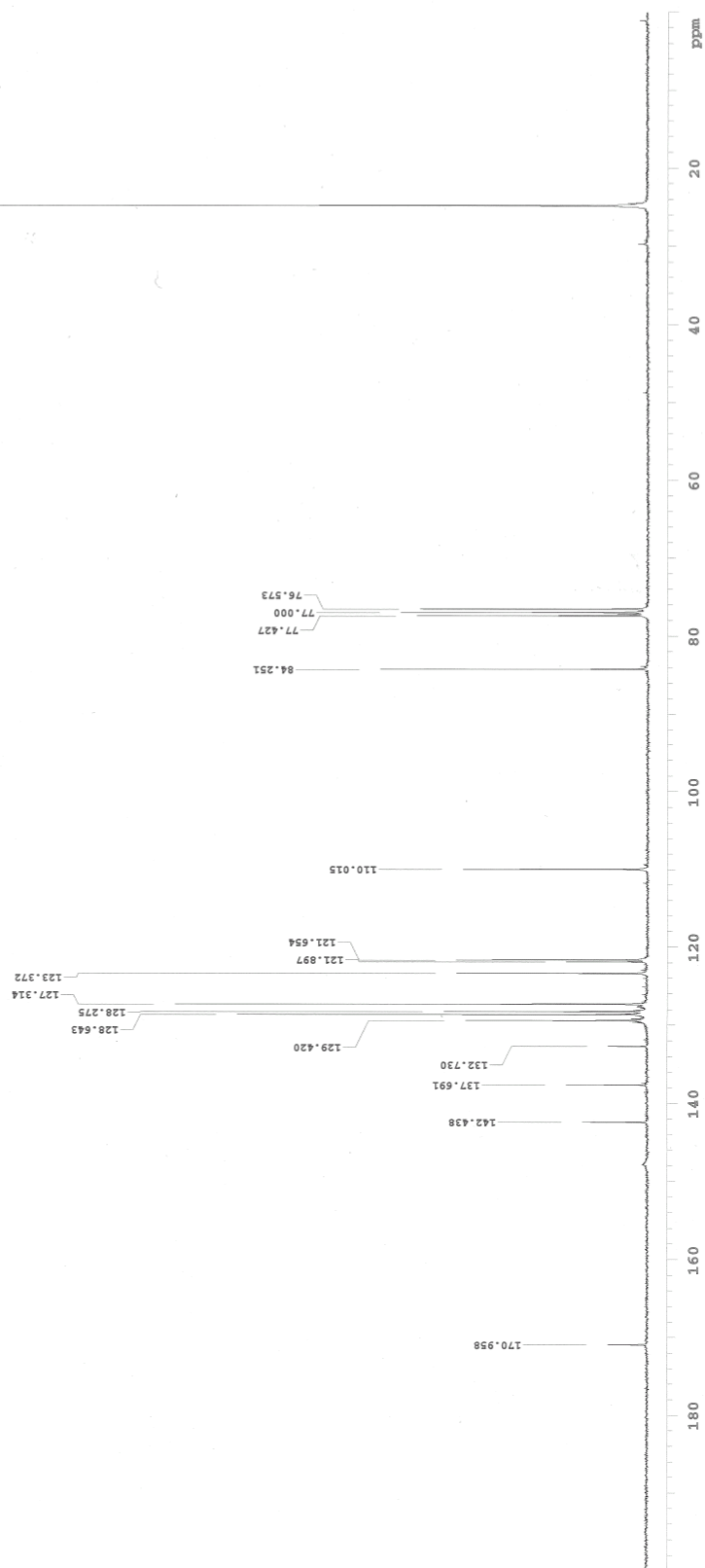
WALTZ-16 modulated

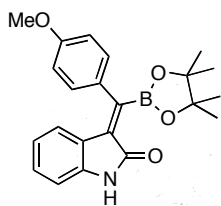
DATA PROCESSING

Line broadening 1.0 Hz

Phase 0.00000000

Total time 745 hr, 37 min, 52 sec





3g

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

QNP1H-300MHz "varian2"

Relax. delay 1.502 sec

Pulse 45.0 degrees

Acq. time 3.200 sec

Width 5000.0 Hz

16 repetitions

OBSERVE H1, 300.0672328 MHz

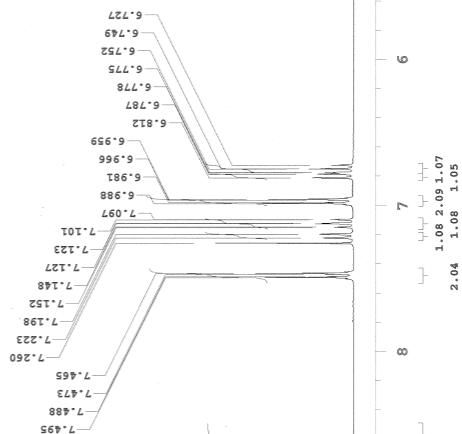
DATA PROCESSING

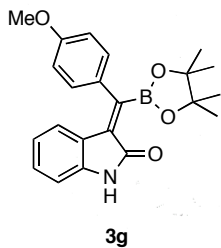
FT size 32768

Total time 1 min, 24 sec

3.873

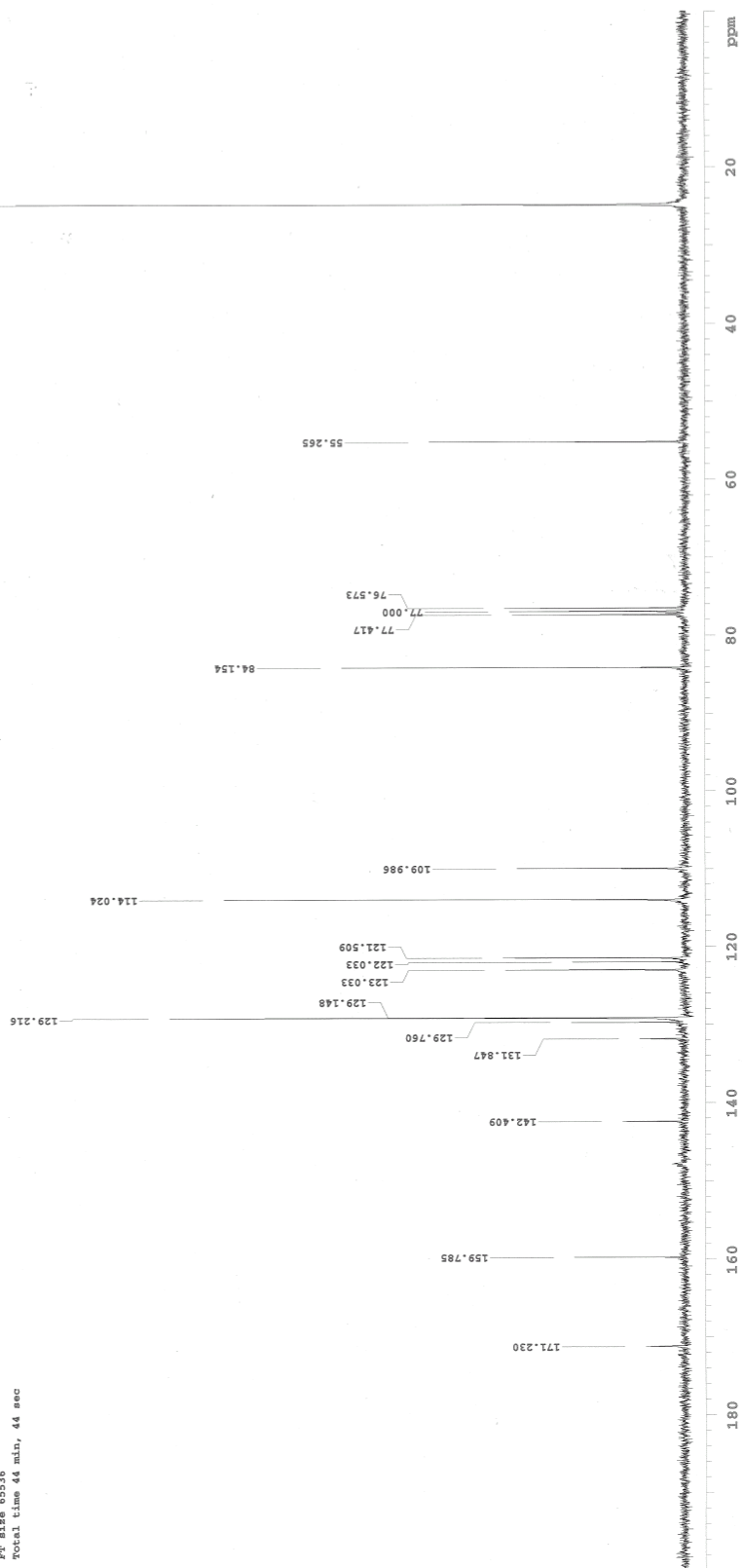
11.96

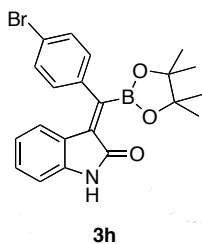




13C OBSERVE

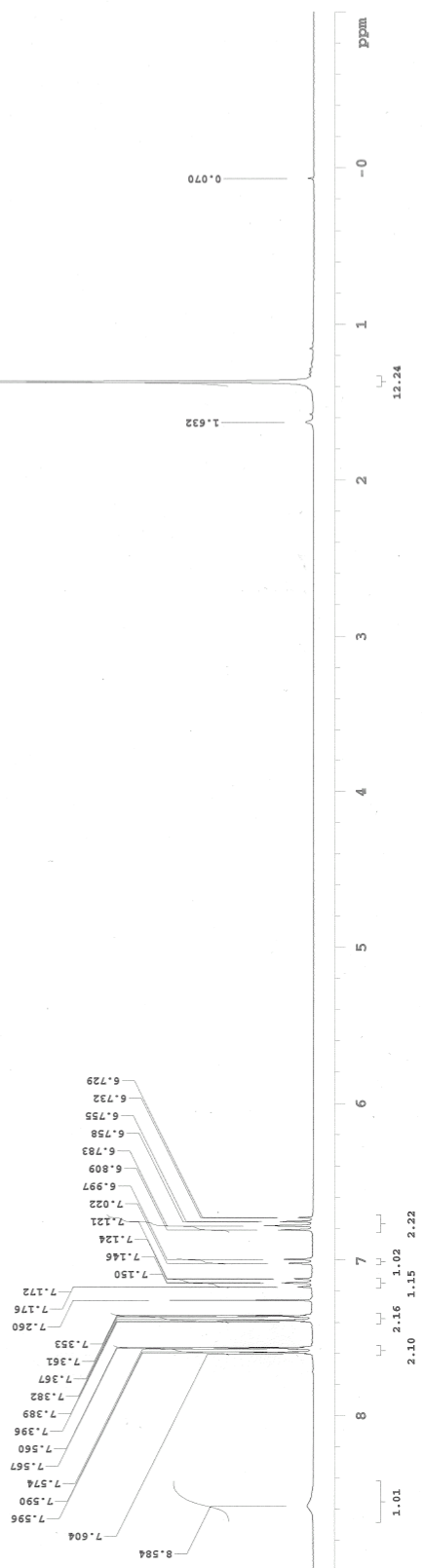
Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 GEMINI-300BB "varian2"
 Relax. delay 1.158 sec
 Pulse 45.0 degrees
 Acq. time 0.542 sec
 Width 24000.0 Hz
 484 repetitions
 OBSERVE c13, 75.4519732 MHz
 DECOUPLE H1, 300.0667335 MHz
 Power 37 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 65536
 Total time 44 min, 44 sec

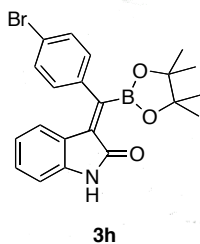




STANDARD 1H OBSERVE

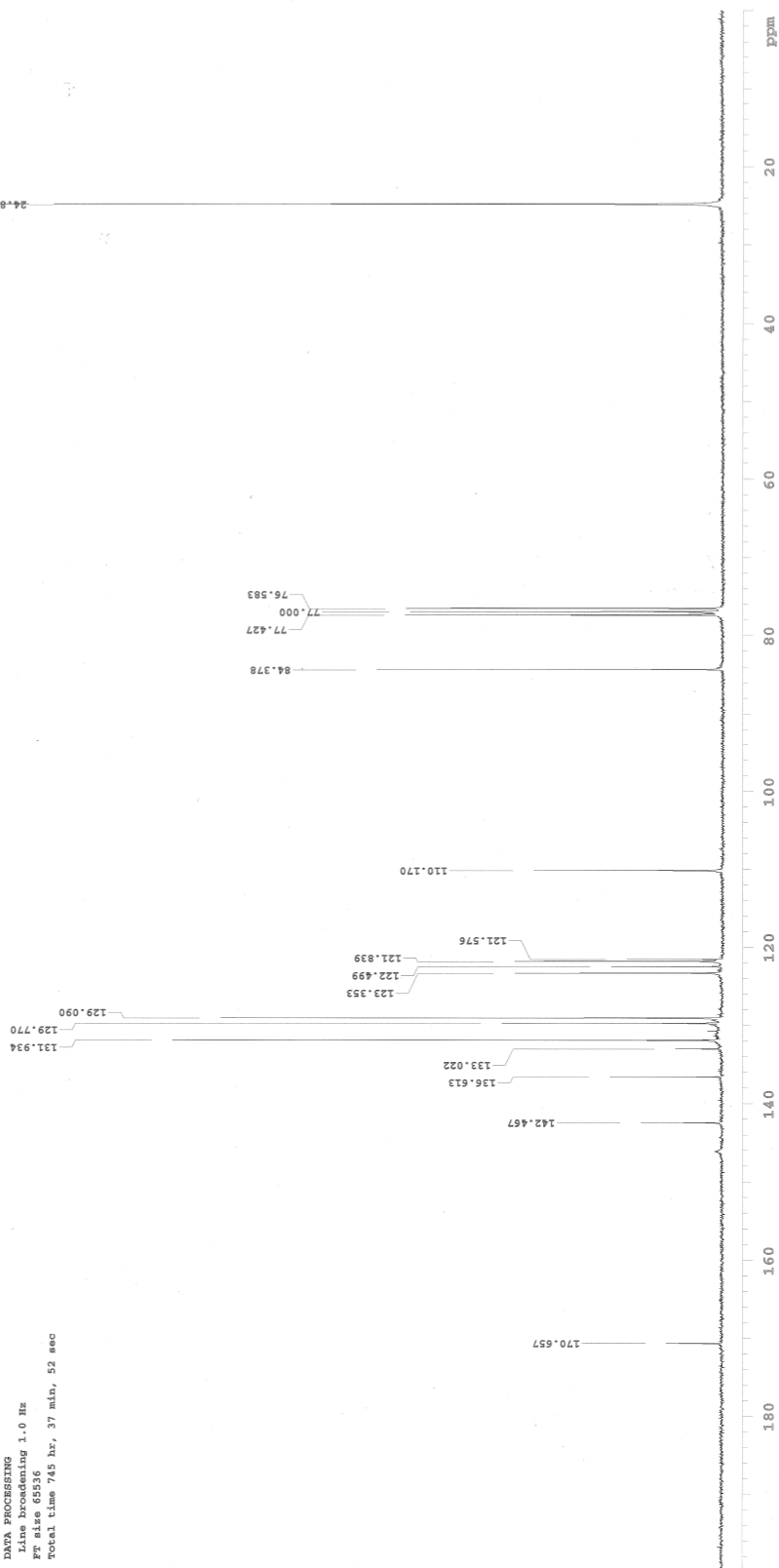
Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 GEMINI-300BB "varian2"
 Relax. delay 1.502 sec
 Pulse 45.0 degrees
 Acq. time 3.200 sec
 Width 5000.0 Hz
 16 repetitions
 OBSERVE HL 300.0672331 MHz
 DATA PROCESSING
 FT size 32768
 Total time 1 min, 24 sec

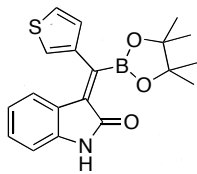




13C OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 GEMINI-300BB "varian2"
 Relax. delay 1.158 sec
 Pulse 45.0 degrees
 Acq. time 0.842 sec
 Width 24000.0 Hz
 11720 repetitions
 OBSERVE C13, 75.4519703 MHz
 DECOUPLE H1, 300.0687335 MHz
 Power 37 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 Gain 6.595
 Total time 745 hr, 37 min, 52 sec

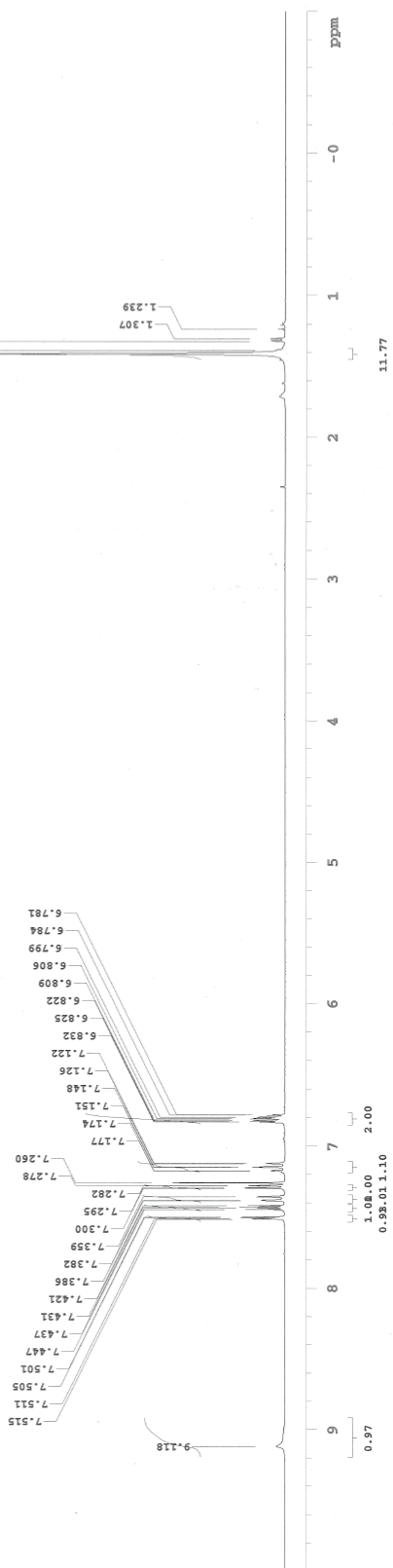


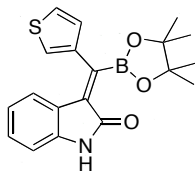


3i

STANDARD IN OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient Temperature
GEMIN-300DBB "variant2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE ML, 300.0672328 MHz
DATA PROCESSING
Ft size 32768
Total time 1 min, 24 sec

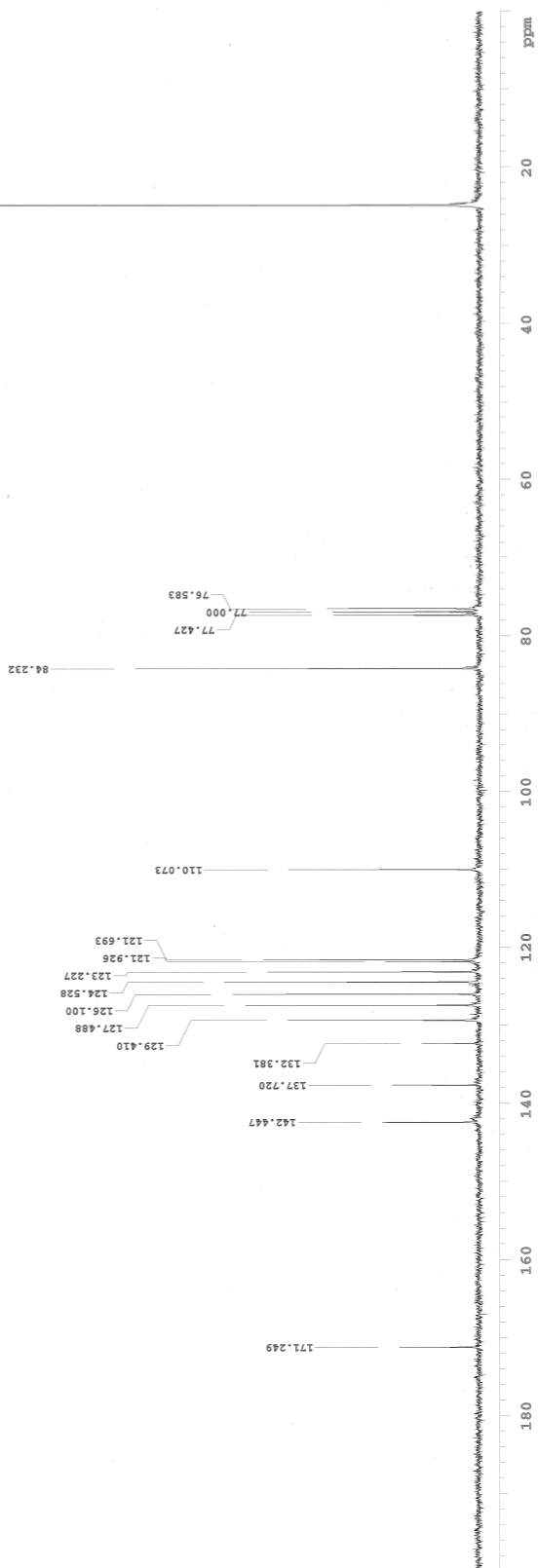


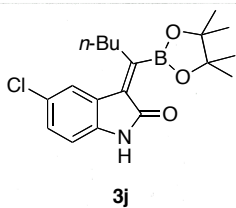


3i

13C OBSERVE

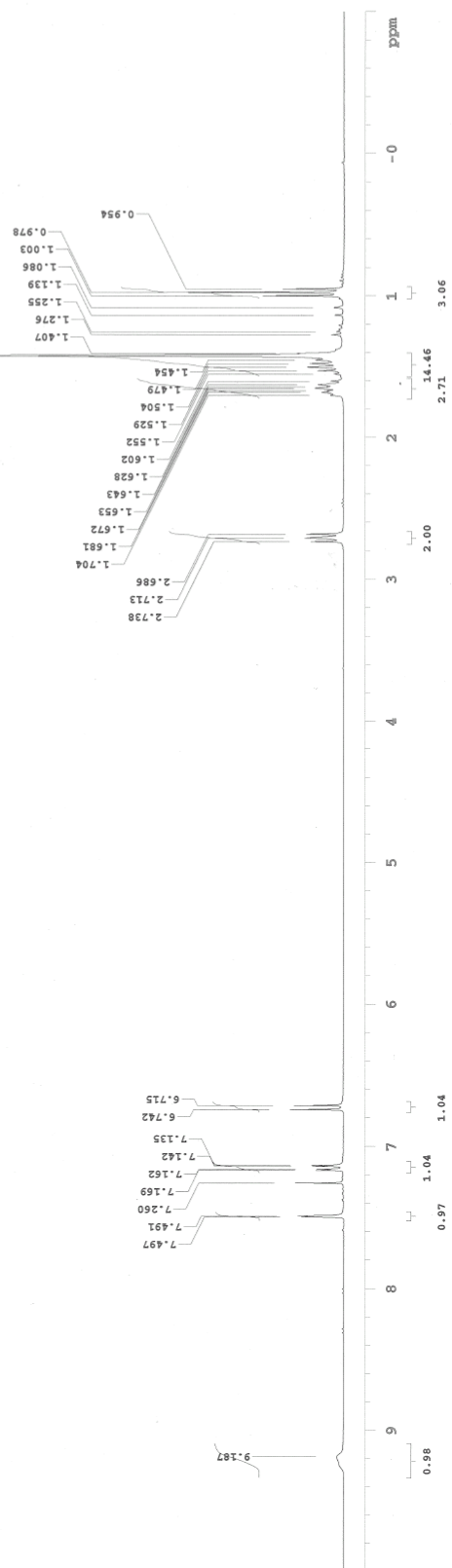
Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 GEMINI-300DB "varian2"
 Relax. delay 1.158 sec
 Pulse 45.0 degrees
 Acq. time 0.842 sec
 Width 24000.0 Hz
 680 repetitions
 OBSERVE C13, 75.4519732 MHz
 DECOUPLE H1, 300.0687335 MHz
 Power 37 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 65536
 Total time 743 hr, 37 min, 52 sec

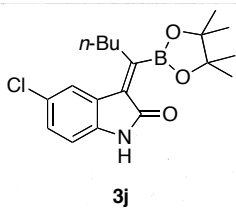




STANDARD 1H OBSERVE

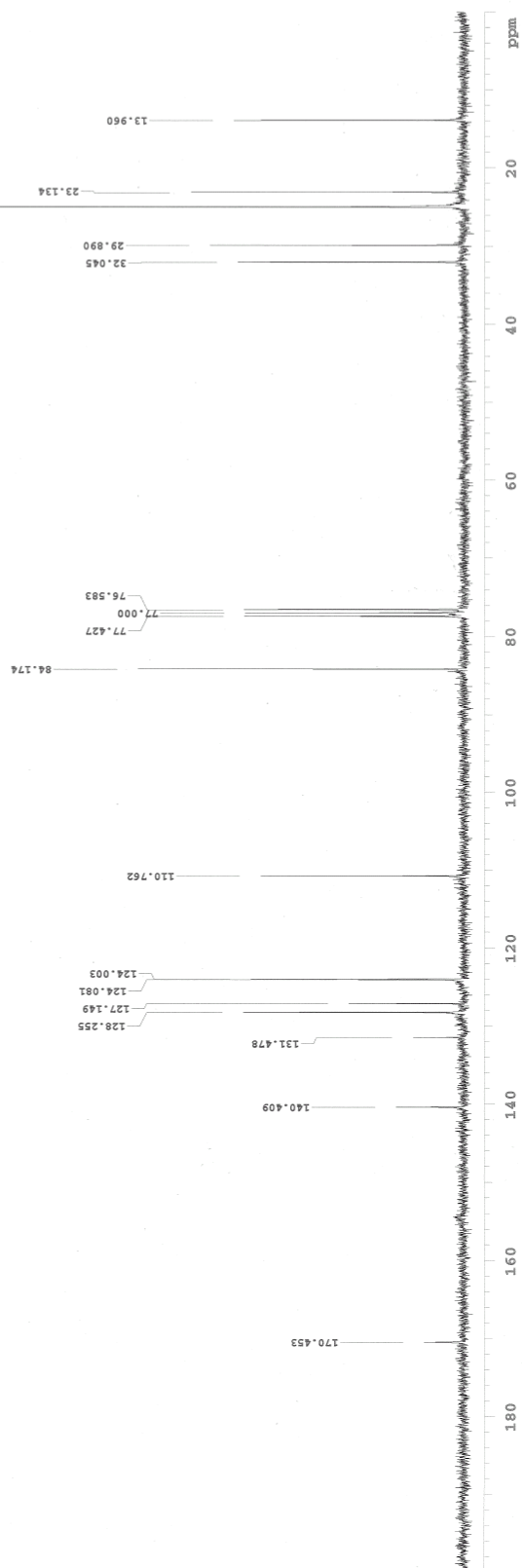
Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient Temperature
 GEMIN-300DB "variant2"
 Relax. delay 1.502 sec
 Pulse 45.0 degrees
 Acq. time 3.200 sec
 Width 5000.0 Hz
 16 repetitions
 OBSERVE M1, 300.0672331 MHz
 DATA PROCESSING
 FT size 32768
 Total time 1 min, 24 sec

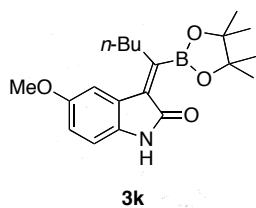




13C OBSERVE

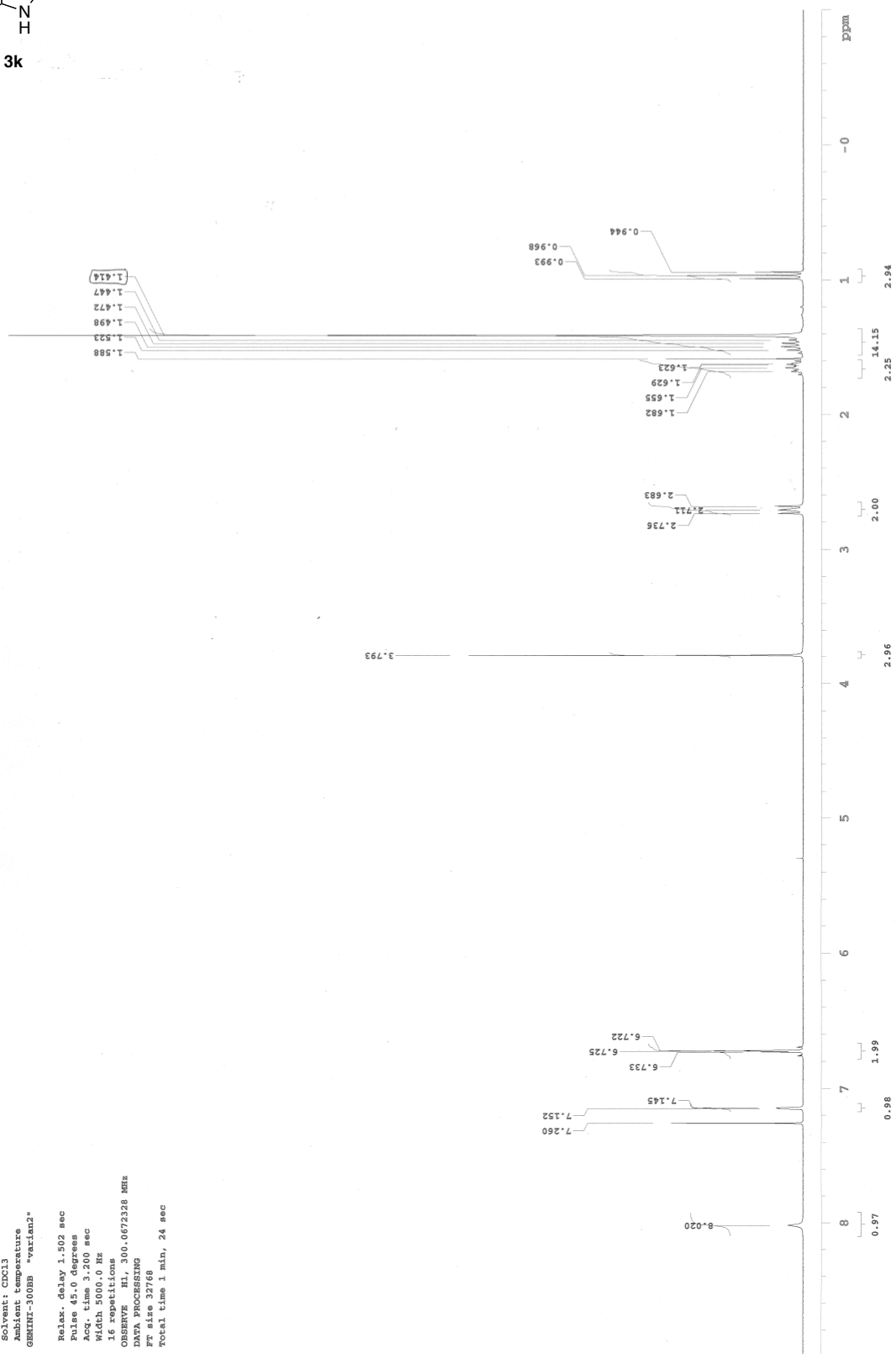
Pulse sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 GEMINI-300BB "variant2"
 Relax. delay 1.158 sec
 Pulse 45.0 degrees
 Acq. time 0.842 sec
 Width 24000.0 Hz
 600 repetitions
 OBSERVE C13, 75.4519703 MHz
 DECOUPLE H1, 300.0667335 MHz
 Power 37 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 65536
 Total time 745 hr, 37 min, 52 sec

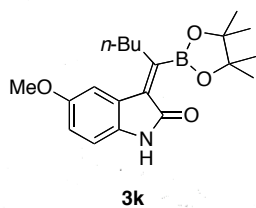




STANDARD IN OBSERVE

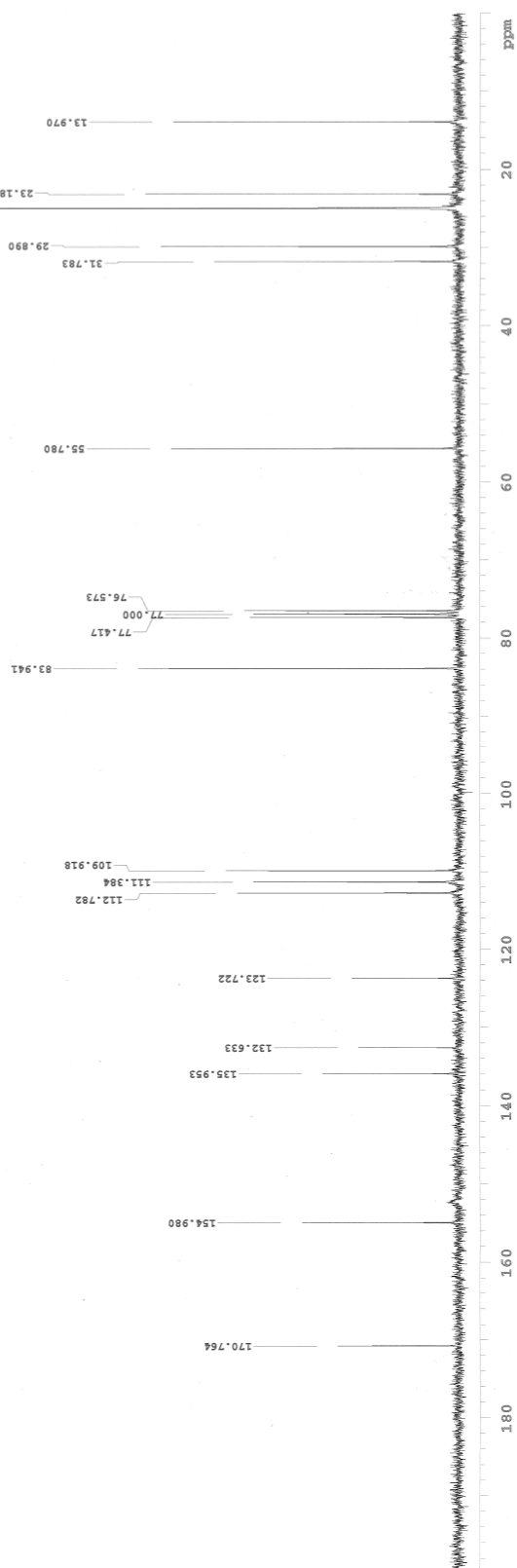
Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 GEMINI-300BB "varian2"
 Relax. delay 1.502 sec
 Pulse 45.0 degrees
 Acq. time 3.200 sec
 Width 5000.0 Hz
 16 repetitions
 OBSERVE H1, 300.0672328 MHz
 DATA PROCESSING
 F1 size 32768
 Total time 1 min, 24 sec

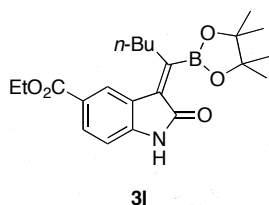




13C OBSERVE

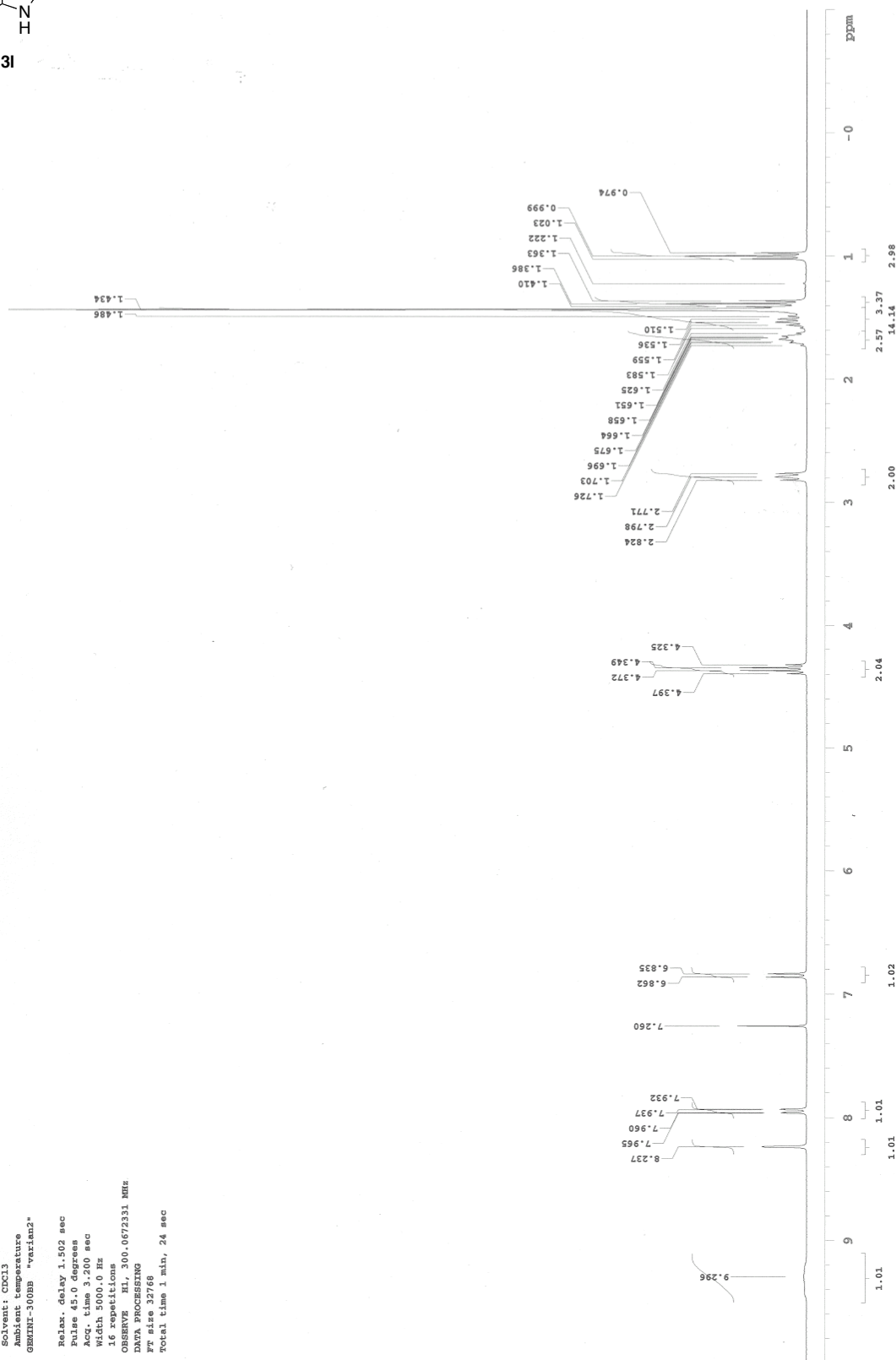
Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 GEMINI-300DB "varian2"
 Relax. delay 1.158 sec
 Pulse 45.0 degrees
 Acq. time 0.842 sec
 Width 24000.0 Hz
 348 repetitions
 OBSERVE C13, 75.4519718 MHz
 DECOUPLE H1, 300.0687335 MHz
 Power 37 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 65536
 Total time 22 min, 22 sec

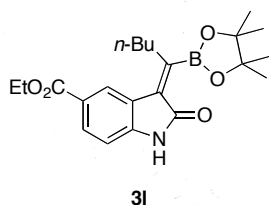




STANDARD IN OBSERVE

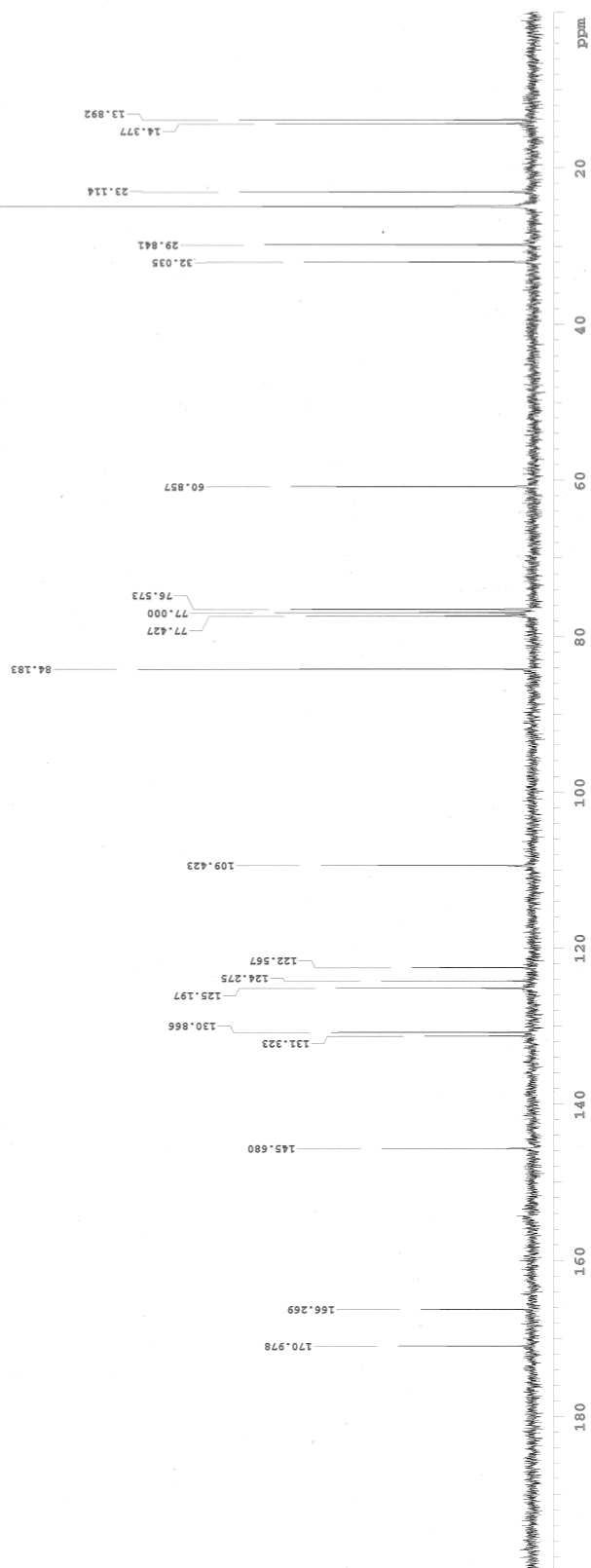
Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 GEMINI-300DB "varian2"
 Relax. delay 1.502 sec
 Pulse 45.0 degrees
 Acq. time 3.200 sec
 Width 5000.0 Hz
 16 repetitions
 OBSERVE H1, 300.0672331 MHz
 DATA PROCESSING
 FT size 32768
 Total time 1 min, 24 sec

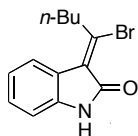




13C OBSERVE

Pulse sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 GEMINI-300BB "variant2"
 Relax. delay 1.158 sec
 Pulse 45.0 degrees
 Acq. time 0.842 sec
 Width 24000.0 Hz
 436 repetitions
 OBSERVE C13, 75.4519710 MHz
 DECOUPLE H1, 300.0667335 MHz
 Power 37 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 65536
 Total time 22 min, 22 sec





7

STANDARD IN OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

GEMIN-300DB "varian2"

Relax. delay 1.502 sec

Pulse 45.0 degrees

Acq. time 3.200 sec

Width 5000.0 Hz

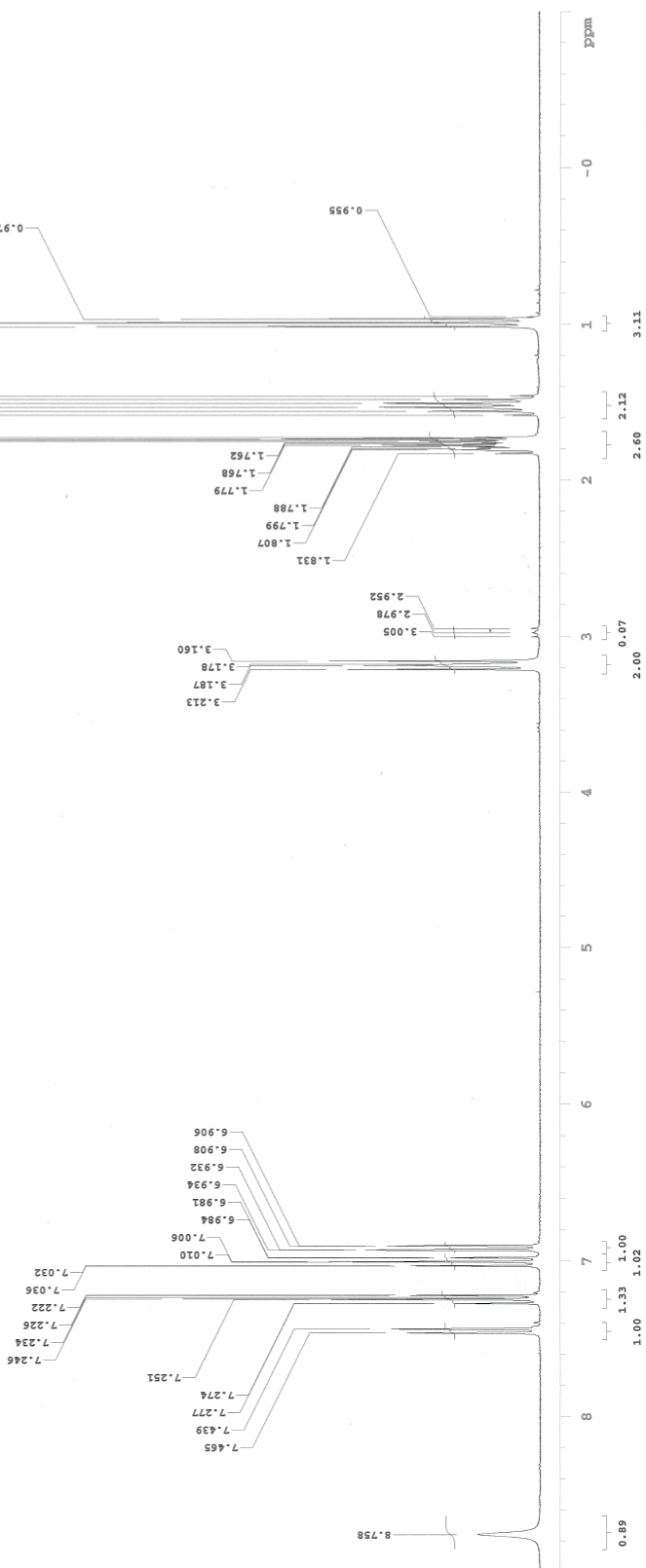
16 repetitions

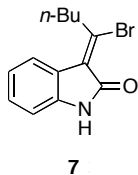
OBSERVE H1, 300.0672369 MHz

DATA PROCESSING

FT size 32768

Total time 1 min, 24 sec





13C OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 GEMIN-300DB "variant2"
 Relax. delay 1.158 sec
 Pulse 45.0 degrees
 Acq. time 0.842 sec
 Width 24000.0 Hz
 472 repetitions
 OBSERVE C13, 75.4519696 MHz
 DECOUPLE H1, 300.0687385 MHz
 Power 37 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 65536
 Total time 7 hr, 27 min, 22 sec

