Supplementary Information

Stereoselective synthesis of vinyl-substituted (*Z*)-stilbenes by rhodium-catalysed addition of arylboronic acids to allenic alcohols

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General. Infrared spectra were recorded on a Shimadzu FTIR-8100 spectrometer. ^{1}H and ^{13}C NMR spectra were recorded on a Varian Gemini 2000 (^{1}H at 300 MHz and ^{13}C at 75 MHz), a JNM-ECS 400 (^{1}H at 400 MHz and ^{13}C at 100 MHz) or a Bruker AVANCE 500 (^{1}H at 500MHz and ^{13}C at 125MHz) spectrometer using $CHCl_3$ (^{1}H, $\delta = 7.26$) and $CDCl_3$ (^{13}C, $\delta = 77.0$) as an internal standard. Low-resolution mass spectra were recorded on a Shimadzu PARVUM 2 (GC/MS). High-resolution mass spectra were recorded on a JEOL JMS-SX102A. All reactions were carried out under an argon atmosphere unless otherwise noted. Flash column chromatography was performed with basic silica gel NH-DM1020 (Fuji Silysia Chemical Ltd) or silica gel 60 N (Kanto). Preparative thin-layer chromatography was performed with silica gel 60 PF254 (Merck).

Materials. Unless otherwise noted, all chemicals were obtained from commercial suppliers and used as received. Anhydrous MeOH (Nacalai) was purchased and distilled from magnesium. $[Rh(OH)(cod)]_2$ was prepared according to the literature procedure.[1] Allenic alcohols (**1a**, **1c**, **1d**, **1e** and **4b**) were prepared from the corresponding propargyl alcohols by the literature procedures.[2,3] Allenic alcohols (**1b** and **4a**) were prepared from the corresponding allenic esters[4] according to the reported procedure.[5] The analytical data of compounds (**1a**,[2] **1b**,[6] **3ac**,[7] **3ad**,[8] **4a**,[3] and **4b**[3]) have been already reported.

1c: IR (neat): 3397, 3032, 2930, 1950, 1703, 1599, 1495, 1456, 1375, 1152 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 1.44$ (6H, s), 5.79 (1H, d, $J = 6.6$ Hz), 6.33 (1H, d, $J = 6.6$ Hz), 7.13–7.25 (1H, m), 7.25–7.35 (4H, m); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 30.4, 70.5, 98.2, 105.3, 126.8, 127.3, 128.8, 134.2, 202.1$; MS ($EI^+$): m/z (%) 174 ($M^+$, 5), 116 (100), 89 (8), 59 (87); HRMS (EI^+): Calcd for $C_{12}H_{14}O$, M^+ 174.1045. Found m/z 174.1047.

1d: IR (neat): 3347, 2930, 2874, 1952, 1491, 1092, 1013 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta =$ 4.20–4.33 (2H, m), 5.73–5.85 (1H, m), 6.23–6.33 (1H, m), 7.17–7.35 (4H, m); ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 60.4, 96.4, 96.5, 128.2, 129.0, 132.5, 133.0, 204.5$; MS ($EI^+$): m/z (%) 180 ($M^+$, 28), 149 (86), 127 (31), 115 (100); HRMS (EI^+): Calcd for $C_{10}H_9ClO$, M^+ 180.0342. Found m/z 180.0345.

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1e: IR (neat): 3386, 2838, 1948, 1607, 1512, 1464, 1441, 1300, 1248, 1173, 1032 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ = 3.80 (3H, s), 4.18–4.32 (2H, m), 5.73–5.82 (1H, m), 6.26–6.34 (1H, m), 6.80–6.90 (2H, m), 7.19–7.30 (2H, m); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 55.5, 60.7, 96.0, 97.0, 114.4, 126.2, 128.1, 159.2, 203.7; MS (EI^+): m/z (%) 176 (M^+, 37), 145 (100), 115 (13), 102 (18); HRMS (EI^+): Calcd for $C_{11}H_{12}O_2$, M^+ 176.0837. Found m/z 176.0836.

Typical procedure for the rhodium-catalysed reaction of arylboronic acids to allenic alcohols: An oven-dried flask was charged with **2a** (27.1 mg, 0.20 mmol), $B(OH)_3$ (123.6 mg, 2.0 mmol) and a solution of **1a** (29.4 mg, 0.20 mmol) in MeOH (2.0 mL). Then, $[Rh(OH)(cod)]_2$ (2.3 mg, 5.0 μmol) was added and the flask was flushed with argon. After stirred at room temperature for 3 h, the reaction mixture was diluted with ethyl acetate (10 mL) and passed through a pad of basic silica gel (Fuji Silysia Chemical Ltd., NH-DM1020). The filtrate was concentrated under reduced pressure and the residue was purified by preparative thin-layer chromatography (hexane/ethyl acetate 50:1) to give the product **3aa** as a colorless oil (31.6 mg, 0.14 mmol, 71%, *Z*/*E* = 98:2).

3aa: IR (neat): 3061, 3022, 2922, 1601, 1493, 1447 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ = 2.10 (3H, s), 4.67 (1H, d, *J* = 17.1Hz), 5.10 (1H, d, *J* = 10.5 Hz), 6.63 (1H, s), 6.73 (1H, dd, *J* = 17.1, 10.5 Hz), 6.81–6.92 (2H, m), 7.00–7.15 (4H, m), 7.15–7.35 (3H, m); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 19.2, 115.9, 126.3, 127.0, 127.5, 128.1, 128.9, 129.4, 130.3, 131.5, 136.2, 136.8, 137.3, 141.1; MS (EI^+): m/z (%) 220 (M^+, 100), 205 (79), 129 (54), 105 (74); HRMS (EI^+): Calcd for $C_{17}H_{16}$, M^+ 220.1252; Found m/z 220.1251.

3ab: IR (KBr): 3029, 2921, 1599, 1493, 1445, 1408, 1076 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ = 2.35 (3H, s), 4.85 (1H, d, *J* =17.1Hz), 5.15 (1H, d, *J* = 10.5Hz), 6.58 (1H, s), 6.73 (1H, dd, *J* = 17.1, 10.5 Hz), 6.87–7.00 (4H, m), 7.05–7.20 (4H, m), 7.23–7.33 (1H, m); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 21.7, 116.5, 126.7, 127.0, 128.1, 128.2, 128.8, 129.6, 130.2, 131.5, 136.9, 137.9, 138.5, 142.0; MS (EI^+): m/z (%) 220 (M^+, 100), 205 (77), 128 (54), 105 (84); HRMS (EI^+): Calcd for $C_{17}H_{16}$, M^+ 220.1252. Found m/z 220.1254.

3ac: IR (KBr): 2919, 1599, 1512, 1445, 1109 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ = 2.40 (3H, s), 4.87 (1H, d, *J* = 17.1 Hz), 5.15 (1H, d, *J* = 10.5 Hz), 6.59 (1H, s), 6.73 (1H, dd, *J* = 17.1, 10.5 Hz), 6.88–6.98 (2H, m), 7.13–7.15 (5H, m), 7.16–7.24 (2H, m); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 21.5, 116.4, 127.0, 128.1, 129.60, 129.64, 131.5, 134.9, 137.0, 141.9, 142.1; MS (EI^+): m/z (%) 220 (M^+, 96), 205 (82), 128 (49), 105 (100); HRMS (EI^+): Calcd for $C_{17}H_{16}$, M^+ 220.1252. Found m/z 220.1251.

3ae: IR (neat): 3084, 2834, 1597, 1491, 1248, 1097 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ = 3.73 (3H, s), 4.83 (1H, d, *J* = 17.1 Hz), 5.16 (1H, d, *J* = 9.6 Hz), 6.75 (1H, s), 6.80 (1H, dd, *J* = 17.1, 10.2 Hz), 6.95–7.20 (8H, m), 7.35–7.45 (1H, m); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 55.9, 111.6, 115.5, 121.3, 126.8, 127.0, 128.1, 129.0, 129.7, 131.1, 132.2, 137.1, 138.5, 141.0, 157.2; MS (EI^+): m/z (%) 236 (M^+, 100), 221 (31), 202 (35), 121 (71); HRMS (EI^+): Calcd for $C_{17}H_{16}O$, M^+ 236.1201. Found m/z 236.1202.

3af: IR (KBr): 3017, 2934, 1603, 1509, 1441, 1287, 1244, 1183, 1173, 1030 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ = 3.85 (3H, s), 4.89 (1H, dd, *J* = 17.4 Hz), 5.15 (1H, dd, *J* = 11.1 Hz), 6.59 (1H, s), 6.73 (1H, dd, *J* = 17.1, 10.5Hz), 6.89–6.99 (4H, m), 7.05–7.15 (5H, m); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 55.4, 114.4, 116.4, 127.0, 128.1, 129.6, 130.1, 130.9, 131.6, 137.0, 141.5, 142.2, 159.0; MS (EI^+): m/z (%) 236 (M^+, 100), 221 (31), 205 (34), 121 (89); HRMS (EI^+): Calcd for $C_{17}H_{16}O$, M^+ 236.1201. Found m/z 236.1202.

3ag: IR (KBr): 3092, 3046, 1597, 1485, 1445, 1406, 1389, 1069 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ = 4.83 (1H, d, *J* = 17.4 Hz), 5.17 (1H, d, *J* = 10.5 Hz), 6.63 (1H, s), 6.73 (1H, dd, *J* = 16.5, 10.5 Hz), 6.85–6.98 (2H, m), 7.03–7.20 (5H, m), 7.50–7.59 (2H, m); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 116.6, 121.5, 127.3, 128.2, 129.5, 131.6, 132.1, 132.2, 136.4, 136.9, 140.5, 141.4; MS (EI^+): m/z (%) 284 (M^+, 50), 269 (10), 205 (100), 169 (40); HRMS (EI^+): Calcd for $C_{16}H_{13}Br$, M^+ 284.0201. Found m/z 284.0203.

3ah: IR (neat): 2951, 1723, 1607, 1435, 1277, 1103 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ = 3.94 (3H, s), 4.79 (1H, d, *J* = 17.1 Hz), 5.17 (1H, dd, *J* = 10.5 Hz), 6.65 (1H, s), 6.74 (1H, dd, *J* = 17.1, 10.5 Hz), 6.85–6.95 (2H, m), 7.05–7.15 (3H, m), 7.24–7.32 (2H, m), 8.04–8.12 (2H, m); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 52.3, 116.7, 127.3, 128.2, 129.3, 129.5, 130.0, 130.2, 132.1, 136.3, 140.8, 141.2, 143.3, 167.1; MS (EI^+): m/z (%) 264 (M^+, 89), 233 (17), 205 (100), 149 (44); HRMS (EI^+): Calcd for $C_{18}H_{16}O_2$, M^+ 264.1150. Found m/z 264.1151.

3ai: IR (neat): 3075, 2926, 1599, 1491, 1449, 1225 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ = 5.14 (1H, d, *J* = 16.5 Hz), 5.24 (1H, d, *J* = 10.2 Hz), 6.73 (1H, s), 6.76 (1H, dd, *J* = 16.8, 10.5 Hz), 6.86–6.92 (1H, m), 6.99–7.21 (6H, m), 7.34–7.42 (1H, m); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 116.5, 126.0, 127.28, 127.34, 127.35, 128.0, 129.4, 133.9, 134.1, 136.3, 137.9, 141.2; MS (EI^+): m/z (%) 212 (M^+, 100), 197 (38), 178 (87), 128 (66); HRMS (EI^+): Calcd for $C_{14}H_{12}S$, M^+ 212.0660. Found m/z 212.0663.

3aj: IR (KBr): 3102, 2924, 1597, 1489, 1444, 997 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ = 5.01 (1H, d, *J* = 17.1 Hz), 5.18 (1H, d, *J* = 9.6 Hz,), 6.64 (1H, s), 6.72 (1H, dd, *J* = 17.1, 11.4 Hz), 6.85–7.05 (3H, m), 7.05–7.23 (3H, m), 7.23–7.43 (1H, m); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 116.3, 123.7, 125.9, 127.2, 128.2, 129.1, 129.4, 132.5, 136.7, 136.9, 137.6, 141.4; MS (EI^+): m/z (%) 212 (M^+, 100), 197 (35), 178 (70), 128 (40); HRMS (EI^+): Calcd for $C_{14}H_{12}S$, M^+ 212.0660. Found m/z 212.0664.

3ak: IR (neat): 3024, 2926, 1599, 1493, 1449 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ = 5.19 (1H, d, *J* = 10.8 Hz), 5.52 (1H, d, *J* = 17.1 Hz), 6.58 (1H, dd, *J* = 17.1, 10.8 Hz), 6.65 (1H, s), 6.72 (1H, d, *J* = 16.5 Hz), 7.06 (1H, d, *J* = 16.5 Hz), 7.10–7.43 (10H, m); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 116.7, 125.9, 126.7, 127.3, 127.8, 128.3, 128.4, 128.8, 130.0, 132.6, 137.5, 137.6, 137.8, 138.2; MS (EI^+): m/z (%) 232 (M^+, 100), 215 (41), 141 (83), 128 (94); HRMS (EI^+): Calcd for $C_{18}H_{16}$, M^+ 232.1252. Found m/z 232.1254.

3al: IR (neat): 3023, 2957, 2926, 2857, 1597, 1491, 1466, 1445, 1379, 1076, 1030 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 0.92 (3H, t, *J* = 7.2 Hz), 1.27–1.56 (4H, m), 2.05–2.24 (2H, m), 5.17 (1H, d, *J* = 11.1 Hz), 5.50 (1H, d, *J* = 17.1 Hz), 5.91 (1H, dt, *J* = 15.9, 7.2 Hz), 6.36 (1H, d, *J* = 15.6 Hz), 6.53 (1H, s) 6.54 (1H, dd, *J* = 17.1, 10.8 Hz), 7.16–7.42 (5H, m); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.2, 22.5, 31.6, 33.1, 115.8, 126.5, 126.9, 128.1, 128.2, 129.8, 135.8, 137.7, 138.1, 139.1; MS (EI^+): m/z (%) 212 (M^+, 100), 197 (37), 178 (84), 128 (59); HRMS (EI^+): Calcd for $C_{16}H_{20}$, M^+ 212.1565. Found m/z 212.1567.

3ba: IR (neat): 3020, 2923, 1603, 1491, 1445, 1369 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$): δ = 2.08 (3H, s), 2.17 (3H, d, *J* = 0.7Hz), 4.55 (1H, d, *J* = 1.9 Hz), 5.05-5.07 (1H, m), 6.75 (1H, s), 6.82–6.84 (2H, m), 7.02 (1H, dd, *J* = 7.4, 1.4Hz), 7.05-7.11(3H, m), 7.18–7.27 (3H, m); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 19.4, 21.0, 116.9, 126.3, 126.9, 127.2, 127.4, 128.2, 129.2, 129.8, 130.3, 136.5, 137.3, 139.4, 142.5, 144.3; MS (EI^+): m/z (%) 234 (M^+, 70), 219 (100), 204 (50), 115 (38); HRMS (EI^+): Calcd for $C_{18}H_{18}$, M^+ 234.1409. Found m/z 234.1405.

3ca: IR (neat): 3060, 2923, 1599, 1489, 1447, 1375 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 1.46 (3H, s), 1.83 (3H, s), 2.15 (3H, s), 5.96–6.00 (1H, m), 6.50 (1H, s), 6.82–6.86 (2H, m), 7.02–7.11 (4H, m), 7.15–7.26 (3H, m); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 18.8, 19.4, 27.9, 126.3, 126.4, 127.3, 128.0, 128.3, 128.5, 129.0, 129.8, 130.2, 135.3, 135.9, 137.5, 140.2, 140.4; MS (EI^+): m/z (%) 248 (M^+, 62), 233 (100), 218 (37), 115 (39); HRMS (EI^+): Calcd for $C_{19}H_{20}$, M^+ 248.1565. Found m/z 248.1564.

3da: IR (KBr): 3002, 2921, 1584, 1487, 1455, 1090 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ = 2.09 (3H, s), 4.70 (1H, d, *J* = 17.1 Hz), 5.14 (1H, d, *J* = 11.1 Hz), 6.58 (1H, s), 6.71 (1H, dd, *J* = 17.1, 10.2 Hz), 6.73–6.82 (2H, m), 7.00–7.10 (3H, m), 7.19–7.35 (3H, m); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 19.3, 116.7, 126.6, 127.9, 128.5, 129.5, 130.2, 130.3, 130.6, 132.8, 135.5, 136.2, 137.1, 140.9, 141.9; MS (EI^+): m/z (%) 254 (M^+, 63), 219 (100), 204 (72), 105 (47); HRMS (EI^+): Calcd for $C_{17}H_{15}Cl$, M^+ 254.0862. Found m/z 254.0860.

3ea: IR (KBr): 3007, 2965, 1595, 1509, 1460, 1254, 1179, 1028 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ = 2.11 (3H, s), 3.72 (3H, s), 4.62 (1H, d, *J* = 17.7 Hz), 5.05 (1H, d, *J* = 10.2 Hz), 6.58 (1H, s), 6.60–6.84 (5H, m), 7.06 (1H, d, *J* = 6.6 Hz), 7.18–7.30 (3H, m); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 19.1, 55.1, 113.6, 114.7, 126.3, 127.4, 129.5, 129.6, 130.1, 130.2, 131.0, 136.3, 137.5, 139.0, 141.2, 158.6; MS (EI^+): m/z (%) 250 (M^+, 100), 235 (28), 219 (36), 134 (37); HRMS (EI^+): Calcd for $C_{18}H_{18}O$, M^+ 250.1358. Found m/z 250.1356.

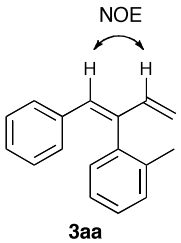
5aa: IR (neat): 2924, 2850, 1631, 1592, 1487, 1448, 986, 909 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ = 1.03–1.28 (3H, m), 1.28–1.42 (2H, m), 1.64–1.72 (1H, m), 1.72–1.80 (4H, m), 2.17 (3H, s), 2.55–2.65 (1H, m), 4.64 (1H, d, *J* = 17.5 Hz), 5.09 (1H, d, *J* = 10.6 Hz), 5.24 (1H, d, *J* = 9.4 Hz), 6.91 (1H, dd, *J* = 17.2, 10.6 Hz), 7.05 (1H, d, *J* = 7.1 Hz), 7.12–7.23 (3H, m); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 19.5, 25.9, 26.0, 33.3, 36.6, 116.2, 125.3, 126.8, 129.6, 130.0, 133.0, 136.3, 137.6, 139.1, 141.1; MS (EI^+): m/z (%) 226 (M^+, 77), 211 (32), 129 (100), 115 (35); HRMS (EI^+): Calcd for $C_{17}H_{22}$, M^+ 226.1722. Found m/z 226.1704.

5ba: IR (neat): 3015, 2957, 1593, 1487, 1456, 1379, 1044 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 0.95 (3H, t, *J* = 7.1 Hz), 1.35–1.51 (4H, m), 2.19 (3H, s), 2.31–2.38 (2H, m), 4.66 (1H, d, *J* = 17.2, Hz), 5.10 (1H, d, *J* = 10.6 Hz), 5.41 (1H, t, *J* = 7.7 Hz), 6.91 (1H, d, *J* = 17.2, 10.6 Hz), 7.06 (1H, d, *J* = 7.1 Hz), 7.13–7.24 (3H, m); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.2, 19.8, 22.6, 27.5, 32.1, 116.4, 125.5, 127.0, 129.8, 130.2, 133.0, 133.5, 136.5, 139.6, 141.4; MS (EI^+): m/z (%) 200 (M^+, 36), 157 (38), 143 (47), 129 (100); HRMS (EI^+): Calcd for $C_{15}H_{20}$, M^+ 200.1565. Found m/z 200.1572.

Stereochemistries of the products were determined by NOE or NOESY experiments. Curved arrows shown below indicate the observed NOE or NOESY.

[Compound 3aa]

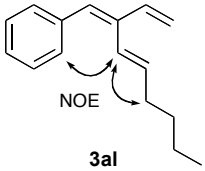
The following results of **3aa** (major product) suggested that the stereochemistry of the double bond was (Z)-configuration.



3aa

[Compound 3al]

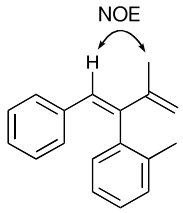
The following results of **3al** (major product) suggested that the stereochemistry of the double bond was (*Z*)-configuration.



3al

[Compound 3ba]

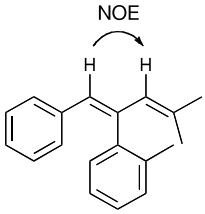
The following results of **3ba** (major product) suggested that the stereochemistry of the double bond was (*Z*)-configuration.



3ba

[Compound 3ca]

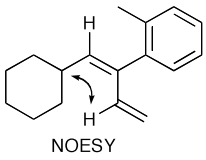
The following results of **3ca** (major product) suggested that the stereochemistry of the double bond was (*Z*)-configuration.



3ca

[Compound 5aa]

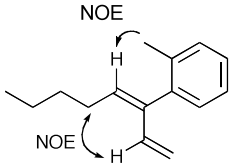
The following results of **5aa** (major product) suggested that the stereochemistry of the double bond was (*E*)-configuration.



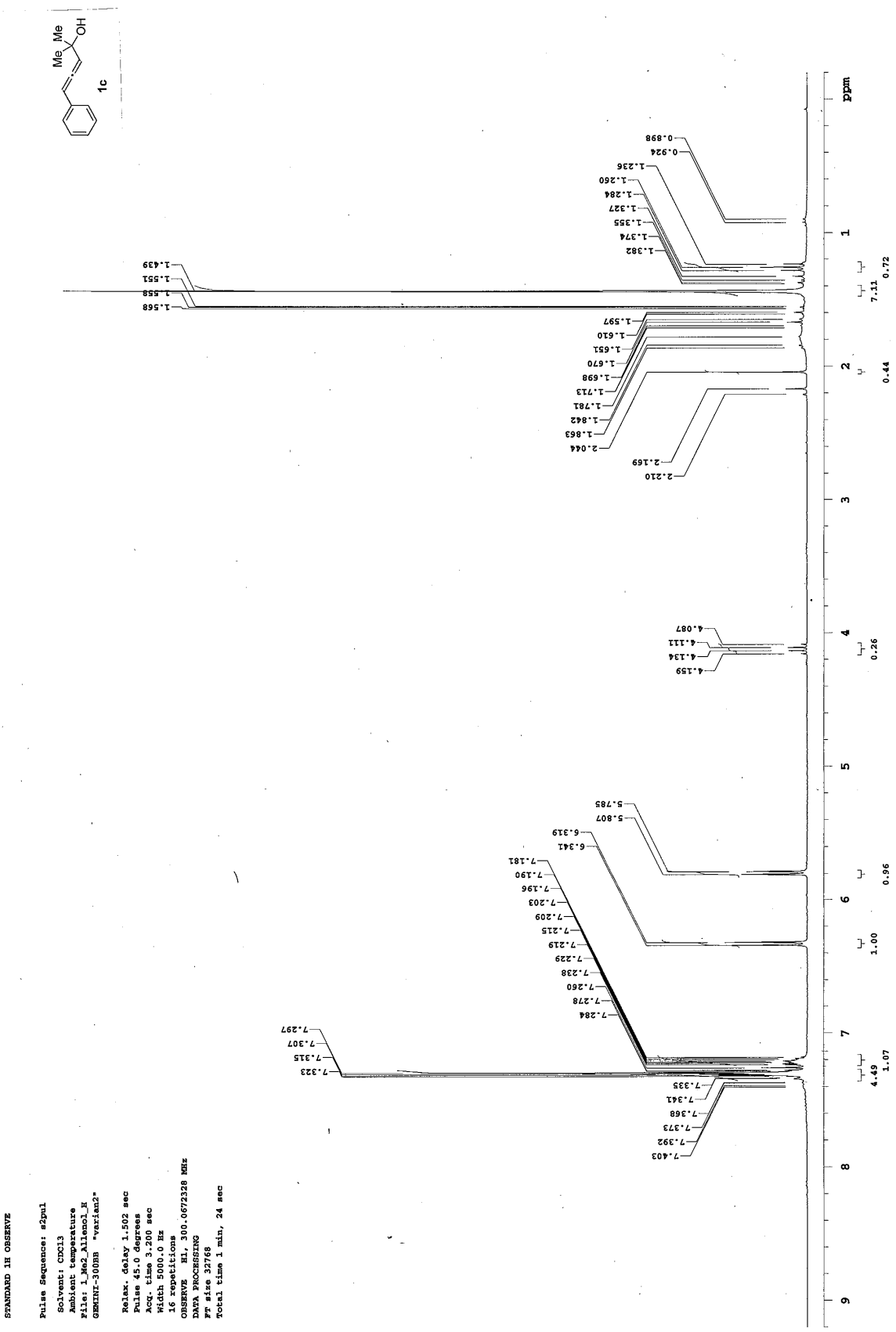
5aa

[Compound 5ba]

The following results of **5ba** (major product) suggested that the stereochemistry of the double bond was (*E*)-configuration.



5ba

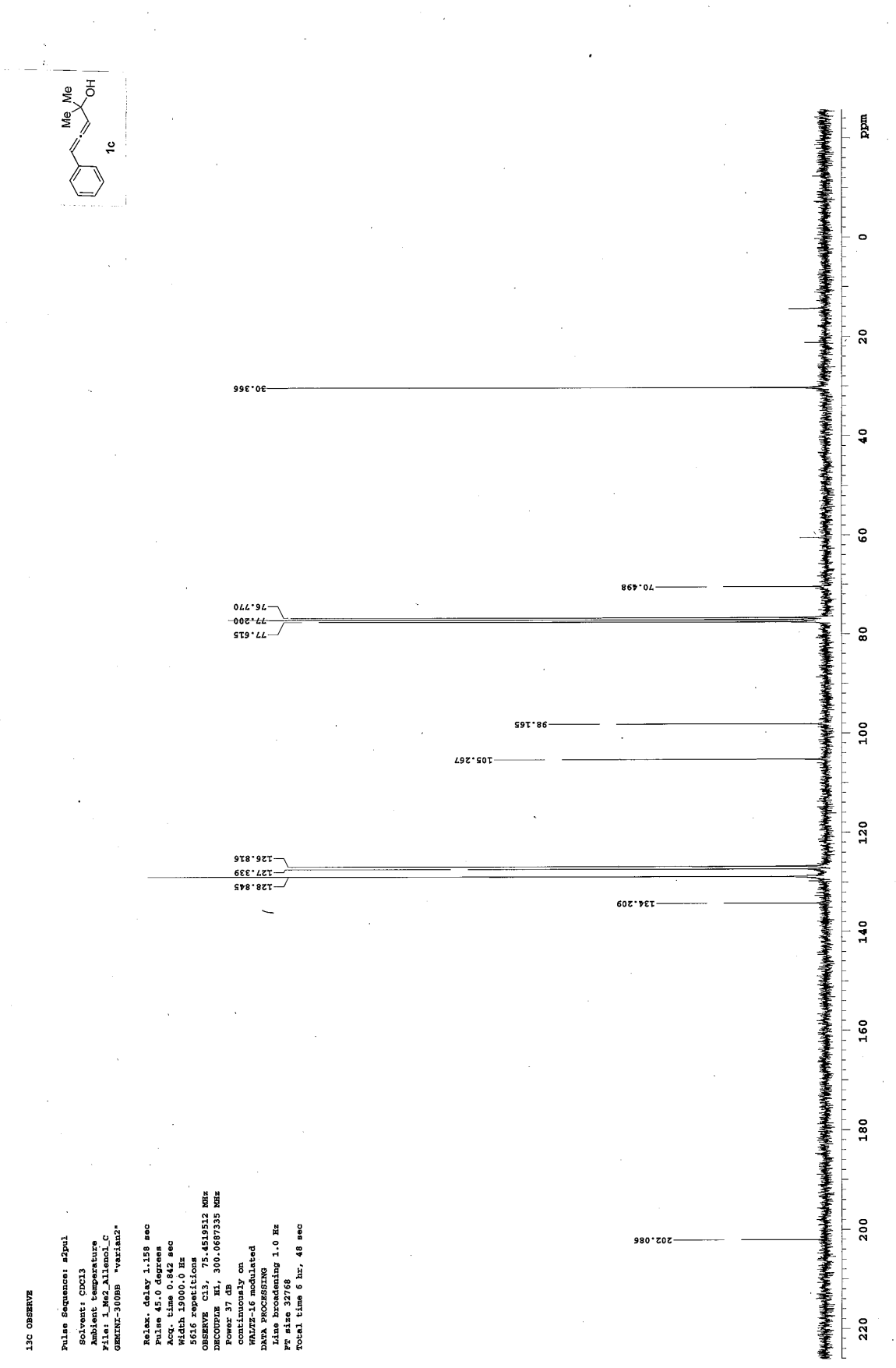


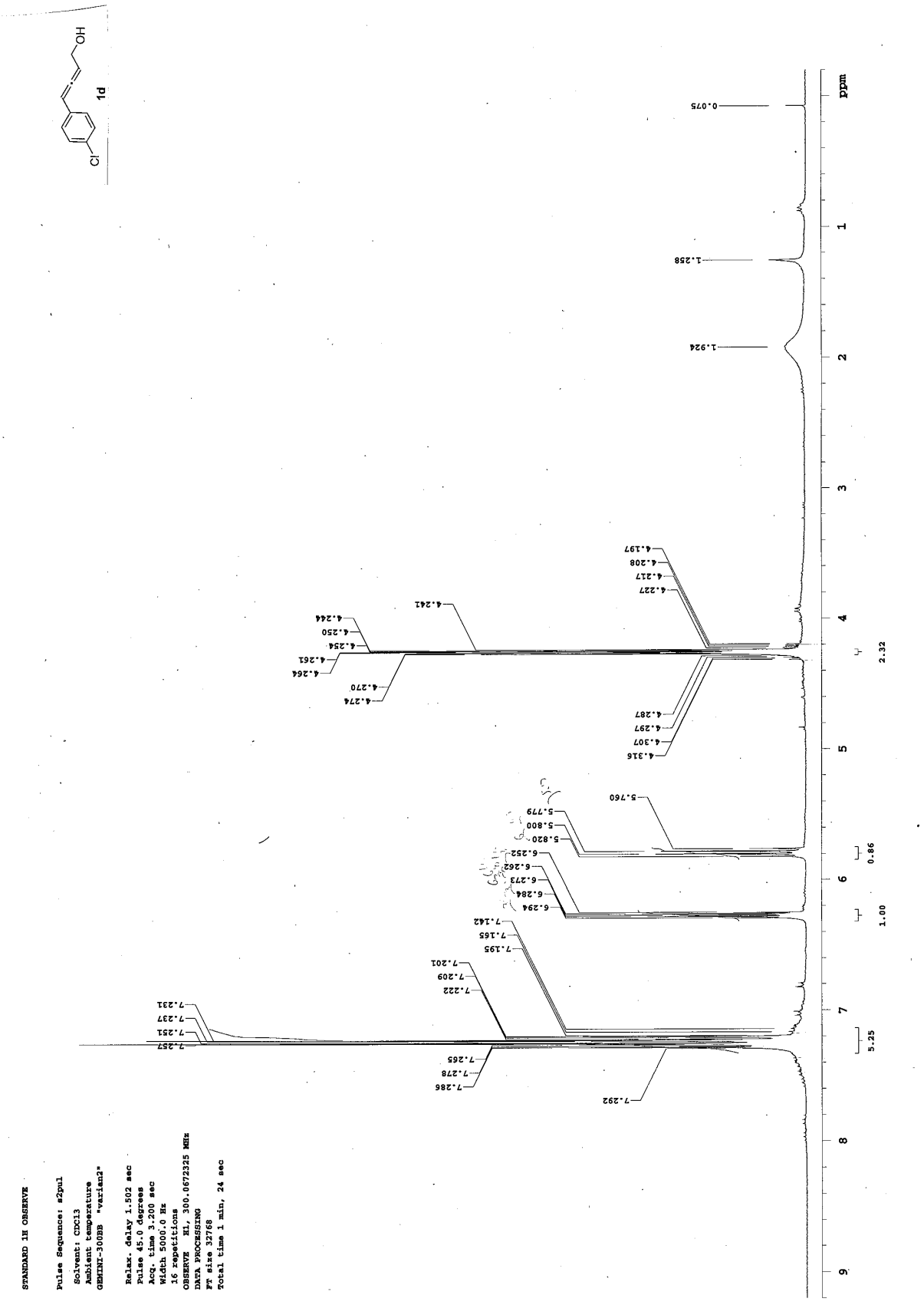
STANDARD 1H OBSERVE

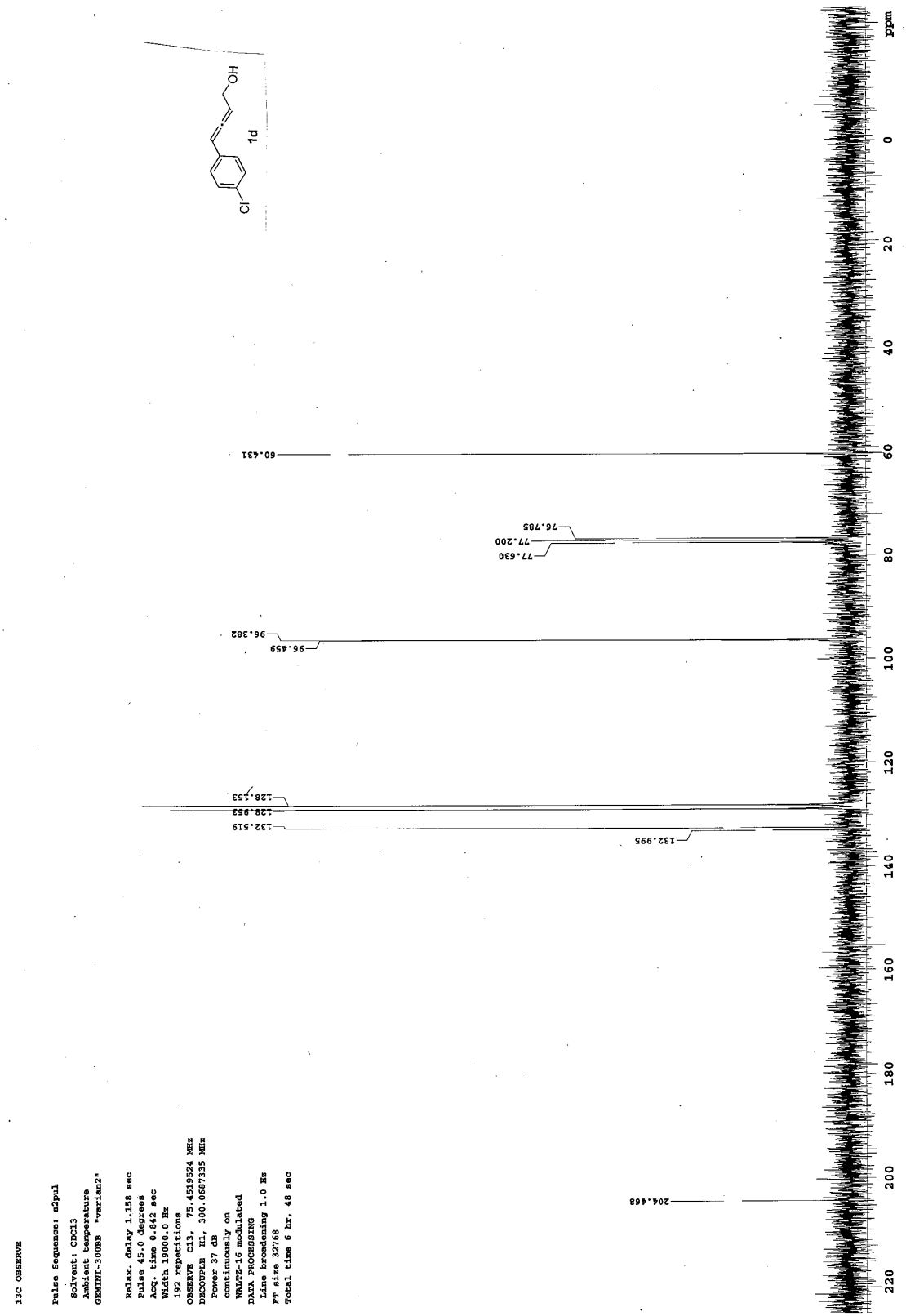
Pulse Sequence: s2pul

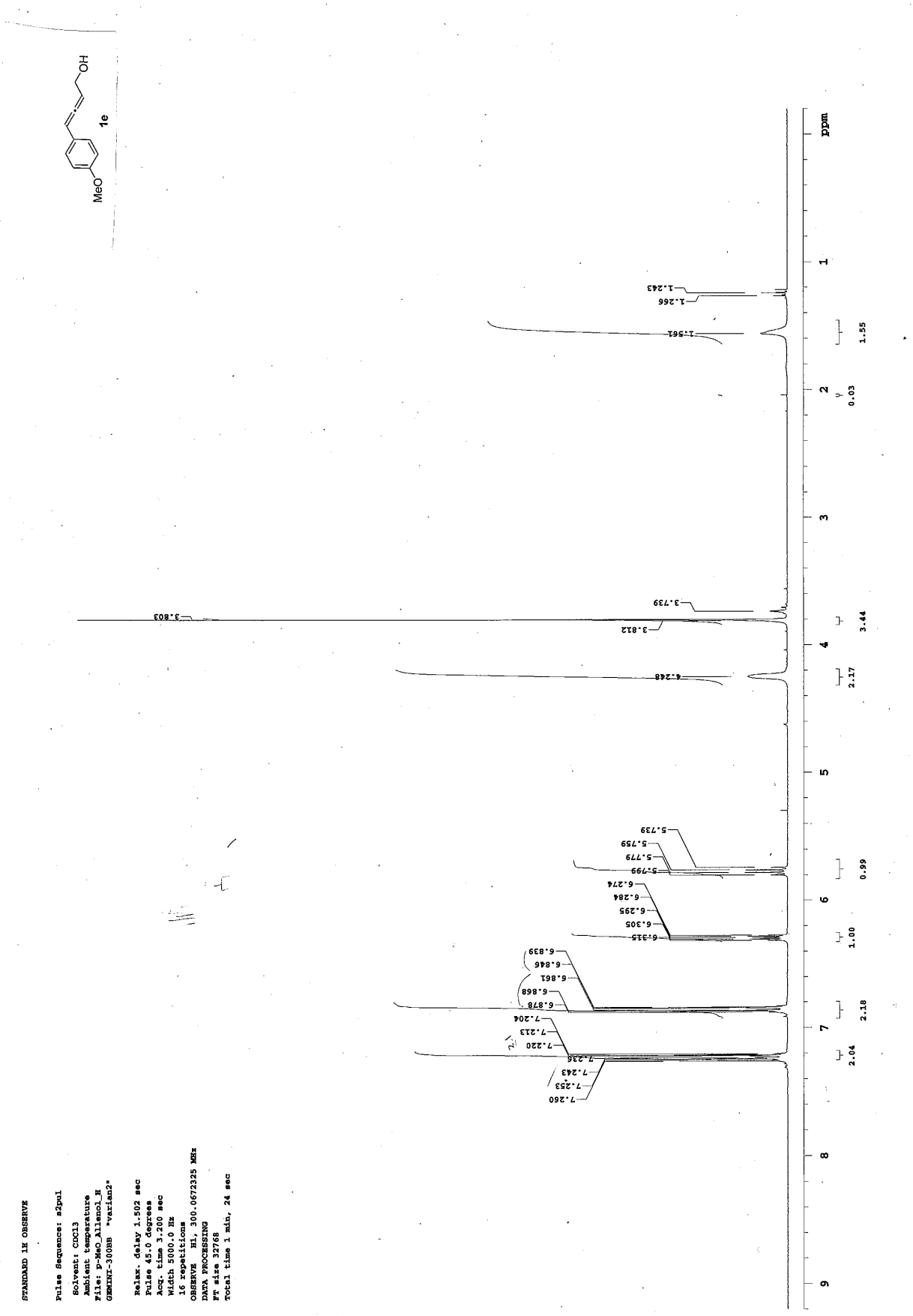
Solvent: CDCl3
Ambient temperature
File: 1_Me2_Allenol_H
GEMINI-300BB "varian2"

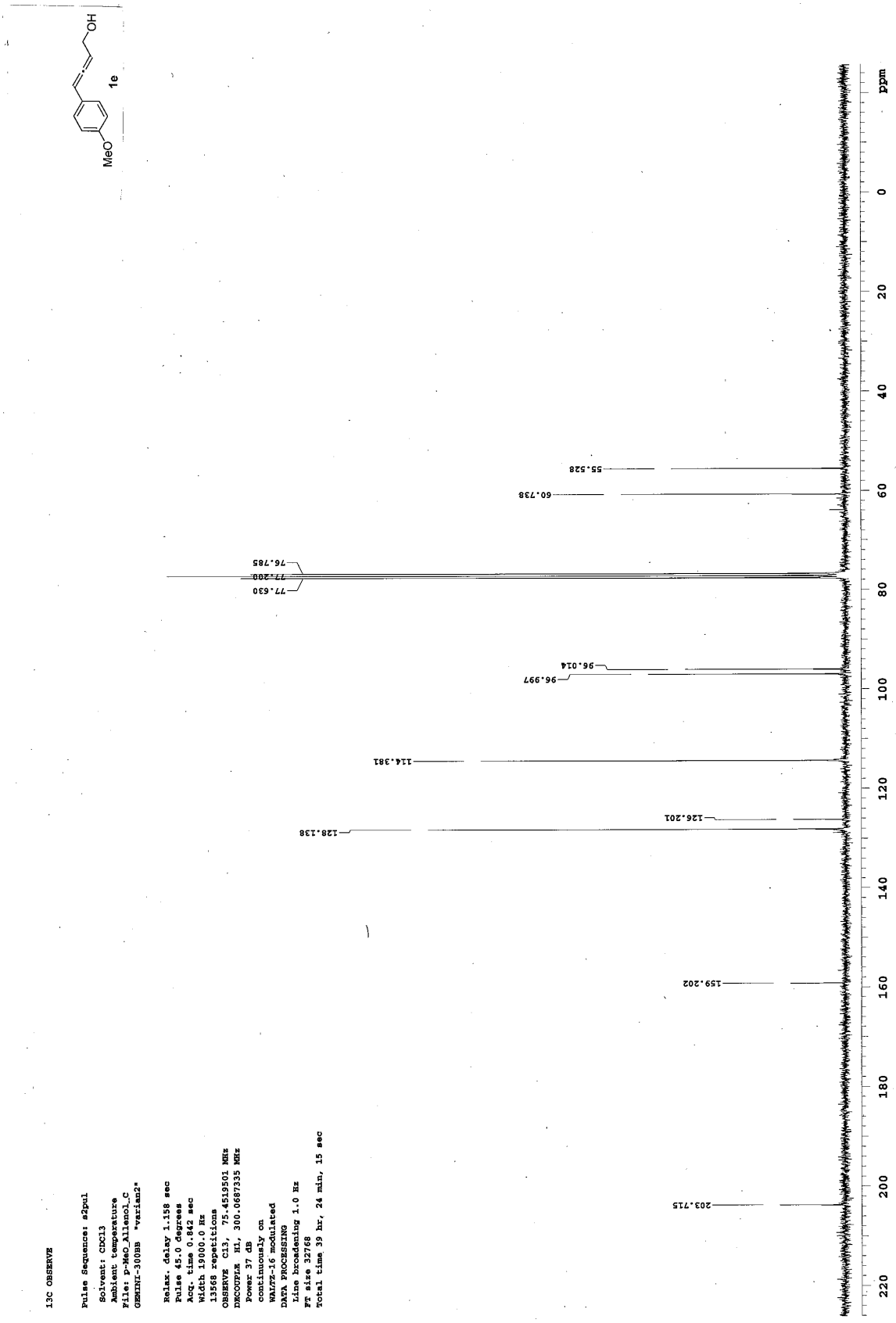
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672328 MHz
DATA PROCESSING
Ft size 32768
Total time 1 min, 24 sec

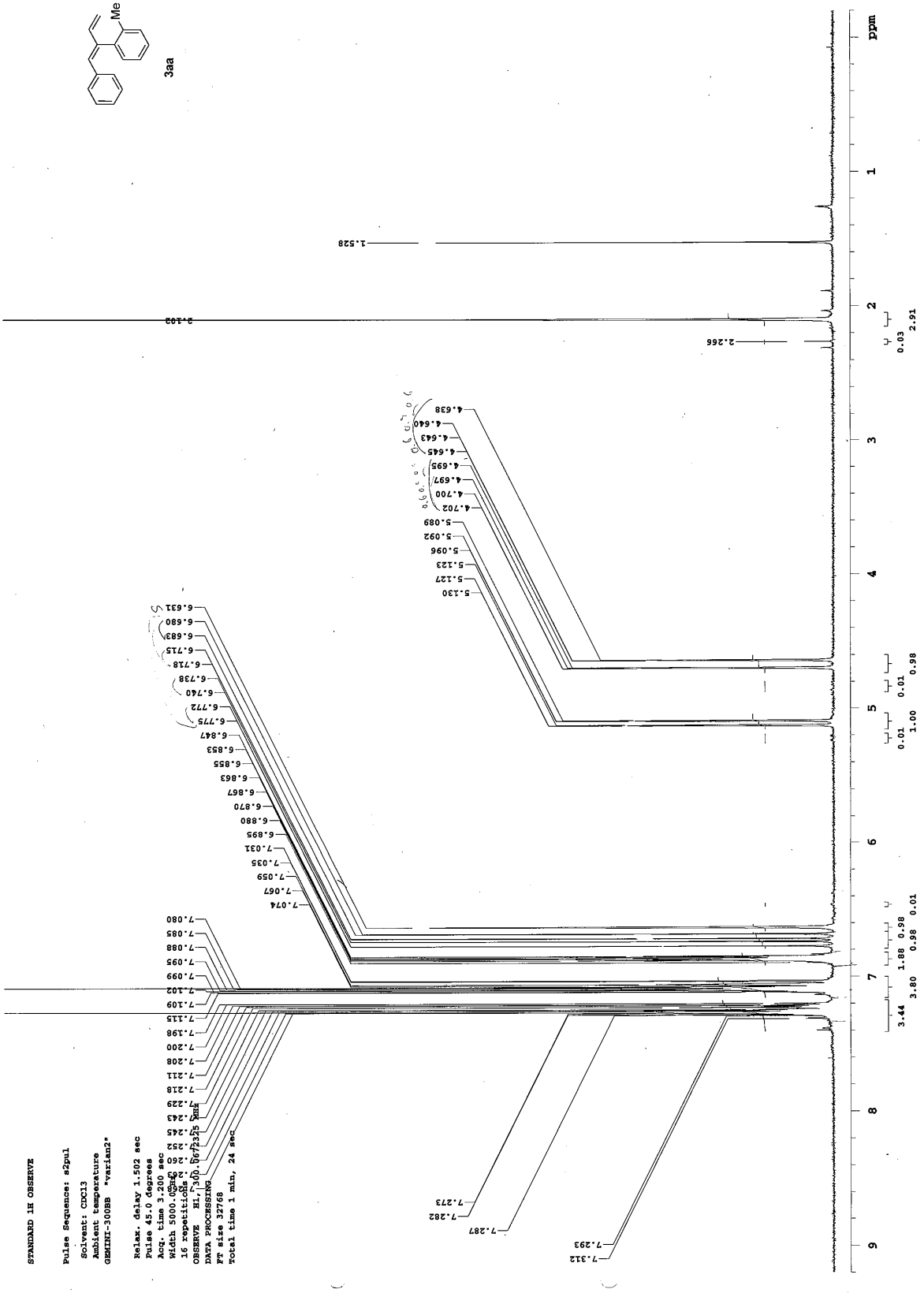

Me
Me
OH
1c
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 1_Me2_1ineol_C
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
5616 repetitions
OBSERVE C13, 75.4519512 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
line broadening 1.0 Hz
FT size 32768
Total time 6 hr, 48 sec
30.366
70.498
76.770
77.200
77.615
98.165
105.267
126.816
127.339
128.845
134.209
202.086
ppm
0
20
40
60
80
100
120
140
160
180
200
220

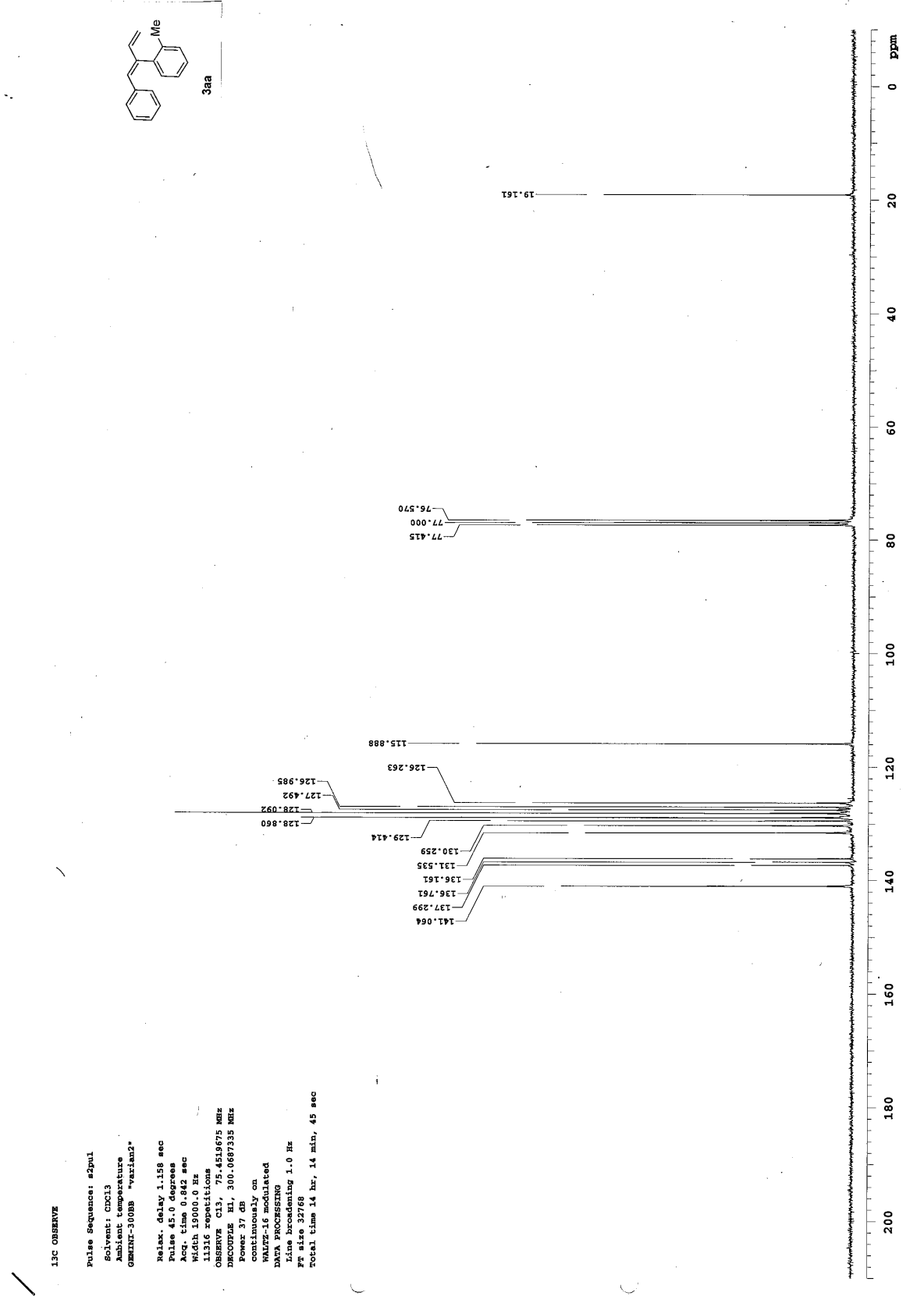

1d
OH
Cl
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672325 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec
ppm
0.075
1.258
1.924
4.197
4.208
4.217
4.227
4.241
4.244
4.250
4.254
4.261
4.264
4.270
4.274
4.287
4.297
4.307
4.316
5.760
5.779
5.800
5.820
6.252
6.262
6.273
6.284
6.294
7.142
7.165
7.195
7.201
7.209
7.222
7.231
7.237
7.251
7.257
7.265
7.278
7.286
7.292
2.32
0.86
1.00
5.25

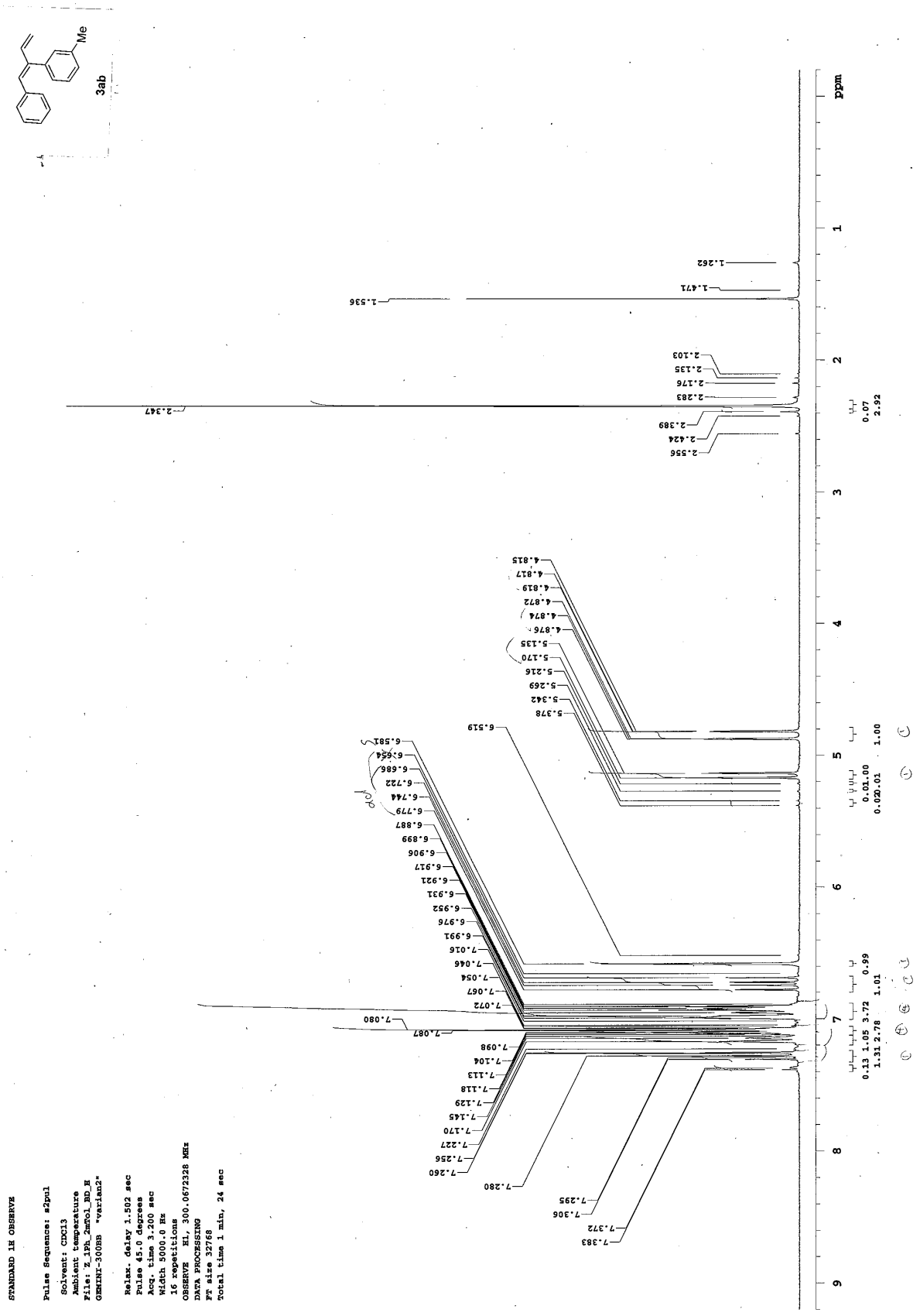

1d
OH
Cl
60.431
76.785
77.200
77.630
96.382
96.459
128.153
128.953
132.519
132.995
204.468
ppm
0
20
40
60
80
100
120
140
160
180
200
220
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
192 repetitions
OBSERVE C13, 75.4519524 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 6 hr, 48 sec

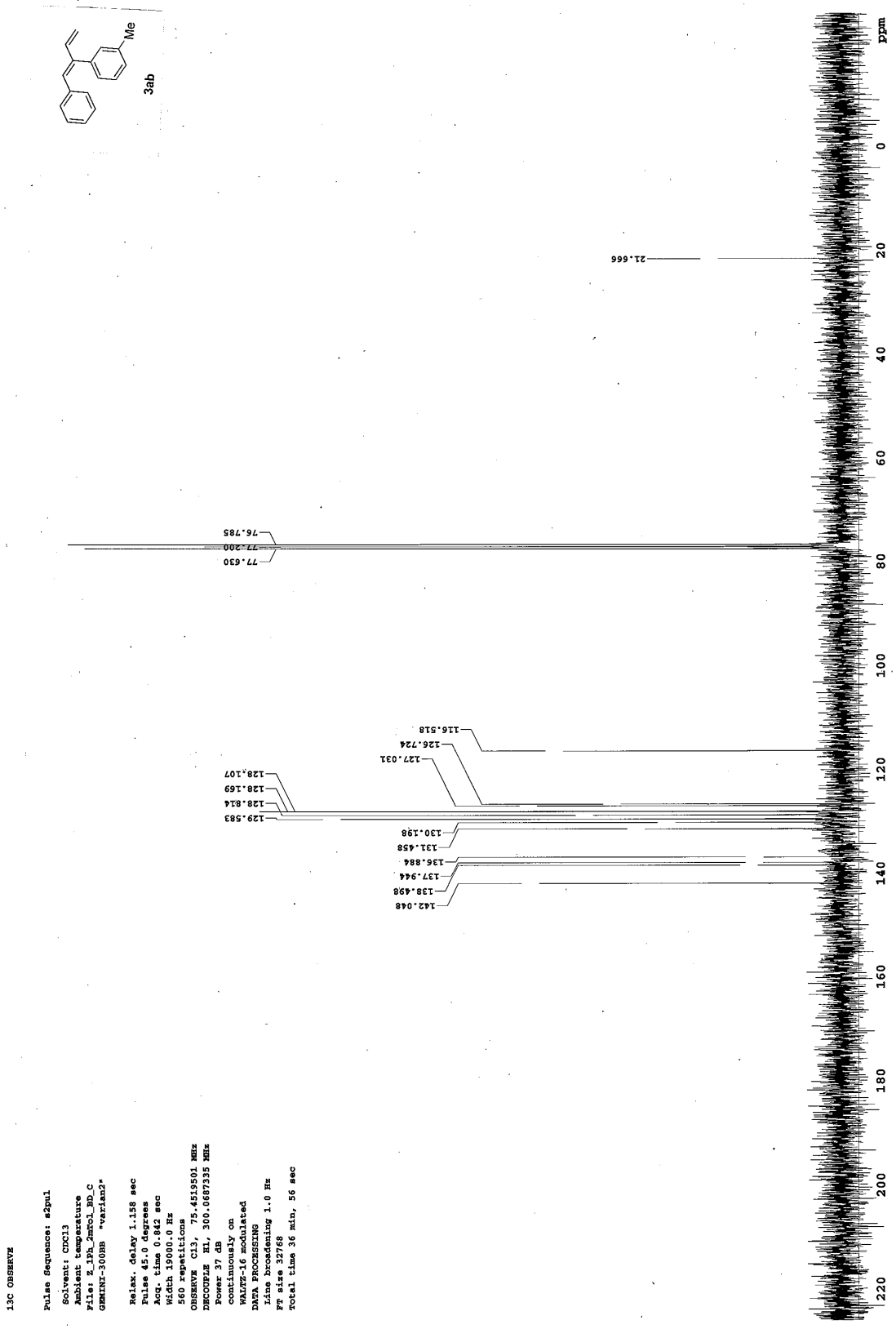

OH
1e
MeO
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: p-MeO_Allenol_H
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672325 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec
1.243
1.266
1.561
3.739
3.803
3.812
4.248
5.739
5.759
5.779
5.799
6.274
6.284
6.295
6.305
6.315
6.839
6.846
6.861
6.868
6.878
7.204
7.213
7.220
7.236
7.243
7.253
7.260
ppm
1.55
0.03
3.44
2.17
0.99
1.00
2.18
2.04

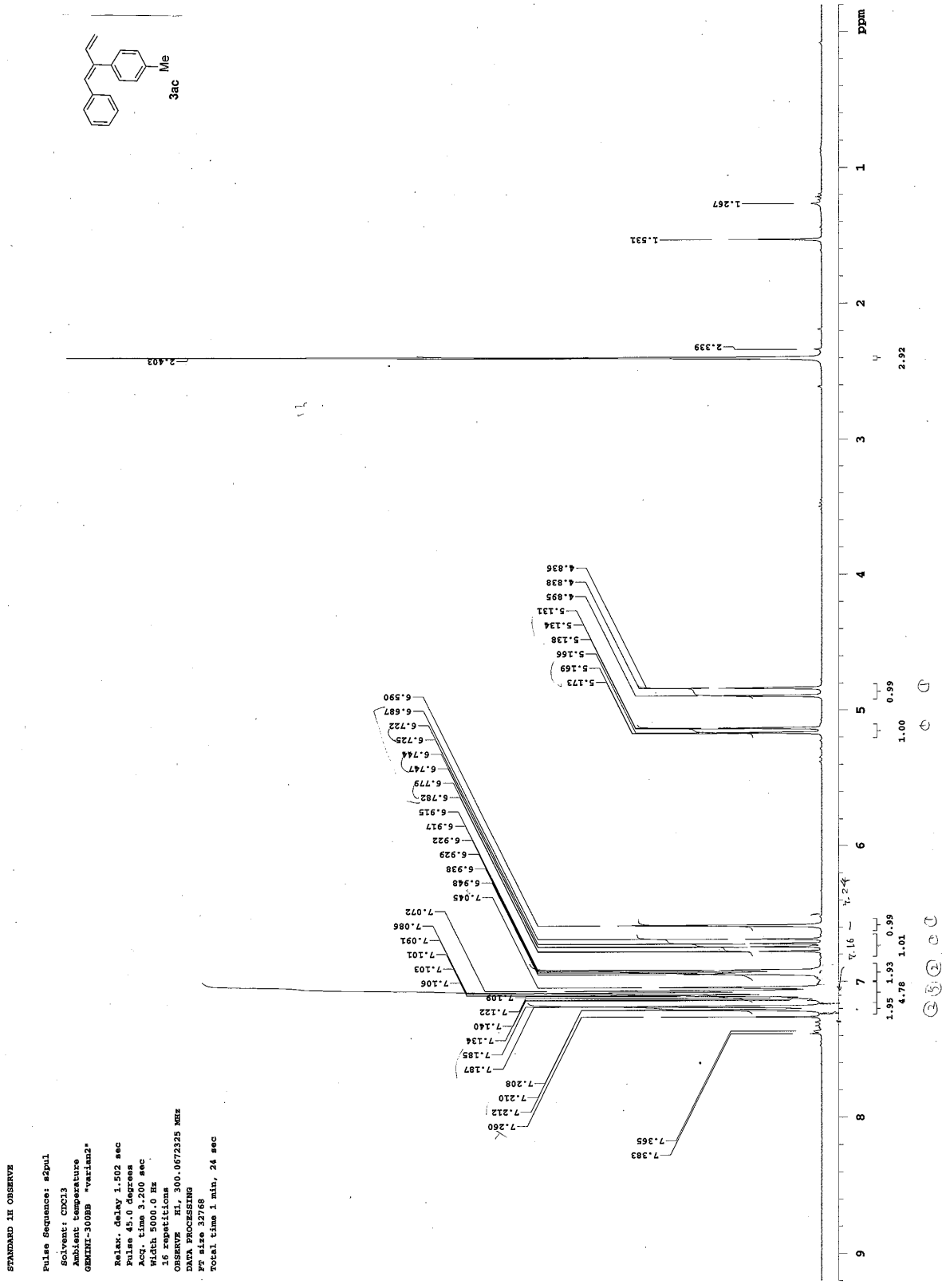

OH
MeO
1e
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: p-MeO_Allenol_C
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
13568 repetitions
OBSERVE C13, 75.4519501 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 39 hr, 24 min, 15 sec
55.528
60.738
76.785
77.200
77.630
96.014
96.997
114.381
126.201
128.138
159.202
203.715
ppm

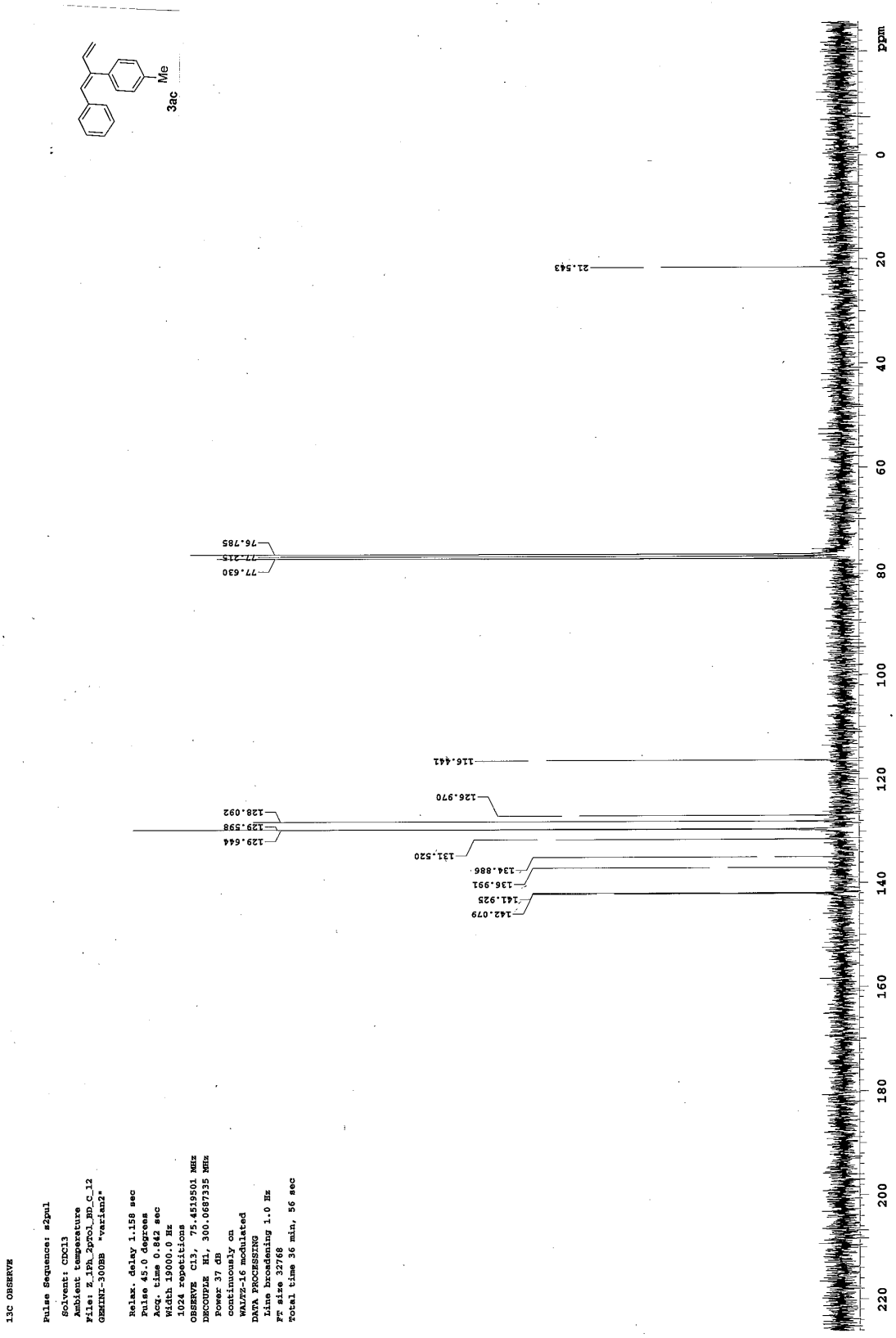

Me
3aa
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672325 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec
1.528
2.102
2.266
4.638
4.640
4.643
4.645
4.695
4.697
4.700
4.702
5.089
5.092
5.096
5.123
5.127
5.130
6.631
6.680
6.683
6.715
6.738
6.740
6.772
6.775
6.847
6.853
6.855
6.863
6.867
6.870
6.880
6.895
7.031
7.035
7.059
7.067
7.074
7.080
7.085
7.088
7.095
7.099
7.102
7.109
7.115
7.198
7.200
7.208
7.211
7.218
7.229
7.243
7.245
7.252
7.260
7.273
7.282
7.287
7.293
7.312
ppm
0.03
2.91
0.01
0.01
1.00
0.98
3.44
1.88
0.98
0.98
3.80
0.01

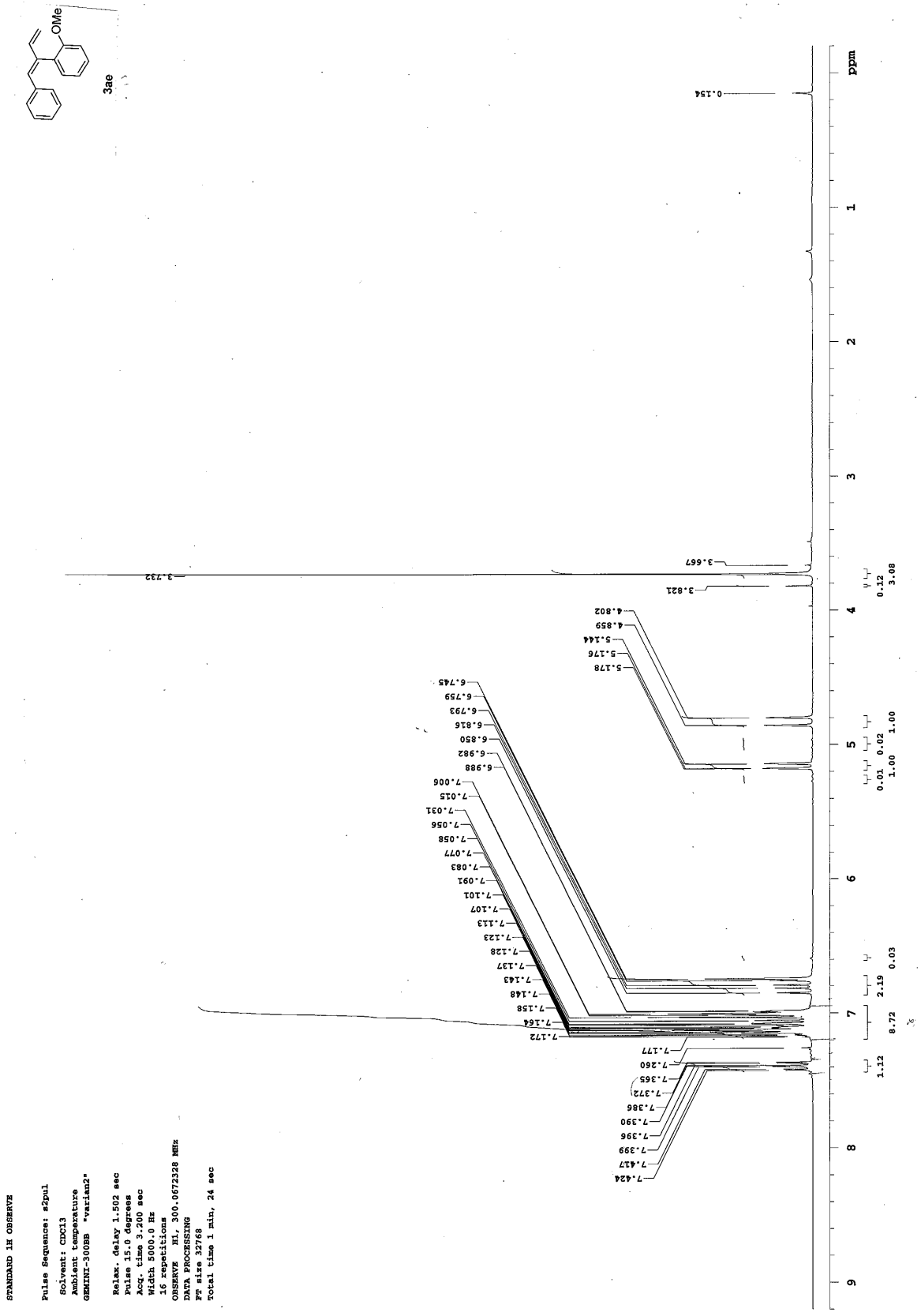

Me
3aa
19.161
76.570
77.000
77.415
115.888
126.263
126.985
127.492
128.092
128.860
129.414
130.259
131.535
136.161
136.761
137.299
141.064
ppm

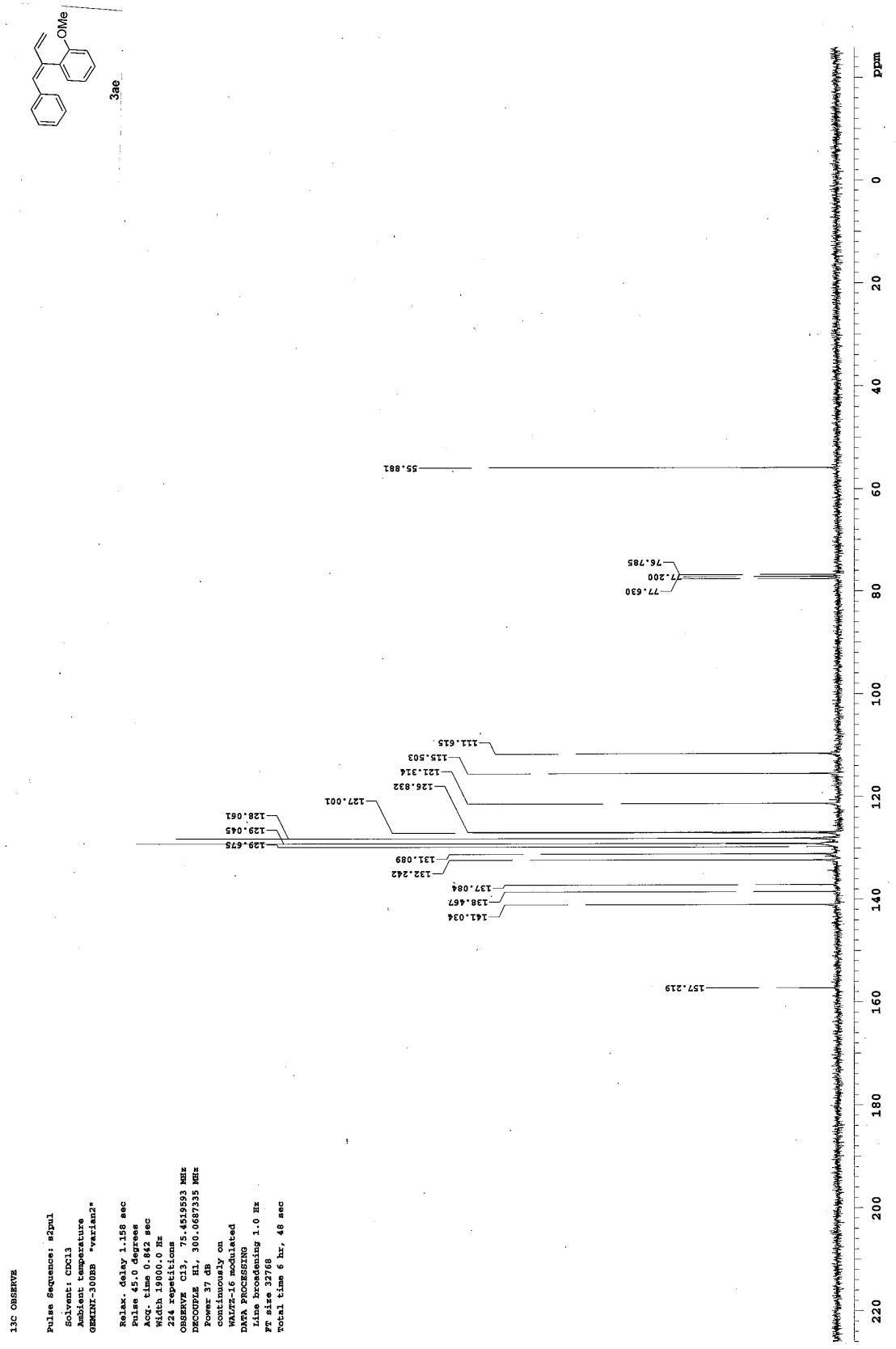

3ab
Me
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: Z_1Ph_2mTol_BD_E
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672328 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec
ppm

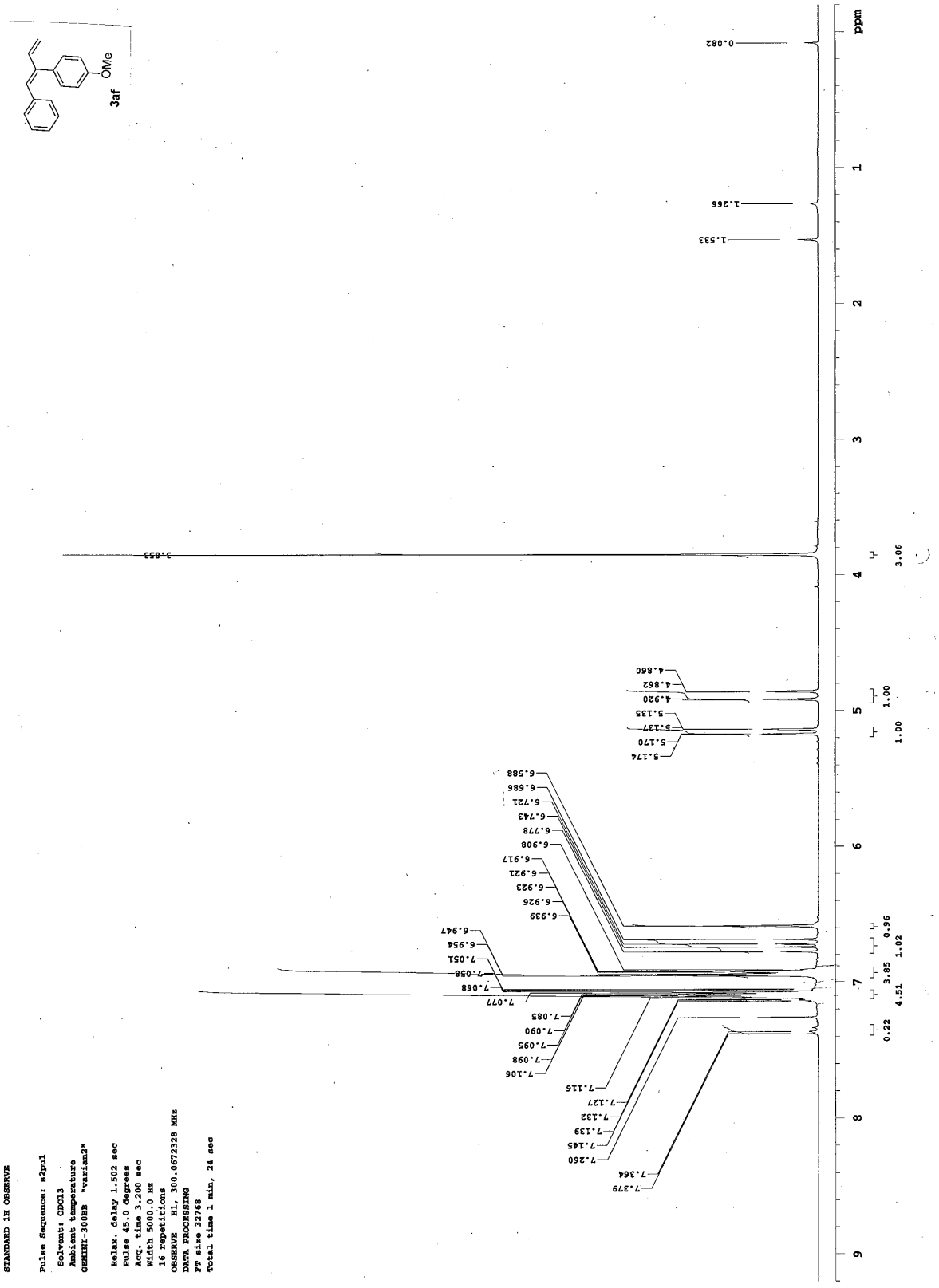

Me
3ab
21.666
76.785
77.200
77.630
116.518
126.724
127.031
128.107
128.169
128.814
129.583
130.198
131.458
136.884
137.944
138.498
142.048
ppm
0
20
40
60
80
100
120
140
160
180
200
220
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 2_1Ph_2mTol_3D_C
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
560 repetitions
OBSERVE C13, 75.4519501 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 36 min, 56 sec

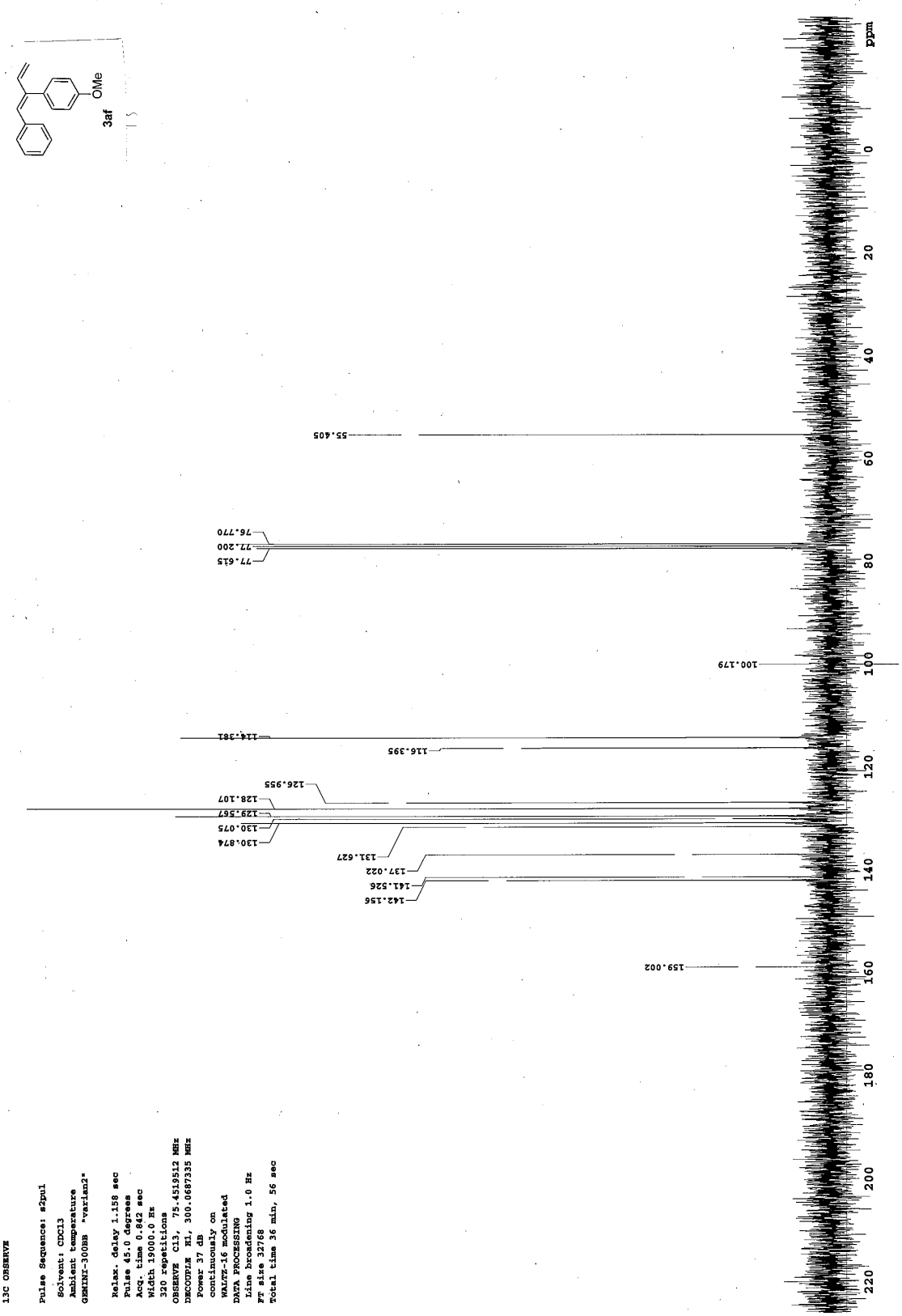

Me
3ac
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672325 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec
ppm

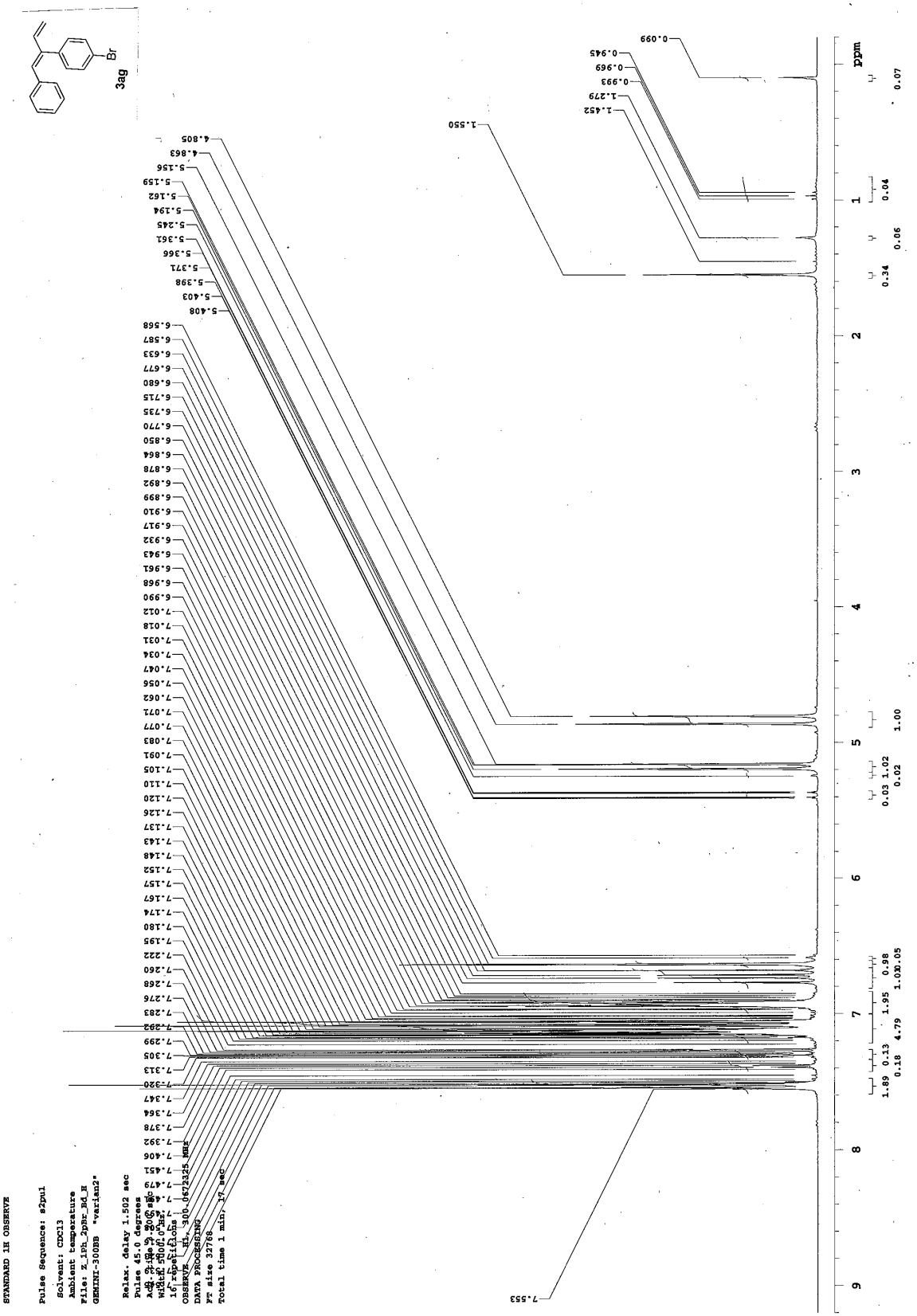

Me
3ac
21.543
76.785
77.215
77.630
116.441
126.970
128.092
129.598
129.644
131.520
134.886
136.991
141.925
142.079
ppm
0
20
40
60
80
100
120
140
160
180
200
220
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: Z_1Ph_2pTol_BD_C_12
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
1024 repetitions
OBSERVE C13, 75.4519501 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 36 min, 56 sec

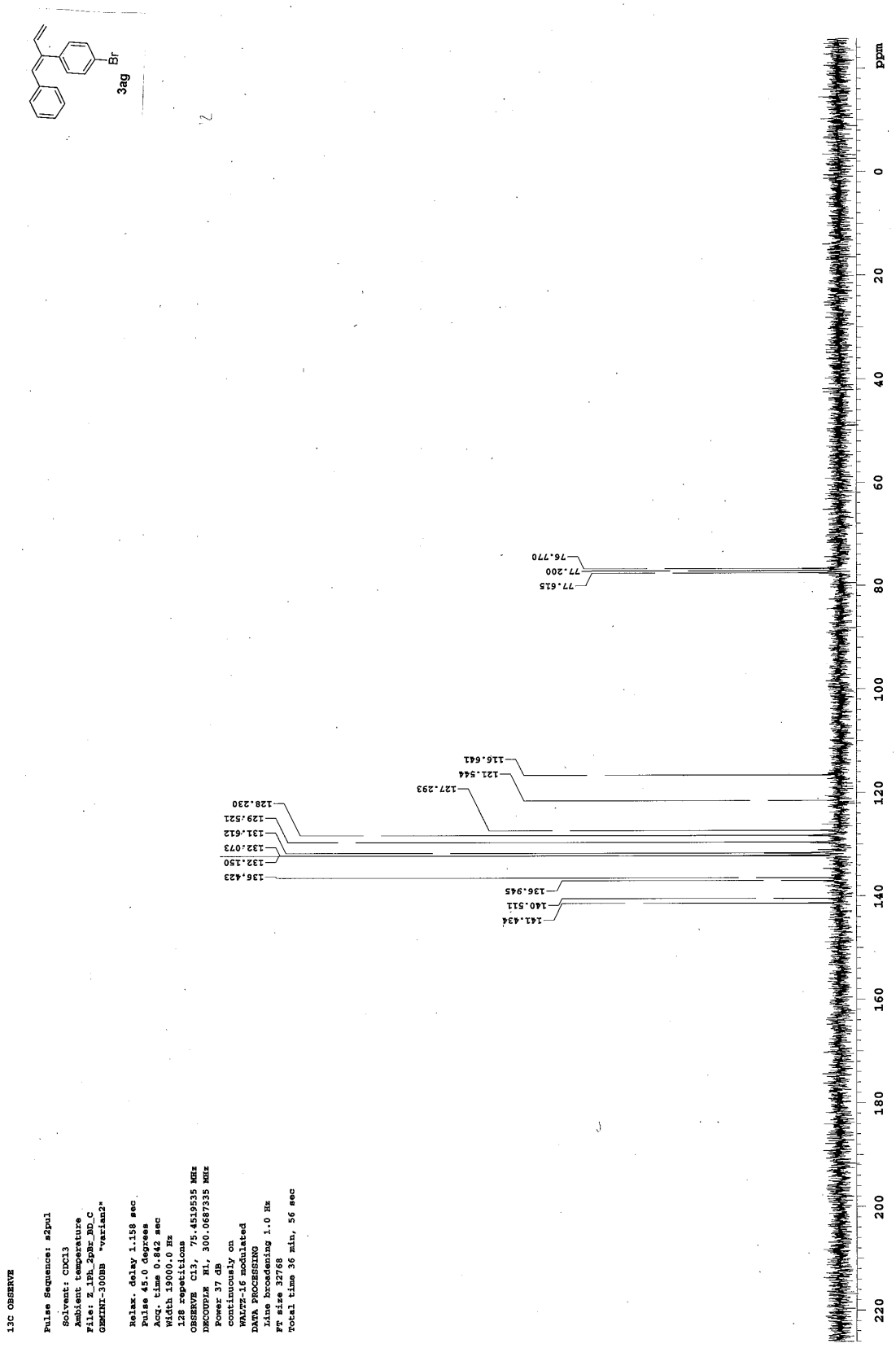

OMe
3ae
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 15.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672328 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec
ppm

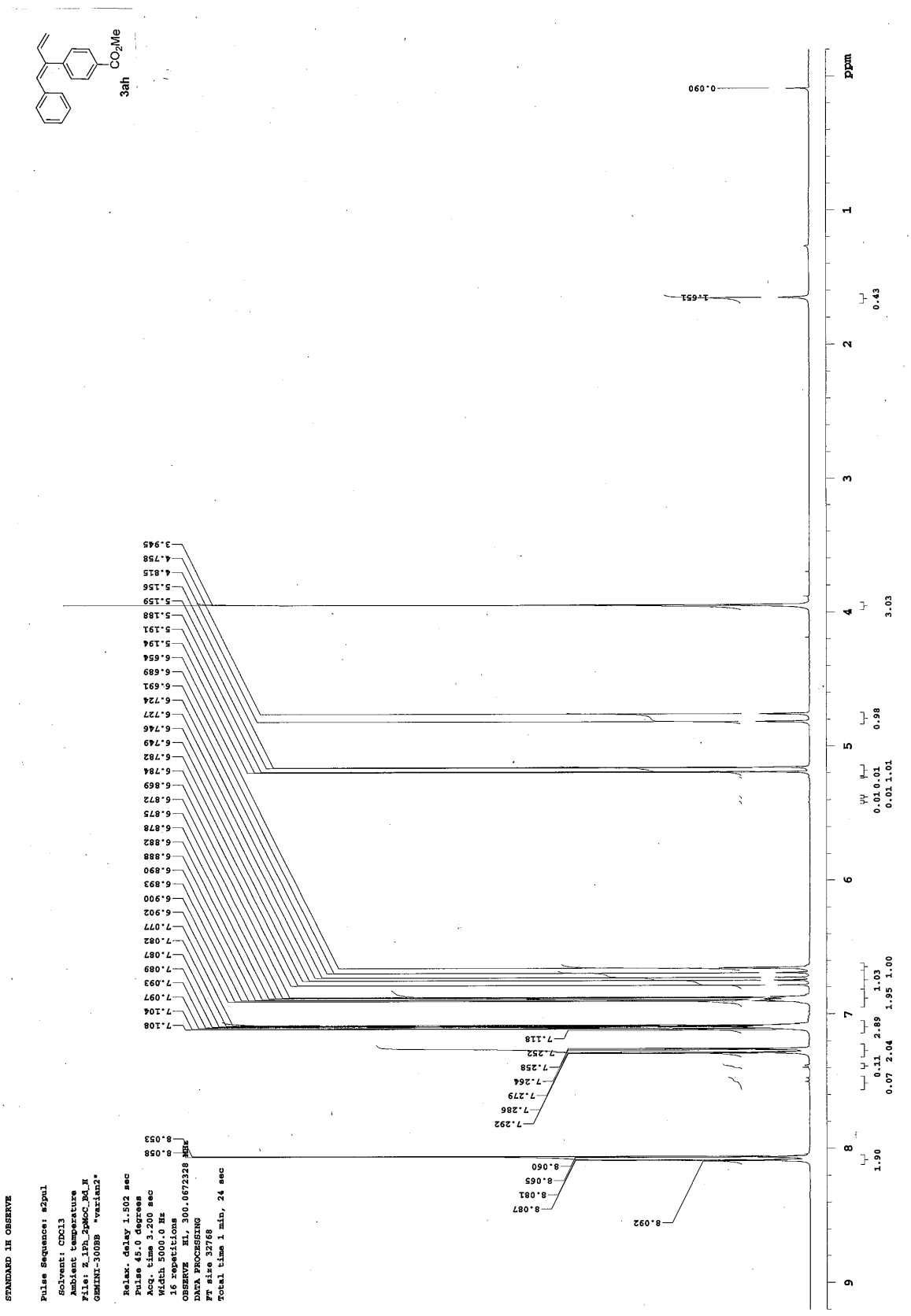

OMe
3ae
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
224 repetitions
OBSERVE C13, 75.4519593 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 6 hr, 48 sec
55.881
76.785
77.200
77.630
111.615
115.503
121.314
126.832
127.001
128.061
129.045
129.675
131.089
132.242
137.084
138.467
141.034
157.219
ppm

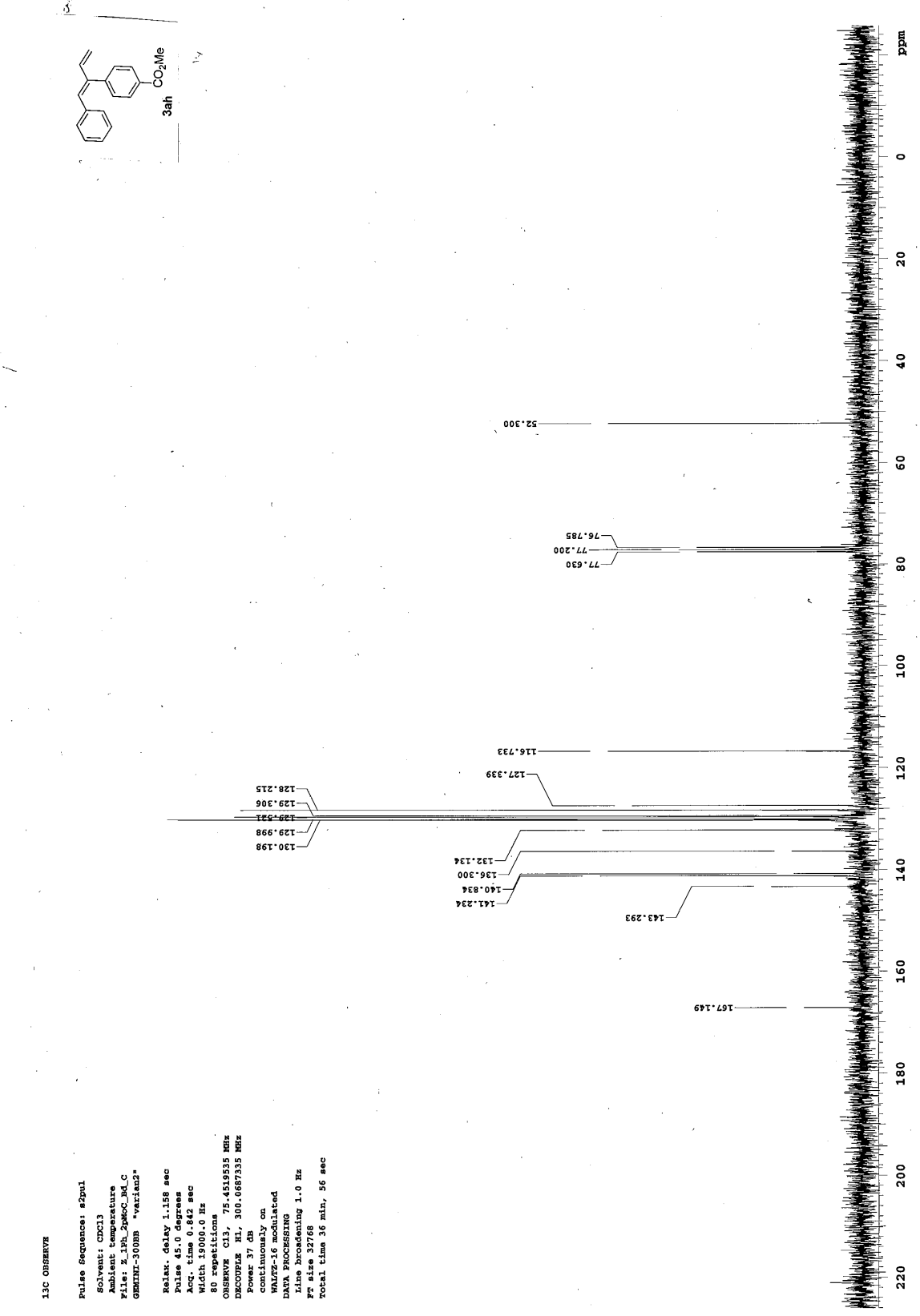

3af
OMe
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672328 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec
ppm


OMe
3af
ppm
0
20
40
60
80
100
120
140
160
180
200
220
55.405
76.770
77.200
77.615
100.179
114.381
116.395
126.955
128.107
129.567
130.075
130.874
131.627
137.022
141.526
142.156
159.002
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
320 repetitions
OBSERVE C13, 75.4519512 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 36 min, 56 sec


3ag
Br
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 2_1Fn_2pbr_Bd_E
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 9.006 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672325 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 17 sec
ppm


3ag
Br
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 2_1Ph_2pBr_BD_C
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
128 repetitions
OBSERVE C13, 75.45195535 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 36 min, 56 sec
141.434
140.511
136.945
136.423
132.150
132.073
131.612
129.521
128.230
127.293
121.544
116.641
77.615
77.200
76.770
ppm
220
200
180
160
140
120
100
80
60
40
20
0

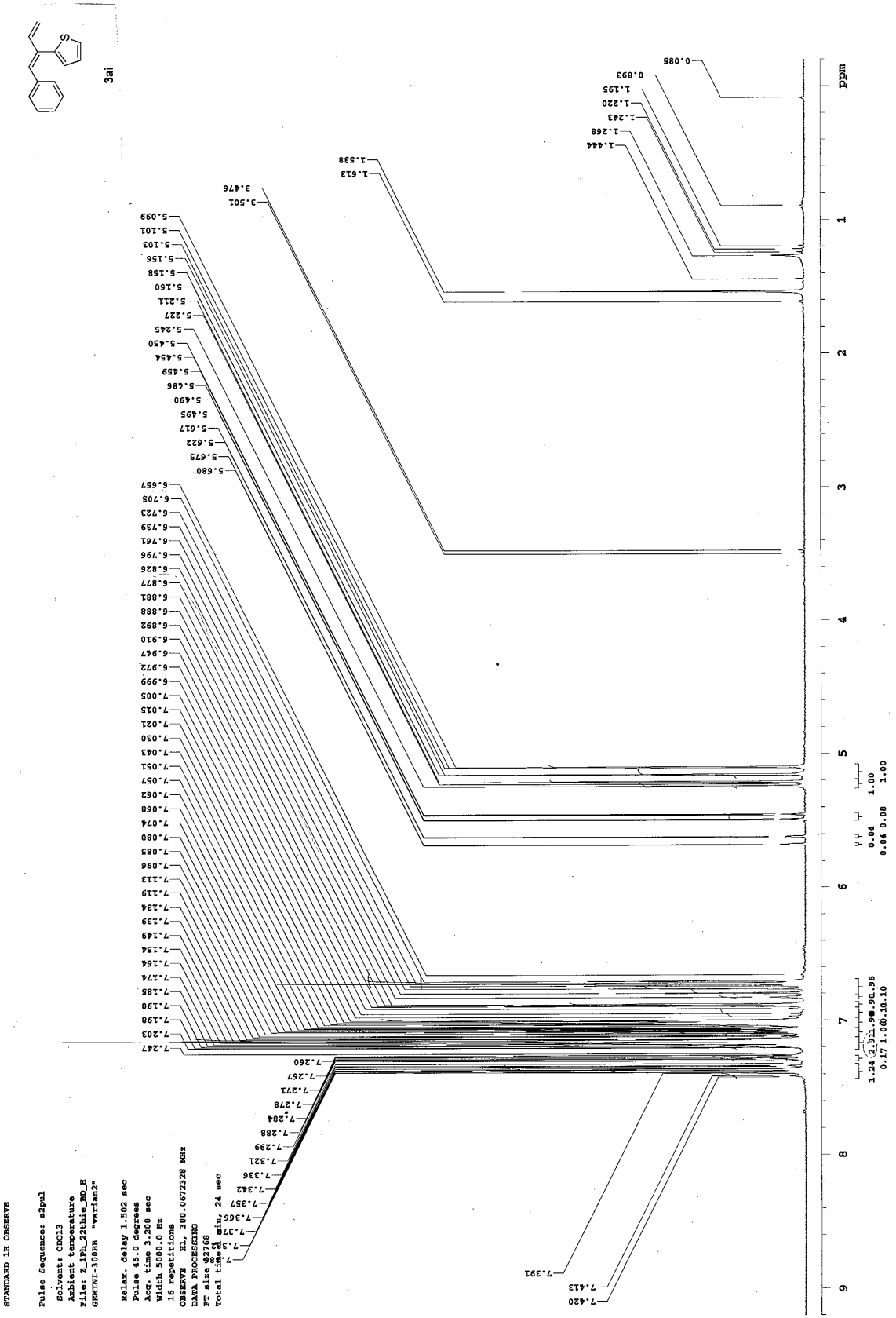

3ah
CO2Me
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 2_1Ph_2pMOC_BG_R
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672328 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec
ppm

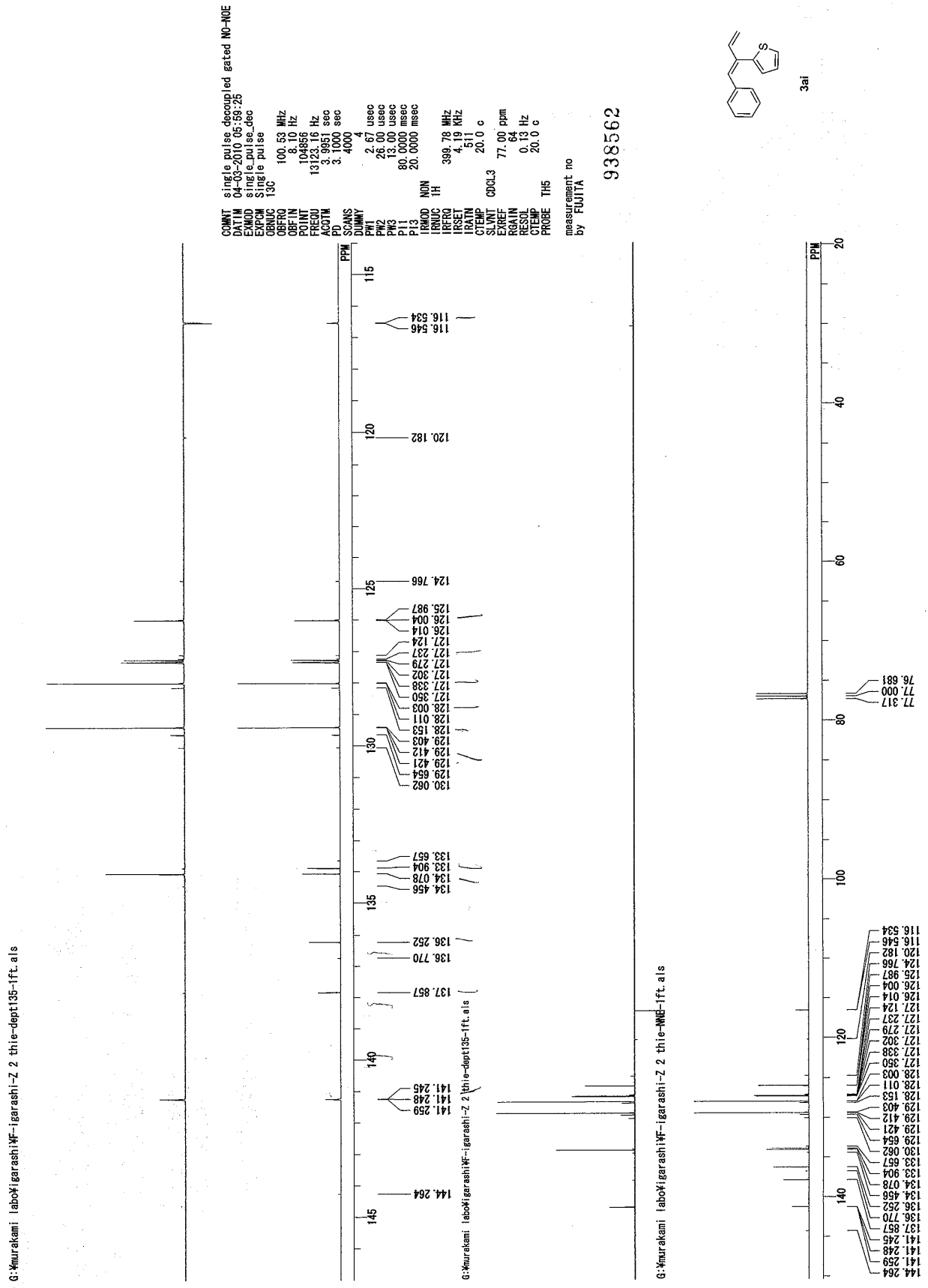


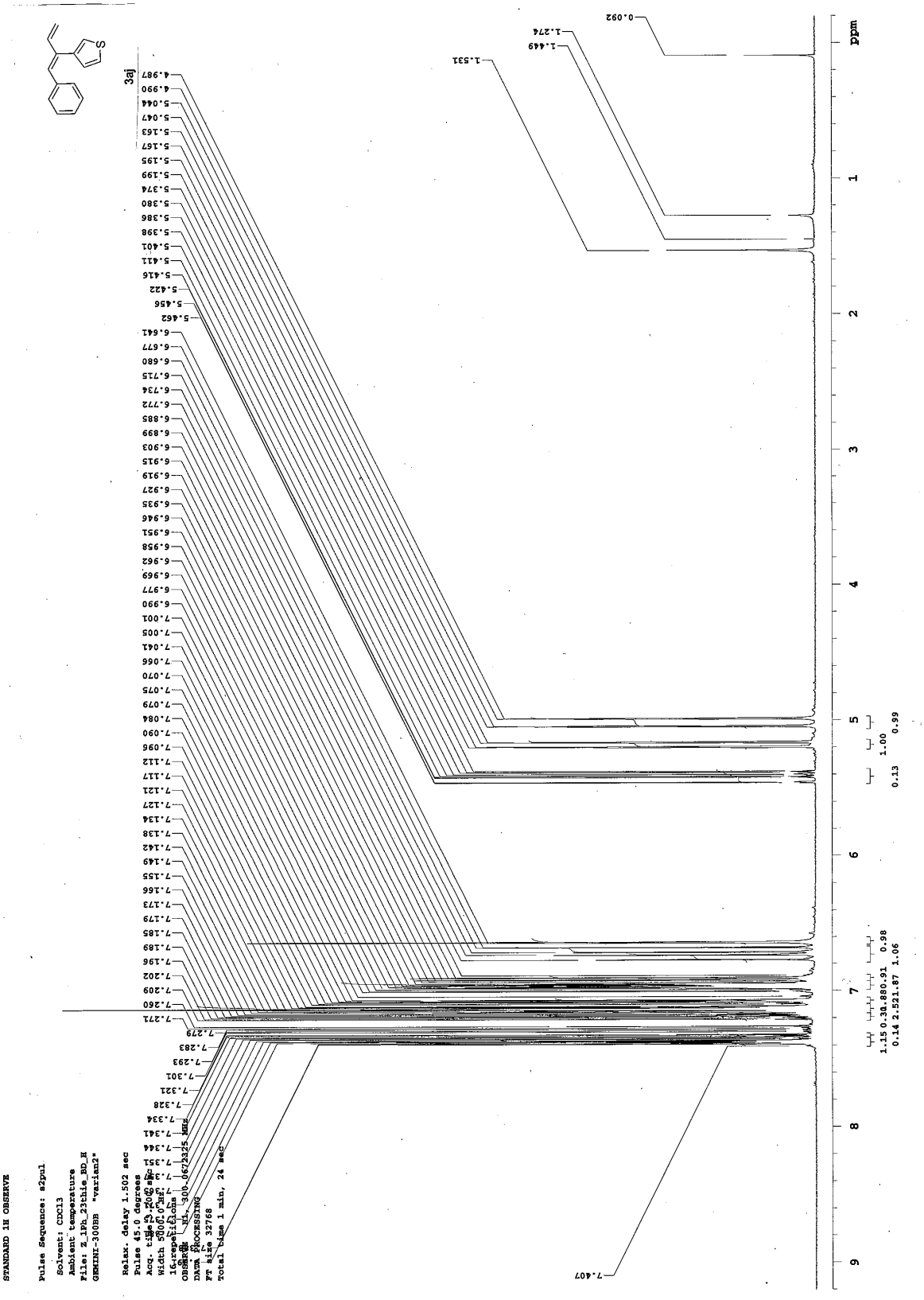
13C OBSERVE

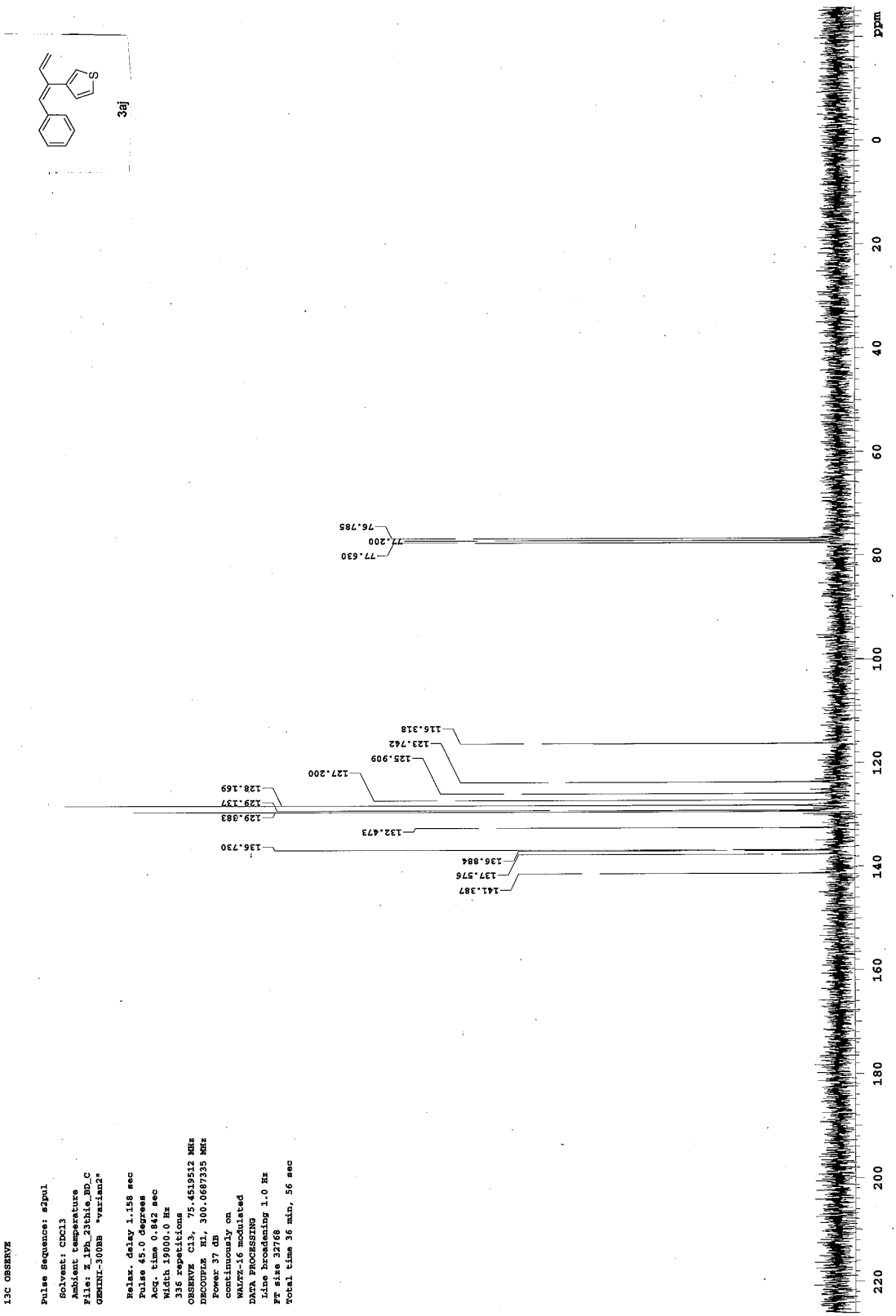
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: Z_1FB_2pMOC_B4_C
GEMINI-300BB "varian2"

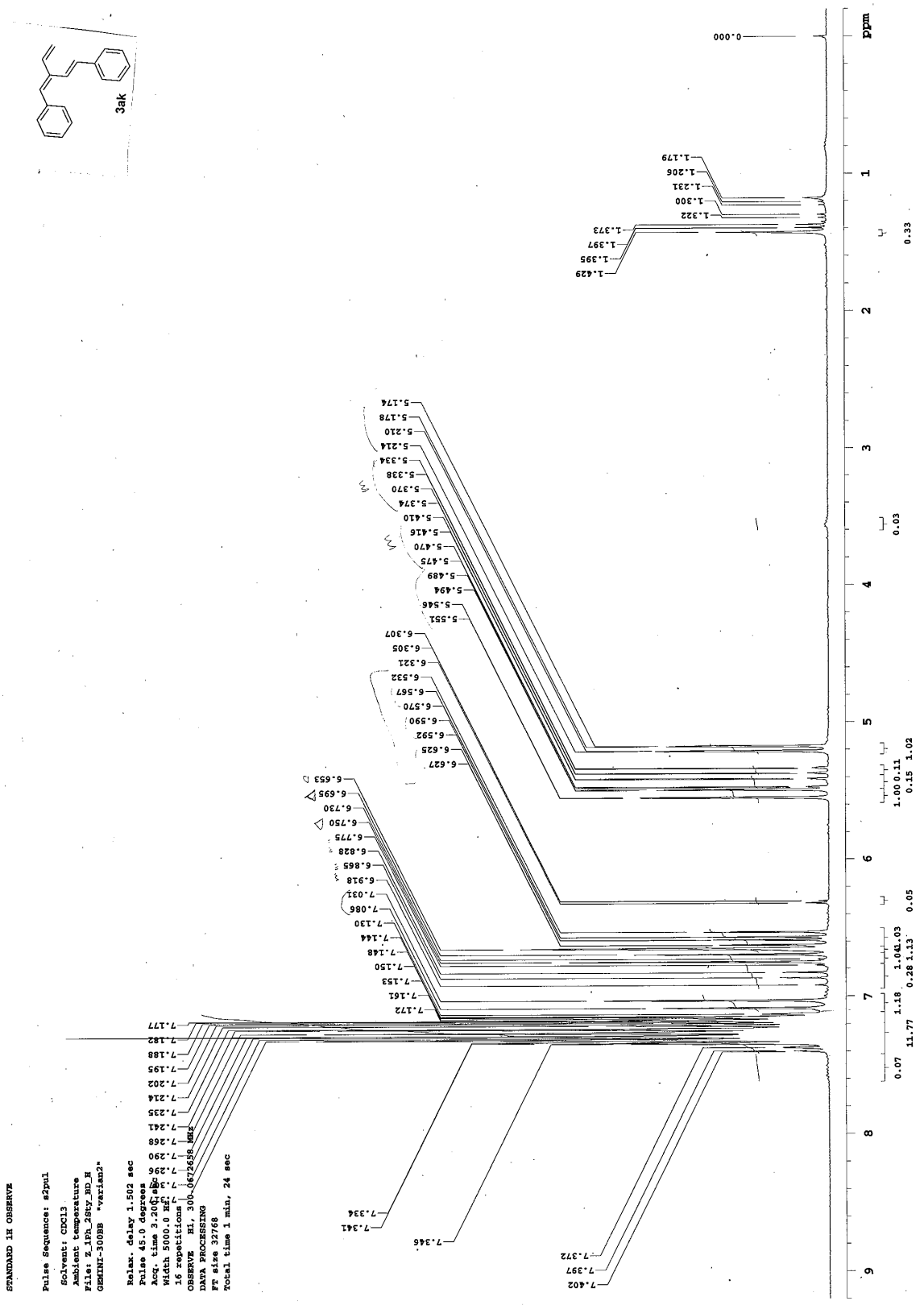
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
80 repetitions
OBSERVE C13, 75.4519535 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 36 min, 56 sec

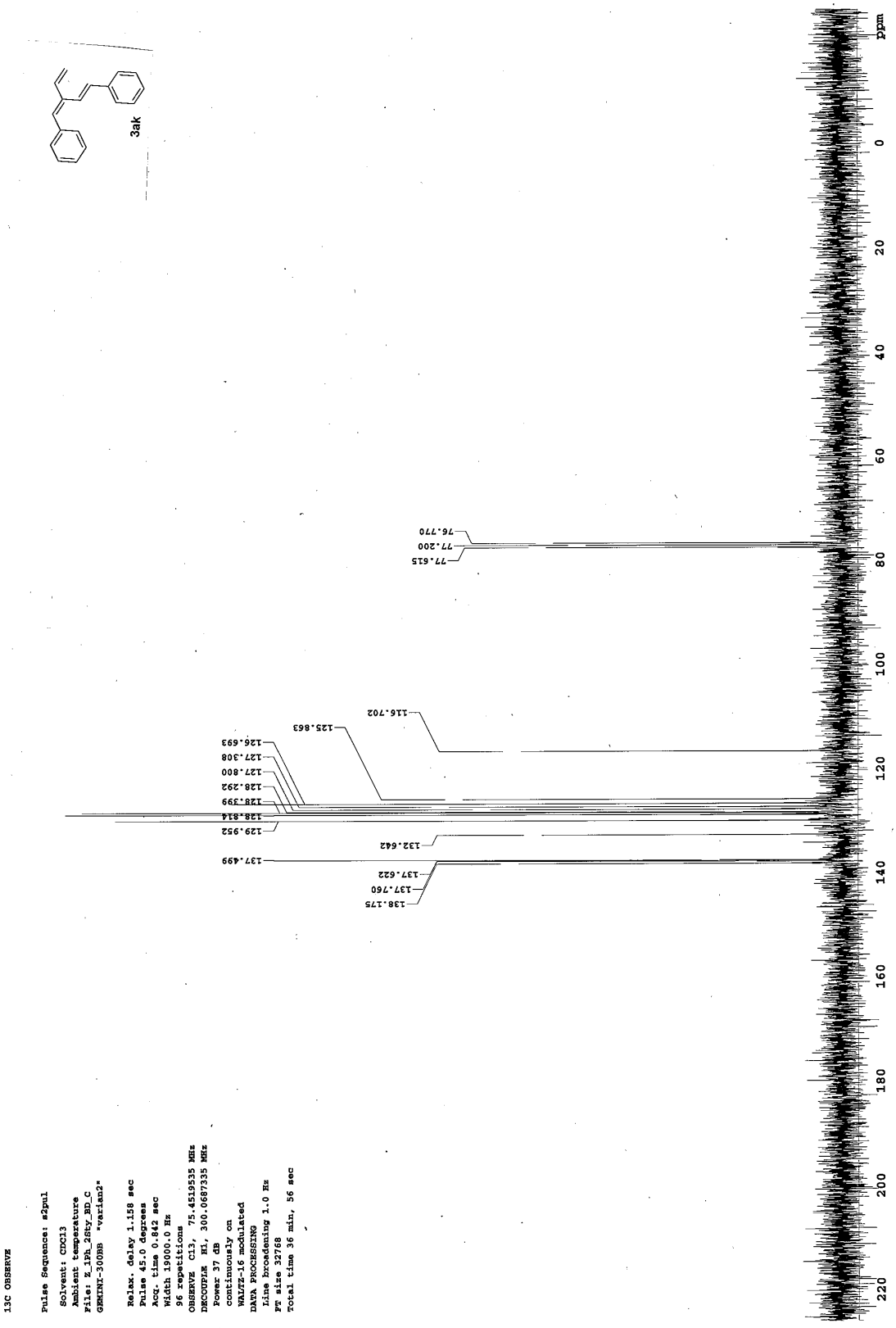

3ai
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 2_1Ph_2thie_BD_H
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672328 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 24 sec
ppm

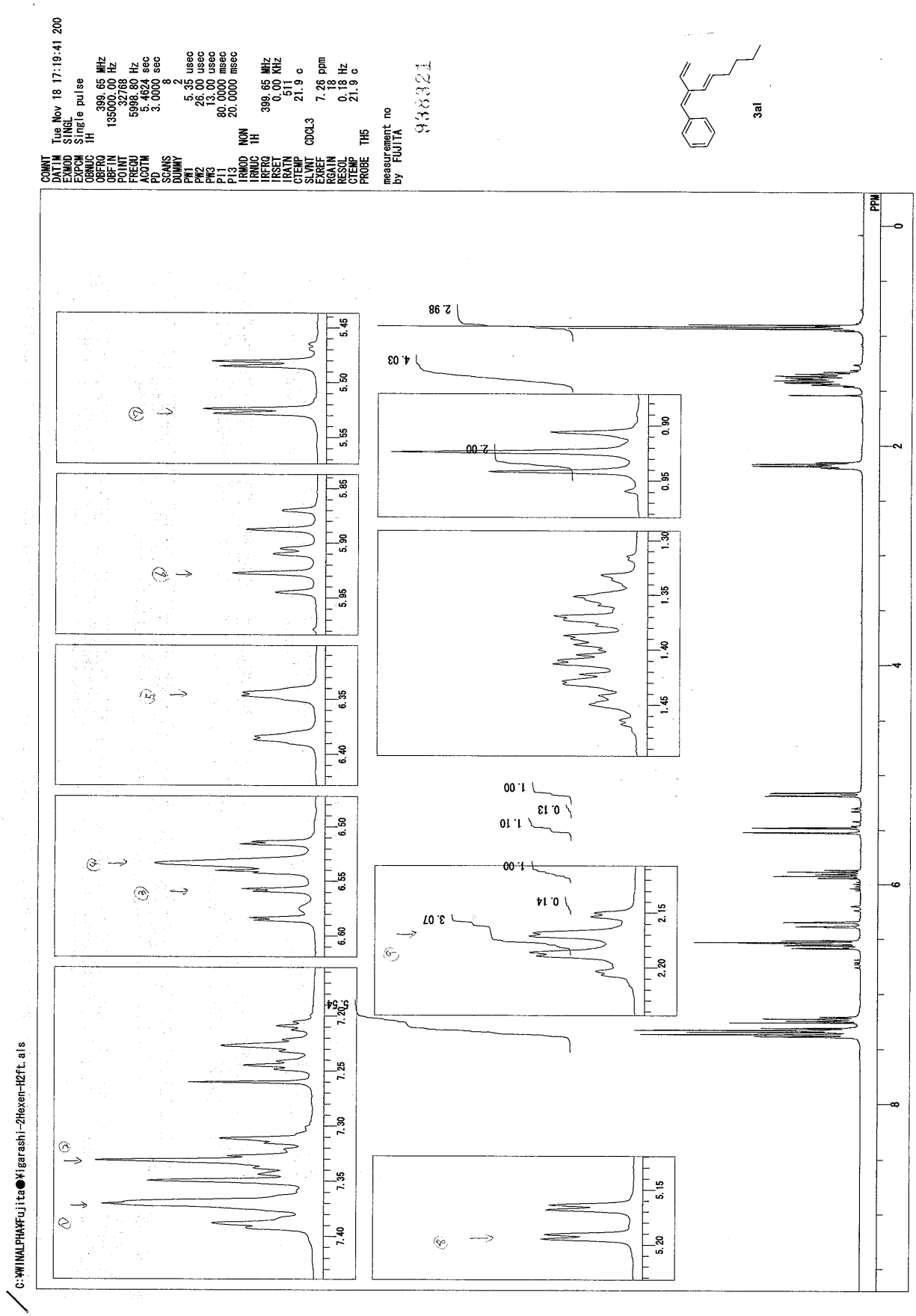

3ai
938562
G:¥murakami labo¥igarashi¥MF-igarashi-Z 2 thie-dept135-1ft.als
G:¥murakami labo¥igarashi¥MF-igarashi-Z 2 thie-NMR-1ft.als

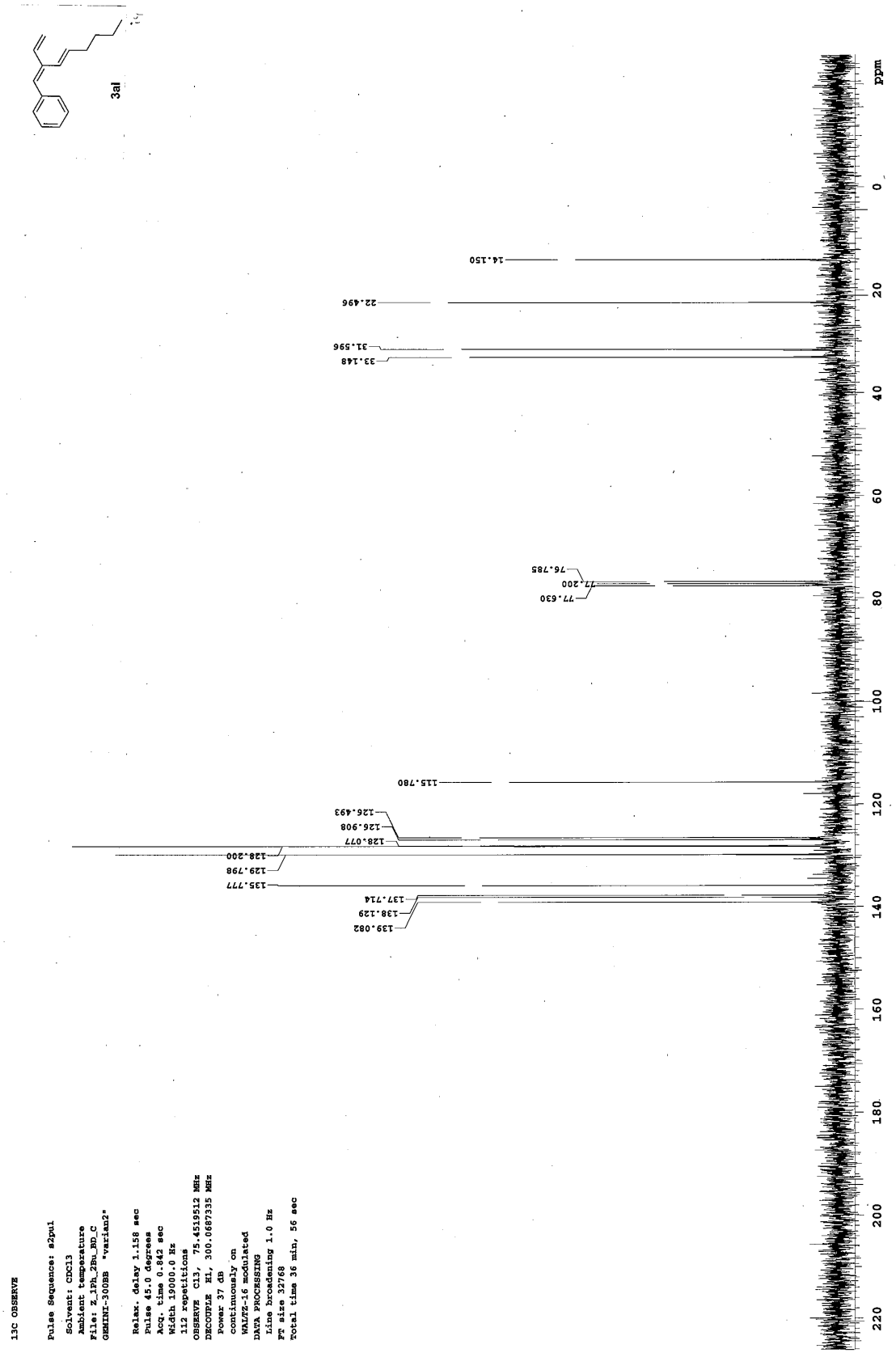

3aj
S
SPANNARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 2_1Ph_2Sthie_BD_H
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 5.440 sec
Width 5001.6 Hz
16 repetitions
OBSERVE H1, 300.0673325 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec
0.092
1.274
1.449
1.531
7.407
ppm
1.15 0.38 0.80 0.91 0.98
0.14 2.52 1.87 1.06
0.13 1.00 0.99

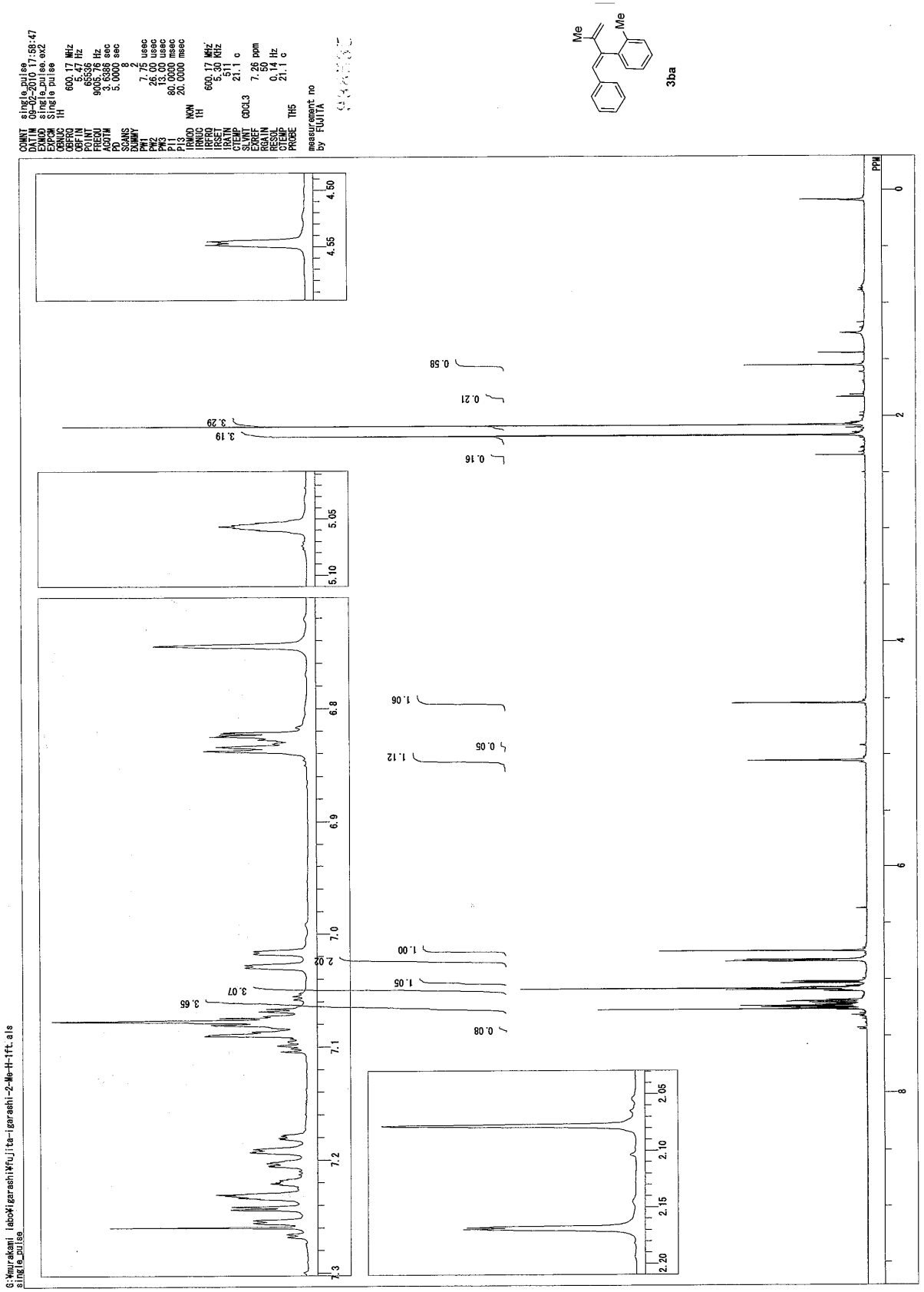

3aj
S
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: Z_1Ph_2Sthie_BD_C
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
336 repetitions
OBSERVE C13, 75.4519512 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 36 min, 56 sec
141.387
137.576
136.884
136.730
132.473
129.383
129.137
128.169
127.200
125.909
123.742
116.318
77.630
77.200
76.785
ppm
220
200
180
160
140
120
100
80
60
40
20
0

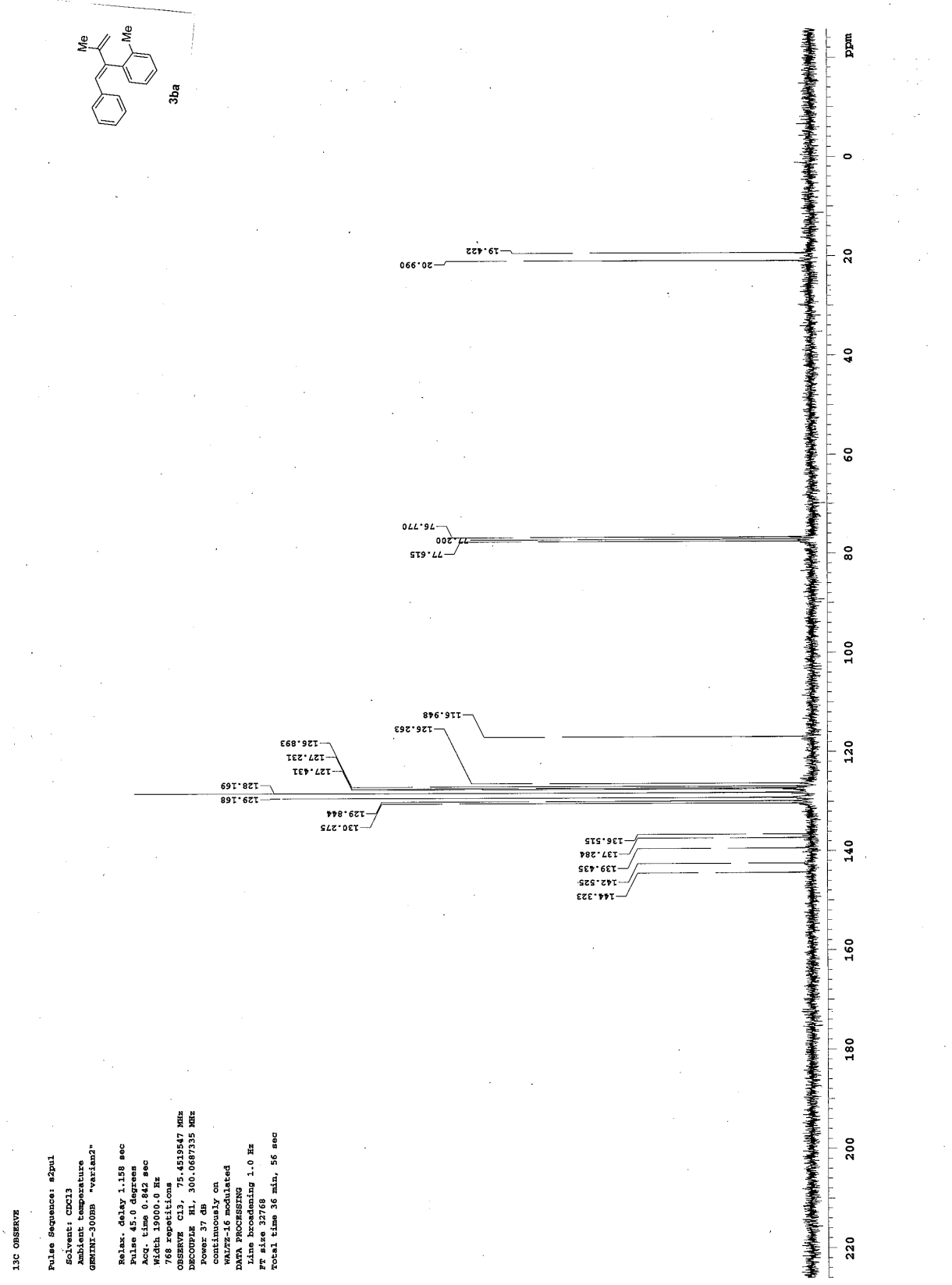

3ak
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: Z_1PA_28ty_RD_H
GEMINI-300BB "varian2"
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672658 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec
ppm

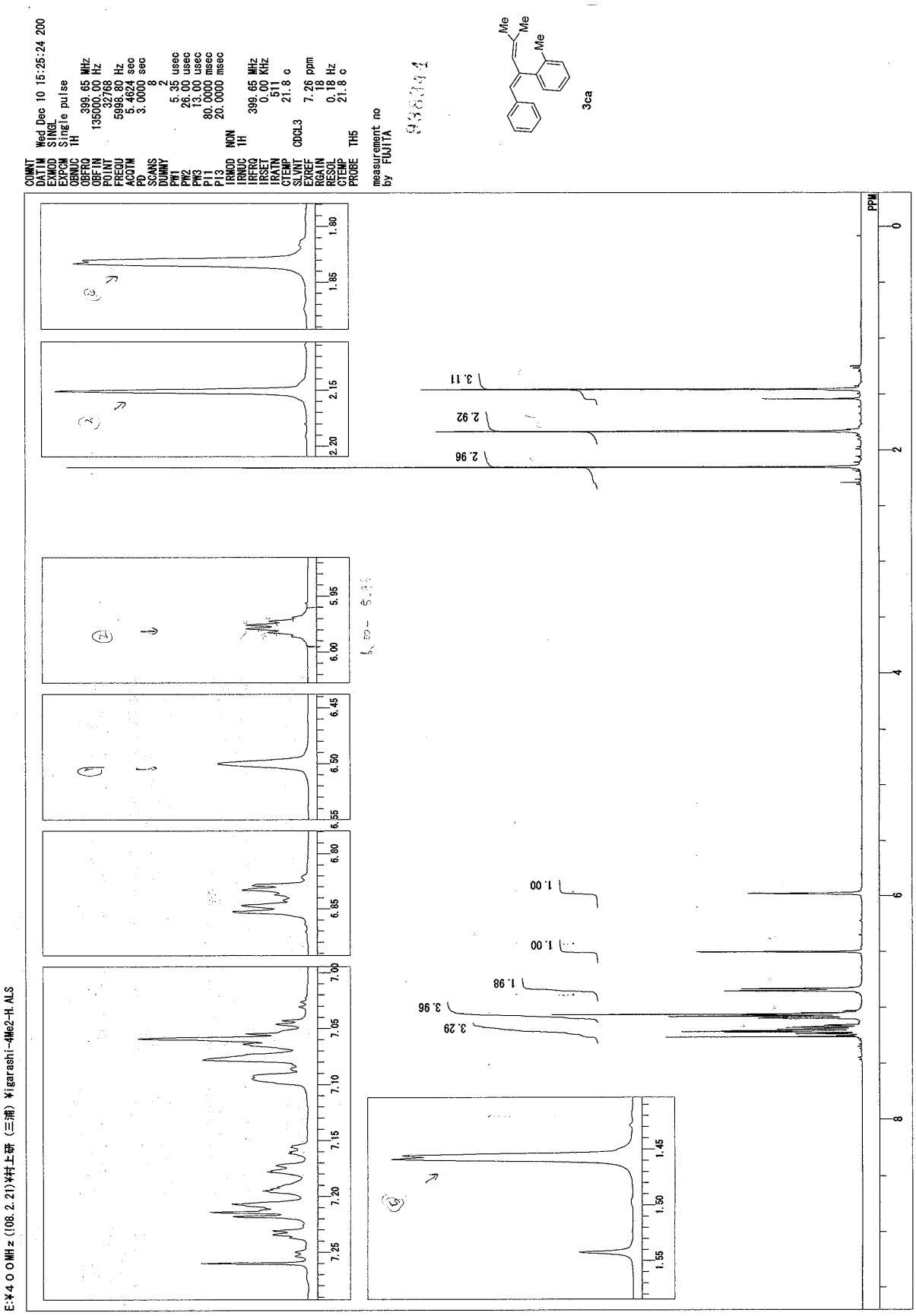

3ak
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: Z_1Ph_2Sty_BD_C
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
96 repetitions
OBSERVE C13, 75.4519535 MHz
DECOUPLE H1, 300.0687335 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 36 min, 56 sec
77.615
77.200
76.770
116.702
125.863
126.693
127.308
127.800
128.292
128.399
128.814
129.952
132.642
137.499
137.622
137.760
138.175
ppm

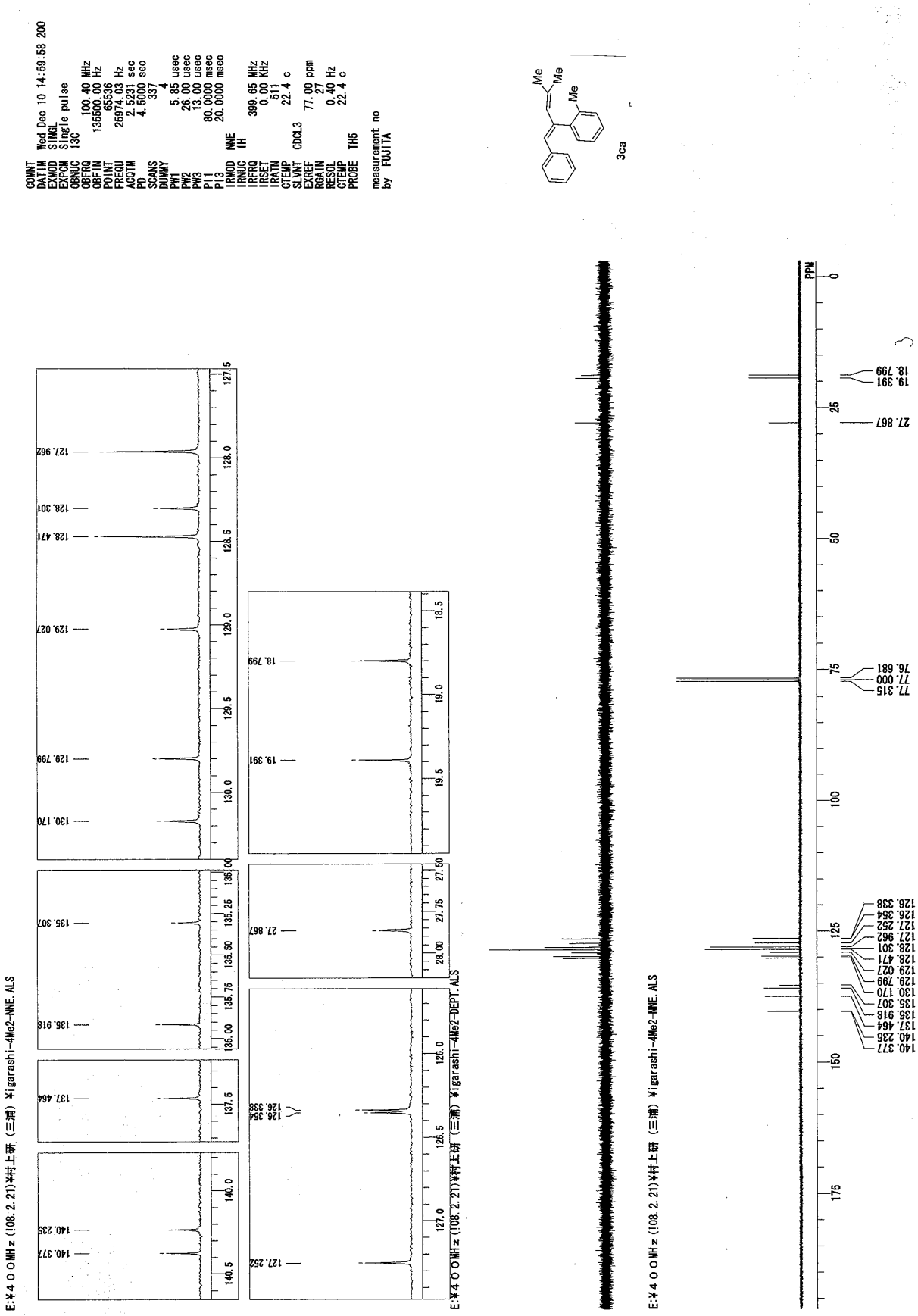

COMMT Tue Nov 18 17:19:41 200
DATIM
EXMOD SINGL
EXPCM Single pulse
OBNUC 1H
OBFRQ 399.65 MHz
OBFIN 135000.00 Hz
POINT 32768
FREQU 5998.80 Hz
ACQTM 5.4624 sec
PD 3.0000 sec
SCANS 8
DUMMY 2
PW1 5.35 usec
PW2 26.00 usec
PW3 13.00 usec
P11 80.0000 msec
P13 20.0000 msec
IRMOD NON
IRNUC 1H
IRFRQ 399.65 MHz
IRSET 0.00 KHz
IRATN 511
CTEMP 21.9 c
SLVNT CDCL3
EXREF 7.26 ppm
RGAIN 18
RESOL 0.18 Hz
CTEMP 21.9 c
PROBE TH5
measurement no
by FUJITA
3al
PPM
2.98
4.03
2.00
1.00
0.13
1.10
1.00
0.14
3.07
1.00

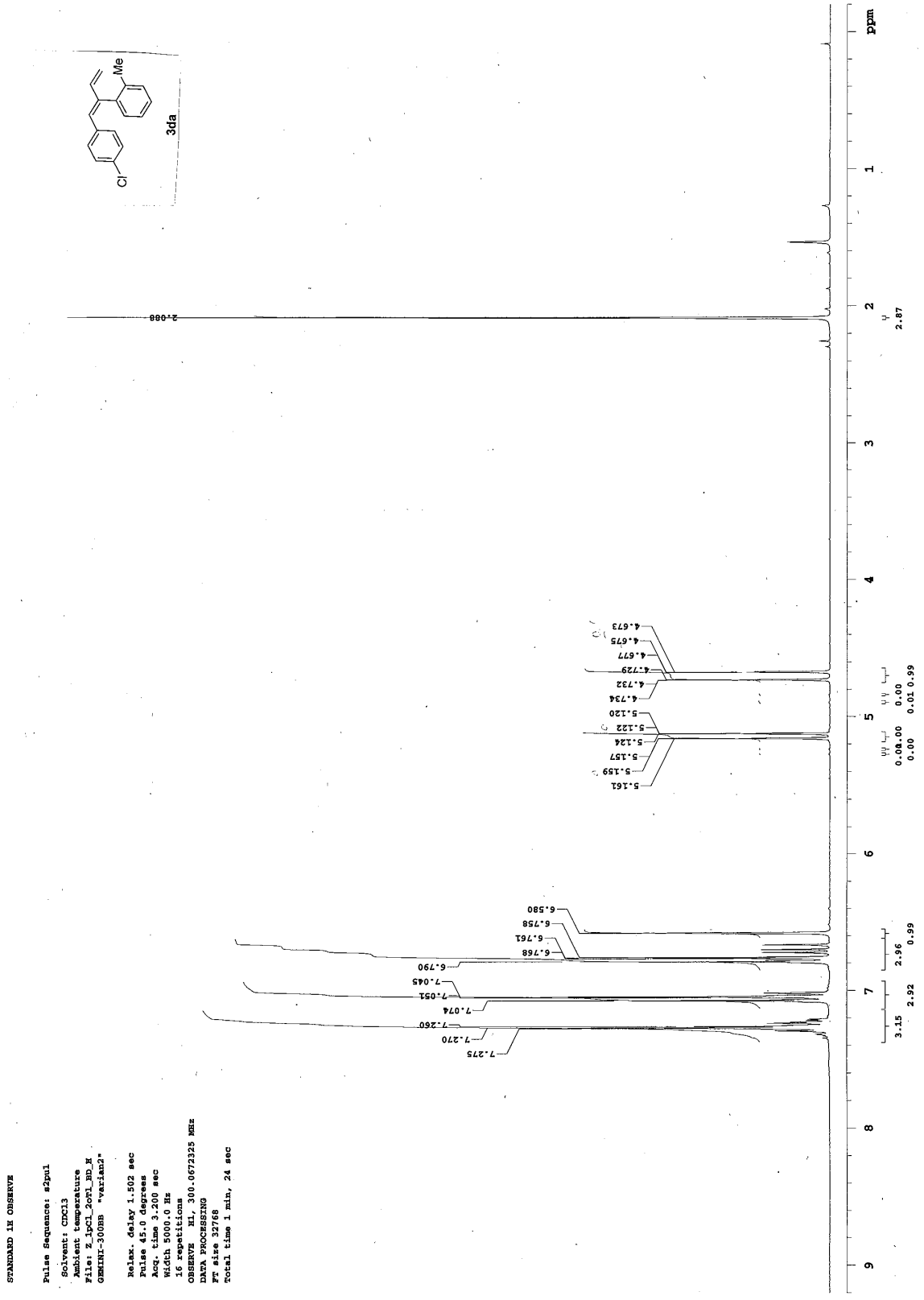

3al
14.150
22.496
31.596
33.148
76.785
77.200
77.630
115.780
126.493
126.908
128.077
128.200
129.798
135.777
137.714
138.129
139.082
ppm
0
20
40
60
80
100
120
140
160
180
200
220
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: Z_1Ph_2Bn_RD_C
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
112 repetitions
OBSERVE C13, 75.4519512 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 36 min, 56 sec


Me
Me
3ba
COMNT single_pulse
DATIM 09-02-2010 17:58:47
EXMOD single_pulse.ex2
EXPNO 1
OBNUC 1H
OBFRQ 600.17 MHz
OBFIN 5.47 Hz
POINT 65536
FREQU 9005.76 Hz
ACQTM 3.6388 sec
PD 5.0000 sec
SS 2
DUMMY 2
PW1 7.75 usec
PW2 26.00 usec
PW3 13.00 usec
P11 80.0000 msec
P12 20.0000 msec
TRMOD NON
IRNUC 1H
IRFRQ 600.17 MHz
IRSET 5.30 KHz
IRATN 511
CTEMP 21.1 c
SLVNT CDCL3
CSHIFT 7.26 ppm
RGAIN 50
RESOL 0.14 Hz
OTEMP 21.1 c
PROBE TH5
measurement no
by TOUTIA
G:¥murakami labo¥igarashi¥fujita-igarashi-2-Me-H-1ft.als
single_pulse
PPM
0.58
0.21
3.29
3.19
0.16
1.06
0.65
1.12
1.00
1.05
0.80
3.07
3.66


Me
Me
3ba
19.422
20.990
76.770
77.200
77.615
116.948
126.263
126.893
127.231
127.431
128.168
129.168
129.844
130.275
136.515
137.284
139.435
142.525
144.323
ppm
0
20
40
60
80
100
120
140
160
180
200
220
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
768 repetitions
OBSERVE C13, 75.4519547 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 36 min, 56 sec


Me
Me
Me
3ca
3.11
2.92
2.96
1.00
1.00
1.98
3.96
3.29
PPM

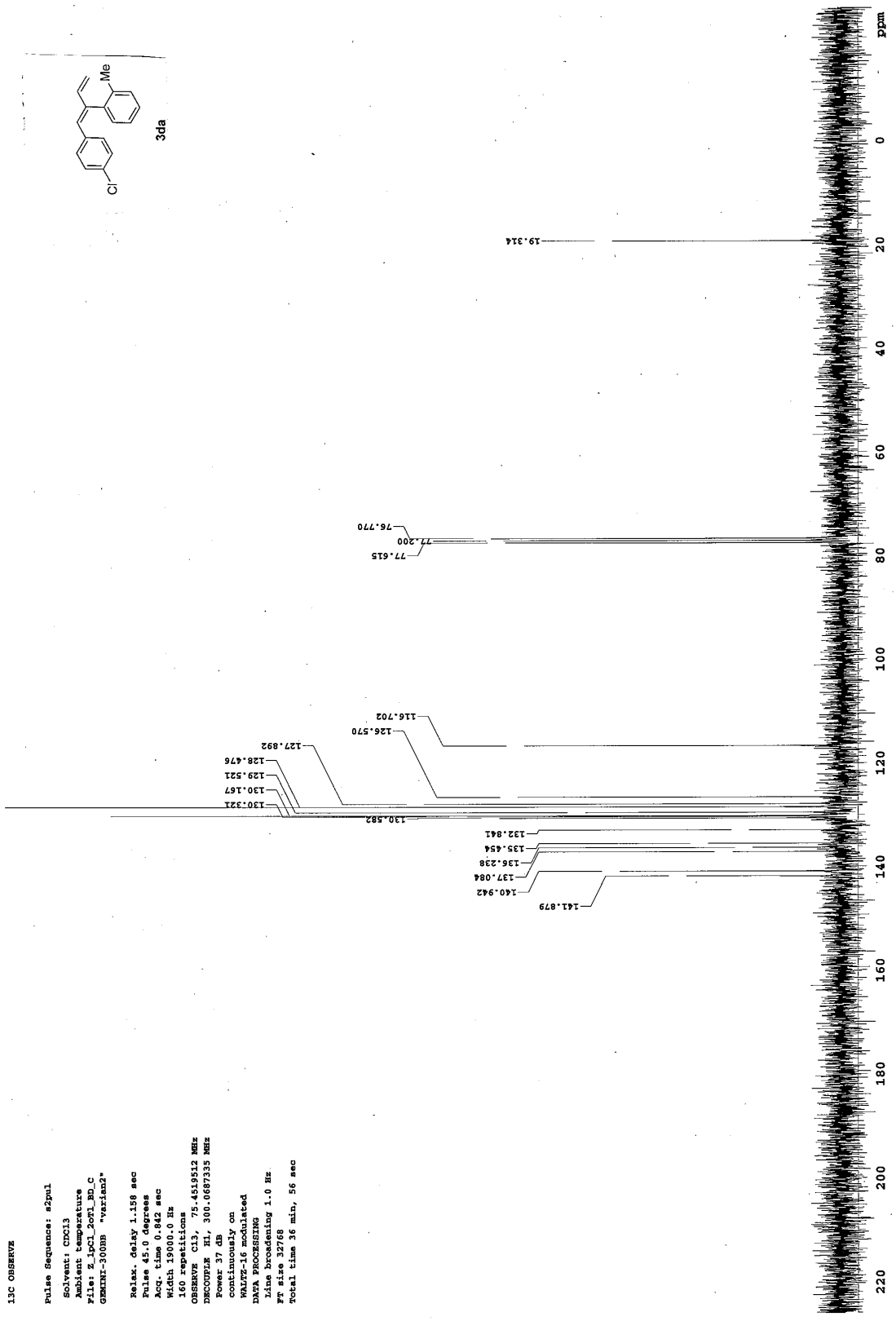

COMNT
DATIM Wed Dec 10 14:59:58 200
EXMOD SINGL
EXPCM Single pulse
OBNUC 13C
OBFRQ 100.40 MHz
OBFIN 135500.00 Hz
POINT 65536
FREQU 25974.03 Hz
ACQTM 2.5231 sec
PD 4.5000 sec
SCANS 337
DUMMY 4
PW1 5.85 usec
PW2 26.00 usec
PW3 13.00 usec
P11 80.0000 msec
P13 20.0000 msec
IRMOD NNE
IRNUC 1H
IRFRQ 399.65 MHz
IRSET 0.00 KHz
IRATN 511
CTEMP 22.4 c
SLVNT CDCL3
EXREF 77.00 ppm
RGAIN 27
RESOL 0.40 Hz
CTEMP 22.4 c
PROBE TH5
measurement no
by FUJITA
Me
Me
Me
3ca
E:¥400MHz (108.2.21)¥村上研 (三浦) ¥igarashi-4Me2-NME.ALS
E:¥400MHz (108.2.21)¥村上研 (三浦) ¥igarashi-4Me2-DEPT.ALS
E:¥400MHz (108.2.21)¥村上研 (三浦) ¥igarashi-4Me2-NME.ALS
PPM

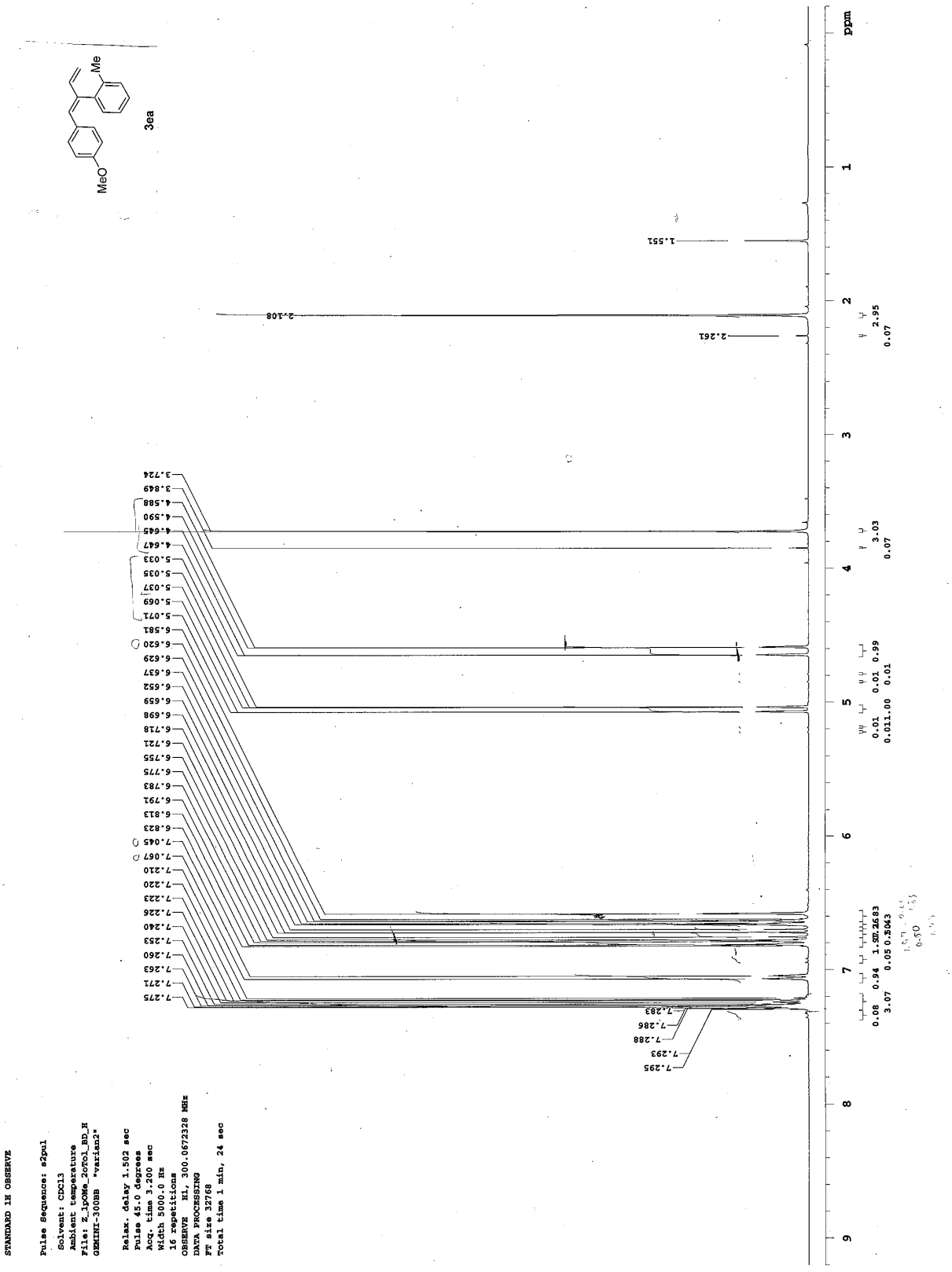


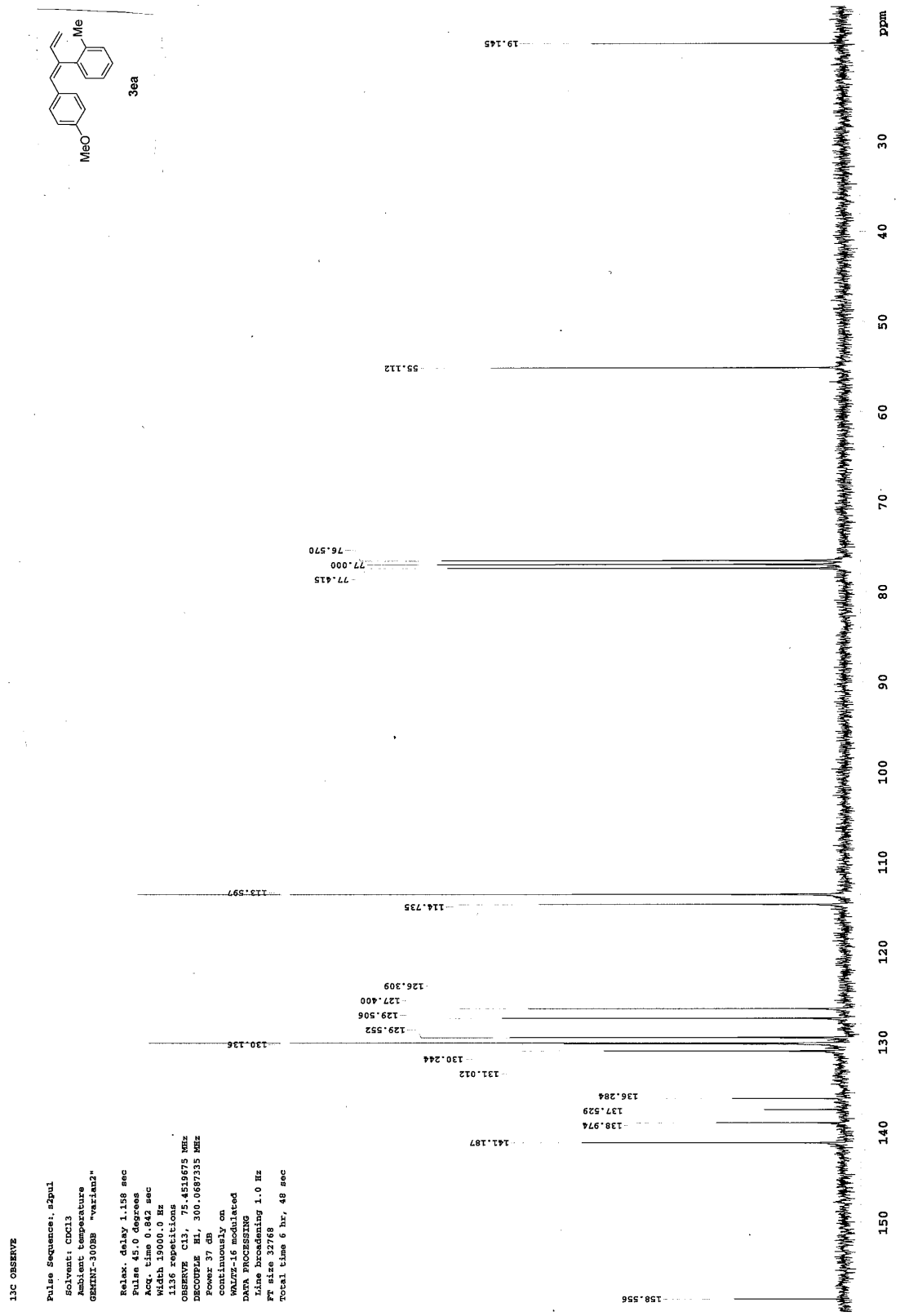
STANDARD 1H OBSERVE

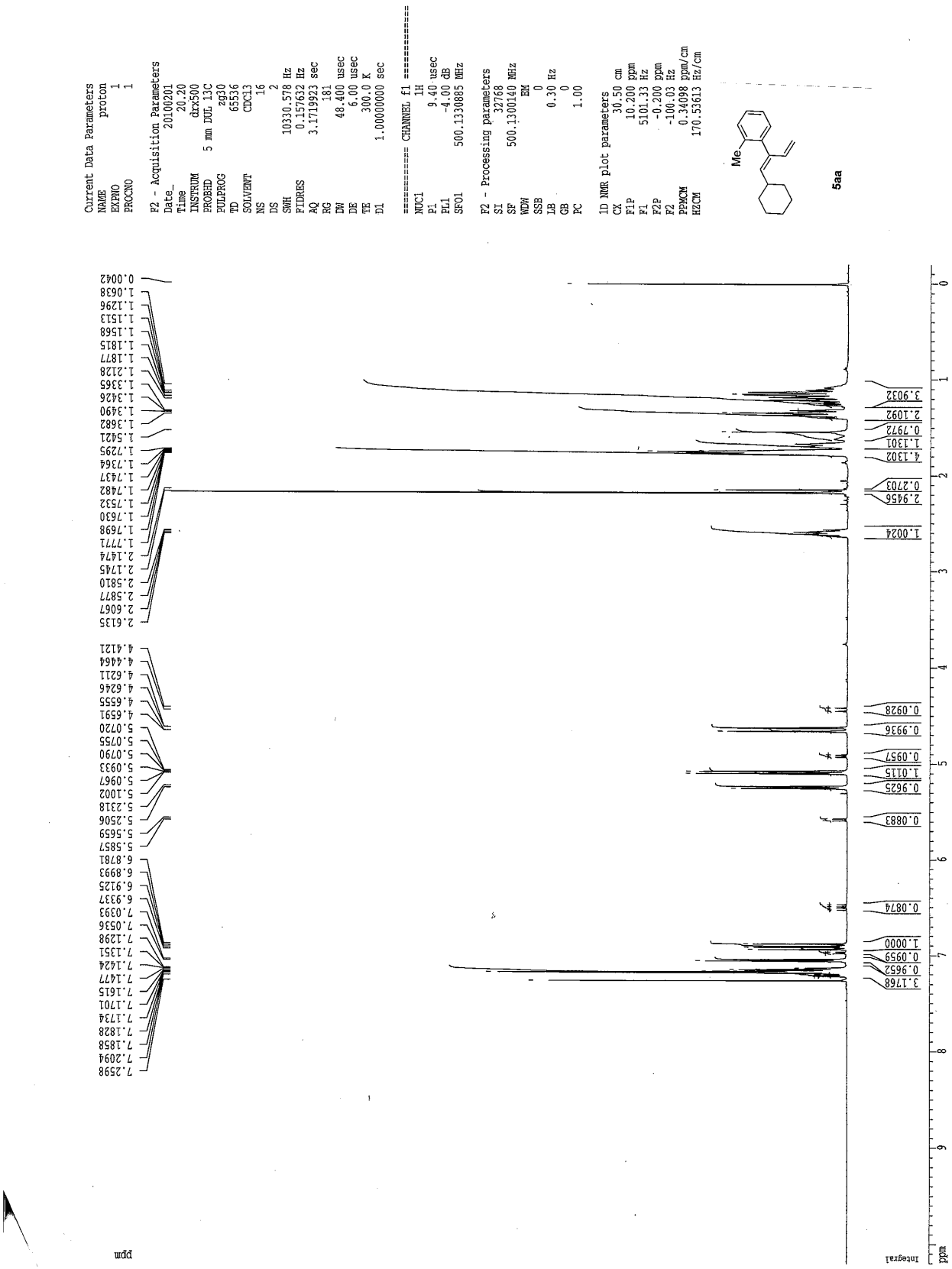
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: Z_1pc1_2of1_RD_K
GEMINI-300BB "varian2"

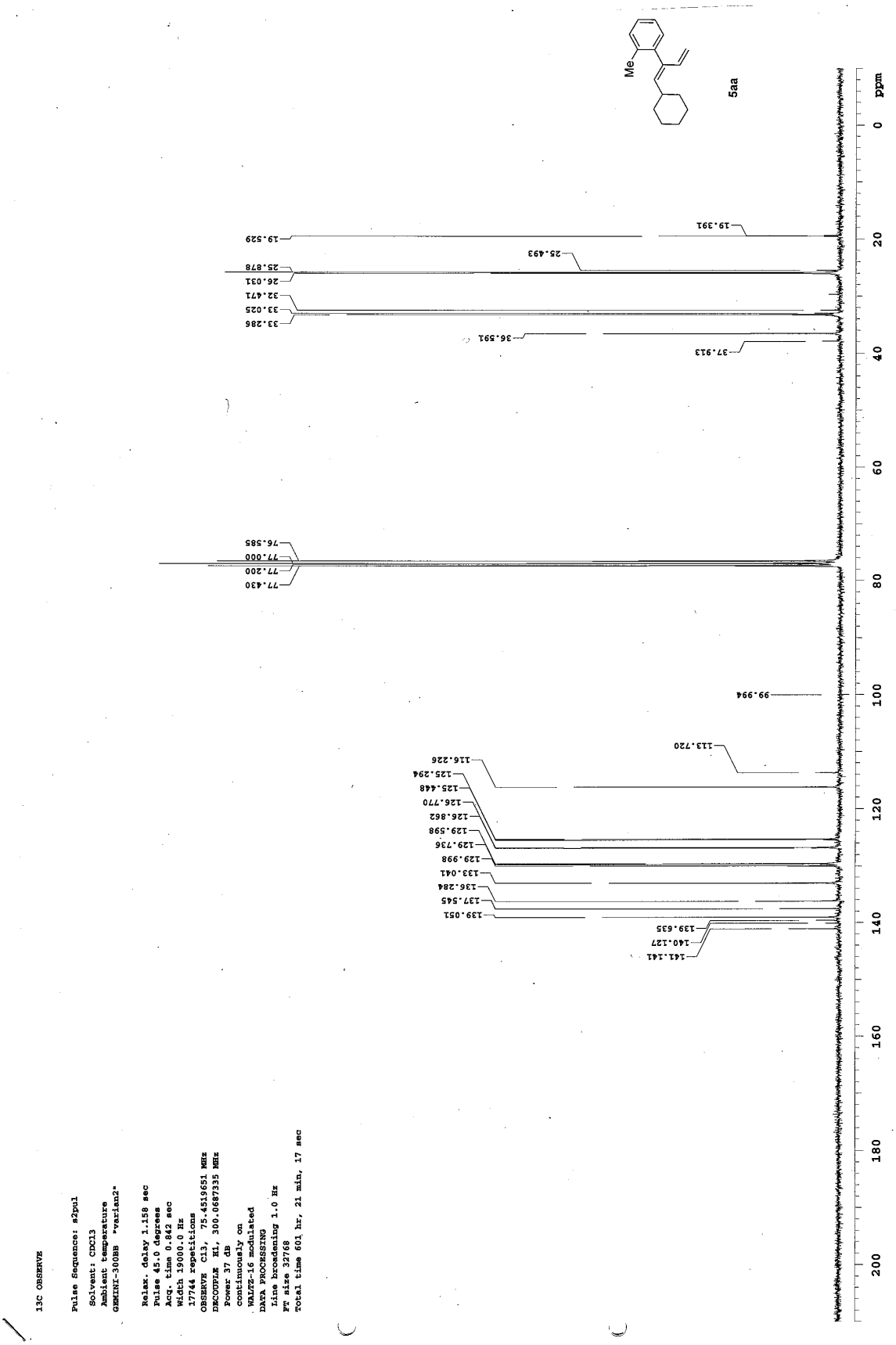
Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.200 sec
Width 5000.0 Hz
16 repetitions
OBSERVE H1, 300.0672325 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 24 sec


3da
Me
Cl
19.314
76.770
77.200
77.615
116.702
126.570
127.892
128.476
129.521
130.167
130.321
130.582
132.841
135.454
136.238
137.084
140.942
141.879
ppm
0
20
40
60
80
100
120
140
160
180
200
220
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 2_1pCl_2oTl_BD_C
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
160 repetitions
OBSERVE C13, 75.4519512 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 36 min, 56 sec


Me
MeO
3ea
STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: Z_1pOMe_2oTol_BD_H
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
ppm


Me
MeO
3ea
19.145
55.112
76.570
77.000
77.415
113.597
114.735
126.309
127.400
129.506
129.552
130.136
130.244
131.012
136.284
137.529
138.974
141.187
158.556
ppm


Current Data Parameters
NAME proton
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100201
Time 20.20
INSTRUM drx500
PROBHD 5 mm DUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 181
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
========== CHANNEL f1 ==========
NUC1 1H
P1 9.40 usec
PL1 -4.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300140 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 30.50 cm
F1P 10.200 ppm
F1 5101.33 Hz
F2P -0.200 ppm
F2 -100.03 Hz
PPMCM 0.34098 ppm/cm
HZCM 170.53613 Hz/cm
Me
5aa
0.0042
1.0638
1.1296
1.1513
1.1568
1.1815
1.1877
1.2128
1.3365
1.3426
1.3490
1.3682
1.5421
1.7295
1.7364
1.7437
1.7482
1.7532
1.7630
1.7698
1.7771
2.1474
2.1745
2.5810
2.5877
2.6067
2.6135
4.4121
4.4464
4.6211
4.6246
4.6555
4.6591
5.0720
5.0755
5.0790
5.0933
5.0967
5.1002
5.2318
5.2506
5.5659
5.5857
6.8781
6.8993
6.9125
6.9337
7.0393
7.0536
7.1298
7.1351
7.1424
7.1477
7.1615
7.1701
7.1734
7.1828
7.1858
7.2094
7.2598
3.9032
2.1092
0.7972
1.1301
4.1302
0.2703
2.9456
1.0024
0.0928
0.9936
0.0957
1.0115
0.9625
0.0883
0.0874
1.0000
0.0959
0.9652
3.1768
ppm
Integral

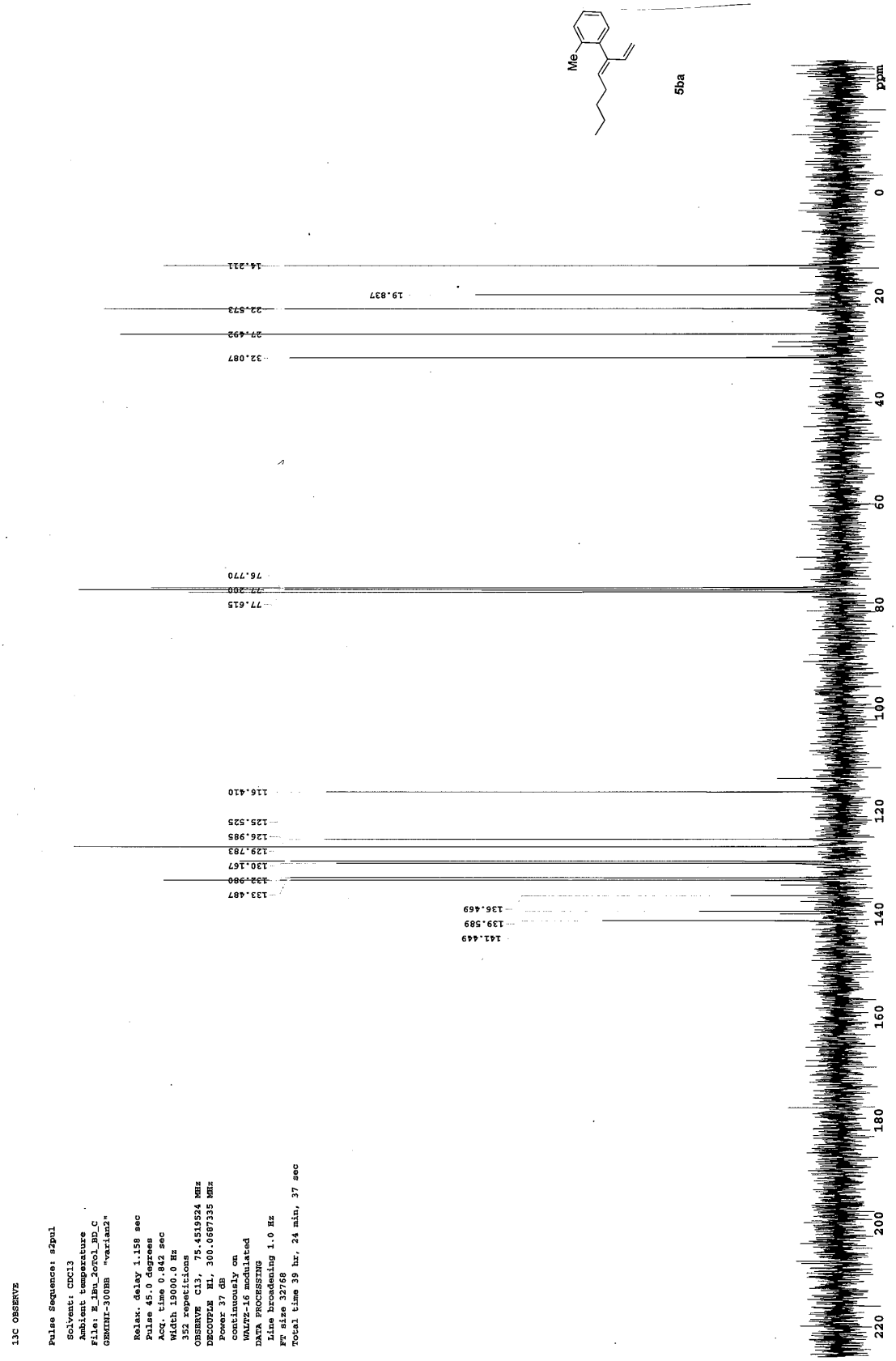


13C OBSERVE

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

Relax. delay 1.150 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
17744 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


Me
5ba
COMMNT single_pulse
DATIM 04-02-2010 09:06:25
EXMOD single_pulse.ex2
EXPCM single pulse
OBFRQ 1H 399.78 MHz
OBSFO 7.29 Hz
OBFIN 32768
POINT 5998.08 Hz
FREQU 5.4631 sec
ACQTM 6.0000 sec
PD 8
SCANS 2
DUMMY 4.82 usec
PW1 26.00 usec
PW2 13.00 usec
PW3 80.0000 msec
P11 20.0000 msec
P13
IRMOD NON
IRNUC 1H 399.78 MHz
IRFRQ 4.19 KHz
IRSET 511
IRATN 19.6 c
CTEMP
SLVNT CDCL3 7.26 ppm
EXREF 32 Hz
RGAIN 0.12 Hz
RESOL 19.6 c
CTEMP
PROBE TH5
measurement no
by FUJITA
G:¥murakami labo¥igarashi¥F-igarashi-Oct-H-1ft.als
single_pulse
PPM


Me
5ba
ppm
0
20
40
60
80
100
120
140
160
180
200
220
14.211
19.837
22.573
27.492
32.087
76.770
77.200
77.615
116.410
125.525
126.985
129.783
130.167
132.980
133.487
136.469
139.589
141.449
13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: K_1Bn_2oTol_BD_C
GEMINI-300BB "varian2"
Relax. delay 1.158 sec
Pulse 45.0 degrees
Acq. time 0.842 sec
Width 19000.0 Hz
352 repetitions
OBSERVE C13, 75.4519524 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 39 hr, 24 min, 37 sec