Supporting Information

Stereoselective Oxindole Synthesis by Palladium-Catalyzed Cyclization Reaction of 2-(Alkynyl)aryl Isocyanates with Amides

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General. All reactions were carried out with standard Schlenk techniques under an argon atmosphere. ^{1}H and ^{13}C NMR spectra were recorded on a Varian Gemini 2000 (^{1}H at 300.07 MHz and ^{13}C at 75.46 MHz) spectrometer. NMR data were obtained in $CDCl_3$ and acetone-d_6. Proton chemical shifts were referenced to the residual proton signal of the solvent at 7.26 ppm ($CHCl_3$) and 2.09 ppm (acetone). Carbon chemical shifts were referenced to the carbon signal of the solvent at 77.0 ppm ($CDCl_3$) and 205.87 ppm (acetone-d_6). 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 silica gel 60N (Kanto). Preparative thin-layer chromatography was performed with silica 60 PF254 (Merck).

Materials. Toluene was dried and degassed by The Ultimate Solvent System (GlassContour). Anhydrous THF was purchased from Kanto Chemical Co. All other commercially available resources were used without further purification. 2-(Alkynyl)anilines were prepared by Sonogashira reaction[1] of the corresponding 2-iodoaniline derivatives[2] with alkyne. 2-(Alkynyl)aryl isocyanates were synthesized from the corresponding 2-(alkynyl)aniline according to the reported procedure.[3] The analytical data of compounds **1a**,[4] **1b**,[4] **1c**,[4] **1d**,[6] **1e**,[4] **1f**,[4] **1g**,[5] **1h**,[6] **1i**,[6] **1j**,[6] **1k**,[5] **1n**,[4] **1o**,[4] **1p**,[4] **1q**,[4] **1r**,[4] and **5**[7] have been already reported.

1l: IR (neat): 3013, 2250, 1592, 1507, 1424, 1248 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ = 5.64 (dd, *J* = 11.1, 2.4 Hz, 1H), 5.87 (dd, *J* = 17.7, 2.1 Hz, 1H), 6.09 (dd, *J* = 17.4, 11.1 Hz, 1H), 7.04 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.14 (td, *J* = 7.5, 1.5 Hz, 1H), 7.26 (td, *J* = 7.6, 1.8 Hz, 1H), 7.43 (dd, *J* = 7.5, 1.8 Hz, 1H); ^{13}C NMR ($CDCl_3$): δ = 85.3, 96.2, 116.4, 120.7, 123.5, 125.4, 127.3, 128.2, 129.4, 132.1, 135.0; HRMS (EI^+): Calcd for $C_{11}H_7NO$, M^+ 169.0528. Found m/z 169.0526.

1m: IR (KBr): 2274, 1509, 1489, 1387, 1090 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ = 6.93 (d, *J* = 8.7 Hz, 1H), 7.33–7.43 (m, 4H), 7.56–7.64 (m, 2H), 7.66 (d, *J* = 2.1 Hz, 1H); ^{13}C NMR ($CDCl_3$): δ = 83.5, 98.7, 118.3, 121.9, 122.8, 124.9, 127.5, 128.5, 129.3, 131.6, 132.3, 133.9, 134.7; HRMS (EI^+): Calcd for $C_{15}H_8BrNO$, M^+ 296.9789. Found m/z 296.9791.

General procedure for the palladium-catalyzed cyclization reaction of 1 with 2 (Tables 1 and 3)

To an oven-dried flask was added $Pd_2dba_3{\cdot}CHCl_3$ (2.0 mg, 0.20 μmol, 2 mol % Pd), a phosphine ligand (0.40 μmol, 2 mol %), amide **2**, and a solution of substrate **1** (0.20 mmol, 1.0 equiv) in dry toluene (4.0 mL). The reaction mixture was stirred at 100 °C for 3–12 h under an argon atmosphere, and then quenched with addition of water (4.0 mL) and brine (4.0 mL). The resulting aqueous solution was extracted with ethyl acetate (3 x 10 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 reprecipitation (CH_2Cl_2/hexane), preparative thin-layer chromatography, or silica gel column chromatography to give the product **3**.

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3aa: Purified by reprecipitation. IR (KBr): 3158, 1742, 1636, 1466, 1163 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 1.03 (t, *J* = 7.2 Hz, 3H), 1.51–1.66 (m, 2H), 1.69–1.84 (m, 2H), 3.21–3.35 (m, 2H), 6.95 (d, *J* = 7.5 Hz, 1H), 7.11 (dt, *J* = 8.1, 0.9 Hz, 1H), 7.26 (dt, *J* = 7.7, 0.9 Hz, 1H), 7.45 (d, *J* = 7.5 Hz, 1H), 9.16 (br s, 1H), 13.52 (br s, 1H); ^{13}C NMR ($CDCl_3$): δ = 13.8, 23.0, 29.2, 29.9, 109.6, 110.5, 115.5 (q, *J* = 287.0 Hz), 121.8, 122.3, 122.7, 127.9, 138.2, 154.0, 155.4 (q, *J* = 38.1 Hz), 170.8; HRMS (EI^+): Anal. Calcd for $C_{15}H_{15}F_3N_2O_2$: C, 57.69; H, 4.84; F, 18.25; N, 8.97. Found: C, 57.56; H, 4.63; F, 18.21; N, 8.84.

3ba: Purified by reprecipitation. IR (KBr): 3029, 1748, 1636, 1526, 1468 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 1.16 (t, *J* = 7.5 Hz, 3H), 1.76–1.92 (m, 2H), 3.21–3.33 (m, 2H), 6.95 (d, *J* = 7.8 Hz, 1H), 7.11 (t, *J* = 7.5 Hz, 1H), 7.26 (t, *J* = 7.5 Hz, 1H), 7.46 (d, *J* = 7.8 Hz, 1H), 8.80 (br s, 1H), 13.52 (br s, 1H); ^{13}C NMR ($CDCl_3$): δ = 14.1, 20.8, 31.8, 109.8, 110.5, 115.5 (q, *J* = 287.0 Hz), 121.8, 122.3, 122.7, 128.0, 138.3, 153.6, 155.4 (q, *J* = 38.0 Hz), 170.8; HRMS (EI^+): Calcd for $C_{14}H_{13}F_3N_2O_2$, M^+ 298.0929. Found m/z 298.0930.

3ca: Purified by preparative thin-layer chromatography (chloroform/acetone = 10:1). IR (KBr): 3169, 1750, 1669, 1464, 1285 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 1.51 (d, *J* = 7.2 Hz, 6H), 3.77 (sept, *J* = 6.9 Hz, 1H), 6.96 (d, *J* = 7.2 Hz, 1H), 7.12 (t, *J* = 7.5 Hz, 1H), 7.28 (td, *J* = 7.8, 1.2 Hz, 1H), 7.51 (d, *J* = 7.5 Hz, 1H), 9.27 (br s, 1H), 13.42 (br s, 1H); ^{13}C NMR ($CDCl_3$): δ = 17.5, 31.2, 110.5, 111.3, 116.0 (q, *J* = 287.0 Hz), 121.6, 122.7, 123.5, 128.3, 138.4, 155.0 (q, *J* = 36.9 Hz), 157.7, 171.1; HRMS (EI^+): Calcd for $C_{14}H_{13}F_3N_2O_2$, M^+ 298.0929. Found m/z 298.0927.

3da: Purified by reprecipitation. IR (KBr): 3156, 1736, 1673, 1617, 1462, 1264 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 0.74–0.87 (m, 2H), 1.27–1.41 (m, 2H), 2.26–2.41 (m, 1H), 6.93 (d, *J* = 7.8 Hz, 1H), 7.12 (t, *J* = 7.8 Hz, 1H), 7.27 (t, *J* = 7.5 Hz, 1H), 7.74 (d, *J* = 7.5 Hz, 1H), 8.42 (br s, 1H), 13.03 (br s, 1H); ^{13}C NMR ($CDCl_3$): δ = 10.7, 13.4, 110.0, 113.2, 115.6 (q, *J* = 287.0 Hz), 121.6, 122.4, 124.6, 128.2, 138.2, 151.2, 155.0 (q, *J* = 38.0 Hz), 170.5; HRMS (EI^+): Calcd for $C_{14}H_{11}F_3N_2O_2$, M^+ 296.0773. Found m/z 296.0777.

3ea: Purified by preparative thin-layer chromatography (hexane/ethyl acetate = 20:1). IR (KBr): 3179, 1694, 1620, 1466, 1466, 1167 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 1.60 (s, 9H), 6.83–6.94 (m, 1H), 7.09 (t, *J* = 7.5 Hz, 1H), 7.21–7.31 (m, 1H), 7.77 (d, *J* = 8.1 Hz, 1H), 9.59 (br s, 1H), 11.50 (br s, 1H); ^{13}C NMR ($CDCl_3$): δ = 27.5, 38.6, 110.4, 115.8 (q, *J* = 287.1 Hz), 119.3, 120.3, 122.1, 127.8, 129.0, 139.3, 157.9 (q, *J* = 38.0 Hz), 160.5, 171.1; HRMS (EI^+): Calcd for $C_{15}H_{15}F_3N_2O_2$, M^+ 312.1086. Found m/z 312.1086.

3fa: Purified by reprecipitation. IR (KBr): 3025, 1748, 1632, 1464, 1219 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 6.10 (d, *J* = 7.5 Hz, 1H), 6.72 (td, *J* = 7.8, 0.9 Hz, 1H), 6.89 (d, *J* = 7.5 Hz, 1H), 7.15 (td, *J* = 7.5, 1.2 Hz, 1H), 7.42–7.49 (m, 2H), 7.53–7.63 (m, 3H), 8.63 (br s, 1H), 13.12 (br s, 1H); ^{13}C NMR ($CDCl_3$): δ = 110.1, 111.0, 115.4 (q, *J* = 287.0 Hz), 121.7, 122.2, 122.3, 127.4, 128.4, 129.2, 130.1, 132.1, 138.5, 147.2, 154.8 (q, *J* = 38.0 Hz), 170.7; HRMS (EI^+): Calcd for $C_{17}H_{11}F_3N_2O_2$, M^+ 332.0773. Found m/z 332.0775.

3ga: Purified by reprecipitation. IR (KBr): 3021, 1746, 1630, 1510, 1464, 1217 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 3.92 (s, 3H), 6.31 (d, *J* = 7.8 Hz, 1H), 6.75 (t, *J* = 6.6 Hz, 1H), 6.87 (d, *J* = 8.4 Hz, 1H), 7.04–7.10 (m, 2H), 7.14 (td, *J* = 7.5, 0.9 Hz, 1H), 7.35–7.41 (m, 2H), 7.88 (br s, 1H), 13.04 (br s, 1H); ^{13}C NMR ($CDCl_3$): δ = 55.4, 110.1, 111.0, 114.6, 115.4 (q, *J* = 287.0 Hz), 121.9, 122.2, 122.3, 124.1, 128.3, 129.2, 138.4, 147.5, 154.9 (q, *J* = 38.0 Hz), 161.0, 170.7; HRMS (EI^+): Calcd for $C_{18}H_{13}F_3N_2O_3$, M^+ 362.0878. Found m/z 362.0875.

3ha: Purified by silica gel column chromatography (chloroform/ethyl acetate = 10/1). IR (KBr): 3173, 1736, 1676, 1522, 1333 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 6.03 (d, *J* = 6.9 Hz, 1H), 6.75 (t, *J* = 8.1 Hz, 1H), 6.90 (d, *J* = 7.5 Hz, 1H), 7.17 (t, *J* = 7.8 Hz, 1H), 7.60 (d, *J* = 9.0 Hz, 2H), 7.84 (d, *J* = 8.7 Hz, 2H), 7.97 (br s, 1H), 13.15 (br s, 1H); ^{13}C NMR ($CDCl_3$): δ = 110.4, 111.4, 115.3 (q, *J* = 287.0 Hz), 121.1, 122.0, 122.6, 123.7 (q, *J* = 270.9 Hz), 126.3 (q, *J* = 13.8 Hz), 128.2, 129.0, 132.2 (q, *J* = 32.3 Hz), 135.7, 138.7, 145.1, 154.9 (q, *J* = 38.1 Hz), 170.6; HRMS (EI^+): Calcd for $C_{18}H_{10}F_6N_2O_2$, M^+ 400.0646. Found m/z 400.0648.

3ia: Purified by reprecipitation. IR (KBr): 3160, 1750, 1663, 1630, 1464, 1217 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 2.49 (s, 3H), 6.23 (d, *J* = 7.2 Hz, 1H), 6.74 (td, *J* = 7.5, 0.6 Hz, 1H), 6.89 (d, *J* = 8.1 Hz, 1H), 7.15 (td, *J* = 7.5, 0.9 Hz, 1H), 7.30–7.41 (m, 4H), 8.73 (br s, 1H), 13.10 (br s, 1H); ^{13}C NMR ($CDCl_3$): δ = 21.7, 110.1, 110.9, 115.4 (q, *J* = 287.1 Hz), 121.8, 122.2, 122.3, 127.3, 128.3, 129.1, 129.9, 138.5, 140.4, 147.6, 154.8 (q, *J* = 38.0 Hz), 170.9; HRMS (EI^+): Calcd for $C_{18}H_{13}F_3N_2O_2$, M^+ 346.0929. Found m/z 346.0927.

3ja: Purified by silica gel column chromatography (chloroform/ethyl acetate = 10/1). IR (KBr): 3171, 1750, 1638, 1462, 1302, 1210 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 2.29 (s, 3H), 5.91 (d, *J* = 7.5 Hz, 1H), 6.74 (td, *J* = 7.5, 0.9 Hz, 1H), 6.92 (d, *J* = 7.8 Hz, 1H), 7.17 (td, *J* = 7.5, 1.2 Hz, 1H), 7.28 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.34–7.45 (m, 2H), 7.45–7.55 (m, 1H), 9.62 (br s, 1H), 13.25 (br s, 1H); ^{13}C NMR ($CDCl_3$): δ = 19.2, 110.2, 110.8, 115.4 (q, *J* = 287.0 Hz), 121.7, 121.9, 122.6, 126.8, 127.0, 128.4, 130.1, 130.6, 131.9, 135.3, 138.6, 147.1, 154.5 (q, *J* = 38.0 Hz), 171.0; HRMS (EI^+): Calcd for $C_{18}H_{13}F_3N_2O_2$, M^+ 346.0929. Found m/z 346.0928.

3ka: Purified by reprecipitation. IR (KBr): 3166, 1741, 1674, 1636, 1460, 1215 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 6.30 (d, *J* = 7.8 Hz, 1H), 6.80 (t, *J* = 7.5 Hz, 1H), 6.88 (d, *J* = 7.8 Hz, 1H), 7.16 (td, *J* = 7.8, 1.2 Hz, 1H), 7.17 (dd, *J* = 5.4, 1.2 Hz, 1H), 7.49 (dd, *J* = 3.0, 1.2 Hz, 1H), 7.57 (dd, *J* = 5.1, 3.0 Hz, 1H), 8.07 (br s, 1H), 13.06 (br s, 1H); ^{13}C NMR ($CDCl_3$): δ = 110.1, 111.7, 115.4 (q, *J* = 287.0 Hz), 121.7, 122.1, 122.5, 125.6, 126.7, 127.3, 128.6, 131.7, 138.4, 142.4, 154.8 (q, *J* = 38.1 Hz), 170.3; HRMS (EI^+): Calcd for $C_{15}H_9F_3N_2O_2S$, M^+ 338.0337. Found m/z 338.0336.

3la: Purified by reprecipitation. IR (KBr): 3168, 1738, 1671, 1619, 1520, 1462 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 5.92 (d, *J* = 17.1 Hz, 1H), 5.93 (d, *J* = 12.3 Hz, 1H), 6.92 (d, *J* = 7.8 Hz, 1H), 6.99 (t, *J* = 7.8 Hz, 1H), 7.10 (dd, *J* = 16.8, 11.1 Hz, 1H), 7.23 (t, *J* = 7.8 Hz, 1H), 7.76 (d, *J* = 8.1 Hz, 1H), 8.55 (br s, 1H), 13.13 (br s, 1H); ^{13}C NMR ($CDCl_3$): δ = 109.8, 110.3, 115.4 (q, *J* = 287.0 Hz), 121.6, 122.3, 122.8, 125.4, 128.5, 128.6, 138.4, 146.3, 155.5 (q, *J* = 38.0 Hz), 170.7; HRMS (EI^+): Calcd for $C_{13}H_9F_3N_2O_2$, M^+ 282.0616. Found m/z 282.0618.

3ma: Purified by silica gel column chromatography (chloroform/ethyl acetate = 10/1). IR (KBr): 3166, 1755, 1671, 1636, 1466, 1302, 1213 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 6.12 (d, *J* = 2.1 Hz, 1H), 6.76 (d, *J* = 8.4 Hz, 1H), 7.25 (dd, *J* = 8.1, 2.1 Hz, 1H), 7.40–7.46 (m, 2H), 7.56–7.67 (m, 3H), 7.99 (br s, 1H), 13.04 (br s, 1H); ^{13}C NMR ($CDCl_3$): δ = 110.0, 111.6, 115.2, 115.3 (q, *J* = 288.2 Hz), 123.5, 125.1, 127.1, 129.4, 130.6, 130.9, 131.4, 137.3, 148.9, 154.7 (q, *J* = 38.0 Hz), 170.7; HRMS (EI^+): Calcd for $C_{17}H_{10}BrF_3N_2O_2$, M^+ 409.9878. Found m/z 409.9879.

3na: Purified by reprecipitation. IR (KBr): 3160, 1740, 1674, 1632, 1472, 1202 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 1.05 (t, *J* = 7.5 Hz, 3H), 1.55–1.85 (m, 4H), 3.18–3.38 (m, 2H), 6.87 (d, *J* = 8.4 Hz, 1H), 7.23 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.41 (d, *J* = 2.1 Hz, 2H), 9.18 (br s, 1H), 13.49 (br s, 1H); ^{13}C NMR ($CDCl_3$): δ = 13.7, 22.9, 29.1, 29.9, 108.8, 111.2, 115.4 (q, *J* = 287.1 Hz), 122.4, 123.3, 127.6, 128.2, 136.4, 155.5 (q, *J* = 38.0 Hz), 155.6, 170.6; HRMS (EI^+): Calcd for $C_{15}H_{14}ClF_3N_2O_2$, M^+ 346.0696. Found m/z 346.0702.

3oa: Purified by reprecipitation. IR (KBr): 3164, 1728, 1674, 1528, 1480, 1204 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 1.03 (t, *J* = 6.9 Hz, 3H), 1.52–1.66 (m, 2H), 1.70–1.82 (m, 2H), 3.20–3.30 (m, 2H), 3.82 (s, 3H), 6.80 (dd, *J* = 8.7, 2.1 Hz, 1H), 6.84 (d, *J* = 8.4 Hz, 1H), 7.03 (d, *J* = 2.1 Hz, 1H), 8.92 (br s, 1H), 13.59 (br s, 1H); ^{13}C NMR ($CDCl_3$): δ = 13.8, 23.0, 29.1, 29.8, 55.9, 109.6, 109.9, 110.5, 112.5, 115.5 (q, *J* = 287.0 Hz), 122.8, 132.0, 154.1, 155.4 (q, *J* = 38.0 Hz), 155.8, 170.8; HRMS (EI^+): Calcd for $C_{16}H_{17}F_3N_2O_3$, M^+ 342.1191. Found m/z 342.1193.

3pa: Purified by reprecipitation. IR (KBr): 3168, 1744, 1671, 1636, 1474, 1254 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 1.07 (t, *J* = 6.9 Hz, 3H), 1.41 (t, *J* = 7.2 Hz, 3H), 1.61–1.86 (m, 4H), 3.31–3.38 (m, 2H), 4.39 (q, *J* = 7.2Hz, 2H), 6.99 (d, *J* = 8.4 Hz, 1H), 8.01 (d, *J* = 7.5 Hz, 1H), 8.19 (s, 1H), 9.35 (br s, 1H), 13.37 (br s, 1H); ^{13}C NMR ($CDCl_3$): δ = 13.6, 14.3, 22.9, 29.0, 30.0, 61.1, 108.7, 110.0, 115.4 (q, *J* = 287.1 Hz), 121.6, 123.6, 125.1, 130.0, 141.7, 155.4 (q, *J* = 38.0 Hz), 155.5, 166.2, 171.2; HRMS (EI^+): Calcd for $C_{18}H_{19}F_3N_2O_4$, M^+ 384.1297. Found m/z 384.1296.

3qa: Purified by reprecipitation. IR (KBr): 3150, 1750, 1676, 1686, 1476 cm^{-1}; ^1H NMR (acetone-d_6): δ = 1.06 (t, *J* = 7.5 Hz, 3H), 1.56–1.94 (m, 4H), 3.34–3.52 (m, 2H), 7.25 (d, *J* = 8.4 Hz, 2H), 7.69 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.94 (s, 1H), 10.57 (br s, 1H), 13.79 (br s, 1H); ^{13}C NMR (acetone-d_6): δ = 13.6, 22.9, 29.5, 30.0, 105.8, 108.5, 111.5, 116.0 (q, *J* = 287.0 Hz), 119.4, 123.1, 125.8, 132.6, 143.0, 155.5 (q, *J* = 38.0 Hz), 156.3, 170.8; HRMS (EI^+): Calcd for $C_{16}H_{14}F_3N_3O_2$, M^+ 337.1038. Found m/z 337.1037.

3ra: Purified by recrystallization from toluene. IR (KBr): 3162, 1740, 1665, 1622, 1264 cm^{-1}; ^1H NMR (DMSO-d_6): δ = 1.02 (t, *J* = 7.2 Hz, 3H), 1.48–1.63 (m, 2H), 1.66–1.80 (m, 2H), 3.23–3.33 (m, 2H), 7.65–7.76 (m, 2H), 7.77 (s, 1H), 8.15–8.22 (m, 1H), 8.23–8.31 (m, 1H), 12.11 (br s, 1H), 13.82 (br s, 1H); ^{13}C NMR (DMSO-d_6): δ = 14.6, 23.1, 29.8, 30.0, 110.7, 116.2 (q, *J* = 287.0 Hz), 116.7, 121.1, 123.7, 124.7, 125.4, 128.1, 128.9, 129.8, 135.8, 154.4, 155.3 (q, *J* = 38.0 Hz), 171.4; HRMS (EI^+): Calcd for $C_{19}H_{16}ClF_3N_2O_2$, M^+ 396.0852. Found m/z 396.0851.

3ab: Purified by preparative thin-layer chromatography (chloroform/ethyl acetate = 20:1). IR (KBr): 2959, 1663, 1607, 1466, 1341, 1156 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 0.93 (t, *J* = 7.2 Hz, 3H), 1.38–1.62 (m, 4H), 2.42 (s, 3H), 2.81–2.92 (m, 2H), 6.97 (d, *J* = 7.5 Hz, 1H), 7.03 (td, *J* = 7.8, 1.2 Hz, 1H), 7.17 (td, *J* = 7.8, 1.2 Hz, 1H), 7.23 (d, *J* = 7.8 Hz, 1H), 7.32 (d, *J* = 8.7 Hz, 2H), 7.85 (d, *J* = 8.4 Hz, 2H), 8.59 (br s, 1H), 12.64 (br s, 1H); ^{13}C NMR ($CDCl_3$): δ = 13.7, 21.6, 23.1, 29.3, 29.5, 104.9, 110.4, 121.0, 122.1, 122.3, 126.4, 127.2, 130.0, 137.3, 137.5, 144.4, 156.1, 170.9; HRMS (EI^+): Calcd for $C_{20}H_{22}N_2O_3S$, M^+ 370.1351. Found m/z 370.1350.

3ac: Purified by preparative thin-layer chromatography (chloroform/ethyl acetate = 10:1). IR (KBr): 3025, 1667, 1624, 1464, 1252 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 1.06 (t, *J* = 7.5 Hz, 3H), 1.56–1.74 (m, 4H), 3.35–3.56 (m, 2H), 6.91 (d, *J* = 7.5 Hz, 1H), 7.08 (td, *J* = 7.5, 1.5 Hz, 1H), 7.16 (td, *J* = 7.5, 1.2 Hz, 1H), 7.43–7.63 (m, 4H), 8.10–8.18 (m, 2H), 9.40 (br s, 1H), 13.41 (br s, 1H); ^{13}C NMR ($CDCl_3$): δ = 13.9, 23.2, 29.4, 30.2, 105.9, 109.9, 121.5, 122.2, 123.1, 126.2, 128.0, 128.8, 132.5, 134.1, 137.4, 158.7, 165.5, 171.1; HRMS (EI^+): Calcd for $C_{20}H_{20}N_2O_2$, M^+ 320.1525. Found m/z 320.1524.

3ad: Purified by preparative thin-layer chromatography (chloroform/ethyl acetate = 20:1). IR (KBr): 3140, 1746, 1663, 1464, 1213 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 1.02 (t, *J* = 6.9 Hz, 3H), 1.50–1.85 (m, 4H), 3.23–3.38 (m, 2H), 5.23 (s, 2H), 6.92 (d, *J* = 7.5 Hz, 1H), 7.05 (td, *J* = 7.5, 1.2 Hz, 1H), 7.14 (td, *J* = 7.5, 1.2 Hz, 1H), 7.31–7.46 (m, 6H), 8.77 (br s, 1H), 12.14 (br s, 1H); ^{13}C NMR ($CDCl_3$): δ = 13.9, 23.1, 29.2, 29.4, 67.3, 104.1, 109.9, 121.0, 121.9, 123.1, 125.8, 128.1, 128.3, 128.5, 135.6, 137.1, 152.5, 158.0, 170.5; HRMS (EI^+): Calcd for $C_{21}H_{22}N_2O_3$, M^+ 350.1630. Found m/z 350.1627.

3ae: Purified by preparative thin-layer chromatography (chloroform/acetone = 4:1). IR (KBr): 3297, 1671, 1605, 1559, 1466, 1240 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 1.12 (t, *J* = 7.5 Hz, 3H), 1.52–1.67 (m, 2H), 1.74–1.88 (m, 2H), 3.34–3.46 (m, 2H), 4.51 (d, *J* = 5.7 Hz, 2H), 6.32 (br s, 1H), 6.74–6.82 (m, 1H), 7.01–7.16 (m, 3H), 7.28–7.42 (m, 6H), 12.10 (br s, 1H); ^{13}C NMR ($CDCl_3$): δ = 14.0, 23.2, 29.7, 29.9, 44.0, 101.3, 109.6, 120.5, 121.9, 123.5, 124.8, 127.7, 127.8, 128.9, 135.8, 138.8, 153.3, 161.6, 170.2; HRMS (EI^+): Calcd for $C_{21}H_{23}N_3O_2$, M^+ 349.1790. Found m/z 349.1788.

3af: Purified by preparative thin-layer chromatography (chloroform/ethyl acetate = 5:1). IR (KBr): 3139, 1721, 1615, 1466, 1364 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 0.95 (t, *J* = 7.2 Hz, 3H), 1.42–1.57 (m, 2H), 1.61–1.76 (m, 2H), 2.90–3.01 (m, 2H), 6.62–6.71 (m, 1H), 7.04 (td, *J* = 7.5, 1.2 Hz, 1H), 7.21 (td, *J* = 7.8, 1.2 Hz, 1H), 7.53 (d, *J* = 8.1 Hz, 1H), 7.73–7.82 (m, 2H), 7.88–7.96 (m, 2H), 8.12 (br s, 1H); ^{13}C NMR ($CDCl_3$): δ = 13.9, 22.8, 27.9, 34.1, 110.0, 121.4, 122.0, 123.6, 123.9, 125.8, 129.7, 132.2, 134.1, 140.6, 141.9, 166.5, 167.1; HRMS (EI^+): Calcd for $C_{21}H_{18}N_2O_3$, M^+ 346.1317. Found m/z 346.1316.

3ag: Purified by preparative thin-layer chromatography (chloroform/ethyl acetate = 5:1). IR (KBr): 3304, 1721, 1709, 1615, 1466 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 0.95 (t, *J* = 7.2 Hz, 3H), 1.42–1.58 (m, 2H), 1.63–1.78 (m, 2H), 2.78–2.92 (m, 1H), 2.96–3.10 (m, 1H), 3.65 (d, *J* = 22.2 Hz, 1H), 3.77 (d, *J* = 22.2 Hz, 1H), 6.66 (t, *J* = 7.2 Hz, 2H), 7.03 (t, *J* = 7.8 Hz, 2H), 7.12–7.24 (m, 2H), 7.29 (dd, *J* = 7.5, 0.6 Hz, 1H), 7.50 (d, *J* = 7.5 Hz, 1H), 8.51 (br s, 1H); ^{13}C NMR ($CDCl_3$): δ = 13.9, 23.0, 28.2, 33.4, 36.2, 108.8, 110.0, 121.4, 121.9, 122.4, 123.8, 124.5, 125.0, 125.4, 127.6, 129.6, 140.8, 144.4, 144.5, 165.9, 174.3; HRMS (EI^+): Calcd for $C_{21}H_{20}N_2O_2$, M^+ 332.1525. Found m/z 332.1526.

3ah: Purified by preparative thin-layer chromatography (chloroform/ethyl acetate = 1:1). IR (KBr): 3250, 1750, 1701, 1617, 1468, 1397, 1190 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 1.00 (t, *J* = 7.2 Hz, 3H), 1.45–1.62 (m, 2H), 1.64–1.78 (m, 2H), 2.83–2.95 (m, 2H), 3.99 (t, *J* = 7.5 Hz, 2H), 4.53 (t, *J* = 7.5 Hz, 2H), 6.87 (d, *J* = 7.8 Hz, 1H), 6.99 (t, *J* = 7.8 Hz, 1H), 7.19 (t, *J* = 7.8 Hz, 1H), 7.39 (d, *J* = 7.5 Hz, 1H), 8.59 (br s, 1H); ^{13}C NMR ($CDCl_3$): δ = 14.0, 22.9, 28.9, 32.1, 46.4, 62.9, 109.9, 121.2, 121.7, 122.1, 123.0, 128.7, 140.0, 148.3, 155.7, 166.6; HRMS (EI^+): Calcd for $C_{16}H_{18}N_2O_3$, M^+ 286.1317. Found m/z 286.1315.

3ai: Purified by preparative thin-layer chromatography (hexane/ethyl acetate/triethylamine = 5:5:1). IR (KBr): 3152, 1640, 1578, 1462, 1225 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 0.88 (t, *J* = 7.5 Hz, 3H), 1.34–1.49 (m, 2H), 1.65–1.80 (m, 2H), 2.75–2.86 (m, 2H), 6.99–7.12 (m, 3H), 7.22–7.36 (m, 4H), 7.39–7.49 (m, 2H), 9.30 (br s, 1H), 12.08 (br s, 1H); ^{13}C NMR ($CDCl_3$): δ = 13.6, 22.7, 28.8, 29.4, 95.6, 109.6, 118.5, 120.8, 122.6, 124.4, 126.1, 126.4, 129.2, 135.4, 137.9, 163.4, 171.1; HRMS (EI^+): Calcd for $C_{19}H_{20}N_2O$, M^+ 292.1576. Found m/z 292.1576.

A Straightforward Synthesis of 3aa on Gram Scale (equation 1)

To a cooled solution of triphosgene (1.10 g, 3.72 mmol, 0.37 equiv) in benzene (100 mL) was slowly added a solution of 1-(hex-1-ynyl)aniline **5** (1.73 g, 10.0 mmol, 1.0 equiv), Et_3N (17 mL, 121 mmol, 12 equiv) in benzene (25 mL). The resulting solution was stirred at 0 °C for 2.5 h under an argon atmosphere. After this time, the solution was filtered through a pad of Celite and eluted with Et_2O. The filtrate was concentrated under reduced pressure to give crude product **1a** as a brown oil.

To an oven-dried flask was added $Pd_2dba_3{\cdot}CHCl_3$ (103 mg, 0.10 mmol, 2 mol % Pd), dppf (111 mg, 0.20 mmol, 2 mol %), trifluoroacetamide (**2a**, 1.69 g, 15.0 mmol, 1.5 equiv), crude product **1a**, and dry toluene (200 mL). The reaction mixture was stirred at 100 °C for 3 h under an argon atmosphere, and then cooled gradually to 0 °C. The precipitated yellow solid was filtered and dried under reduced pressure to give **3aa** (2.43 g, 78%). To the filtrate was added water (50 mL) and the resulting aqueous solution was extracted with ethyl acetate (3 x 50 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 reprecipitation (CH_2Cl_2/hexane) to give **3aa** (0.23 g, 7%) [Total 2.65 g, 85% over 2 steps].

General procedure for the hydrolysis of the trifluoroacetyl group (Table 2)

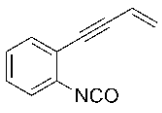
To an oven-dried flask was added **3** (0.15 mmol, 1.0 equiv), K_2CO_3 (107 mg, 5.2 equiv), MeOH (4.5 mL), and H_2O (0.1 mL). The reaction mixture was stirred at room temperature for 50 min under an air atmosphere, and then the solvent was removed under reduced pressure. To the residue was added water (5 mL) and $CHCl_3$ (10 mL), and the resulting aqueous solution was extracted with $CHCl_3$ (3 x 10 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/acetone = 5:1) to give the product **4**.

4a: IR (KBr): 3357, 3196, 1619, 1541, 1466 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 1.00 (t, *J* = 7.5 Hz, 3H), 1.44–1.61 (m, 2H), 1.68–1.82 (m, 2H), 2.67–2.76 (m, 2H), 5.34 (br s, 1H), 6.93–7.08 (m, 3H), 7.20–7.25 (m, 1H), 8.63 (br s, 1H), 9.67 (br s, 1H); ^{13}C NMR ($CDCl_3$): δ = 13.8, 22.7, 28.8, 34.6, 94.7, 109.2, 118.5, 120.8, 122.7, 124.7, 135.4, 163.5, 170.9; HRMS (EI^+): Calcd for $C_{13}H_{16}N_2O$, M^+ 216.1263. Found m/z 216.1267.

4f: IR (KBr): 3353, 3185, 1617, 1545, 1464 cm^{-1}; ^1H NMR (acetone-d_6): δ = 6.24 (d, *J* = 7.8 Hz, 1H), 6.56–6.65 (m, 1H), 6.87–6.98 (m, 2H), 7.12 (br s, 1H), 7.54–7.69 (m, 5H), 9.71 (br s, 2H); ^{13}C NMR (acetone-d_6): δ = 95.2, 109.2, 118.3, 120.2, 123.2, 125.4, 128.3, 129.4, 130.5, 136.7, 136.9, 160.6, 171.1; HRMS (EI^+): Calcd for $C_{15}H_{12}N_2O$, M^+ 236.0950. Found m/z 236.0953.

4k: IR (KBr): 3357, 3187, 1653, 1617, 1561, 1464 cm^{-1}; ^1H NMR (acetone-d_6): δ = 6.54 (d, *J* = 7.8 Hz, 1H), 6.64–6.75 (m, 1H), 6.90–6.99 (m, 2H), 7.07 (br s, 1H), 7.36–7.42 (m, 1H), 7.72–7.79 (m, 1H), 7.86–7.93 (m, 1H), 9.70 (br s, 2H); ^{13}C NMR (acetone-d_6): δ = 96.8, 110.3, 119.5, 121.4, 124.4, 126.5, 128.0, 128.6, 129.0, 138.0, 138.3, 156.7, 172.3; HRMS (EI^+): Calcd for $C_{13}H_{10}N_2OS$, M^+ 242.0514. Found m/z 242.0513.

4n: IR (KBr): 3445, 3187, 1647, 1613, 1534, 1466 cm^{-1}; ^1H NMR ($CDCl_3$): δ = 1.02 (t, *J* = 7.2 Hz, 3H), 1.46–1.62 (m, 2H), 1.66–1.81 (m, 2H), 2.63–2.73 (m, 2H), 5.45 (br s, 1H), 6.86 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.00 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.15 (d, *J* = 1.8 Hz, 1H), 8.91 (br s, 1H), 9.75 (br s, 1H); ^{13}C NMR ($CDCl_3$): δ = 13.8, 22.6, 28.6, 34.4, 94.2, 110.0, 118.3, 122.2, 126.0, 126.1, 133.8, 164.6, 171.0; HRMS (EI^+): Calcd for $C_{13}H_{15}ClN_2O$, M^+ 250.0873. Found m/z 250.0878.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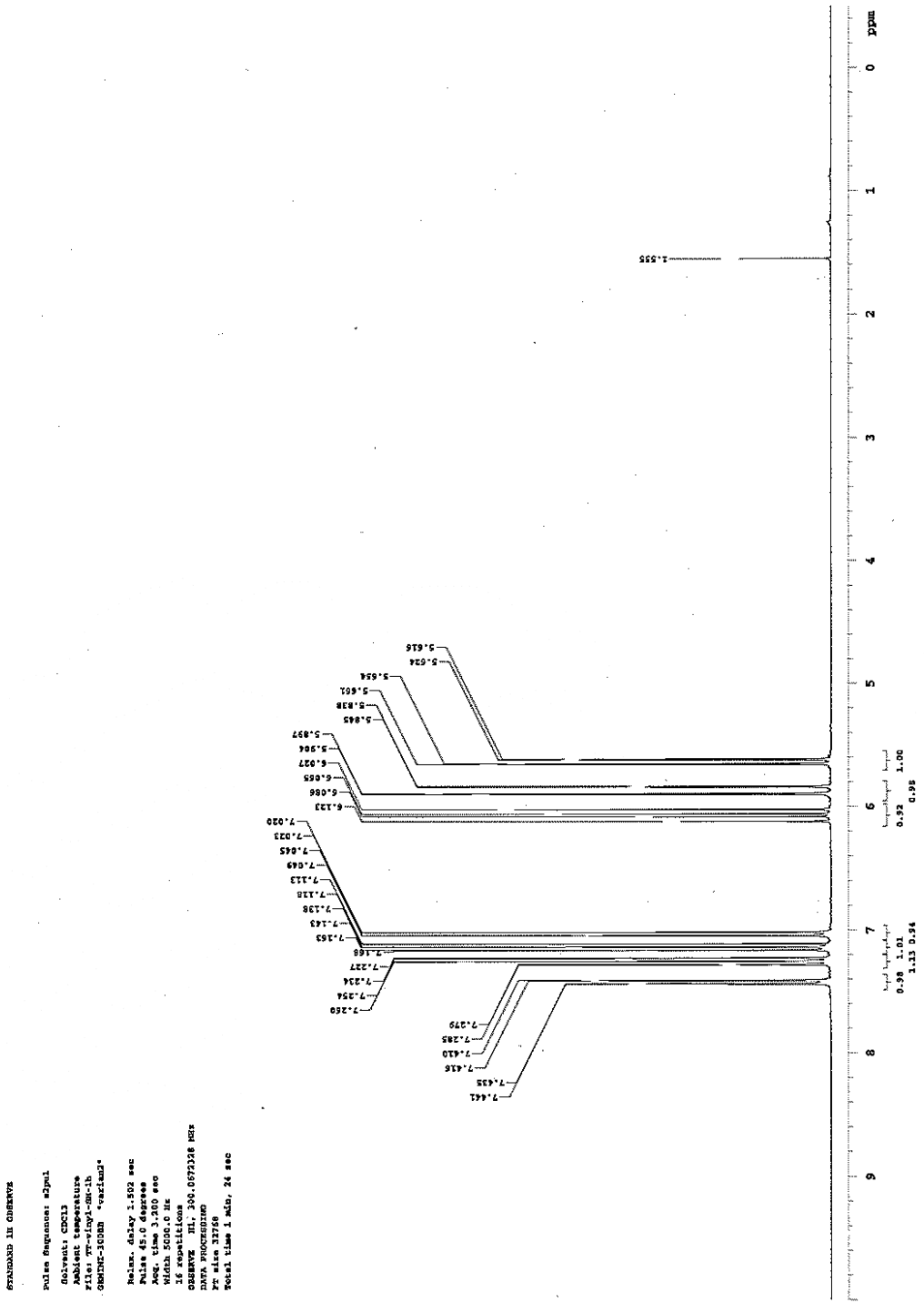
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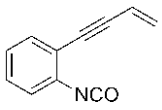

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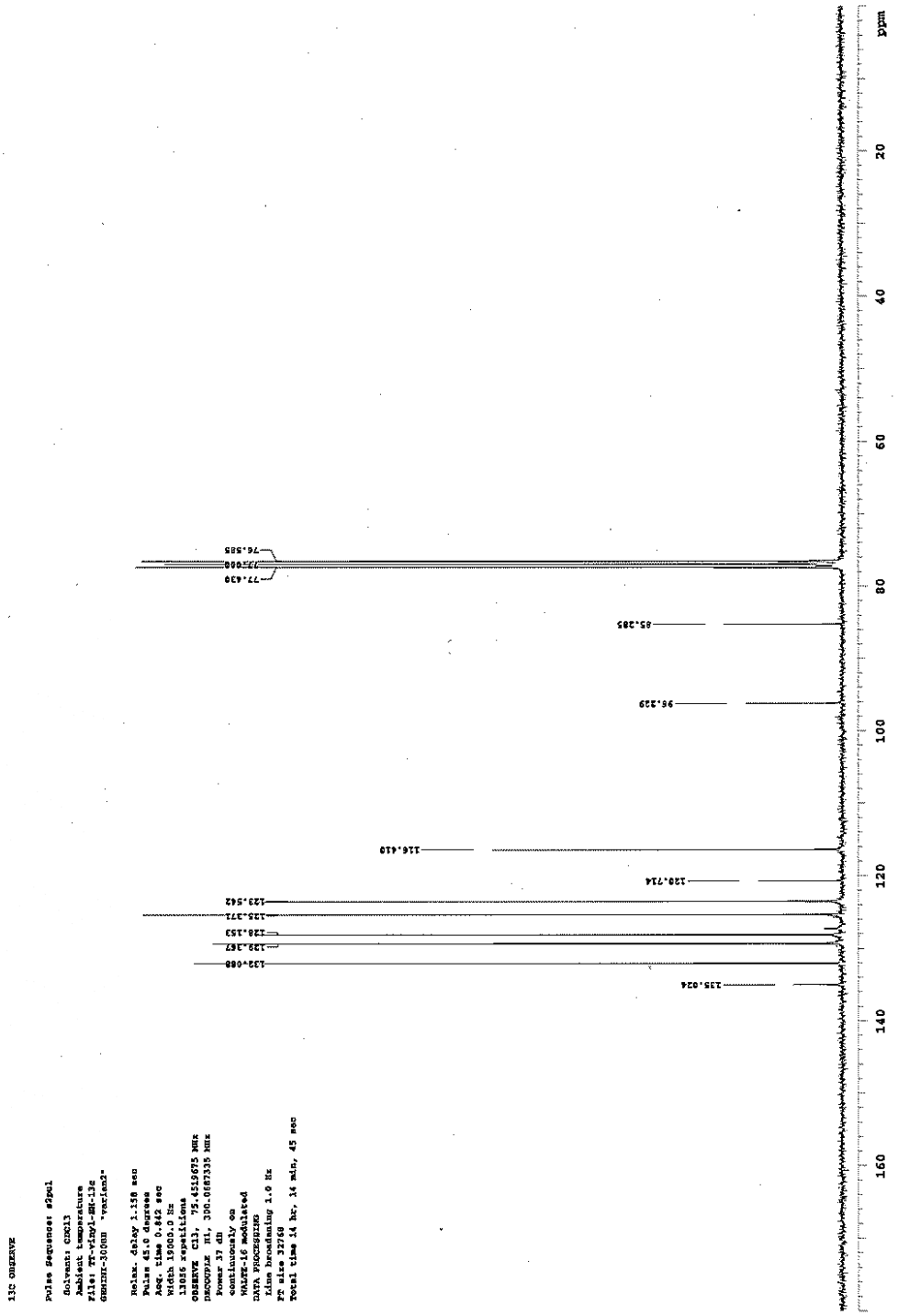

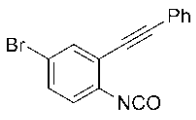
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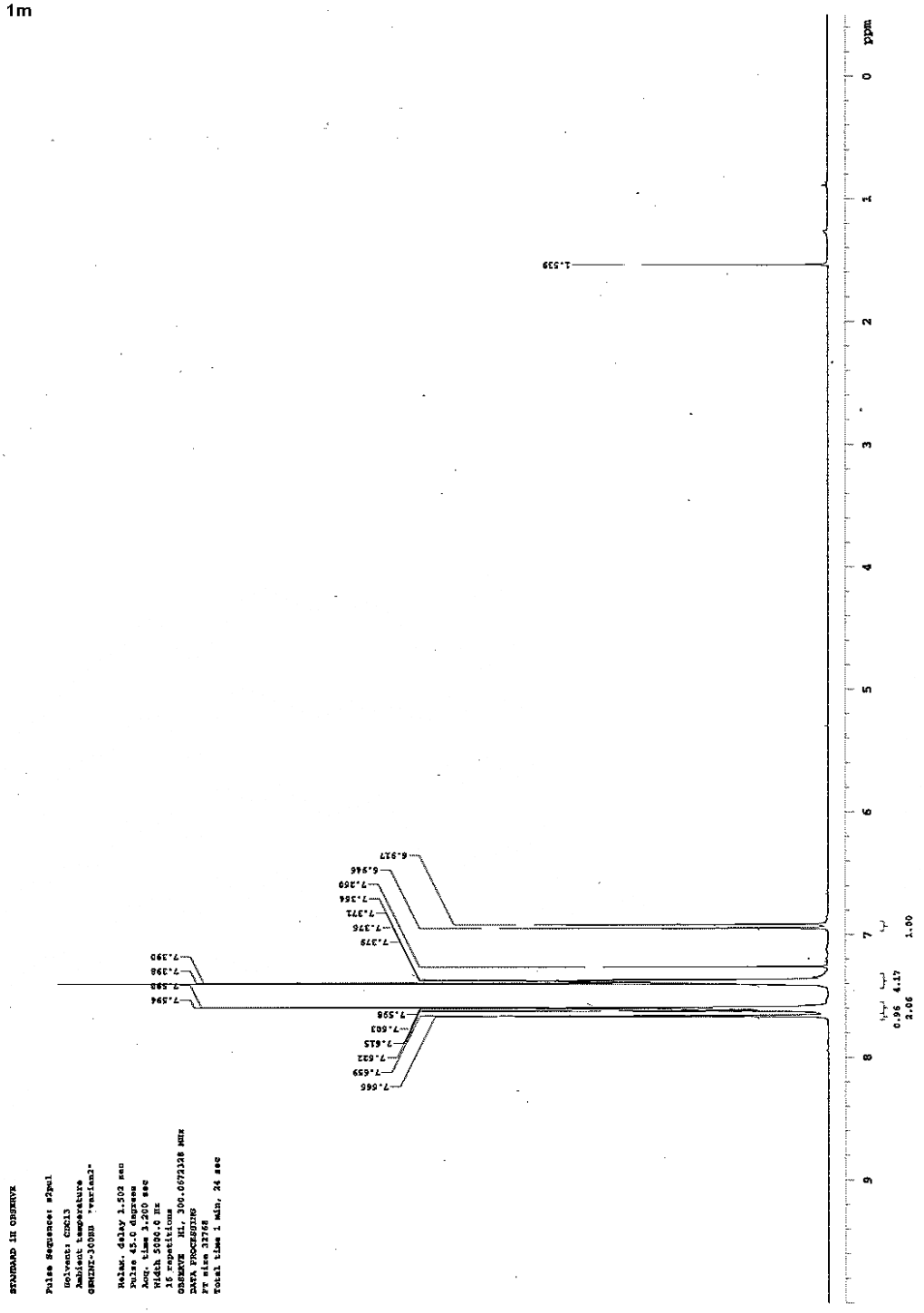
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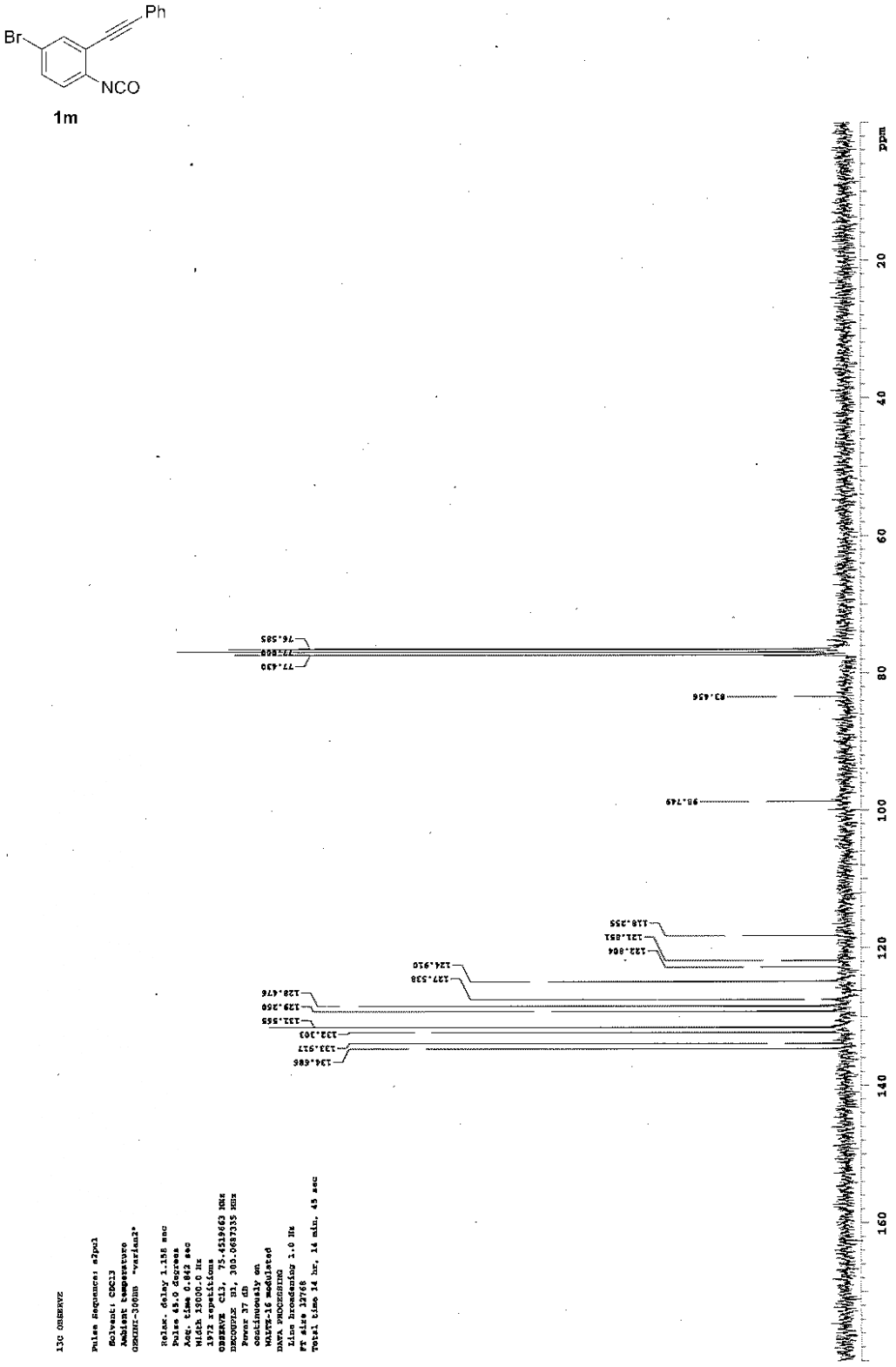
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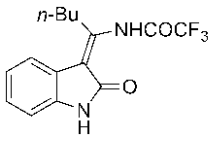
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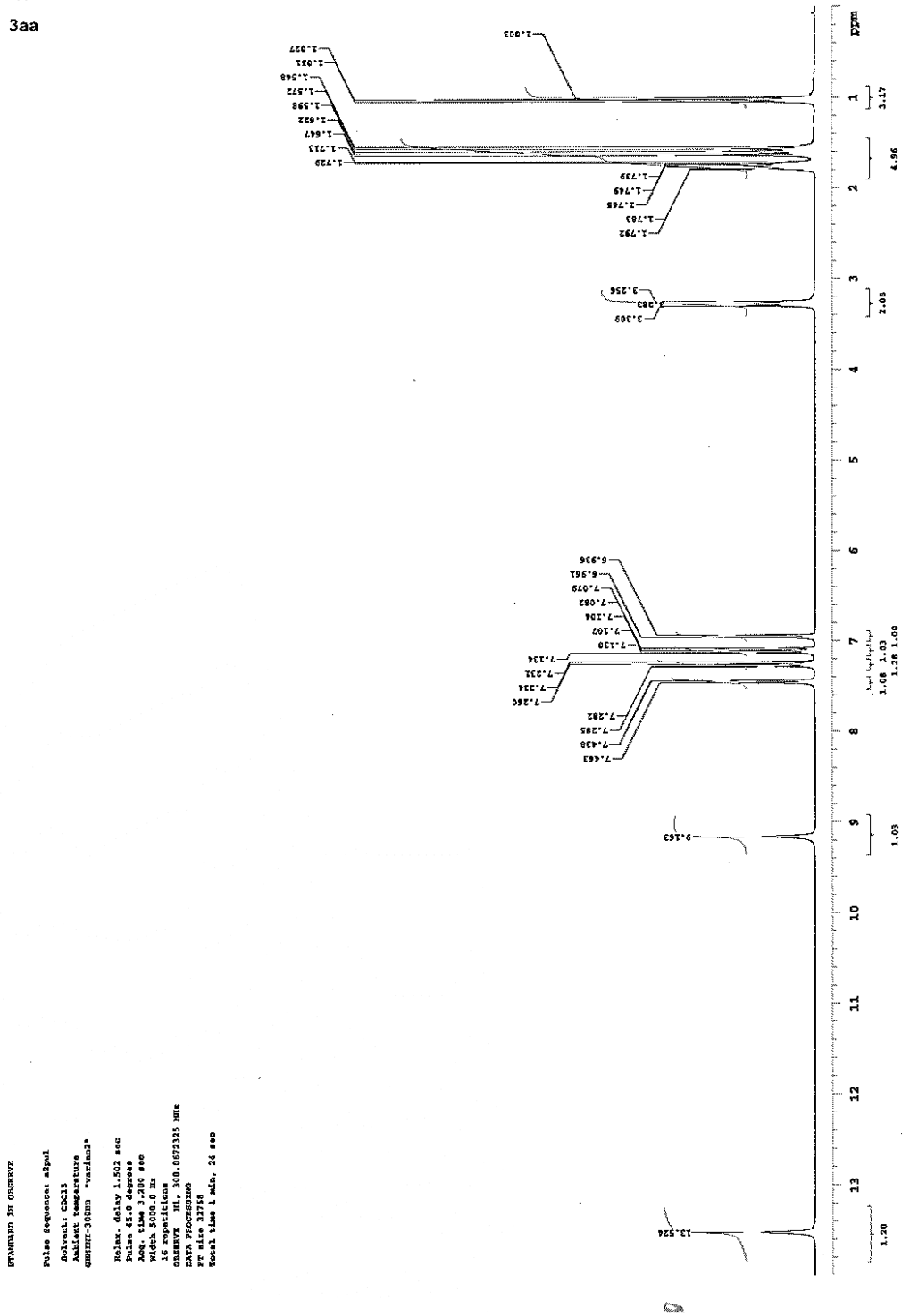
H

3aa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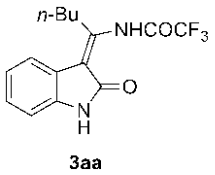
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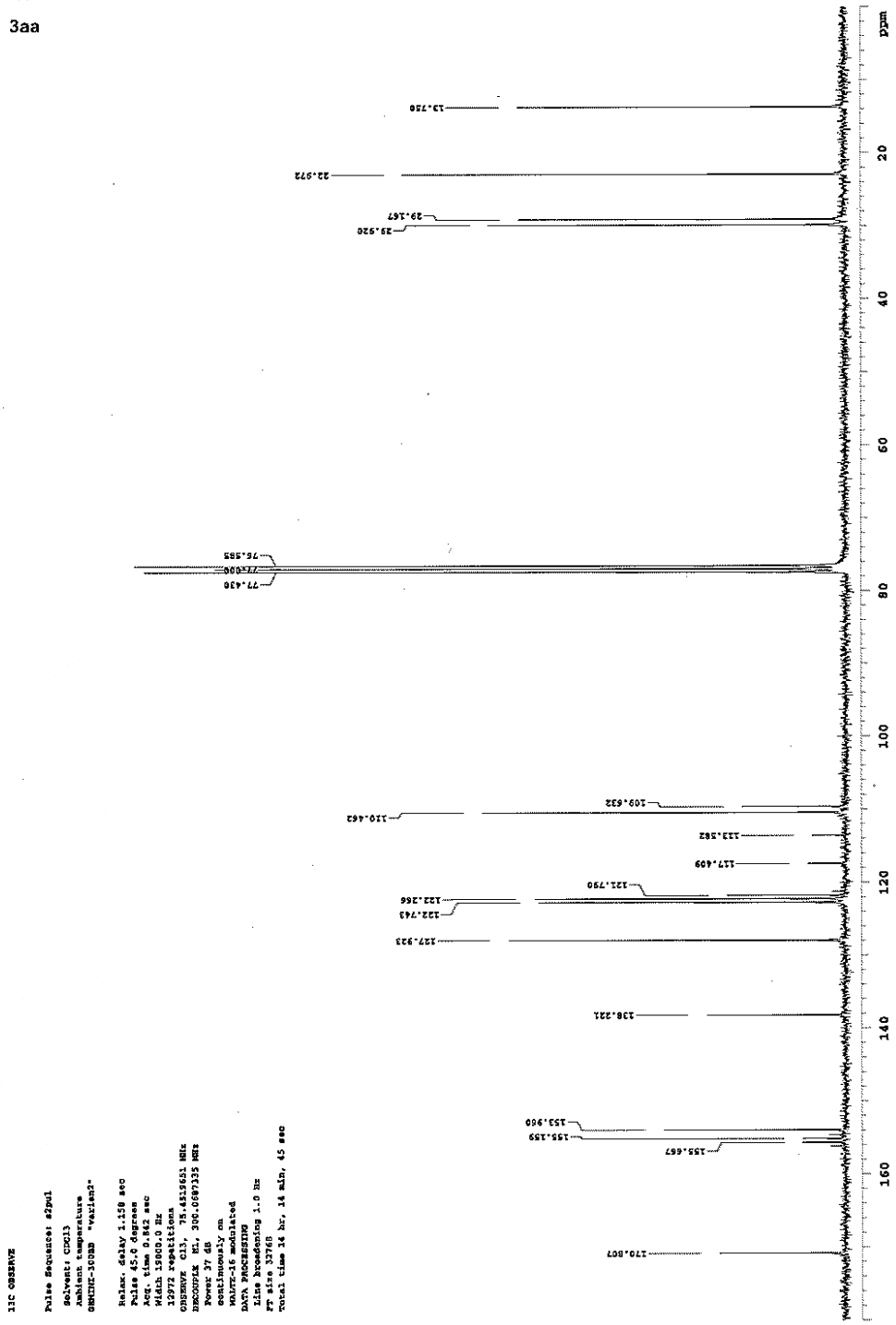
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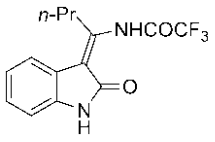
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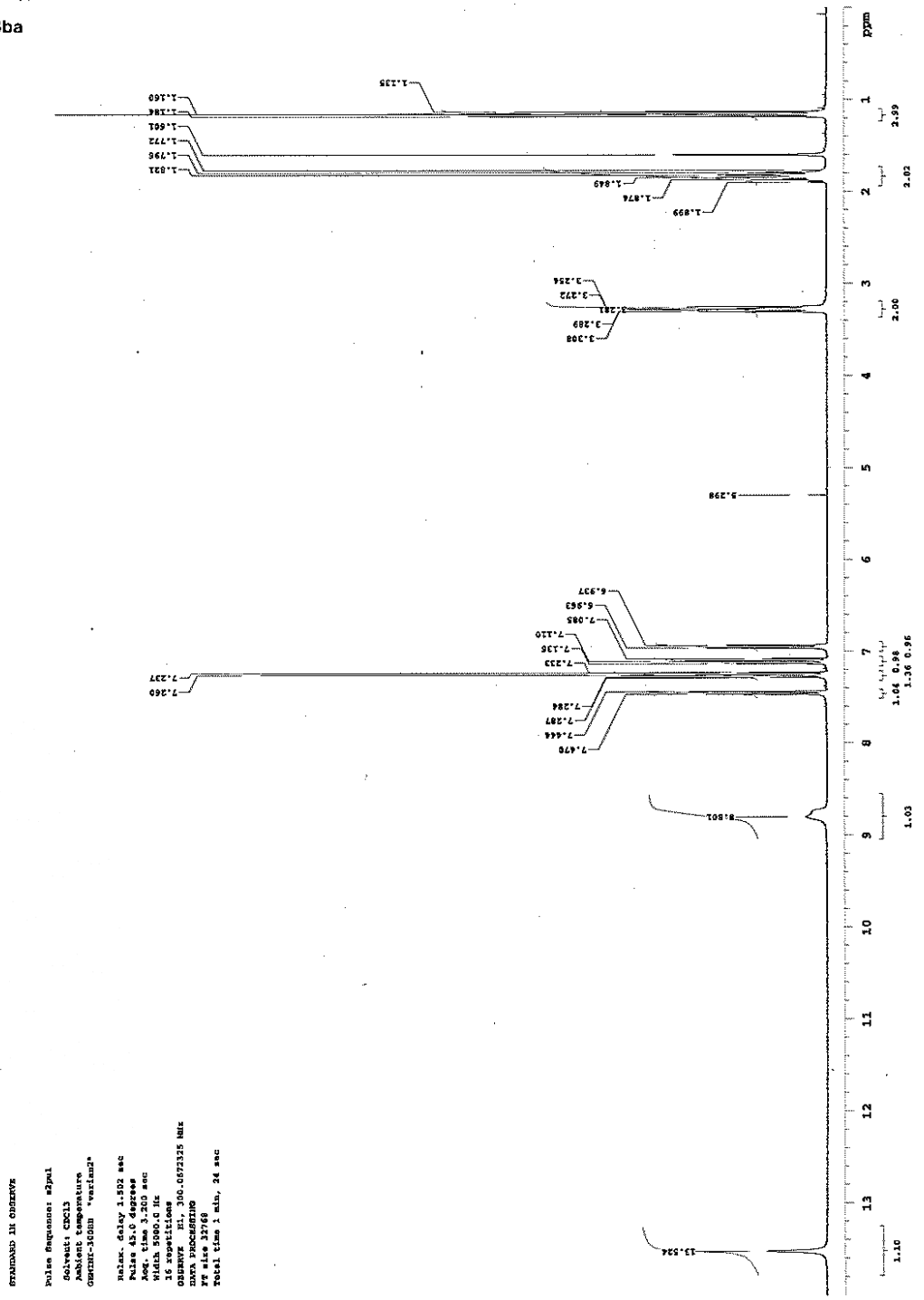
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Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
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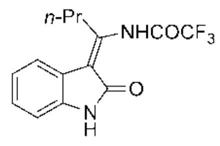
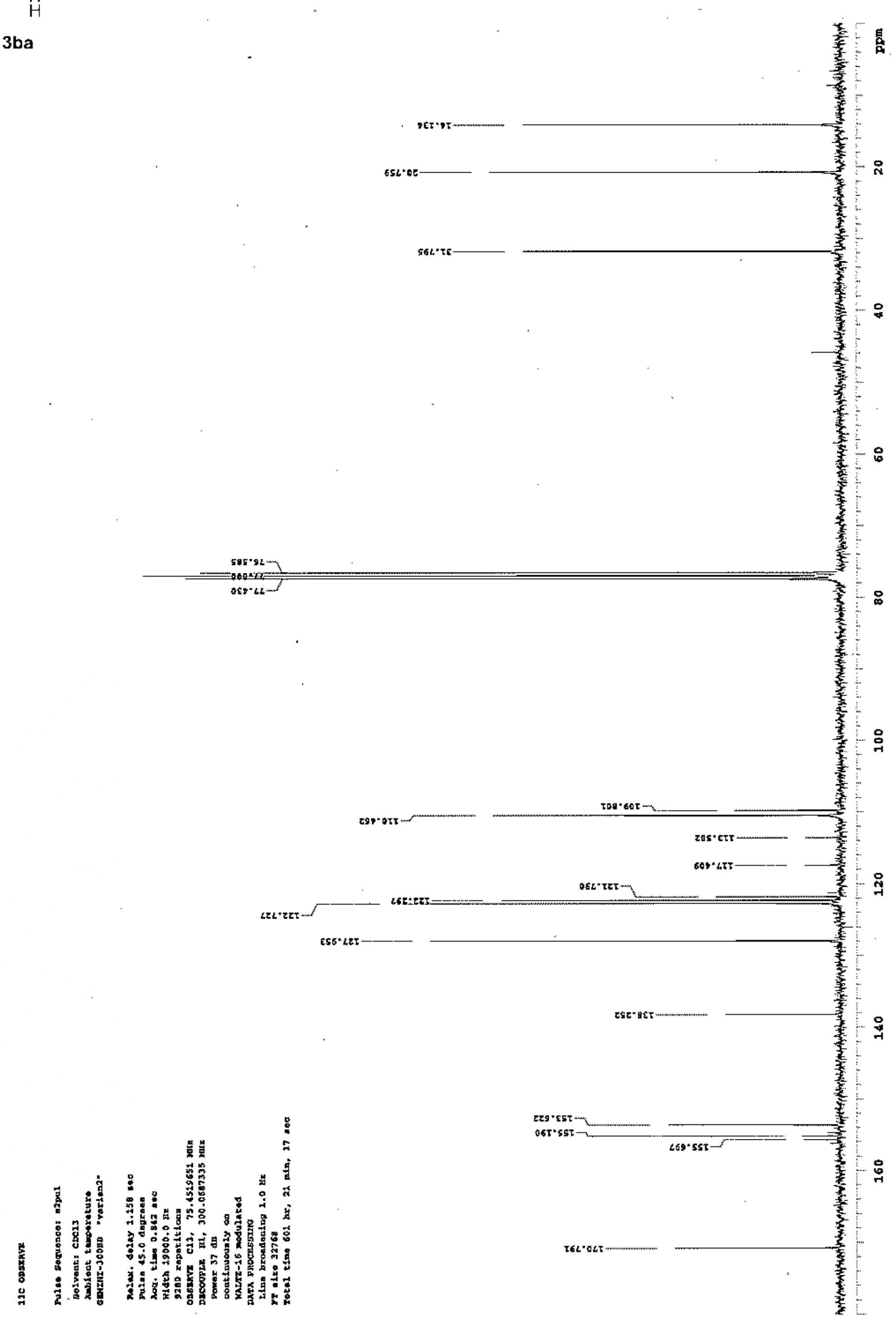
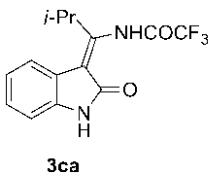


n-Pr
NHCOCF3
O
N
H

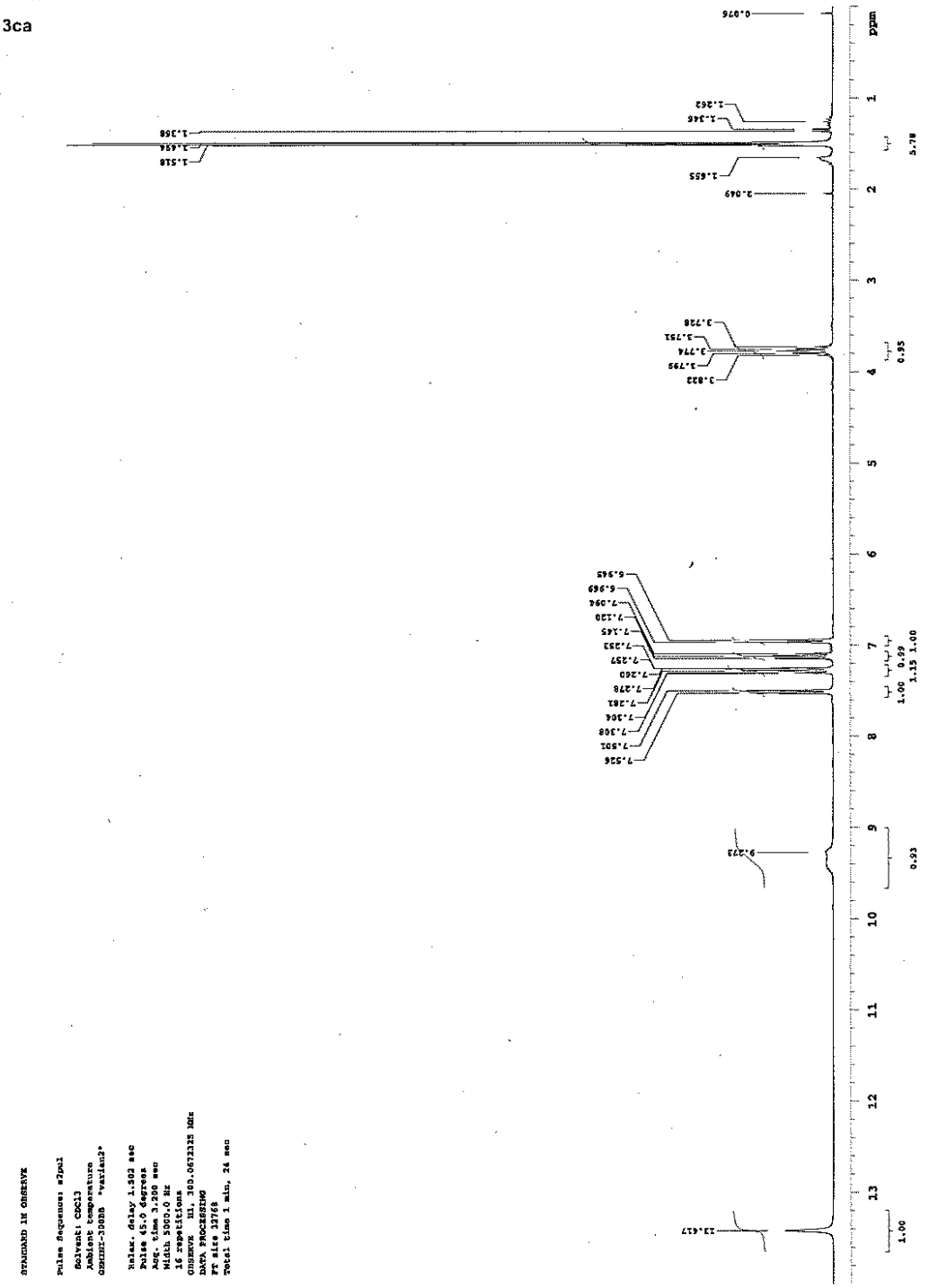
3ba



14.134
20.759
31.795
76.585
77.000
77.430
109.801
110.462
113.582
117.409
121.790
122.397
122.727
127.953
138.252
153.622
155.190
155.697
170.791
ppm
20
40
60
80
100
120
140
160



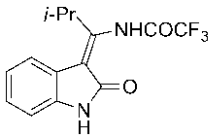
3ca



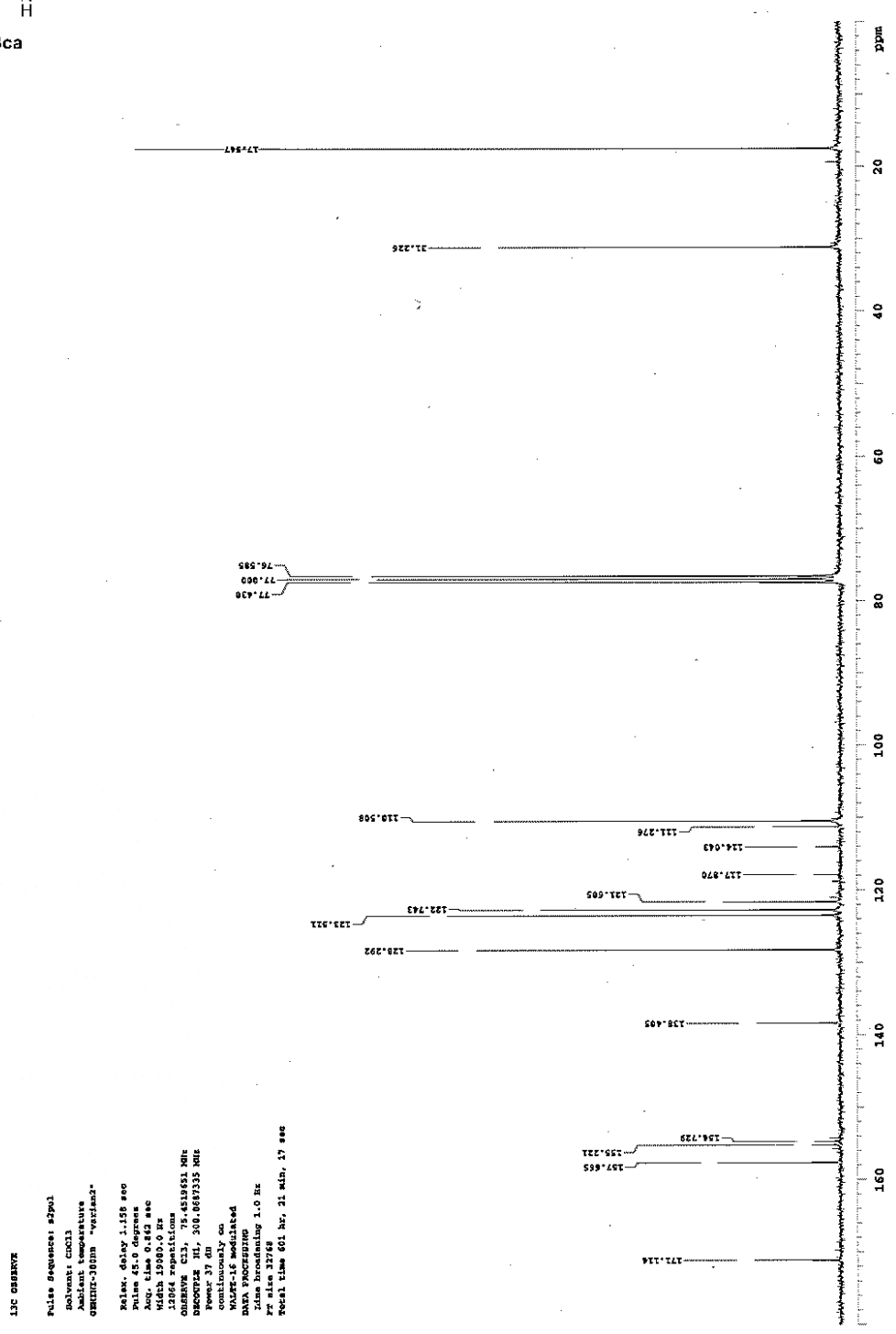
STANDARD 1H OBSERVE

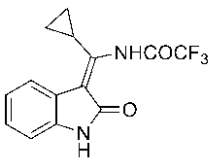
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"

Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5003.0 Hz
16 repetitions
OBSERVE H1, 300.0672325 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec

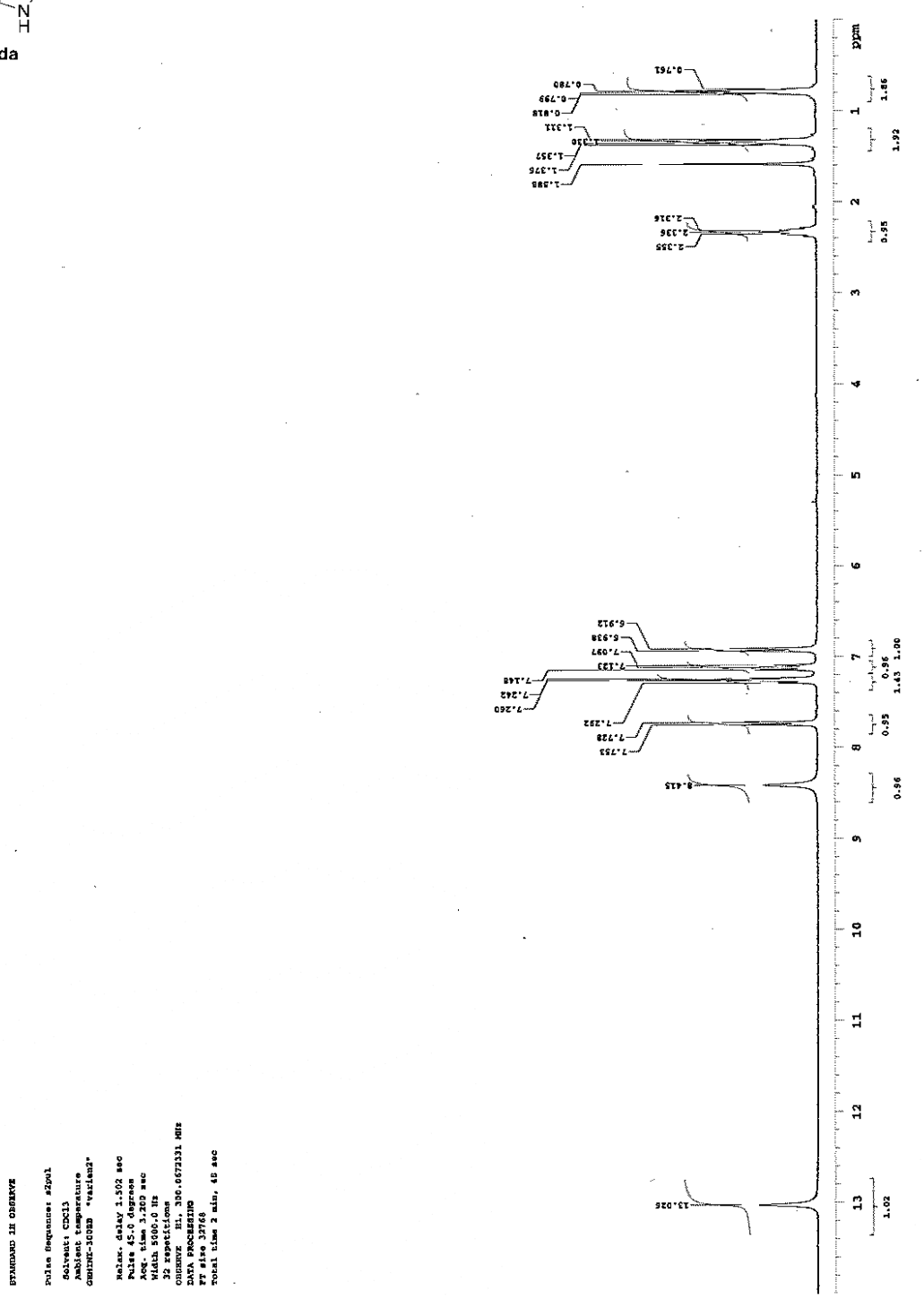

i-Pr
NHCOCF3
O
N
H

3ca


13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "variana"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19980.0 Hz
12064 repetitions
OBSERVE C13, 75.4519651 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 601 hr, 21 min, 17 sec
17.547
31.336
76.585
77.000
77.436
120.508
121.276
124.043
127.870
131.605
132.743
133.521
138.292
138.405
156.729
157.221
157.665
172.134
20
40
60
80
100
120
140
160
ppm



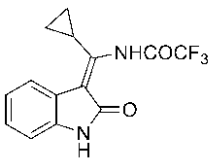
3da



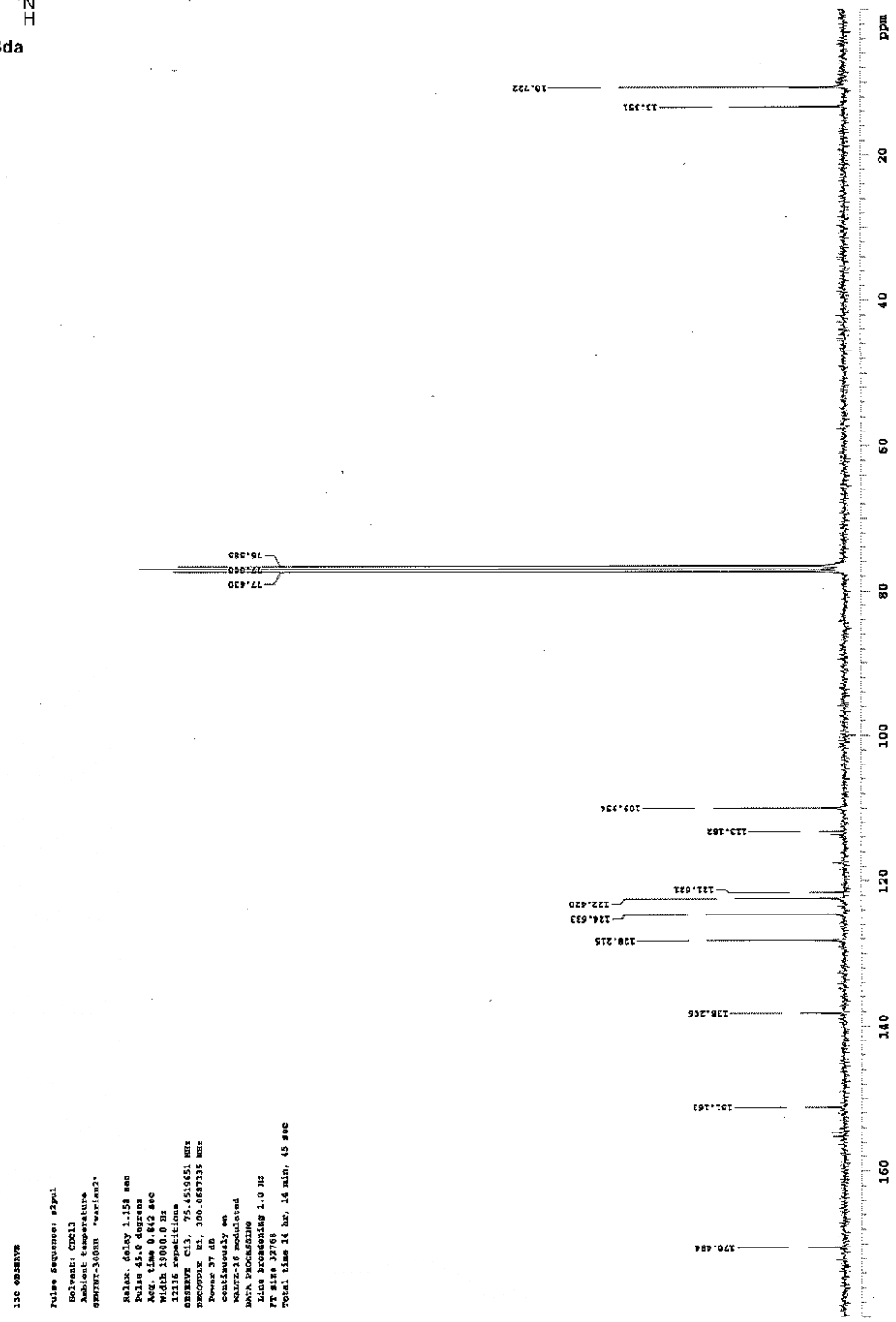
STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"

Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.260 sec
Width 5000.0 Hz
32 repetitions
OBSERVE H1, 300.0672331 MHz
DATA PROCESSING
FT size 32768
Total time 2 min, 48 sec



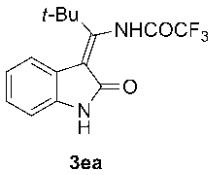
3da



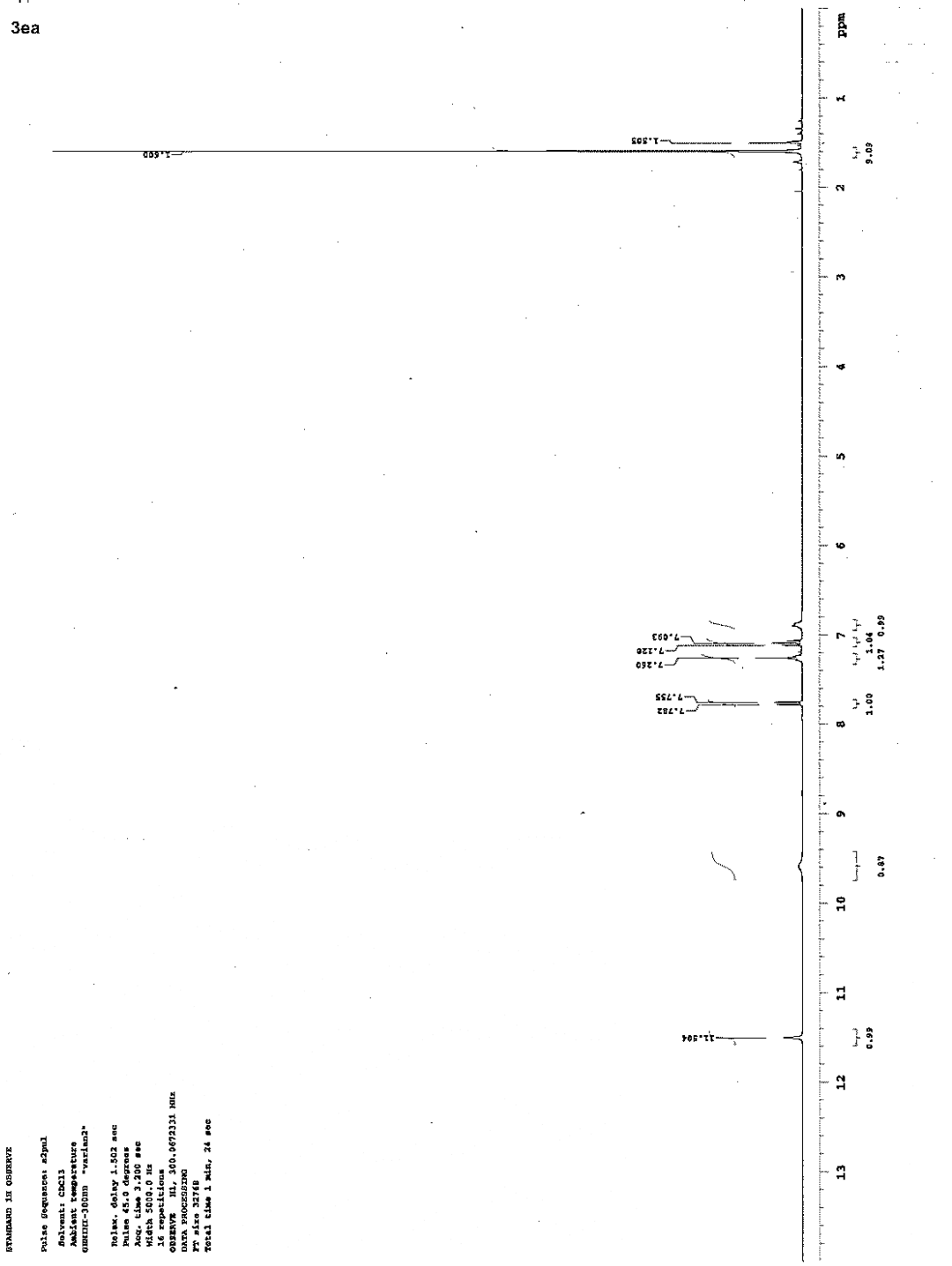
13C OBSERVE

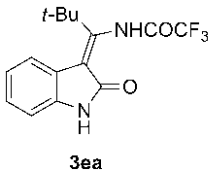
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"

Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 15900.0 Hz
12136 repetitions
OBSERVE C13, 75.4519651 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 14 hr, 14 min, 45 sec

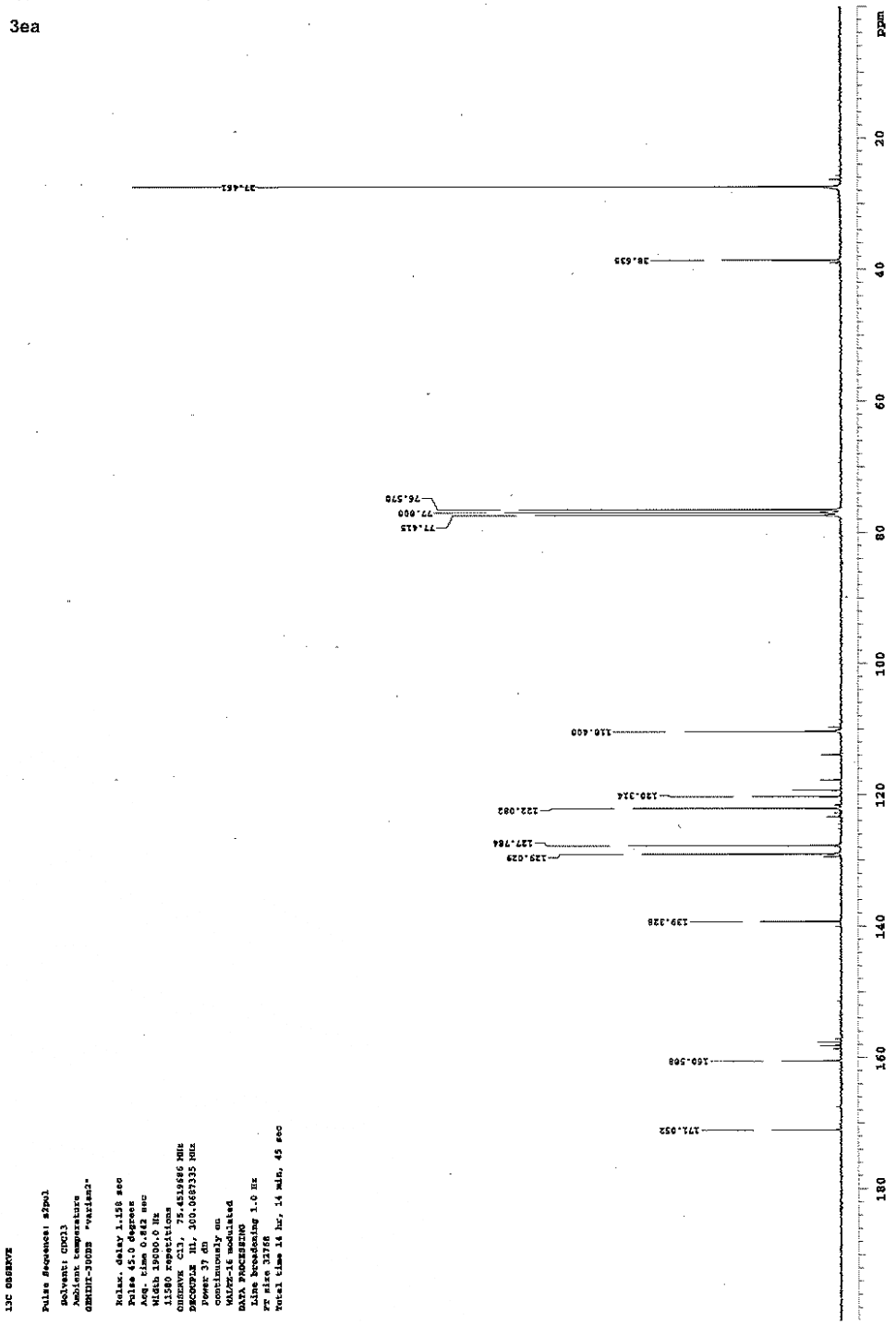

t-Bu
NHCOCF3
O
N
H

3ea


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 5010.0 Hz
16 repetitions
OBSERVE H1, 300.0672331 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec
1.659
1.505
7.093
7.128
7.260
7.755
7.782
11.964
ppm



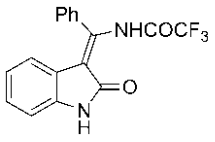
3ea



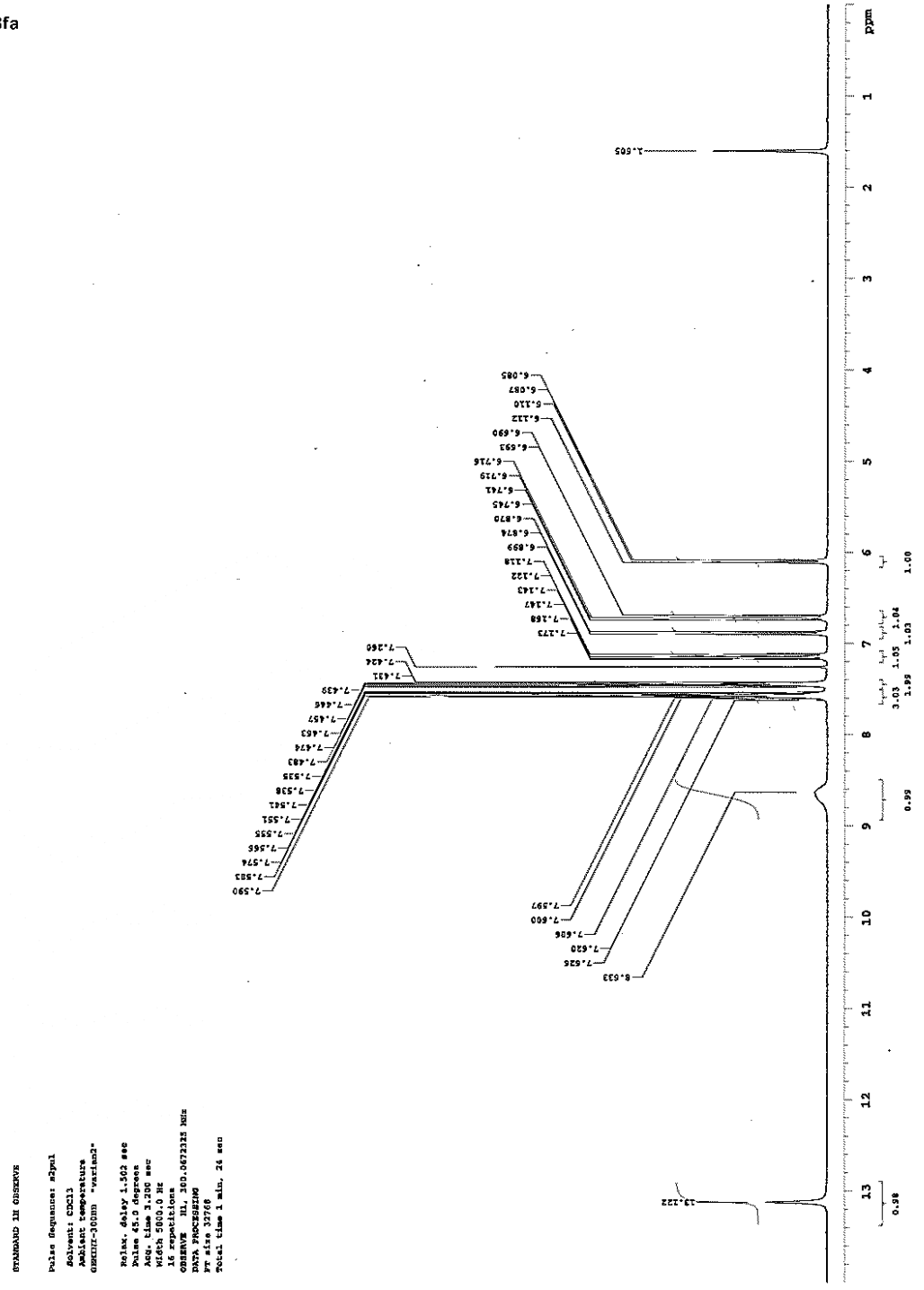
13C OBSERVE

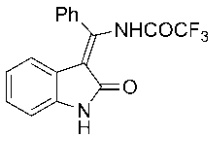
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "variana"

Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
11580 repetitions
OBSERVE C13, 75.4519686 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 14 hr, 14 min, 45 sec

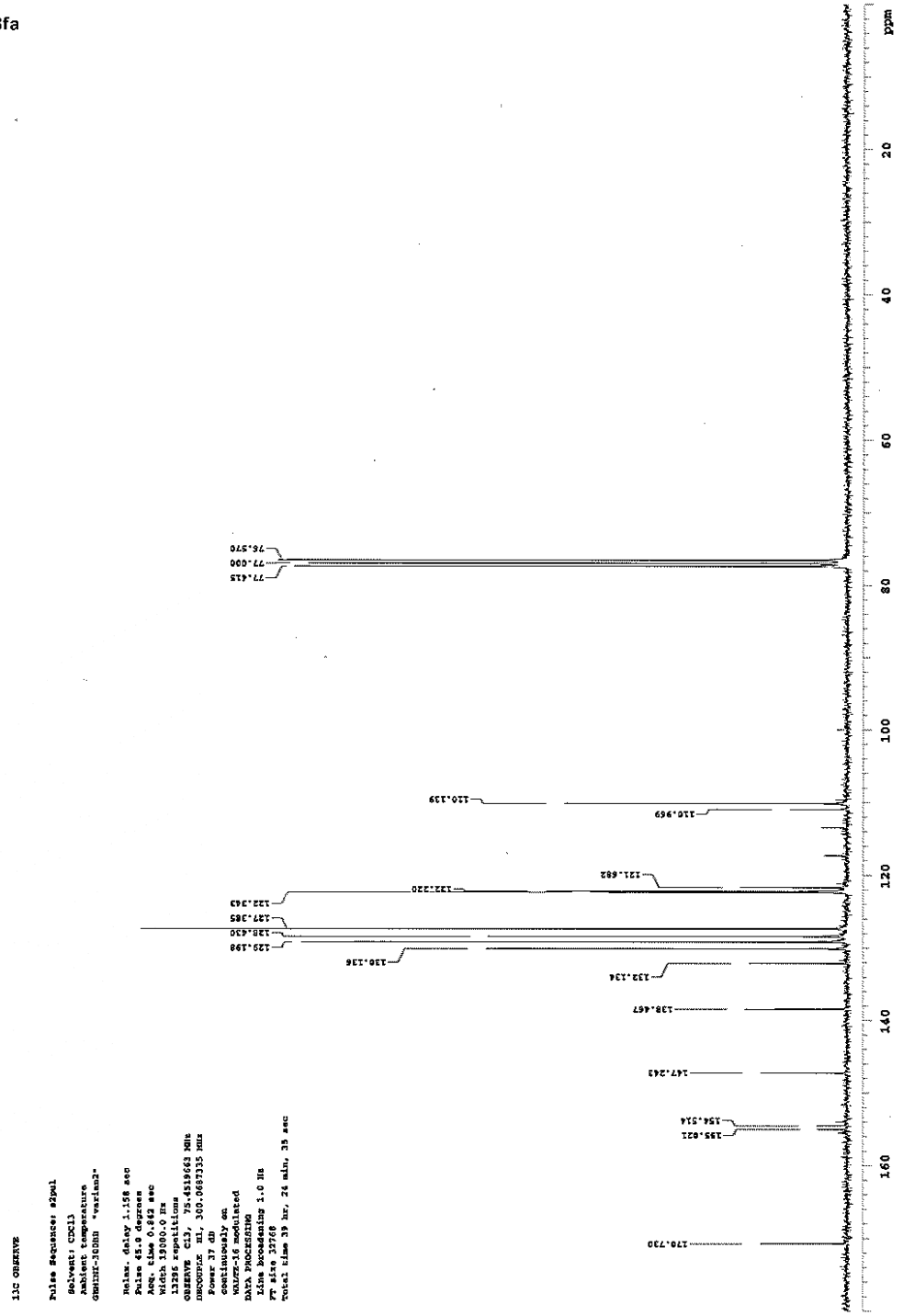

Ph
NHCOCF3
O
N
H

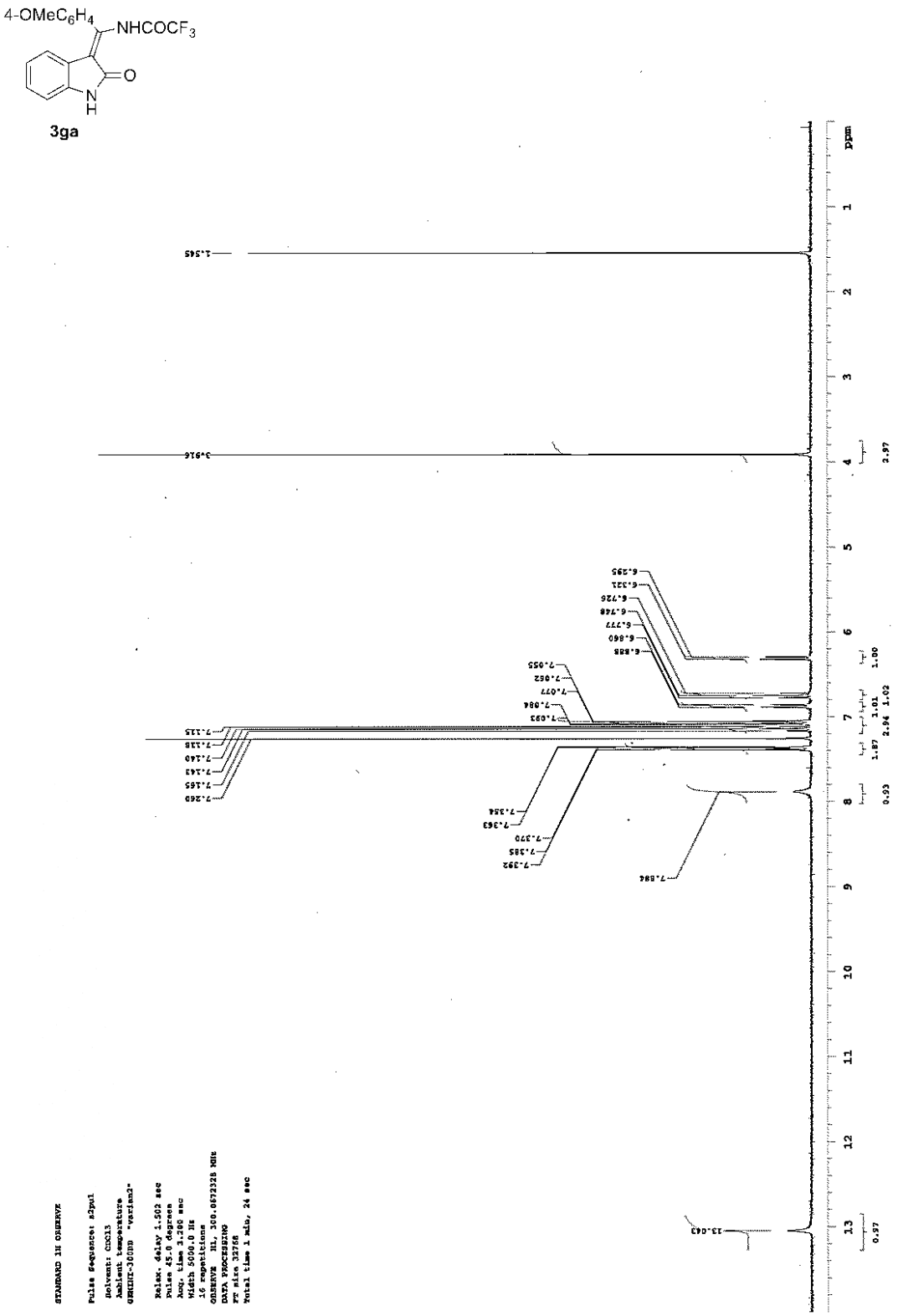
3fa

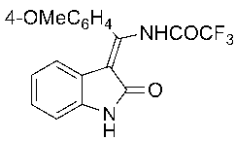

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672325 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec
ppm


Ph
NHCOCF3
O
N
H

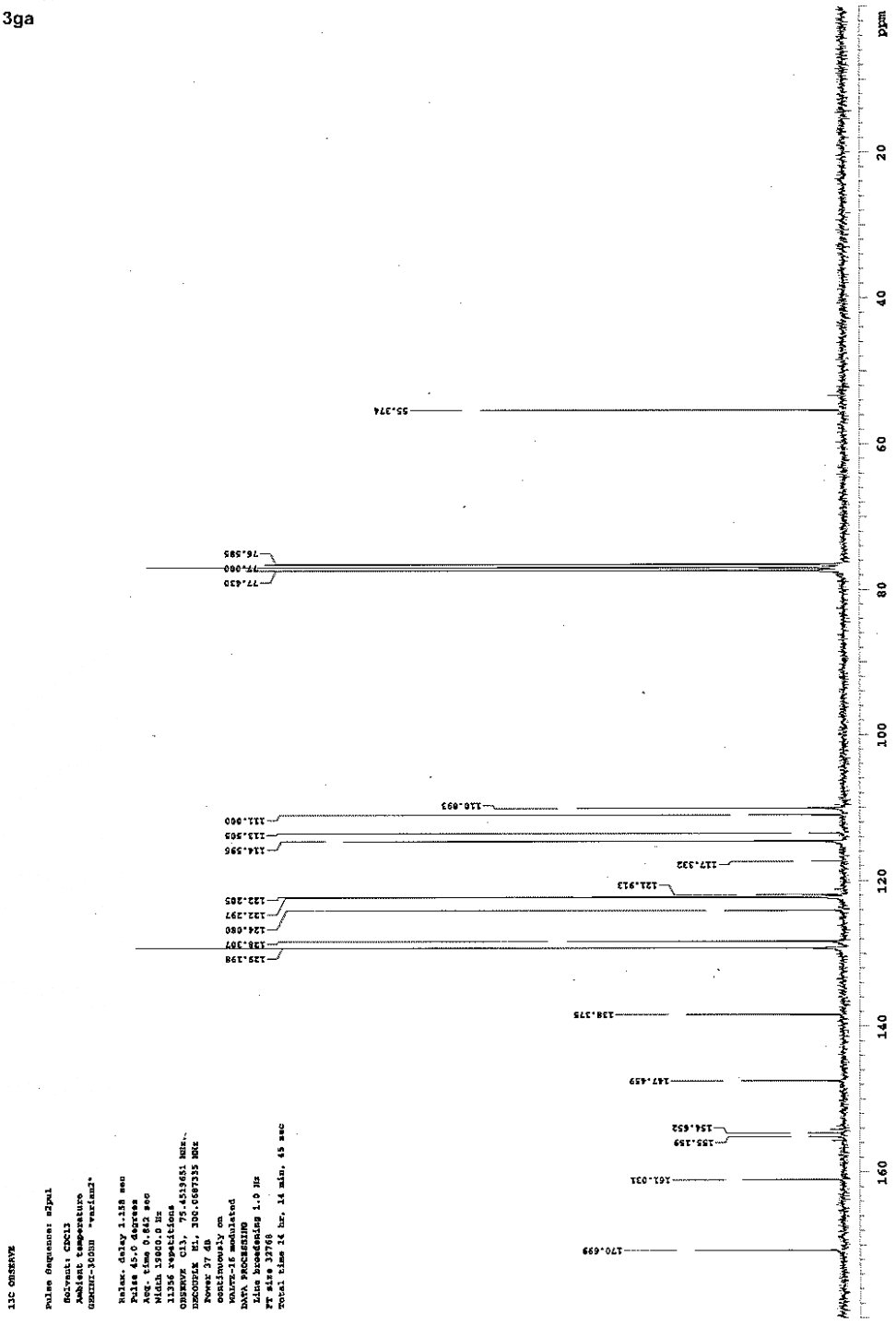
3fa

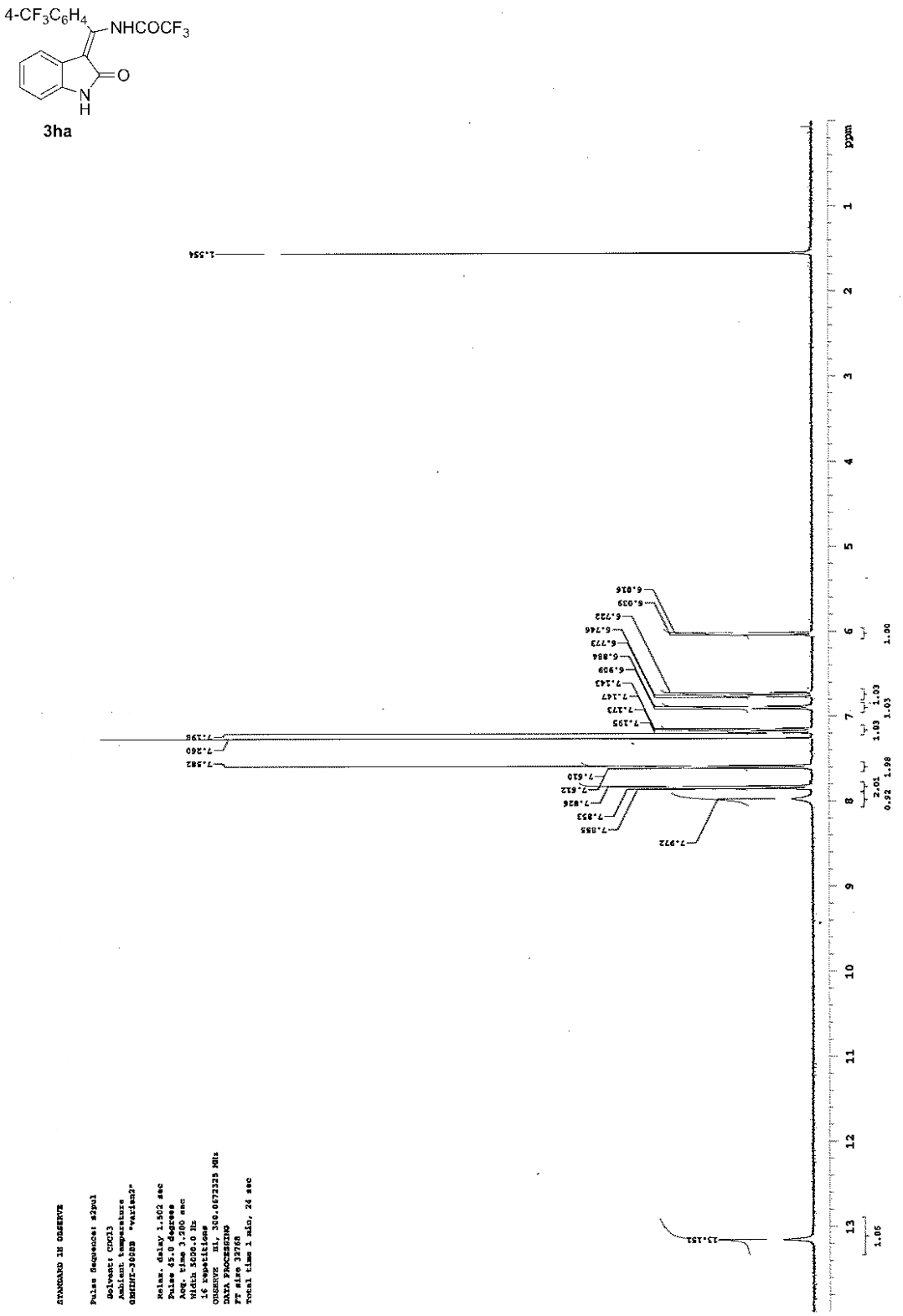

ppm
20
40
60
80
100
120
140
160
176.730
154.514
147.243
138.467
132.134
130.136
129.198
128.430
127.385
122.343
121.682
110.969
110.139
77.415
77.000
76.576

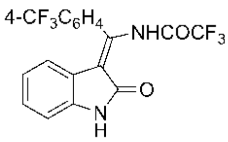

4-OMeC6H4
NHCOCF3
O
N
H
3ga
STANDARD 1H OBSERVE
Pulse sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0572325 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec
ppm


4-OMeC6H4
NHCOCF3
O
N
H

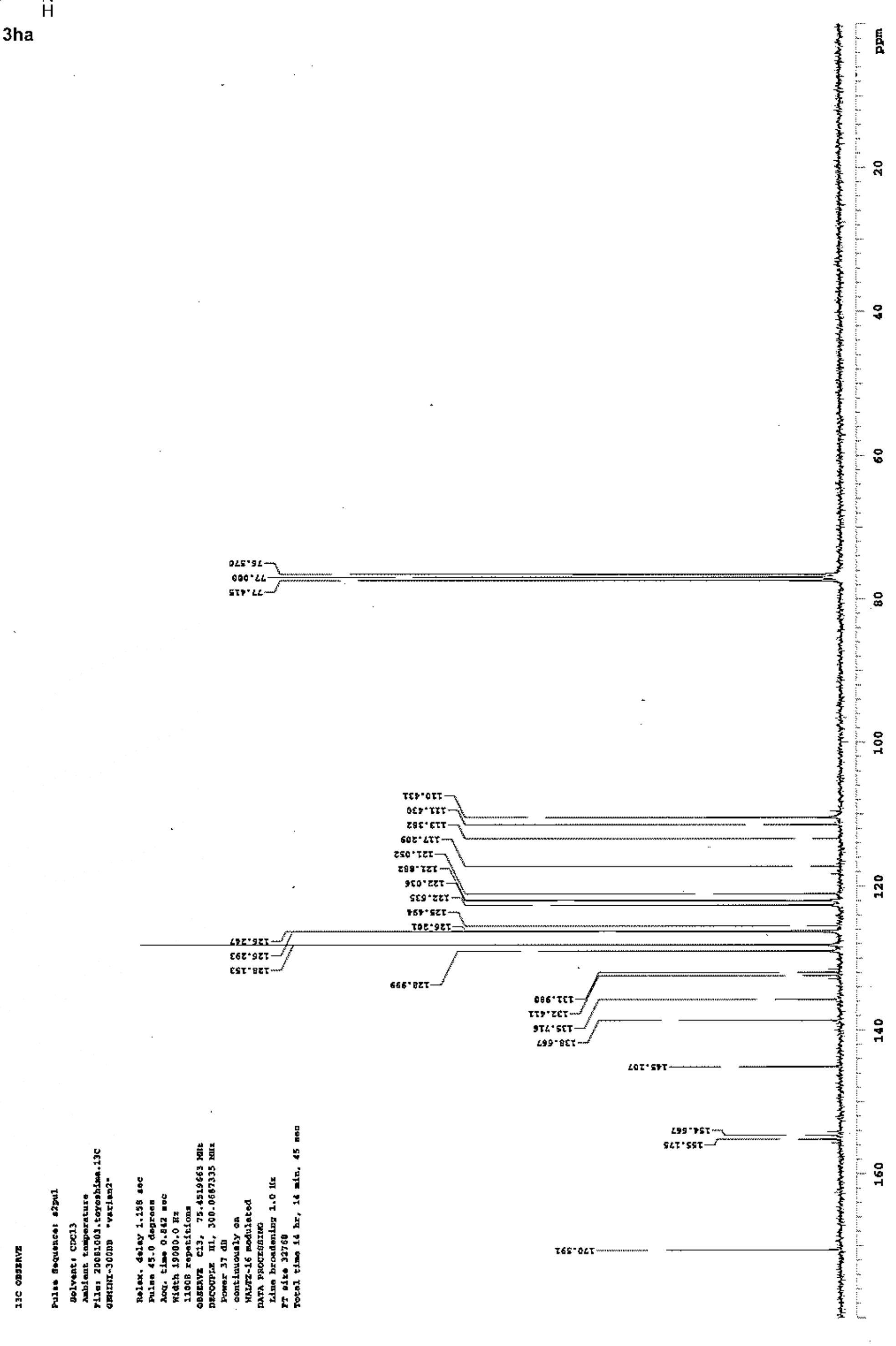
3ga

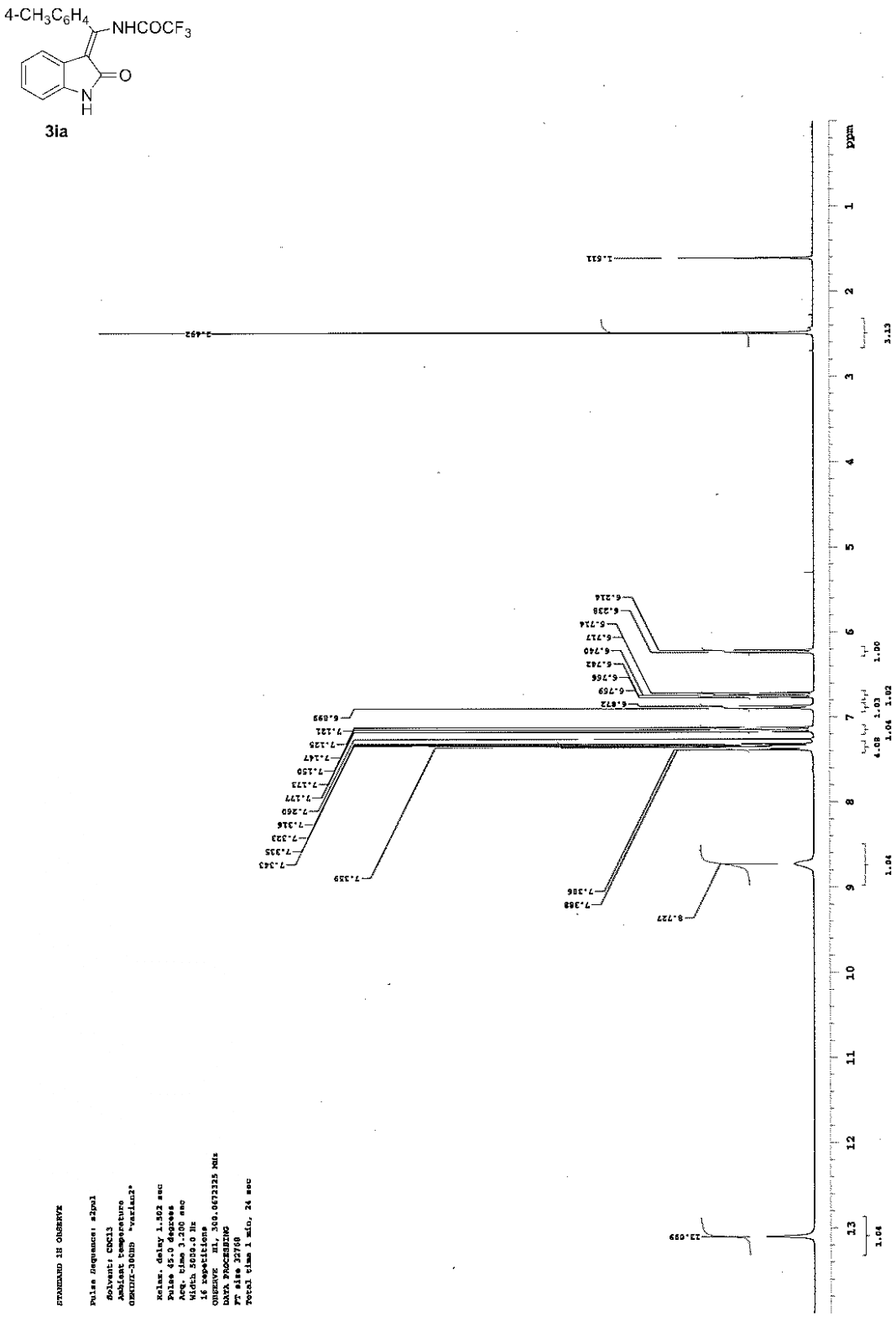

55.374
76.585
77.000
77.430
118.093
121.060
123.305
124.596
127.323
132.913
132.285
132.397
134.080
136.307
135.198
138.375
147.459
154.652
155.159
161.031
170.699
ppm
20
40
60
80
100
120
140
160

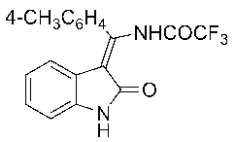

4-CF3C6H4
NHCOCF3
O
N
H
3ha
1.554
6.816
6.839
6.722
6.746
6.773
6.884
6.909
7.143
7.147
7.173
7.195
7.198
7.360
7.382
7.610
7.624
7.826
7.853
7.855
7.972
13.151
ppm
1.00
1.03
1.03
2.01
0.92
1.98
1.05
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.260 sec
Width 5026.0 Hz
16 repetitions
OBSERVE H1, 300.0672325 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec


4-CF3C6H4
NHCOCF3
O
N
H

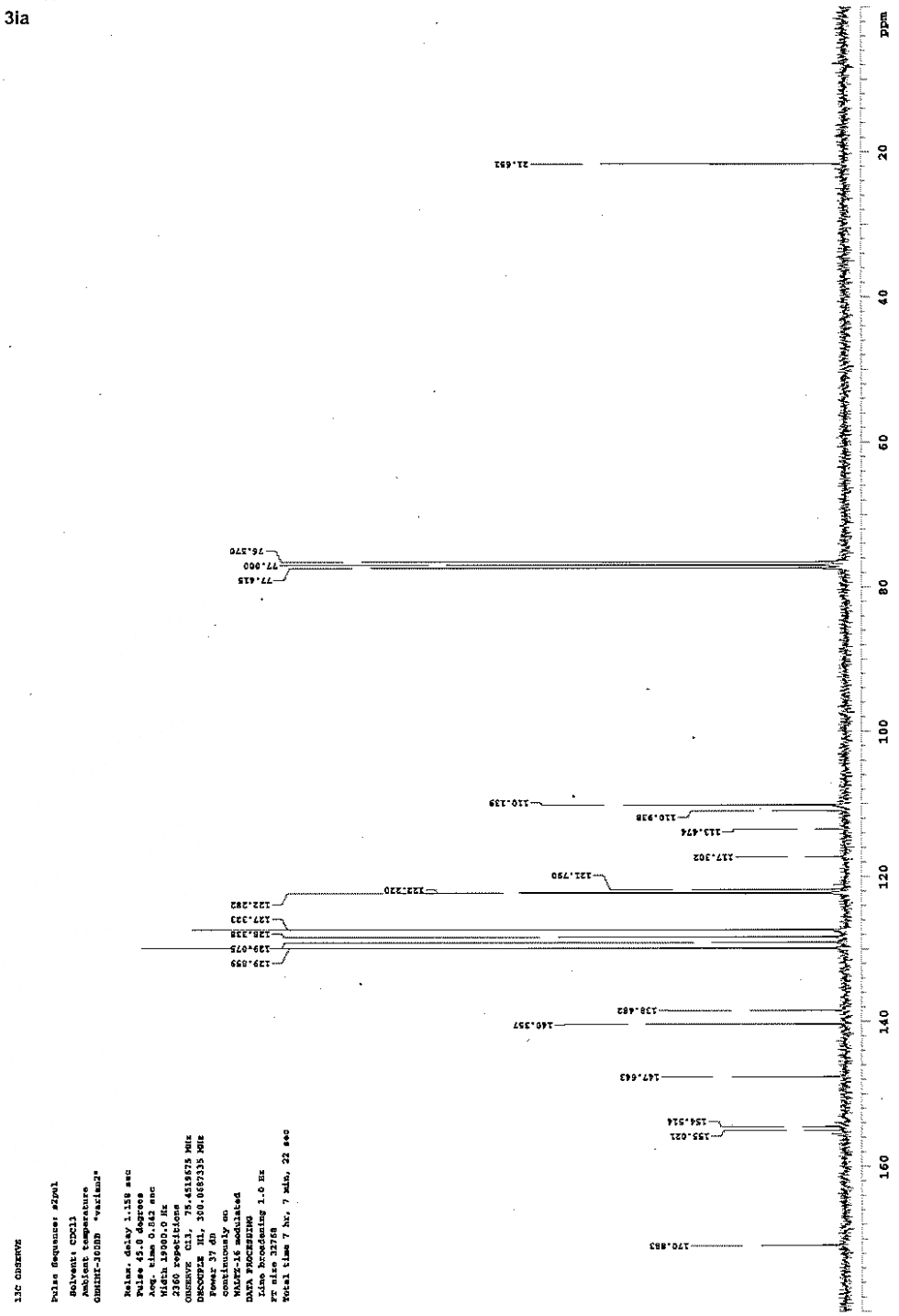
3ha

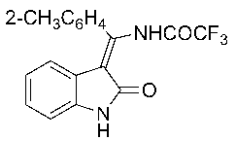

13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 20081003.toyoshima.13C
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
11008 repetitions
OBSERVE C13, 75.4519663 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 14 hr, 14 min, 45 sec
170.591
155.175
154.667
145.207
138.667
135.716
132.411
131.980
128.999
128.153
126.293
126.247
126.201
125.454
122.635
122.036
121.882
121.052
117.209
113.382
111.430
110.431
77.415
77.080
76.570
160
140
120
100
80
60
40
20
ppm


4-CH3C6H4
NHCOCF3
O
N
H
3ia
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 5059.0 Hz
16 repetitions
OBSERVE H1, 300.0673325 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec
ppm

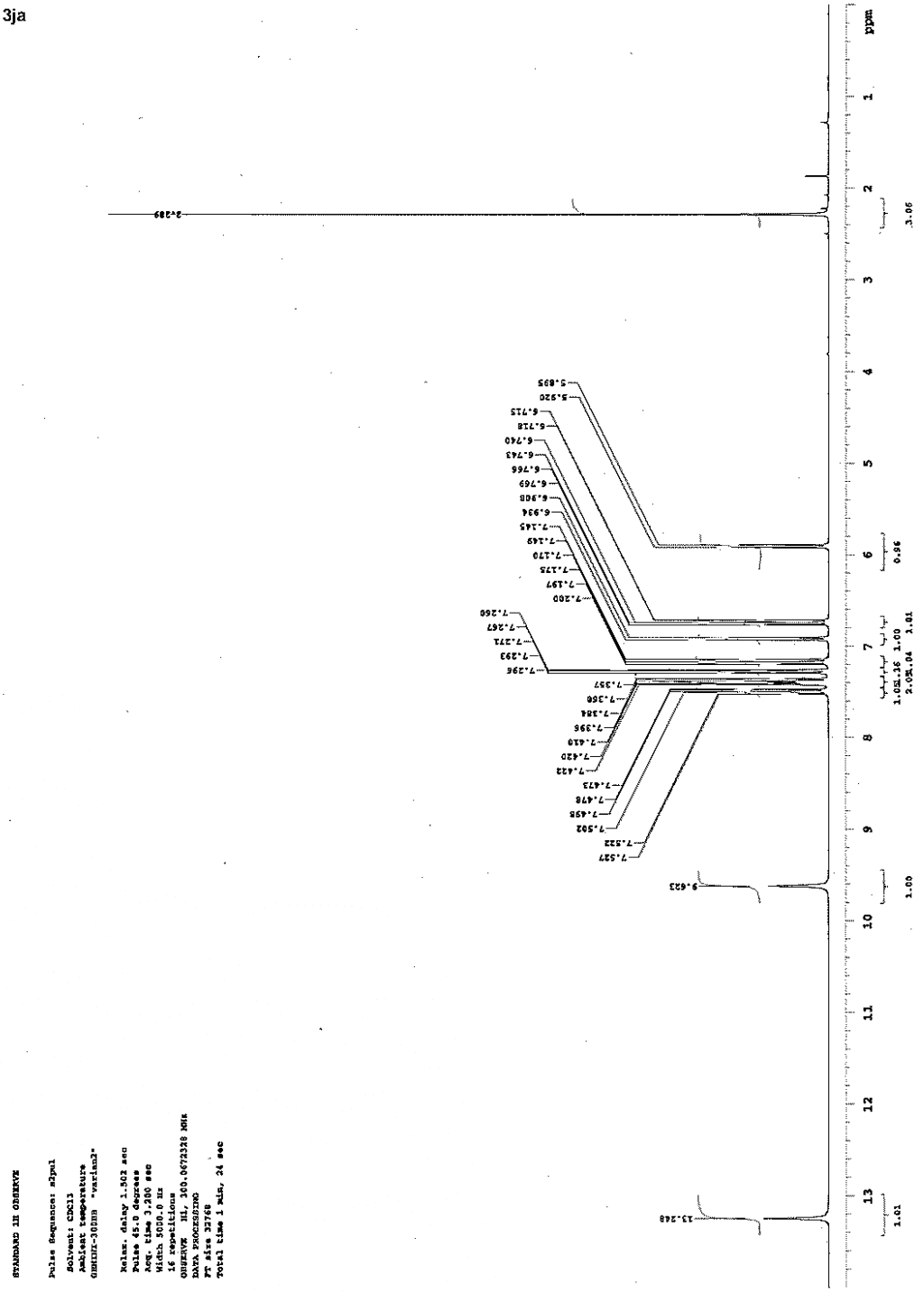

4-CH3C6H4
NHCOCF3
O
N
H

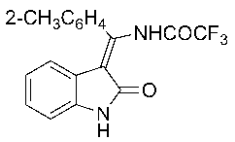
3ia


21.692
76.570
77.000
77.435
110.139
110.938
113.474
117.302
121.750
123.320
127.282
127.323
128.338
129.075
129.859
138.482
140.357
147.663
154.514
155.021
170.883
ppm
20
40
60
80
100
120
140
160

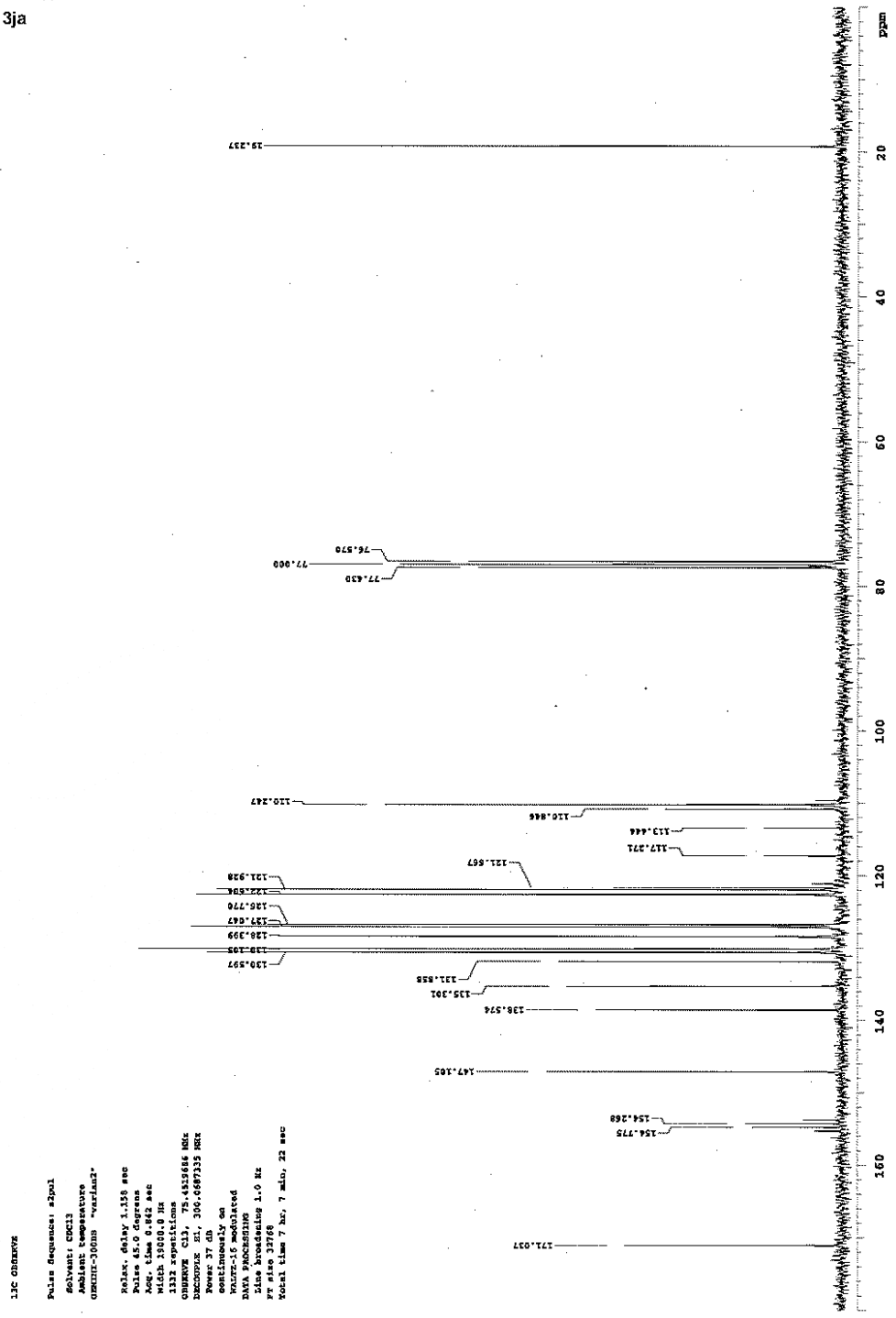

2-CH3C6H4
NHCOCF3
O
N
H

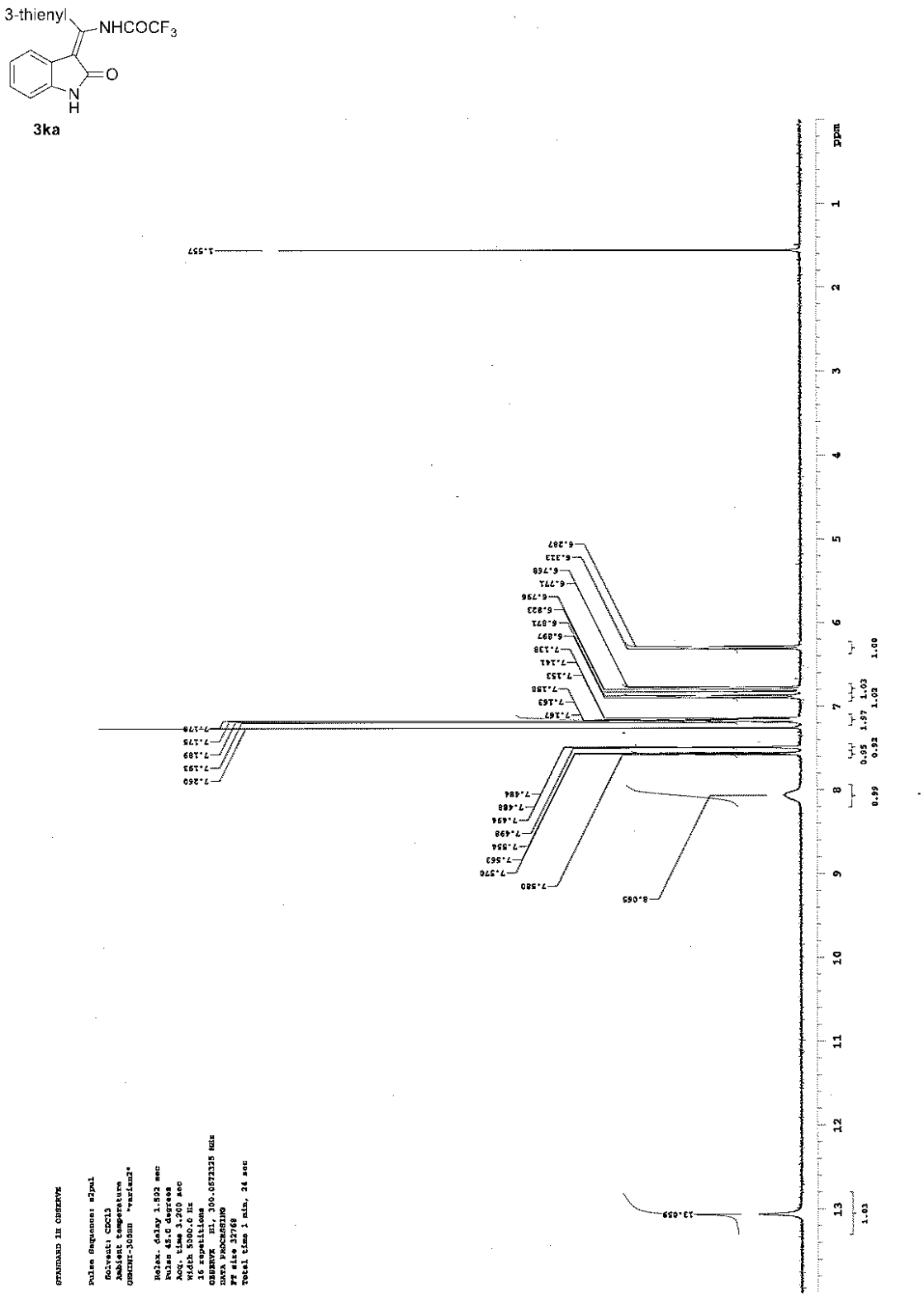
3ja

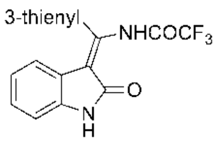

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672328 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec
2.289
5.895
5.920
6.713
6.718
6.740
6.743
6.766
6.769
6.908
6.934
7.145
7.149
7.170
7.175
7.197
7.260
7.266
7.267
7.274
7.293
7.296
7.357
7.360
7.384
7.396
7.418
7.420
7.433
7.473
7.478
7.495
7.502
7.522
7.537
9.633
13.348
ppm
1.05
0.96
1.00
1.01
1.05
2.08
1.00
1.01


2-CH3C6H4
NHCOCF3
O
N
H

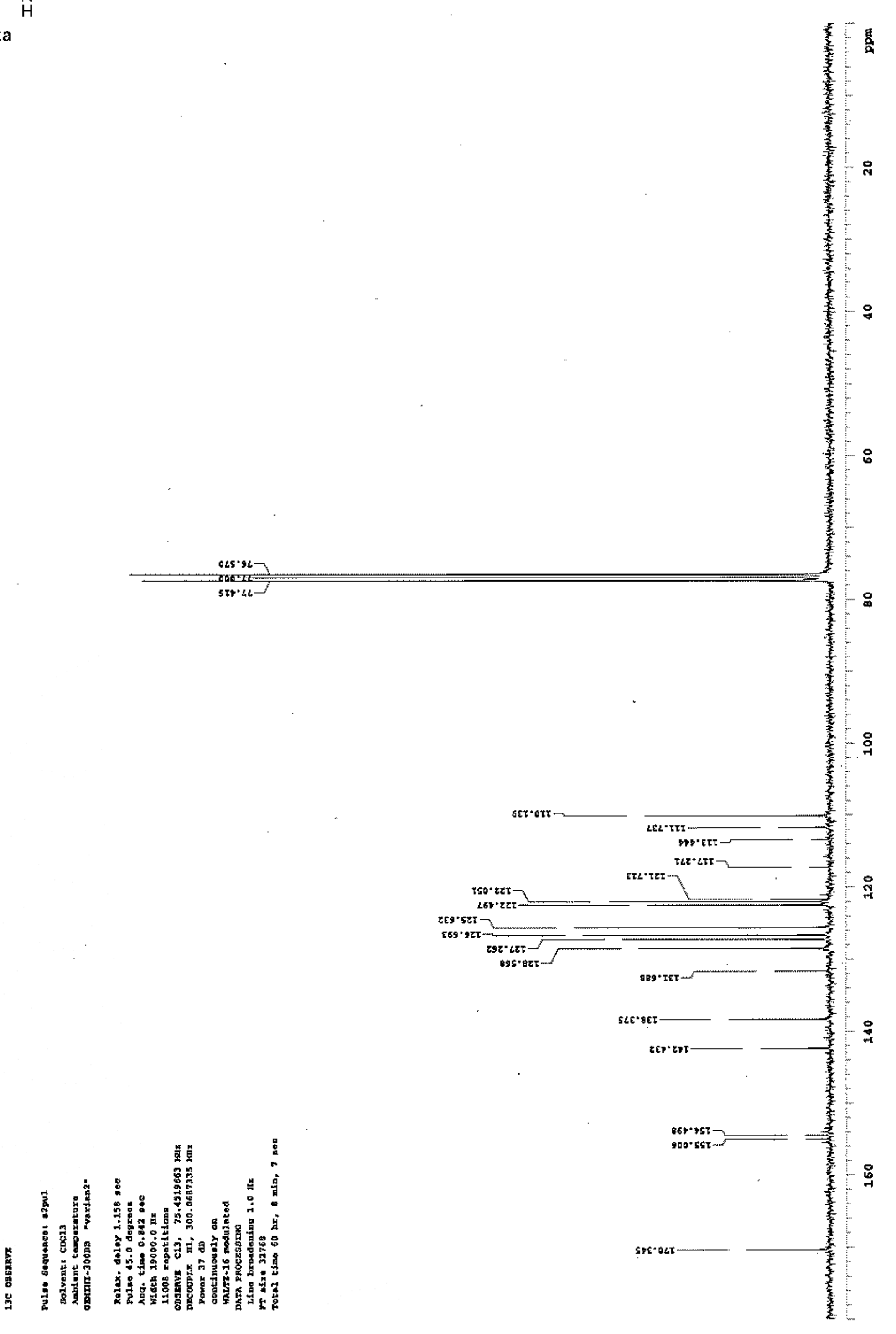
3ja

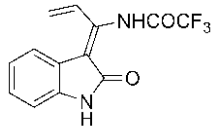

19.337
76.578
77.000
77.420
110.247
110.866
113.444
117.371
121.667
122.938
123.696
126.770
127.647
128.399
129.185
130.597
131.858
135.301
136.574
147.165
154.268
154.775
172.037
ppm
20
40
60
80
100
120
140
160
13C CHDRXVE
Pulse dequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.155 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 15600.0 Hz
1333 repetitions
OBSERVE C13, 75.4825686 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 db
Power 37 db on
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 7 hr, 7 min, 22 sec


3-thienyl
NHCOCF3
O
N
H
3ka
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
OBSERVE H1, 300.0672325 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec
Delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
1.557
6.287
6.323
6.768
6.774
6.796
6.823
6.874
6.897
7.138
7.141
7.153
7.158
7.163
7.167
7.178
7.179
7.189
7.193
7.260
7.370
7.383
7.554
7.558
7.684
7.688
7.694
7.698
8.065
13.059
ppm
1.00
1.03
1.02
1.07
0.92
0.95
0.89
1.03

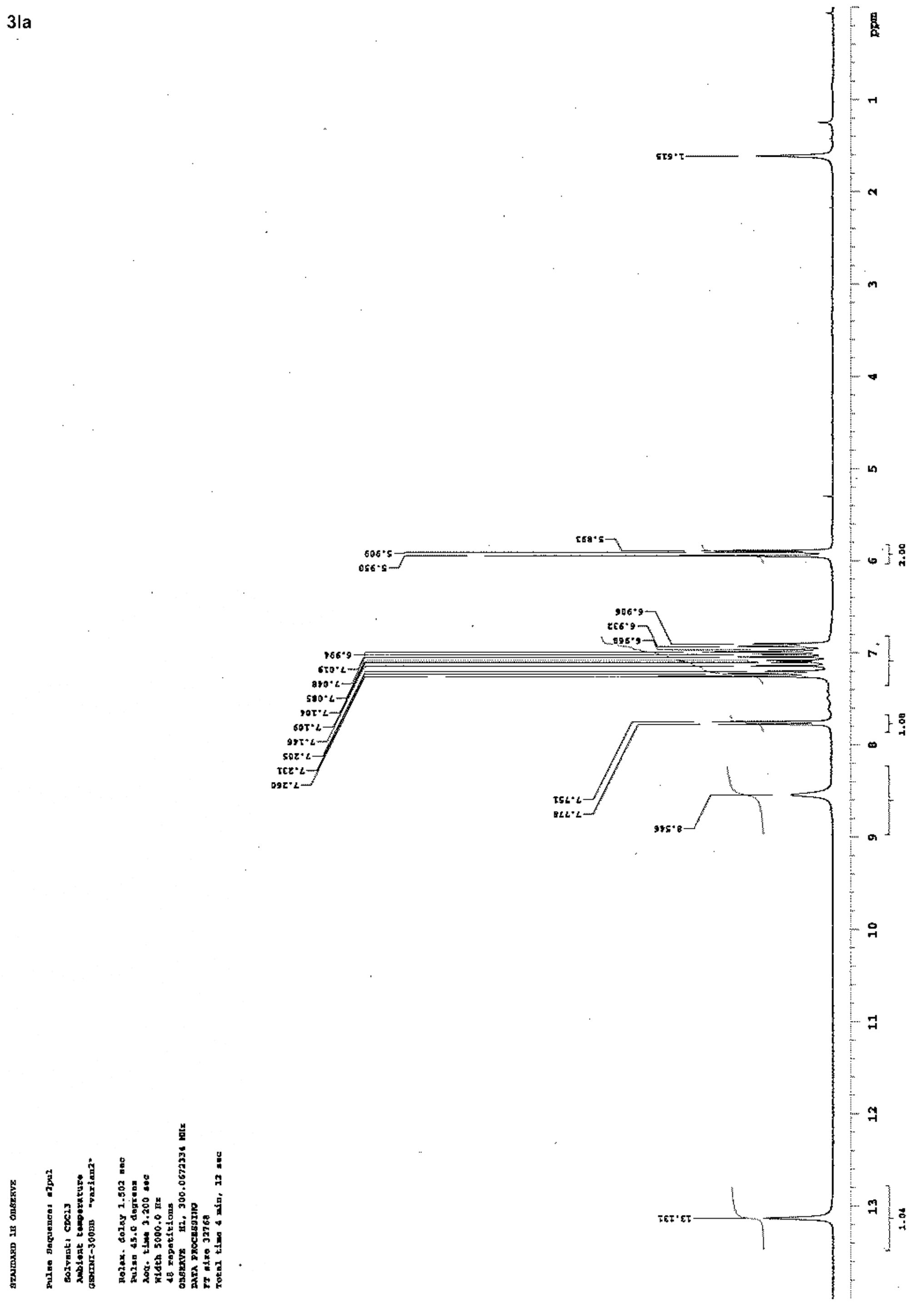


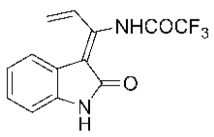
3ka



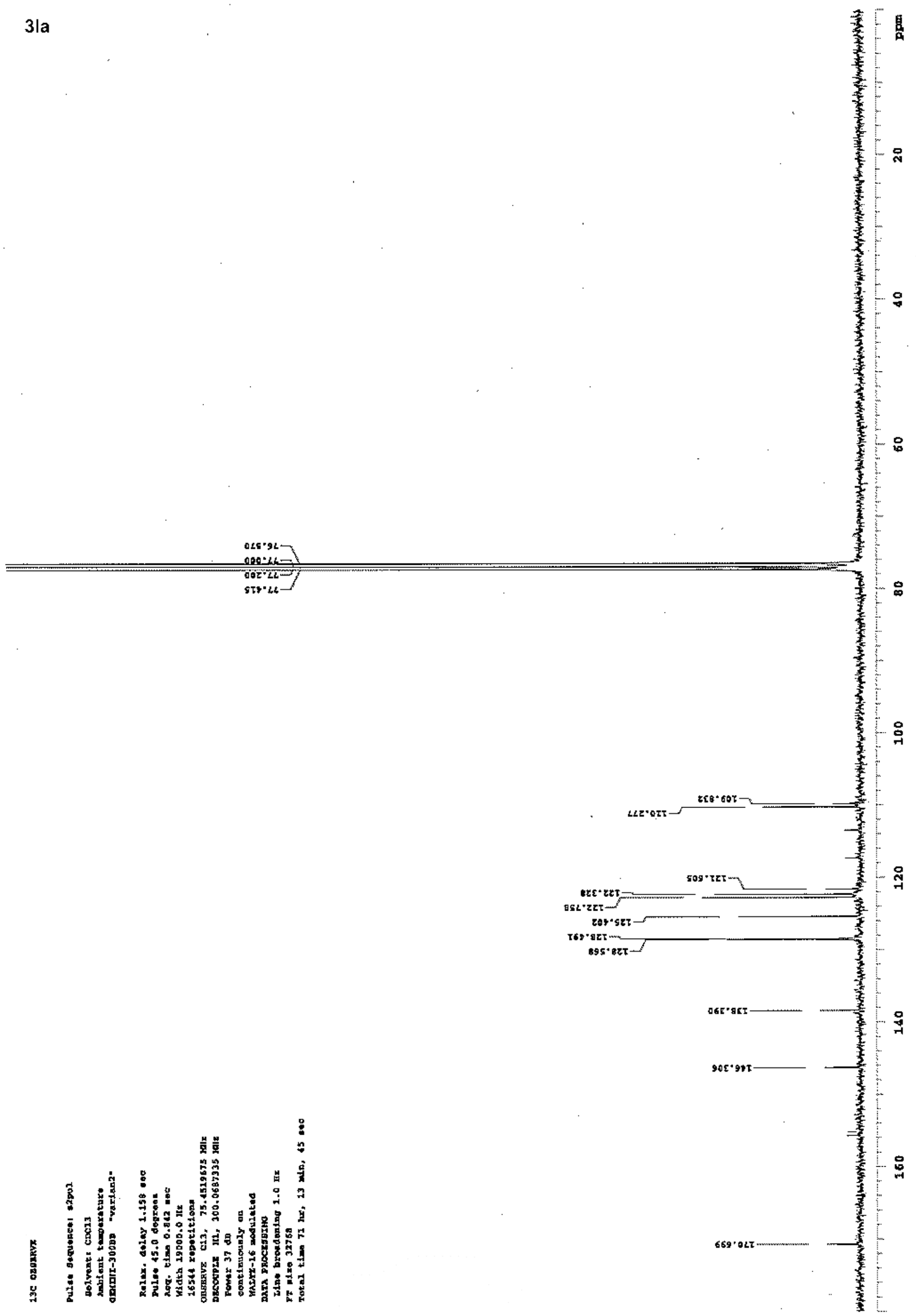


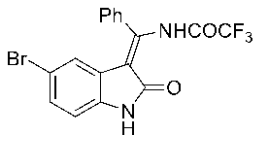
3la



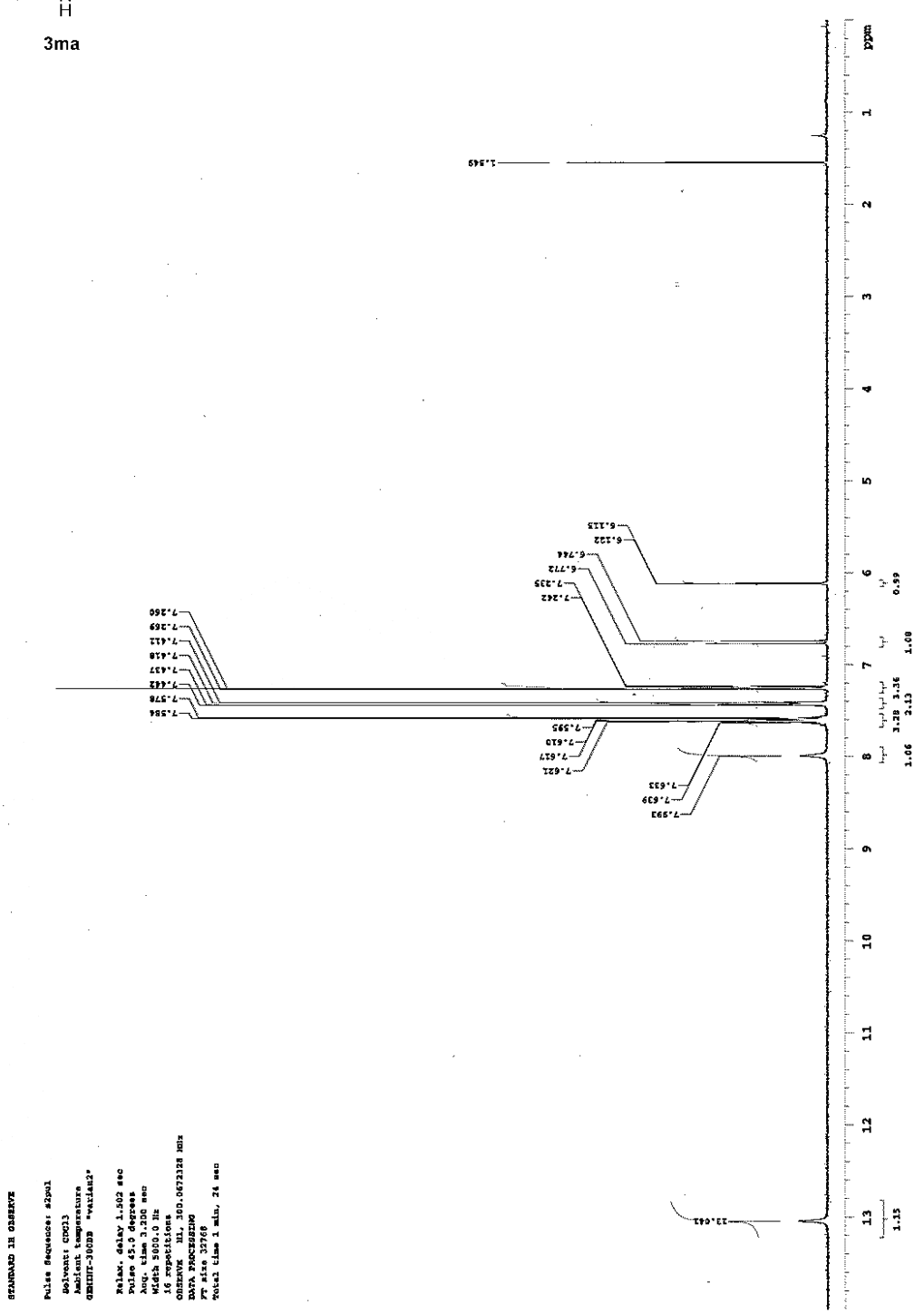


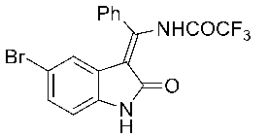
3la



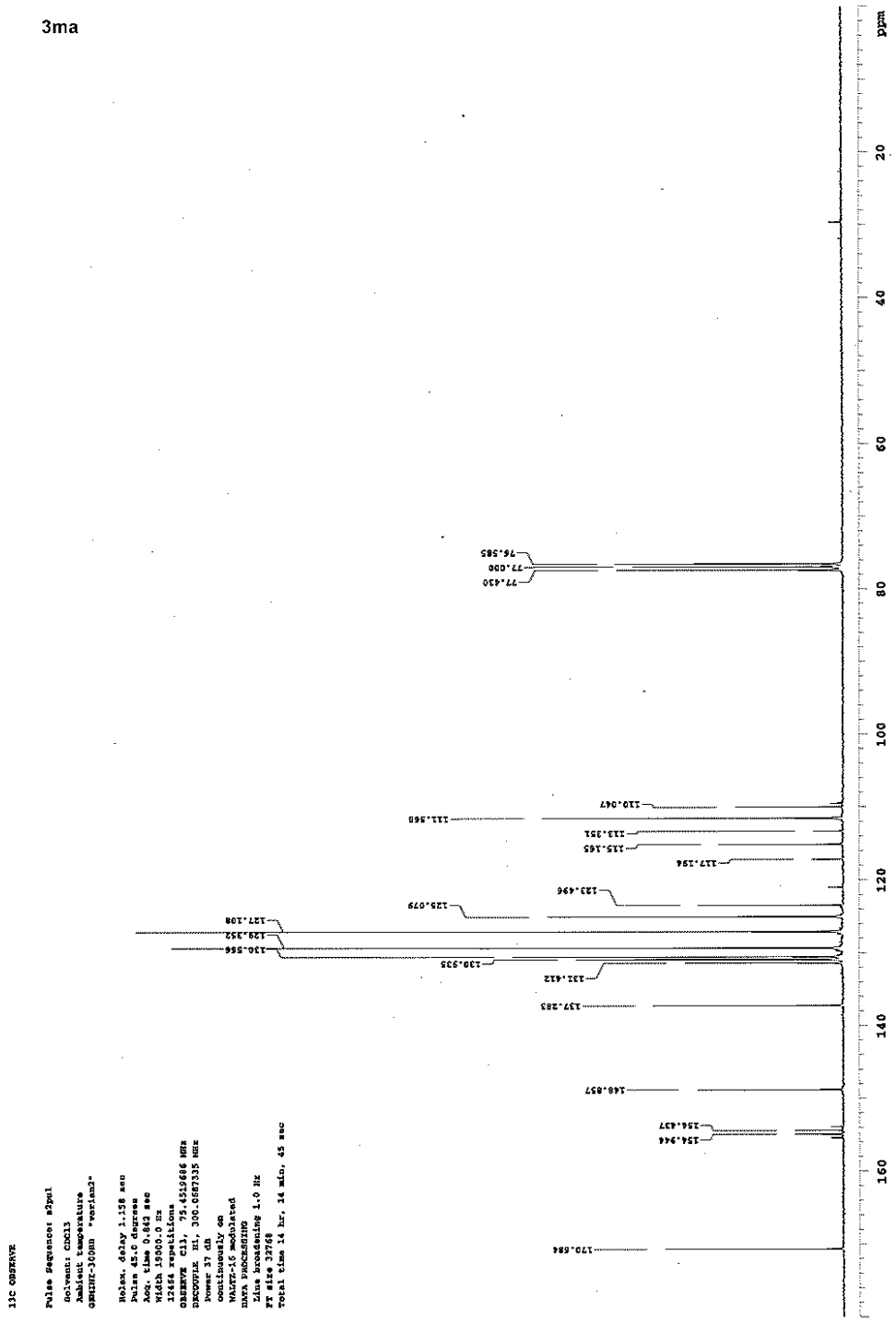

Ph
NHCOCF3
Br
O
N
H

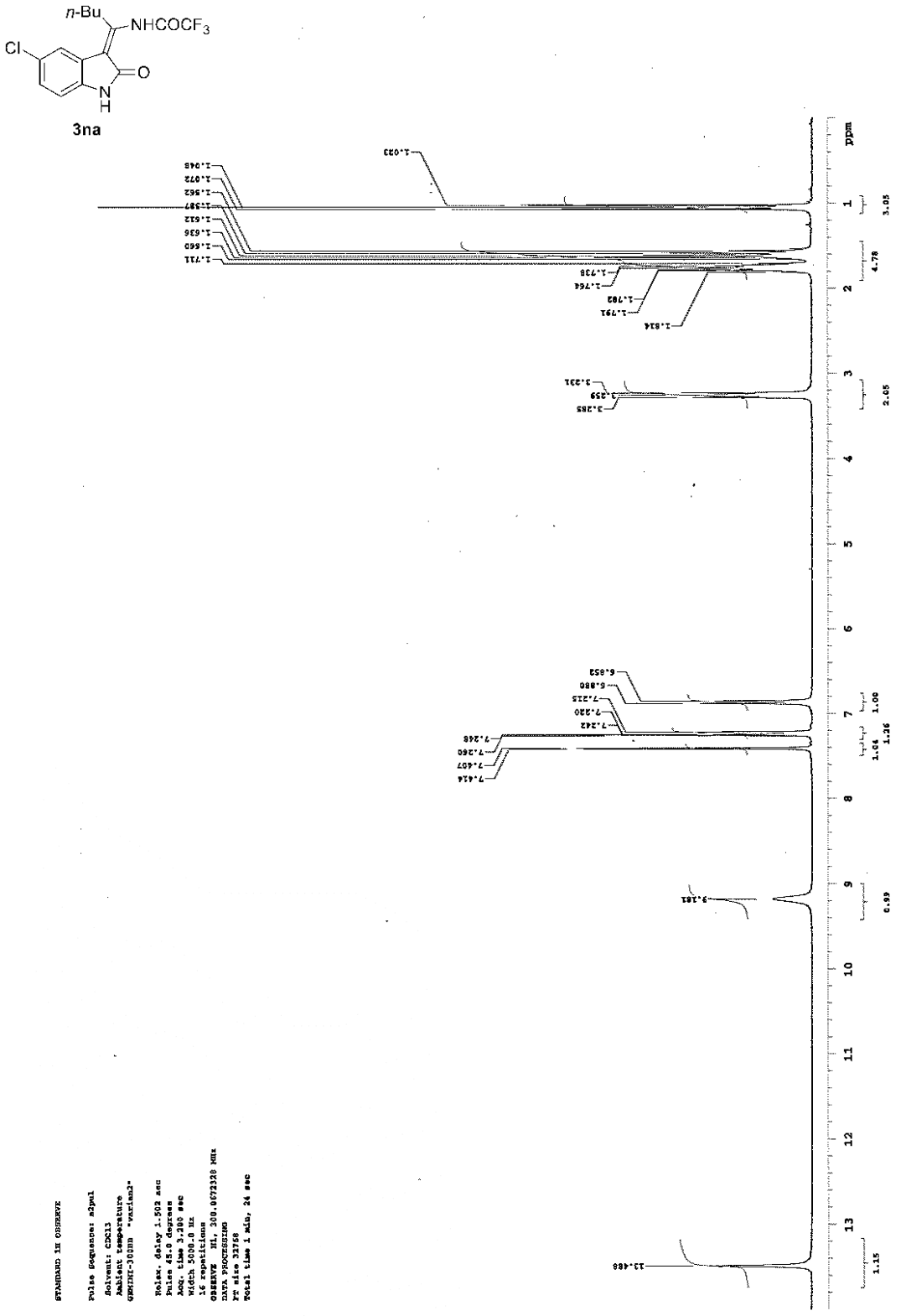
3ma


STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "vnt4m2"
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
ppm


Ph
NHCOCF3
Br
O
N
H

3ma


ppm
20
40
60
80
100
120
140
160
77.430
77.088
76.585
170.684
156.437
154.944
148.857
137.283
132.432
130.535
130.556
129.352
127.138
125.079
123.696
117.194
115.165
113.351
111.368
110.047
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.843 sec
Width 19000.0 Hz
12464 repetitions
OBSERVE C13, 75.4519866 MHz
DECOUPLE H1, 300.0587335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 14 hr, 14 min, 45 sec



STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

QNMNMR-300MM "varian2"

Relax. delay 1.502 sec

Pulse 45.0 degrees

Acq. time 3.200 sec

Width 5000.0 Hz

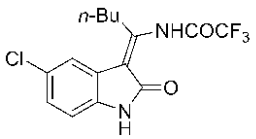
16 repetitions

OBSERVE H1, 300.0672328 MHz

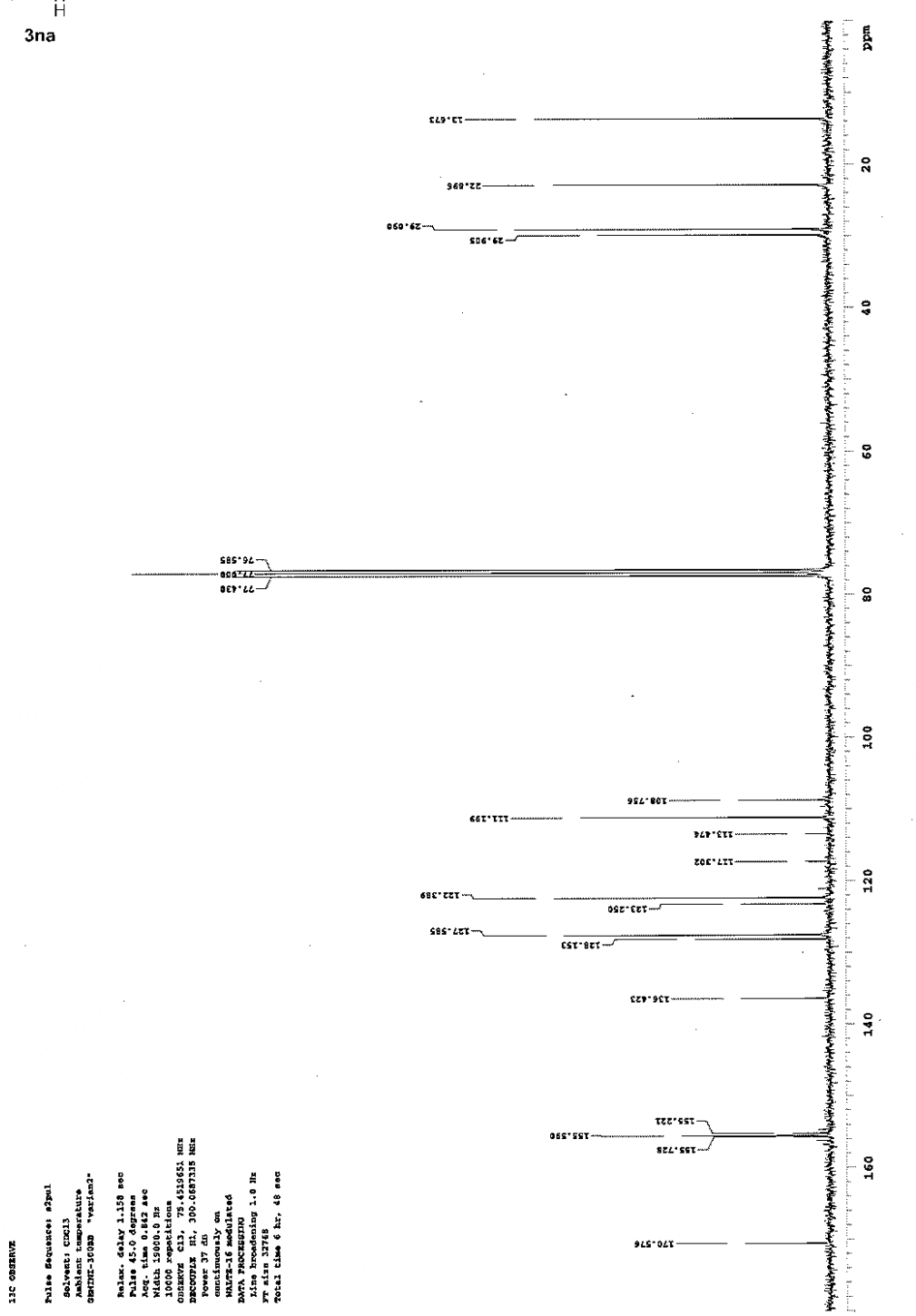
DATA PROCESSING

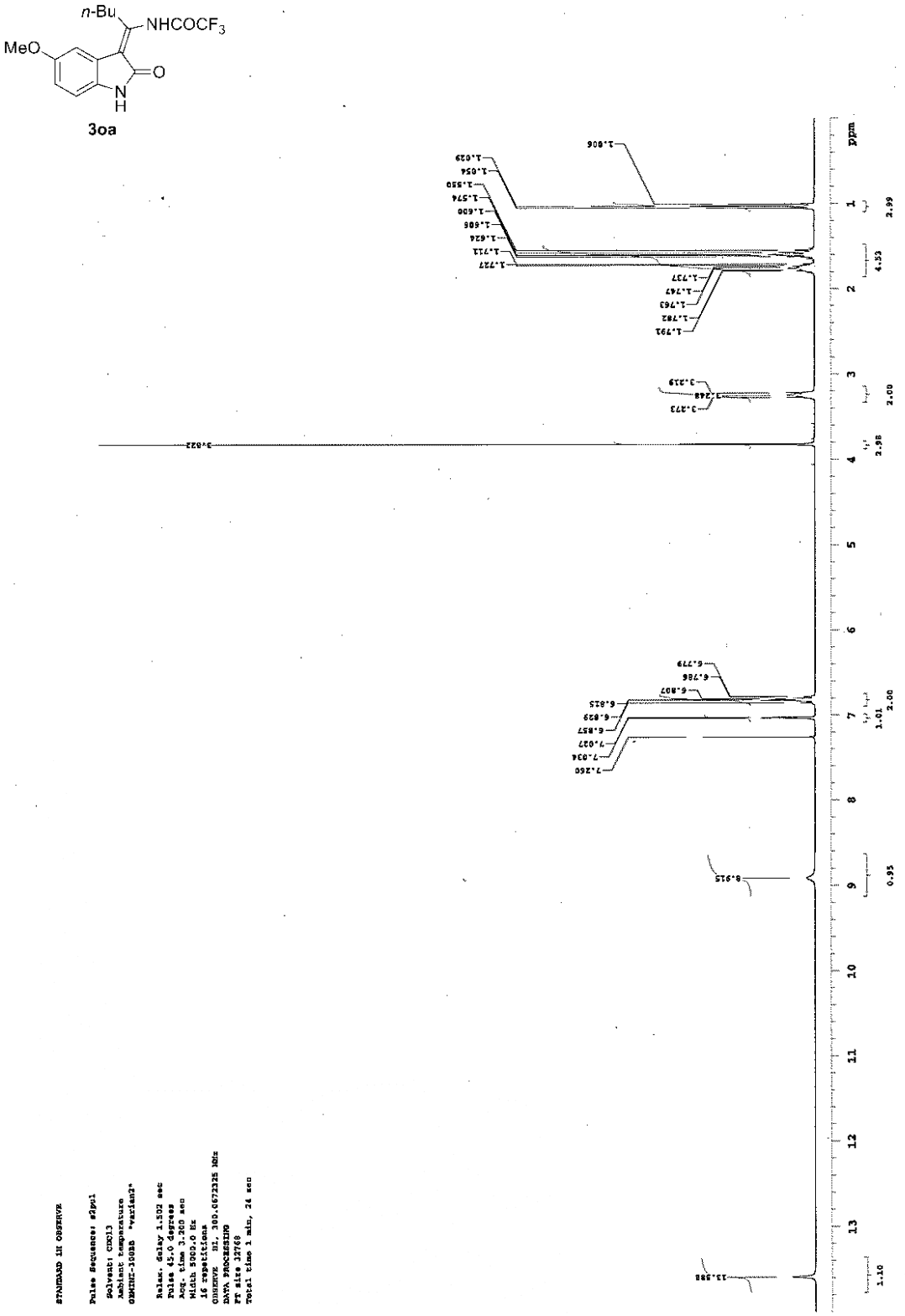
FT size 32768

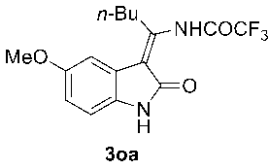
Total time 1 min, 24 sec


n-Bu
NHCOCF3
Cl
O
N
H

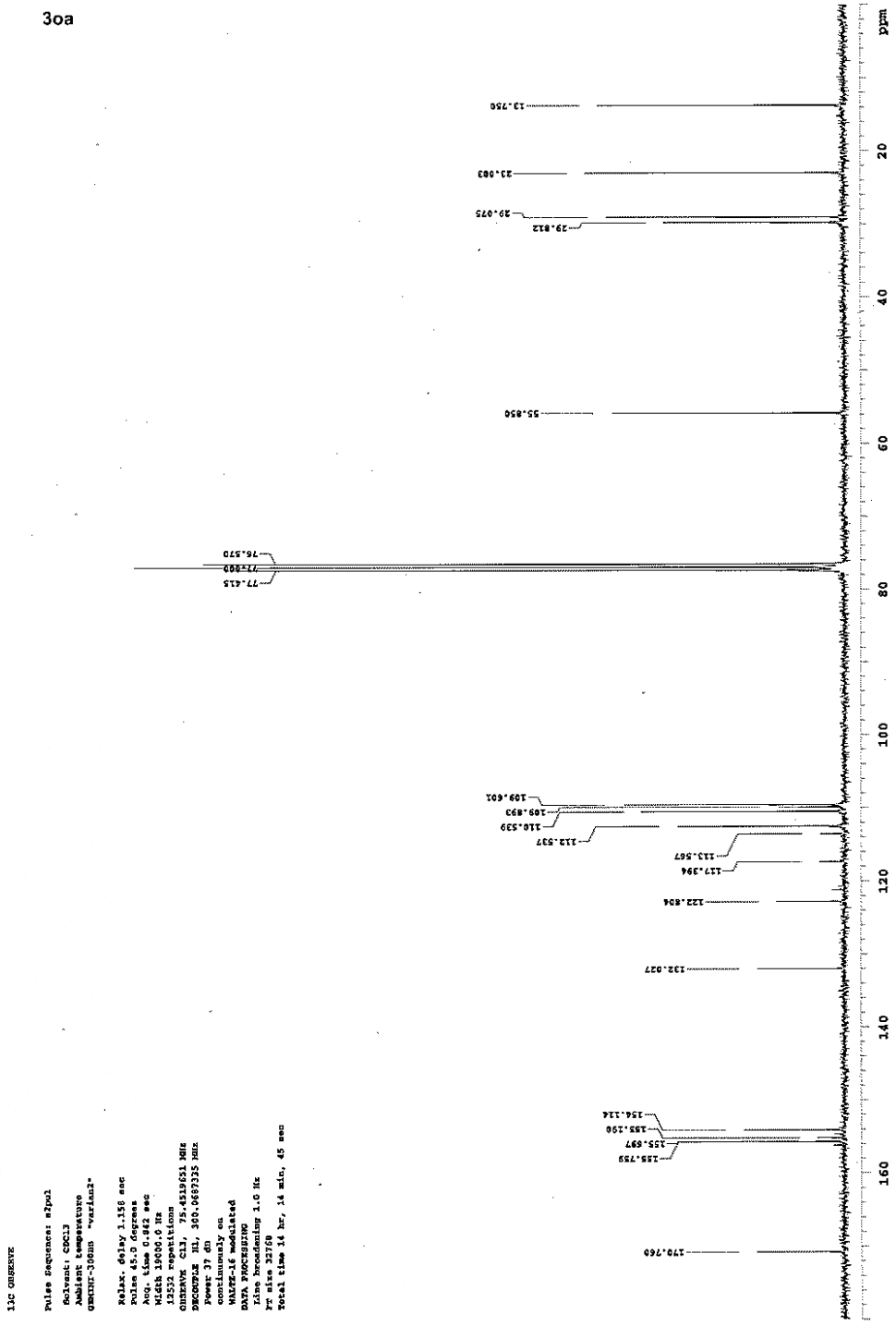
3na


13.673
22.095
29.050
29.905
76.585
77.000
77.438
108.756
111.199
113.474
117.302
123.389
123.250
127.585
128.153
136.423
155.221
155.590
155.728
170.576
ppm
20
40
60
80
100
120
140
160
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300B "varian2"
Relax. delay 1.150 sec
Pulse 45.0 degrees
Acq. time 0.862 sec
Width 19000.0 Hz
10000 repetitions
OBSERVE C13, 75.4519651 MHz
DECOUPLE H1, 300.0687338 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 6 hr, 48 sec


n-Bu
NHCOCF3
MeO
O
N
H
3oa
PROTON 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672325 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec
ppm



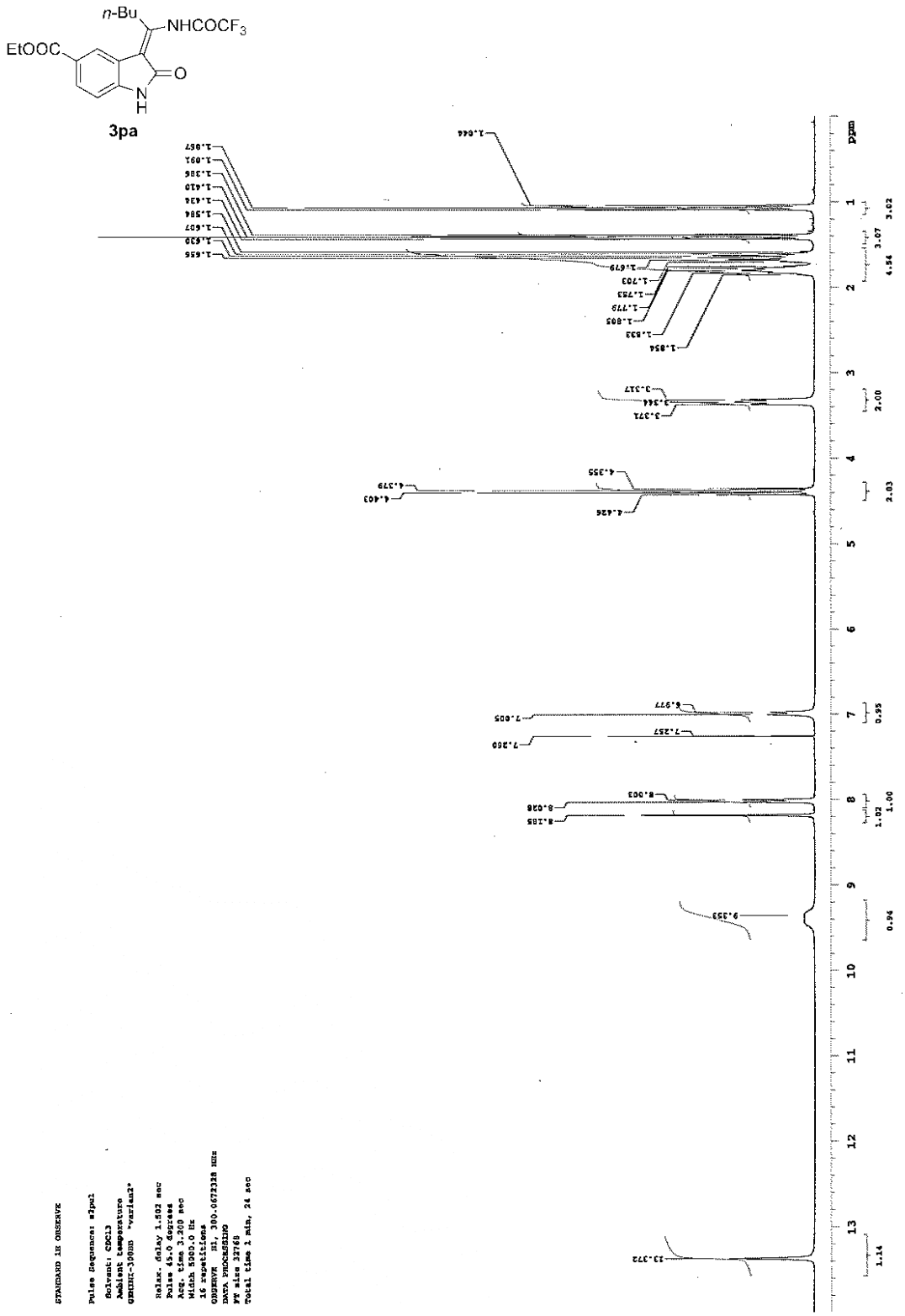
3oa

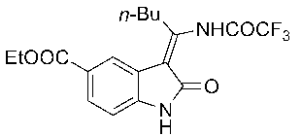


13C OBSERVE

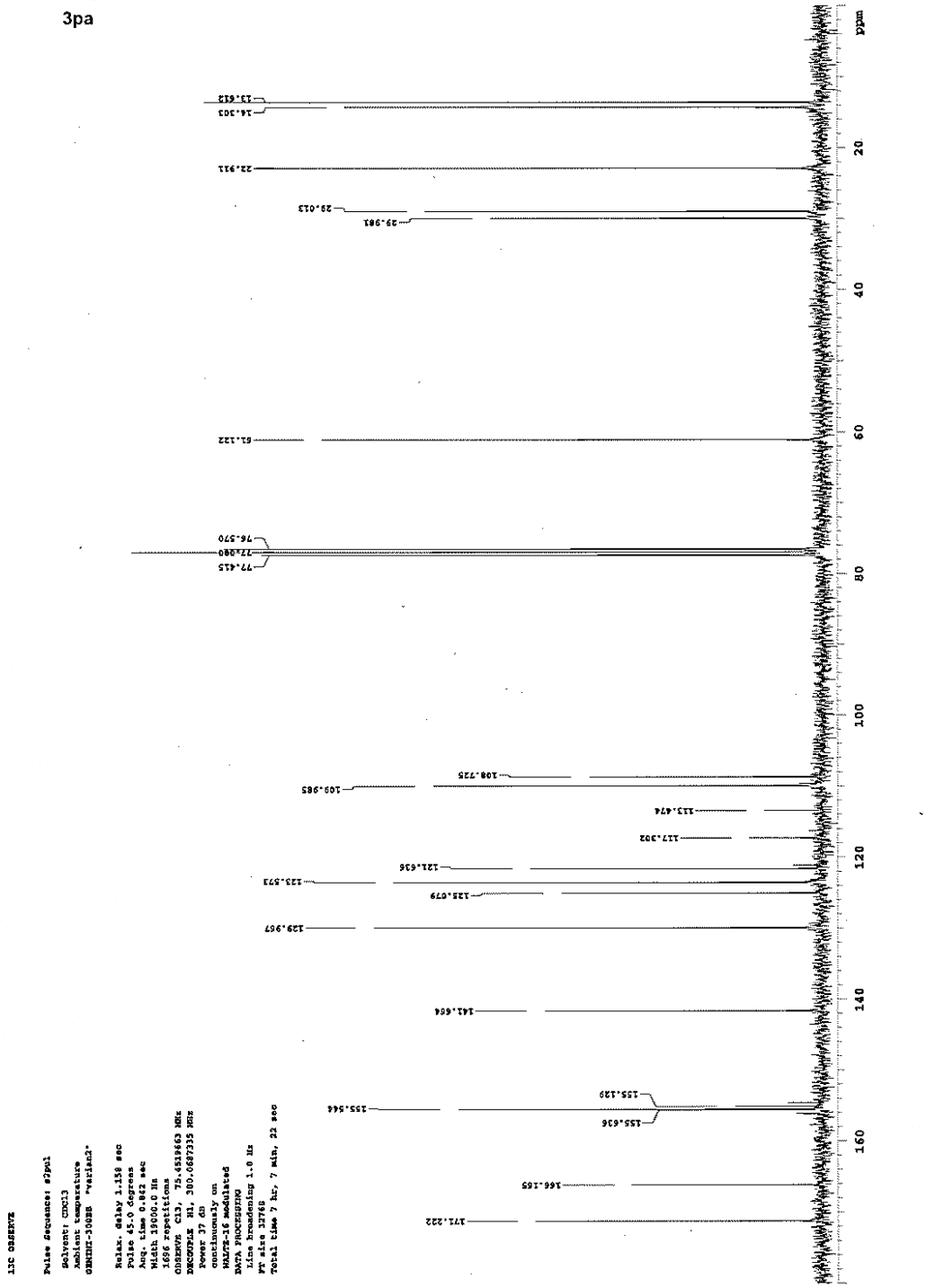
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"

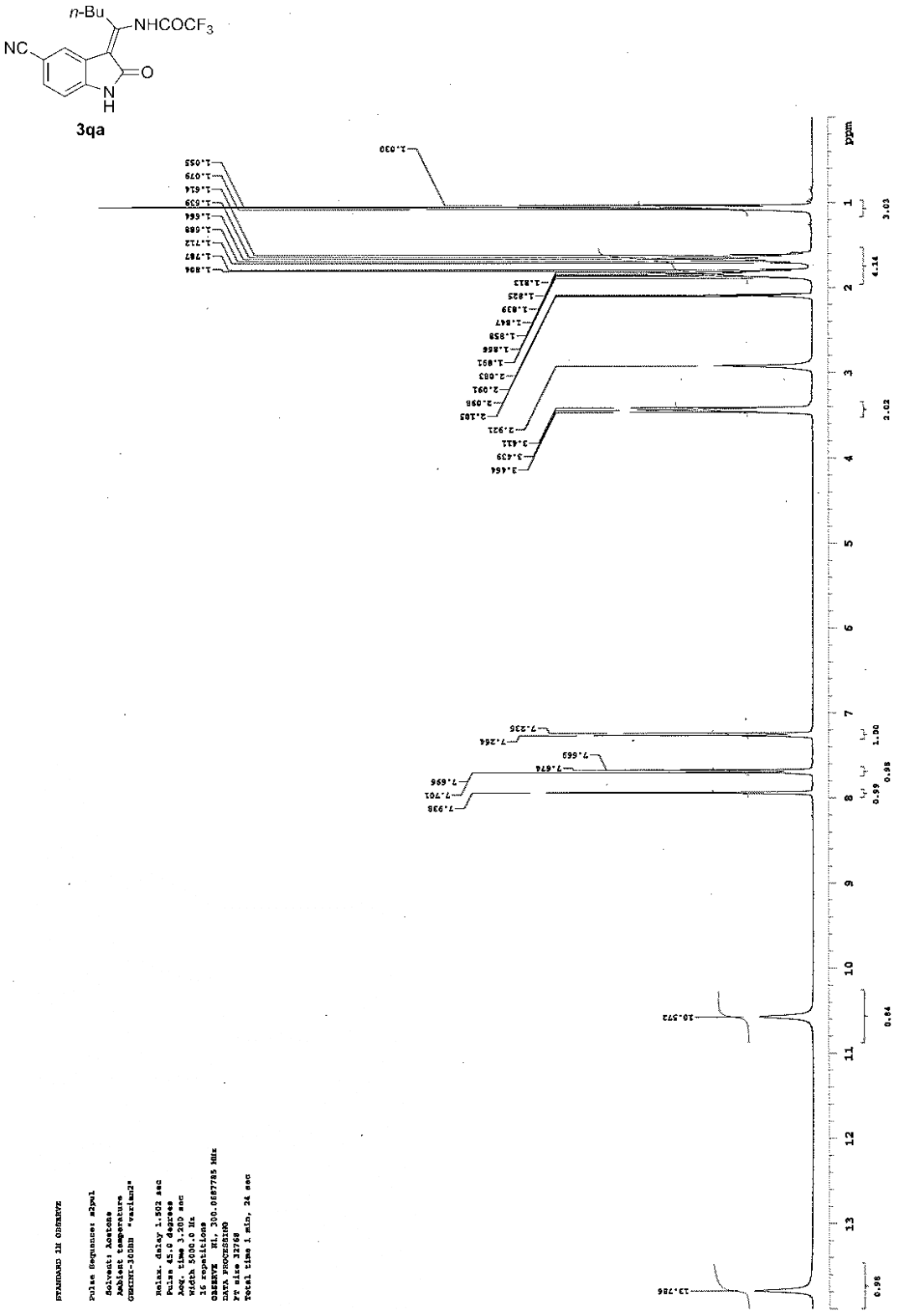
Relax. delay 1.150 sec
Pulse 45.0 degrees
Acq. time 0.882 sec
Width 19000.0 Hz
13532 repetitions
OBSERVE C13, 75.4519651 MHz
DECOUPLE H1, 300.0687325 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 14 hr, 14 min, 45 sec

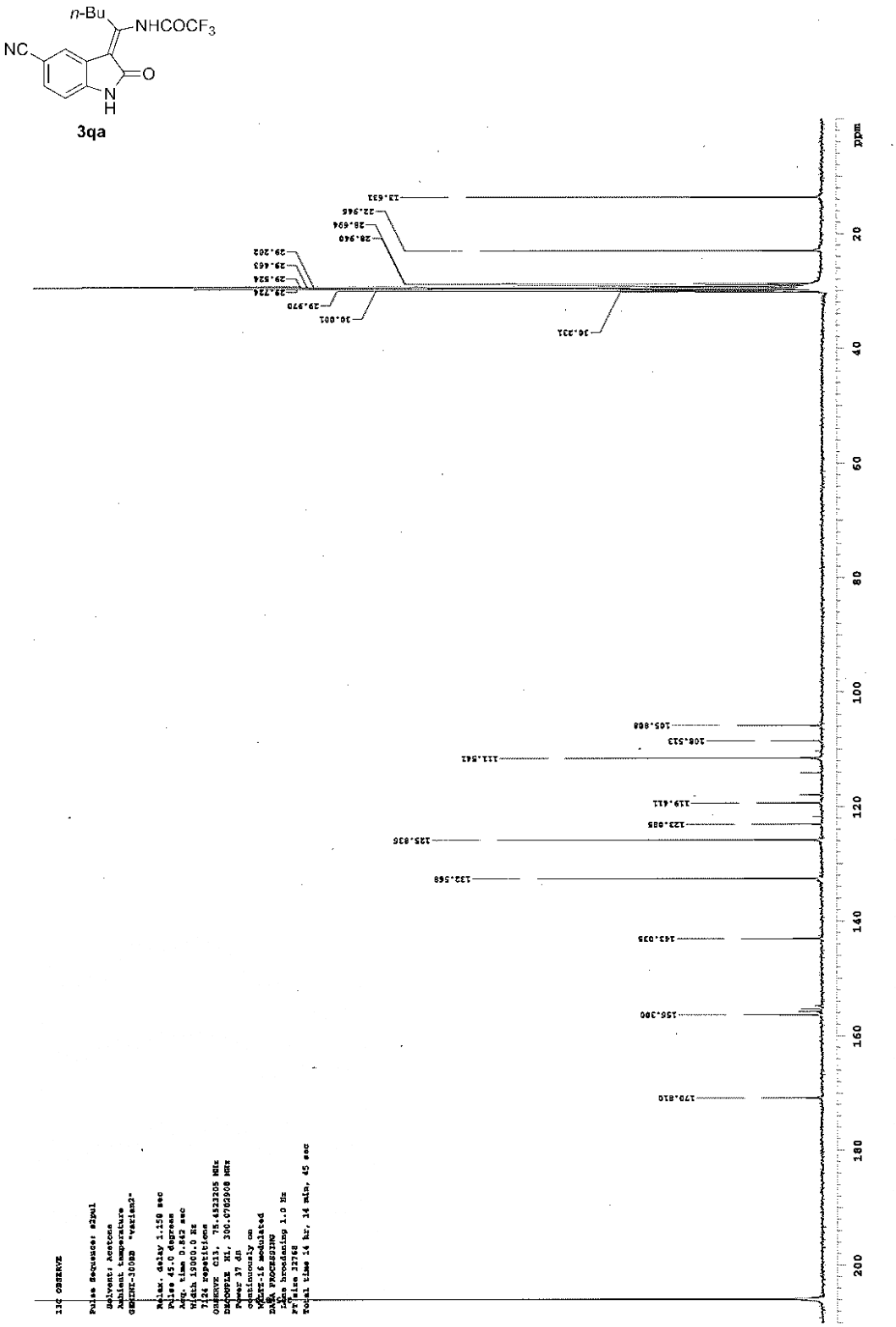

n-Bu
NHCOCF3
EtOOC
O
N
H
3pa
1.044
1.857
1.892
1.386
1.410
1.434
1.584
1.607
1.630
1.656
1.679
1.703
1.733
1.779
1.805
1.833
1.854
3.317
3.344
3.374
4.355
4.379
4.403
4.426
6.977
7.005
7.357
7.360
8.003
8.036
8.285
9.353
13.372
ppm
3.02
3.07
4.94
2.00
2.03
0.95
1.02
1.00
0.94
1.14
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
OMNMR-300nm "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5980.0 Hz
16 repetitions
OBSERVE H1, 300.0672328 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec

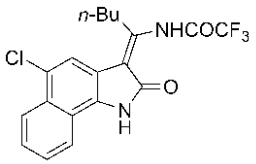

n-Bu
NHCOCF3
EtOOC
O
N
H

3pa

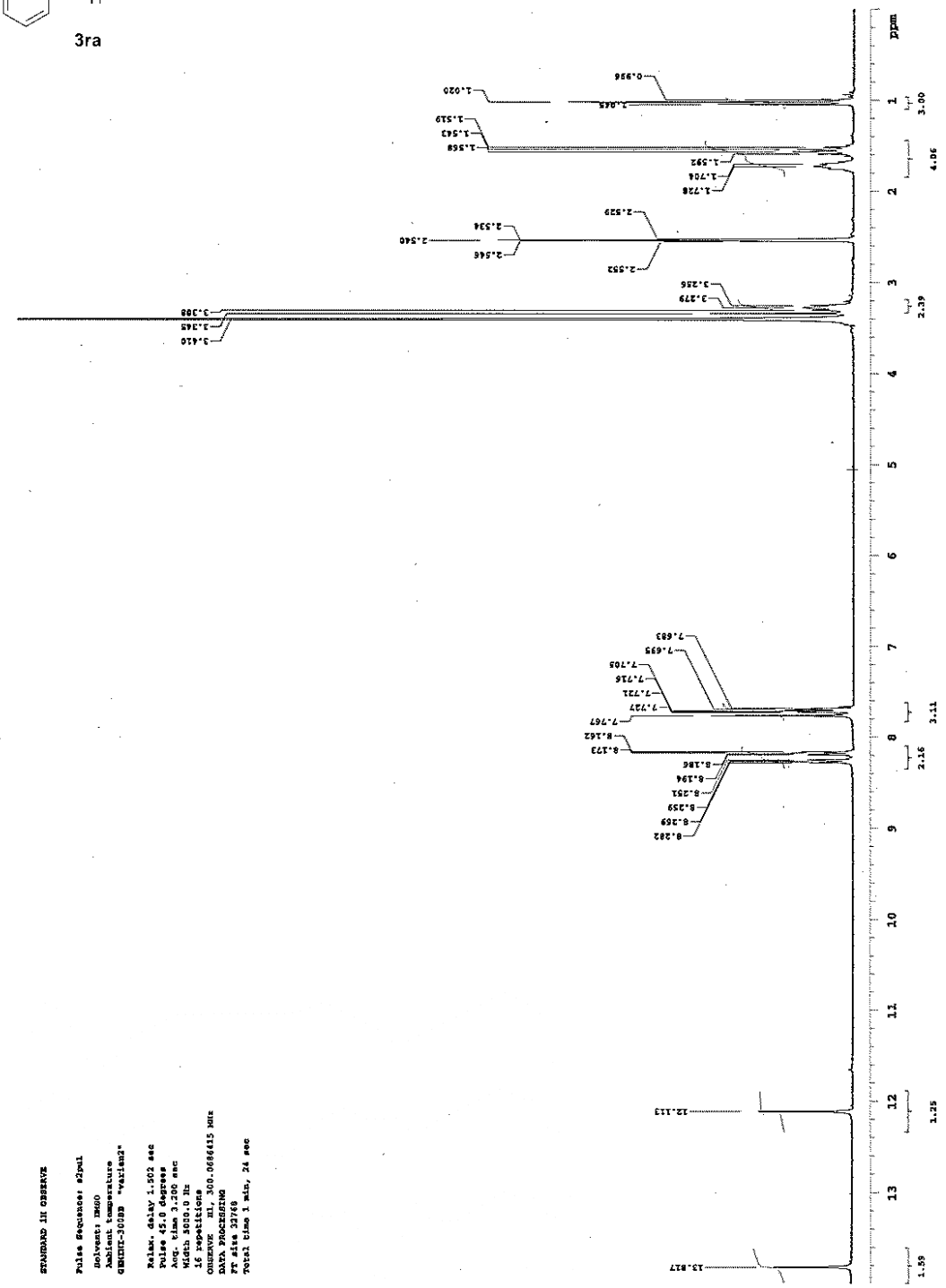

13.632
14.303
22.911
29.013
29.981
61.122
76.570
77.080
77.415
108.725
109.985
113.474
117.302
121.636
123.573
125.079
139.967
141.664
155.139
155.544
155.636
166.163
171.232
ppm
20
40
60
80
100
120
140
160

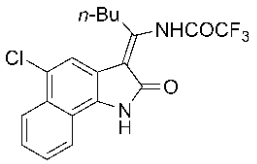

n-Bu
NHCOCF3
NC
O
N
H
3qa
1.030
1.055
1.079
1.614
1.639
1.664
1.688
1.712
1.787
1.804
1.813
1.825
1.839
1.847
1.858
1.868
1.951
2.083
2.091
2.098
2.185
2.921
3.411
3.439
3.464
7.235
7.264
7.663
7.674
7.696
7.701
7.938
10.372
13.786
ppm
3.03
4.14
2.02
1.00
0.98
0.99
0.84
0.98


n-Bu
NHCOCF3
NC
O
N
H
3qa
13.631
22.945
28.694
28.940
29.202
29.463
29.524
29.734
29.970
30.001
36.331
105.868
108.513
111.841
119.611
123.085
135.836
152.568
163.035
176.300
179.810
ppm

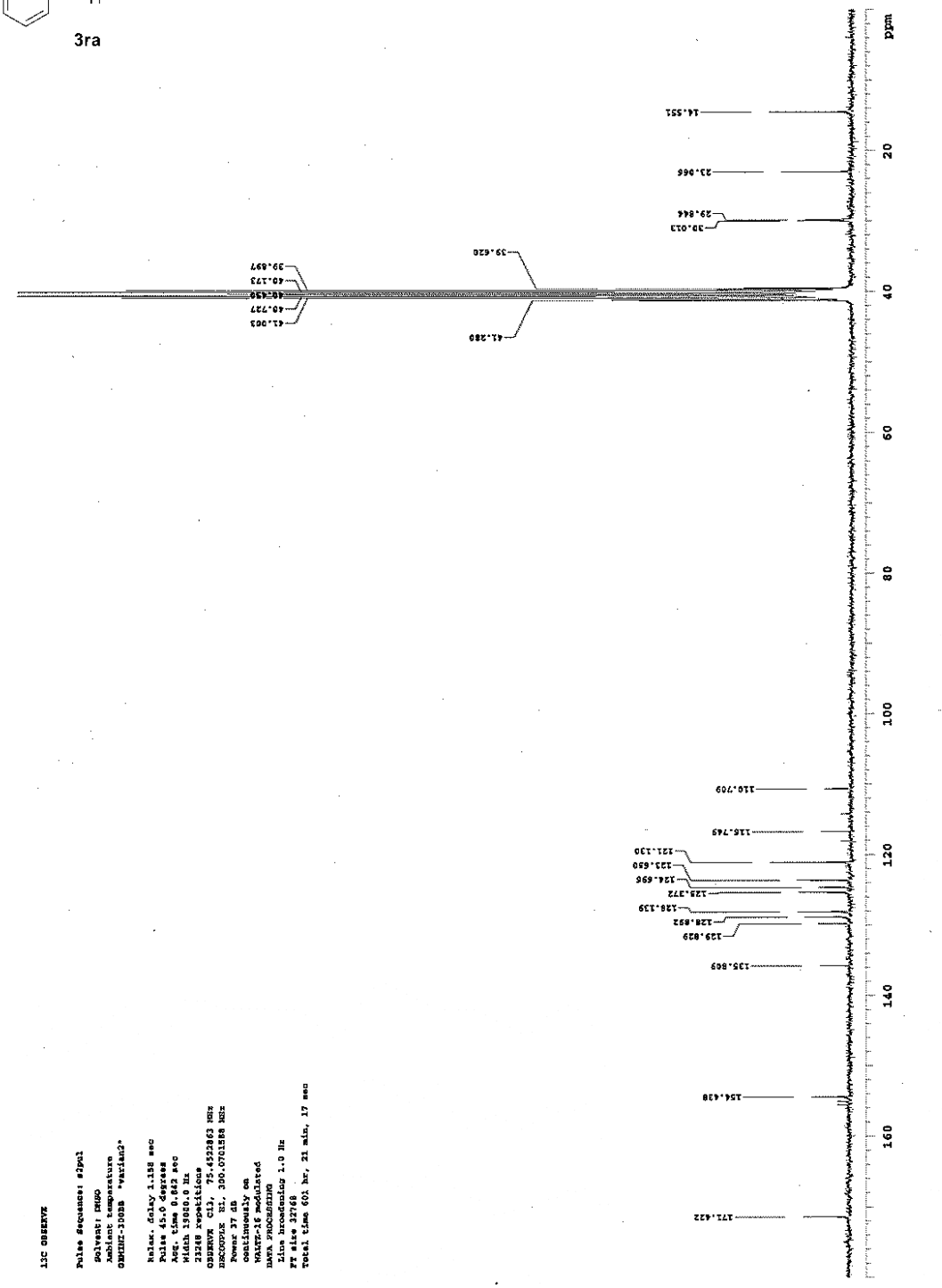

n-Bu
NHCOCF3
Cl
O
N
H

3ra


STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
OMNMR-300SB "valiant"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 3000.0 Hz
16 repetitions
OBSERVE H1, 300.0686415 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec
ppm



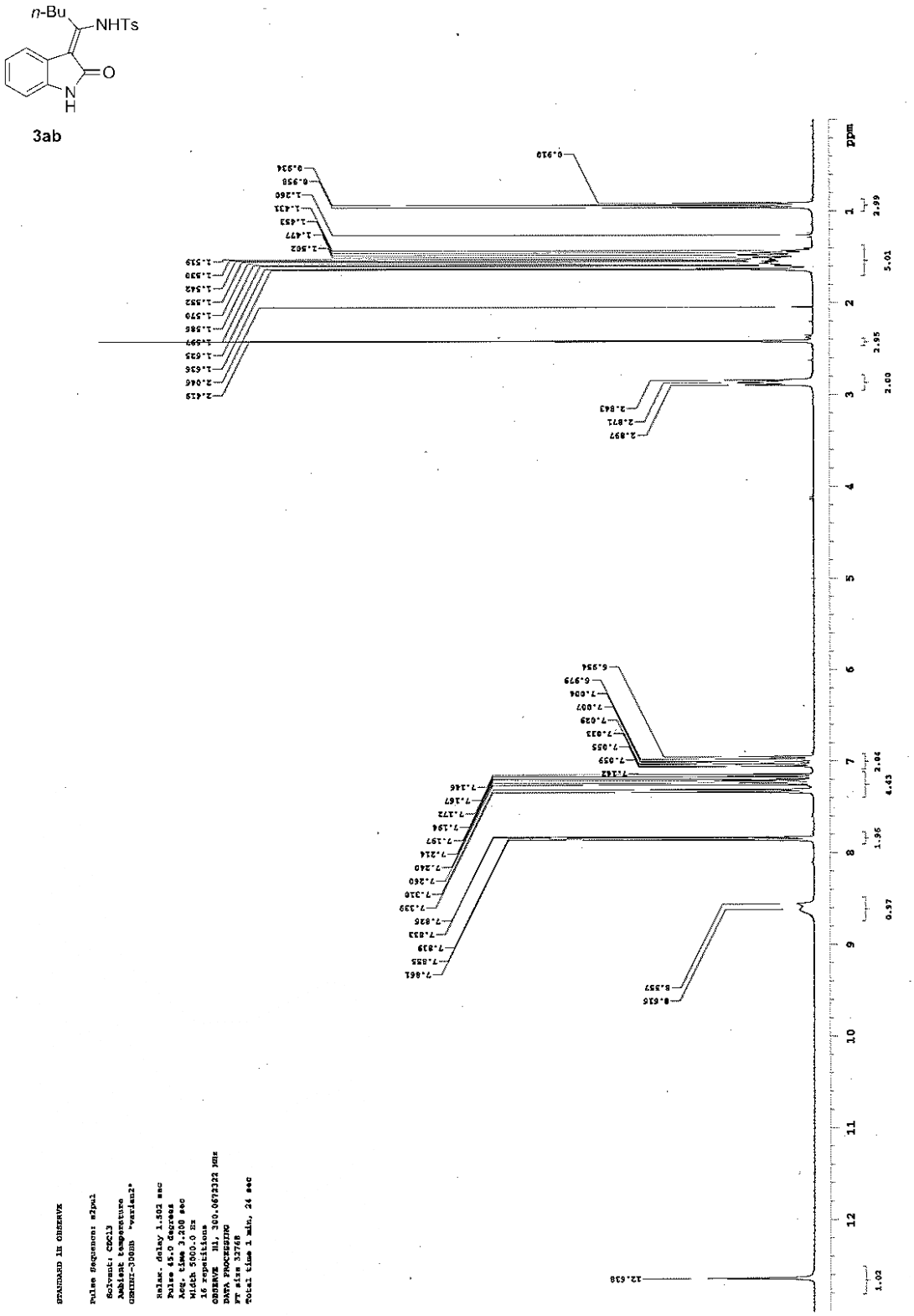
3ra

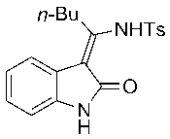


13C OBSERVE

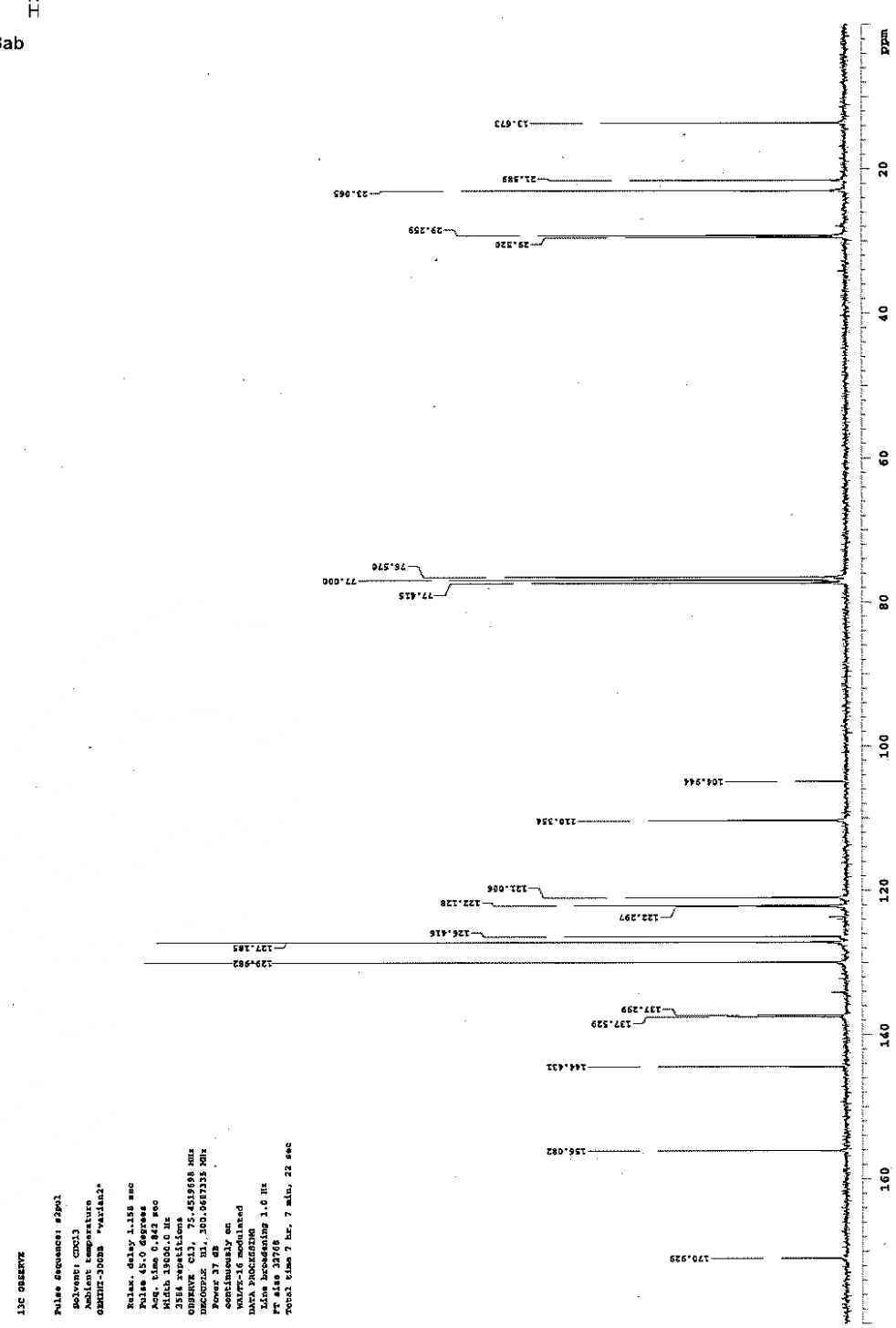
Pulse sequence: s2pul
Solvent: DMSO
Ambient temperature
CHMIMI-300B "varian2"

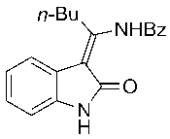
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.862 sec
Width 19020.0 Hz
23240 repetitions
OBSERVE C13, 75.4522863 MHz
DECOUPLE H1, 300.0701588 MHz
Power 37 dB
continuously on
WALTZ16 modulated
GARP decoupling
Line broadening 1.0 Hz
FT size 32768
Total time 601 hr, 21 min, 17 sec


n-Bu
NHTs
O
N
H
3ab
ppm

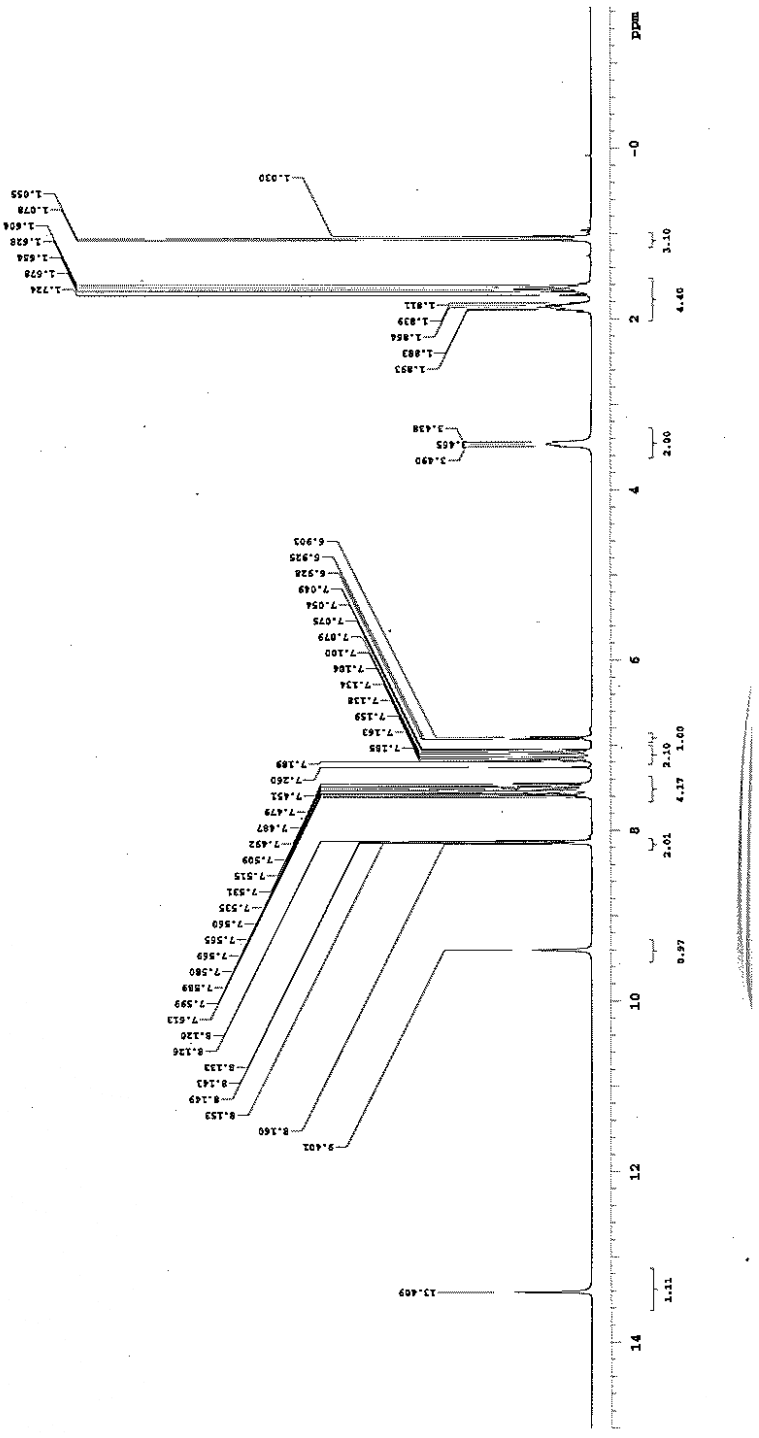

n-Bu
NHTs
O
N
H

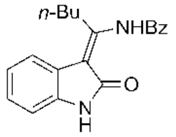
3ab


13.673
21.389
33.965
39.259
39.326
76.576
77.000
77.415
104.944
118.354
132.006
132.128
132.397
136.416
137.185
139.982
137.299
137.529
144.633
156.082
170.935
ppm
20
40
60
80
100
120
140
160

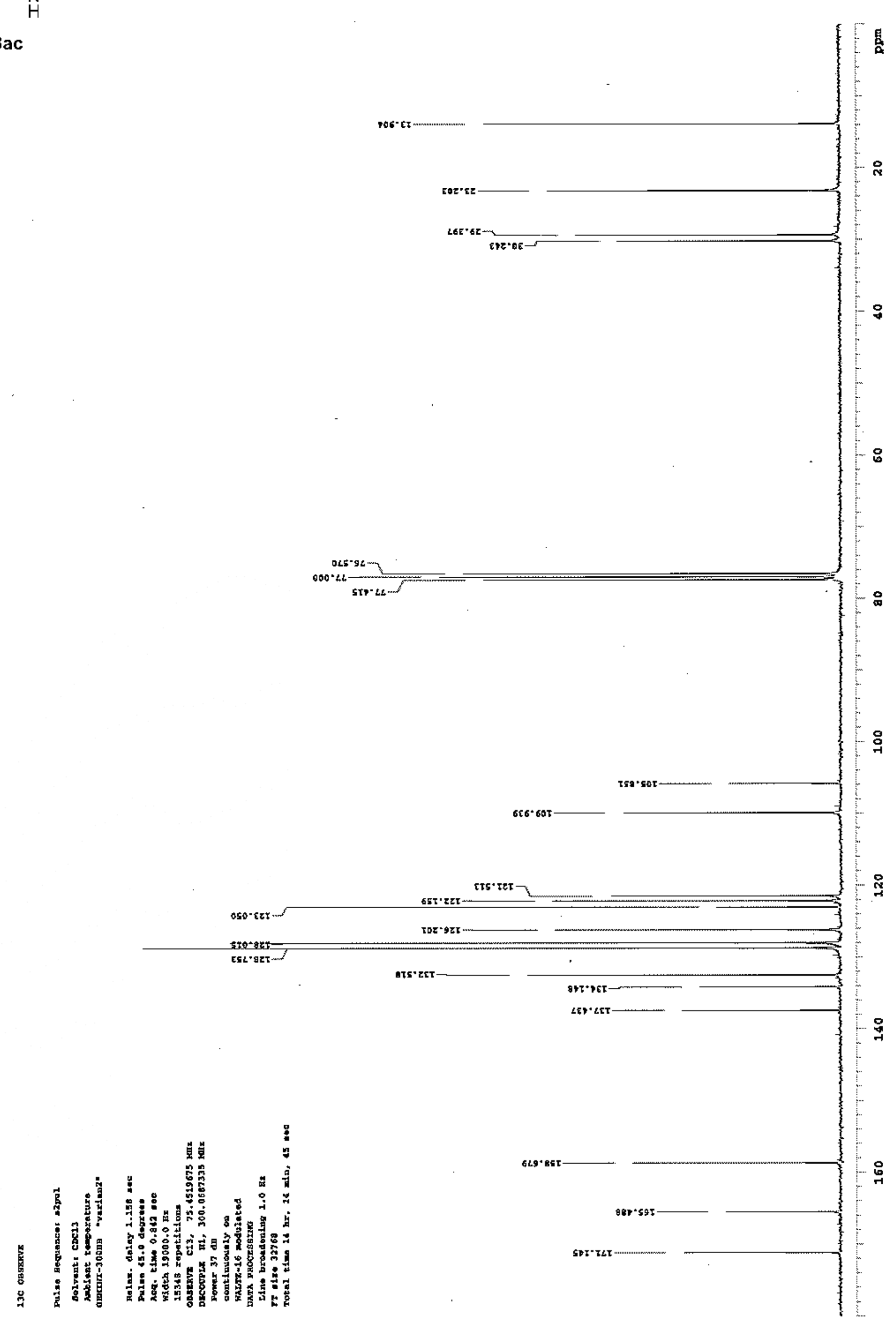


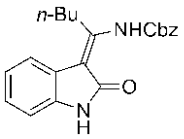
3ac



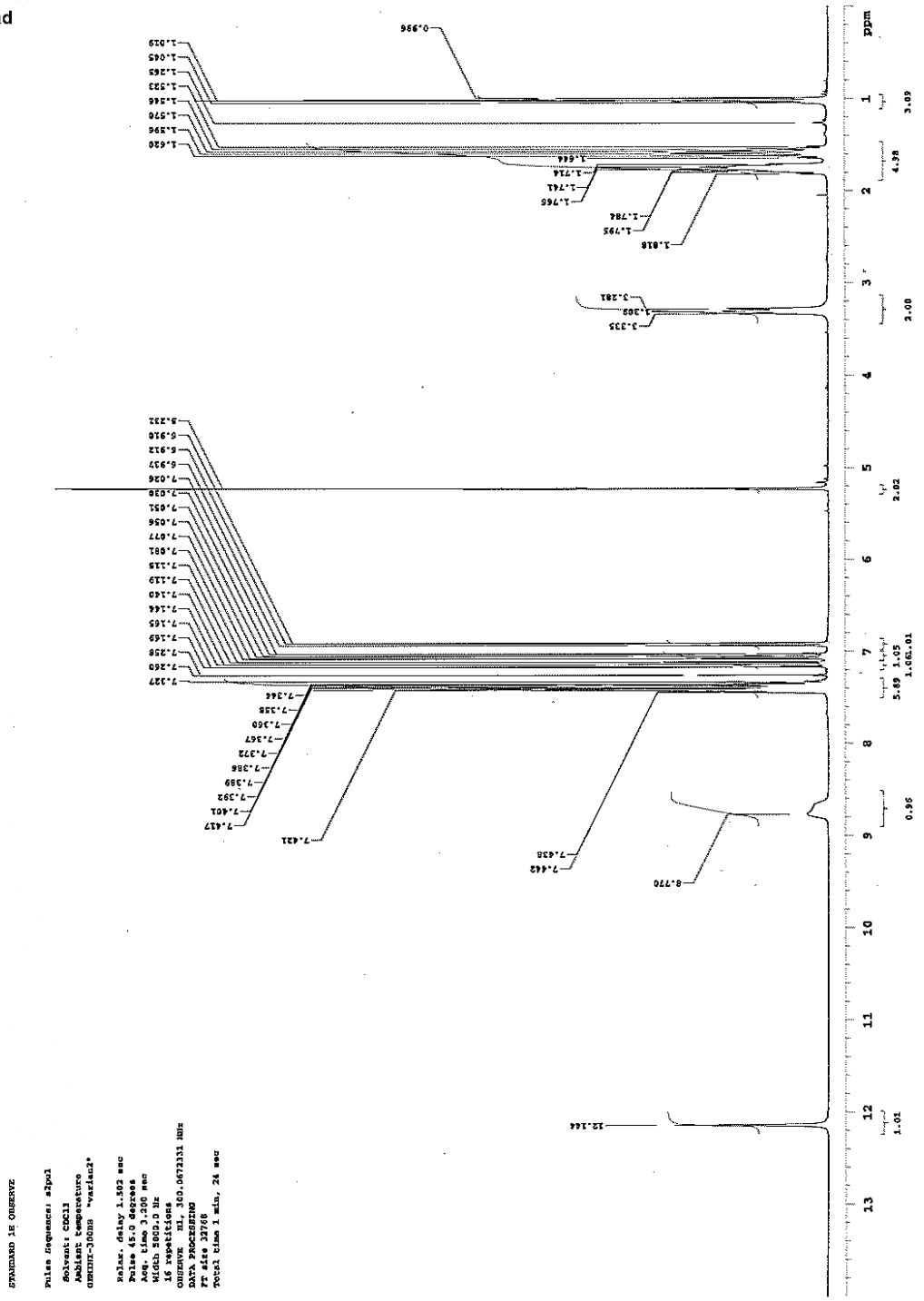


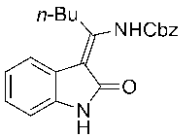
3ac



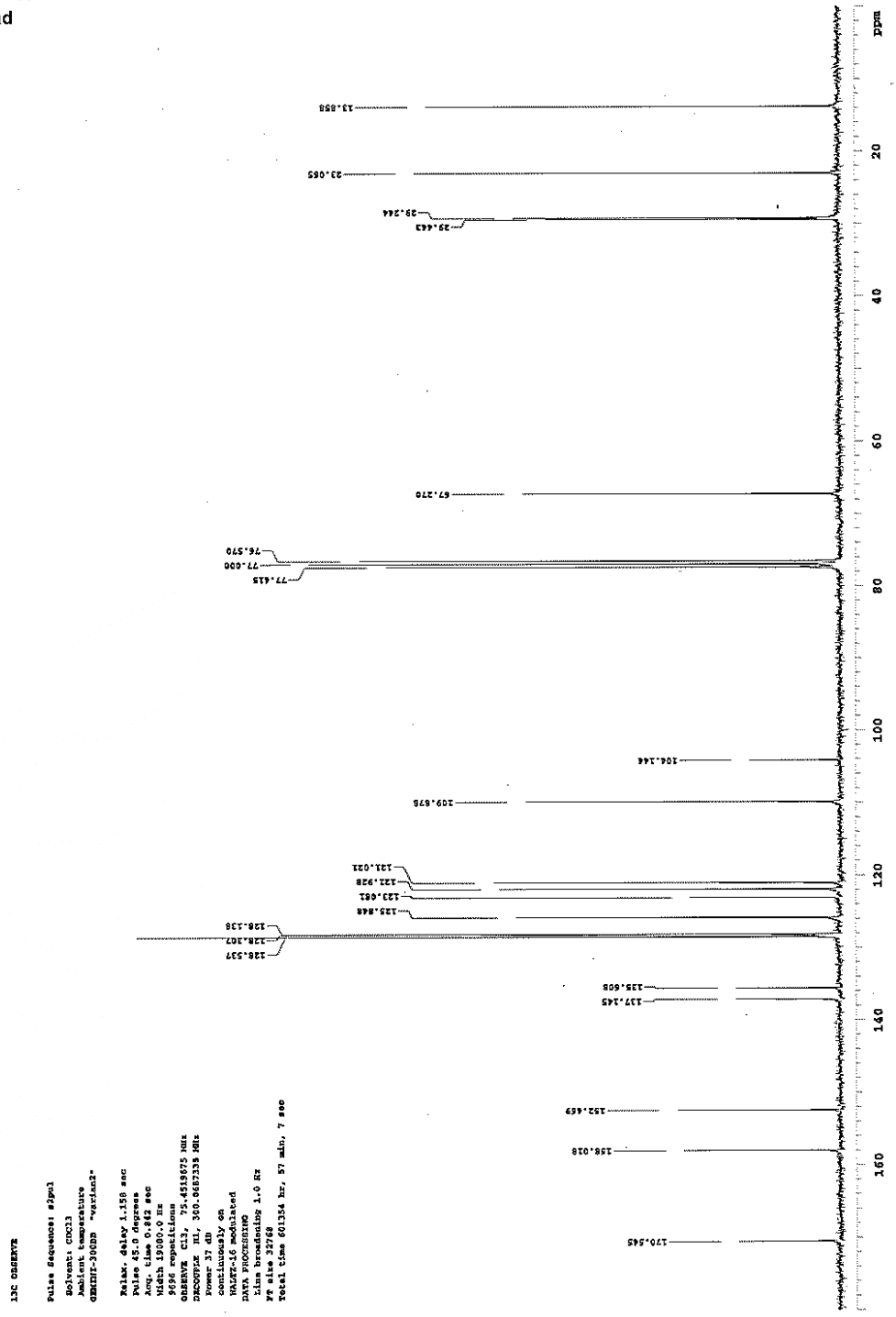

n-Bu
NHCbz
O
N
H

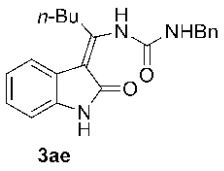
3ad


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 5009.0 Hz
16 repetitions
OBSERVE H1, 300.0672331 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec
ppm

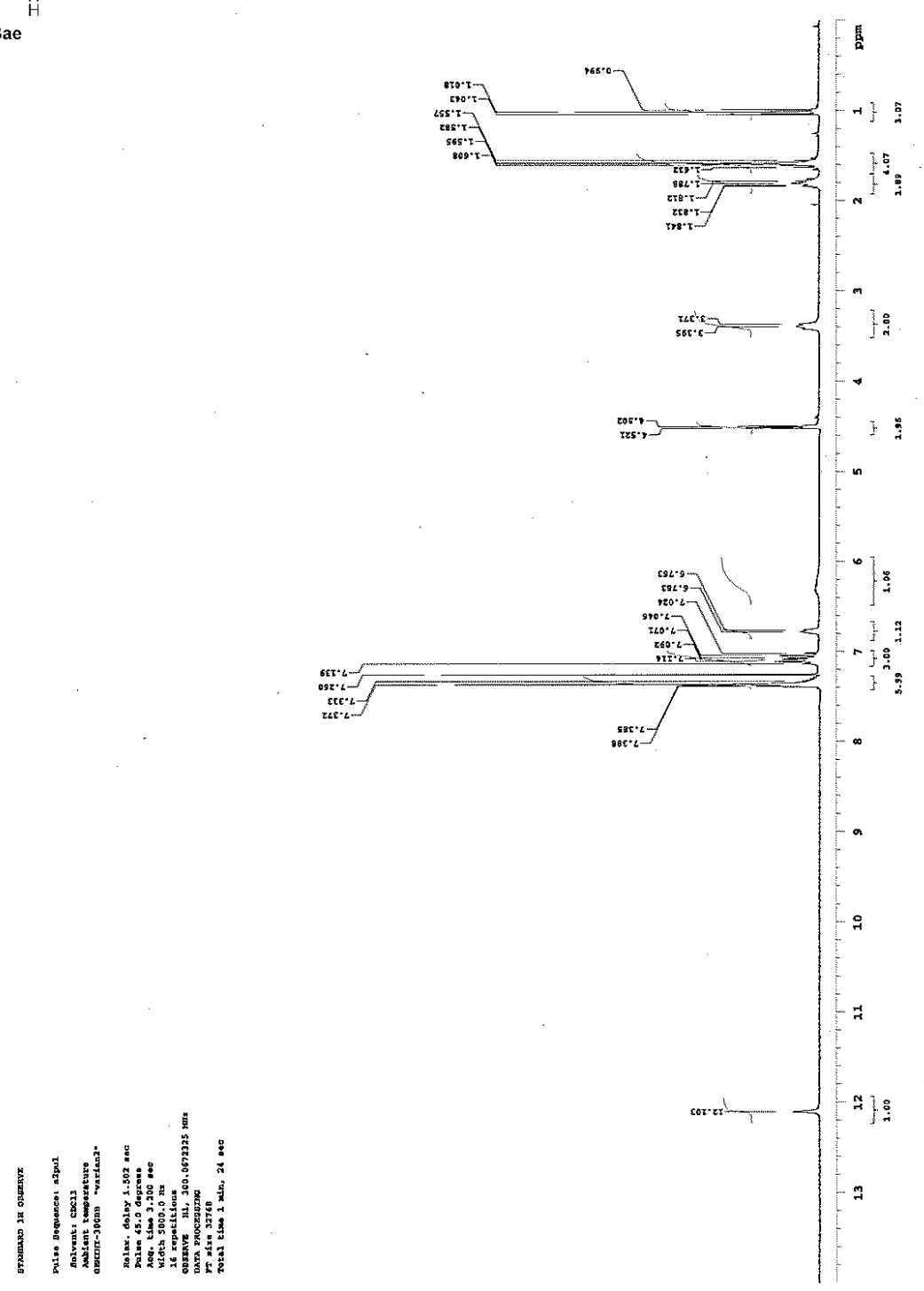

n-Bu
NHCbz
O
N
H

3ad


13.858
23.065
29.244
29.443
67.270
76.570
77.000
77.415
104.144
109.678
122.031
122.928
123.081
125.848
128.138
128.307
128.537
135.605
137.145
152.469
158.018
170.545
ppm
20
40
60
80
100
120
140
160



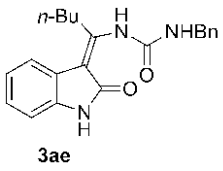
3ae



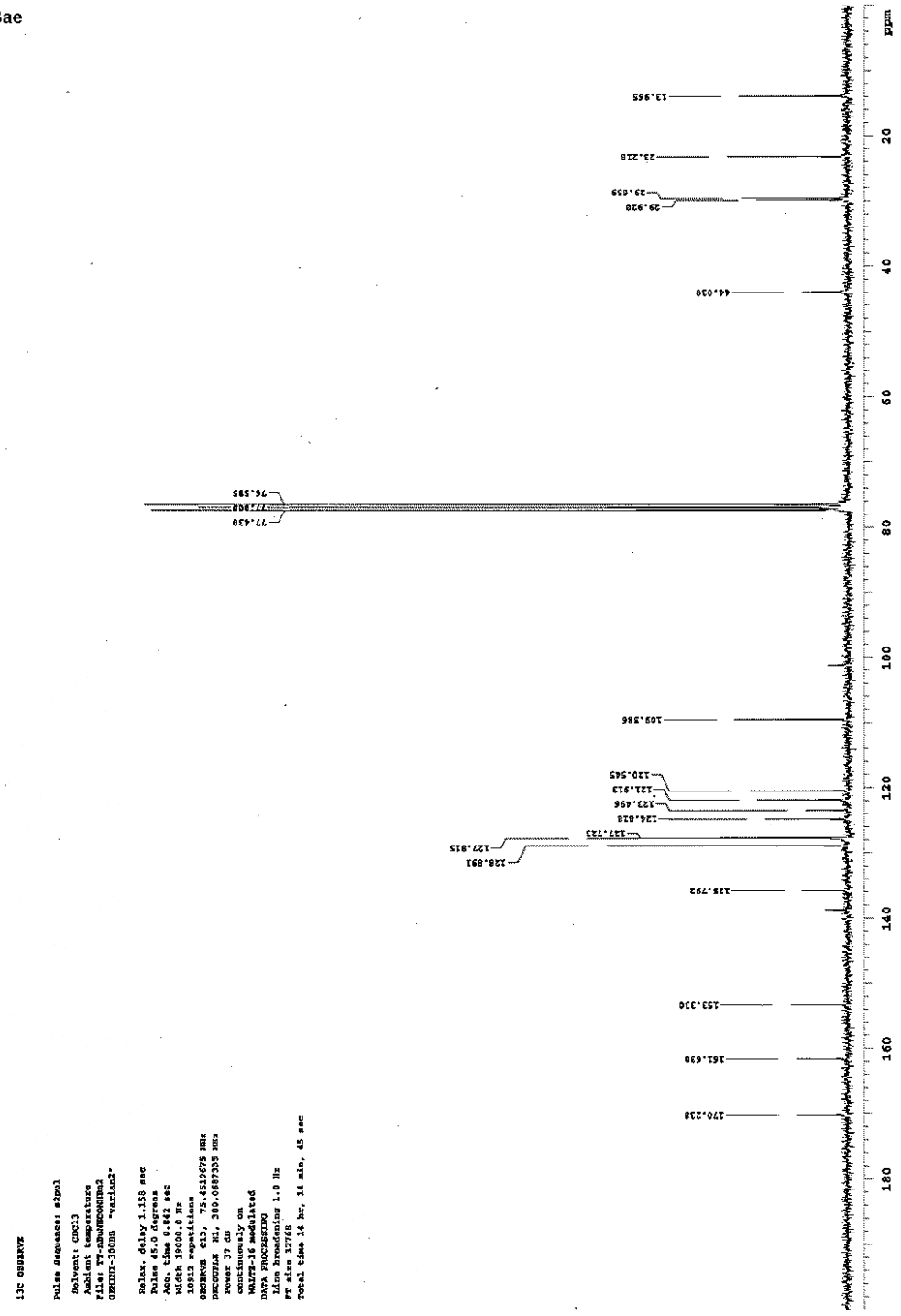
STANDARD 1H OBSERVE

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 H1, 300.0672325 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec



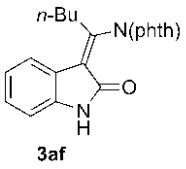
3ae



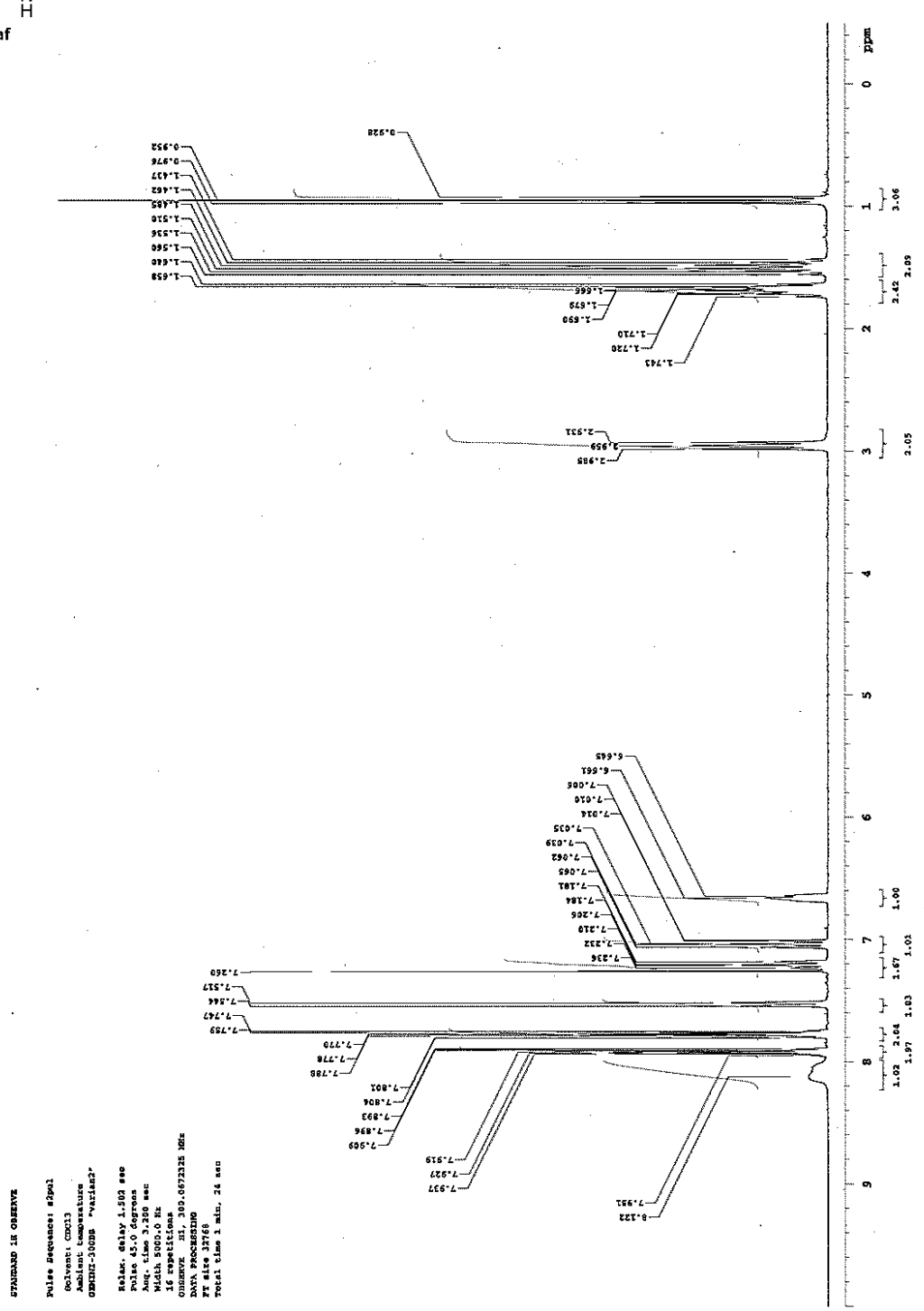
13C OBSERVE

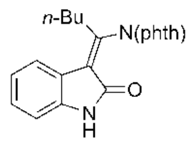
Pulse sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: YY-SHAMCHAMB2
GEMINI-300BB "varian"

Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
10513 repetitions
OBSERVE C13, 75.4519675 MHz
DECOUPLE H1, 300.0687335 MHz
Power 39 dB
continuously on
WALTZ16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 14 hr, 14 min, 45 sec


n-Bu
N(phth)
O
N
H

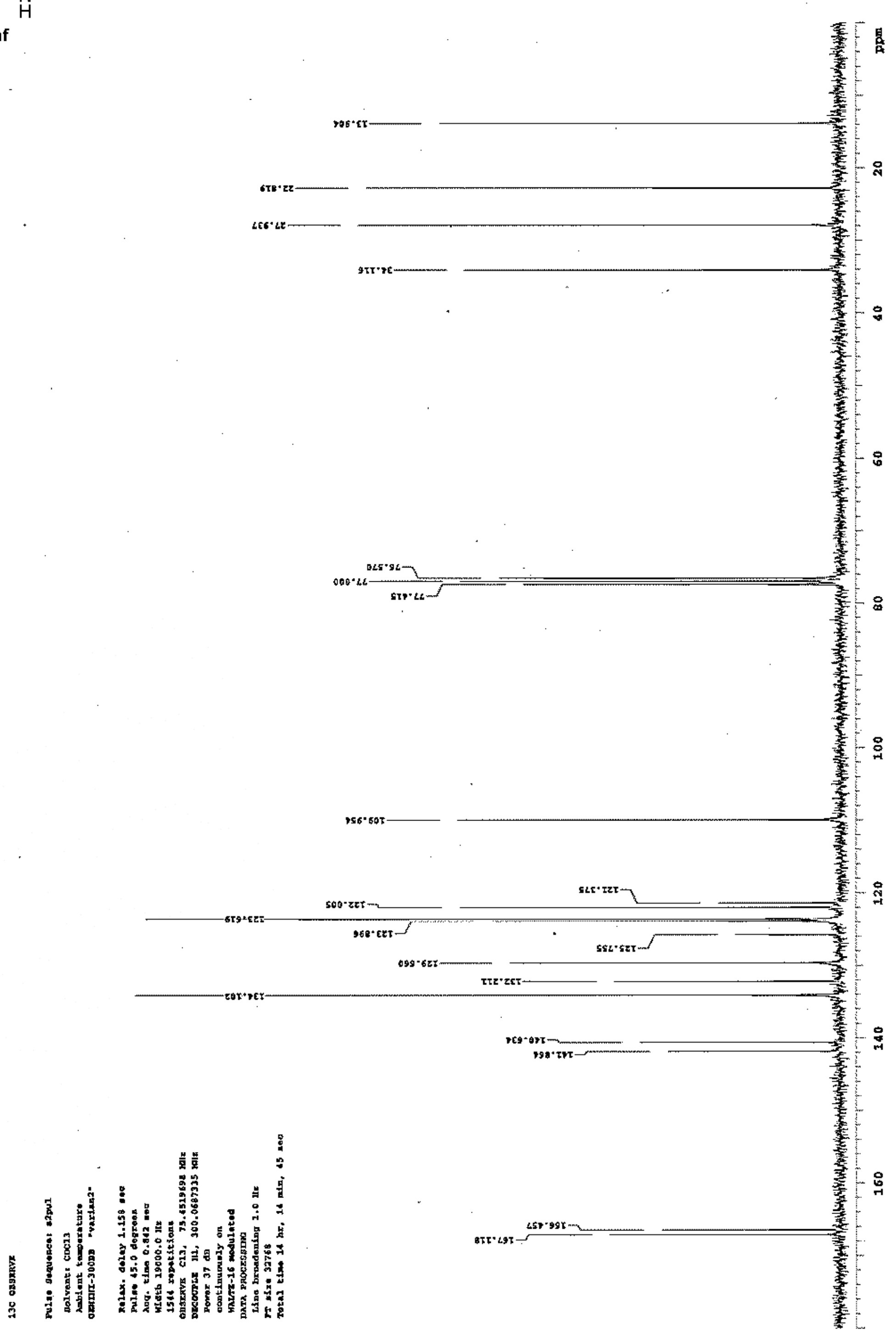
3af


STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
CHMIN2-300B "variab2"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672325 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec
ppm

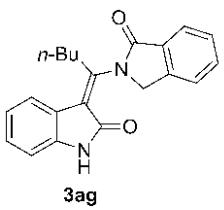


n-Bu
N(phth)
O
N
H

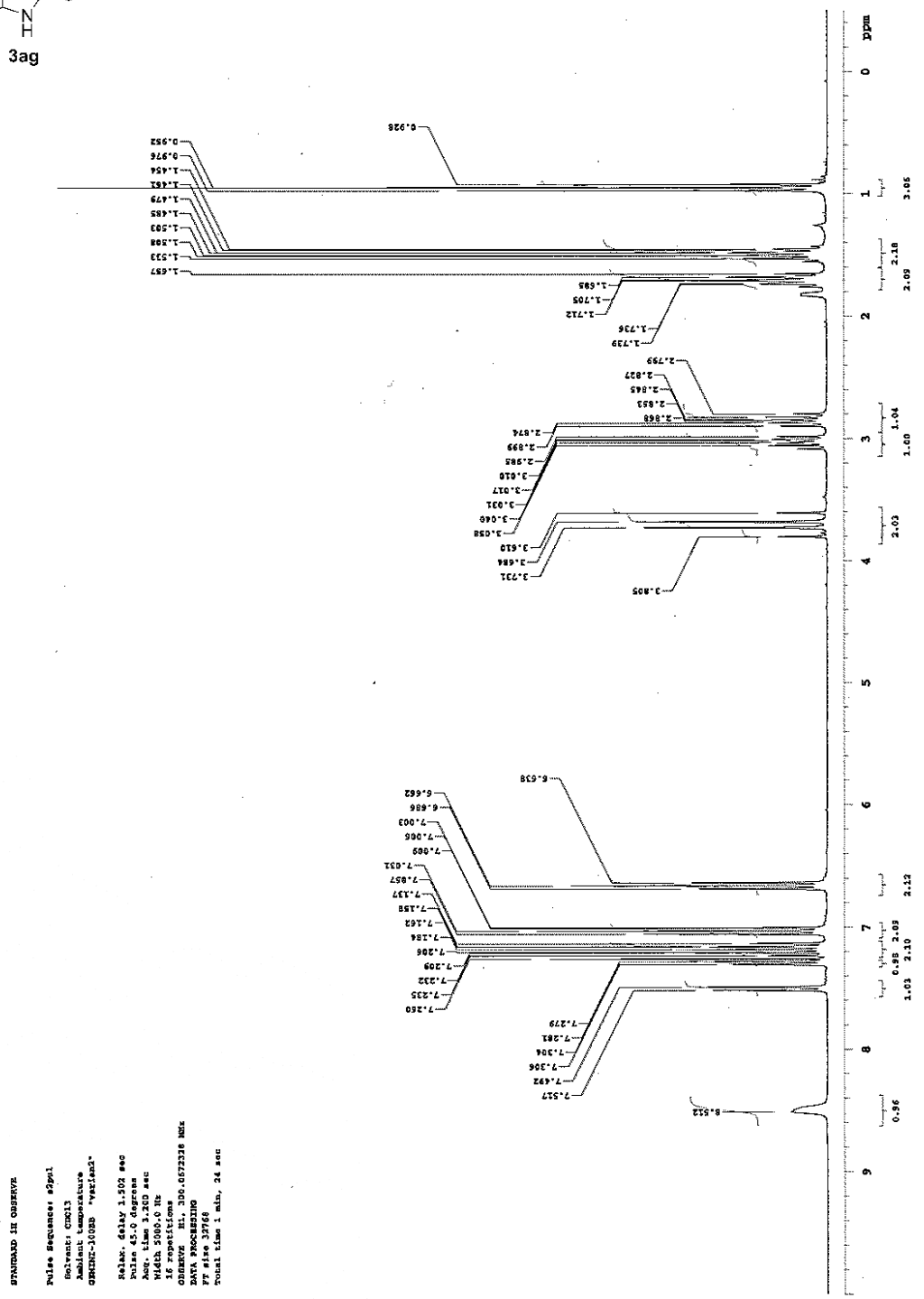
3af



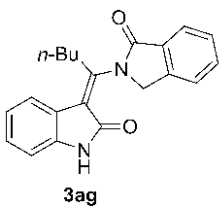
13.984
22.819
27.937
34.116
76.570
77.000
77.415
109.954
121.375
122.005
123.619
123.896
125.755
129.660
132.211
134.103
140.634
141.864
166.457
167.118
20
40
60
80
100
120
140
160
ppm
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
1544 repetitions
OBSERVE C13, 75.4519698 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 14 hr, 14 min, 45 sec



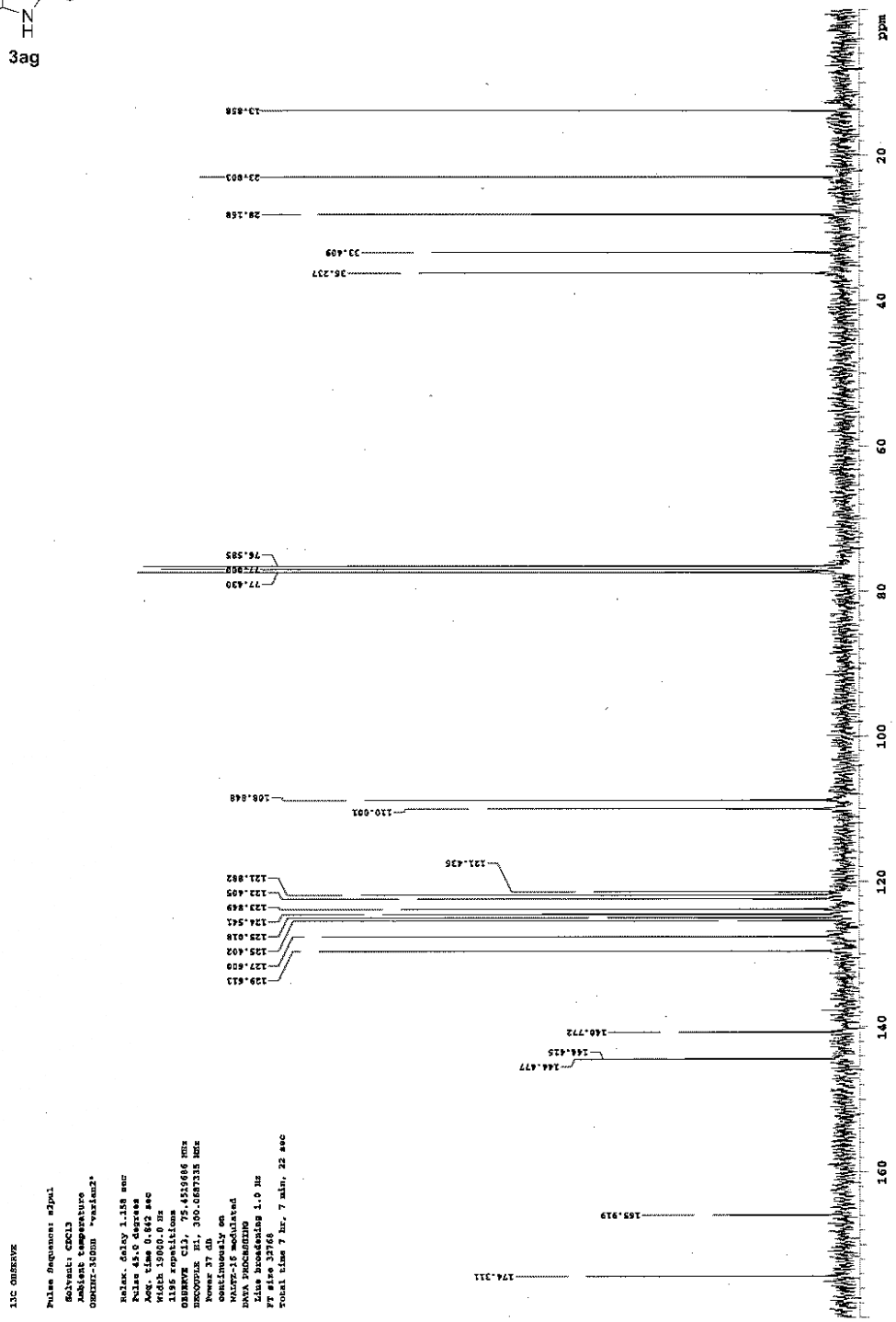
3ag




```
$STANDARD IN CDCl3CV2

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
CHMINZ-300B  "variansa"

Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.260 sec
Width 5000.0 Hz
16 repetitions
OBSERVE   H1, 300.0572336 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec
```



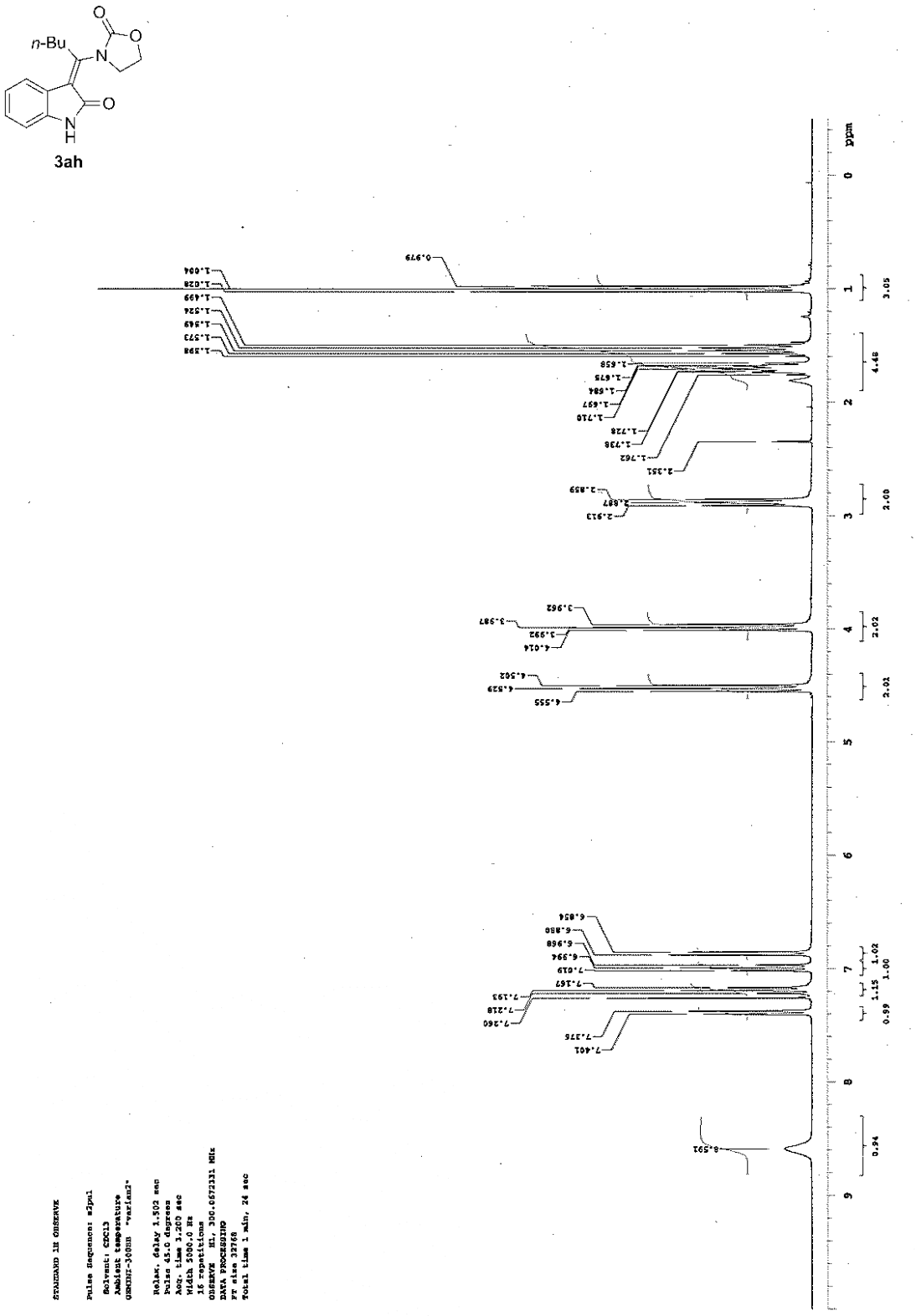
3ag



13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
OMNI07-500SB *variant2*

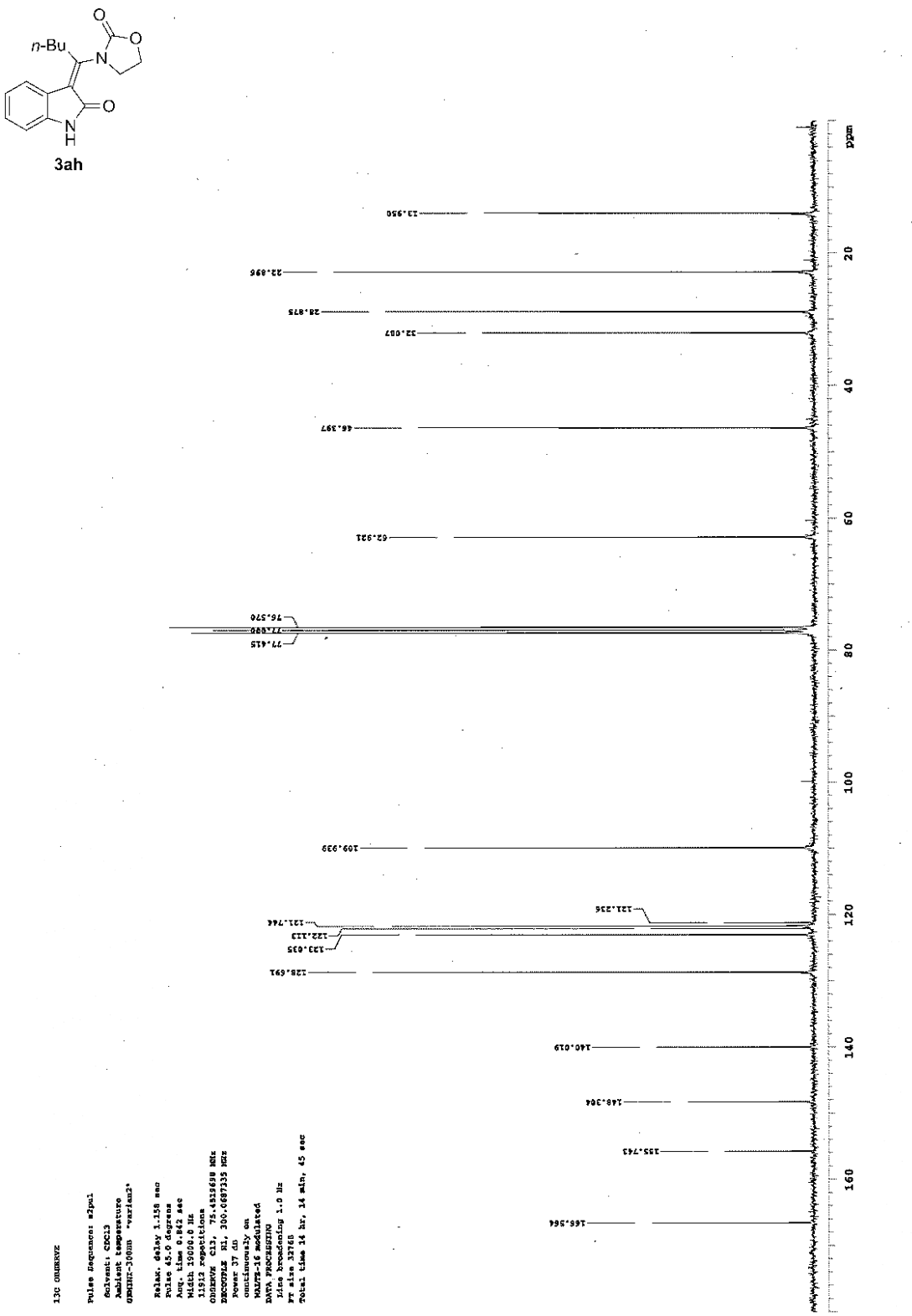
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.862 sec
Width 19000.0 Hz
1195 repetitions
OBSERVE C13, 75.4519686 MHz
DECOUPLE H1, 300.0697335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
LB 1.00 Hz
FT size 32768
Total time 7 hr, 7 min, 22 sec

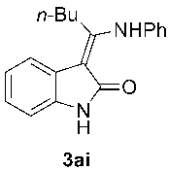


STANDARD 1H OBSERVE

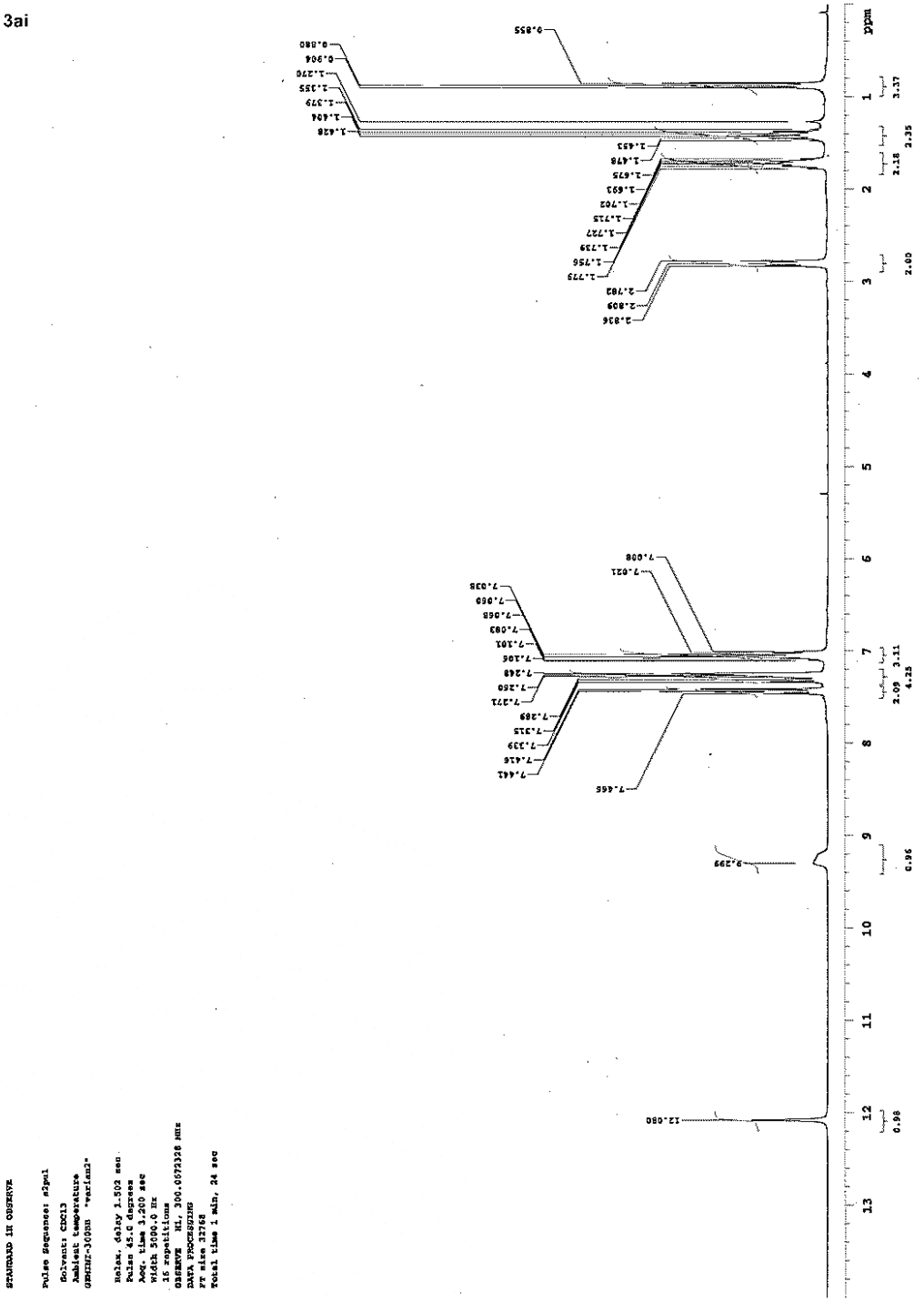
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
CHKOSI-300US "variant"

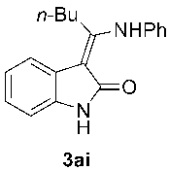
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 1.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0572331 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec


n-Bu
O
O
N
O
N
H
3ah
13.950
22.696
28.875
33.087
46.397
62.921
76.570
77.000
77.415
109.939
121.336
121.744
122.113
123.035
128.691
140.019
148.304
155.743
166.564
ppm
20
40
60
80
100
120
140
160
130 OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "variasz"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
11513 repetitions
OBSERVE C13, 75.4512698 MHz
DECOUPLE H1, 300.0697335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
Data processing
Line broadening 1.0 Hz
FT size 32768
Total time 14 hr, 14 min, 45 sec

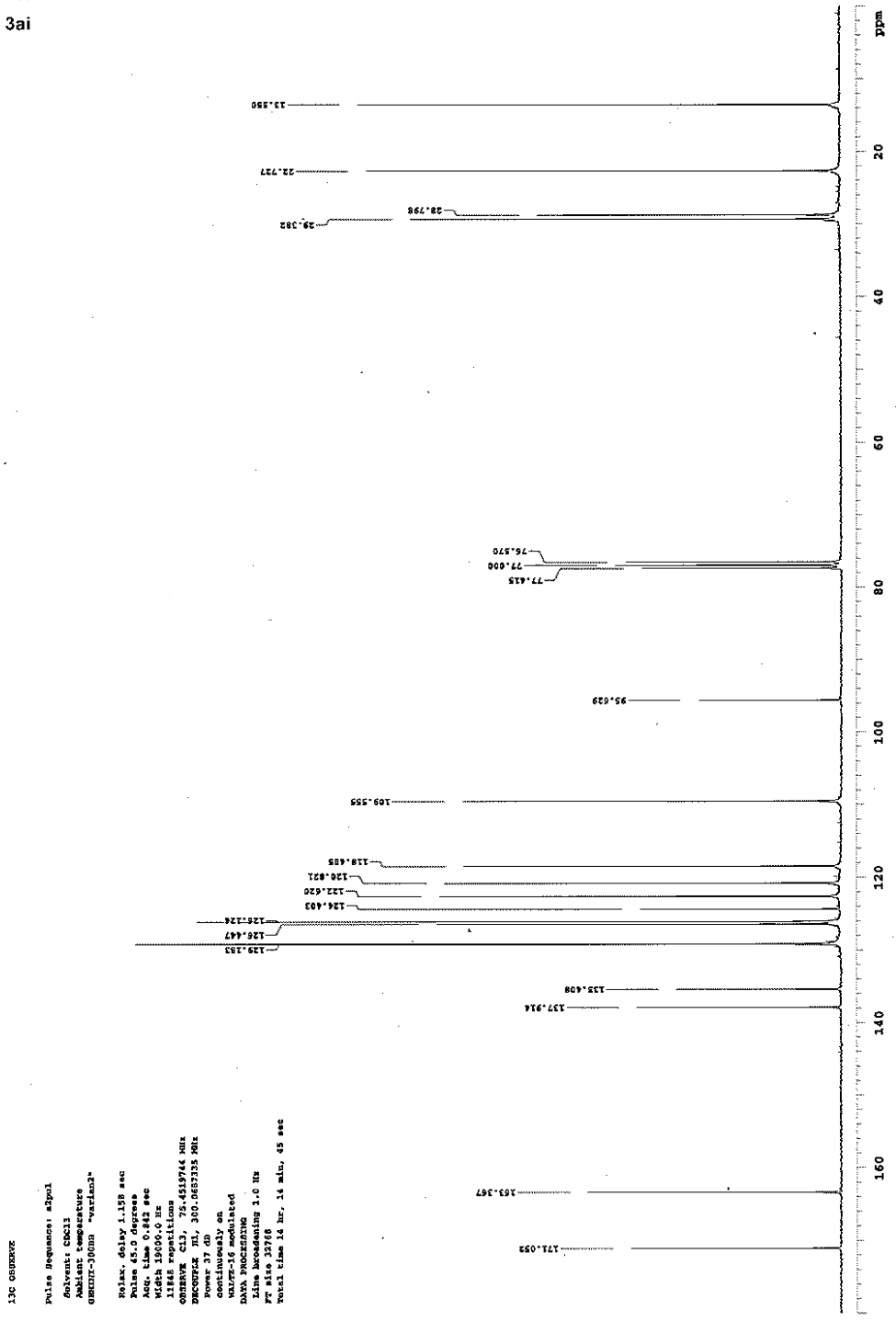

n-Bu
NHPh
O
N
H

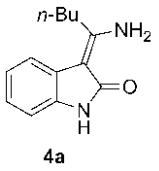
3ai


STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0573328 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec
ppm

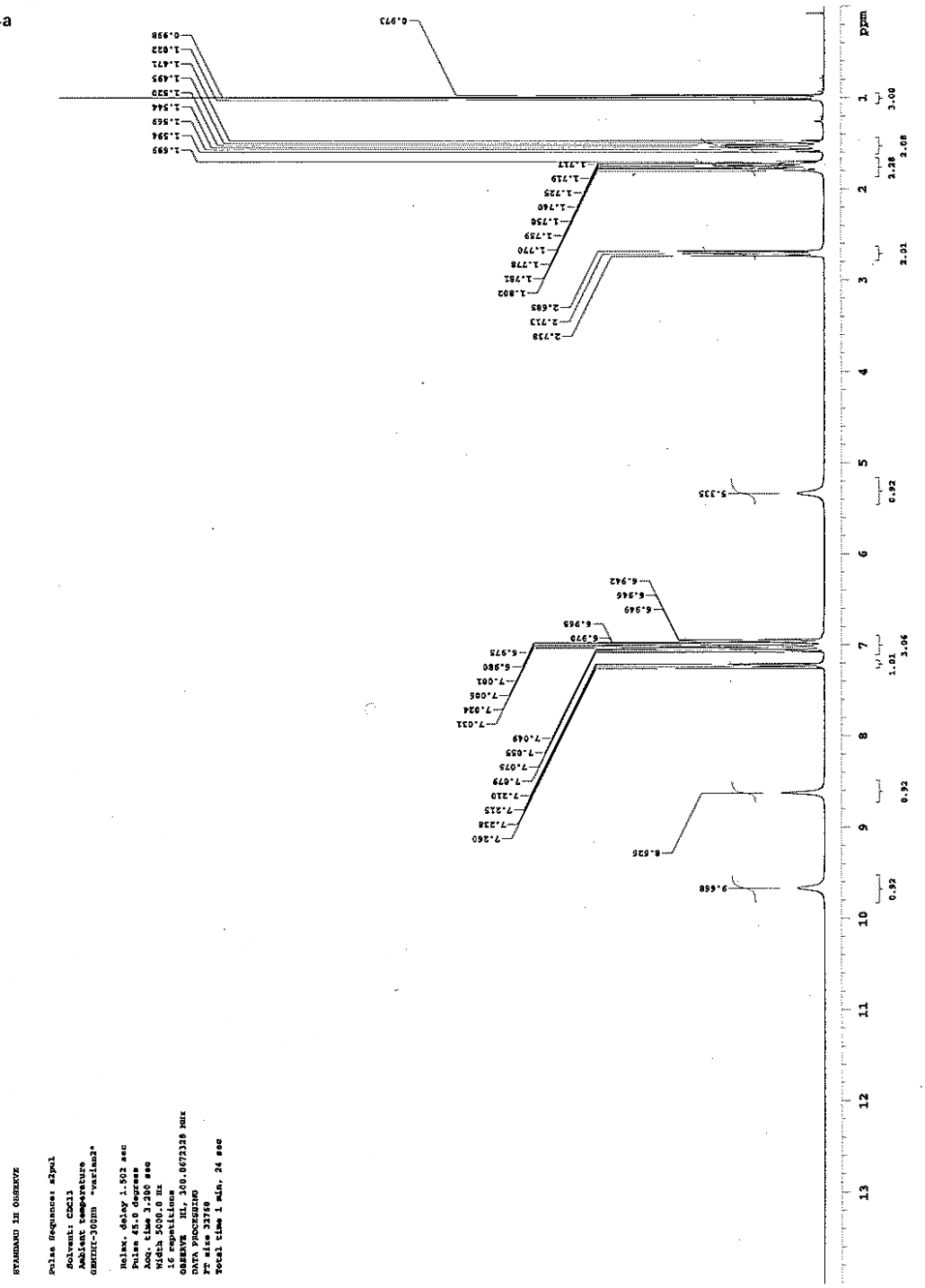

n-Bu
NHPh
O
N
H

3ai


13.350
22.737
28.798
29.282
75.570
77.000
77.425
95.629
109.555
118.685
120.821
122.620
124.403
126.124
126.447
129.383
133.608
137.914
163.367
172.053
ppm
20
40
60
80
100
120
140
160
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 19000.0 Hz
11848 repetitions
OBSERVE C13, 75.4519744 MHz
DECOUPLE H1, 300.0657335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 14 hr, 14 min, 45 sec



4a



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

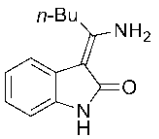
16 repetitions

OBSERVE H1, 300.0672328 MHz

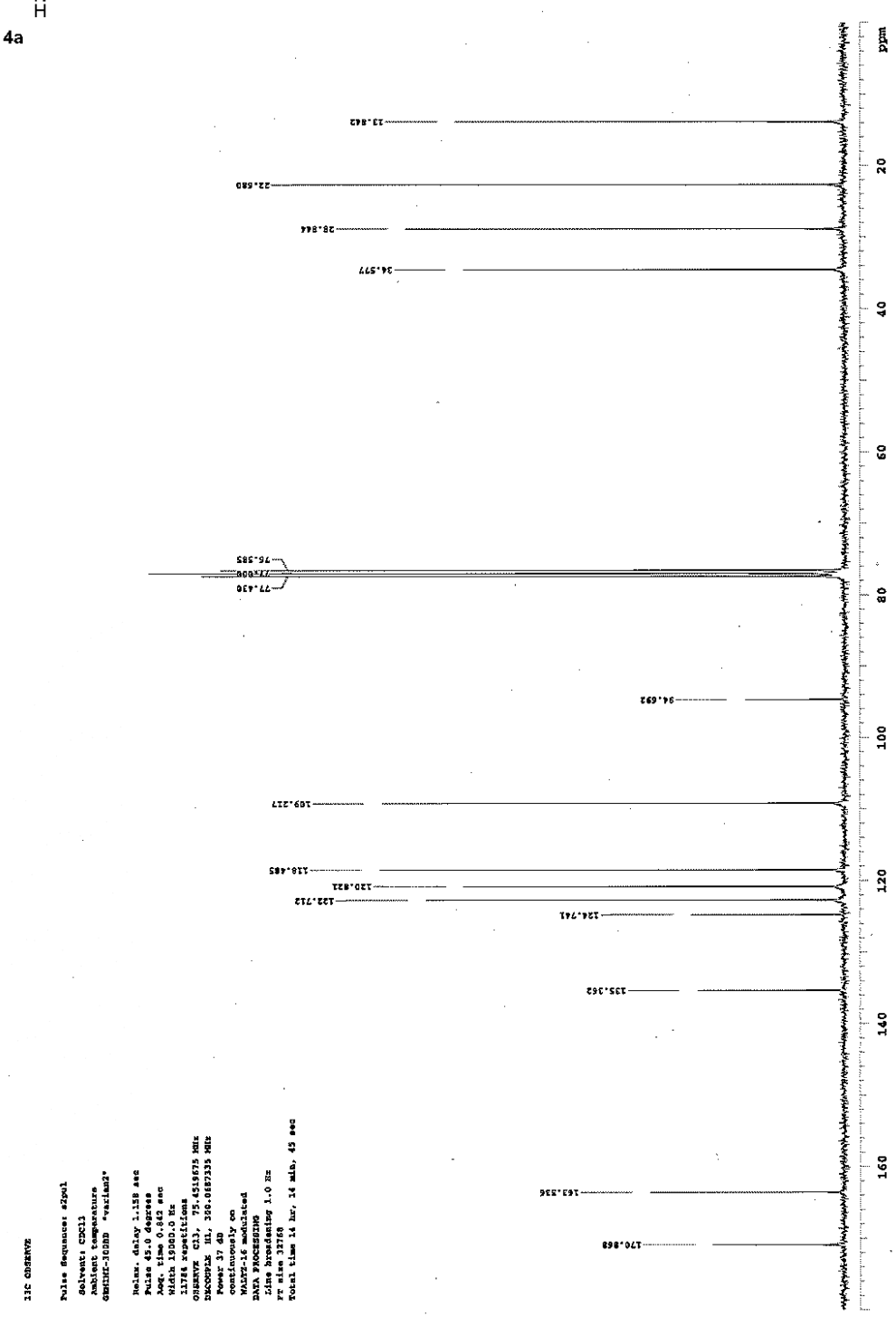
DATA PROCESSING

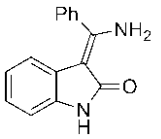
FT size 32768

Total time 1 min, 24 sec

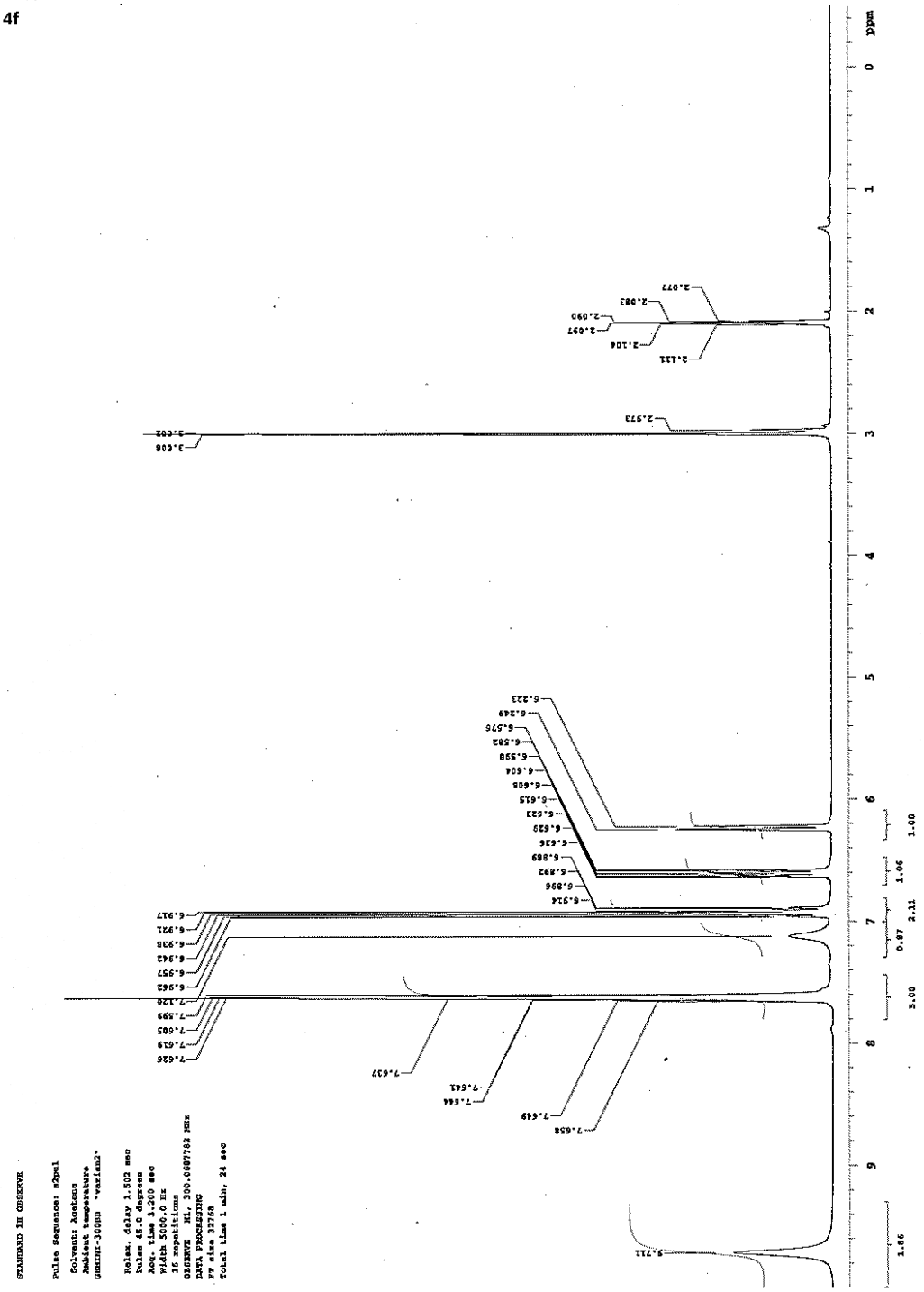

n-Bu
NH2
O
N
H

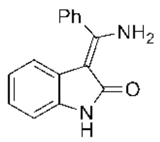
4a


13.843
22.680
28.844
34.577
76.585
77.006
77.430
94.692
109.217
118.685
120.821
122.712
124.741
135.362
163.336
170.869
ppm
20
40
60
80
100
120
140
160
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 19060.0 Hz
11784 repetitions
OBSERVE C13, 75.4519675 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 14 hr, 14 min, 45 sec

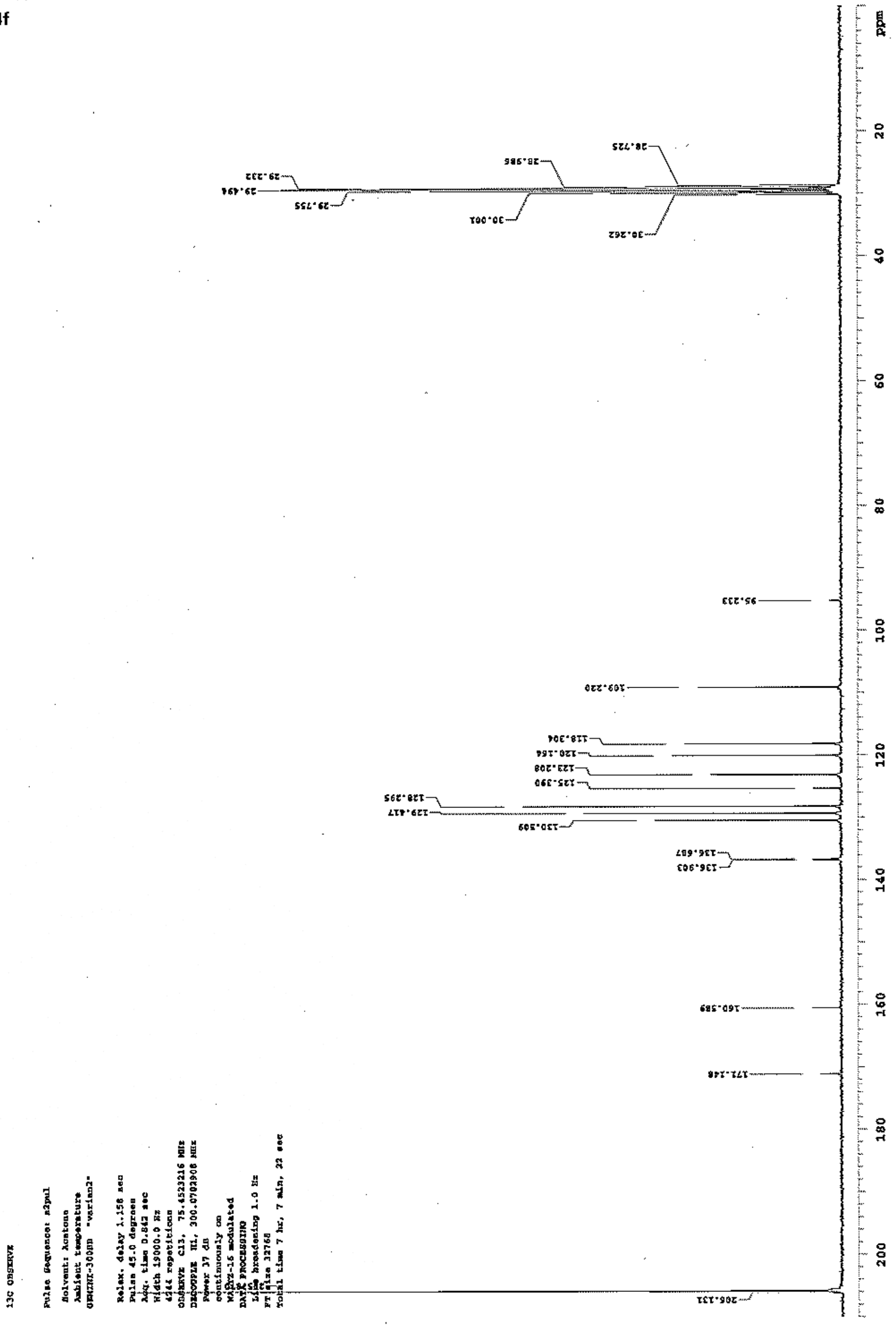

Ph
NH2
O
N
H

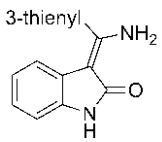
4f


STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: Acetone
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, 300.0687782 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec
ppm
2.077
2.083
2.090
2.097
2.104
2.111
2.573
3.082
3.088
6.323
6.349
6.576
6.582
6.598
6.604
6.608
6.615
6.623
6.629
6.636
6.889
6.892
6.896
6.924
6.917
6.921
6.938
6.943
6.957
6.963
7.126
7.599
7.605
7.619
7.636
7.637
7.644
7.661
7.649
7.658
9.711
1.00
1.06
2.11
0.87
5.00
1.86

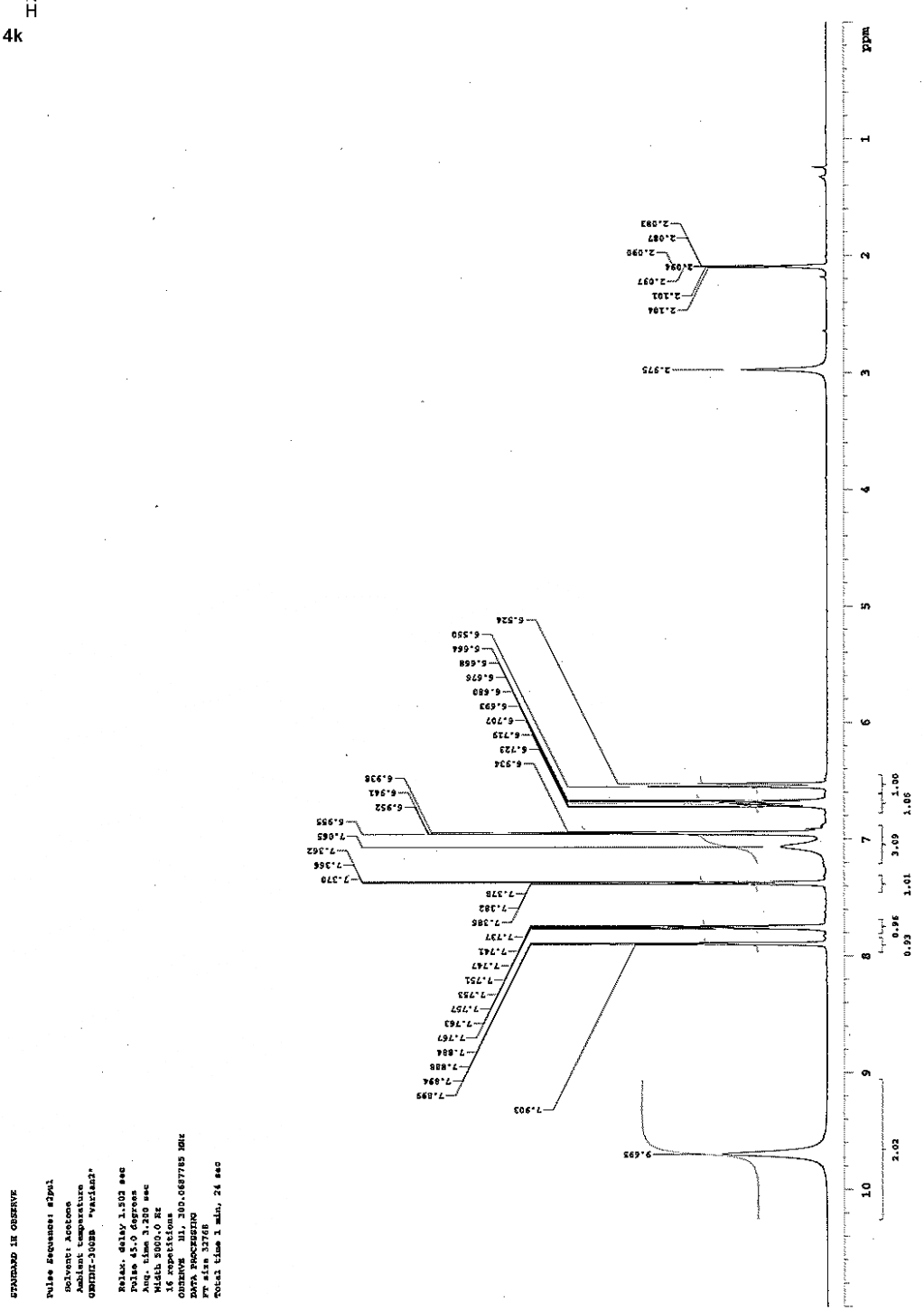

Ph
NH2
O
N
H

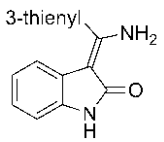
4f


28.725
28.986
29.233
29.494
29.755
30.001
30.262
95.233
109.230
118.304
120.164
123.208
125.390
128.295
129.417
130.309
136.687
136.903
160.589
171.148
206.131
20
40
60
80
100
120
140
160
180
200
ppm

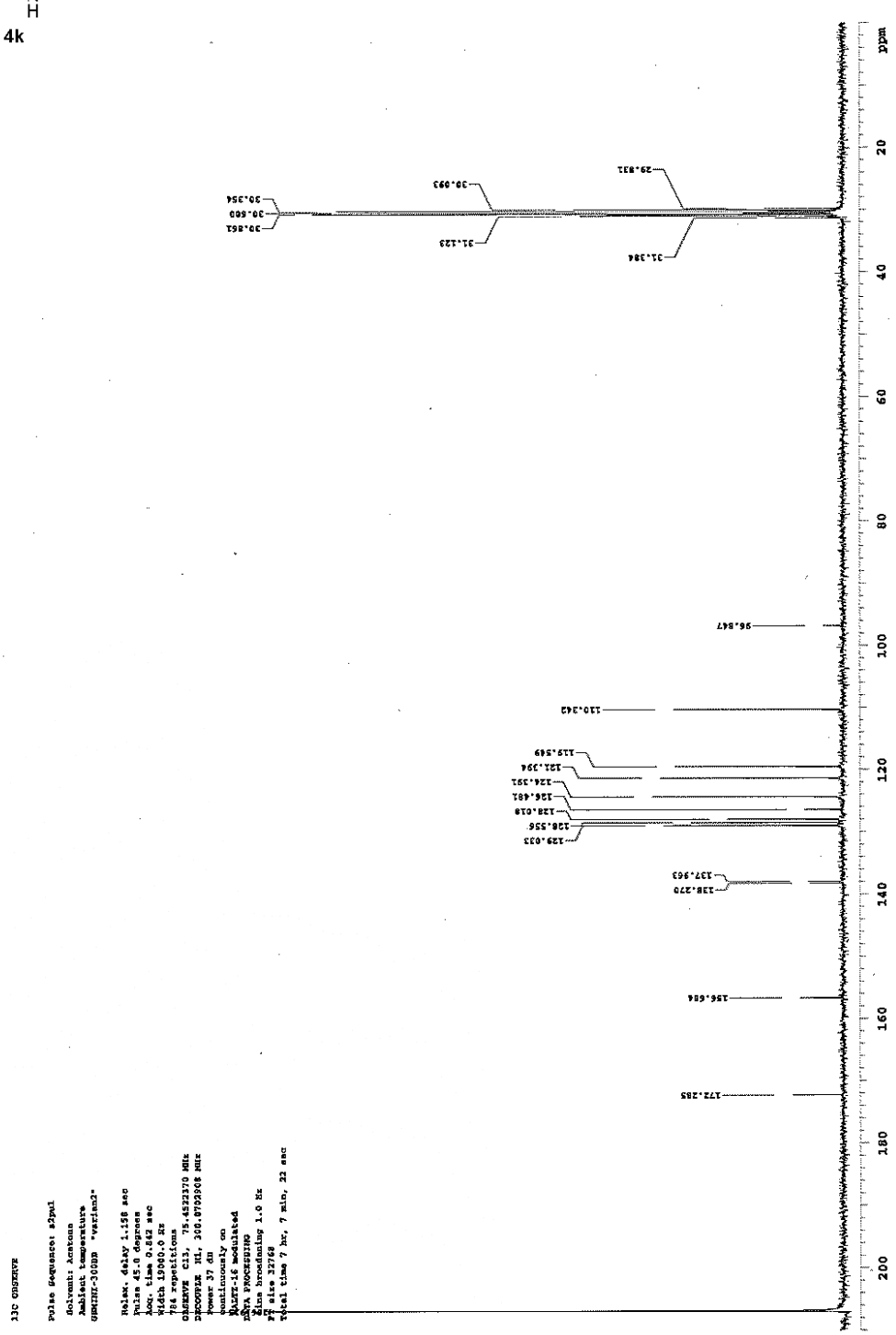

3-thienyl
NH2
O
N
H

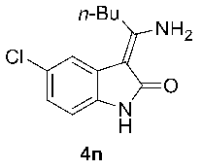
4k


STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: Acetone
Ambient temperature
OMNMR-300SB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0687785 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec
ppm

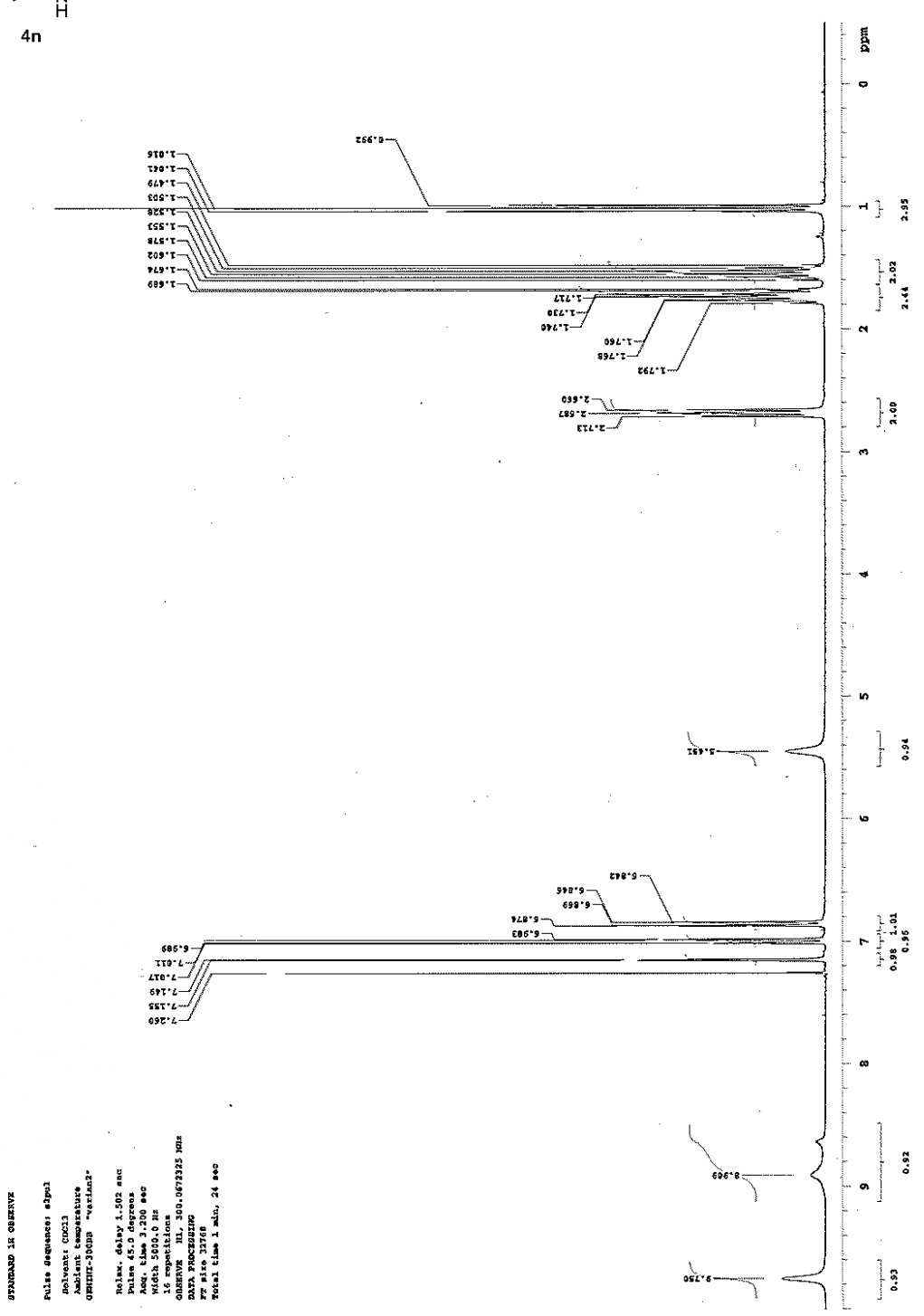

3-thienyl
NH2
O
N
H

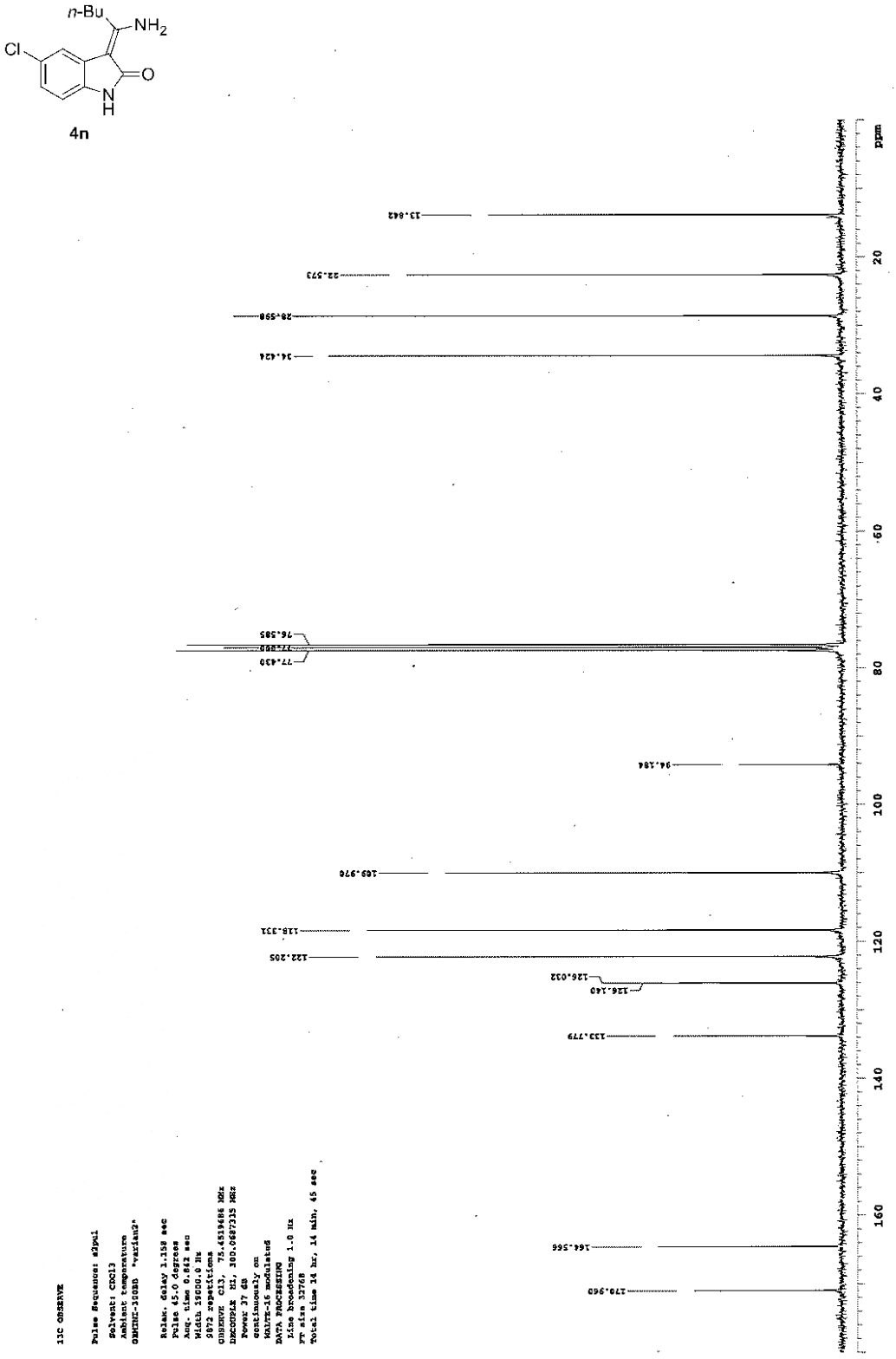
4k


13C OBSERVE
Pulse Sequence: s2pul
Solvent: Acetone
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.862 sec
Width 19060.0 Hz
784 repetitions
OBSERVE C13, 75.4522370 MHz
DECOUPLE H1, 300.0702908 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
EXP. PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 7 hr, 7 min, 22 sec
29.831
30.093
30.354
30.560
30.862
31.123
31.384
96.847
130.343
132.394
139.033
137.963
138.370
156.654
172.285
ppm


n-Bu
NH2
Cl
O
N
H

4n


STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
OMNMR-300SB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5600.0 Hz
16 repetitions
OBSERVE H1, 300.0672325 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec
ppm


n-Bu
NH2
Cl
O
N
H
4n
13.843
22.573
28.598
34.424
76.585
77.000
77.430
94.184
169.976
118.331
122.205
126.032
126.140
133.779
164.566
170.960
ppm
20
40
60
80
100
120
140
160
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
OMNMR-300BB "varian2"
Relax. delay 1.198 sec
Pulse 45.0 degrees
Acq. time 0.862 sec
Width 19000.0 Hz
5872 repetitions
OBSERVE C13, 75.4539686 MHz
DECOUPLE H1, 300.0687315 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 14 hr, 14 min, 45 sec