Electronic Supplementary Material (ESI) for Medicinal Chemistry Communications

Synthesis of Chiral α-Amino Acid-derived 1*H*-1,2,4-Triazoles and 1,2,4-Triazines

Ebrahim H. Ghazvini Zadeh,[a] Bahaa El-Dien M. El-Gendy,[a,b] Alex G. Pop,[a] and Alan R. Katritzky*[,a]

[a]Center for Heterocyclic Compounds, Department of Chemistry, University of Florida, Gainesville, FL 32611-7200. [b]Department of Chemistry, Faculty of Science, Benha University, Benha, Egypt.

katritzky@chem.ufl.edu

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Experimental Section

Melting points were determined on a capillary point apparatus equipped with a digital thermometer and are uncorrected. The ^{1}H and ^{13}C NMR spectra for starting materials were recorded on a Varian Gemini instrument, operating at 300 MHz for ^{1}H and 75 MHz for ^{13}C with TMS as an internal reference.

General Procedure for the Synthesis Aminoacylamidrazones 7a-n, 7f':

Method A: A solution of *N*-protected amino acylbenzotriazole **5a-n, 5f'** (1.0 mmol), 2-pyridylamidrazone **6** (1.0 mmol) and diisopropylethylamine (1.0 mmol) in acetonitrile (10 mL) was stirred at room temperature for 5 h. The precipitate was collected on a Buchner funnel and was washed with diethylether (20 mL), water (30 mL) and methanol (5 mL). The solid was dried *in vacuo* to afford the desired aminoacylamidrazones **7** as a white solid.

Method B: A solution of **5a-n, 5f'** (1.0 mmol) and **6** (1.0 mmol) was heated to reflux in acetonitrile (5 mL) for 1 h. The mixture was worked up as in A.

(*S*)-Benzyl (1-(2-(amino(pyridin-2-yl)methylene)hydrazinyl)-3-methyl-1-oxobutan-2-yl)carbamate (7a). White microcrystals (73%); mp 203.0–205.0 °C. Two rotamers (2:1): ^{1}H NMR (DMSO-d_6, 300 MHz) δ Two rotamers: 10.08 (s, 0.3H), 9.95 (s, 0.7H), 8.60-8.56 (m, 1H), 8.08 (t, J = 8.4 Hz, 1H), 7.93-7.80 (m, 1H), 7.49-7.43 (m, 2H), 7.41-7.23 (m, 5H), 6.74 (s, 1H), 6.68 (s, 1H), 5.05-4.99 (m, 2H), 3.94-.85 (m, 1H), 2.30-2.15 (m, 0.3H), 2.05-1.90 (m, J = 6.9 Hz, 0.7H), 1.00-0.87 (m, 6H). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 172.4, 167.1, 156.2, 150.5, 148.1, 146.3, 142.5, 137.1, 137.0, 136.8, 128.3, 127.8, 127.7, 124.6, 124.5, 120.6, 119.2, 65.4, 65.3, 59.6, 56.0, 30.3, 29.5, 19.6, 19.3, 19.1, 18.7. Anal. Calcd. for $C_{19}H_{23}N_5O_3$ (369.43): C, 61.77; H, 6.28; N, 18.96. Found: C, 61.88; H, 6.51; N, 18.60.

(*S*)-Benzyl(1-(2-(amino(pyridin-2-yl)methylene)hydrazinyl)-1-oxo-3-phenylpropan-2-yl)-carbamate (7b). White microcrystals (72%); mp 168.0–170.0 ˚C. Two rotamers (1:1): ^{1}H NMR (DMSO-d_6, 300 MHz) δ Two rotamers: 10.10 (s, 0.5H), 10.02 (s, 0.5H), 8.59 (d, J = 4.8 Hz, 1H), 8.10 (dd, J = 8.1, 3.6 Hz, 1H), 7.94-7.85 (m, 1H), 7.70 (d, J = 8.4 Hz, 0.5H), 7.53 (d, J = 9.0 Hz, 0.5H), 7.50-7.45 (m, 1H), 7.38-7.12 (m, 9H), 6.73-6.65 (m, 2H), 5.38-5.15 (m, 0.5H), 5.00-4.90 (m, 2H), 4.38 (sextet, J = 4.5 Hz, 0.5H), 3.14-2.95 (m, 1H), 2.91-2.70 (m, 1H). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 172.4, 166.4, 156.0, 150.8, 148.2, 148.1, 146.4, 143.0, 138.6, 137.2, 136.9, 129.1, 129.3, 128.3, 128.3, 128.1, 127.6, 127.5, 127.4, 126.3, 125.0, 120.6, 65.2, 65.1, 55.0, 52.5, 38.0, 37.0. Anal. Calcd. for $C_{23}H_{23}N_5O_3$ (417.47): C, 66.17; H, 5.55; N, 16.78. Found: C, 66.08; H, 5.55; N, 16.87.

Benzyl (2-(2-(amino(pyridin-2-yl)methylene)hydrazinyl)-2-oxoethyl)carbamate (7c). White microcrystals (83%); mp 153.0–155.0 ˚C. Two rotamers (2:1): ^{1}H NMR (DMSO-d_6, 300 MHz) δ 10.11 (s, 0.7H), 9.89 (br s, 0.3H), 8.57 (t, J = 5.1 Hz, 1H), 8.08 (d, J = 8.1 Hz, 1H), 7.86 (q, J = 7.8 Hz, 1H), 7.54 -7.25 (m, 7H), 6.70-6.60 (m, 2H), 5.07-5.03 (m, 2H), 4.16 (d, J = 6.0 Hz, 1.3H), 3.74 (d, J = 6.0 Hz, 0.7H). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 170.0, 164.4, 156.6, 150.3, 148.0, 142.8, 137.2, 136.8, 128.3, 127.7, 124.5, 120.5, 120.2, 65.3, 42.2, 41.9. Anal. Calcd. for $C_{16}H_{17}N_5O_3$ (327.35): C, 58.71; H, 5.23; N, 21.39. Found: C, 58.63; H, 5.27; N, 21.42.

Benzyl ((2*S*,3*S*)-1-(2-(amino(pyridin-2-yl)methylene)hydrazinyl)-3-methyl-1-oxo pentan-2-yl)carbamate (7d). White microcrystals (71%); mp 223.0–224.0 °C. Two rotamers (2:1): ^{1}H NMR (DMSO-d_6, 300 MHz) δ 10.07 (br s, 0.4H), 9.96 (br s, 0.6H), 8.59 (d, J = 3.3 Hz, 1H), 8.08 (t, J = 8.1 Hz, 1H), 7.89 (q, J = 7.5 Hz, 1H), 7.50 (d, J = 9.3 Hz, 1H), 7.47-7.44 (m, 1H), 7.39-7.20 (m, 5H), 6.75 (br s, 1H), 6.67 (br s, 1H), 5.05-5.00 (m, 2H), 3.96 (t, J = 9.0 Hz, 1H), 1.98-1.88 (m, 0.33H), 1.82-1.70 (m, 0.67H), 1.60-1.40 (m, 1H), 1.28-1.10 (m, 1H), 0.95-0.78 (m,

6H). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 172.3, 167.2, 156.1, 150.5, 148.0, 146.3, 142.4, 137.1, 136.8, 128.3, 127.6, 124.6, 124.4, 120.6, 119.9, 65.4, 58.3, 55.0, 36.1, 24.6, 23.9, 15.8, 15.4, 11.3, 10.7. Anal. Calcd. for $C_{20}H_{25}N_5O_3$ (383.45): C, 62.65; H, 6.57; N, 18.26. Found: C, 62.96; H, 6.71; N, 18.32.

(*S*)-Benzyl (1-(2-(amino(pyridin-2-yl)methylene)hydrazinyl)-3-(1*H*-indol-3-yl)-1-oxopropan-2-yl)carbamate (7e). White microcrystals (69%); mp 171.0–173.0 °C. Two rotamers (1:1): ^{1}H NMR (DMSO-d_6, 300 MHz) δ 10.82 (d, J = 3.9 Hz, 1H), 10.07 (br s, 1H), 8.60 (t, J = 4.5 Hz, 1H), 8.09 (t, J = 9.9 Hz, 1H), 7.92-7.78 (m, 1H), 7.2-7.40 (m, 3H), 7.40-7.26 (m, 5H), 7.20 (d, J = 2.1 Hz, 1H), 7.09-6.97 (m, 2H), 6.85 (t, J = 7.5 Hz, 1H), 6.74 (br s, 2H), 5.30 (td, J = 9.6, 4.2 Hz, 0.5H), 5.03-4.86 (m, 2H), 4.49-4.40 (m, 0.5H), 3.24-3.09 (m, 1H), 3.05-2.91 (m, 1H). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 173.0, 167.6, 155.9, 150.5, 148.2, 148.1, 146.7, 143.3, 137.2, 136.8, 136.1, 128.3, 128.3, 127.6, 127.5, 127.3, 123.9, 120.9, 120.8, 120.6, 118.4, 118.2, 118.1, 111.3, 110.5, 110.1, 65.3, 65.1, 52.0, 46.2, 26.3. Anal. Calcd. for $C_{25}H_{26}N_6O_3$ (456.51): C, 65.78; H, 5.30; N, 18.41. Found: C, 65.57; H, 5.24; N, 18.44.

(*S*)-Benzyl (1-(2-(amino(pyridin-2-yl)methylene)hydrazinyl)-1-oxopropan-2-yl)carbamate (7f). White microcrystals (80%); mp 141.0–143.0 °C. Two rotamers (1:1): ^{1}H NMR (DMSO-d_6, 300 MHz) δ 10.02 (br s, 0.5H), 9.96 (br s, 0.5H), 8.60-8.56 (m, 1H), 8.08 (t, J = 7.5 Hz, 1H), 7.87 (t, J = 7.5 Hz, 1H), 7.58-7.42 (m, 2H), 7.40-7.20 (m, 5H), 6.80-6.65 (m, 2H), 5.03 (s, 2H), 5.10-4.90 (m, 0.5H), 4.18 (quin, J = 7.2 Hz, 0.5H), 1.32 (d, J = 7.2 Hz, 1.5H), 1.27 (d, J = 7.2 Hz, 1.5H). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 168.2, 155.7, 150.4, 148.0, 142.6, 136.9, 128.3, 127.7, 124.6, 124.4, 120.5, 120.0, 65.4, 65.2, 49.2, 47.1, 18.5, 17.0. Anal. Calcd. for $C_{17}H_{19}N_5O_3$ (341.37): C, 59.81; H, 5.61; N, 20.52. Found: C, 59.67; H, 5.66; N, 20.55.

Benzyl (1-(2-(amino(pyridin-2-yl)methylene)hydrazinyl)-1-oxopropan-2-yl)carbamate (7f'). White microcrystals (79%); mp 178.0–179.0 °C. Two rotamers (1:1): ^{1}H NMR (DMSO-d_6, 300 MHz) δ 10.02 (s, 0.5H), 9.97 (s, 0.5H), 8.57 (s, 1H), 8.08 (t, J = 8.1 Hz, 1H), 7.86 (t, J = 7.8 Hz, 1H), 7.54 (d, J = 6.9 Hz, 0.5H), 7.50-7.21 (m, 6.5H), 6.70 (d, J = 12.6 Hz, 2H), 5.03 (s, 2H), 4.99 (quint, J = 6.9 Hz, 0.5H), 4.18 (quint, J = 6.9 Hz, 0.5H), 1.33 (d, J = 7.2 Hz, 1.5H), 1.28 (d, J = 7.2 Hz, 1.5). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 173.4, 168.3, 155.7, 155.7, 150.5, 150.4, 148.1, 146.4, 142.6, 137.2, 137.1, 136.9, 136.8, 128.3, 127.7, 124.6, 124.5, 120.5, 120.0, 65.4, 65.2, 49.2, 47.1, 18.4, 17.0. Anal. Calcd. for $C_{17}H_{19}N_5O_3$ (341.37): C, 59.81; H, 5.61; N, 20.52. Found: C, 60.16; H, 5.70; N, 20.55.

(*S*)-(9*H*-Fluoren-9-yl)methyl (1-(2-(amino(pyridin-2-yl)methylene)hydrazinyl)-1-oxo-3-(trityloxy)propan-2-yl)carbamate (7g). White microcrystals (79%); mp 224.0–226.0 °C. Two rotamers (3:2): ^{1}H NMR (DMSO-d_6, 300 MHz) δ 10.08 (s, 0.4H), 9.95 (s, 0.6H), 8.58-8.55 (m, 1H), 8.08 (t, J = 9.9 Hz, 1H), 7.90-7.84 (m, 3H), 7.76 (t, J = 6.0 Hz, 2H), 7.62 (d, J = 8.7 Hz, 0.6H) 7.48-7.38 (m, 3H), 7.34-7.29 (m, 2.4H), 6.73-6.67 (m, 2H), 5.03 (dd, J = 8.4, 5.7 Hz, 0.4H), 4.29-4.22 (m, 3H), 3.91 (t, J = 8.7 Hz, 0.6H), 2.23 (sextet, J = 6.9 Hz, 0.4H), 2.00 (sextet, J = 6.9 Hz, 0.6H), 1.00-0.85 (m, 6H). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 172.4, 167.0, 156.4, 150.5, 148.0, 146.2, 143.9, 143.7, 140.6, 136.8, 128.9, 127.6, 127.2, 127.0, 125.4, 124.6, 121.3, 120.5, 120.0, 65.7, 59.6, 46.7, 30.2, 19.3, 18.8. Anal. Calcd. for $C_{26}H_{27}N_5O_3$ (457.54): C, 68.25; H, 5.95; N, 15.31. Found: C, 68.53; H, 6.06; N, 15.23.

(9*H*-Fluoren-9-yl)methyl (2-(2-(amino(pyridin-2-yl)methylene)hydrazinyl)-2-oxoethyl)-carbamate (7h). White microcrystals (75%); mp 195.0–197.0 °C. Two rotamers (1:1): ^{1}H NMR (DMSO-d_6, 300 MHz) δ 10.12 (s, 0.7H), 9.89 (s, 0.3H), 8.57 (s, 1H), 8.09 (d, J = 7.8 Hz, 1H), 7.91-7.77 (m, 3H), 7.75 (d, J = 5.4 Hz, 2H), 7.70-7.57 (m, 0.5H), 7.44-7.23 (m, 5.5H), 6.70-6.62

(m, 2H), 4.30-4.16 (m, 5H), 3.76-3.74 (m, 0.7H), 3.65-3.55 (m, 0.3H). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 170.7, 165.2, 157.3, 151.0, 148.7, 144.6, 143.4, 141.4, 137.5, 128.3, 127.8, 126.0, 125.1, 120.8, 66.3, 47.4, 42.5. Anal. Calcd. for $C_{23}H_{21}N_5O_3$ (415.46): C, 66.49; H, 5.09; N, 16.86. Found: C, 66.54; H, 5.16; N, 16.62.

(*S*)-(9*H*-Fluoren-9-yl)methyl (1-(2-(amino(pyridin-2-yl)methylene)hydrazinyl)-4-methyl-1-oxopentan-2-yl)carbamate (7i). White microcrystals (71%); mp 179.0–181.0°C. Two rotamers (1:1): ^{1}H NMR (DMSO-d_6, 300 MHz): δ 10.01 (s, 1H), 8.58 (s, 1H), 8.11 (t, J = 7.5 Hz, 1H), 8.00-7.60 (m, 6H), 7.58-7.20 (m, 5H), 6.79 (br s, 1H), 6.72 (br s, 1H), 5.22-5.07 (m, 0.5H), 4.27 (s, 3H), 3.70-3.40 (m, 0.5H), 1.88-1.40 (m, 3H), 1.08-0.75 (m, 6H). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 173.6, 168.1, 156.1, 150.6, 148.1, 146.4, 143.9, 143.8, 142.7, 140.7, 136.8, 129.0, 127.6, 127.0, 125.4, 124.5, 120.7, 120.6, 120.9, 120.1, 119.8, 66.4, 65.6, 52.0, 49.0, 46.7, 41.0, 24.5, 24.3, 23.4, 23.0, 21.7, 21.3. Anal. Calcd. for $C_{23}H_{21}N_5O_3$ (471.56): C, 68.77; H, 6.20; N, 14.85. Found: C, 68.53; H, 6.32; N, 14.69.

(S)-(9*H*-fluoren-9-yl)methyl (1-(2-(amino(pyridin-2-yl)methylene)hydrazinyl)-3-(tert-butoxy)-1-oxopropan-2-yl)carbamate (7j). White microcrystals (77%); mp 186.0–188.0 °C. Two rotamers (1:1): ^{1}H NMR (DMSO-d_6, 300 MHz): δ 10.11 (br s, 0.4H), 9.95 (br s, 0.6H), 8.58 (t, J = 4.8 Hz, 1H), 8.10 (d, J = 7.8 Hz, 1H), 7.95-7.80 (m, 3H), 7.75 (d, J = 7.5 Hz, 2H), 7.57-7.30 (m, 6H), 6.74 (br s, 1H), 6.68 (br s, 1H), 4.38-4.16 (m, 3H), 3.72-3.42 (m, 3H), 1.09 (d, J = 9.6 Hz, 9H). ^{13}C NMR (DMSO-d_6, 75 MHz): δ 148.7, 143.3, 140.1, 138.1, 137.5, 129.6, 128.0, 122.1, 120.7, 110.4, 28.0. Anal. Calcd. for $C_{28}H_{31}N_5O_4$ (501.59): C, 67.05; H, 6.23; N, 13.96. Found: C, 67.46; H, 6.35; N, 13.98.

(*S*)-(9*H*-Fluoren-9-yl)methyl (1-(2-(amino(pyridin-2-yl)methylene)hydrazinyl)-1-oxo-3-phenylpropan-2-yl)carbamate (7k). White microcrystals (76%); mp 207.0–209.0 °C. Two

rotamers (1:1): ^{1}H NMR (DMSO-d_6, 300 MHz) δ 10.11 (s, 0.5H), 10.0 (s, 0.5H), 8.59 (d, J = 3.9 Hz, 1H), 8.10 (d, J = 7.5 Hz, 1H), 7.92-7.82 (m, 3H), 7.70-7.65 (m, 2H), 7.53-7.17 (m, 11H), 6.80-6.65 (m, 2H), 5.30-5.17 (m, 0.5H), 4.39 (sextet, J = 4.5 Hz, 0.5H), 4.25-4.10 (m, 3H), 3.14-2.80 (m, 2H). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 172.3, 167.2, 155.8, 150.5, 148.1, 148.1, 146.7, 143.8, 143.7, 143.0, 140.6, 138.6, 138.0, 136.8, 129.3, 129.1, 128.1, 128.1, 127.6, 127.0, 126.3, 125.3, 124.7, 124.5, 120.6, 120.1, 65.7, 65.6, 55.3, 53.2, 46.5, 37.8, 36.5. Anal. Calcd. for $C_{30}H_{27}N_5O_3$ (505.58): C, 71.27; H, 5.38; N, 13.85. Found: C, 71.09; H, 5.34; N, 13.77.

(*S*,*Z*)-(9*H*-Fluoren-9-yl)methyl (1-(2-(amino(pyridin-2-yl)methylene)hydrazinyl)-3-(1*H*-indol-3-yl)-1-oxopropan-2-yl)carbamate (7l). White microcrystals (67%); mp 158.0–160.0 °C. Two rotamers (1:1): ^{1}H NMR (DMSO-d_6, 300 MHz) δ 10.85 (br s, 1H), 10.09 (s, 1H), 8.60 (t, J = 4.2 Hz, 1H), 8.13-8.05 (m , 1H), 7.89-7.87 (m, 2H), 7.86 (d, J = 8.4 Hz, 1H), 7.76-7.59 (m, 3H), 7.50-7.23 (m, 7H), 7.10-6.98 (m, 2H), 6.84 (t, J = 7.5 Hz, 1H), 6.80-6.70 (m, 2H), 5.50-5.25 (m, 0.5H), 4.50-4.40 (m, 0.5H), 4.35-3.93 (m, 3H), 3.30-2.94 (m, 2H). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 173.1, 167.7, 155.9, 150.5, 150.5, 148.1, 148.1, 146.7, 143.8, 143.8, 143.3, 140.7, 136.8, 136.7, 136.1, 127.6, 127.3, 127.1, 125.4, 124.6, 124.6, 123.9, 120.9, 120.8, 120.6, 120.3, 120.1, 118.6, 118.4, 118.2, 118.1, 111.3, 110.6, 110.2, 65.7, 65.6, 64.9, 54.5, 52.1, 46.6, 28.0, 26.9, 15.2. Anal. Calcd. for $C_{32}H_{28}N_6O_3$ (544.62): C, 70.57; H, 5.18; N, 15.43. Found: C, 70.20; H, 5.29; N, 15.71.

***t*-Butyl(2-(2-(amino(pyridin-2-yl)methylene)hydrazinyl)-2-oxoethyl)carbamate (7m)**. White microcrystals (91%); mp 159.0–161.0 °C. Two rotamers (2:1): ^{1}H NMR (DMSO-d_6, 300 MHz) δ 10.05 (s, 0.7H), 9.83 (br s, 0.3H), 8.57 (t, J = 4.5 Hz, 1H), 8.08 (d, J = 8.1 Hz, 1H), 7.90-7.82 (m, 1H), 7.48-7.42 (m, 1H), 6.98 (t, J = 5.4 Hz, 0.3H), 6.75 (t, J = 5.4 Hz, 0.7H), 6.67 -6.63 (m, 2H), 4.09 (d, J = 6.0 Hz, 1.3H), 3.64 (d, J = 6.0 Hz, 0.7H), 1.40 (s, 9H). ^{13}C NMR (DMSO-

d_6, 75 MHz) δ 170.2, 165.0, 155.8, 150.4, 150.3, 147.9, 146.4, 142.6, 136.8, 124.6, 124.4, 120.4, 120.1, 77.7, 42.3, 41.4, 28.1. Anal. Calcd. for $C_{13}H_{19}N_5O_3$ (293.33): C, 53.23; H, 6.53; N, 23.88. Found: C, 53.42; H, 6.51; N, 23.97.

(*S*)-*t*-Butyl(1-(2-(amino(pyridin-2-yl)methylene)hydrazinyl)-1-oxopropan-2-yl)carbamate (7n). White microcrystals (71%); mp 208.0–210.0 °C. Two rotamers (1:1): ^{1}H NMR (DMSO-d_6, 300 MHz) δ 9.95 (s, 0.54H), 9.81 (s, 0.46H), 8.57 (t, J = 4.5 Hz, 1H), 8.10 (t, J = 8.7 Hz, 1H), 7.89-7.82 (m, 1H), 7.48-7.42 (m, 1H), 6.85 (t, J = 4.8 Hz, 0.44H), 6.77 (t, J = 4.8 Hz, 0.56H), 6.66 (s, 0.86H), 6.62 (s,1.14H), 3.23 (sextet, J = 6.6 Hz, 2H), 2.77 (t, J = 6.9 Hz, 1H), 2.36 (t, J = 6.9 Hz, 1H), 1.46-1.31 (m, 9H). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 172.9, 166.7, 156.1, 151.3, 151.2, 148.7, 148.6, 146.6, 142.9, 137.5, 137.4, 125.2, 125.0, 121.1, 120.9, 78.3, 37.5, 36.4, 33.5, 28.9. Anal. Calcd. for $C_{14}H_{21}N_5O_3$ (307.36): C, 54.71; H, 6.89; N, 22.79. Found: C, 54.67; H, 6.95; N, 22.66.

General Procedure for the Synthesis of Aminoacyl-1*H*-1,2,4-triazoles 8a-p, and 8f':

Method A: A solution of acylamidrazone **7** (2.0 mmol) in glacial acetic acid (1.00 mL) was heated to reflux for 1 h. The solution was poured over cold brine (20 mL) and the precipitate was filtered and washed with water. The resulting white solid was suspended in methanol (5 mL) and was filtered and dried *in vacuo* to afford the desired product as white microcrystals. In cases where a precipitate was not formed upon pouring the reaction mixture over cold brine, the mixture was extracted using DCM (20 mL × 3), and the organic layer was evaporated to afford **8**.

For the synthesis of **8m,n**, HOAc (0.2 mL) was added to a solution of **7m,n** (1.0 mmol) in ethanol (10 mL) which was heated to reflux for 2 h. The solvent was evaporated under reduced pressure and the resulting solid was suspended in saturated Na_2CO_3/Ether (1:1, 20 mL). The

suspension was vigorously stirred for 10 min, and the solid was filter and dried to afford the desired product.

For the synthesis of **8o,p**, (1.0 mmol) in HOAc (1 mL) was subjected to microwave irradiation of 100 W for 30 min at 180 °C. The solution was evaporated under reduced pressure, and the resulting solid was stirred in diethyl ether for 20 min at rt. The precipitate was filtered and dried *in vacuo* to afford **8o,p** as a white solid.

Method B: A solution of **7** (2.0 mmol) in glacial acetic acid (1.00 mL) was irradiated (50 W, + cooling) at 130 °C for 5 min. The mixture was worked up as in A.

(*S*)-Benzyl (2-methyl-1-(5-(pyridin-2-yl)-1*H*-1,2,4-triazol-3-yl)propyl)carbamate (8a). White microcrystals (87%); mp 127.0–129.0 °C. ^{1}H NMR (DMSO-d_6, 300 MHz) δ 14.51 (br s, 1H), 8.70 (s, 1H), 8.04 (d, J = 7.5 Hz, 1H), 8.00-7.87 (m, 1H), 7.65-7.45 (m, 2H), 7.40-7.20 (m, 5H), 5.04 (s, 1H), 5.04 (s, 1H), 4.55 (t, J = 8.1 Hz, 1H), 2.25-2.15 (m, 1H), 0.94 (d, J = 6.6 Hz, 3H), 0.82 (d, J = 6.6 Hz, 3H). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 164.2, 156.1, 149.5, 146.0, 137.5, 137.0, 128.3, 127.7, 124.9, 127.6, 121.2, 65.3, 55.0, 31.6, 19.4, 18.6. Anal. Calcd. for $C_{19}H_{21}N_5O_2$ (351.41): C, 64.94; H, 6.02; N, 19.93. Found: C, 65.16; H, 6.13; N, 19.66.

(*S*)-Benzyl (2-phenyl-1-(5-(pyridin-2-yl)-1*H*-1,2,4-triazol-3-yl)ethyl)carbamate (8b). White microcrystals (82%); mp 134.0–136.0 °C. ^{1}H NMR (DMSO-d_6, 300 MHz) δ 14.56 (br s, 1H), 8.70 (br s, 1H), 8.06 (t, J = 7.5 Hz, 1H), 8.10-7.94 (m, 2H), 7.88 (d, J = 8.4 Hz, 1H), 7.53 (t, J = 5.7 Hz, 1H), 7.38-7.14 (m, 9H), 5.10-4.90 (m, 3H), 3.29-3.03 (m, 2H). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 164.7, 155.7, 154.0, 149.5, 146.3, 138.4, 137.8, 137.2, 129.2, 128.2, 128.1, 127.6, 127.4, 126.2, 125.0, 121.2, 65.1, 51.2. Anal. Calcd. for $C_{23}H_{21}N_5O_2$ (399.46): C, 69.16; H, 5.30; N, 17.53. Found: C, 69.49; H, 5.26; N, 17.44.

Benzyl (5-(pyridin-2-yl)-1*H*-1,2,4-triazol-3-yl)methylcarbamate (8c): White microcrystals (90%); mp 191.0–193.0 °C. ^{1}H NMR (DMSO-d_6, 300 MHz) δ 14.57 (br s, 1H), 8.69 (d, J = 4.2 Hz, 1H), 8.08 (m, 2H), 7.83 (t, J = 6.0 Hz, 1H), 7.51 (t, J = 5.7 Hz, 1H), 7.42-7.25 (m, 5H), 5.07 (s, 2H), 4.33 (d, J = 5.7 Hz, 2H). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 161.9, 156.3, 154.1, 149.5, 146.2, 137.8, 137.2, 128.3, 127.7, 125.0, 121.2, 65.4, 38.4. Anal. Calcd. for $C_{16}H_{15}N_5O_2$ (309.33): C, 62.13; H, 4.89; N, 22.64. Found: C, 62.12; H, 4.84; N, 22.81.

Benzyl ((1*S*,2*S*)-2-methyl-1-(5-(pyridin-2-yl)-1*H*-1,2,4-triazol-3-yl)butyl)carbamate (8d). White microcrystals (81%); mp 109.0–111.0 °C. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 11.90-10.95 (br, 1H), 8.69 (d, J = 4.5 Hz, 1H), 8.21 (d, J = 7.8 Hz, 1H), 7.79 (t, J = 7.5 Hz, 1H), 7.35-7.26 (m, 5H), 6.54 (d, J = 9.3 Hz, 1H), 5.19-5.00 (m, 3H), 2.10-1.95 (m, 1H), 1.65-152 (m, 1H), 1.22-1.10 (m, 1H), 0.94-0.85 (m, 6H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 174.2, 162.6, 156.4, 155.9, 149.2, 146.7, 137.8, 136.4, 128.4, 128.0, 124.8, 122.2, 66.9, 53.9, 39.4, 25.0, 15.4, 11.4. Anal. Calcd. for $C_{20}H_{23}N_5O_2$ (365.43): C, 65.73; H, 6.34; N, 19.16. Found: C, 65.74; H, 6.38; N, 19.14.

(*S*)-Benzyl (2-(1*H*-indol-3-yl)-1-(5-(pyridin-2-yl)-1*H*-1,2,4-triazol-3-yl)ethyl)carbamate (8e). White microcrystals (72%); mp 88.0–90.0 °C. ^{1}H NMR (DMSO-d_6, 300 MHz) δ 13.42 (br s, 1H), 8.45 (s, 2H), 8.03 (d, J = 7.8 Hz,1H), 7.61 (t, J = 7.5 Hz, 1H), 7.38 (d, J = 7.5 Hz, 1H), 7.28-6.78 (m, 9H), 6.58 (s, 1H), 6.36 (d, J = 7.2 Hz , 1H), 5.50-5.30 (m, 1H), 5.03 (d, J = 12.0 Hz, 1H), 4.94 (d, J = 12.0 Hz, 1H), 3.50-3.20 (m, 2H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 162.5, 156.4, 156.3, 149.4, 147.1, 137.5, 136.5, 136.0, 128.5, 128.1, 127.8, 124.7, 123.5, 122.1, 121.7, 119.3, 118.6, 111.2, 110.2, 66.9, 50.3, 30.8. Anal. Calcd. for $C_{25}H_{22}N_6O_2.⅓H_2O$ (438.49): C, 67.55; H, 5.14; N, 18.91. Found: C, 67.81; H, 5.08; N, 18.38.

(*S*)-Benzyl 1-(3-(pyridin-2-yl)-1*H*-1,2,4-triazol-5-yl)ethylcarbamate (8f). White microcrystals (86%); mp 156.0–158.0 °C. ^{1}H NMR (DMSO-d_6, 300 MHz) δ 14.53 (br s, 1H),

8.68 (d, J = 4.2 Hz, 1H), 8.04 (d, J = 7.8 Hz, 1H), 7.96 (t, J = 7.5 Hz, 1H), 7.82 (br s, 1H), 7.49 (t, J = 5.7 Hz, 1H), 7.42-7.22 (m, 5H), 5.04 (s, 2H), 4.85 (quin, J = 7.2 Hz, 1H), 1.47 (d, J = 7.2 Hz, 3H). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 155.6, 149.5, 137.6, 137.1, 128.3, 127.7, 124.6, 121.2, 65.3, 44.8, 20.1. Anal. Calcd. for $C_{17}H_{17}N_5O_2$ (323.36): C, 63.15; H, 5.30; N, 21.66. Found: C, 63.10; H, 5.32; N, 21.82.

Benzyl (1-(5-(pyridin-2-yl)-1*H*-1,2,4-triazol-3-yl)ethyl)carbamate (8f'). White microcrystals (85%); mp 160.0–162.0 °C. ^{1}H NMR (DMSO-d_6, 300 MHz) δ 8.65 (d, J = 4.5 Hz, 1H), 8.10-8.00 (m, 2H), 7.91 (t, J = 6.9 Hz, 1H), 7.50-7.20 (m, 7H), 5.15-4.98 (m, 2H), 4.87 (quint, J = 7.5 Hz, 1H), 1.46 (d, J = 6.9 Hz, 3H). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 156.9, 149.5, 147.2, 143.9, 142.6, 139.4, 137.4, 137.4, 135.4, 133.2, 129.5, 128.9, 127.7, 127.3, 124.5, 124.3, 123.9, 122.2, 121.4, 121.2, 121.2, 120.7, 120.0, 109.8, 47.2, 46.0, 24.3, 22.7, 22.2, 21.8. Anal. Calcd. for $C_{17}H_{17}N_5O_2$ (323.36): C, 63.15; H, 5.30; N, 21.66. Found: C, 63.50; H, 5.28; N, 21.84.

(*S*)-(9*H*-Fluoren-9-yl)methyl (2-methyl-1-(5-(pyridin-2-yl)-1*H*-1,2,4-triazol-3-yl)propyl)carbamate (8g). White microcrystals (90%); mp 169.0–171.0 °C. ^{1}H NMR (DMSO-d_6, 300 MHz) δ 14.23 (br s, 1H), 8.69 (d, J = 4.2 Hz, 1H), 8.05 (d, J = 7.8 Hz, 1H), 7.96 (t, J = 7.8 Hz, 1H), 7.87 (d, J = 7.7 Hz, 3H), 7.75 (t, J = 8.7 Hz, 2H), 7.49 (t, J = 5.7 Hz, 1H), 7.45-7.35 (m, 2H), 7.33-7.25 (m, 2H), 4.54 (t, J = 8.1 Hz, 1H), 4.33-4.15 (m, 3H), 2.23 (sextet, J = 7.2 Hz, 1H), 0.97 (d, J = 6.6 Hz, 3H), 0.82 (d, J = 6.6 Hz, 3H).^{13}C NMR (DMSO-d_6, 75 MHz) δ 156.9, 146.5, 147.2, 143.9, 124.6, 139.4, 137.4, 137.4, 125.4, 133.2, 129.5, 128.9, 127.7, 124.5, 124.3, 123.9, 122.2, 121.4, 121.2, 121.2, 120.7, 120.0, 109.8, 47.2, 46.0, 24.3, 22.7, 22.2, 21.8. Anal. Calcd. for $C_{26}H_{25}N_5O_2$ (439.52): C, 71.05; H, 5.73; N, 15.93. Found: C, 70.73; H, 5.70; N, 15.64.

(9*H*-Fluoren-9-yl)methyl (3-(pyridin-2-yl)-1*H*-1,2,4-triazol-5-yl)methylcarbamate (8h). White microcrystals (89%); mp 203.0–205.0 °C. ^{1}H NMR (DMSO-d_6, 300 MHz) δ 14.56 (br s,

1H), 8.69 (d, J = 4.2 Hz, 1H), 8.05 (d, J = 7.8 Hz, 1H), 8.00-7.91 (m, 2H), 7.88 (d, J = 7.5 Hz, 2H), 7.74 (d, J = 7.2 Hz, 2H), 7.50 (t, J = 5.7 Hz, 1H), 7.40 (t, J = 7.2 Hz, 2H), 7.31 (t, J = 7.2 Hz, 2H), 4.44-4.19 (m, 5H). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 161.9, 156.3, 154.1, 149.5, 146.2, 143.9, 140.7, 137.8, 127.6, 127.1, 125.3, 125.0, 121.2, 120.1, 65.7, 46.7, 38.4. Anal. Calcd. for $C_{23}H_{19}N_5O_2$ (397.44): C, 69.51; H, 4.82; N, 17.62. Found: C, 69.20; H, 4.65; N, 17.62.

(*S*)-(9*H*-Fluoren-9-yl)methyl (3-methyl-1-(5-(pyridin-2-yl)-1*H*-1,2,4-triazol-3-yl)butyl)carbamate (8i). White microcrystals (80%); mp 109.0–111.0 °C. ^{1}H NMR (DMSO-d_6, 300 MHz) δ 9.97 (br s, 1H), 8.58 (br s, 1H), 8.07 (t, J = 7.8 Hz, 1H), 7.88-7.25 (m, 9H), 6.76 (s, 1H), 6.69 (s, 1H), 5.18-5.00 (m, 1H), 4.38-4.10 (m, 3H), 1.82-1.40 (m, 3H), 1.00-0.80 (m, 6H). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 155.8, 149.5, 143.9, 140.7, 127.6, 127.0, 125.3, 121.2, 120.1, 65.5, 46.7, 24.2, 22.8, 21.7. Anal. Calcd. for $C_{27}H_{27}N_5O_2$ (439.52): C, 71.50; H, 6.00; N, 15.44. Found: C, 71.13; H, 6.23; N, 15.32.

(*R*)-(9*H*-Fluoren-9-yl)methyl (2-(tert-butoxy)-1-(5-(pyridin-2-yl)-1*H*-1,2,4-triazol-3-yl)ethyl)carbamate (8j). White microcrystals (84%); mp 108.0–111.0 °C. ^{1}H NMR (DMSO-d_6, 300 MHz) δ 8.72 (s, 1H), 8.19 (d, J = 7.5 Hz, 1H), 7.81 (t, J = 7.5 Hz, 1H), 7.73 (d, J = 7.5 Hz, 2H), 7.60 (t, J = 7.5 Hz, 2H, 7.41-7.22 (m, 6H), 6.27 (d, J = 6.6 Hz, 1H), 5.30-5.20 (m, 1H), 4.49 (d, J = 6.6 Hz, 2H), 4.23 (t, J = 6.6 Hz, 1H), 3.98-3.88 (m, 1H), 3.83-3.77 (m, 1H), 1.13 (s, 9H). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 156.4, 147.3, 144.0, 141.4, 137.4, 127.8, 127.2, 125.3, 124.8, 121.8, 120.1, 73.9, 67.3, 63.4, 50.3, 47.4, 27.6. Anal. Calcd. for $C_{28}H_{29}N_5O_3$ (483.58): C, 69.55; H, 6.04; N, 14.48. Found: C, 69.38; H, 6.26; N, 14.02.

(*S*)-(9*H*-Fluoren-9-yl)methyl (2-phenyl-1-(5-(pyridin-2-yl)-1*H*-1,2,4-triazol-3-yl)ethyl)carbamate (8k). White microcrystals (90%); mp 178.0–180.0 °C. ^{1}H NMR (DMSO-d_6, 300 MHz) δ 14.55 (br s, 1H), 8.72 (s, 1H), 8.08 (d, J = 7.2 Hz, 1H), 8.00 (t, J = 7.8 Hz, 1.5H),

7.88 (d, *J* = 7.5 Hz, 2H), 7.67 (d, *J* = 7.2 Hz, 2H), 7.54 (t, *J* = 5.1 Hz, 1H), 7.43-7.17 (m, 9.5H), 7.32-7.15 (m, 7H), 5.05-4.90 (m, 1H), 4.30-4.10 (m, 3H), 3.39-3.05 (m, 2H). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 155.6, 149.5, 143.8, 140.6, 137.6, 129.2, 128.1, 127.6, 127.0, 126.2, 125.3, 121.2, 120.0, 65.6, 46.6. Anal. Calcd. For $C_{30}H_{25}N_5O_2$ (487.57): C, 73.90; H, 5.28; N, 14.36. Found: C, 73.58; H, 5.28; N, 14.15.

(*S*)-(9*H*-Fluoren-9-yl)methyl (2-(1*H*-indol-3-yl)-1-(3-(pyridin-2-yl)-1*H*-1,2,4-triazol-5-yl)ethyl)carbamate hydrate (8l). White microcrystals (81%); mp 133.0–135.0 °C. ^{1}H NMR (DMSO-d_6, 300 MHz): δ 10.83 (s, 1H), 8.69 (d, *J* = 4.5 Hz, 1H), 8.09 (d, *J* = 4.8 Hz, 1H), 8.04-7.92 (m, 2H), 7.87 (d, *J* = 7.5 Hz, 2H), 7.69 (d, *J* = 7.8 Hz, 2H), 7.61 (d, *J* = 7.5 Hz, 1H), 7.46-7.22 (m, 5H), 7.10 (d, *J* = 2.1 Hz, 1H), 7.05 (t, *J* = 7.2 Hz, 1H), 6.96 (t, *J* = 7.5 Hz, 1H), 5.03 (q, *J* = 8.4 Hz, 1H), 4.25-4.04 (m, 3H), 3.40 (dd, *J* = 14.7, 6.3 Hz, 1H), 3.24 (dd, *J* = 14.7 Hz, 6.3 Hz, 1H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 162.5, 156.6, 156.3, 149.3, 147.0, 144.0, 143.8, 141.3, 137.7, 136.0, 127.7, 127.1, 125.2, 124.8, 123.5, 122.1, 121.9, 120.0, 119.5, 118.6, 11.3, 110.3, 67.1, 50.3, 47.2, 30.7. Anal. Calcd. for $C_{32}H_{26}N_6O_2.H_2O$ (544.62): C, 70.57; H, 5.18; N, 15.43. Found: C, 70.20; H, 5.29; N, 15.71.

***t*-Butyl ((5-(pyridin-2-yl)-1*H*-1,2,4-triazol-3-yl)methyl)carbamate (8m)**. White microcrystals (79%); mp 194.0–196.0 °C. ^{1}H NMR (DMSO-d_6, 300 MHz): δ 14.31 (br s, 1H), 8.67 (d, *J* = 4.5 Hz, 1H), 8.04 (d, *J* = 7.2 Hz, 1H), 8.00 (t, *J* = 7.8 Hz, 1H), 7.48 (t, *J* = 6.0 Hz, 1H), 7.34 (t, *J* = 5.7 Hz, 1H), 4.24 (d, *J* = 5.7 Hz, 2H), 1.39 (s, 9H). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 170.2, 165.0, 155.8, 150.5, 150.3, 148.0, 147.9, 146.7, 142.6, 136.8, 124.6, 124.4, 120.4, 120.1, 78.0, 77.8, 42.4, 41.5, 28.2. Anal. Calcd. for $C_{13}H_{17}N_5O_2$ (275.31): C, 56.72; H, 6.22; N, 25.44. Found: C, 56.59; H, 6.33; N, 24.27.

(*S*)-*t*-Butyl (1-(5-(pyridin-2-yl)-1*H*-1,2,4-triazol-3-yl)ethyl)carbamate (8n). White microcrystals. (83%), mp 162.0.–164.0 °C. ^{1}H NMR (DMSO-d_6, 300 MHz) δ 8.74 (d, J = 3.9 Hz, 1H), 8.20 (d, J = 8.1 Hz, 1H), 7.86 (t, J = 7.5 Hz, 1H), 7.39 (t, J = 6.9 Hz, 1H), 5.60 (d, J = 7.5 Hz, 1H), 5.09 (br s, 1H), 1.62 (d, J = 6.9 Hz, 3H), 1.45 (s, 9H). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 155.5, 149.6, 137.6, 124.8, 122.0, 44.9, 28.5, 21.4. HRMS Calcd. for $C_{13}H_{17}N_5O_2$ $[M+H]^+$: 290.1612. Found $[M+H]^+$: 290.1604

***N*-((3-(Pyridin-2-yl)-1*H*-1,2,4-triazol-5-yl)methyl)acetamide (8o).** White solid (91%); mp178.0–180.0 °C. ^{1}H NMR (DMSO-d_6, 300 MHz) δ 14.65-14.20 (m, 1H), 8.66 (s, 1H), 8.43 (br s, 1H), 8.04 (d, J = 7.8 Hz, 1H), 7.95 (s, 1H), 7.47 (s, 1H), 4.38 (s, 2H), 1.88 (s, 3H). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 169.2, 161.7, 154.2, 149.5, 146.0, 137.8, 125.0, 121.2, 36.5, 22.6. Anal. Calcd. for $C_{10}H_{11}N_5O$ (217.23): C, 51.06; H, 4.71; N, 29.77. Found: C, 51.01; H, 4.62; N, 29.50.

(*S*)-*N*-(1-(3-(Pyridin-2-yl)-1*H*-1,2,4-triazol-5-yl)ethyl)acetamide (8p). White solid (90%); mp 195.0–196.0 °C. ^{1}H NMR (DMSO-d_6, 300 MHz) δ 8.67 (dd, J = 3.9, 0.9 Hz, 1H), 8.37 (d, J = 8.1 Hz, 1H), 8.05 (d, J = 8.1 Hz, 1H), 7.94 (t, J = 7.5 Hz, 1H), 7.47 (d, 6.3 Hz, 1H), 5.08 (quint, J = 7.2 Hz, 1H), 1.86 (s, 3H), 1.44 (d, J = 7.2 Hz, 3H). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 168.6, 149.5, 137.5, 127.6, 121.2, 42.4, 22.6, 20.2. Anal. Calcd. for $C_{11}H_{13}N_5O$ (231.26): C, 57.06; H, 5.67; N, 30.28. Found: C, 57.06; H, 5.67; N, 30.25.

General Procedure for the Synthesis of Amino acyl hydrazide 9a-g, 9a', 9f': Hydrazine hydrate (1.0 mmol) was added to a solution of *N*-protected amino acylbenzotriazole **5** (1.0 mmol) in THF (10 mL). The solution was stirred for 15 min at room temperature. The solvent was removed under reduced pressure and the resulting crude mixture was dissolved in ethyl acetate and washed with water (20 mL x 2). The organic layer was dried over anhydrous sodium sulfate

and was dried under reduced pressure. The resulting solid was suspended in ether and the insoluble solid was collected on a Buchner funnel to afford the desired product as white microcrystals.

(*S*)-Benzyl (1-hydrazinyl-3-methyl-1-oxobutan-2-yl)carbamate (9a). White microcrystals (86%); mp 163.0–165.0 °C (Lit. mp 178.0 °C)[25]. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7 7.79 (br s, 1H), 7.34-7.26 (m, 5H), 5.56 (d, J = 9.0 Hz, 1H), 5.15-5.05 (m, 2H), 3.94 (dd, J = 9.0, 7.2 Hz, 1H), 2.13-2.04 (m, 1H), 0.95-0.92 (m, 6H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 172.2, 156.1, 136.1, 128.7, 128.4, 128.2, 64.3, 59.5, 31.0, 19.3, 18.2. Anal. Calcd. for $C_{13}H_{19}N_3O_3$ (265.31): C, 58.85; H, 7.22; N, 15.84. Found: C, 59.00; H, 7.47; N, 15.64.

Benzyl (1-hydrazinyl-3-methyl-1-oxobutan-2-yl)carbamate (1:1) (9a'). White microcrystals (78%); mp 126.0–128.0 °C. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.40-8.20 (br s, 1H), 7.40-7.26 (m, 5H), 5.84 (d, J = 9.0 Hz, 1H), 5.11 (d, J = 12.0 Hz, 1H), 5.04 (d, J = 12.3 Hz, 1H), 4.15-3.85 (m, 3H), 2.05 (sextet, J = 6.0 Hz, 1H), 0.91 (d, J = 6.6 Hz, 6H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 172.3, 156.7, 136.2, 128.6, 128.3, 128.1, 67.2, 59.4, 31.0, 19.3, 18.2. Anal. Calcd. for $C_{13}H_{19}N_3O_3$ (265.31): C, 58.85; H, 7.22; N, 15.84. Found: C, 58.61; H, 6.97; N, 16.01.

(*S*)-Benzyl (1-hydrazinyl-1-oxo-3-phenylpropan-2-yl)carbamate (9b). White microcrystals (94%); mp 153.0–155.0 °C (Lit. mp 164.0–165.0 °C)[23]. ^{1}H NMR (DMSO-d_6, 300 MHz) δ 9.27 (s, 1H), 7.56 (d, J = 8.7 Hz, 1H), 7.36-7.10 (m, 10H), 4.94 (s, 2H), 4.30-4.18 (m, 3H), 2.97-2.75 (m, 2H). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 170.8, 155.7, 138.1, 137.0, 129.2, 128.3, 128.1, 127.7, 127.5, 126.3, 65.2, 55.0, 37.8. Anal. Calcd. for $C_{10}H_{13}N_3O_3$ (313.36): C, 65.16; H, 6.11; N, 13.41. Found: C, 64.88; H, 6.12; N, 13.32.

Benzyl (2-hydrazinyl-2-oxoethyl)carbamate (9c). White microcrystals (86%); mp 163.0–165.0 °C (Lit. mp 115.0 °C)[26]. ^{1}H NMR (DMSO-d_6, 300 MHz) δ 9.07 (s, 1H), 7.45 (t, J = 6.3

Hz, 1H), 7.37-7.28 (m, 5H), 5.02 (s, 2H), 4.20 (s, 2H), 3.58 (d, J = 6.3 Hz, 2H). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 168.5, 156.4, 137.0, 128.3, 127.7, 65.5, 42.3. Anal. Calcd. for $C_{10}H_{13}N_3O_3$ (313.36): C, 53.81; H, 5.87; N, 18.82. Found: C, 53.52; H, 5.77; N, 18.68.

(*S*)-Benzyl (1-hydrazinyl-4-methyl-1-oxopentan-2-yl)carbamate (9d). White microcrystals (87%); mp 159.0–161.0 °C. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.85 (br s, 1H), 7.40-7.26 (m, 5H), 5.58 (d, J = 8.7 Hz, 1H), 5.14-5.03 (m, 2H), 3.99 (dd, J = 9.0, 7.2 Hz, 1H), 1.82-1.78 (m, 1H), 1.60-1.45 (m, 1H), 1.16-1.06 (m, 1H), 0.92-0.85 (m, 6H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 172.2, 156.5, 136.2, 128.7, 128.4, 128.2, 67.3, 58.6, 37.2, 24.9, 15.6, 11.3. Anal. Calcd. for $C_{14}H_{21}N_3O_3$ (279.34): C, 60.20; H, 7.58; N, 15.04. Found: C, 60.37; H, 7.90; N, 14.89.

(*S*)-Benzyl (1-hydrazinyl-3-(1*H*-indol-3-yl)-1-oxopropan-2-yl)carbamate (9e). White microcrystals (90%); mp 163.0–165.0 °C. ^{1}H NMR (DMSO-d_6, 300 MHz) δ 10.81 (br s, 1H), 9.26 (br s, 1H), 7.62 (d, J = 7.5 Hz, 1H), 7.43 (d, J = 8.4 Hz, 1H), 7.34-7.24 (m, 6H), 7.14 (d, J = 1.5 Hz, 1H), 7.06 (t, J = 7.2 Hz, 1H), 6.97 (t, J = 7.2 Hz, 1H), 5.00-4.89 (m, 2H), 4.25-4.21 (m, 3H), 3.04 (dd, J = 14.4, 5.1 Hz, 1H), 2.91 (dd, J = 14.6, 9.5 Hz, 2H). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 171.1, 155.7, 137.0, 136.0, 128.3, 127.7, 127.5, 127.2, 123.8, 120.8, 118.5, 118.2, 111.3, 110.1, 65.2, 54.2, 28.1. Anal. Calcd. for $C_{19}H_{20}N_4O_3$ (352.40): C, 64.76; H, 5.72; N, 15.90. Found: C, 64.94; H, 5.89; N, 15.95.

(*S*)-Benzyl (1-hydrazinyl-1-oxopropan-2-yl)carbamate (9f). White microcrystals (78%); mp 111.0–113.0 °C (Lit. mp 138.5 °C)[26]. ^{1}H NMR (DMSO-d_6, 300 MHz) δ 7.79 (br s, 1H), 7.40-7.20 (m, 5H), 5.43 (d, J = 7.2 Hz, 1H), 5.15-5.04 (m, 2H), 4.30-4.15 (m, 1H), 1.38 (d, J = 7.2 Hz, 3H). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 173.1, 156.1, 136.1, 128.7, 128.4, 128.3, 67.4, 49.4, 18.5. Anal. Calcd. for $C_{14}H_{21}N_3O_3$ (237.26): C, 55.69; H, 6.37; N, 17.71. Found: C, 56.01; H, 6.38; N, 17.90.

(*R* and *S*)-Benzyl (1-hydrazinyl-1-oxopropan-2-yl)carbamate (1:1) (9f´). White microcrystals (75%); mp 116.0–118.0 °C (Lit. mp 119.0-120.0 °C)[25]. ^{1}H NMR (DMSO-d_6, 300 MHz) δ 9.08 (s, 1H), 7.44-7.30 (m, 6H), 5.00 (s, 2H), 4.19 (s, 2H), 4.00 (quint, J = 7.5 Hz, 1H), 1.18 (d, J = 7.2 Hz, 3H). ^{13}C NMR (DMSO-d_6, 75 MHz): δ 171.8, 155.5, 137.0, 128.3, 127.7, 65.3, 48.8, 18.4. Anal. Calcd. for $C_{14}H_{21}N_3O_3$ (237.26): C, 55.69; H, 6.37; N, 17.71. Found: C, 55.83; H, 6.51; N, 17.65.

(*S*)-(9*H*-Fluoren-9-yl)methyl (1-hydrazinyl-4-methyl-1-oxopentan-2-yl)carbamate (9g). White microcrystals (75%); mp 165.0–167.0 °C. ^{1}H NMR (DMSO-d_6, 300 MHz) δ 9.13 (br s, 1H), 7.87 (d, J = 7.5 Hz, 2H), 7.71 (d, J = 7.5 Hz, 2H), 7.50-7.27 (m, 5H), 4.28-4.15 (m, 5H), 4.03-3.95 (m, 1H), 1.57-1.30 (m, 3H), 1.00-0.75 (m, 6H). ^{13}C NMR (DMSO-d_6, 75 MHz): δ 171.5, 155.8, 143.9, 143.8, 140.7, 127.6, 127.0, 125.3, 120.1, 65.5, 51.7, 46.7, 41.0, 24.2, 22.9, 21.6. Anal. Calcd. for $C_{21}H_{25}N_3O_3$ (367.45): C, 68.64; H, 6.86; N, 11.44. Found: C, 68.94; H, 7.10; N, 11.62.

General Procedure for the Synthesis of 3,6-Disubstituted-1,2,4-triazine 11:

Method A: *N*-Cbz-aminoacylhydrazide **9** (2.0 mmol) was heated in a mixture of ethanol (2.00 mL) and glacial AcOH (0.50 mL) at 60 °C, until a clear solution resulted. α-bromo ketones **10** (1.0 mmol) was then added to the solution which was irradiated by μwave (50 W, 95 °C) for 1 h. NaOAc (2.0 mmol) was added to the brownish solution which was irradiated under the same conditions for 1h. The solution was dried and the crude solid was dissolved in DCM (20 mL), and was washed with brine (2 × 20 mL), sat. Na_2CO_3 (3 × 20 mL) and water (20 mL). The elution was dried over anhydrous Na_2SO_4 and was evaporated under reduced pressure. The resulting solid was purified using column chromatography (hexanes: ethyl acetate, 3:1) to afford the title product **11**.

Method B: *N*-Cbz-aminoacylhydrazide **9** (2.0 mmol) was heated in a mixture of ethanol (3.00 mL) and glacial AcOH (1.00 mL) at 60 °C, until a clear solution resulted. NaOAc (1.1 mmol) and α-bromo ketones **10** (1.0 mmol) were added, and the mixture was heated under reflux for 7 h. The solution was poured onto ice/H_2O and neutralized with $NaHCO_3$. The solution was extracted with DCM (3 x 20 mL), the organic layer dried over anhydrous Na_2SO_4 and the solvent evaporated under reduced pressure. The resulting crude solid was purified using column chromatography (hexanes: ethyl acetate, 3:1) to afford the title product **11**.

(*S*)-Benzyl (1-(6-(4-bromophenyl)-1,2,4-triazin-3-yl)ethyl)carbamate (11a). Yellow microcrystals (61%); mp 96.0–98.0 °C. ^{1}H NMR (DMSO-d_6, 300 MHz) δ 9.43 (s, 1H), 8.18 (d, J = 7.5 Hz, 2H), 8.04 (d, J = 8.4 Hz, 1H), 7.82 (d, J = 8.1 Hz, 2H), 7.44-7.26 (m, 5H), 5.05 (d, J = 12.3 Hz, 1H), 4.99 (d, J = 12.6 Hz, 1H), 4.76 (t, J = 7.5 Hz, 1H), 2.35-2.18 (m, 1H), 0.99 (d, J = 6.3 Hz, 3H), 0.81 (d, J = 6.3 Hz, 3H). ^{13}C NMR (DMSO-d_6, 75 MHz): δ 167.6, 156.3, 154.5, 147.7, 137.0, 132.3, 132.2, 128.9, 128.3, 127.8, 127.7, 124.8, 65.4, 61.6, 31.8, 19.4, 18.7. Anal. Calcd. for $C_{21}H_{21}BrN_4O_2$ (441.33): C, 57.15; H, 4.80; N, 12.70. Found: C, 57.44; H, 4.76; N, 12.15.

(*S*)-Benzyl (1-(6-(4-bromophenyl)-1,2,4-triazin-3-yl)-2-phenylethyl)carbamate hydrate (11b). Yellow microcrystals (40%); mp 141.0–143.0 °C. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.90 (s, 1H), 7.96 (d, J = 8.7 Hz, 2H), 7.70 (d, J = 8.7 Hz, 2H), 7.40-7.14 (m, 8H), 7.01 (br s, 2H), 5.96 (br, 1H), 5.88 (d, J = 8.1 Hz, 1H), 5.65-5.50 (m, 1H), 5.12 (d, J = 12.3 Hz, 1H), 5.06 (d, J = 12.6 Hz, 1H), 3.42 (dd, J = 13.7, 5.9 Hz, 1H), 3.30 (dd, J = 13.7, 6.8 Hz, 1H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 166.9, 155.9, 155.2, 146.2, 136.2, 136.1, 132.9, 131.9, 128.7, 128.5, 128.3, 127.1, 126.3, 67.2, 56.5, 41.5. Anal. Calcd. for $C_{25}H_{21}BrN_4O_2.H_2O$ (507.39): C, 59.18; H, 4.57; N, 11.04. Found: C, 59.34; H, 4.03; N, 11.01.

Benzyl (6-(4-bromophenyl)-1,2,4-triazin-3-yl)methylcarbamate (11c). White microcrystals (43%); mp 132.0–134.0 °C. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.95 (s, 1H), 7.95 (d, J = 8.4 Hz, 2H), 7.70 (d, J = 8.4 Hz, 2H), 7.50-7.26 (m, 5H), 5.96 (br, 1H), 5.17 (s, 2H), 4.89 (d, J = 5.7 Hz, 2H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 164.4, 156.7, 155.5, 146.6, 136.5, 132.9, 131.9, 128.7, 128.4, 128.3, 126.2, 67.3, 45.4. Anal. Calcd. for $C_{18}H_{15}BrN_4O_2$ (399.25): C, 54.15; H, 3.79; N, 14.03. Found: C, 54.23; H, 3.69; N, 13.96.

(*S*)-Benzyl (1-(6-(4-bromophenyl)-1,2,4-triazin-3-yl)ethyl)carbamate (11d). Yellow microcrystals (28%); mp 139.0–140.0 °C. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.94 (s, 1H), 7.96 (dt, J = 8.7, 2.1 Hz, 2H), 7.70 (dt, J = 8.7, 2.1 Hz, 2H), 7.45-7.25 (m, 5H), 5.99 (d, J = 4.8 Hz, 1H), 5.33 (quint, J = 7.2 Hz, 1H), 5.16 (d, J = 12.3 Hz, 1H), 5.09 (d, J = 12.3 Hz, 1H), 1.65 (d, J = 7.5 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 168.2, 155.4, 155.3, 146.6, 136.5, 132.8, 132.0, 128.6, 128.4, 128.2, 126.1, 67.0, 51.7, 21.9. Anal. Calcd. for $C_{19}H_{17}BrN_4O_2$ (413.28): C, 52.22; H, 4.15; N, 13.56. Found: C, 55.20; H, 4.06; N, 13.16.

Benzyl (1-(6-(4-bromophenyl)-1,2,4-triazin-3-yl)ethyl)carbamate (11d´). Yellow microcrystals (30%); mp 57.0–59.0 °C. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.95 (s, 1H), 7.86 (d, J = 8.4 Hz, 2H), 7.71 (d, J = 8.4 Hz, 2H), 7.40-7.26 (m, 5H), 5.94 (d, J = 7.5 Hz, 1H), 5.33 (quint, J = 7.2 Hz, 1H), 5.13 (dd, J = 19.8, 12.3 Hz, 2H), 1.65 (d, J = 6.9 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 168.2, 155.3, 146.6, 136.5, 132.8, 132.0, 128.6, 128.4, 128.3, 126.1, 67.1, 51.7, 22.0. Anal. Calcd. for $C_{19}H_{17}BrN_4O_2$ (413.28): C, 55.22; H, 4.15; N, 13.56. Found: C, 55.30; H, 4.03; N, 13.40.

General Procedure for the Synthesis of 3,5,6-Trisubstituted-1,2,4-triazine 13: A solution of *N*-Cbz-aminoacylhydrazide **9** (1.0 mmol), acenaphthenequinone **12** (1.0 mmol), and ammonium acetate (2.0 mmol) was subjected to microwave irradiation of 100 W at 180 °C for

10 min. The black solution was poured onto ice/water and the aqueous solution was washed with DCM (20 mL × 3). The organic layer was dried over sodium sulfate (anhyd.) and was evaporated under reduced pressure. The resulting crude solid was purified using column chromatography (hexanes: ethyl acetate; 3:1) to afford the desired product.

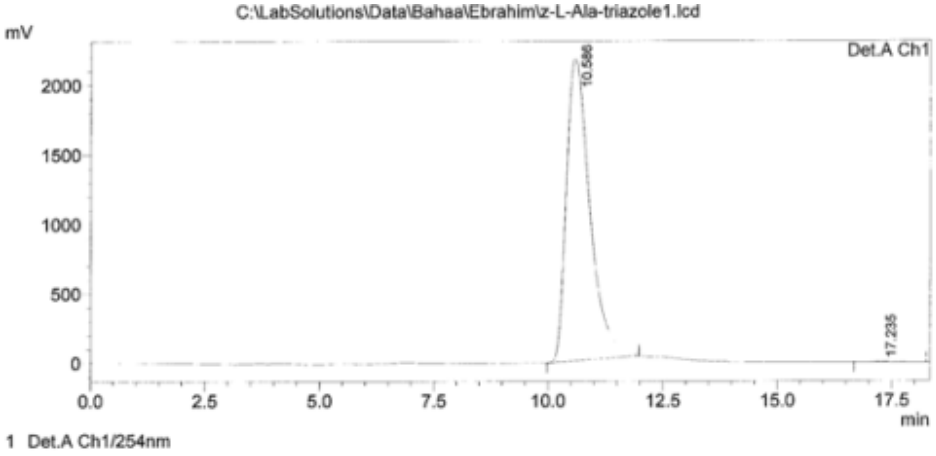
(*S*)-Benzyl (1-(acenaphtho[1,2-*e*][1,2,4]triazin-9-yl)-2-methylpropyl)carbamate (13a). Orange microcrystals (68%); mp 108.0–110.0 °C. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.43 (d, J = 6.9 Hz, 1H), 8.39 (d, J = 7.2 Hz, 1H), 8.19 (d, J = 8.1 Hz, 1H), 8.11 (d, J = 8.4 Hz, 1H), 7.83 (dt, J = 8.1, 1.2 Hz, 2H), 7.42-7.26 (m, 5H), 6.17 (d, J = 9.3 Hz, 1H), 5.25 (dd, J = 9.0, 5.4 Hz, 1H), 5.17 (s, 2H), 1.05 (d, J = 6.6 Hz, 3H), 0.98 (d, J = 6.6 Hz, 3H).^{13}C NMR ($CDCl_3$, 75 MHz) δ 165.4, 157.4, 156.6, 155.7, 136.7, 134.3, 132.6, 130.5, 130.0, 129.6, 128.9, 128.7, 128.3, 125.5, 123.9, 67.1, 61.1, 34.2, 19.8, 17.7. Anal. Calcd. for $C_{25}H_{22}N_4O_2$ (410.48): C, 73.15; H, 5.40; N, 13.65. Found: C, 73.46; H, 5.38; N, 13.52.

Benzyl (1-(acenaphtho[1,2-*e*][1,2,4]triazin-9-yl)-2-methylpropyl)carbamate (13a'). Orange oil (64%). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.45 (d, J = 7.2 Hz, 1H), 8.41 (d, J = 6.9 Hz, 1H), 8.21 (d, J = 8.4 Hz, 1H), 8.13 (d, J = 8.1 Hz, 1H), 7.84 (t, J = 7.5 Hz, H), 7.42-7.26 (m, 5H), 6.16 (d, J = 9.3 Hz, 1H), 5.25 (dd, J = 9.3, 5.7 Hz, 1H), 5.17 (s, 2H), 2.44 (sextet, J = 6.6 Hz, 1H), 1.05 (d, J = 6.6 Hz, 3H), 0.99 (d, J = 6.9 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 165.4, 157.4, 156.5, 155.7, 136.7, 134.3, 132.5, 130.4, 130.0, 129.6, 129.1, 128.9, 128.6, 128.3, 125.5, 123.9, 67.0, 61.1, 34.2, 19.7, 17.7. Anal. Calcd. for $C_{25}H_{22}N_4O_2$ (410.48): C, 73.15; H, 5.40; N, 13.65. Found: C, 73.32; H, 5.44; N, 13.52.

(S)-Benzyl (1-(acenaphtho[1,2-e][1,2,4]triazin-9-yl)-2-phenylethyl)carbamate (13b). Orange microcrystals (61%); mp 112.0–114.0 °C. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 88.49 (d, J = 4.2 Hz, 1H), 8.42 (d, J = 6.9 Hz, 1H), 8.24 (d, J = 8.4 Hz, 1H), 8.16 (d, J = 8.1 Hz, 1H), 7.87 (t,

J = 7.2 Hz, 2H), 7.42-7.00 (m, 10 H), 6.14 (d, *J* = 8.4 Hz, 1H), 5.67 (q, *J* = 7.5 Hz, 1H), 5.19-5.08 (m, 2H), 3.50 (dd, *J* = 13.9, 5.9 Hz, 1H), 3.38 (dd, *J* = 13.7, 6.8 Hz, 1H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 165.1, 157.5, 156.0, 155.8, 136.5, 134.4, 132.7, 130.5, 130.0, 129.7, 128.7, 129.5, 129.2, 129.1, 129.0, 128.6, 128.5, 128.2, 126.8, 125.7, 124.1, 109.8, 66.9, 57.0, 41.7. Anal. Calcd. for $C_{29}H_{22}N_4O_2$ (458.52): C, 75.97; H, 4.84; N, 12.22. Found: C, 75.57; H, 4.78; N, 12.12.

HPLC Chromatogram of **8f**.



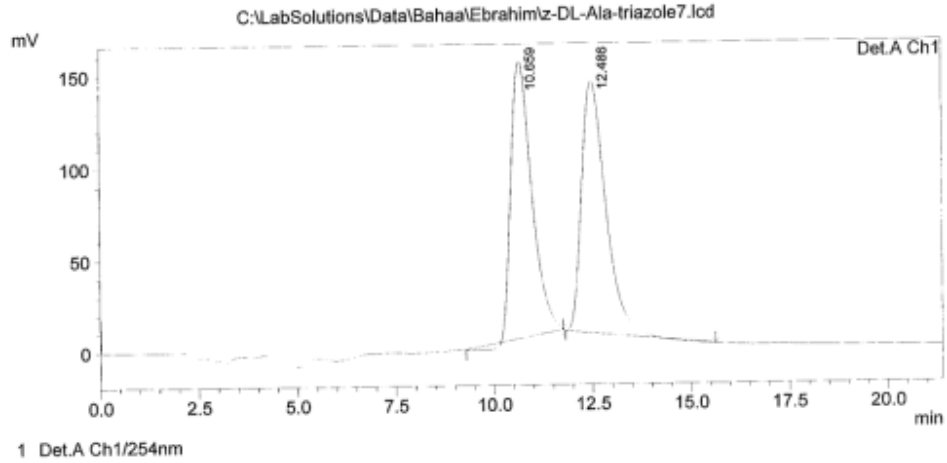
PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.586	77222832	2175176	99.883	99.889
2	17.235	90801	2420	0.117	0.111
Total		77313632	2177596	100.000	100.000

HPLC Chromatogram of **8f'**.

<Chromatogram>

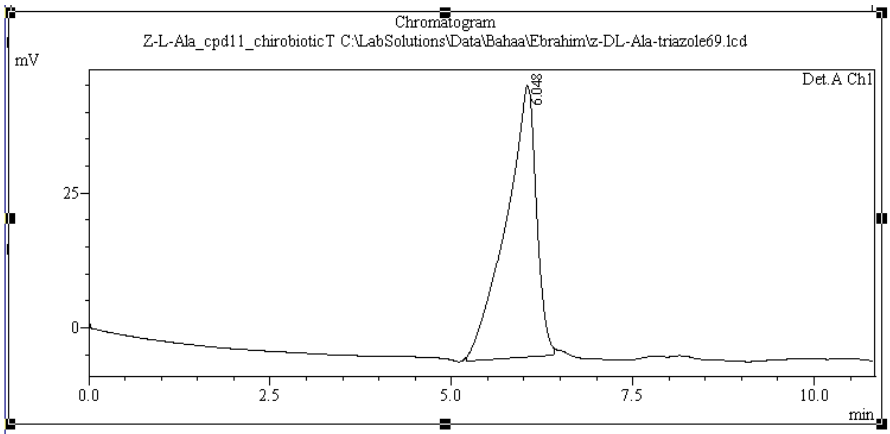


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.659	5479794	150537	48.650	52.404
2	12.486	5783915	136727	51.350	47.596
Total		11263709	287264	100.000	100.000

HPLC Chromatogram of **11d**.



PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.048	1466181	50376	100.000	100.000
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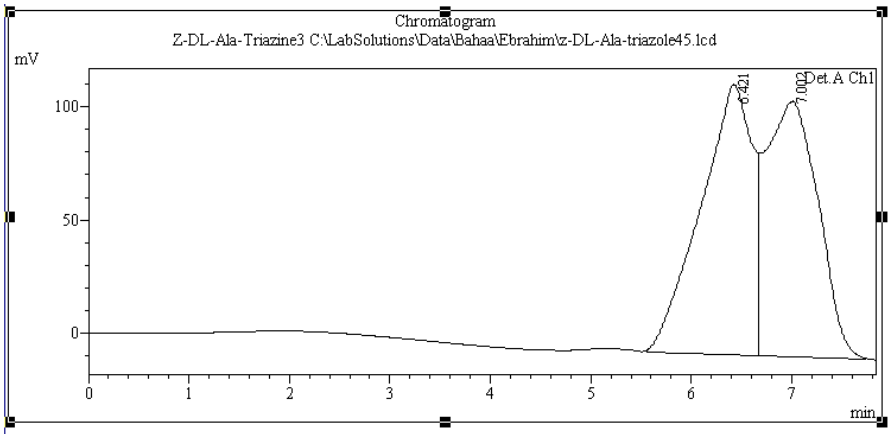
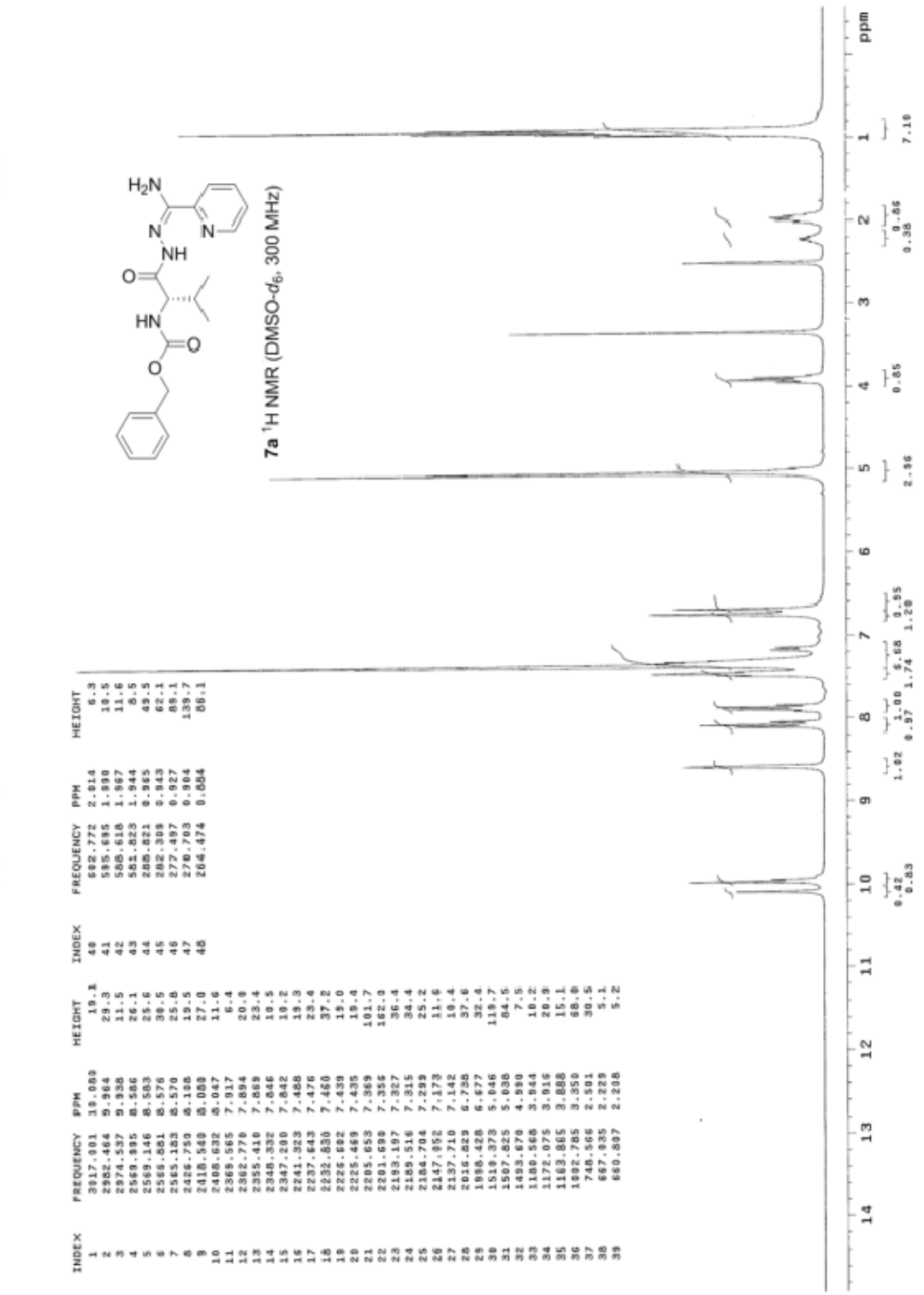
HPLC Chromatogram of **11d'**.



PeakTable

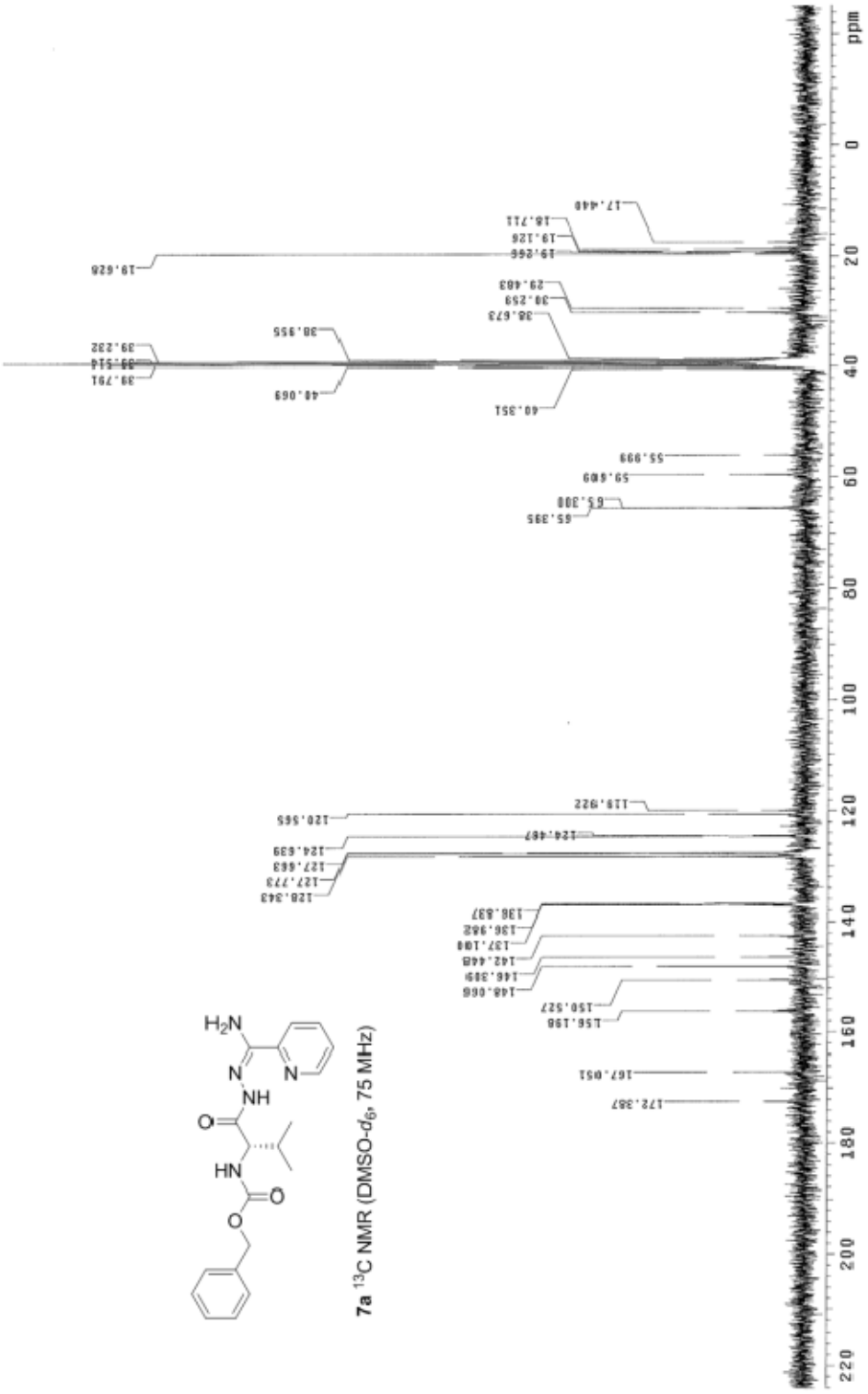
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.421	4259074	119226	51.206	51.433
2	7.002	4058420	112583	48.794	48.567
Total		8317494	231809	100.000	100.000

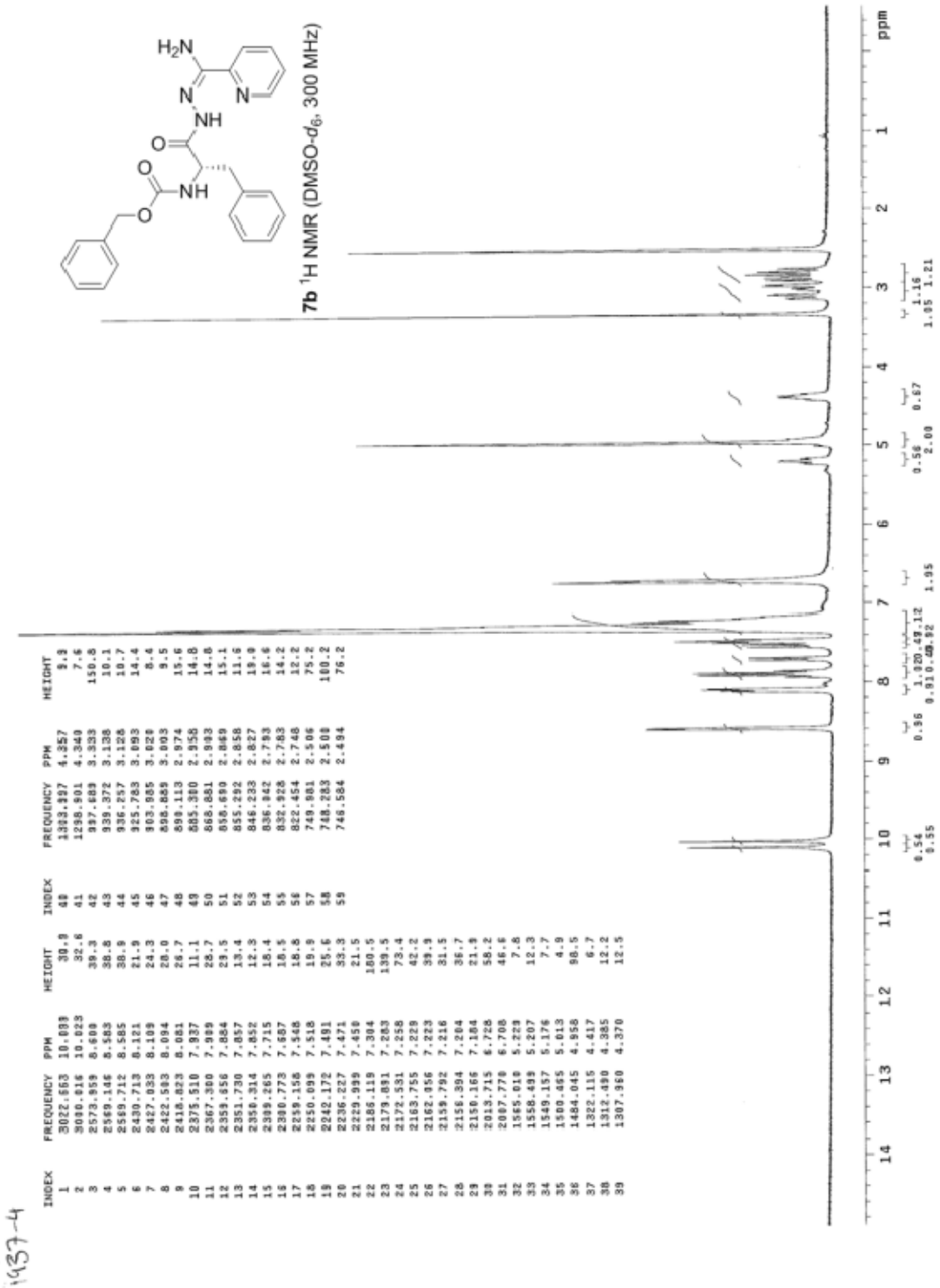


INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	3017.001	10.059	19.1	40	602.772	2.014	6.3
2	2982.464	9.964	29.3	41	595.695	1.990	10.5
3	2974.537	9.938	11.5	42	588.618	1.967	11.6
4	2569.895	8.586	26.1	43	581.823	1.944	8.5
5	2569.146	8.583	25.6	44	288.821	0.965	49.5
6	2565.881	8.576	30.5	45	282.308	0.943	62.1
7	2565.183	8.570	25.8	46	277.497	0.927	89.1
8	2426.750	8.108	19.5	47	270.703	0.904	139.7
9	2418.548	8.080	27.0	48	264.474	0.884	86.1
10	2408.632	8.047	11.6				
11	2369.565	7.917	6.4				
12	2362.770	7.894	20.0				
13	2355.410	7.869	23.4				
14	2348.332	7.846	10.5				
15	2347.200	7.842	10.2				
16	2241.323	7.488	19.3				
17	2237.643	7.476	23.4				
18	2232.836	7.460	37.2				
19	2226.692	7.439	19.0				
20	2225.469	7.435	19.4				
21	2205.653	7.369	101.7				
22	2201.698	7.356	182.0				
23	2193.197	7.327	36.4				
24	2189.516	7.315	34.4				
25	2184.704	7.299	25.2				
26	2147.852	7.173	11.6				
27	2137.710	7.142	10.4				
28	2016.829	6.738	37.6				
29	1998.428	6.677	32.4				
30	1510.373	5.046	118.7				
31	1507.825	5.038	84.5				
32	1493.670	4.990	7.5				
33	1180.568	3.944	10.2				
34	1172.075	3.916	20.9				
35	1163.865	3.888	15.1				
36	1092.785	3.650	88.8				
37	748.566	2.501	30.5				
38	667.035	2.229	5.1				
39	660.807	2.208	5.2				

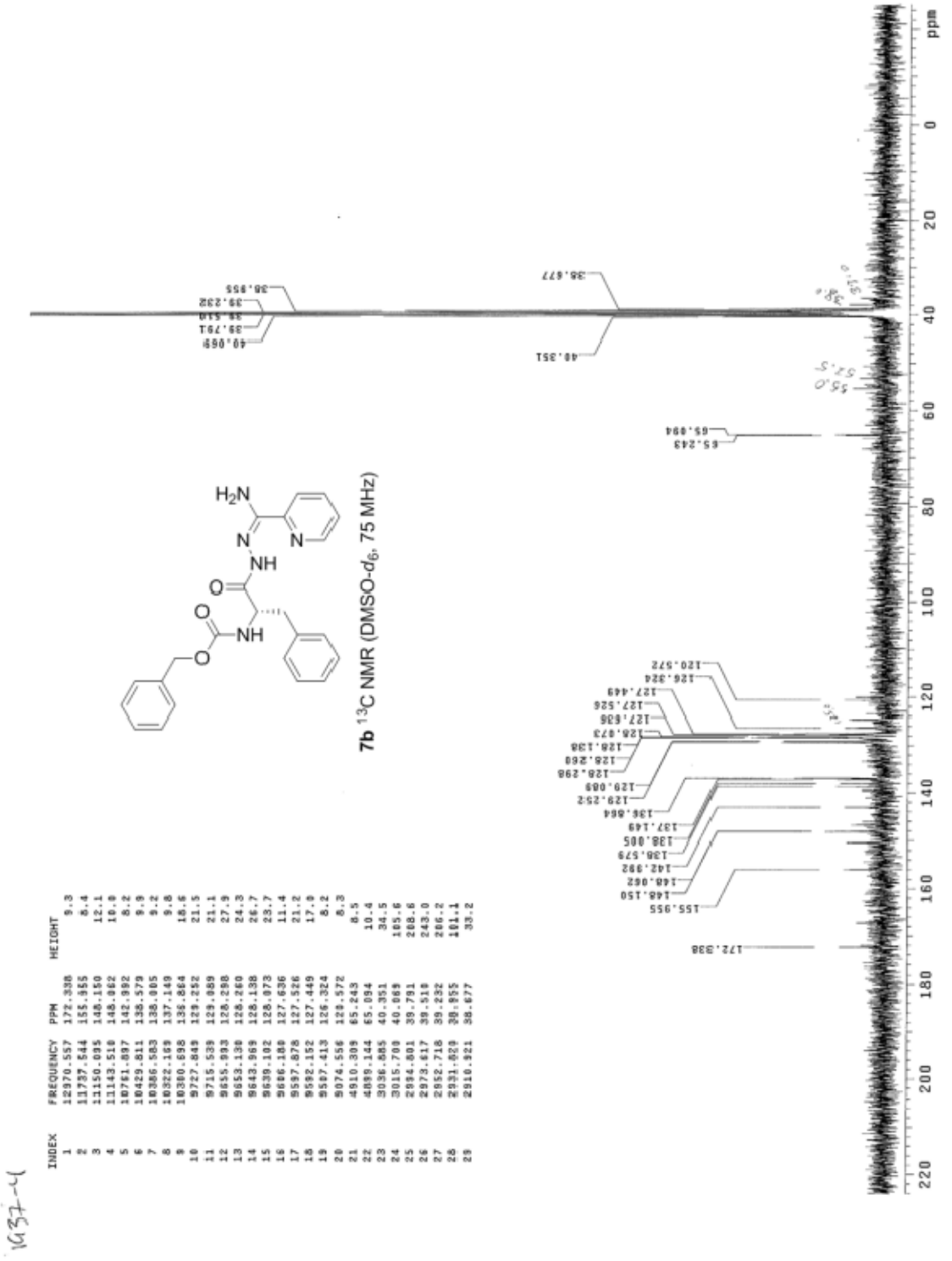
A137-1



7a ^{13}C NMR (DMSO-d_6, 75 MHz)

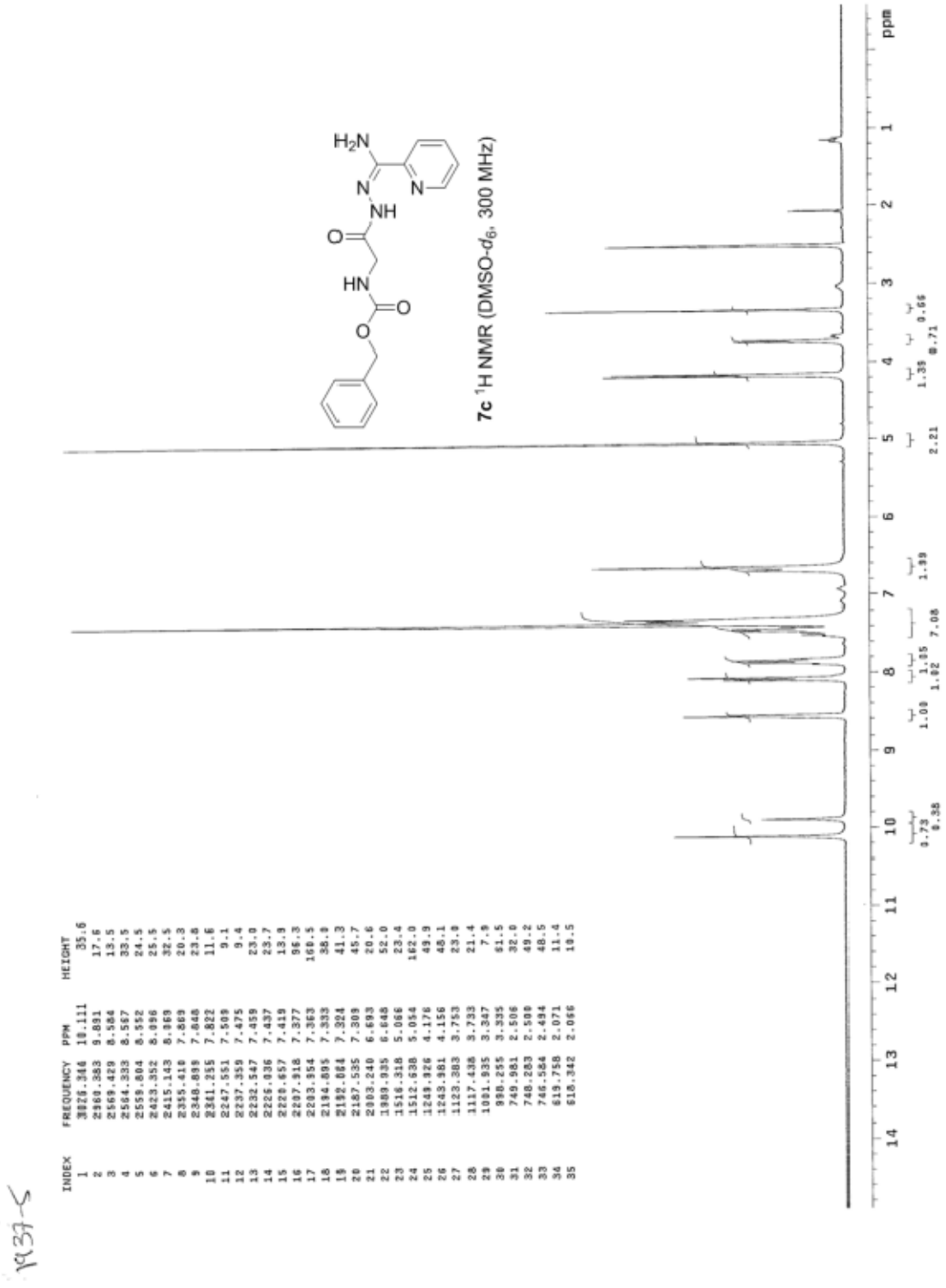


INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	3022.663	10.080	39.8	40	1393.397	4.357	9.9
2	3030.816	10.023	32.6	41	1298.901	4.349	7.6
3	2573.959	8.600	39.3	42	937.689	3.333	150.8
4	2569.146	8.583	38.8	43	939.372	3.138	10.1
5	2569.712	8.585	38.9	44	936.257	3.126	10.7
6	2430.713	8.121	21.9	45	925.783	3.093	14.4
7	2427.033	8.109	24.3	46	903.985	3.020	8.4
8	2422.583	8.094	28.0	47	898.889	3.003	9.5
9	2416.823	8.081	26.7	48	890.113	2.974	15.6
10	2375.510	7.937	11.1	49	885.380	2.956	14.8
11	2367.300	7.909	28.7	50	868.881	2.903	14.8
12	2359.656	7.884	23.5	51	850.690	2.869	15.1
13	2351.730	7.857	13.4	52	855.292	2.858	11.6
14	2350.314	7.852	12.3	53	846.238	2.827	19.9
15	2309.265	7.715	18.4	54	836.842	2.793	16.6
16	2300.773	7.687	10.5	55	832.928	2.783	14.2
17	2259.158	7.548	18.8	56	822.454	2.748	12.2
18	2250.099	7.518	19.9	57	749.881	2.506	75.2
19	2242.172	7.491	25.6	58	748.283	2.500	100.2
20	2236.227	7.471	33.3	59	748.584	2.494	76.2
21	2229.999	7.450	21.5				
22	2186.119	7.304	180.5				
23	2179.891	7.283	139.5				
24	2172.531	7.258	73.4				
25	2163.755	7.229	42.2				
26	2162.456	7.223	39.9				
27	2159.792	7.216	31.5				
28	2158.394	7.204	36.7				
29	2150.166	7.184	21.9				
30	2013.715	6.728	58.2				
31	2007.770	6.708	46.6				
32	1565.018	5.229	7.8				
33	1558.499	5.207	12.3				
34	1549.157	5.176	7.7				
35	1500.465	5.013	4.9				
36	1484.045	4.958	98.5				
37	1322.115	4.417	6.7				
38	1312.490	4.385	12.2				
39	1307.360	4.370	12.5				



INDEX	FREQUENCY	PPM	HEIGHT
1	12970.557	172.338	9.3
2	11737.544	155.955	8.4
3	11150.095	148.150	12.1
4	11143.510	148.062	10.8
5	10761.697	142.992	8.2
6	10429.811	138.579	9.9
7	10386.583	138.065	9.2
8	10322.169	137.149	9.8
9	10300.698	136.864	15.6
10	9727.849	129.252	21.5
11	9715.539	129.089	21.1
12	9655.093	128.298	27.9
13	9653.138	128.260	24.3
14	9643.969	128.138	26.7
15	9639.102	128.073	23.7
16	9606.180	127.636	11.4
17	9597.878	127.526	21.2
18	9592.152	127.449	17.9
19	9587.413	126.324	8.2
20	9074.556	128.572	8.3
21	4910.309	65.243	8.5
22	4899.144	65.094	10.4
23	3036.685	40.351	34.5
24	3015.700	40.869	165.6
25	2934.601	39.791	268.6
26	2973.617	39.518	243.0
27	2952.718	39.232	206.2
28	2931.829	38.955	161.1
29	2910.921	38.677	33.2

1937-4



INDEX	FREQUENCY	PPM	HEIGHT
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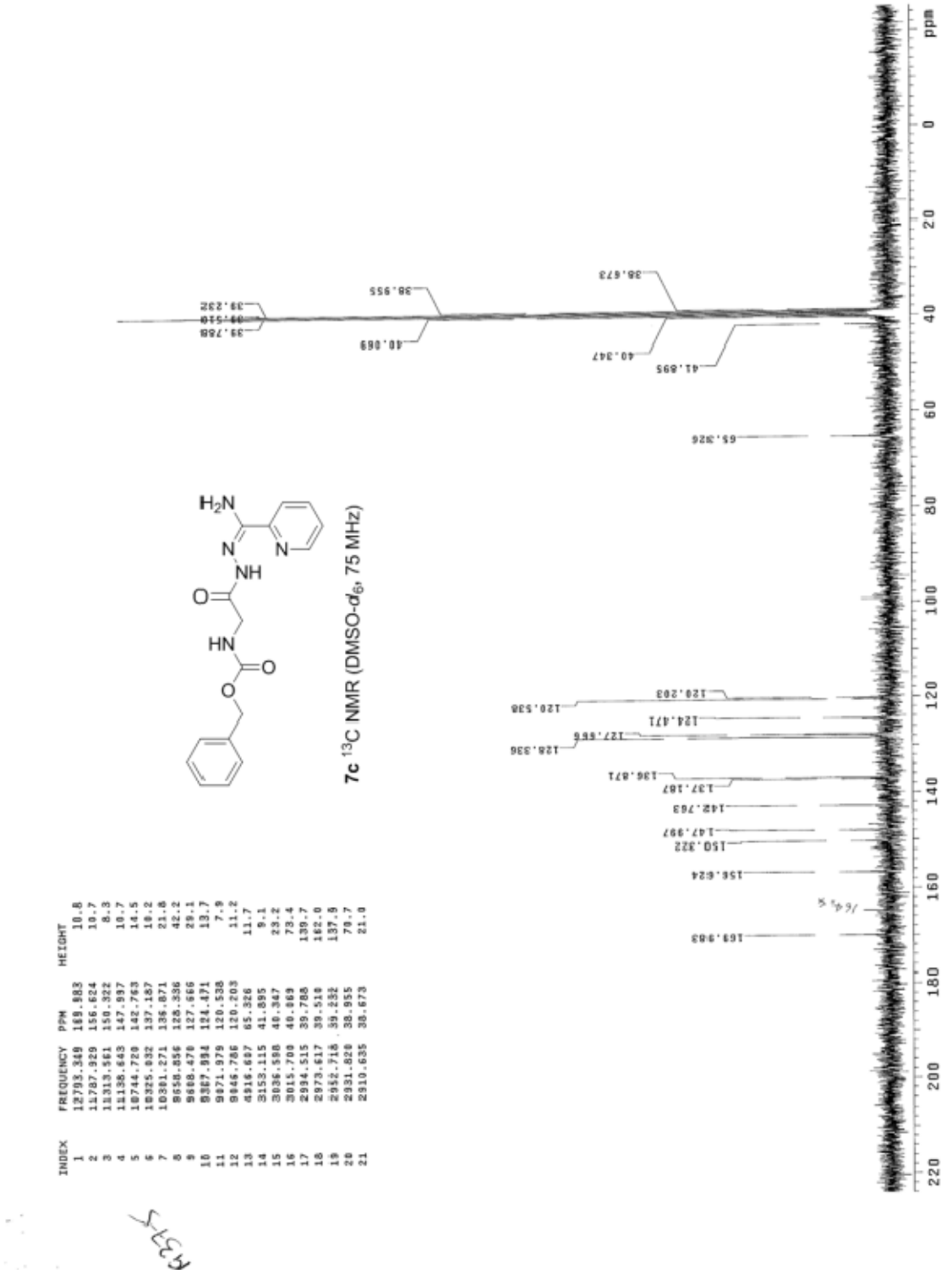
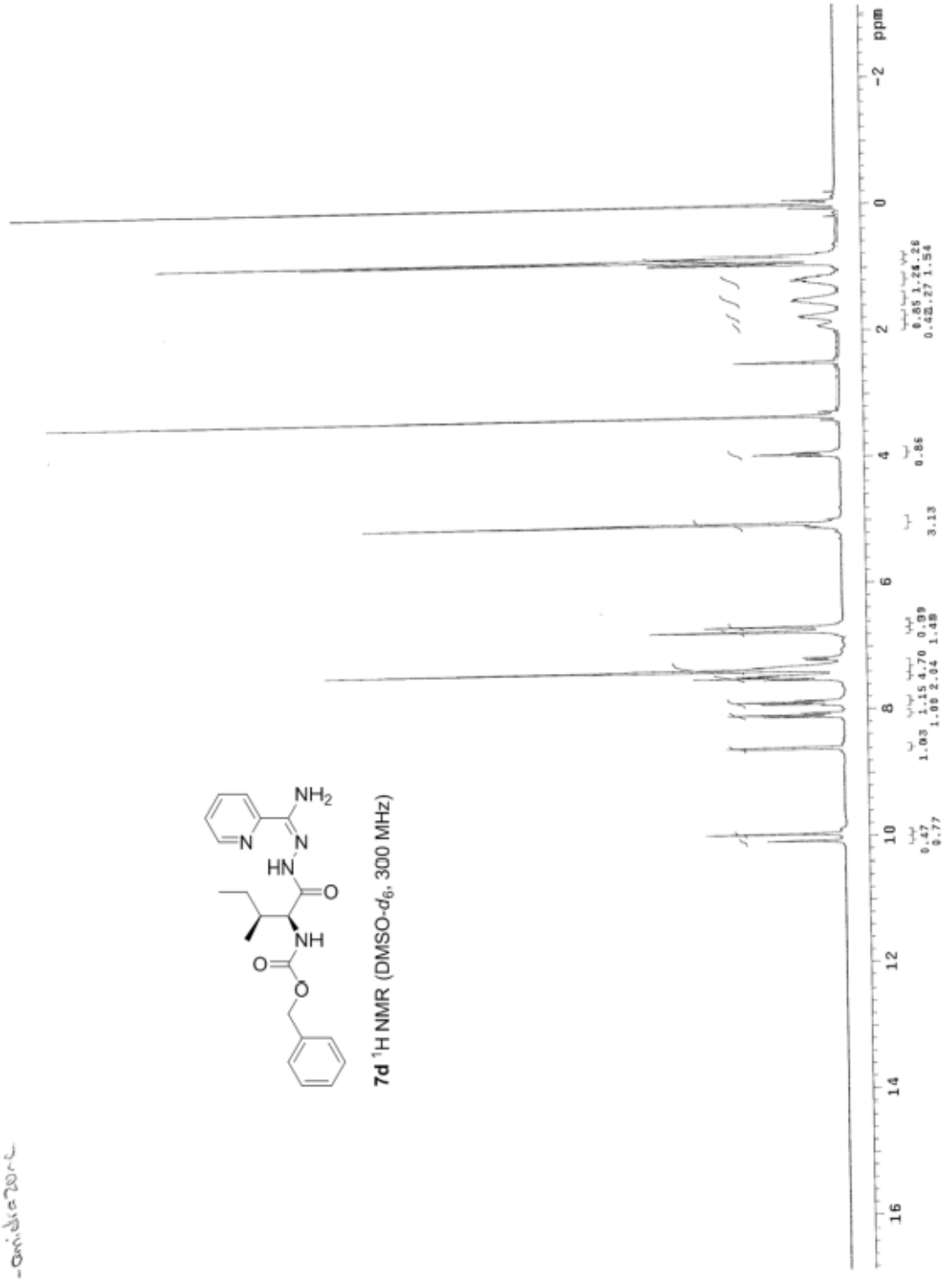
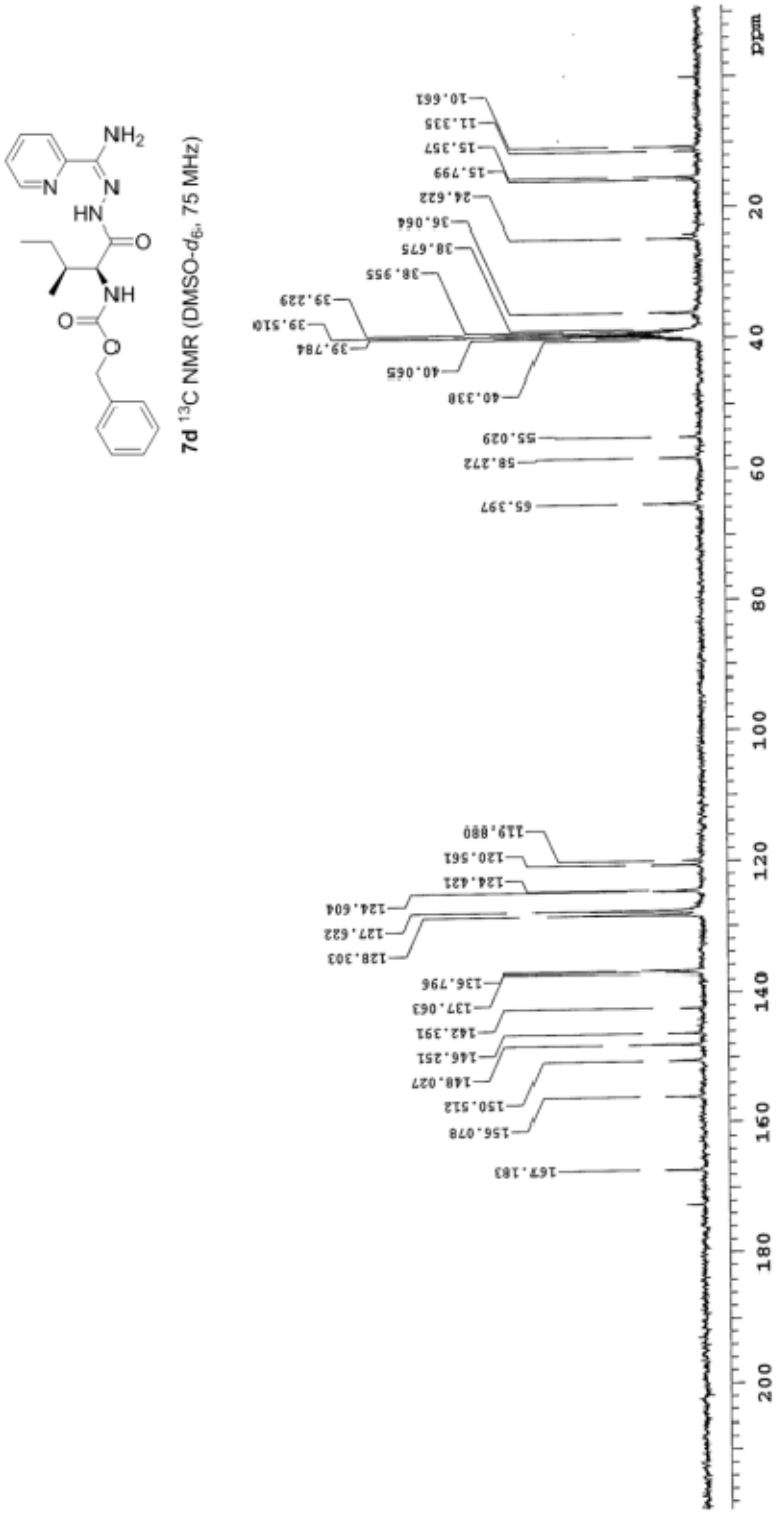
4	2564.333	8.567	33.5
5	2559.804	8.552	24.5
6	2423.352	8.096	25.5
7	2415.143	8.069	32.5
8	2359.418	7.869	20.3
9	2348.898	7.848	23.8
10	2341.255	7.822	11.6
11	2247.551	7.509	9.1
12	2237.359	7.475	9.4
13	2232.547	7.459	23.0
14	2226.036	7.437	23.7
15	2220.657	7.419	13.9
16	2207.918	7.377	96.3
17	2203.354	7.363	160.5
18	2194.895	7.333	38.8
19	2192.884	7.324	41.3
20	2187.535	7.309	45.7
21	2003.240	6.693	20.6
22	1989.935	6.648	52.0
23	1516.318	5.066	23.4
24	1512.638	5.054	162.0
25	1249.326	4.176	49.9
26	1243.381	4.156	48.1
27	1123.383	3.753	23.8
28	1117.438	3.733	21.4
29	1001.935	3.347	7.9
30	998.255	3.335	61.5
31	749.981	2.506	32.0
32	748.283	2.500	49.2
33	746.584	2.494	48.5
34	619.758	2.071	11.4
35	618.342	2.066	10.5



INDEX	FREQUENCY	PPM	HEIGHT
1	12793.349	169.883	10.8
2	11787.929	156.624	10.7
3	11313.561	150.322	8.3
4	11138.643	147.937	10.7
5	10744.720	142.763	14.5
6	10325.832	137.187	18.2
7	10301.271	136.871	21.8
8	9658.856	128.336	42.2
9	9608.470	127.666	29.1
10	9387.994	124.471	13.7
11	9071.979	120.536	7.9
12	9046.786	120.203	11.2
13	4918.667	65.326	11.7
14	3153.115	41.895	9.1
15	3036.598	40.347	23.2
16	3015.700	40.089	73.4
17	2994.515	39.788	139.7
18	2973.617	39.510	182.0
19	2952.718	39.232	137.8
20	2931.820	38.955	79.7
21	2910.635	38.673	21.0

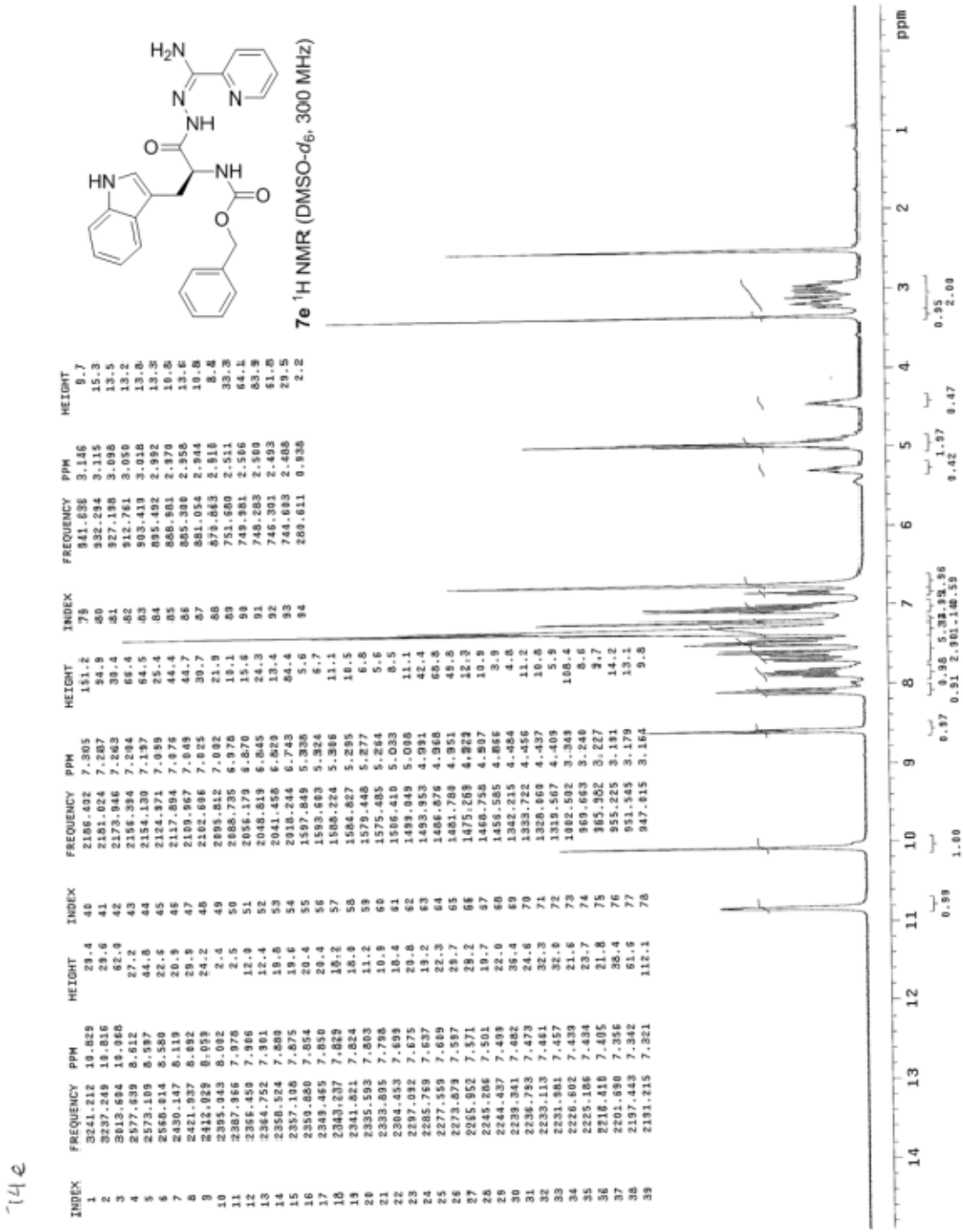


7d ^{1}H NMR (DMSO-d_6, 300 MHz)

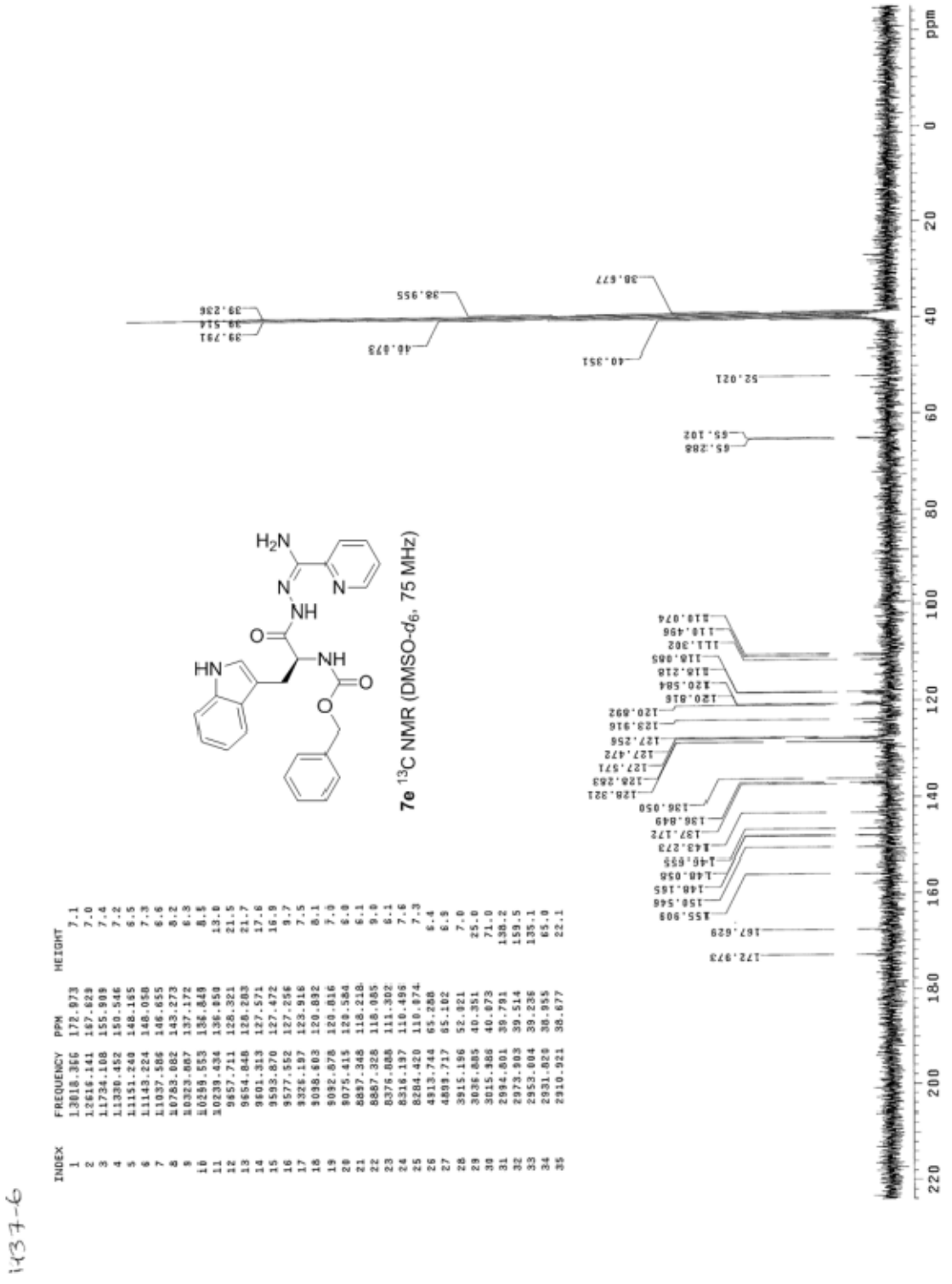


7d ^{13}C NMR (DMSO-d_6, 75 MHz)

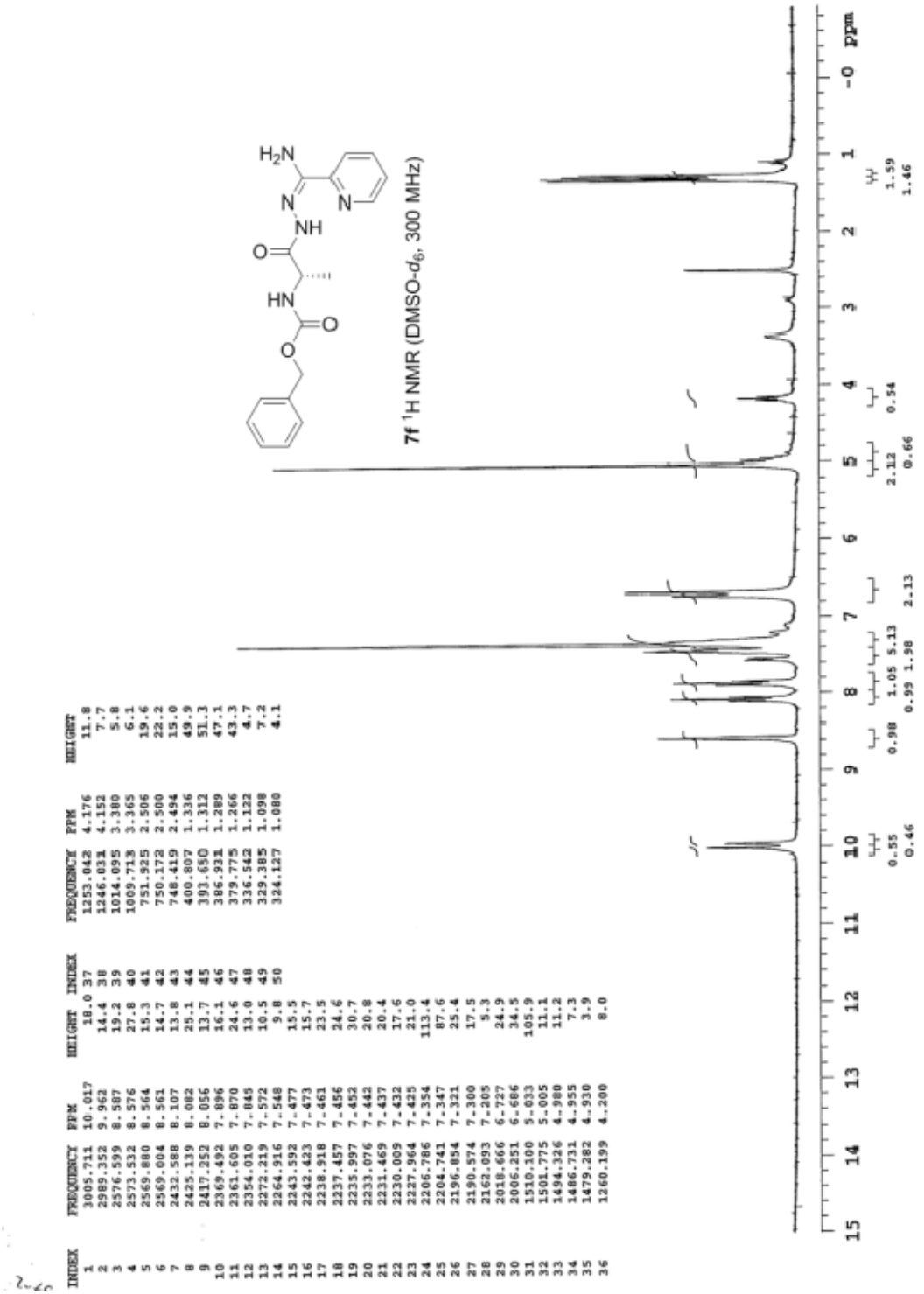
L-Ile-Amidoacene



INDEX	FREQUENCY	PPM	HEIGHT
1	3241.212	10.829	29.4
2	3237.249	10.816	29.6
3	3013.694	10.068	62.0
4	2977.039	8.612	27.2
5	2573.109	8.597	44.8
6	2560.614	8.580	22.5
7	2430.147	8.119	20.5
8	2421.937	8.092	29.9
9	2418.029	8.079	24.2
10	2395.043	8.002	2.4
11	2387.366	7.978	2.5
12	2366.450	7.906	12.9
13	2364.752	7.901	12.4
14	2358.524	7.880	19.8
15	2357.198	7.875	19.6
16	2350.880	7.854	20.4
17	2349.465	7.850	20.4
18	2343.237	7.829	18.2
19	2341.821	7.824	18.8
20	2335.593	7.803	11.2
21	2333.895	7.798	10.8
22	2304.453	7.699	18.4
23	2297.092	7.675	20.8
24	2285.769	7.637	19.2
25	2277.559	7.609	22.3
26	2273.879	7.597	29.7
27	2265.952	7.571	29.2
28	2245.286	7.501	19.7
29	2244.437	7.498	22.0
30	2239.341	7.482	36.4
31	2236.793	7.473	24.6
32	2233.113	7.461	32.3
33	2231.981	7.457	32.0
34	2226.602	7.439	21.6
35	2225.186	7.434	23.7
36	2218.418	7.405	21.8
37	2201.690	7.356	38.4
38	2197.443	7.342	61.6
39	2191.215	7.321	112.1
40	2186.492	7.305	151.2
41	2181.024	7.287	94.9
42	2173.946	7.263	39.4
43	2156.394	7.204	68.4
44	2154.130	7.197	64.5
45	2124.371	7.099	25.4
46	2117.894	7.076	44.4
47	2109.967	7.049	44.7
48	2102.696	7.025	30.7
49	2095.812	7.002	21.9
50	2088.735	6.978	19.1
51	2056.179	6.870	15.6
52	2048.819	6.845	24.3
53	2041.458	6.820	13.4
54	2018.244	6.743	84.4
55	1597.849	5.338	5.6
56	1593.603	5.324	6.7
57	1588.224	5.306	11.1
58	1584.827	5.295	10.5
59	1579.448	5.277	6.8
60	1575.485	5.264	5.6
61	1506.410	5.033	8.5
62	1499.049	5.008	11.1
63	1493.953	4.991	42.4
64	1486.876	4.968	68.8
65	1481.788	4.951	49.8
66	1475.269	4.929	12.3
67	1468.758	4.907	10.9
68	1456.585	4.866	3.9
69	1342.215	4.484	4.8
70	1333.722	4.456	11.2
71	1328.868	4.437	10.8
72	1319.567	4.409	5.9
73	1002.502	3.349	108.4
74	969.663	3.240	8.6
75	965.982	3.227	9.7
76	955.225	3.191	14.2
77	951.345	3.179	13.1
78	947.815	3.164	9.8
79	941.838	3.146	8.7
80	932.294	3.115	15.3
81	927.198	3.098	13.5
82	912.761	3.050	13.2
83	903.413	3.018	13.8
84	895.492	2.992	13.3
85	888.581	2.970	10.8
86	885.398	2.958	13.6
87	881.054	2.944	10.8
88	879.863	2.940	8.4
89	751.680	2.511	33.3
90	749.981	2.506	64.1
91	748.283	2.500	83.9
92	746.301	2.493	61.8
93	744.603	2.488	29.5
94	280.611	0.938	2.2



INDEX	FREQUENCY	PPM	HEIGHT
1	13018.366	172.973	7.1
2	12616.141	167.629	7.0
3	11734.108	155.909	7.4
4	11330.452	150.546	7.2
5	11151.240	148.165	6.5
6	11143.224	148.058	7.3
7	11037.586	146.655	6.6
8	10783.082	143.273	8.2
9	10323.867	137.172	6.3
10	10269.553	136.849	8.5
11	10239.434	136.850	13.0
12	9957.711	128.321	21.5
13	9854.848	128.203	21.7
14	9601.313	127.571	17.6
15	9593.870	127.472	16.9
16	9577.552	127.256	9.7
17	9326.197	123.916	7.5
18	9098.603	120.832	8.1
19	9092.678	120.816	7.0
20	9075.415	120.584	6.0
21	8887.348	118.218	6.1
22	8887.328	118.085	9.0
23	8376.888	111.302	8.1
24	8316.197	110.496	7.6
25	8284.420	110.474	7.3
26	4813.744	65.288	6.4
27	4899.717	65.102	6.9
28	3915.196	52.021	7.0
29	3036.885	40.351	25.0
30	3015.986	40.073	71.0
31	2994.801	39.791	138.2
32	2973.903	39.514	159.5
33	2953.004	39.236	135.1
34	2931.828	38.955	65.0
35	2910.921	38.677	22.1



INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	3005.711	10.017	18.0	37	1253.042	4.176	11.8
2	2989.352	9.962	14.4	38	1246.031	4.152	7.7
3	2576.599	8.587	19.2	39	1014.095	3.380	5.8
4	2573.532	8.576	27.8	40	1009.713	3.365	6.1
5	2569.880	8.564	15.3	41	751.925	2.506	19.6
6	2569.004	8.561	14.7	42	750.172	2.500	22.2
7	2432.588	8.107	13.8	43	748.419	2.494	15.0
8	2425.139	8.082	25.1	44	400.807	1.336	49.9
9	2417.252	8.056	13.7	45	393.650	1.312	51.3
10	2369.492	7.896	16.1	46	386.931	1.289	47.1
11	2361.605	7.870	24.6	47	379.775	1.266	43.3
12	2354.010	7.845	13.0	48	336.542	1.122	4.7
13	2272.219	7.572	10.5	49	329.385	1.098	7.2
14	2264.916	7.548	9.8	50	324.127	1.080	4.1
15	2243.592	7.477	15.5				
16	2242.423	7.473	15.7				
17	2238.918	7.461	23.5				
18	2237.457	7.456	24.6				
19	2235.997	7.452	30.7				
20	2233.076	7.442	20.8				
21	2231.469	7.437	20.4				
22	2230.009	7.432	17.6				
23	2227.964	7.425	21.0				
24	2206.786	7.354	113.4				
25	2204.741	7.347	87.6				
26	2196.854	7.321	25.4				
27	2190.574	7.300	17.5				
28	2162.093	7.205	5.3				
29	2010.666	6.727	24.9				
30	2006.251	6.686	34.5				
31	1510.100	5.033	105.9				
32	1501.775	5.005	11.1				
33	1494.326	4.980	11.2				
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35	1479.282	4.930	3.9				
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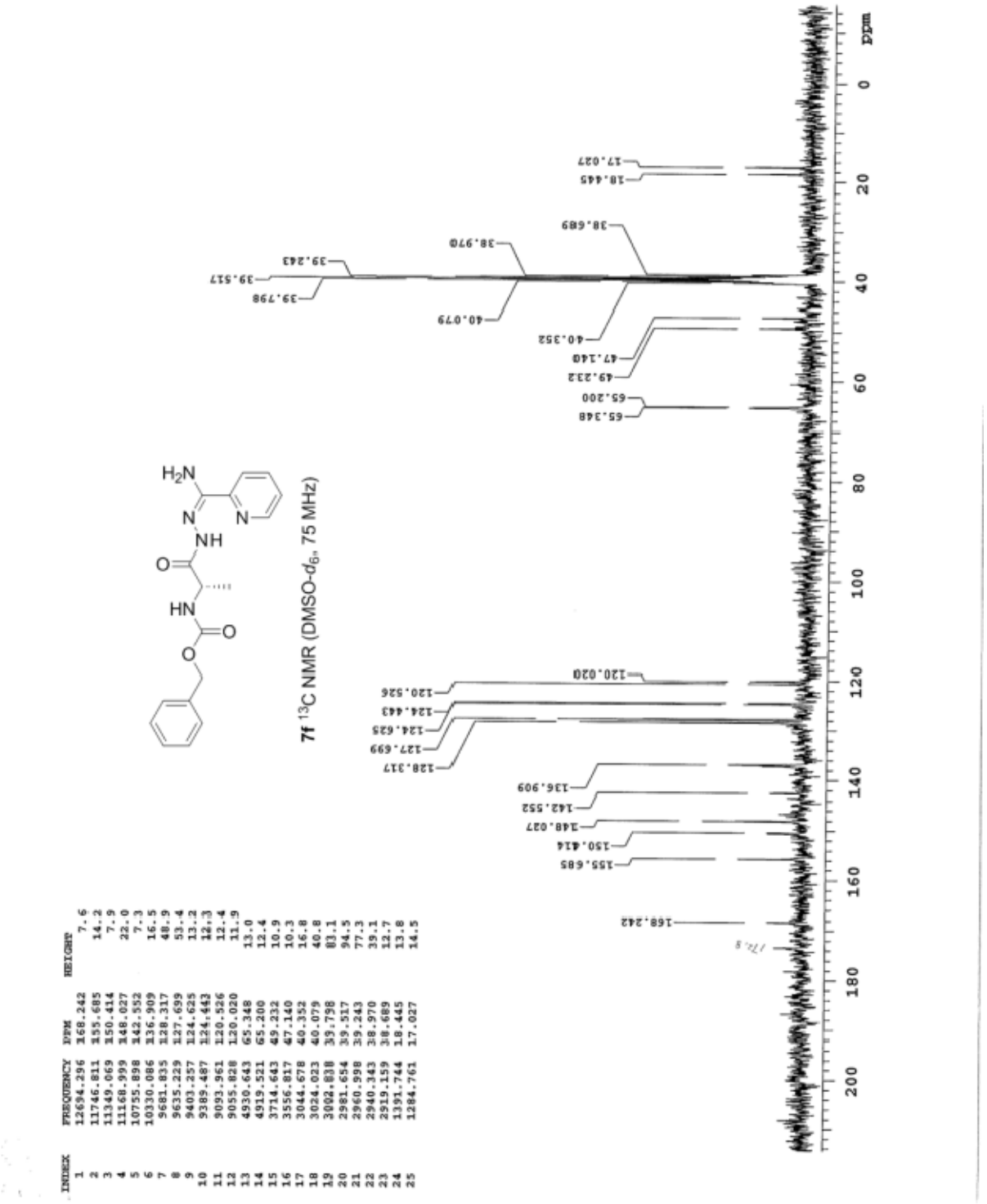
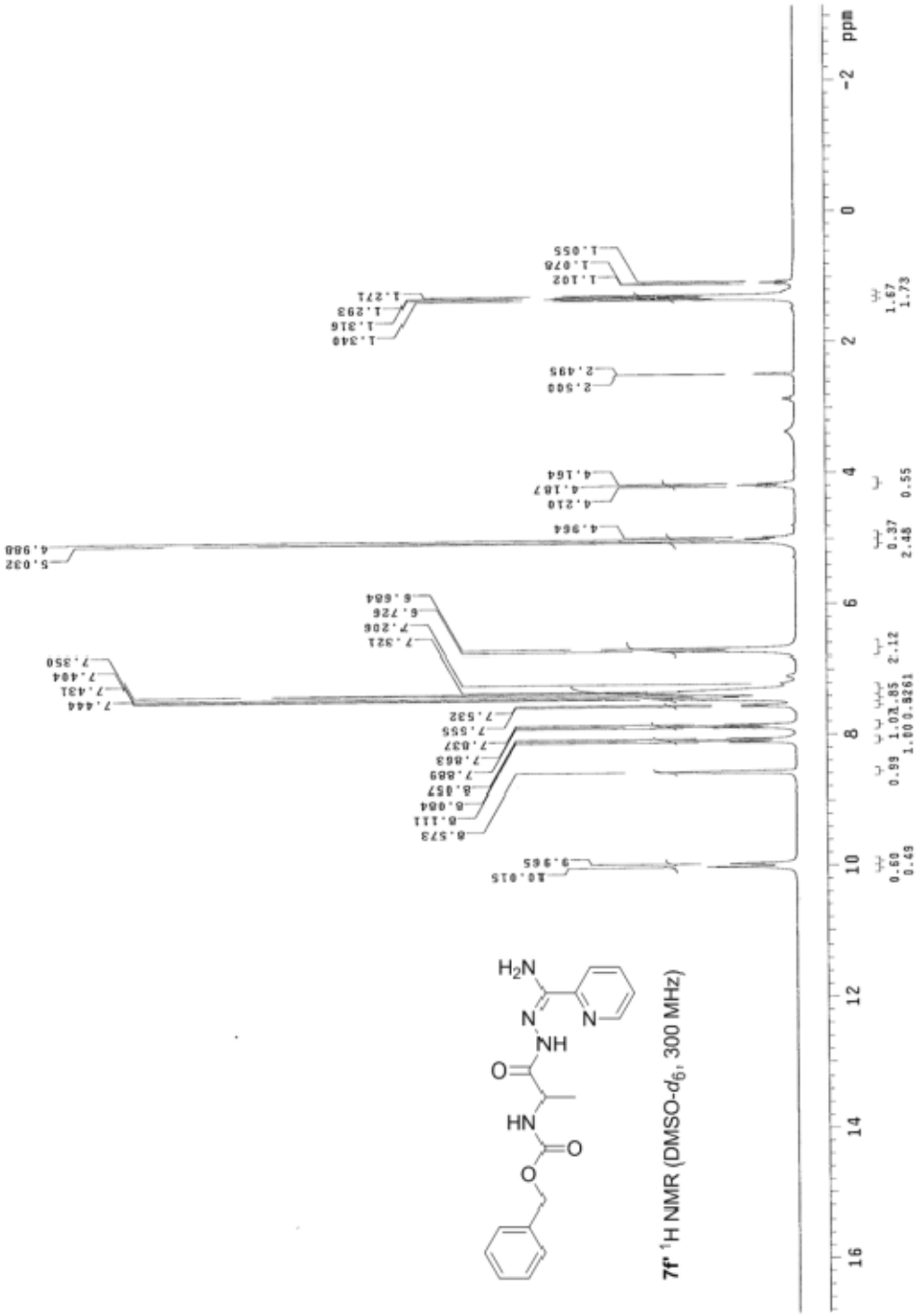
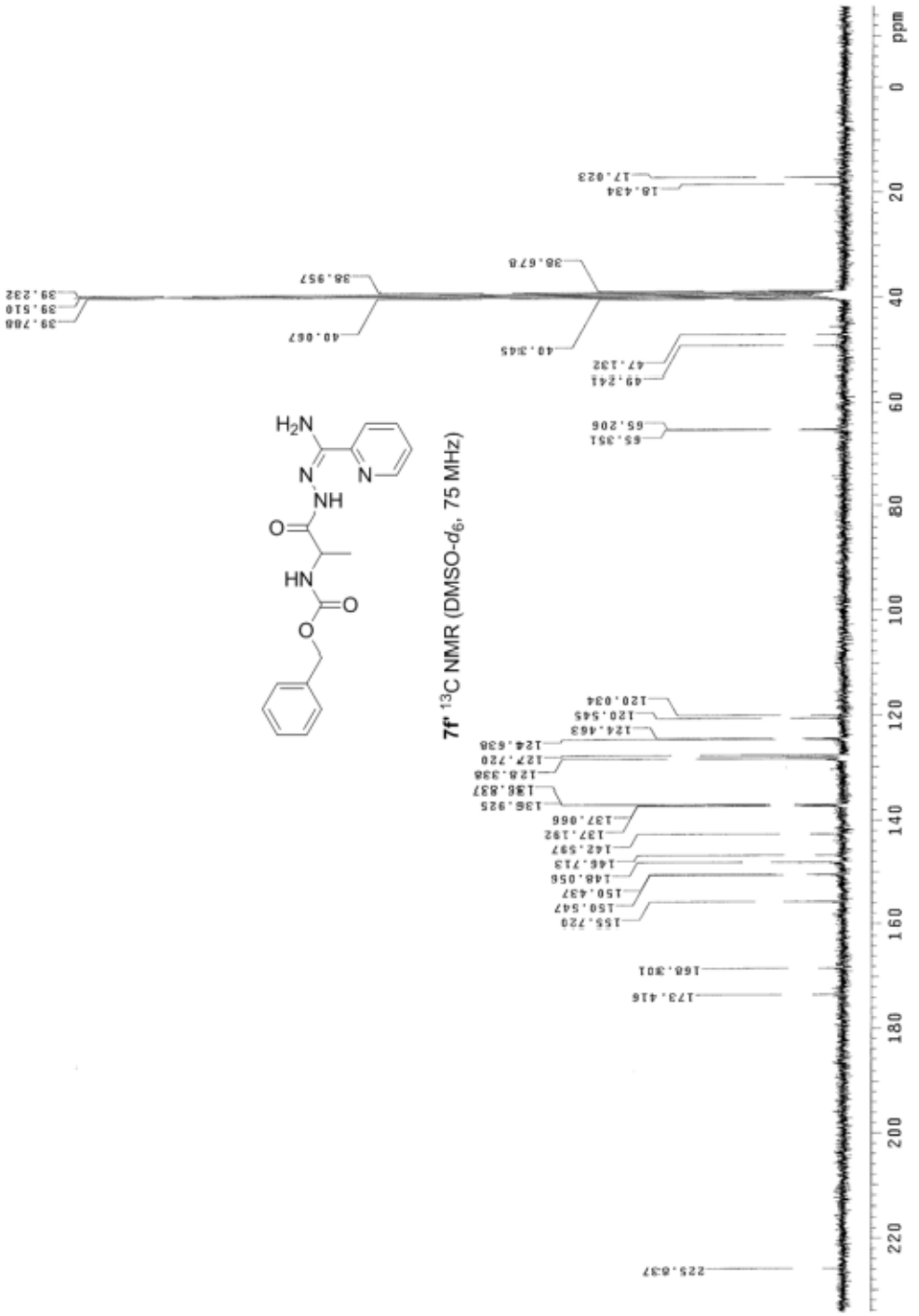


INDEX	FREQUENCY	PPM	HEIGHT
1	12694.296	168.242	7.6
2	11746.811	155.685	14.2
3	11349.069	150.414	7.9
4	11168.999	148.027	22.0
5	10755.898	142.552	7.3
6	10330.086	136.909	16.5
7	9681.835	128.317	48.9
8	9635.229	127.699	53.4
9	9403.257	124.625	13.2
10	9389.487	124.443	12.3
11	9093.961	120.526	12.4
12	9055.828	120.020	11.9
13	4930.643	65.348	13.0
14	4919.521	65.200	12.4
15	3714.643	49.232	10.9
16	3556.817	47.140	10.3
17	3044.678	40.352	16.8
18	3024.023	40.079	40.8
19	3002.838	39.798	83.1
20	2981.654	39.517	94.5
21	2960.998	39.243	77.3
22	2940.343	38.970	39.1
23	2919.159	38.689	12.7
24	1391.744	18.445	13.8
25	1284.761	17.027	14.5


7f' ^{1}H NMR (DMSO-d_6, 300 MHz)
ppm


7f' ^{13}C NMR (DMSO-d_6, 75 MHz)
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N
NH
O
HN
O
O
ppm
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60
80
100
120
140
160
180
200
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39.232
39.510
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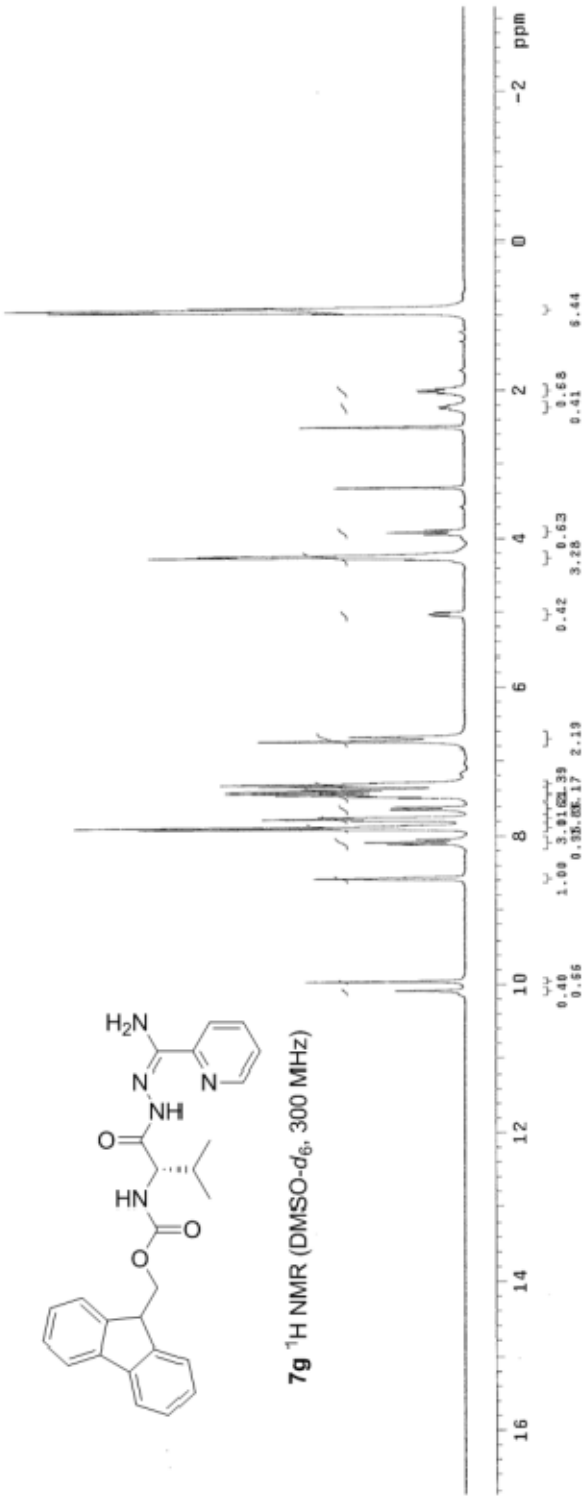
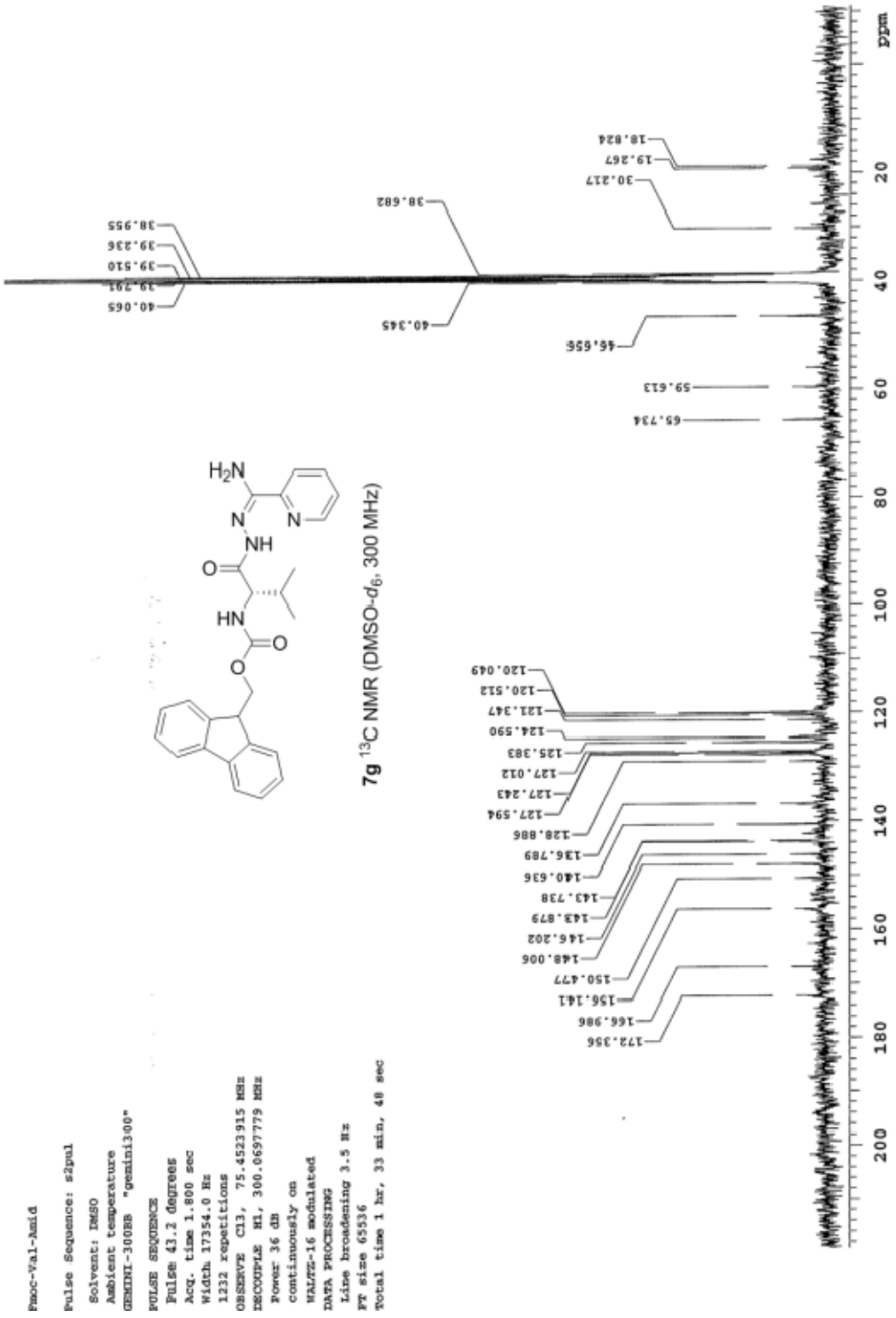


7g ^{1}H NMR (DMSO-d_6, 300 MHz)


7g ^{13}C NMR (DMSO-d_6, 300 MHz)
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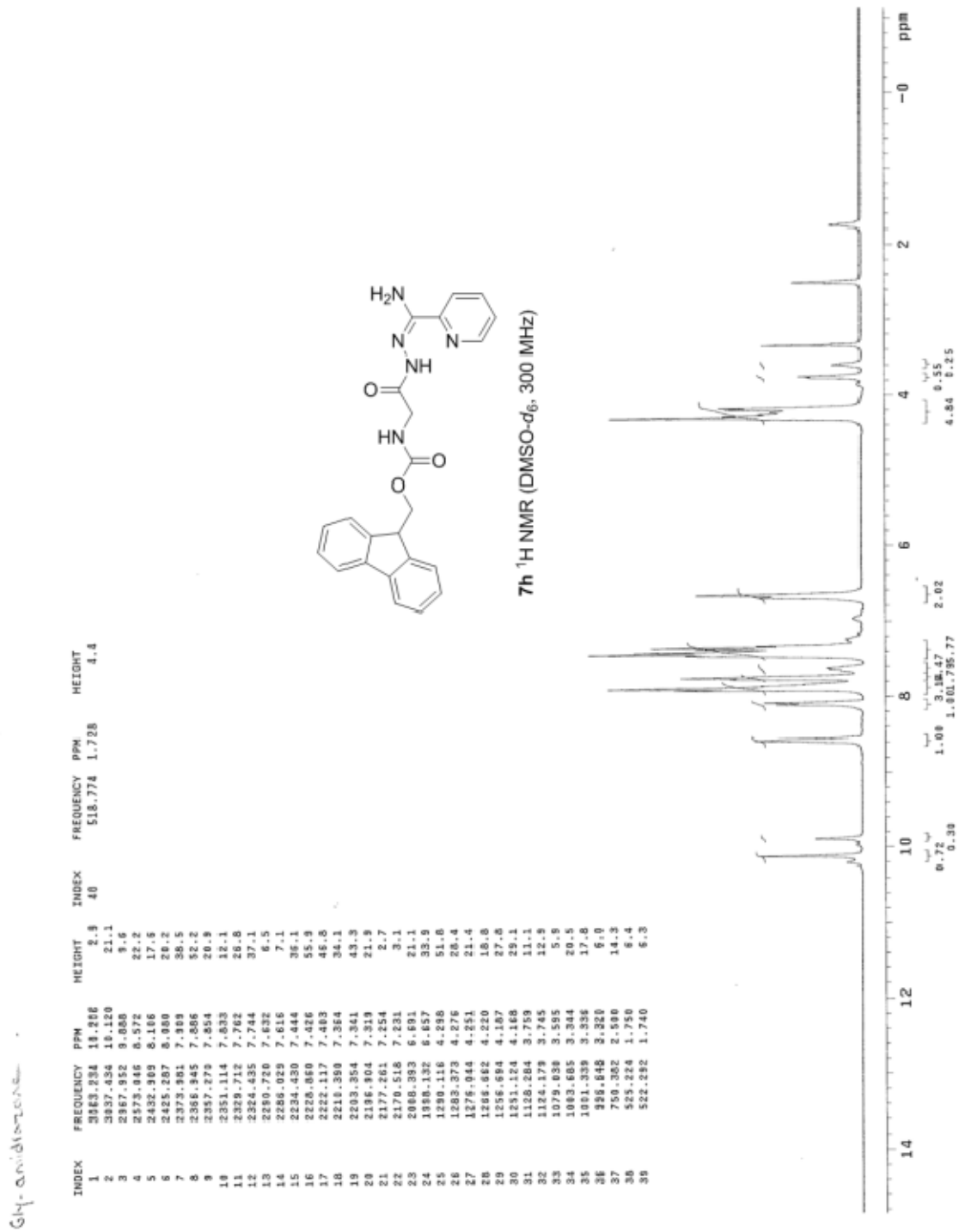
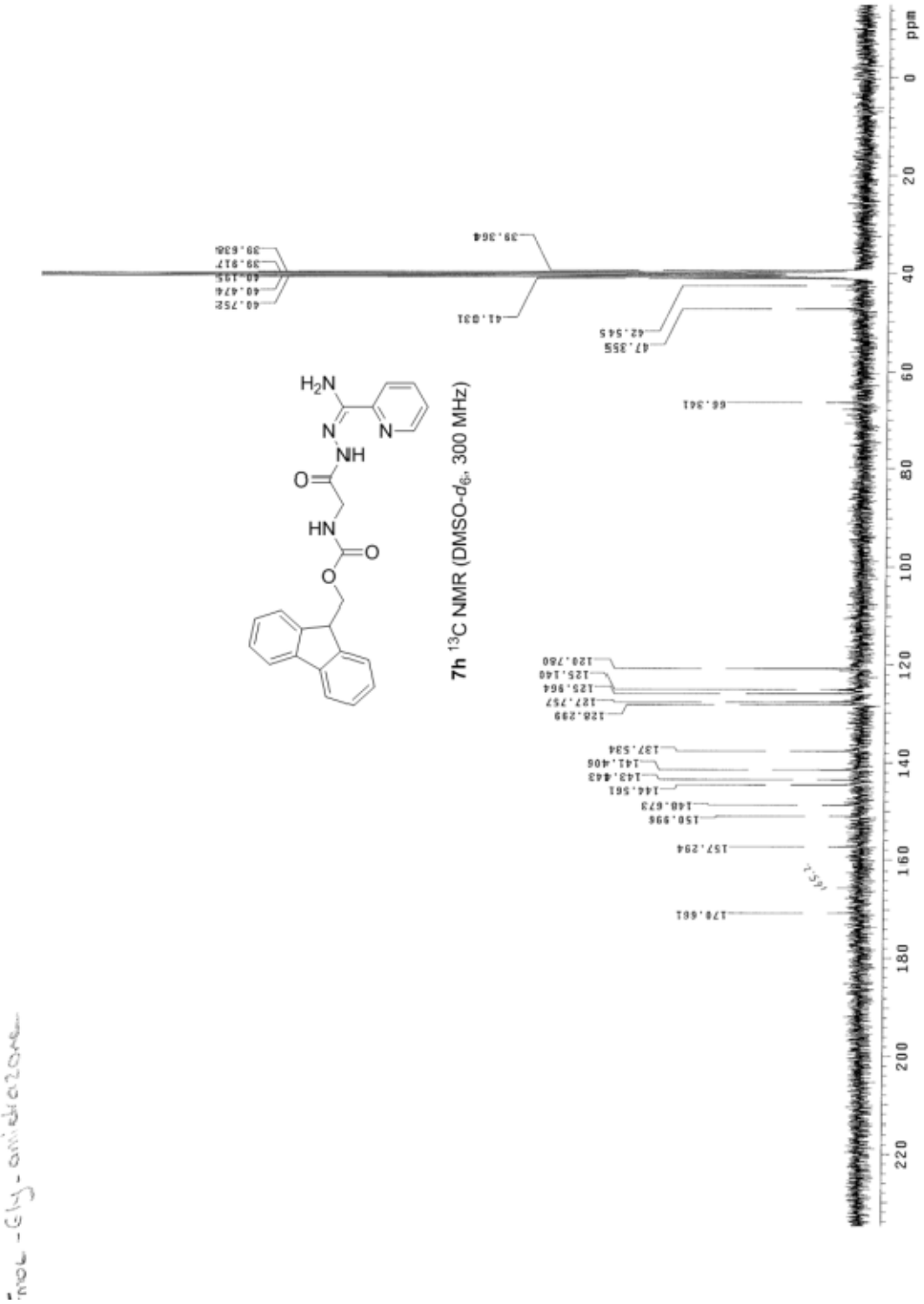
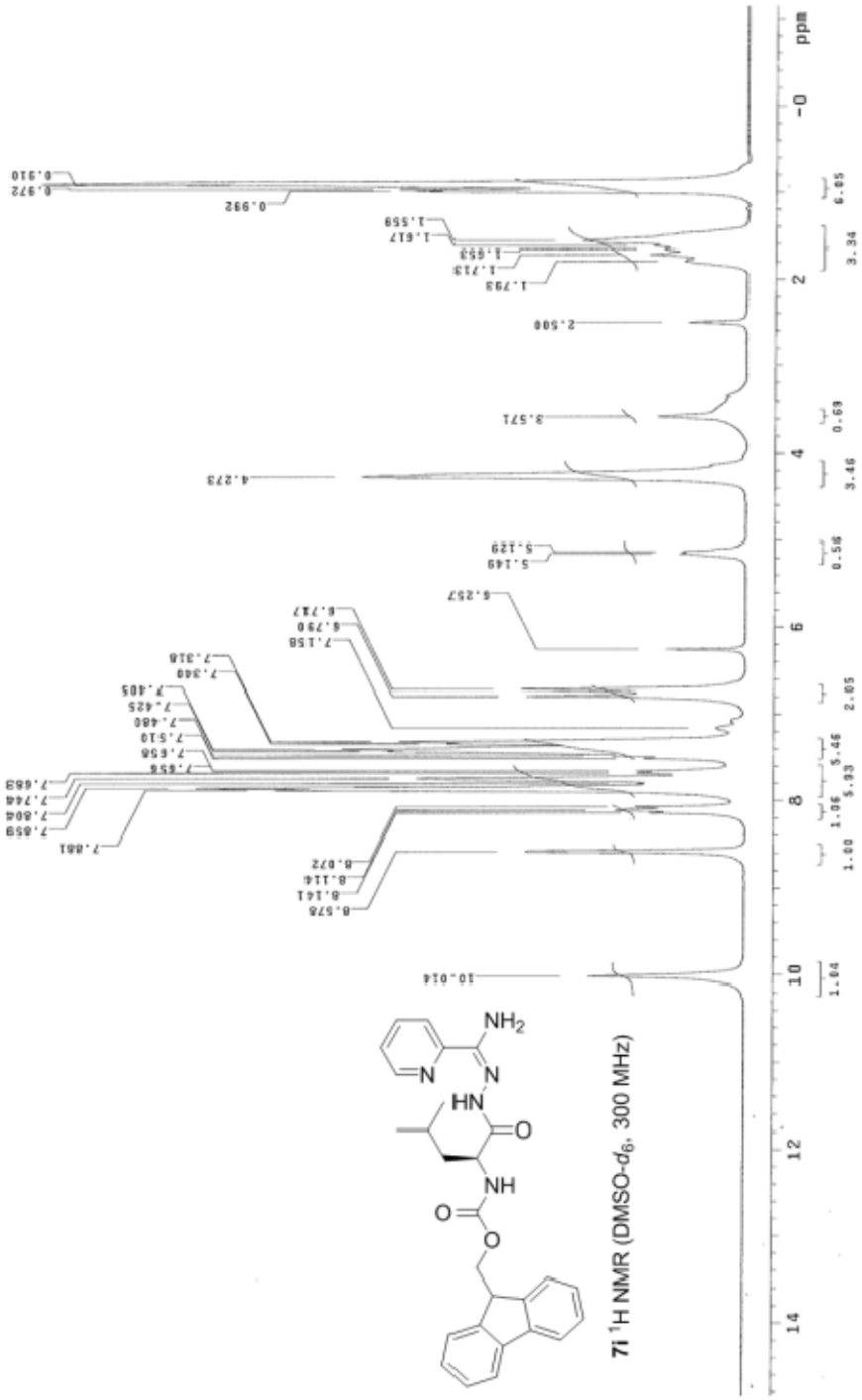
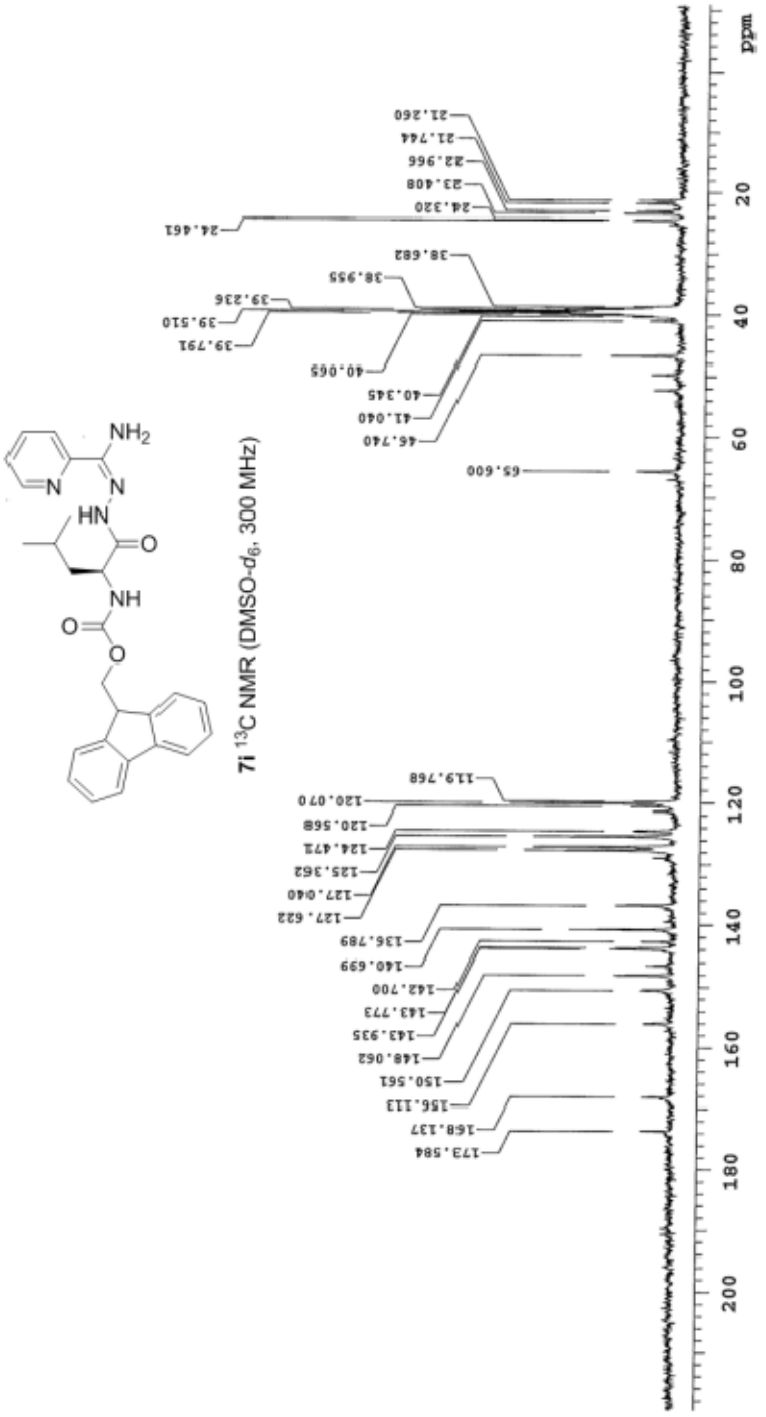
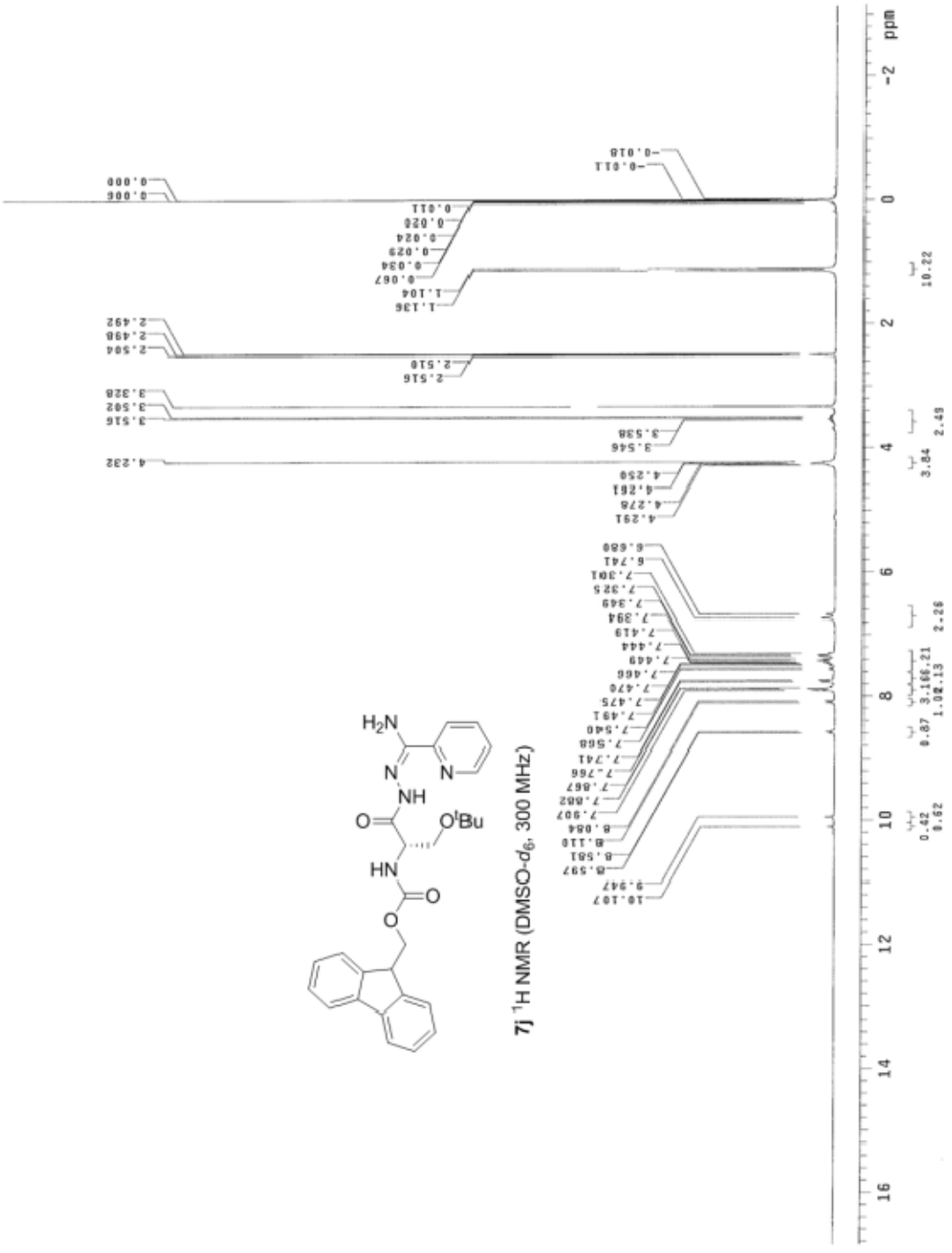
Solvent: DMSO
Ambient temperature
GEMINI-300BB "gemini300"
PULSE SEQUENCE
Pulse 43.2 degrees
Acq. time 1.800 sec
Width 17354.0 Hz
1232 repetitions
OBSERVE C13, 75.4523915 MHz
DECOUPLE H1, 300.0697779 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.5 Hz
FT size 65536
Total time 1 hr, 33 min, 48 sec
ppm


Fmoc-Gly-amidinopyridine
INDEX FREQUENCY PPM HEIGHT INDEX FREQUENCY PPM HEIGHT
1 3563.234 10.266 2.9 40 518.774 1.728 4.4
2 3337.434 10.120 21.1
3 2967.952 9.888 9.6
4 2573.046 8.572 22.2
5 2432.309 8.166 17.6
6 2425.287 8.080 20.2
7 2373.981 7.909 38.5
8 2366.945 7.886 52.2
9 2357.270 7.854 20.9
10 2351.114 7.833 12.1
11 2329.712 7.762 26.8
12 2324.435 7.744 37.1
13 2290.720 7.632 6.5
14 2286.029 7.616 7.1
15 2234.430 7.444 36.1
16 2228.869 7.426 55.9
17 2222.117 7.403 46.8
18 2210.390 7.364 34.1
19 2203.354 7.341 43.9
20 2196.804 7.319 21.9
21 2177.261 7.254 2.7
22 2170.518 7.231 3.1
23 2068.393 6.891 21.1
24 1998.132 6.657 33.9
25 1990.116 4.298 51.6
26 1983.373 4.276 28.4
27 1976.944 4.251 21.4
28 1266.662 4.220 18.8
29 1256.694 4.187 27.6
30 1251.124 4.168 29.1
31 1128.284 3.759 11.1
32 1124.179 3.745 12.9
33 1079.038 3.595 5.9
34 1063.685 3.544 20.5
35 1051.339 3.336 17.8
36 998.648 3.320 6.0
37 750.382 2.500 14.3
38 525.224 1.750 6.4
39 522.282 1.740 6.3
7h ^{1}H NMR (DMSO-d_6, 300 MHz)
H_2N
N
N
NH
O
HN
O
O
14 12 10 8 6 4 2 −0 ppm
0.72 0.30 1.00 3.16.47 1.601.795.77 2.02 0.55 4.84 6.25

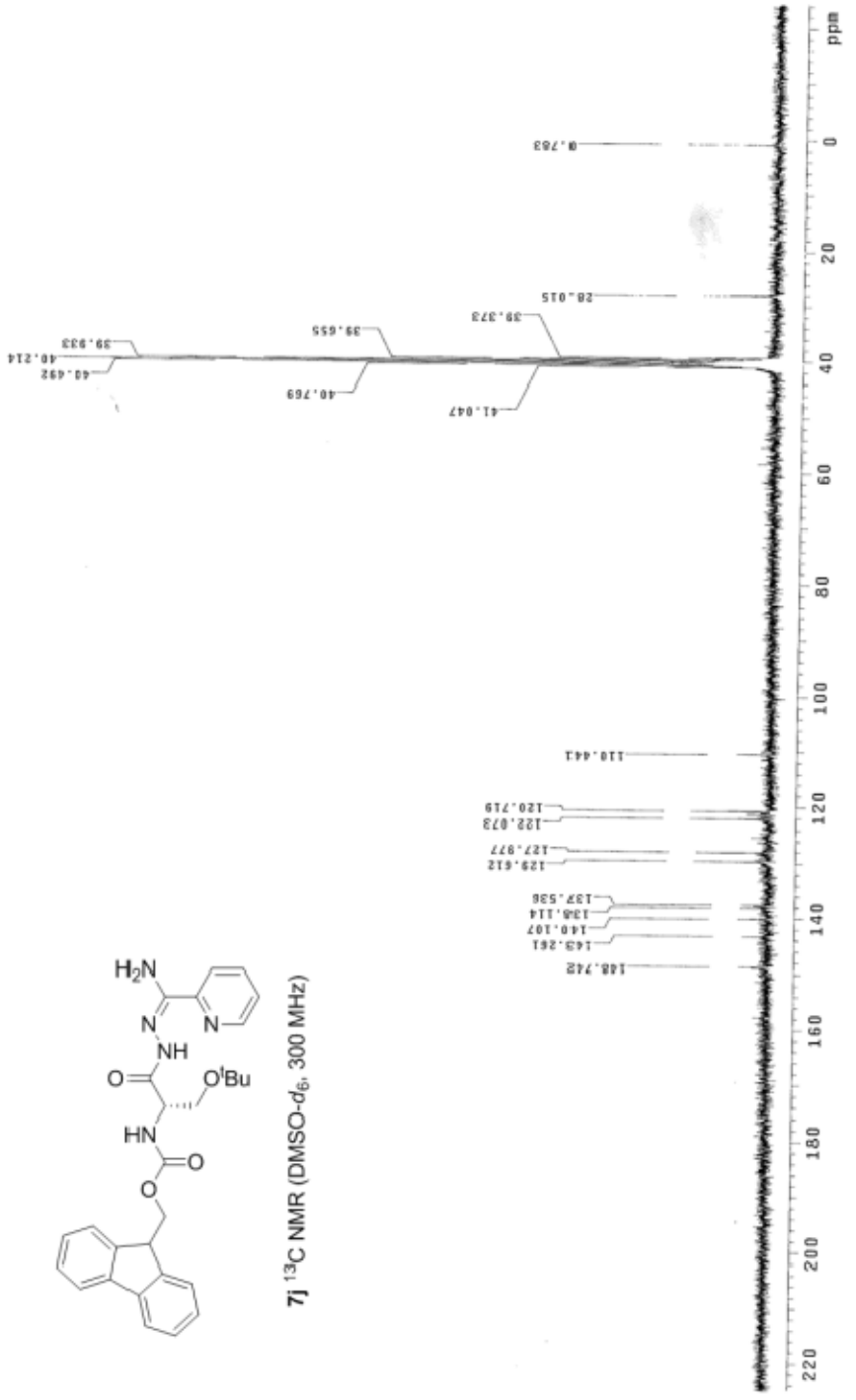

7h ^{13}C NMR (DMSO-d_6, 300 MHz)
ppm


7i ^{1}H NMR (DMSO-d_6, 300 MHz)
NH2
N
N
HN
O
NH
O
O
ppm

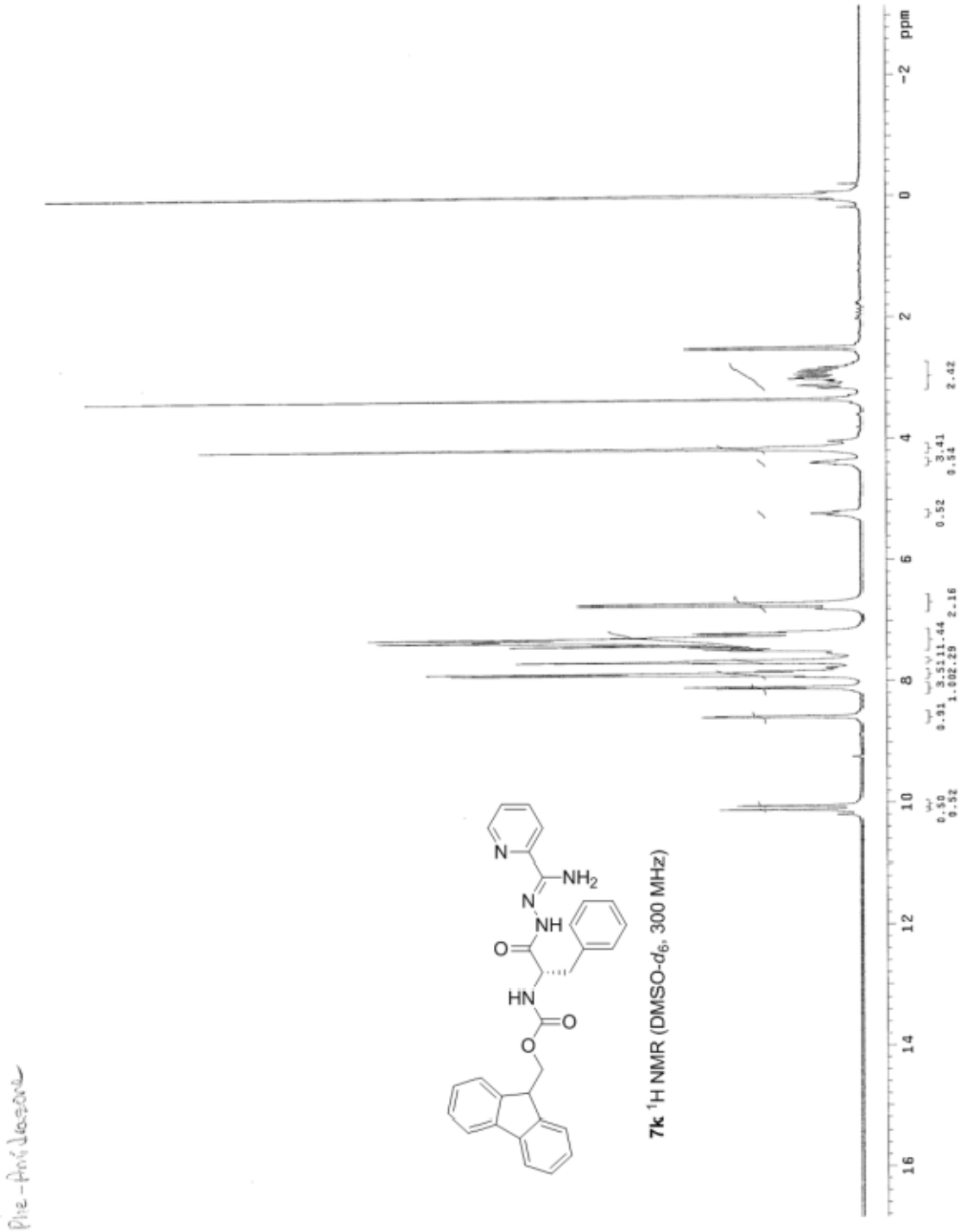

7l ^{13}C NMR (DMSO-d_6, 300 MHz)
NH2
N
N
HN
O
NH
O
O
ppm
20
40
60
80
100
120
140
160
180
200
21.260
21.744
22.966
23.408
24.320
24.461
38.682
38.955
39.236
39.510
39.791
40.065
40.345
41.040
46.740
65.600
119.768
120.070
120.568
124.473
125.362
127.040
127.622
136.789
140.699
142.700
143.773
143.935
148.062
150.561
156.113
168.137
173.584



7j ^{1}H NMR (DMSO-d_6, 300 MHz)

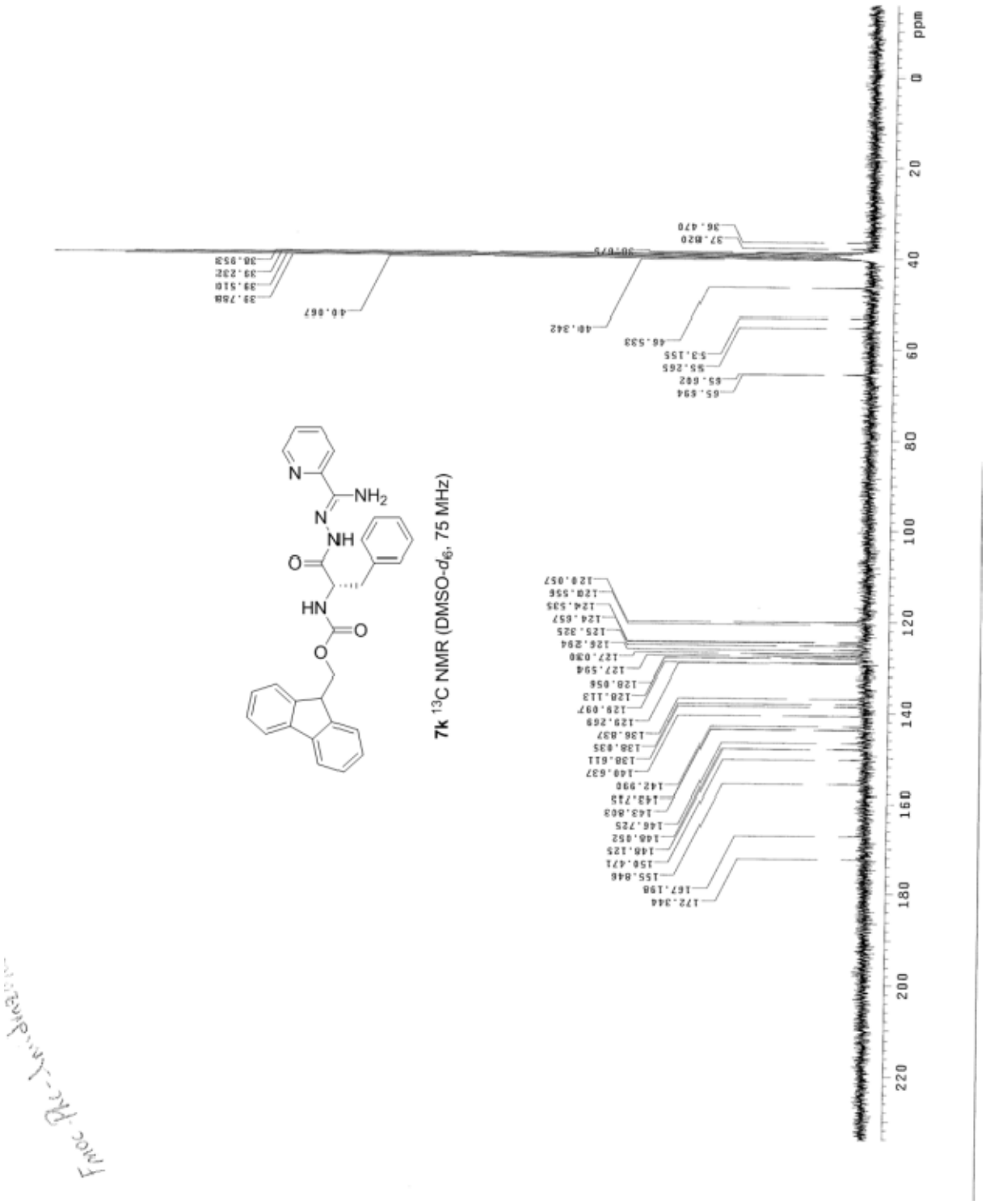


7j ^{13}C NMR (DMSO-d_6, 300 MHz)

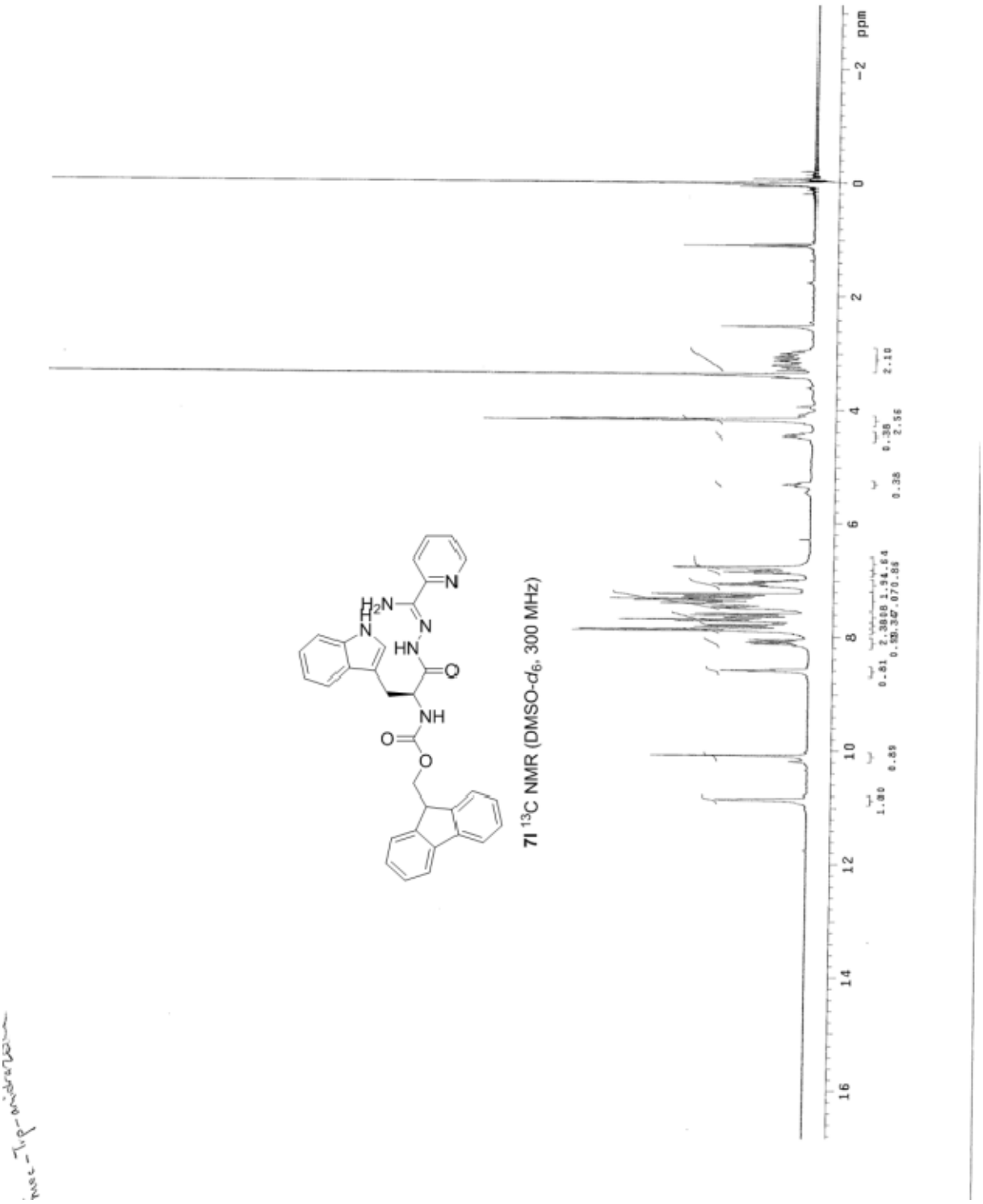


7k ^{1}H NMR (DMSO-d_6, 300 MHz)

Fmoc-Phe-Phe-Hexene



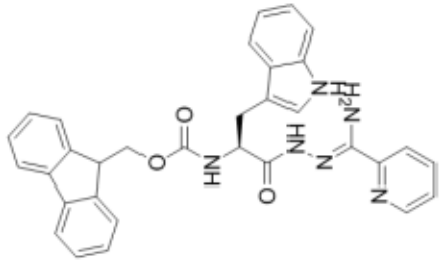
7k ^{13}C NMR (DMSO-d_6, 75 MHz)



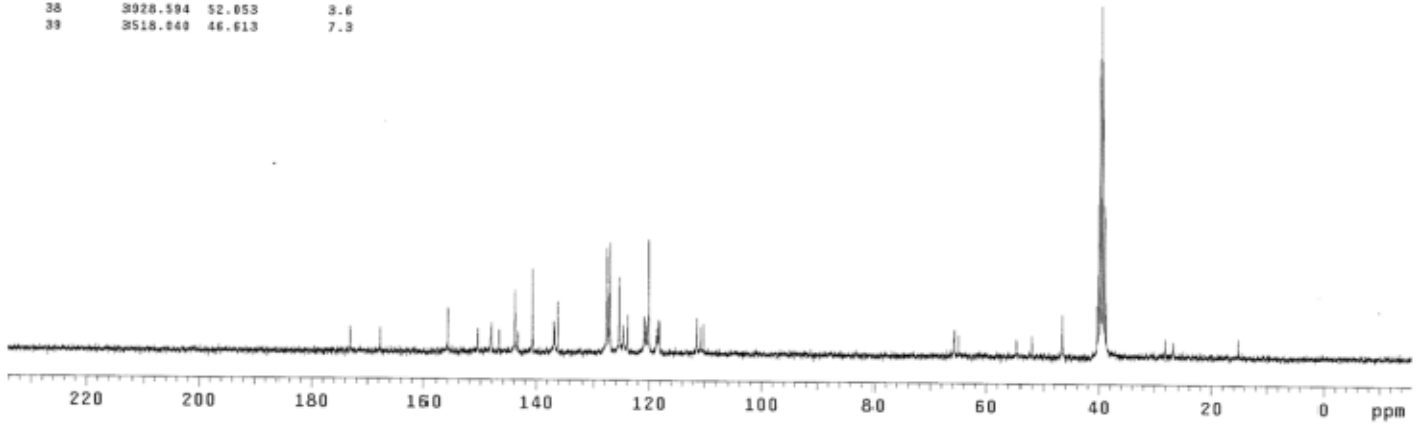
7l ^{13}C NMR (DMSO-d_6, 300 MHz)

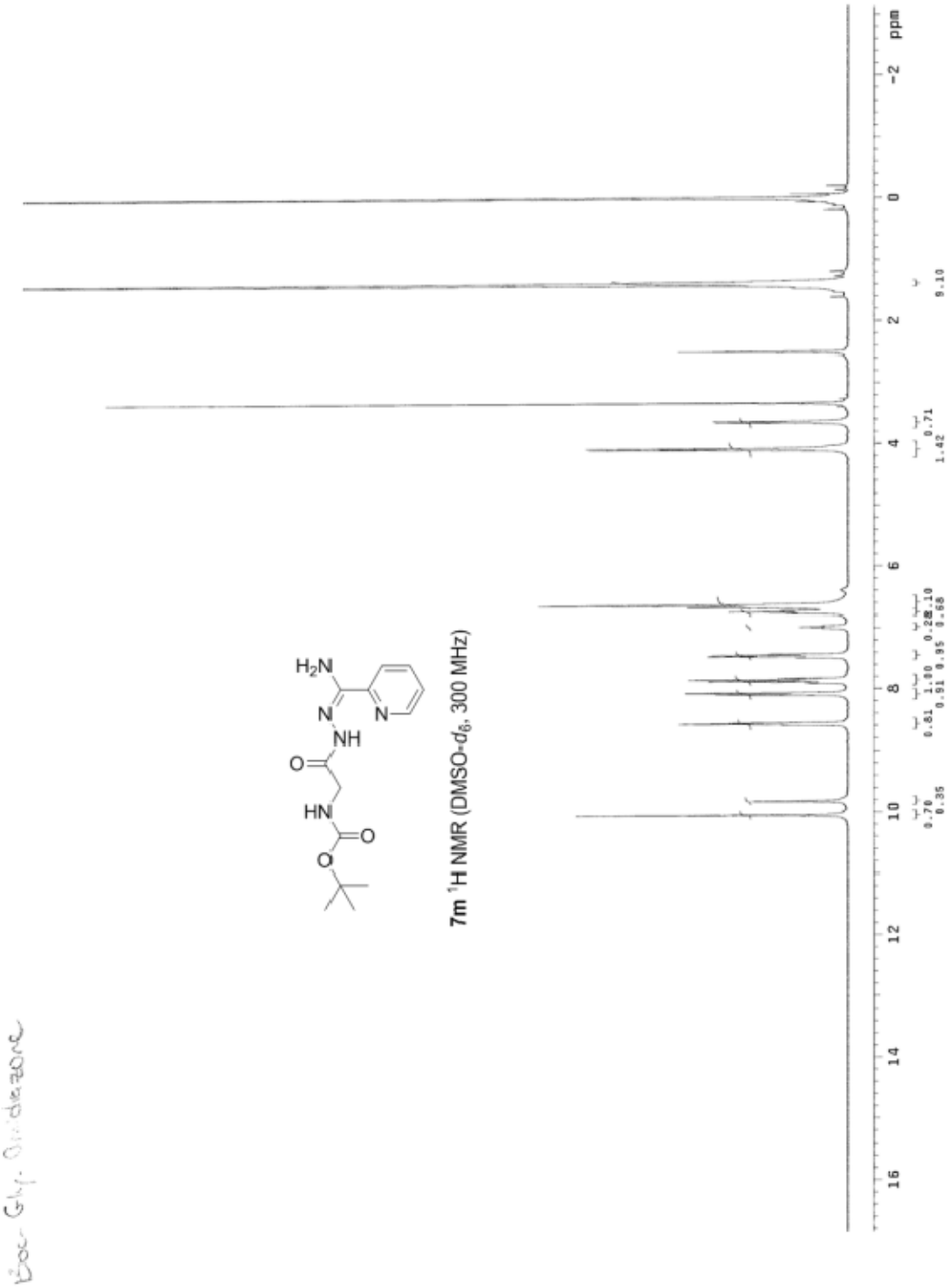
Fmoc-Trp-amidrazone

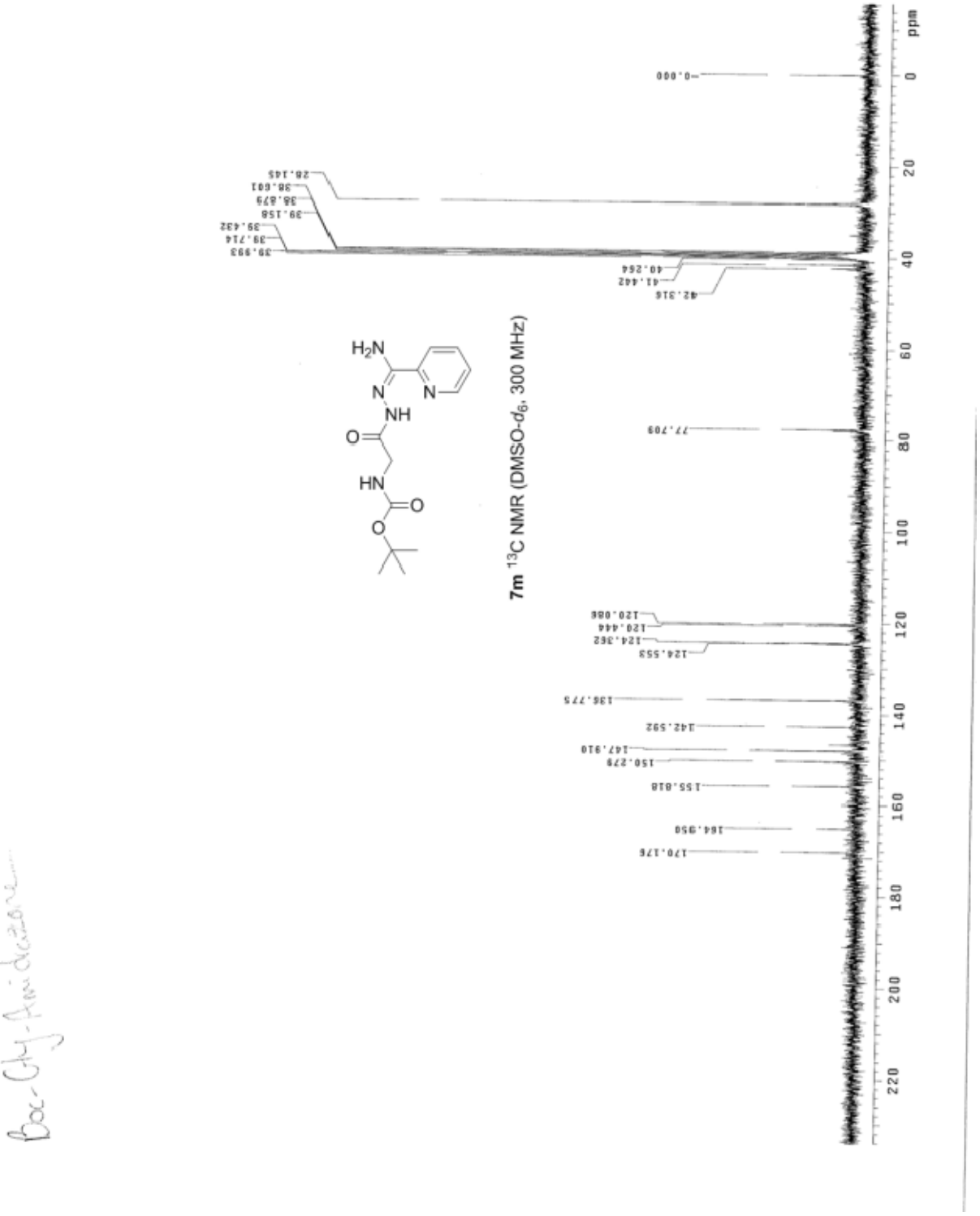
INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	13860.975	173.054	4.4	40	3044.722	40.342	9.0
2	12657.330	167.706	4.2	41	3023.785	40.063	27.0
3	11765.973	155.895	7.8	42	3002.688	39.785	52.7
4	11362.041	150.543	4.0	43	2981.671	39.506	62.8
5	11357.147	150.479	3.9	44	2960.654	39.228	52.2
6	11180.948	148.144	4.3	45	2939.637	38.949	26.7
7	11174.038	148.052	5.2	46	2918.987	38.675	9.6
8	11069.240	146.664	3.9	47	2115.074	28.024	3.3
9	10856.766	143.849	10.6	48	2029.566	26.891	2.7
10	10852.735	143.795	7.1	49	1143.967	15.157	3.3
11	10813.868	143.280	3.6				
12	10616.076	140.660	14.6				
13	10327.019	136.830	5.5				
14	10315.214	136.673	4.6				
15	10270.877	136.086	9.1				
16	9630.862	127.606	18.4				
17	9607.829	127.301	10.5				
18	9598.267	127.068	19.5				
19	9463.013	125.382	13.2				
20	9407.159	124.642	4.9				
21	9359.441	123.890	6.8				
22	9123.859	120.888	6.5				
23	9119.828	120.835	6.4				
24	9101.690	120.594	5.7				
25	9082.976	120.347	4.9				
26	9061.384	120.060	20.0				
27	8954.570	118.645	4.2				
28	8938.448	118.432	4.5				
29	8923.764	118.237	5.9				
30	8913.400	118.100	5.5				
31	8402.367	111.329	6.2				
32	8349.680	110.631	4.6				
33	8315.995	110.184	5.2				
34	4961.889	65.743	3.7				
35	4953.543	65.633	4.5				
36	4898.838	64.908	3.6				
37	4112.854	54.494	2.9				
38	3928.594	52.053	3.6				
39	3518.840	46.613	7.3				



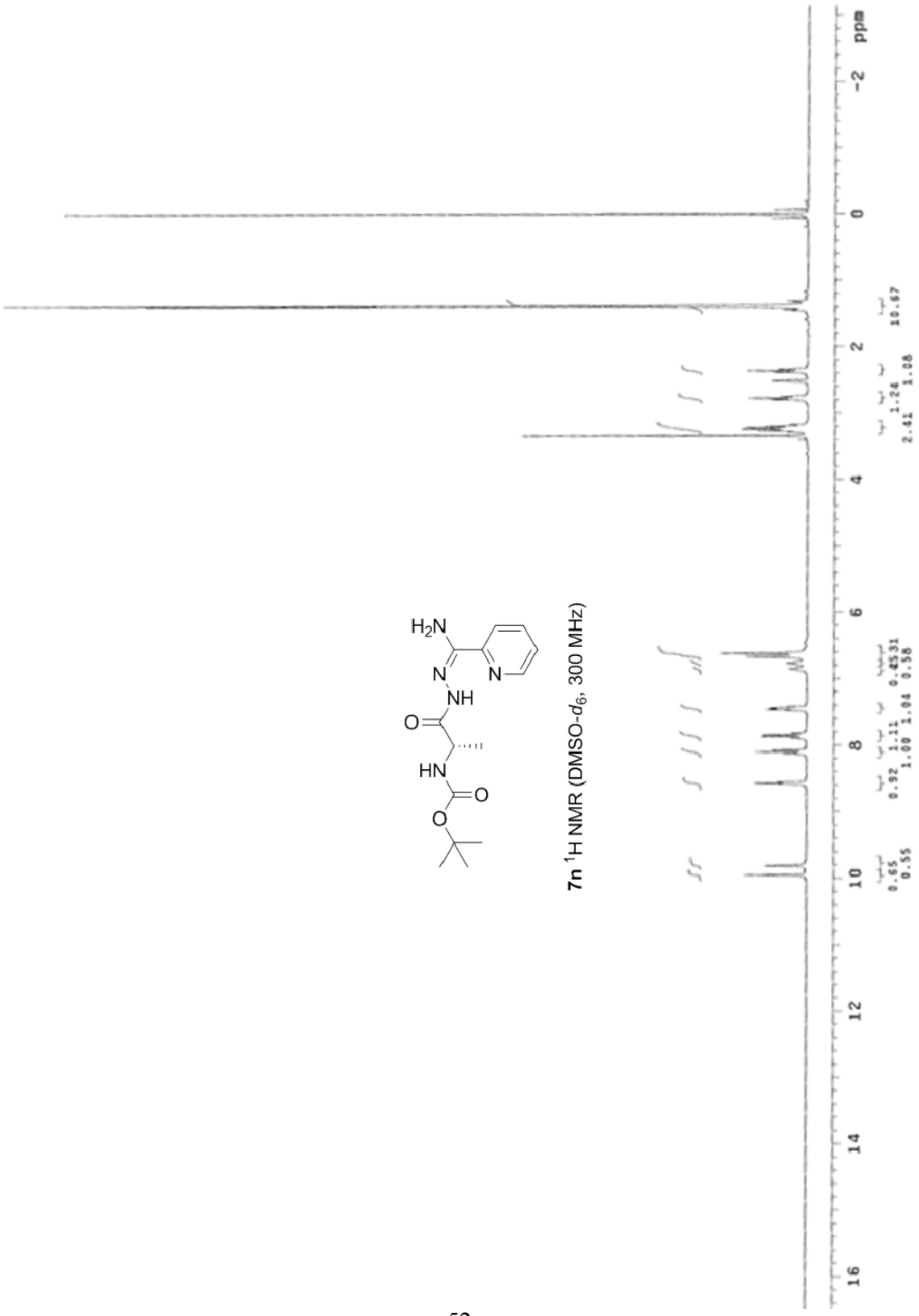
7l ^{1}H NMR (DMSO-d_6, 300 MHz)



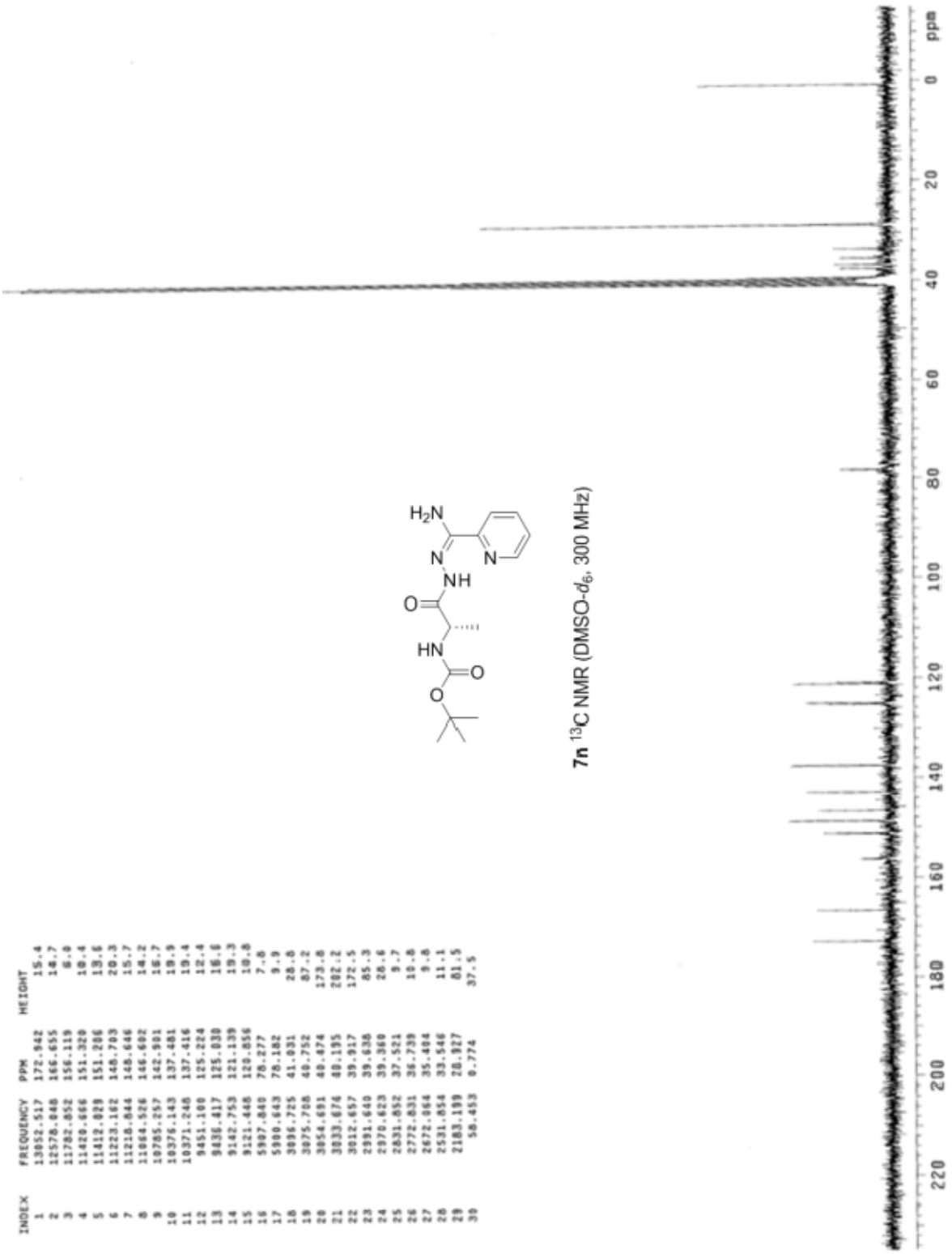

Boc-Gly-Oxidazole
7m ^{1}H NMR (DMSO-d_6, 300 MHz)
H_2N
N
N
NH
O
HN
O
O
ppm
16
14
12
10
8
6
4
2
0
-2



Boc-Gly-Ami dazone

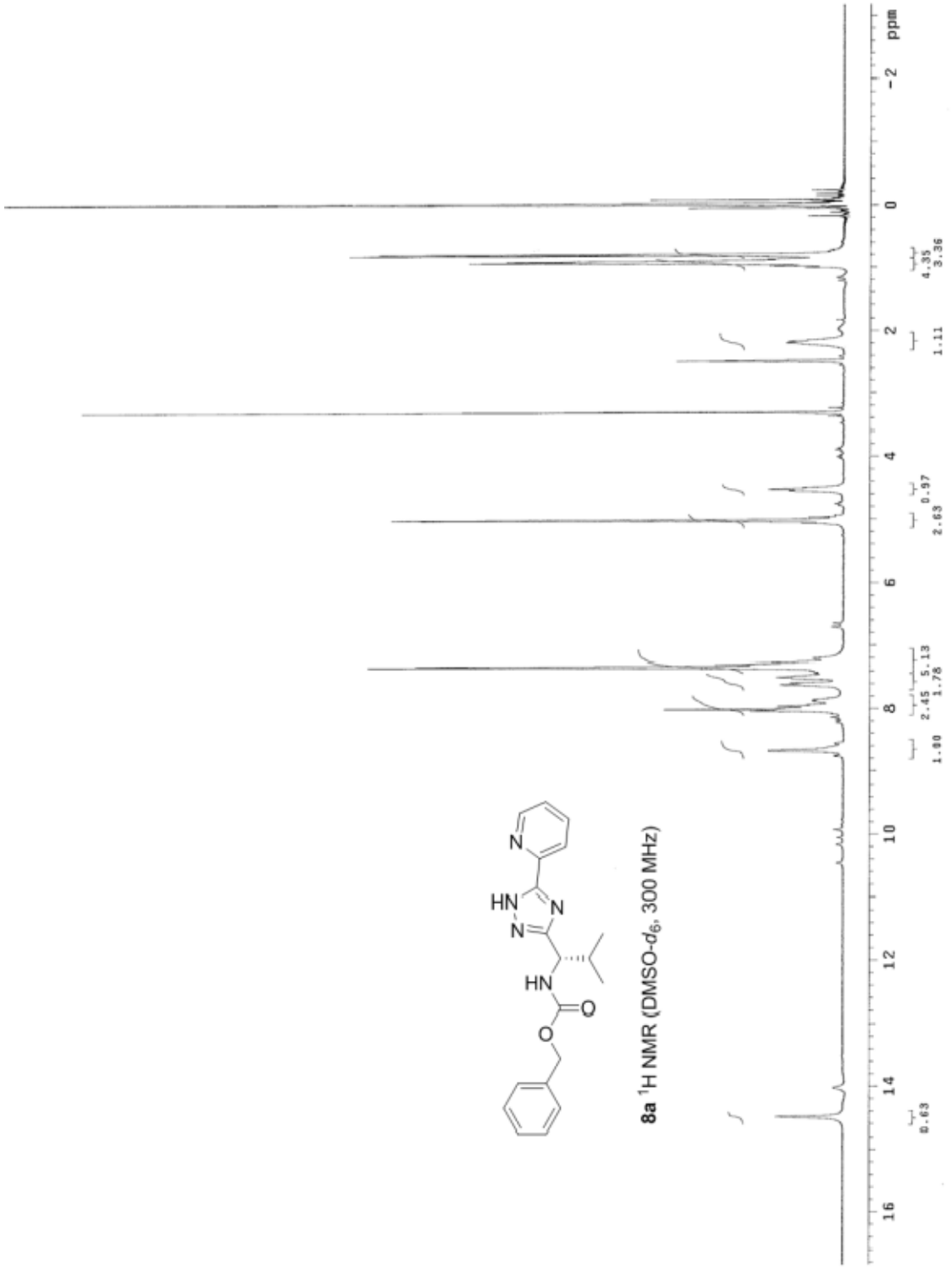


7n ^{1}H NMR (DMSO-d_6, 300 MHz)

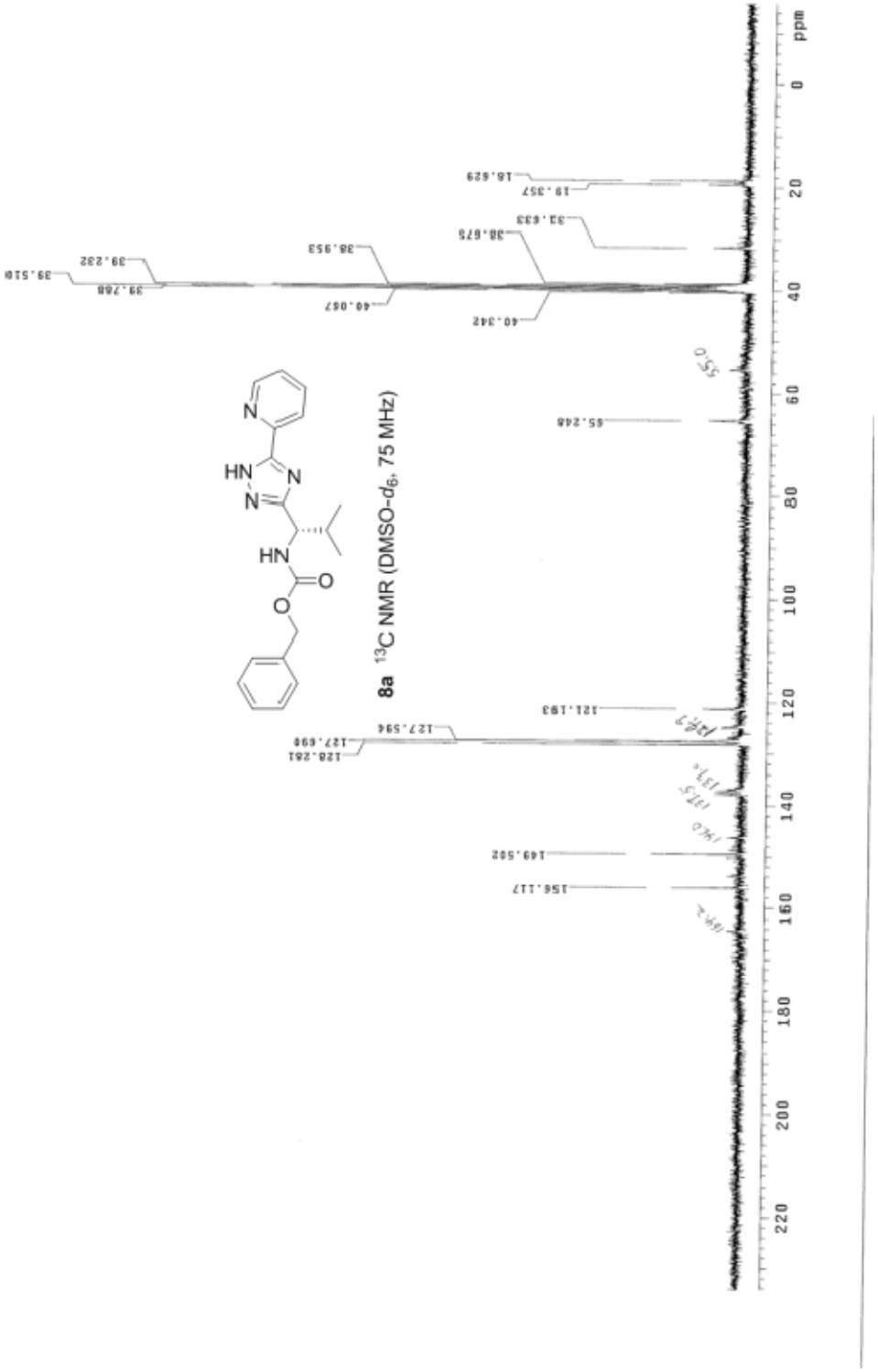


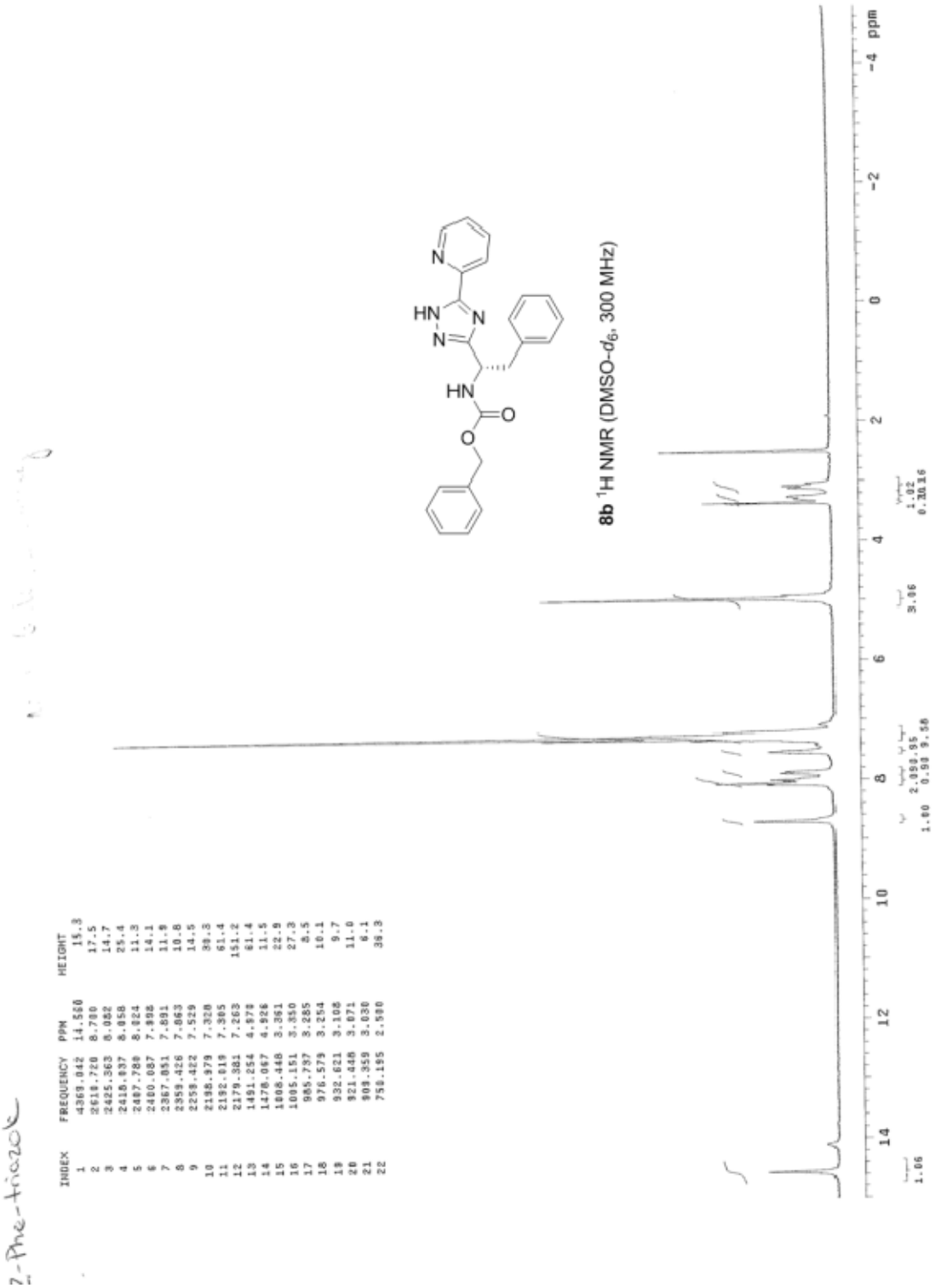
INDEX	FREQUENCY	PPM	HEIGHT
1	13052.517	172.942	15.4
2	12578.848	166.655	14.7
3	11782.852	156.119	6.8
4	11420.666	151.320	10.4
5	11412.828	151.206	13.6
6	11223.162	148.703	20.3
7	11210.844	148.646	15.7
8	11064.526	146.602	14.2
9	10785.257	142.901	16.7
10	10376.143	137.481	19.9
11	10371.246	137.416	19.4
12	9451.100	125.224	12.4
13	9438.417	125.030	16.6
14	9142.753	121.139	19.3
15	9121.446	120.856	10.8
16	5907.840	78.277	7.8
17	5900.643	78.182	9.9
18	3096.725	41.031	28.6
19	3075.708	40.752	87.2
20	3054.691	40.474	173.8
21	3033.674	40.195	282.2
22	3012.657	39.917	172.5
23	2991.640	39.638	85.3
24	2970.623	39.360	28.6
25	2831.852	37.521	9.7
26	2772.831	36.739	10.8
27	2672.064	35.404	9.8
28	2531.854	33.546	11.1
29	2183.199	28.927	81.5
30	58.453	0.774	37.5

7n ^{13}C NMR (DMSO-d_6, 300 MHz)

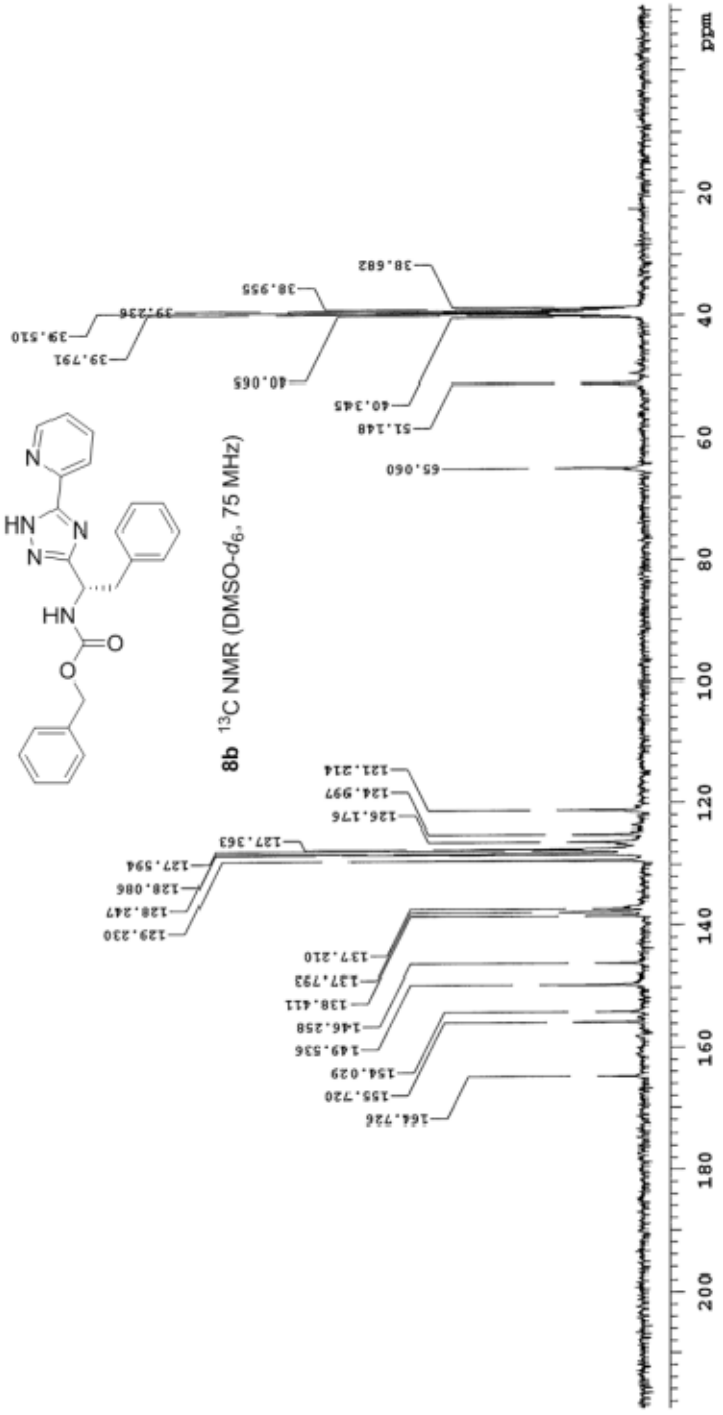


8a ^{1}H NMR (DMSO-d_6, 300 MHz)

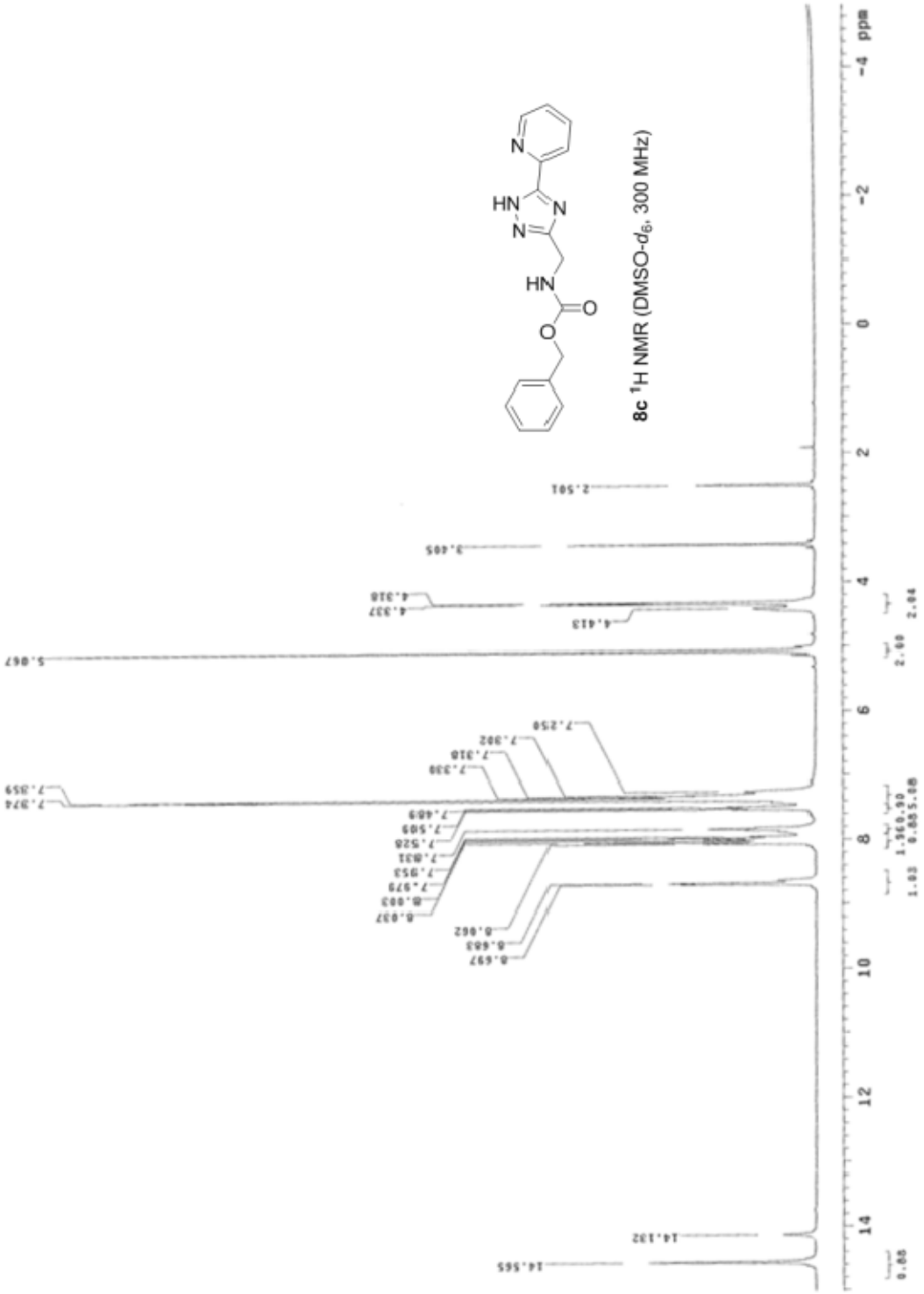

8a ^{13}C NMR (DMSO-d_6, 75 MHz)
HN
N
N
HN
O
O
ppm
0
20
40
60
80
100
120
140
160
180
200
220
16.629
19.357
31.633
38.675
38.953
39.232
39.510
39.788
40.057
40.342
55.246
121.183
127.594
127.698
128.261
169.582
156.117



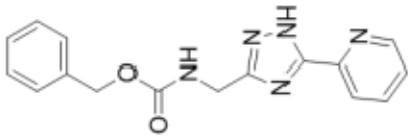
INDEX	FREQUENCY	PPM	HEIGHT
1	4369.042	14.560	15.3
2	2618.720	8.730	17.5
3	2425.363	8.082	14.7
4	2418.037	8.058	25.4
5	2407.788	8.024	11.3
6	2400.087	7.998	14.1
7	2367.051	7.891	11.9
8	2359.426	7.863	10.8
9	2259.422	7.529	14.5
10	2198.979	7.328	38.3
11	2192.619	7.305	61.4
12	2179.381	7.263	151.2
13	1491.254	4.971	61.4
14	1478.067	4.926	11.5
15	1008.448	3.361	22.9
16	1005.151	3.350	27.3
17	985.737	3.285	8.5
18	976.579	3.254	10.1
19	932.621	3.108	9.7
20	921.448	3.071	11.0
21	908.359	3.030	6.1
22	756.195	2.590	36.3



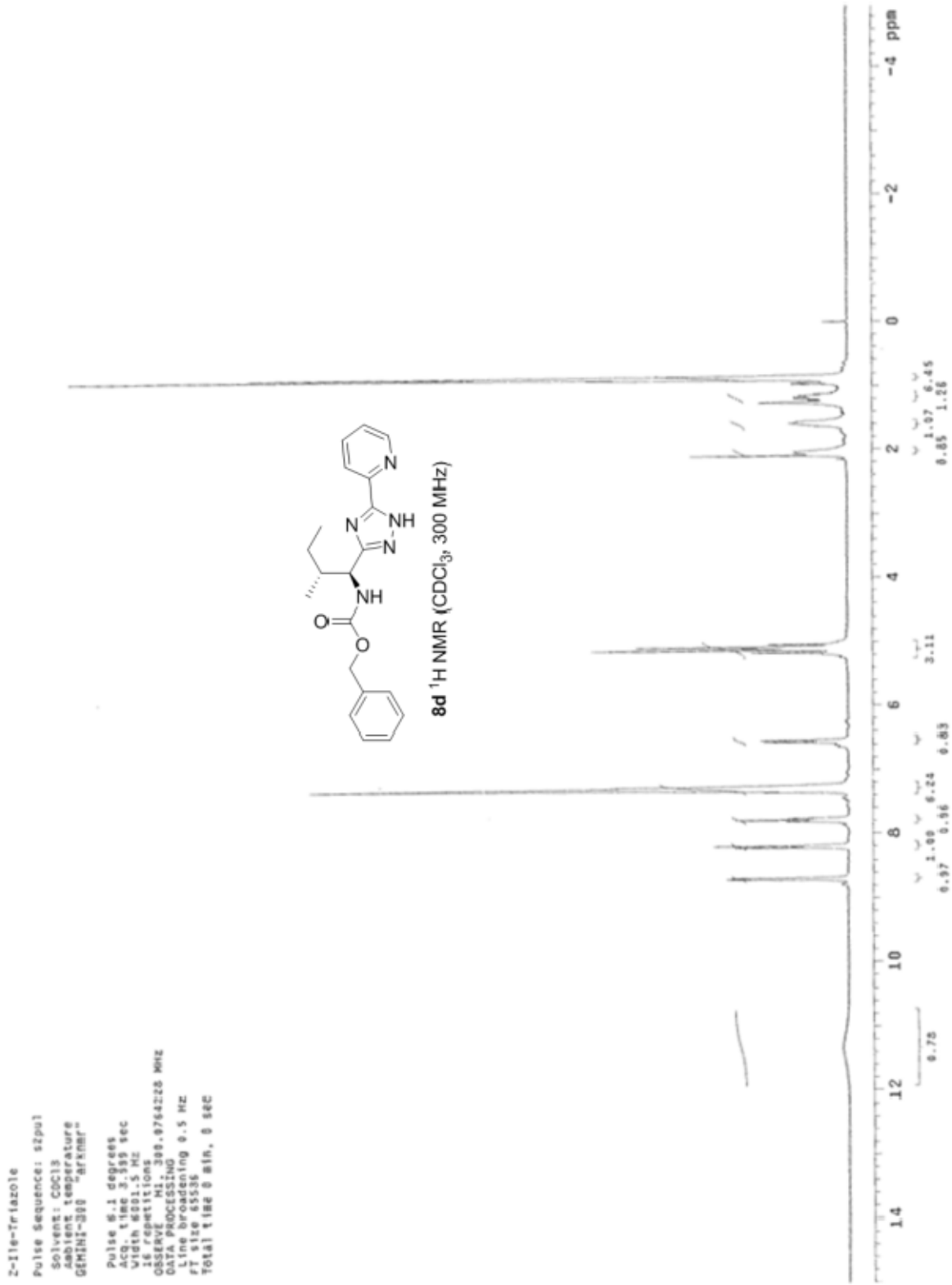
Z-Phe-triazole


8c ^{1}H NMR (DMSO-d_6, 300 MHz)
N
HN
N
N
HN
O
O
2.501
3.405
4.318
4.337
4.413
5.067
7.250
7.302
7.318
7.330
7.359
7.374
7.469
7.509
7.526
7.831
7.953
7.979
8.003
8.037
8.662
8.683
8.697
14.132
14.565
ppm

INDEX	FREQUENCY	PPM	HEIGHT
1	12217.641	161.925	8.5
2	11791.299	156.275	10.6
3	11629.236	154.127	9.3
4	11281.808	149.522	29.5
5	11033.417	146.230	9.0
6	10396.818	137.793	23.4
7	10349.153	137.161	9.0
8	9682.365	128.324	66.0
9	9637.348	127.728	57.8
10	9430.797	124.990	20.1
11	9141.097	121.151	22.8
12	4932.762	65.376	19.2
13	3044.148	40.345	11.1
14	3022.964	40.065	33.4
15	3002.309	39.791	63.9
16	2981.124	39.510	74.3
17	2959.939	39.229	63.3
18	2939.284	38.955	31.8
19	2918.100	38.675	11.5
20	2896.385	38.387	15.8



8c ^{13}C NMR (DMSO-d_6, 75 MHz)

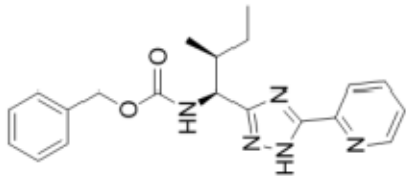

2-Ile-Triazole
Pulse sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300 "arkmar"
Pulse 6.1 degrees
Acq. time 3.999 sec
Width 6001.5 Hz
16 repetitions
OBSERVE H1, 300.0764228 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 0 min, 0 sec
N
NH
N
N
NH
O
O
8d ^{1}H NMR (CDCl$_3$, 300 MHz)
14
12
10
8
6
4
2
0
-2
-4 ppm
0.75
1.00
0.97
6.24
0.96
0.83
3.11
1.07
0.85
6.45
1.26

Ile-~~N2H3~~triazole

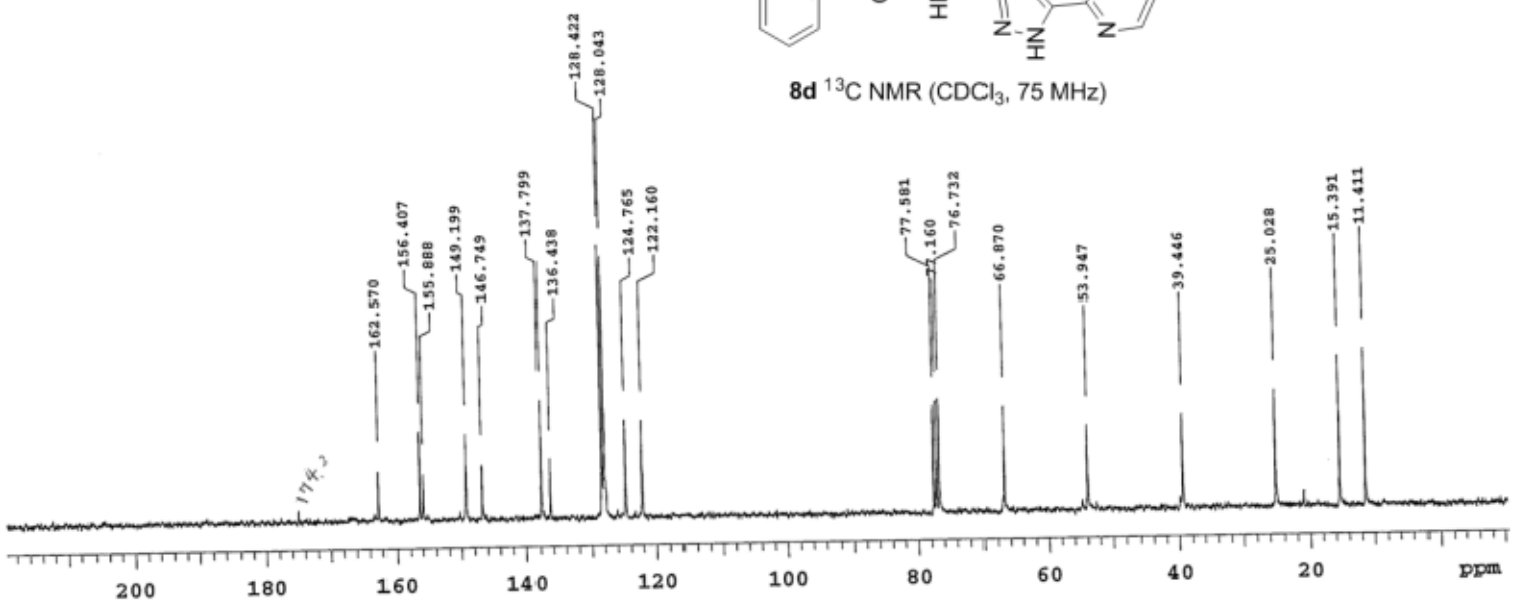
ılse Sequence: s2pul

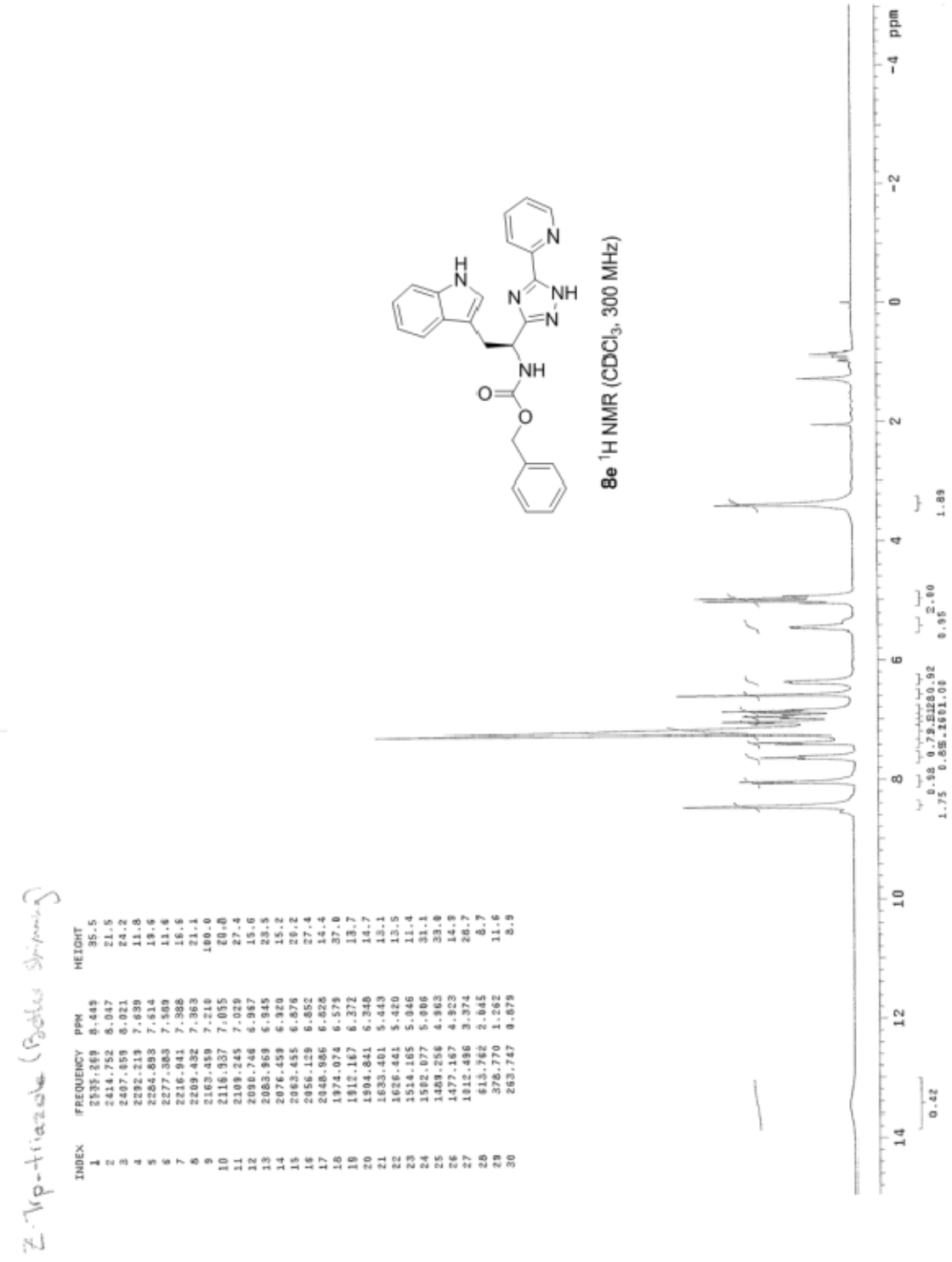
Solvent: CDCl3
Ambient temperature
INOVA-300BB "gemini300"

PULSE SEQUENCE
Pulse 43.2 degrees
Acq. time 1.800 sec
Width 17354.0 Hz
168 repetitions
OBSERVE C13, 75.4519961 MHz
DECOUPLE H1, 300.0683526 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.5 Hz
FT size 65536
Total time 46 min, 54 sec

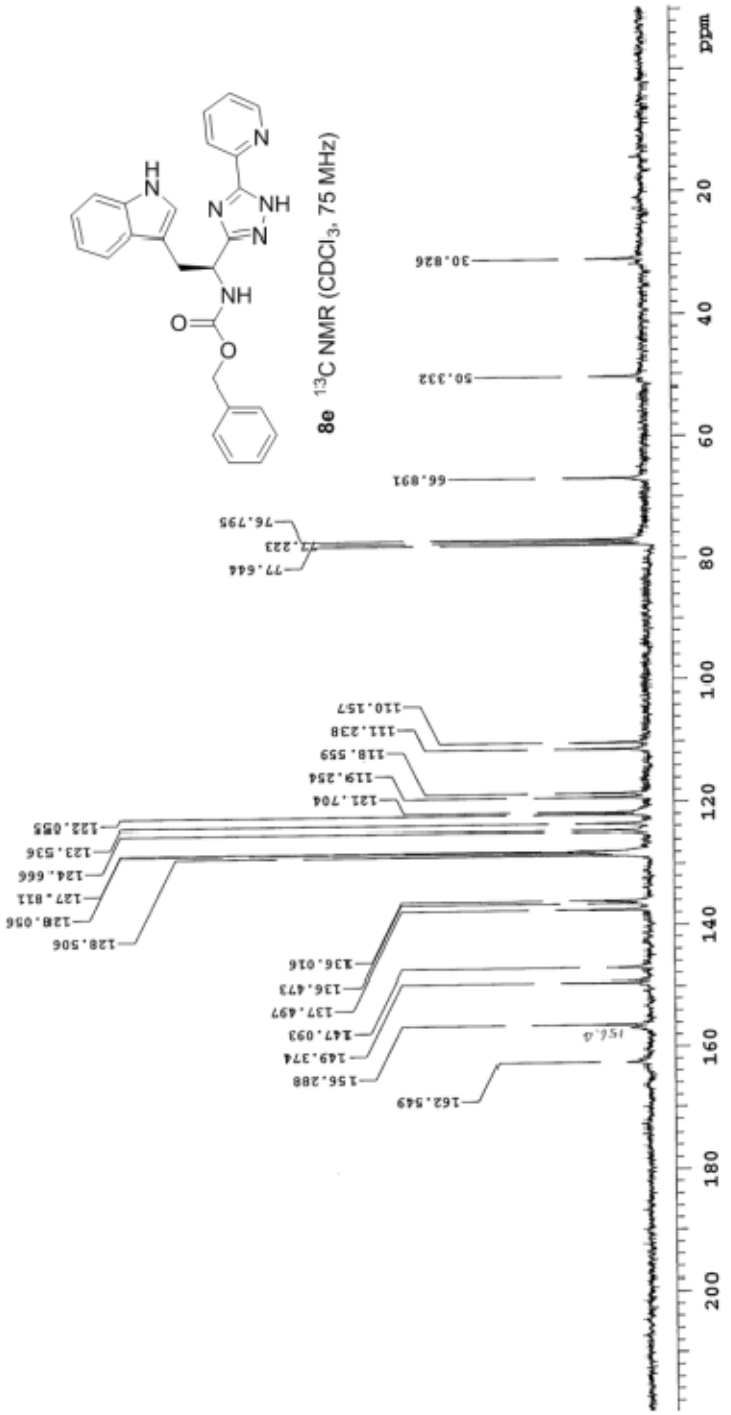


8d ^{13}C NMR ($CDCl_3$, 75 MHz)

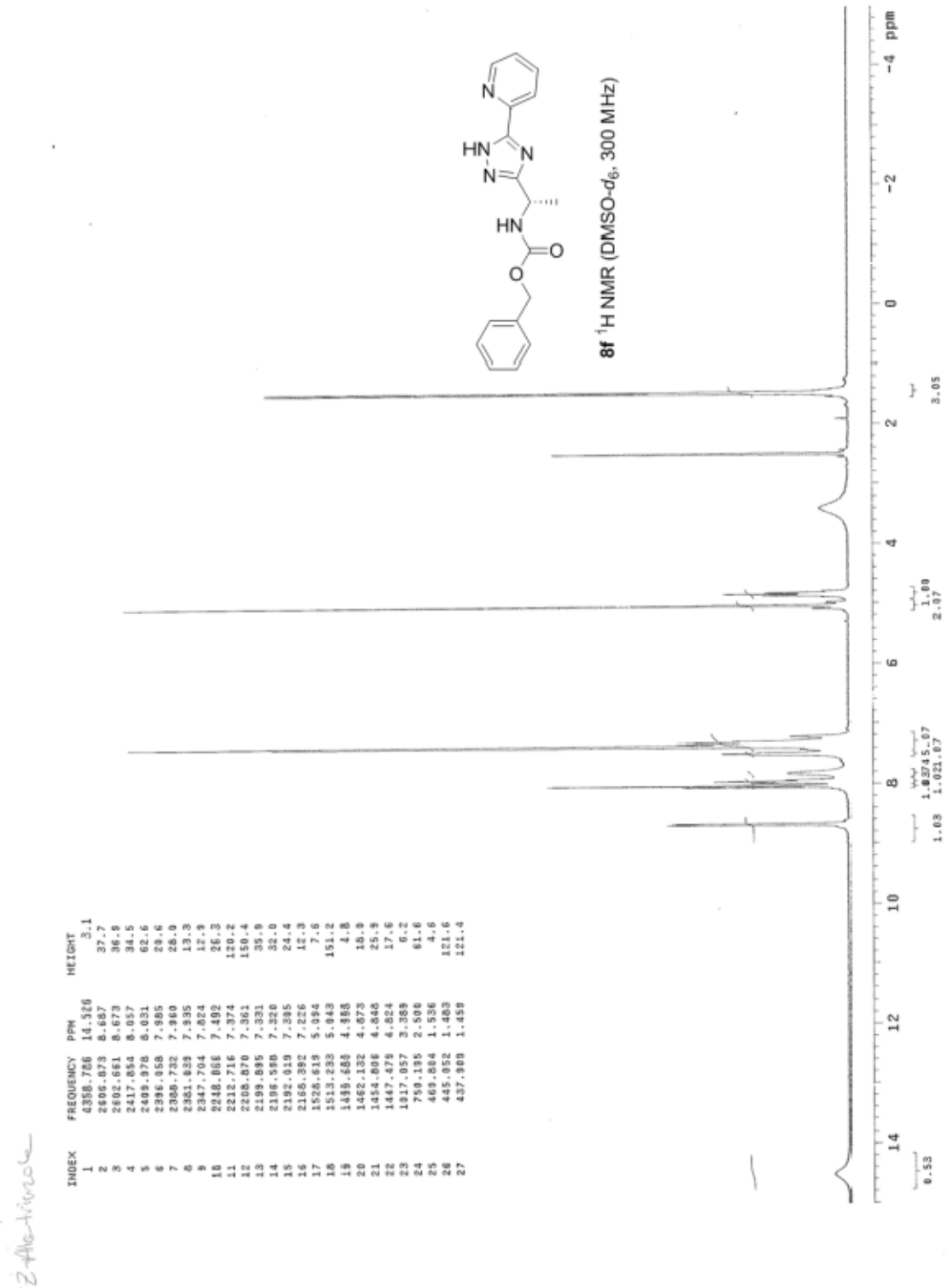




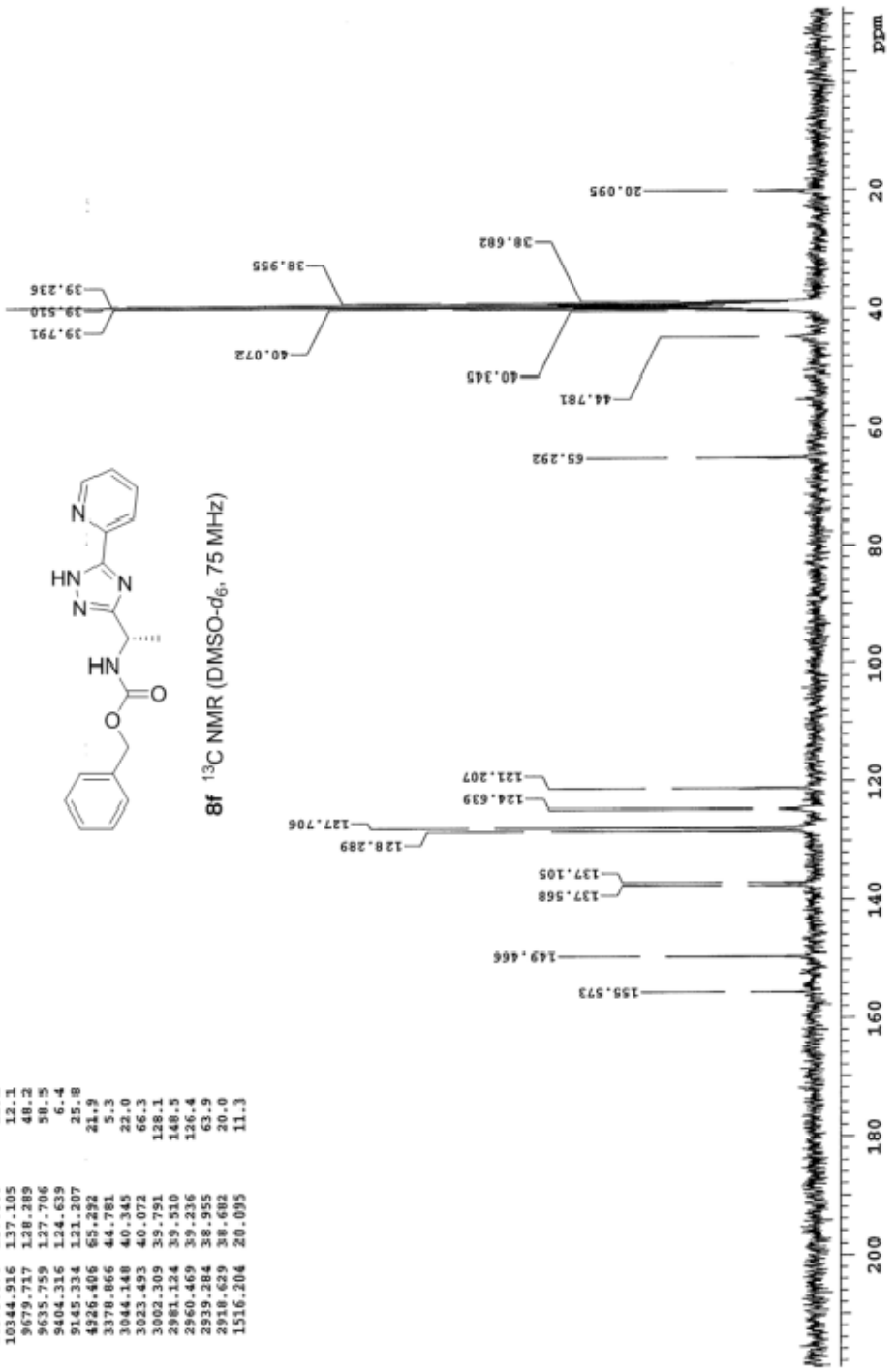
INDEX	FREQUENCY	PPM	HEIGHT
1	2535.269	8.445	35.5
2	2414.752	8.047	21.5
3	2407.659	8.021	24.2
4	2292.219	7.639	11.8
5	2284.893	7.614	19.6
6	2277.383	7.589	11.6
7	2216.941	7.388	16.6
8	2209.432	7.363	21.1
9	2163.459	7.210	100.0
10	2116.937	7.055	29.8
11	2109.245	7.029	27.4
12	2090.746	6.967	15.6
13	2083.969	6.945	23.5
14	2076.459	6.920	15.2
15	2063.455	6.876	29.2
16	2056.129	6.852	27.4
17	2048.986	6.828	14.4
18	1974.074	6.579	37.0
19	1912.167	6.372	13.7
20	1904.841	6.348	14.7
21	1633.401	5.443	13.1
22	1626.461	5.420	13.5
23	1514.165	5.046	11.4
24	1502.077	5.006	31.1
25	1489.256	4.963	33.8
26	1477.167	4.923	14.9
27	1012.496	3.374	26.7
28	613.762	2.045	8.7
29	378.770	1.262	11.6
30	263.747	0.879	8.9



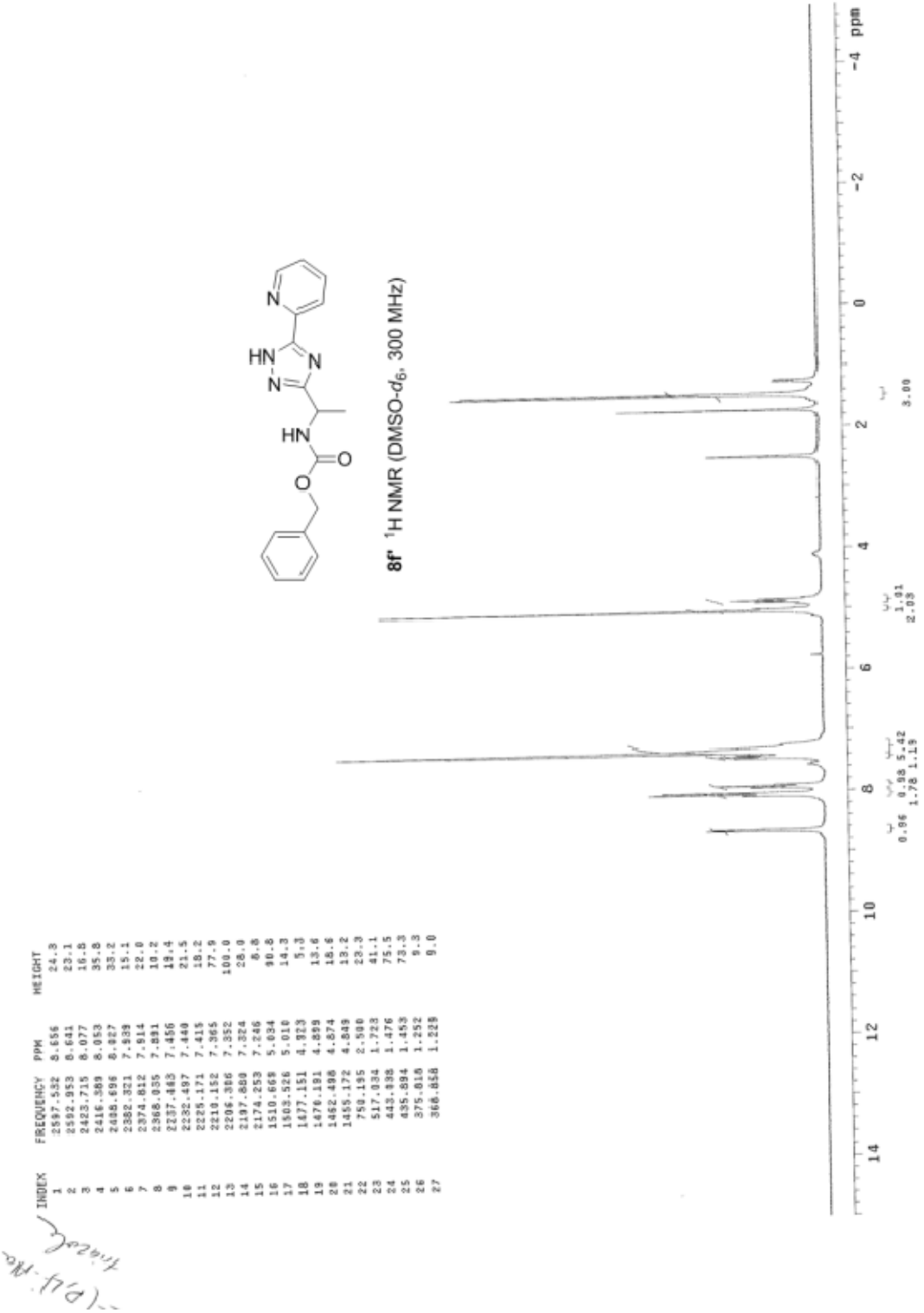
Z-Trp-Triazole



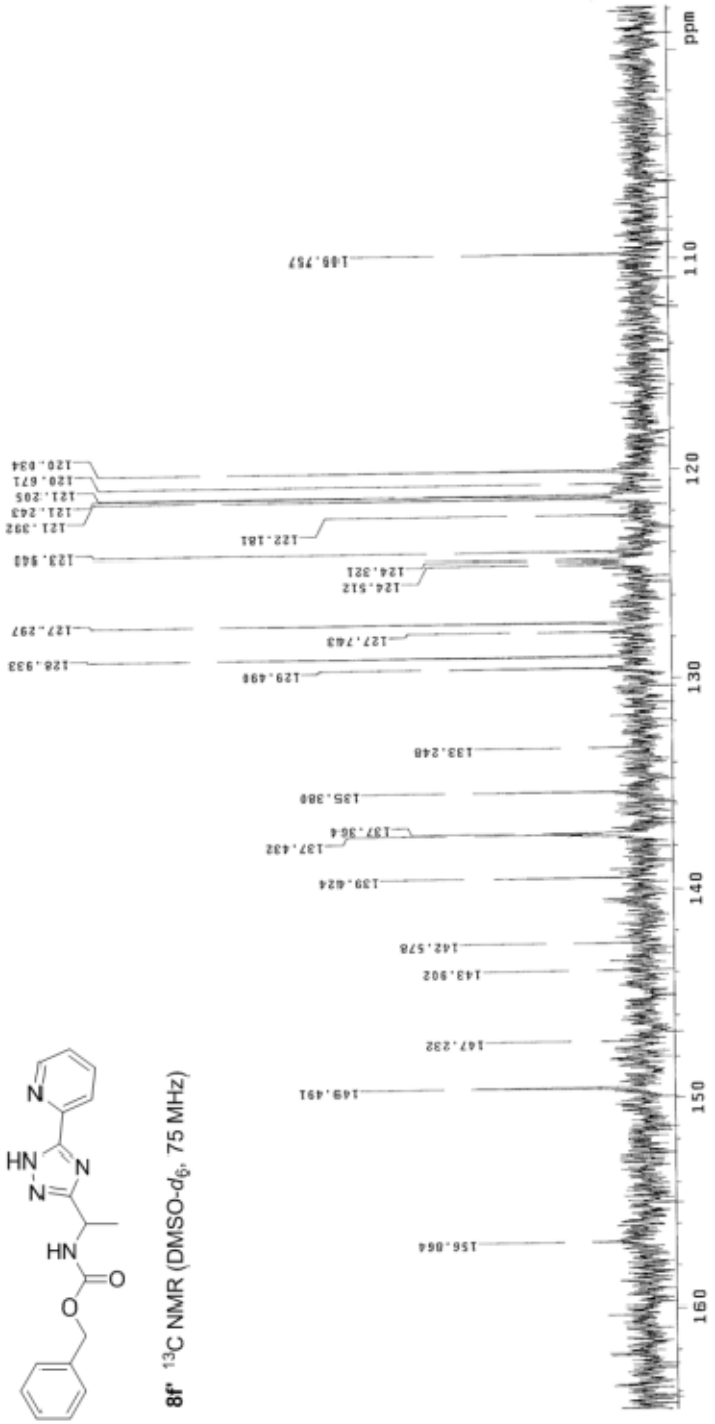
INDEX	FREQUENCY	PPM	HEIGHT
1	4358.786	14.526	3.1
2	2606.373	8.687	37.7
3	2602.661	8.673	36.8
4	2417.654	8.057	34.5
5	2409.378	8.031	62.6
6	2396.958	7.985	29.6
7	2388.732	7.960	28.0
8	2381.829	7.935	13.3
9	2347.704	7.824	12.9
10	2248.086	7.492	26.3
11	2212.716	7.374	120.2
12	2208.870	7.361	150.4
13	2199.895	7.331	35.9
14	2196.580	7.320	32.0
15	2192.019	7.305	24.4
16	2168.382	7.226	12.3
17	1528.618	5.094	7.6
18	1513.238	5.043	151.2
19	1439.680	4.388	1.8
20	1462.132	4.873	18.0
21	1454.806	4.848	25.9
22	1447.479	4.824	17.6
23	1017.057	3.389	6.2
24	750.195	2.500	61.6
25	469.884	1.536	4.6
26	445.052	1.483	121.6
27	437.909	1.459	121.4



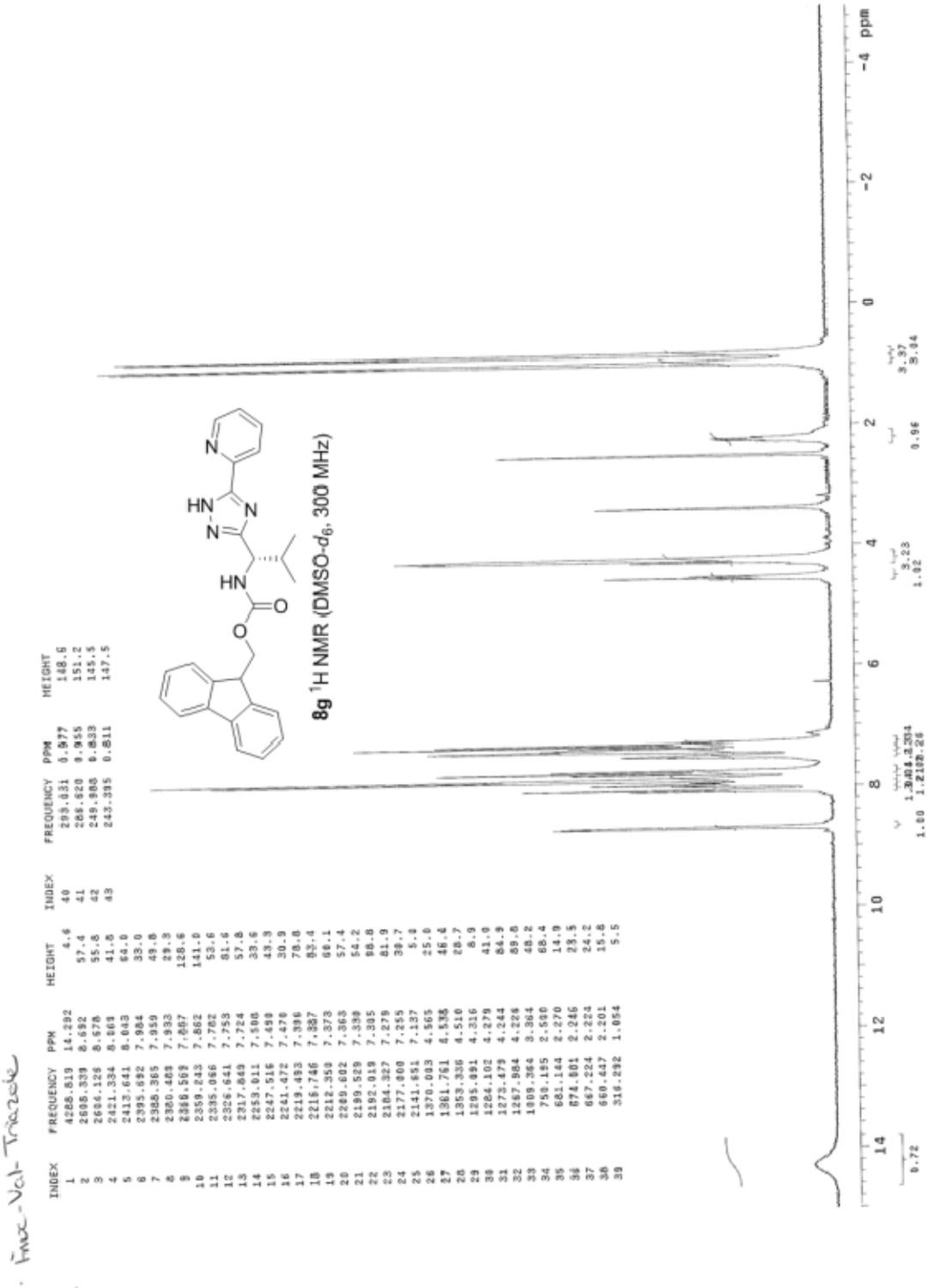
INDEX	FREQUENCY	PPM	HEIGHT
1	11738.337	155.573	11.5
2	11277.571	149.466	26.9
3	10379.870	137.568	12.1
4	10344.916	137.105	12.1
5	9679.717	128.289	48.2
6	9635.759	127.706	58.5
7	9404.316	124.639	6.4
8	9145.334	121.207	25.8
9	4926.406	65.292	24.9
10	3378.866	44.781	5.3
11	3044.148	40.345	22.0
12	3023.493	40.072	66.3
13	3002.309	39.791	128.1
14	2981.124	39.510	148.5
15	2960.469	39.236	126.4
16	2939.284	38.955	63.9
17	2918.629	38.682	20.0
18	1516.204	20.095	11.3



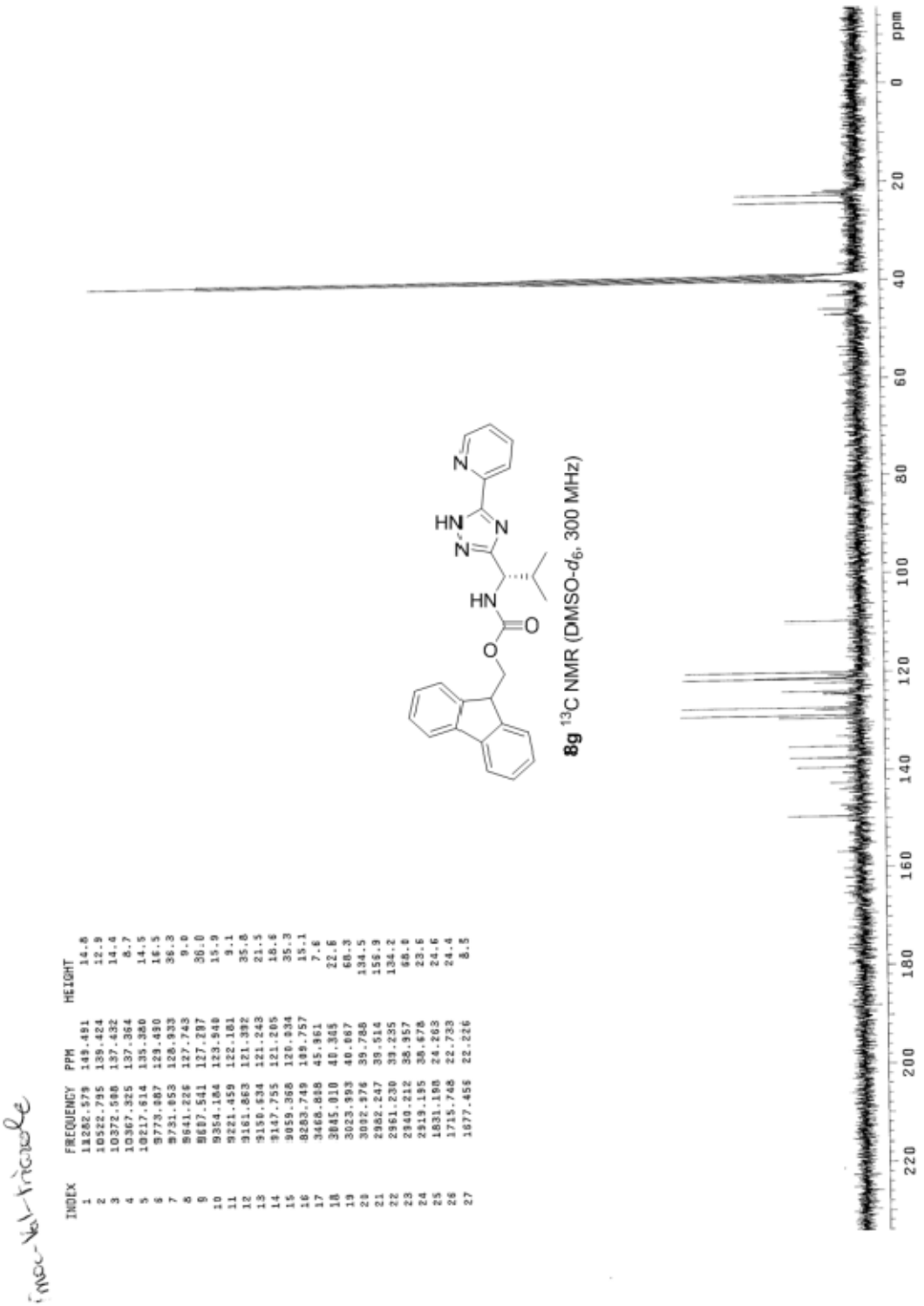
INDEX	FREQUENCY	PPM	HEIGHT
1	2597.532	8.656	24.3
2	2592.953	8.641	23.1
3	2423.715	8.077	16.8
4	2416.369	8.053	35.8
5	2408.696	8.027	33.2
6	2382.321	7.939	15.1
7	2374.812	7.914	22.0
8	2368.055	7.891	10.2
9	2237.483	7.456	19.4
10	2232.497	7.440	21.5
11	2225.171	7.415	18.2
12	2210.152	7.365	77.9
13	2206.386	7.352	100.0
14	2197.880	7.324	28.0
15	2174.253	7.246	6.8
16	1510.668	5.034	90.8
17	1503.526	5.010	14.3
18	1477.151	4.923	5.3
19	1476.191	4.899	13.6
20	1462.498	4.874	18.6
21	1455.172	4.849	13.2
22	759.195	2.530	23.3
23	517.034	1.723	41.1
24	443.938	1.476	75.5
25	435.894	1.453	73.3
26	375.010	1.252	9.3
27	366.858	1.223	9.0



8f ^{13}C NMR (DMSO-d_6, 75 MHz)

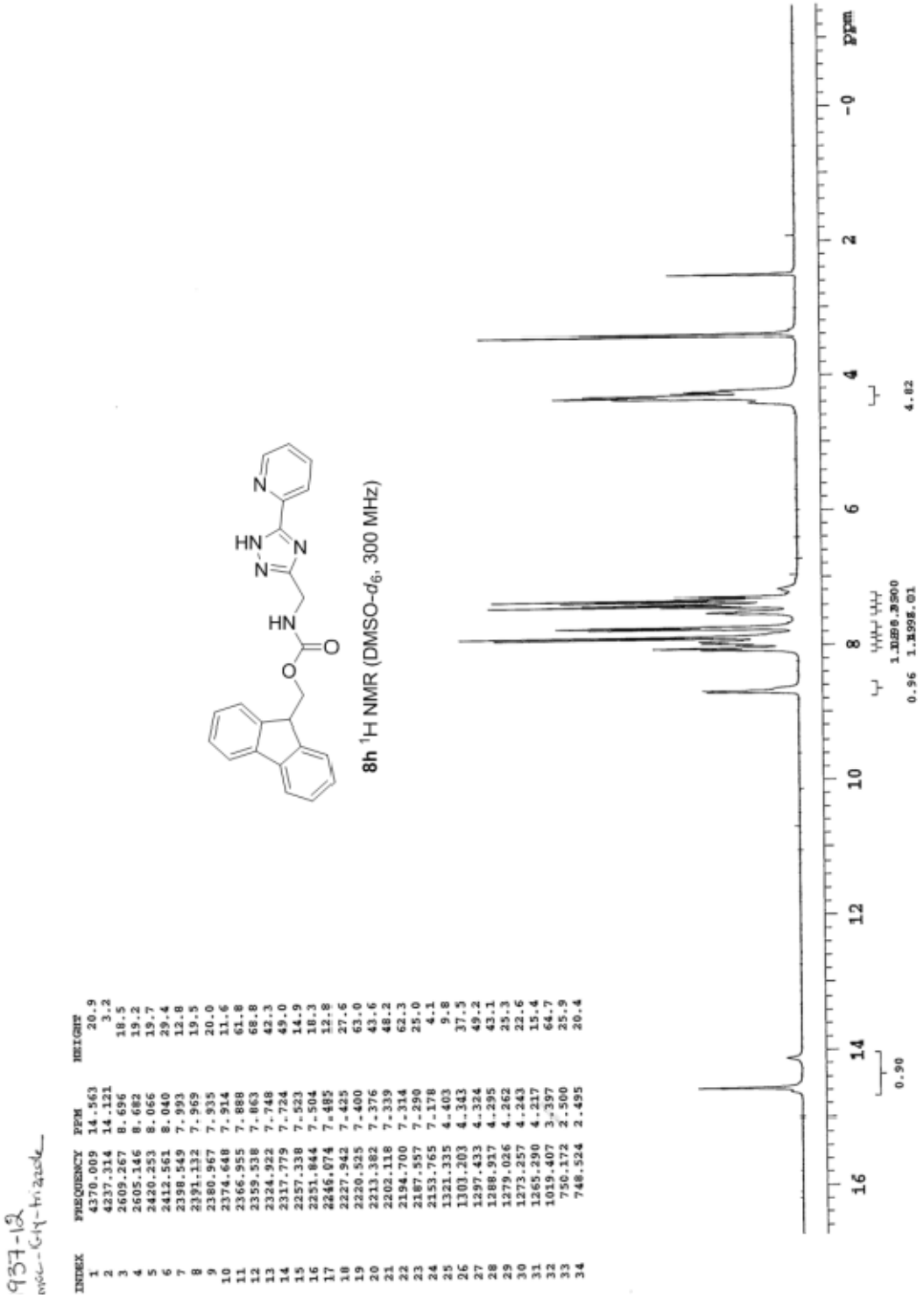


INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	4288.819	14.292	4.6	40	293.031	0.977	148.6
2	2608.339	8.692	57.4	41	286.620	0.955	151.2
3	2604.126	8.678	55.8	42	249.988	0.833	145.5
4	2421.334	8.069	41.8	43	243.335	0.811	147.5
5	2413.641	8.043	64.0				
6	2395.692	7.984	33.0				
7	2388.365	7.959	49.8				
8	2380.488	7.933	29.3				
9	2368.569	7.897	126.6				
10	2359.243	7.862	141.0				
11	2335.066	7.782	53.6				
12	2326.641	7.753	81.6				
13	2317.849	7.724	57.8				
14	2253.011	7.508	33.6				
15	2247.516	7.490	49.3				
16	2241.472	7.470	30.9				
17	2219.493	7.396	78.8				
18	2216.746	7.387	83.4				
19	2212.359	7.373	60.1				
20	2209.602	7.363	57.4				
21	2199.529	7.330	54.2				
22	2192.019	7.305	98.8				
23	2184.327	7.279	81.9				
24	2177.800	7.255	38.7				
25	2141.651	7.137	5.8				
26	1370.003	4.565	25.0				
27	1361.761	4.538	46.4				
28	1353.336	4.510	28.7				
29	1295.991	4.316	6.9				
30	1284.102	4.279	41.0				
31	1273.479	4.244	84.9				
32	1267.984	4.226	89.8				
33	1160.364	3.864	48.2				
34	759.195	2.530	68.4				
35	681.144	2.270	14.9				
36	674.101	2.246	23.5				
37	667.224	2.224	24.2				
38	660.447	2.201	15.8				
39	316.292	1.054	5.5				

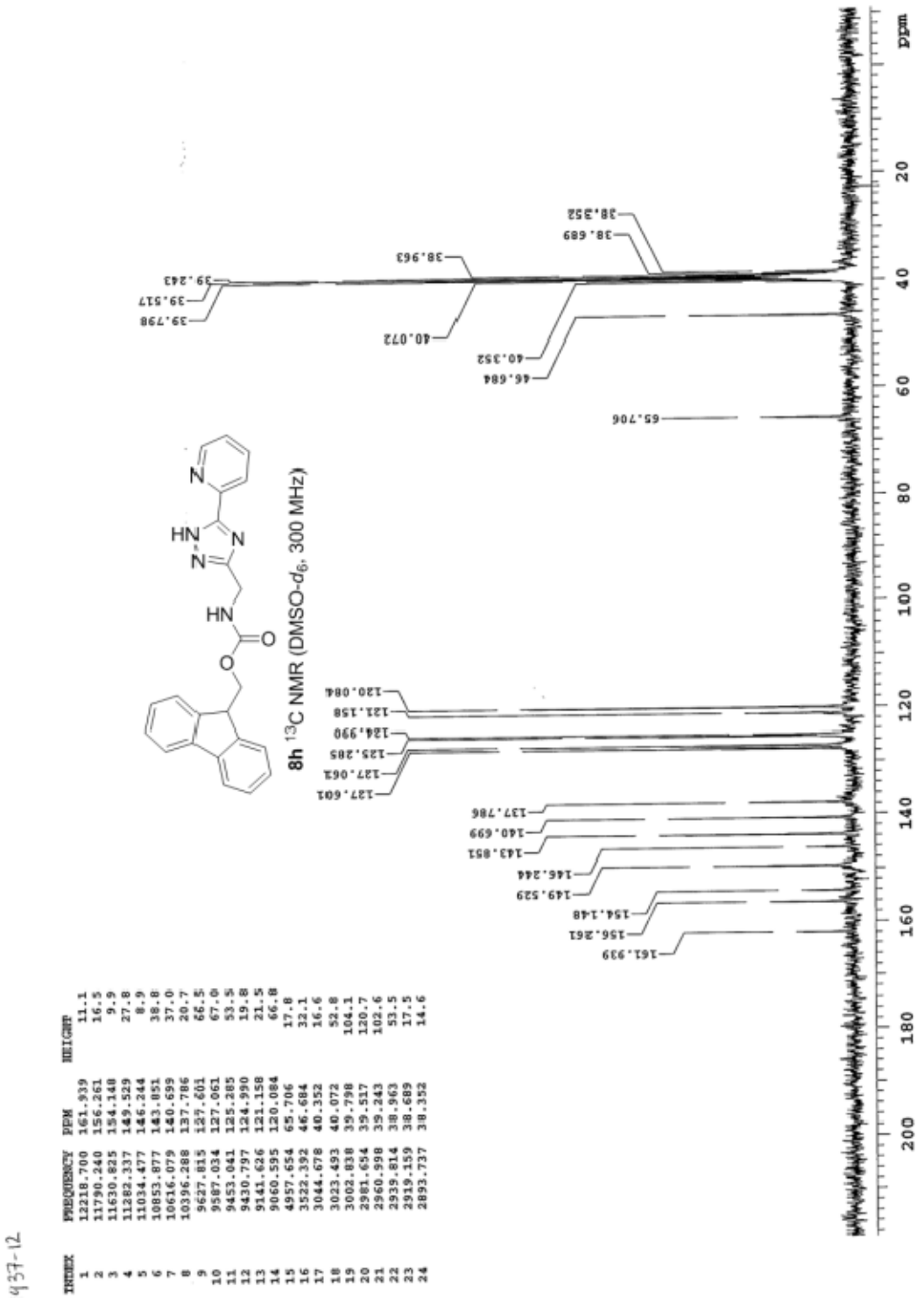


8g ^{13}C NMR (DMSO-d_6, 300 MHz)

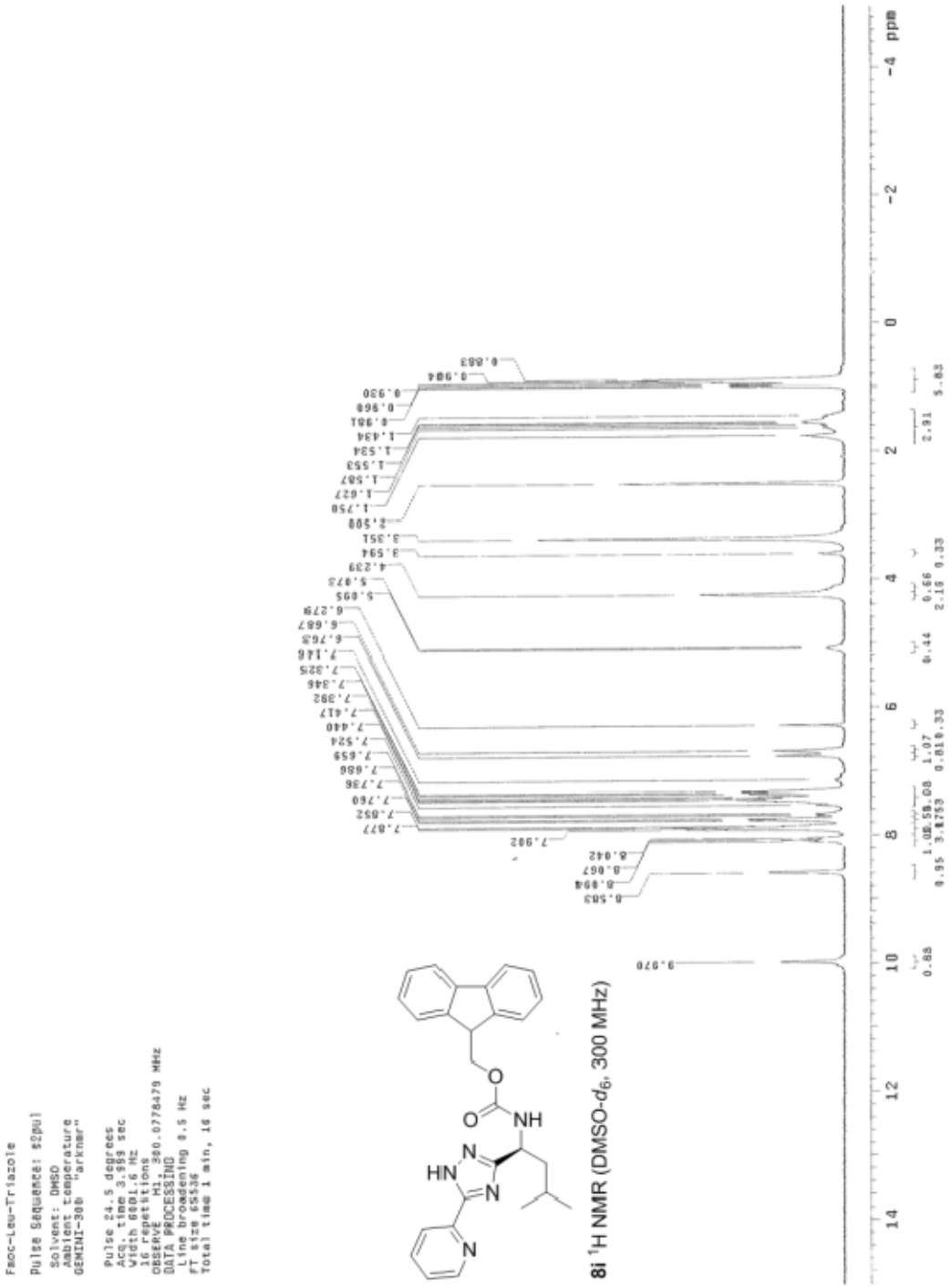
INDEX	FREQUENCY	PPM	HEIGHT
1	18282.578	149.491	14.8
2	18522.795	139.424	12.9
3	10372.598	137.432	14.4
4	10367.325	137.364	8.7
5	10217.614	135.386	14.5
6	9779.887	129.490	16.5
7	9731.853	128.933	36.3
8	9641.226	127.743	9.0
9	9607.541	127.297	36.0
10	9354.184	123.940	15.9
11	9221.459	122.181	9.1
12	9161.863	121.392	35.8
13	9150.634	121.243	21.5
14	9147.755	121.205	18.6
15	9059.368	120.034	35.3
16	8283.749	109.757	15.1
17	3468.688	45.961	7.6
18	3045.010	40.345	22.6
19	3023.993	40.067	68.3
20	3002.976	39.788	134.5
21	2982.247	39.514	156.9
22	2961.230	39.235	134.2
23	2940.212	38.957	68.8
24	2919.195	38.678	23.6
25	1831.188	24.263	24.6
26	1715.748	22.733	24.4
27	1677.456	22.226	8.5

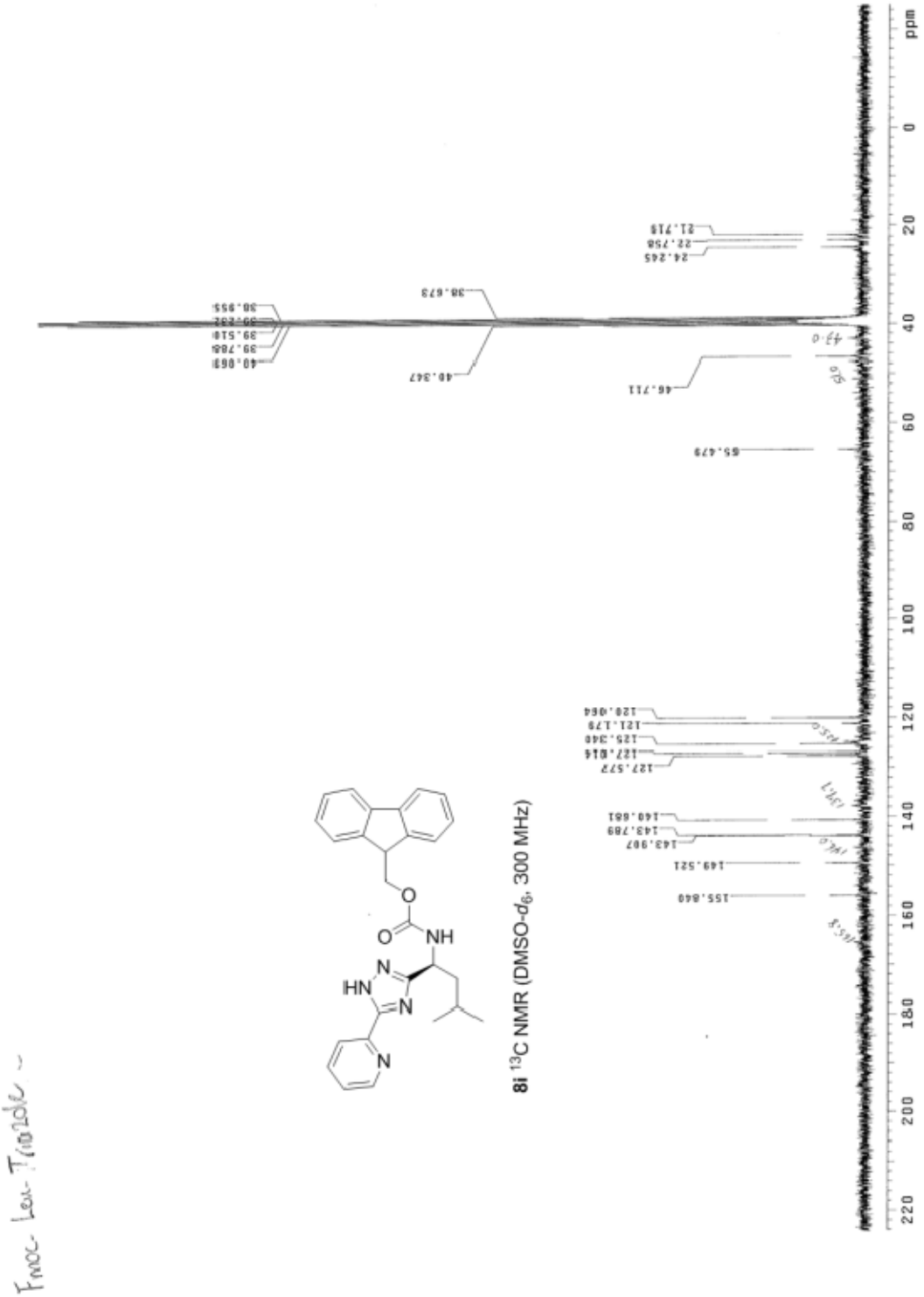


INDEX	FREQUENCY	PPM	HEIGHT
1	4370.009	14.563	20.9
2	4237.314	14.121	3.2
3	2609.267	8.696	18.5
4	2605.146	8.682	19.2
5	2420.253	8.066	19.7
6	2412.561	8.040	29.4
7	2398.549	7.993	12.8
8	2391.132	7.969	19.5
9	2380.967	7.935	20.0
10	2374.648	7.914	11.6
11	2366.955	7.888	61.8
12	2359.538	7.863	68.8
13	2324.922	7.748	42.3
14	2317.779	7.724	49.0
15	2257.338	7.523	14.9
16	2251.844	7.504	18.3
17	2246.074	7.485	12.8
18	2227.942	7.425	27.6
19	2220.525	7.400	63.0
20	2213.382	7.376	43.6
21	2202.118	7.339	48.2
22	2194.700	7.314	62.3
23	2187.557	7.290	25.0
24	2153.765	7.178	4.1
25	1321.335	4.403	9.8
26	1303.203	4.343	37.5
27	1297.433	4.324	49.2
28	1288.937	4.295	43.1
29	1279.026	4.262	25.3
30	1273.257	4.243	22.6
31	1265.290	4.217	15.4
32	1019.407	3.397	64.7
33	750.172	2.500	25.9
34	748.524	2.495	20.4



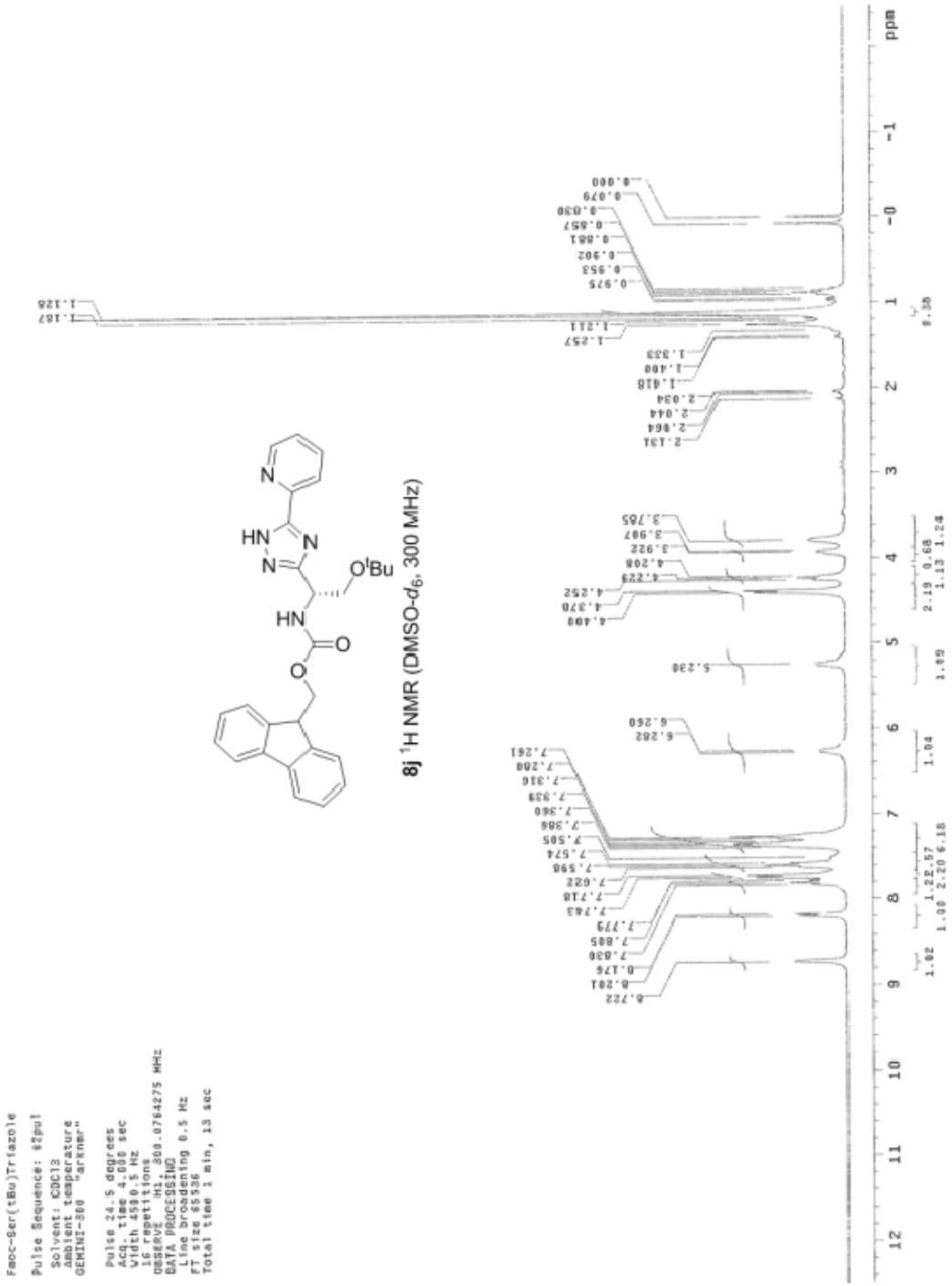
INDEX	FREQUENCY	PPM	HEIGHT
1	12218.700	161.939	11.1
2	11790.240	156.261	16.5
3	11630.825	154.148	9.9
4	11283.337	149.529	27.8
5	11034.477	146.244	8.9
6	10853.877	143.851	38.8
7	10616.079	140.699	37.0
8	10396.288	137.786	20.7
9	9627.815	127.601	66.5
10	9587.034	127.061	67.0
11	9453.041	125.285	53.5
12	9430.797	124.990	19.8
13	9141.626	121.158	21.5
14	9060.595	120.084	66.8
15	4957.654	65.706	17.8
16	3522.392	46.684	32.1
17	3044.678	40.352	16.6
18	3023.493	40.072	52.8
19	3002.838	39.798	104.1
20	2981.654	39.517	120.7
21	2960.998	39.243	102.6
22	2939.814	38.963	53.5
23	2919.159	38.689	17.5
24	2893.737	38.352	14.6


Fmoc-Leu-Triazole
Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
GEMINI-300 "arkmar"
Pulse 24.5 degrees
Acq. time 3.989 sec
Width 6001.6 Hz
16 repetitions
OBSERVE H1, 300.0778479 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 min, 16 sec
O
NH
HN
N
N
N
O
8i ^{1}H NMR (DMSO-d_6, 300 MHz)

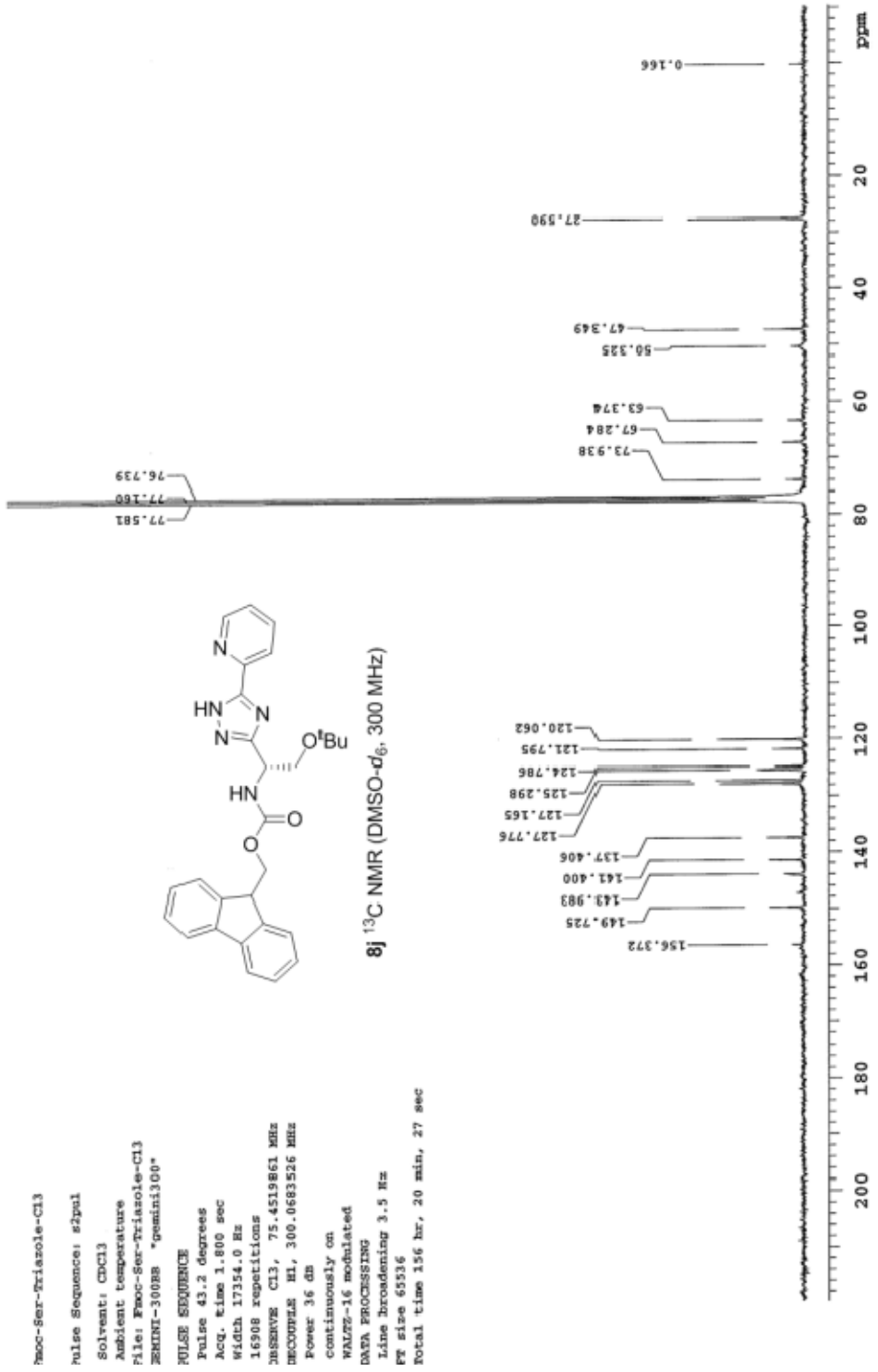


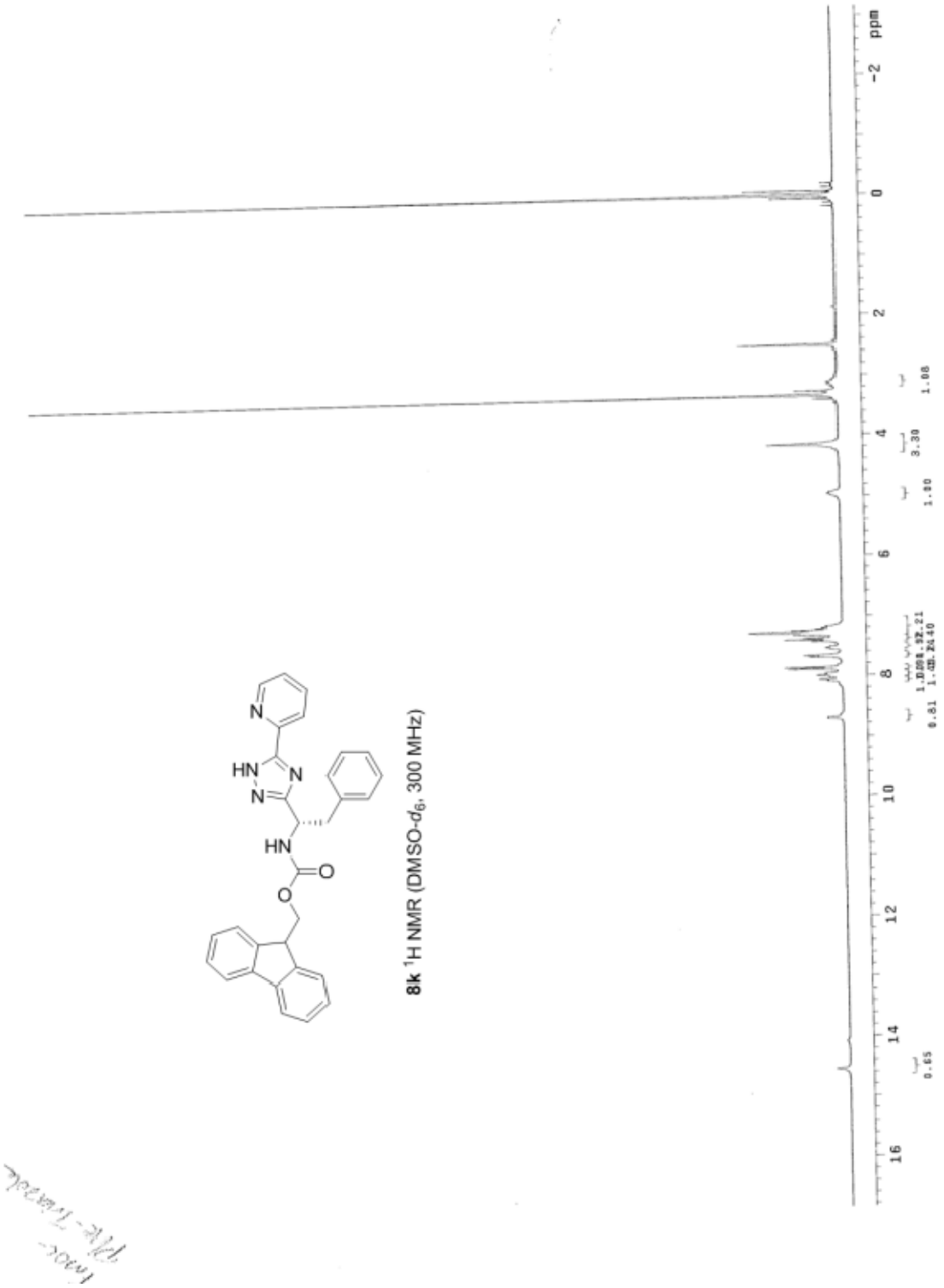
8i ^{13}C NMR (DMSO-d_6, 300 MHz)

Fmoc-Leu-Triazole

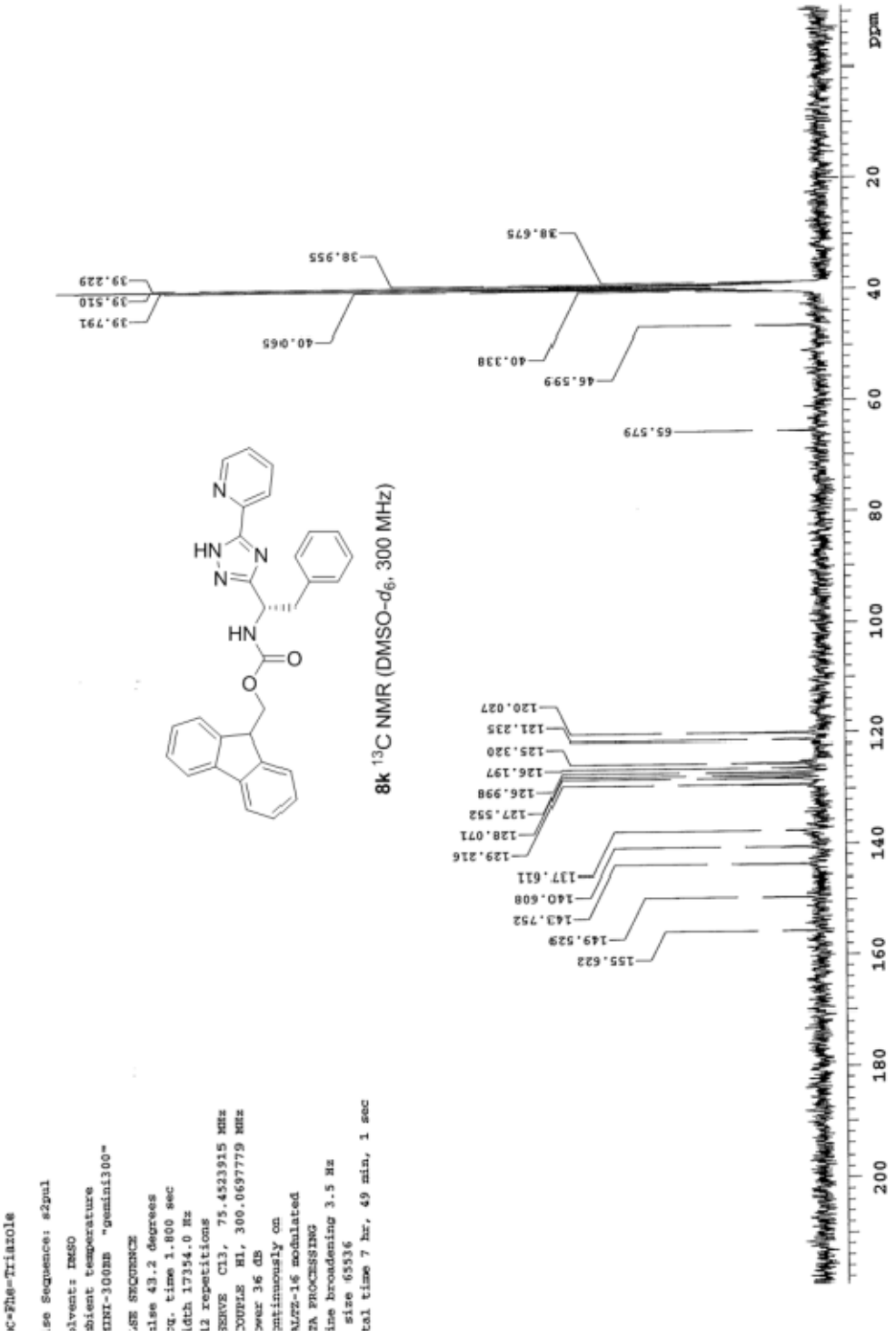


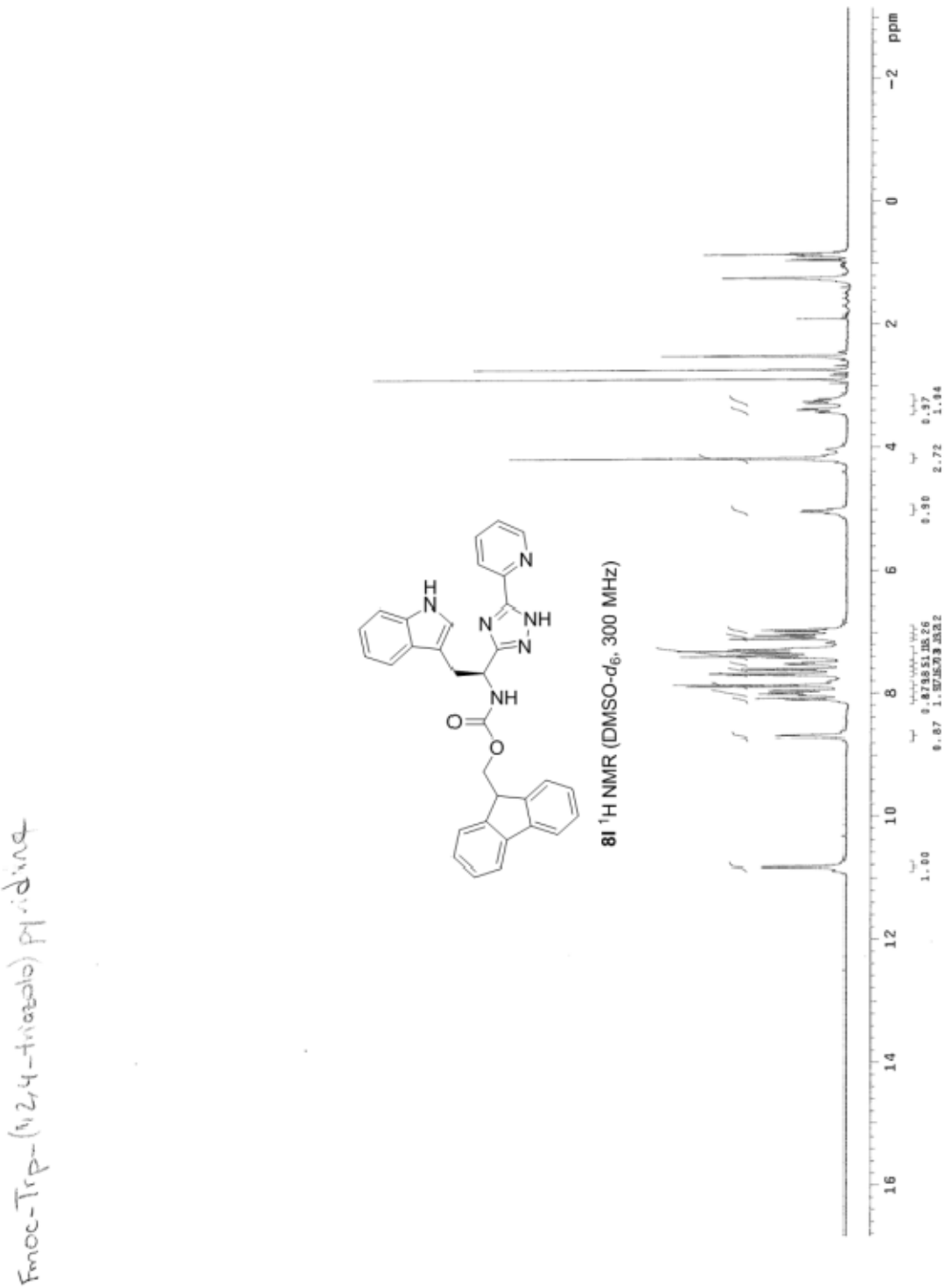
8j ^{1}H NMR (DMSO-d_6, 300 MHz)

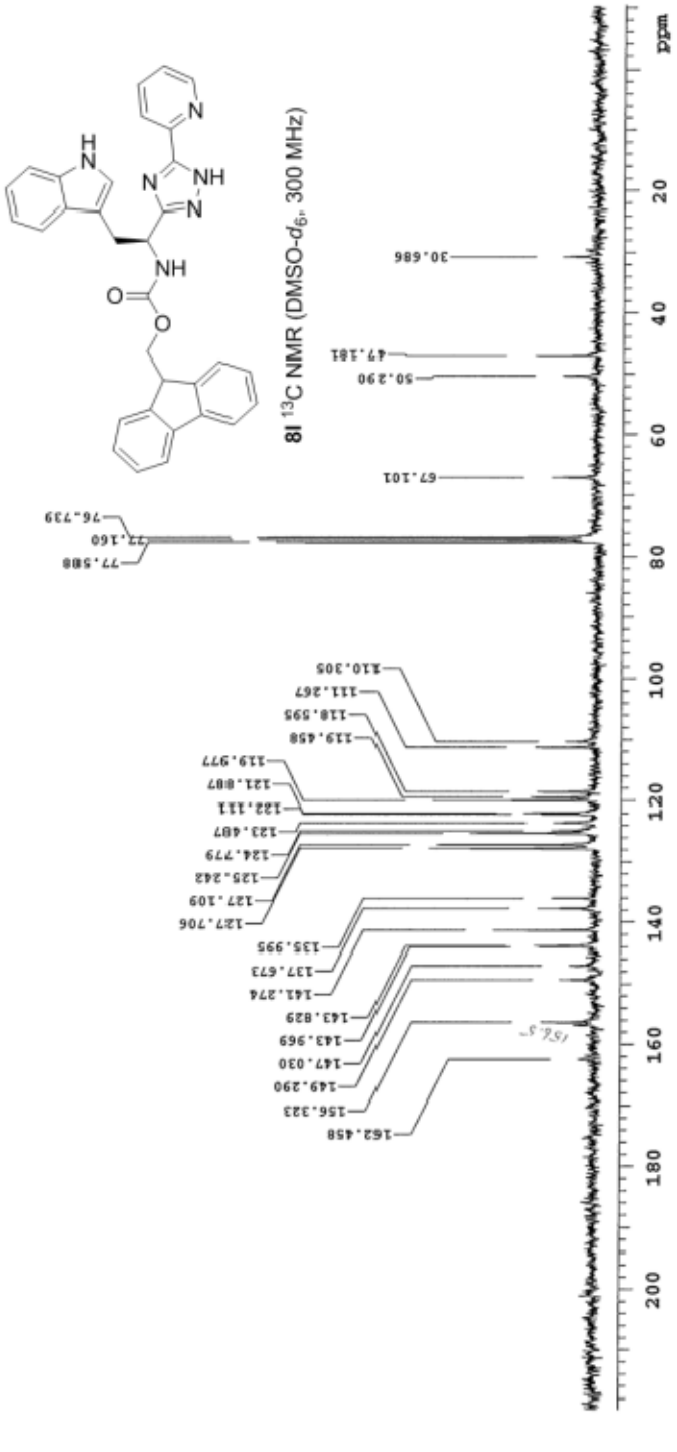

8j ^{13}C NMR (DMSO-d_6, 300 MHz)
ppm
0.166
27.598
47.349
50.335
63.374
67.284
73.938
76.739
77.160
77.581
120.062
121.795
124.786
125.298
127.165
127.776
137.406
143.400
143.983
149.725
156.372
HN
N
N
HN
O
O
O^{t}Bu

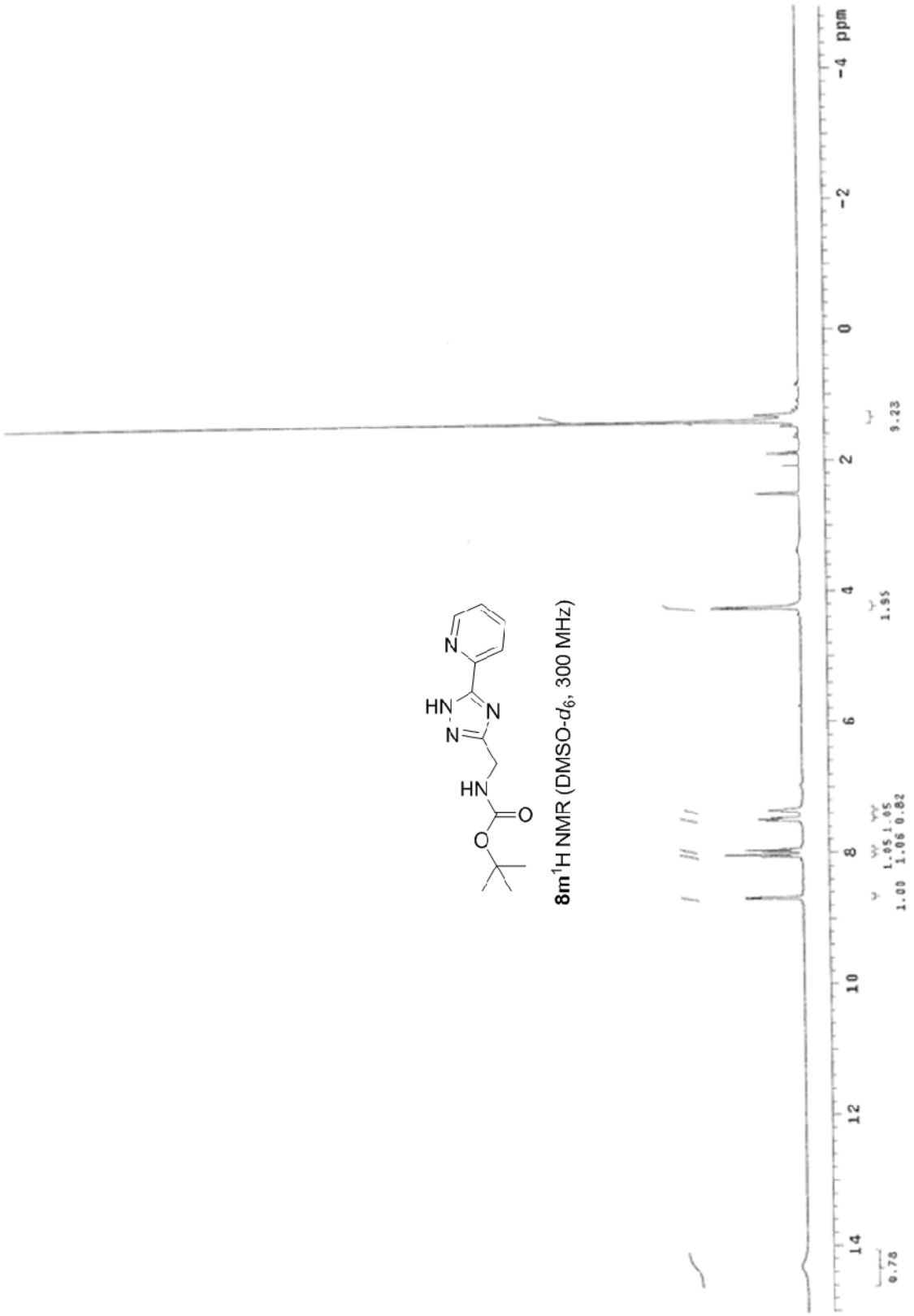


8k ^{1}H NMR (DMSO-d_6, 300 MHz)

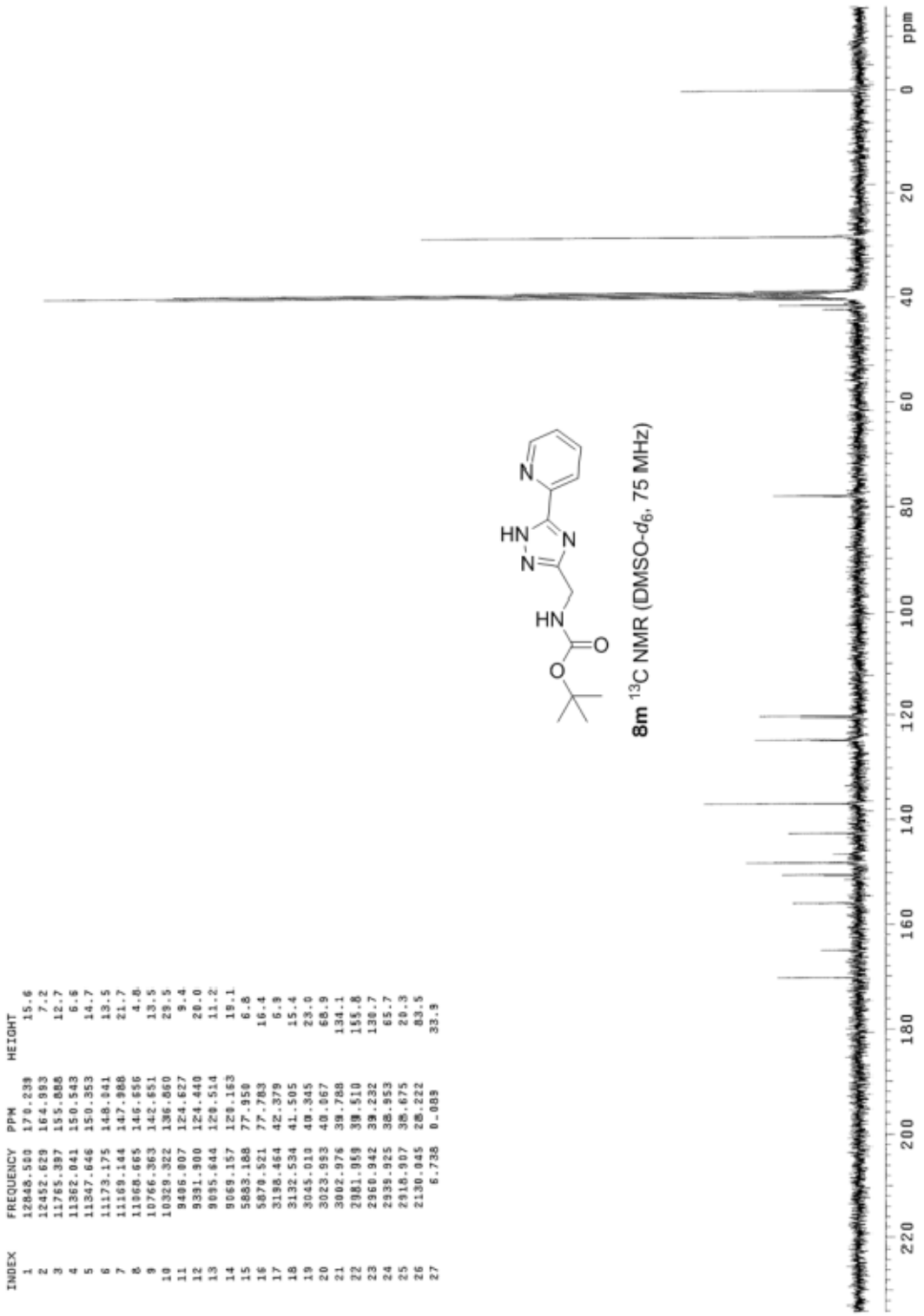

8k ^{13}C NMR (DMSO-d_6, 300 MHz)
ppm
20
40
60
80
100
120
140
160
180
200
38.675
38.955
39.229
39.510
39.791
40.065
40.338
46.599
65.579
120.027
121.235
125.320
126.197
126.998
127.552
128.071
129.316
137.611
140.608
143.752
149.529
155.622
HN
N
N
HN
O
O


Fmoc-Trp-(1,2,4-triazole)-pyridine
8l ^{1}H NMR (DMSO-d_6, 300 MHz)
ppm


8l ^{13}C NMR (DMSO-d_6, 300 MHz)
ppm

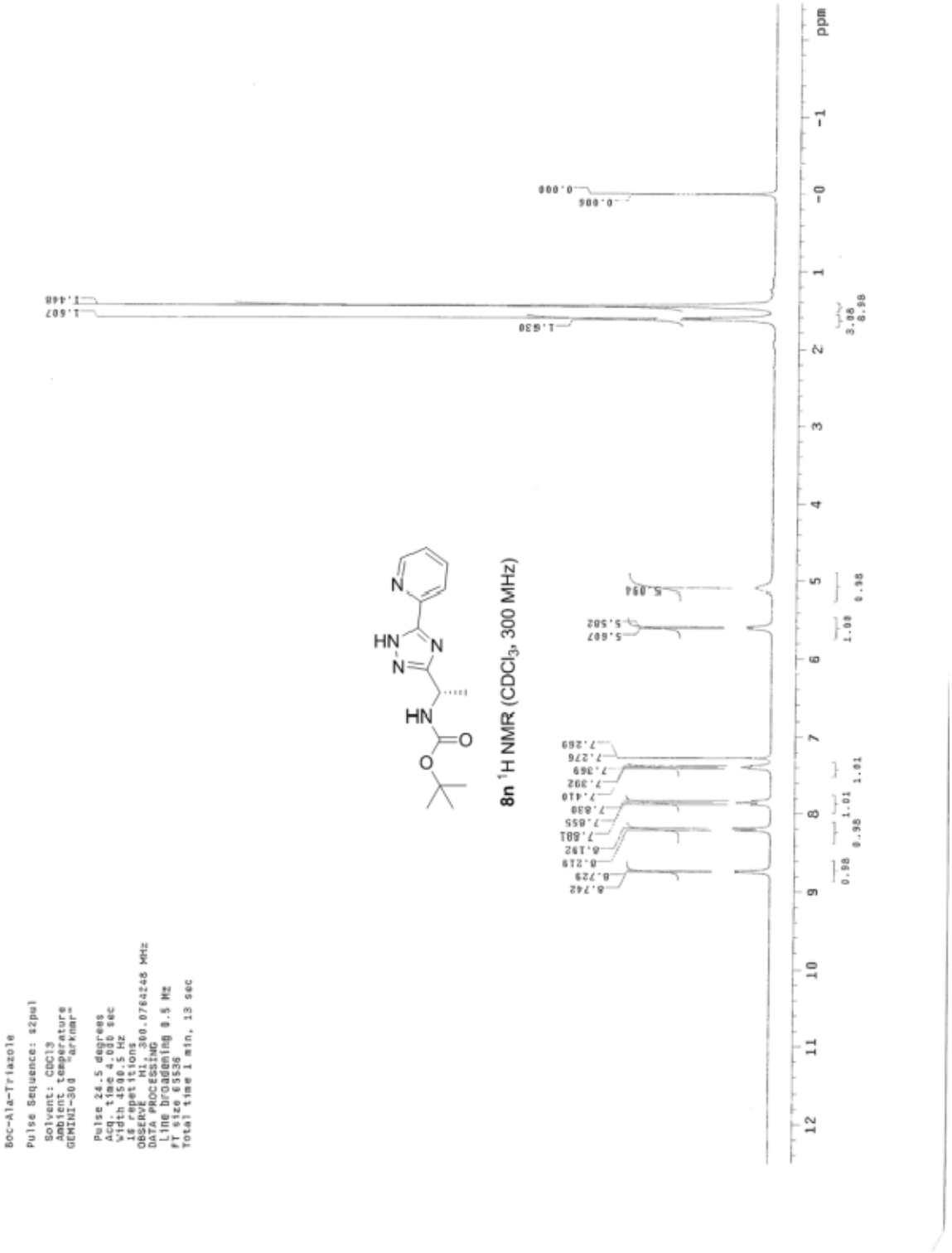


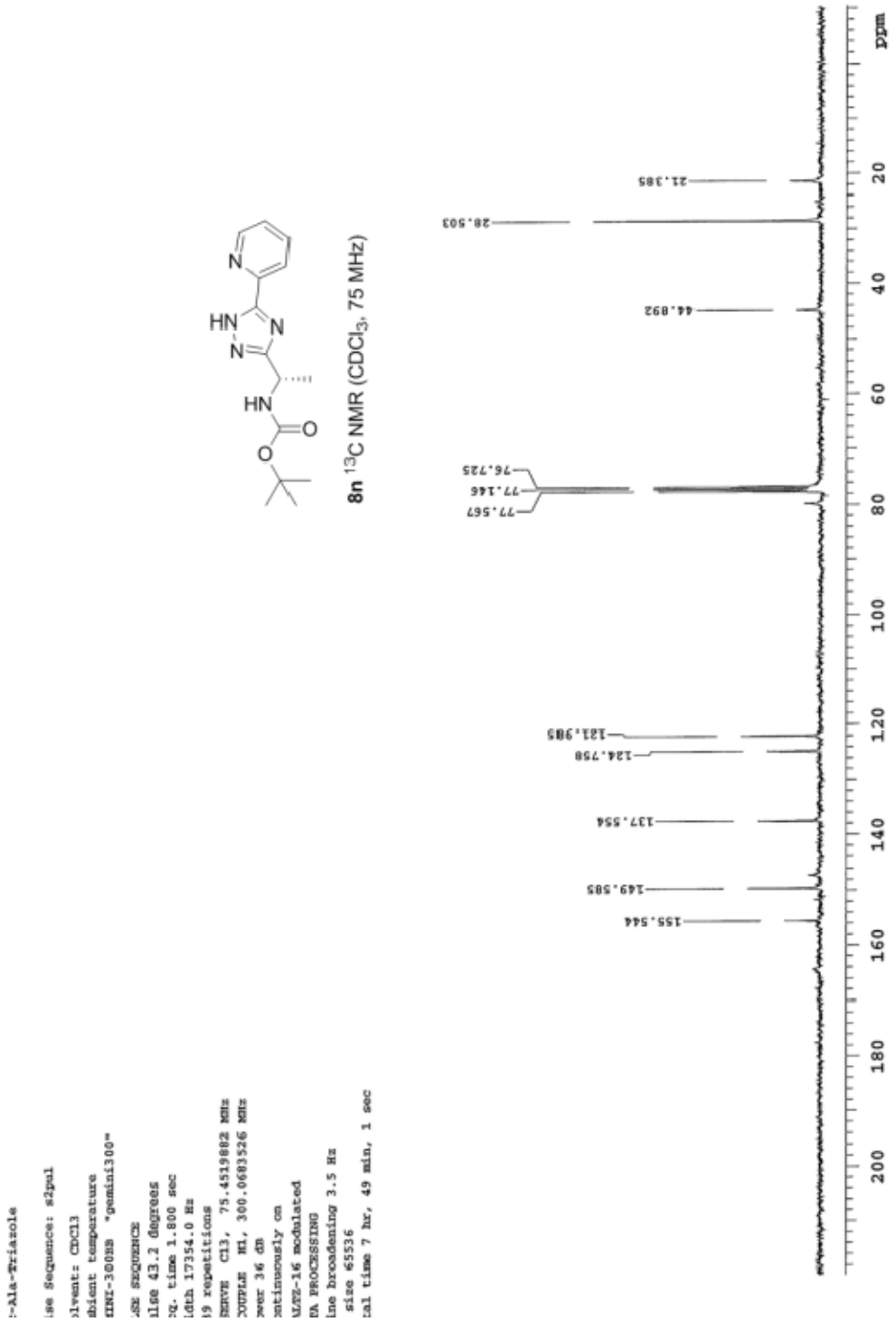
8m ^{1}H NMR (DMSO-d_6, 300 MHz)

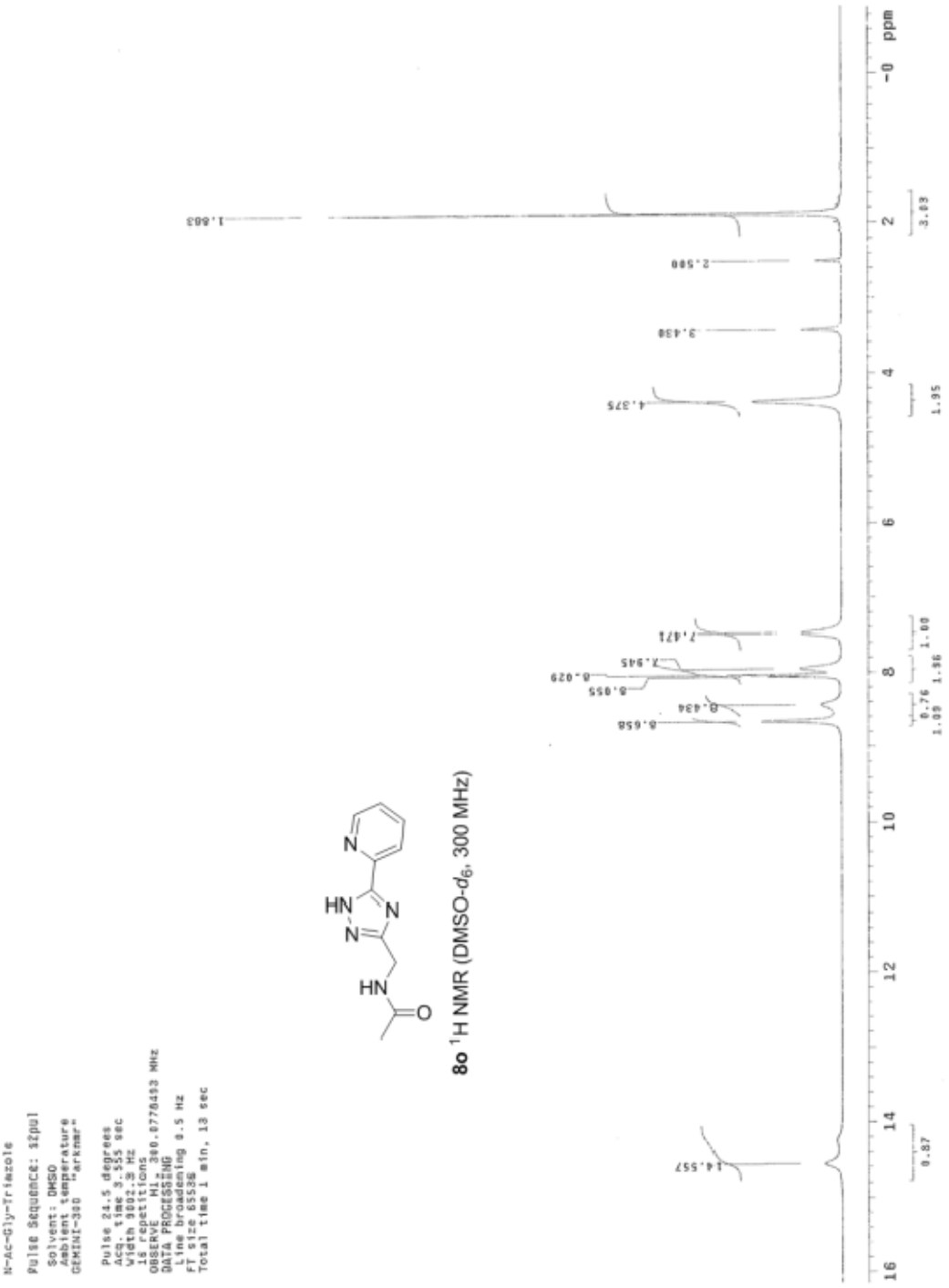


8m ^{13}C NMR (DMSO-d_6, 75 MHz)

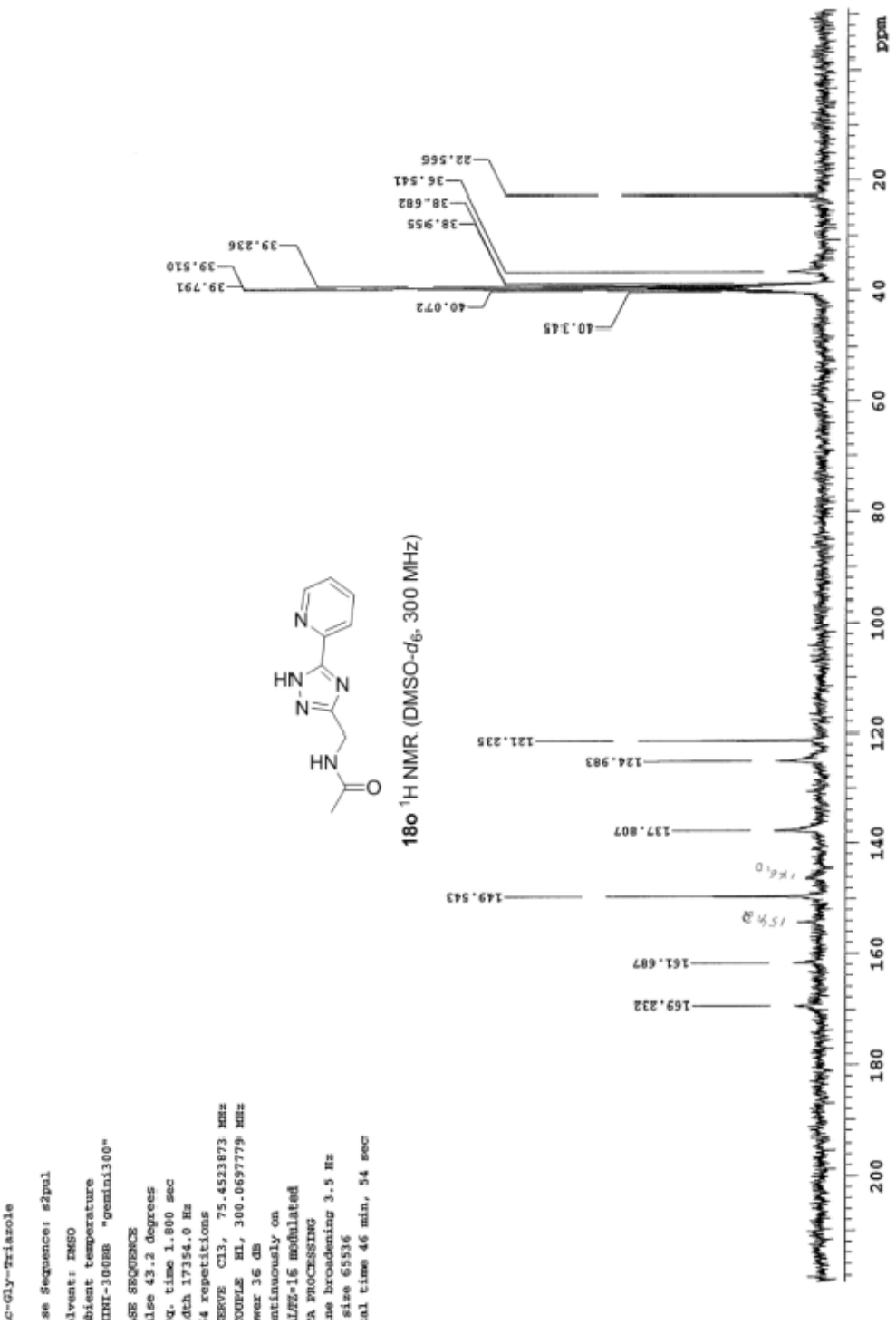
INDEX	FREQUENCY	PPM	HEIGHT
1	12848.510	170.239	15.6
2	12452.629	164.993	7.2
3	11765.397	155.885	12.7
4	11362.041	150.543	6.6
5	11347.646	150.353	14.7
6	11173.175	148.041	13.5
7	11169.144	147.988	21.7
8	11068.665	146.656	4.8
9	10766.363	142.651	13.5
10	10329.322	136.860	29.5
11	9406.807	124.627	9.4
12	9391.900	124.440	20.0
13	9095.644	120.514	11.2
14	9069.157	120.163	18.1
15	5883.188	77.950	6.8
16	5870.521	77.783	16.4
17	3198.464	42.379	6.3
18	3132.534	41.505	15.4
19	3045.010	40.345	23.0
20	3023.993	40.067	68.9
21	3002.976	39.788	134.1
22	2981.959	39.510	155.8
23	2960.942	39.232	130.7
24	2939.925	38.953	65.7
25	2918.907	38.675	29.3
26	2130.045	28.222	83.5
27	6.738	0.089	33.9

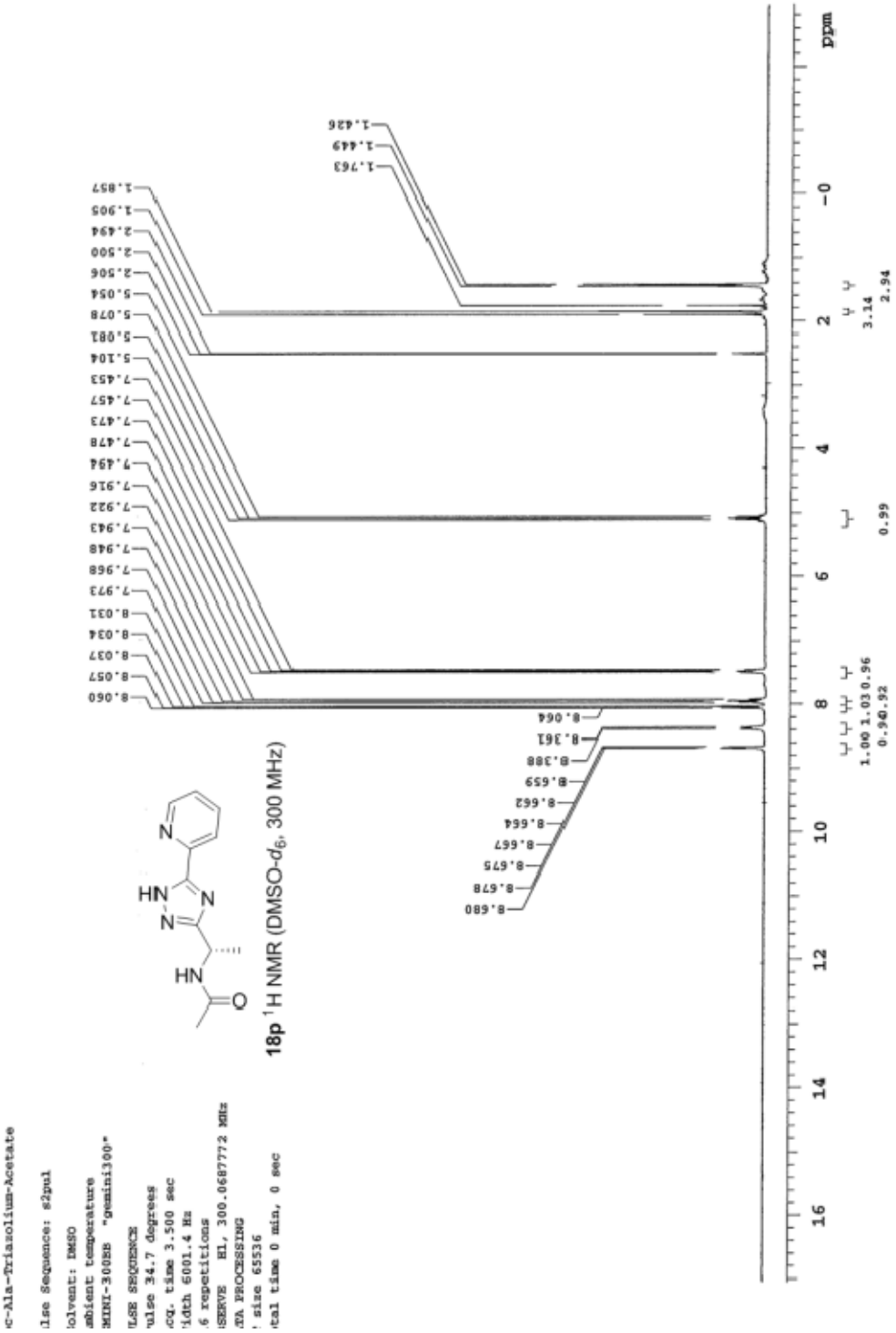

Boc-Ala-Triazole
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300 "arkmar"
Pulse 24.5 degrees
Acq. time 4.000 sec
Width 4500.5 Hz
16 repetitions
OBSERVE H1, 300.0764248 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 min, 13 sec
8n ^{1}H NMR (CDCl$_3$, 300 MHz)
ppm

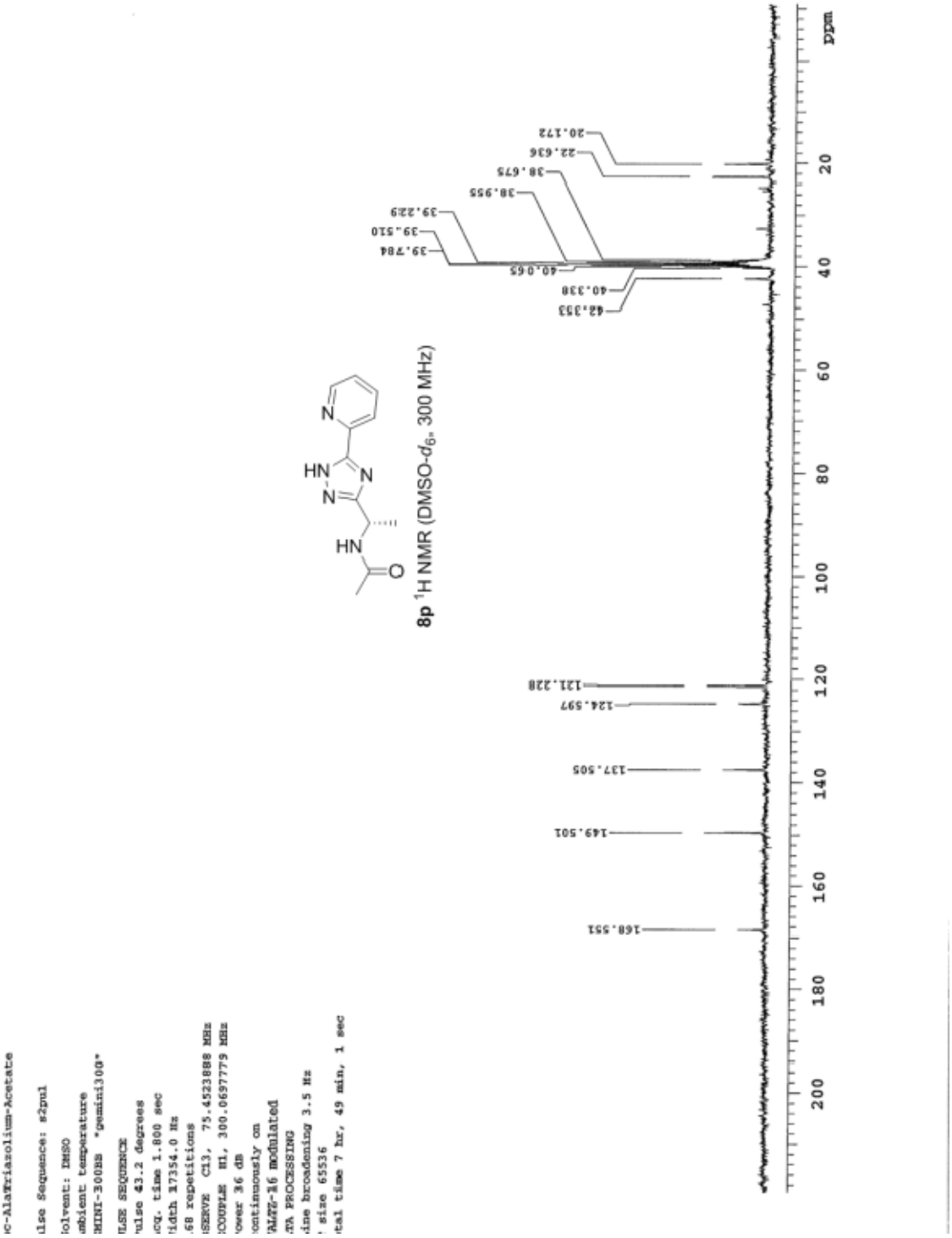

8n ^{13}C NMR ($CDCl_3$, 75 MHz)
HN
N
N
HN
O
O
21.385
28.503
44.892
76.725
77.146
77.567
121.985
124.758
137.554
149.585
155.544
200
180
160
140
120
100
80
60
40
20
ppm

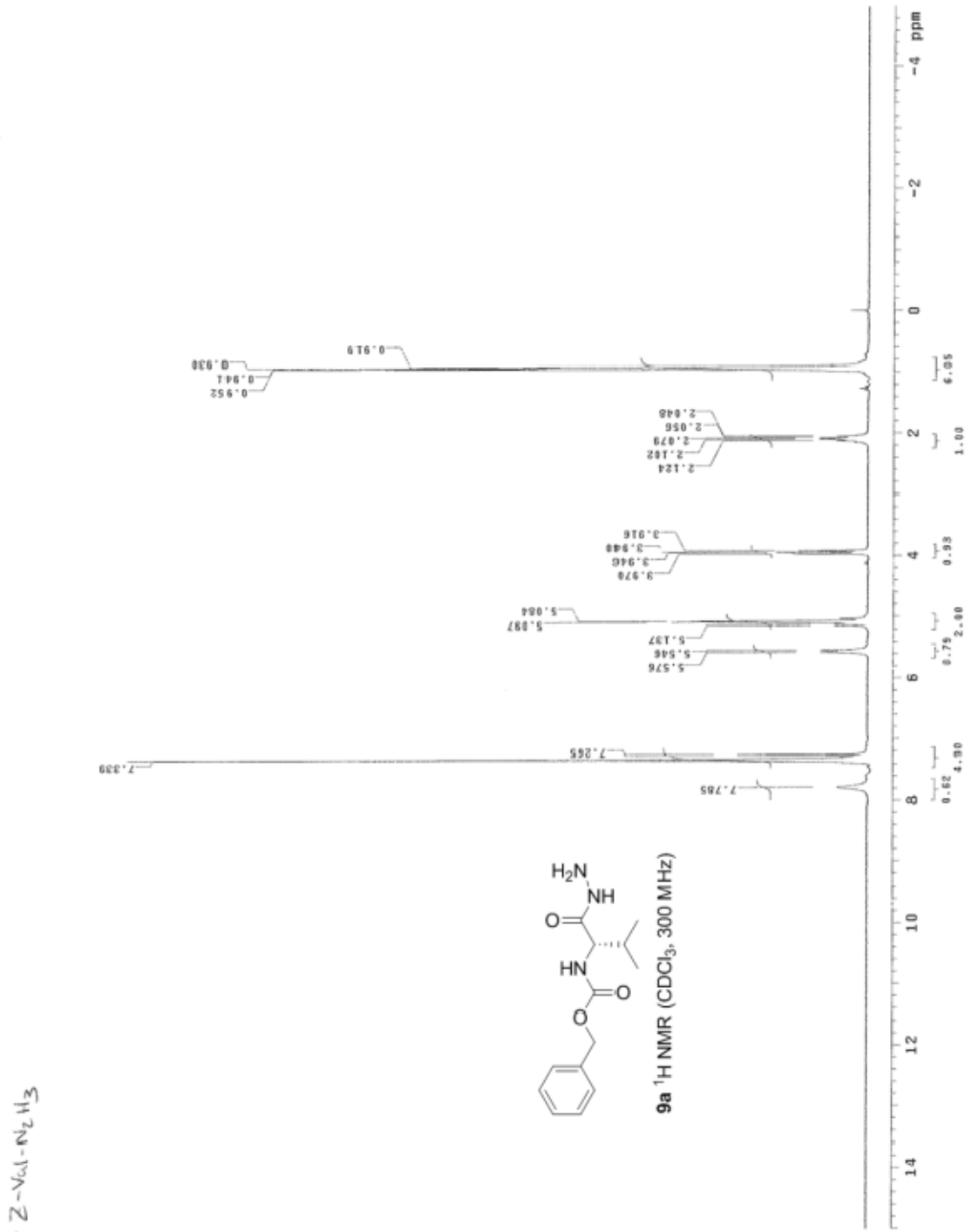


8o ^{1}H NMR (DMSO-d_6, 300 MHz)


18o ^{1}H NMR (DMSO-d_6, 300 MHz)
N
HN
N
N
HN
O
ppm
20
40
60
80
100
120
140
160
180
200
22.565
36.541
38.682
38.955
39.236
39.510
39.791
40.072
40.345
121.235
124.983
137.807
149.543
161.687
169.232

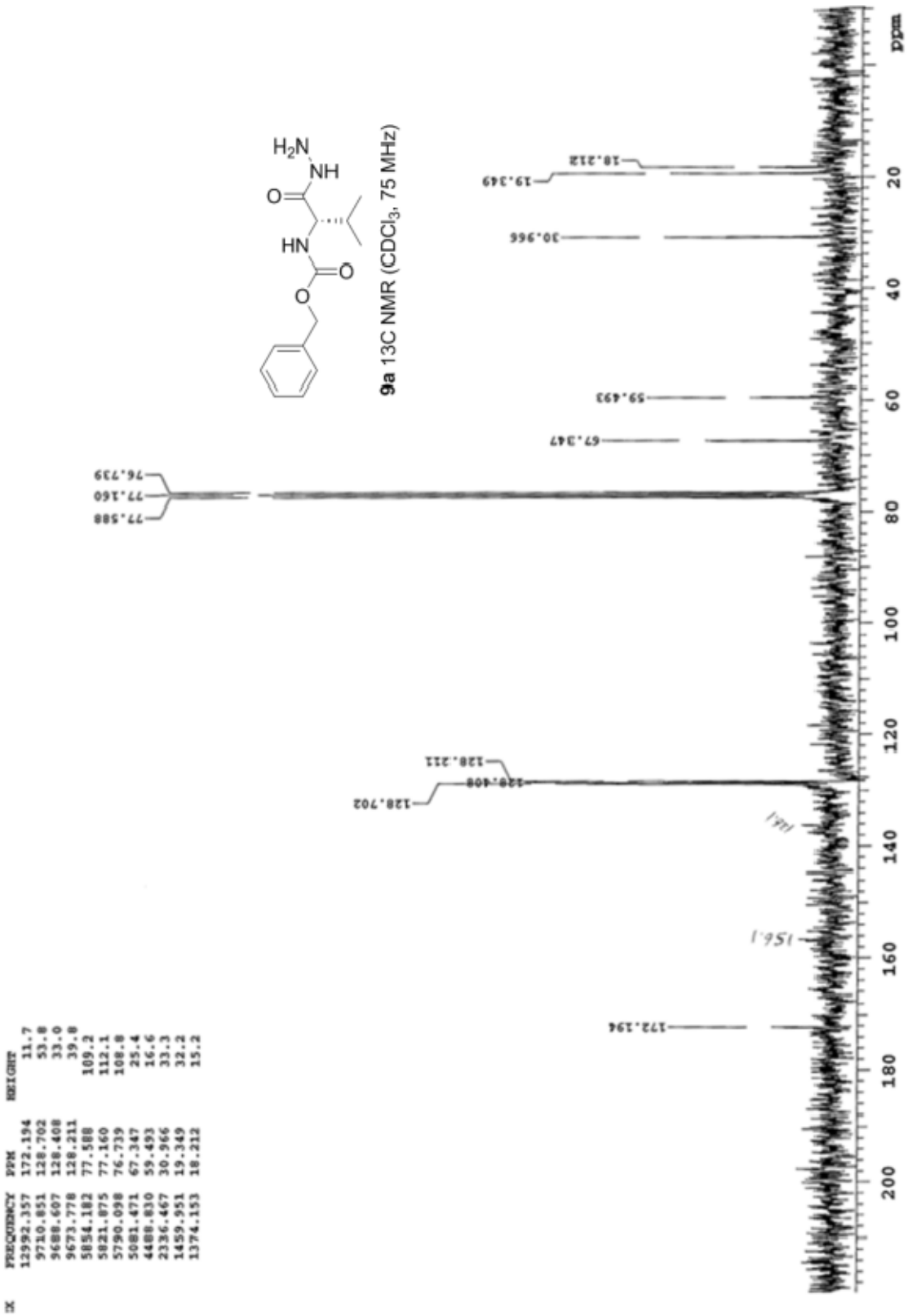

18p ^{1}H NMR (DMSO-d_6, 300 MHz)
HN
N
N
HN
O
ppm
16
14
12
10
8
6
4
2
0
1.00 1.03 0.96
0.94 0.92
3.14
2.94
0.99
8.680
8.678
8.675
8.667
8.664
8.662
8.659
8.388
8.361
8.064
7.857
7.763
7.449
7.436
5.500
5.494
5.078
5.054
5.082
5.104
7.453
7.457
7.473
7.478
7.494
7.916
7.922
7.943
7.948
7.968
7.973
8.031
8.034
8.037
8.057
8.060


8p ^{1}H NMR (DMSO-d_6, 300 MHz)
HN
N
N
HN
O
168.551
149.501
137.505
124.597
121.228
42.353
40.338
40.065
39.784
39.510
39.229
38.955
38.675
22.636
20.172
ppm
200
180
160
140
120
100
80
60
40
20

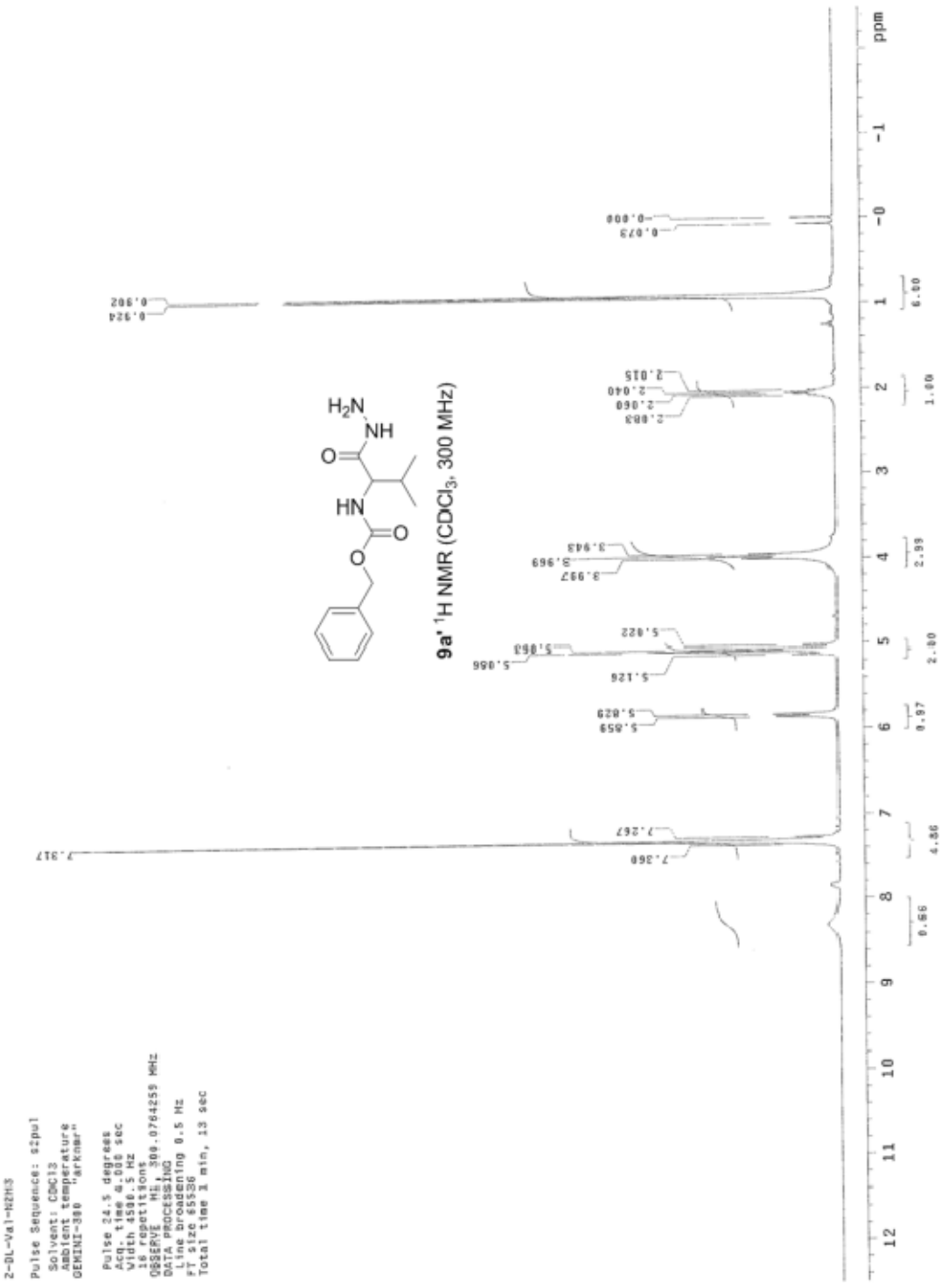


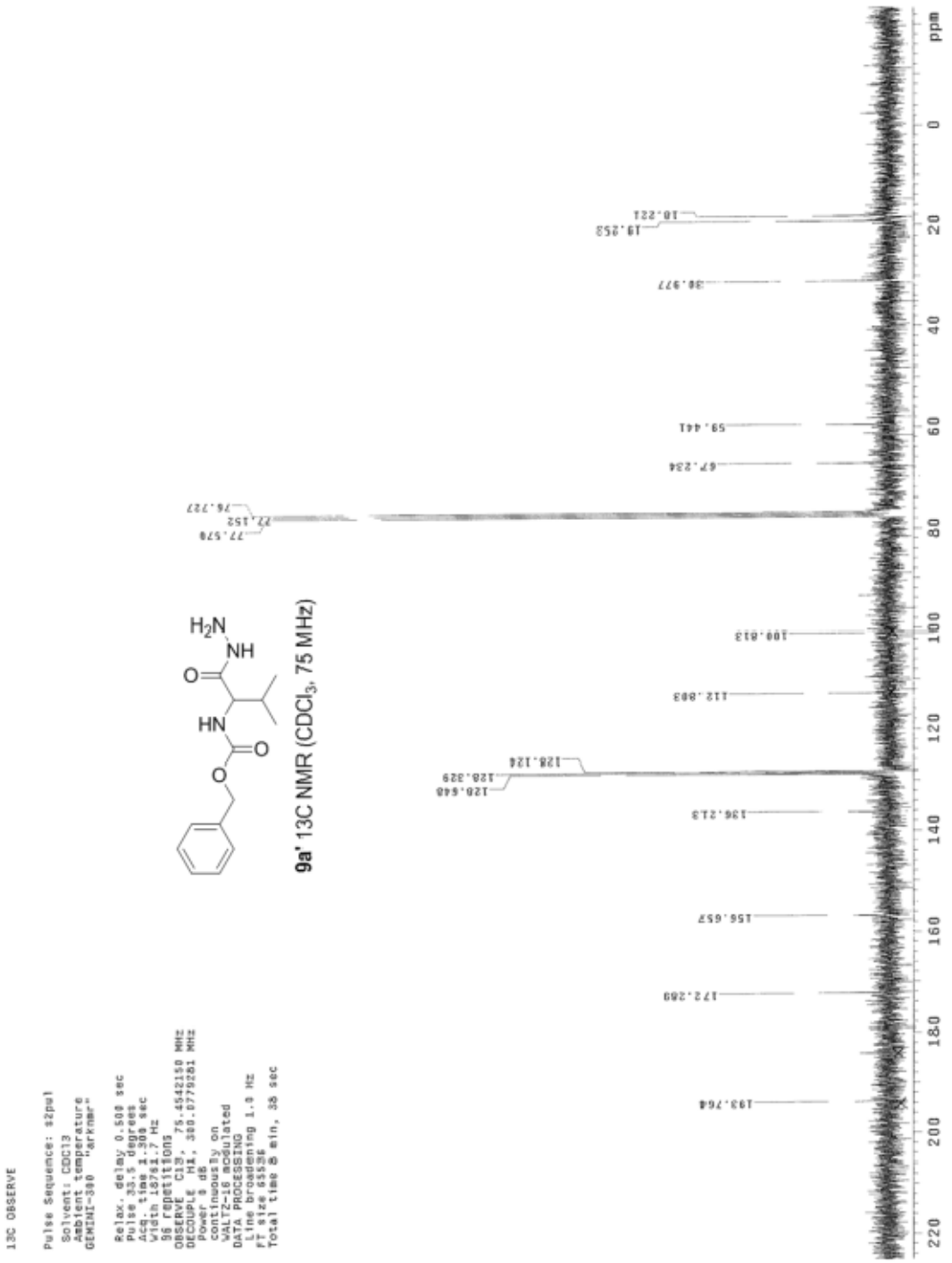
9a ^{1}H NMR ($CDCl_3$, 300 MHz)

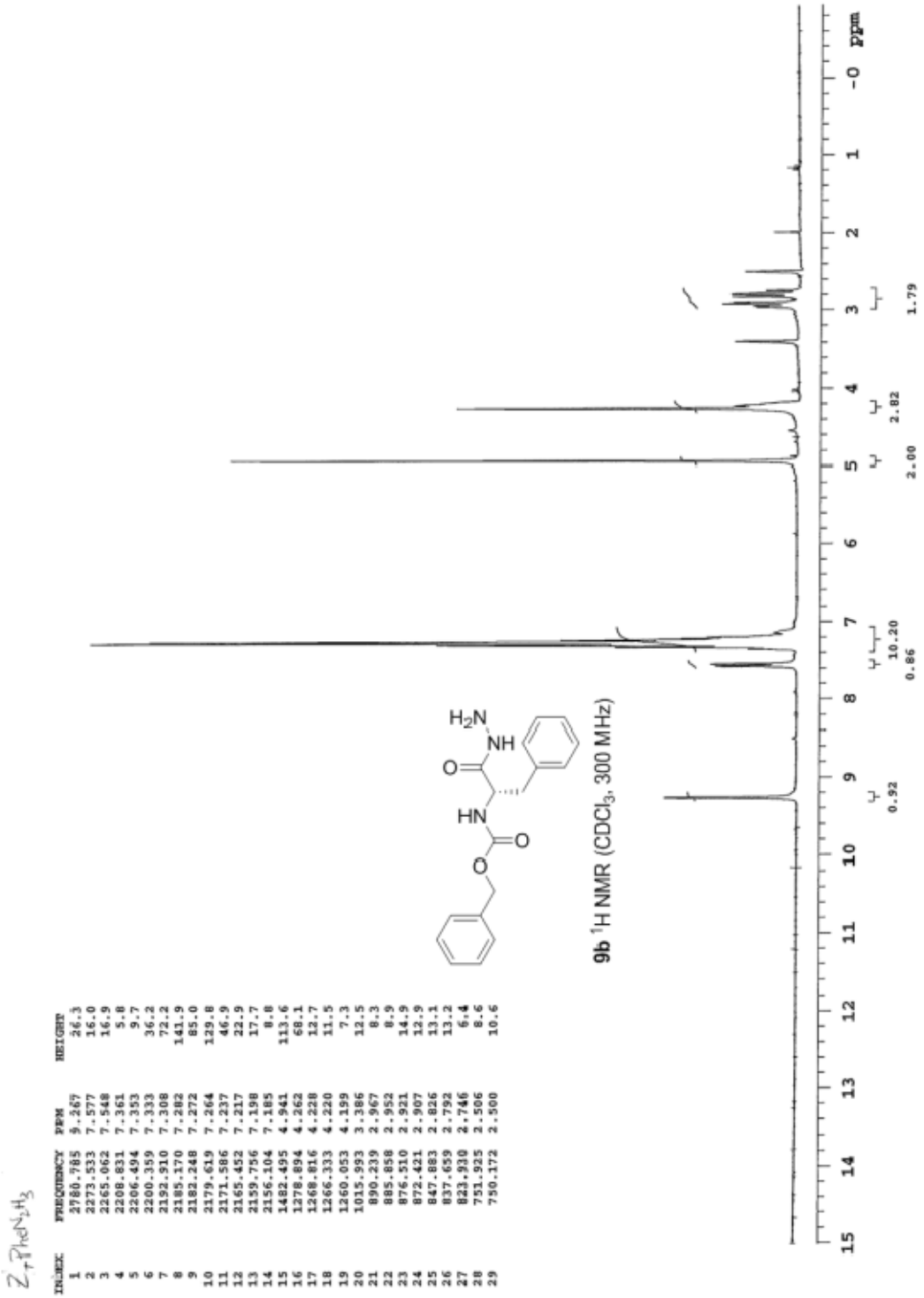
Z-Val-NH_2

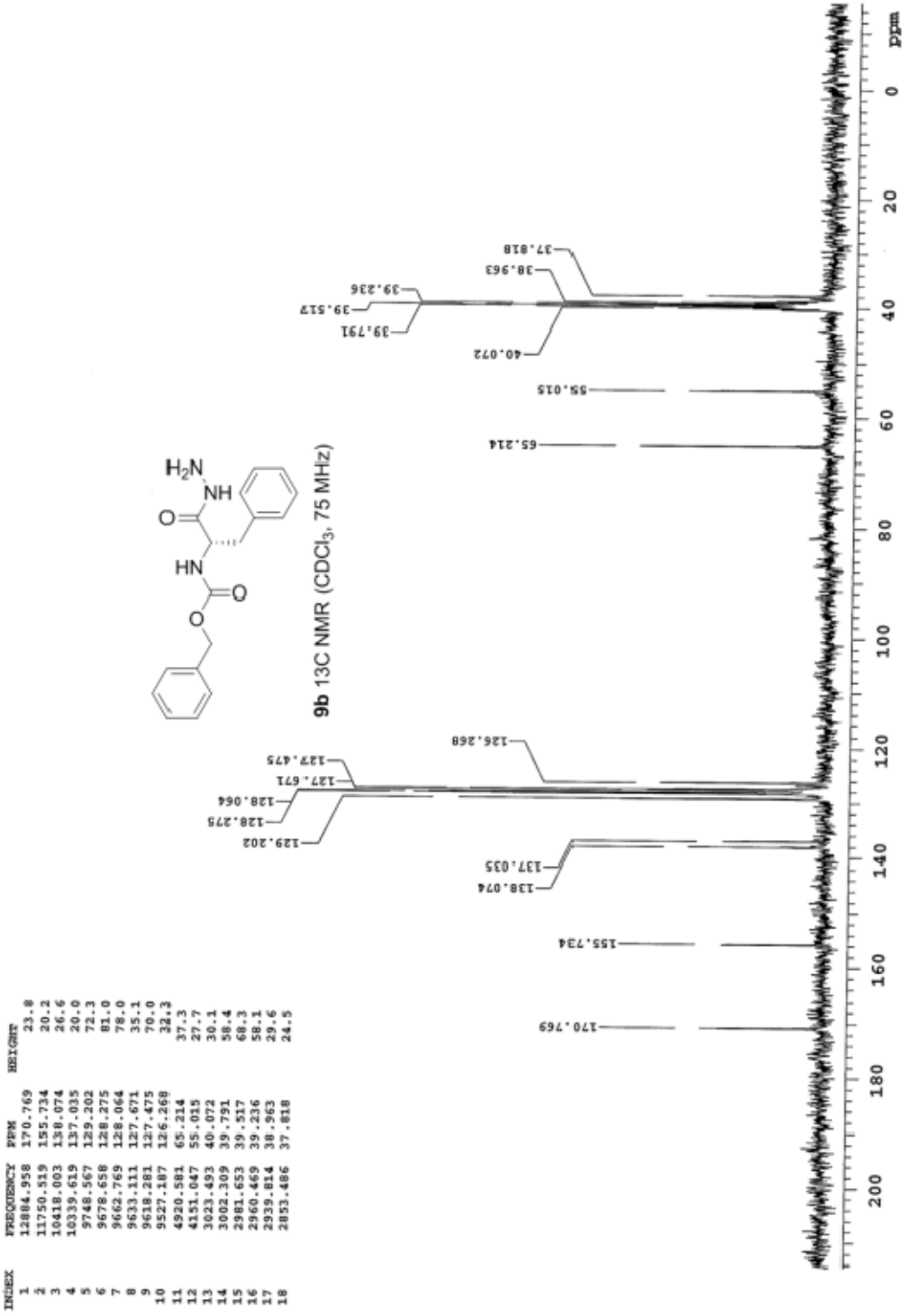


#	FREQUENCY	PPM	HEIGHT
1	12992.357	172.194	11.7
2	9710.851	128.702	53.8
3	9688.607	128.408	33.0
4	9673.778	128.211	39.8
5	5854.182	77.588	109.2
6	5821.875	77.160	112.1
7	5790.098	76.739	108.8
8	5081.471	67.347	25.4
9	4488.830	59.493	16.6
10	2336.467	30.966	33.3
11	1459.951	19.349	32.2
12	1374.153	18.212	15.2


9a' ^{1}H NMR ($CDCl_3$, 300 MHz)
H_2N
NH
O
HN
O
O
ppm
7.317
7.267
7.260
5.859
5.829
5.853
5.822
5.126
5.066
3.943
3.969
3.937
2.083
2.060
2.040
2.015
0.924
0.902
0.073
-0.000
2-DL-Val-NZH3
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300 "arkmar"
Pulse 24.5 degrees
Acq. time 4.008 sec
Width 4501.5 Hz
16 repetitions
OBSERVE H1, 300.0764259 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 2 min, 13 sec


13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300 "arkmar"
Relax. delay 0.500 sec
Pulse 33.5 degrees
Acq. time 1.300 sec
Width 18761.7 Hz
96 repetitions
OBSERVE C13, 75.4542150 MHz
DECOUPLE H4, 300.0779281 MHz
Power 8 dB
continuously on
WALTZ16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 0 min, 38 sec
9a' 13C NMR (CDCl3, 75 MHz)
ppm
0
20
40
60
80
100
120
140
160
180
200
220
19.221
19.353
30.977
59.441
67.234
76.727
77.152
77.578
109.813
112.803
126.640
126.829
128.124
136.213
156.657
172.269
193.764

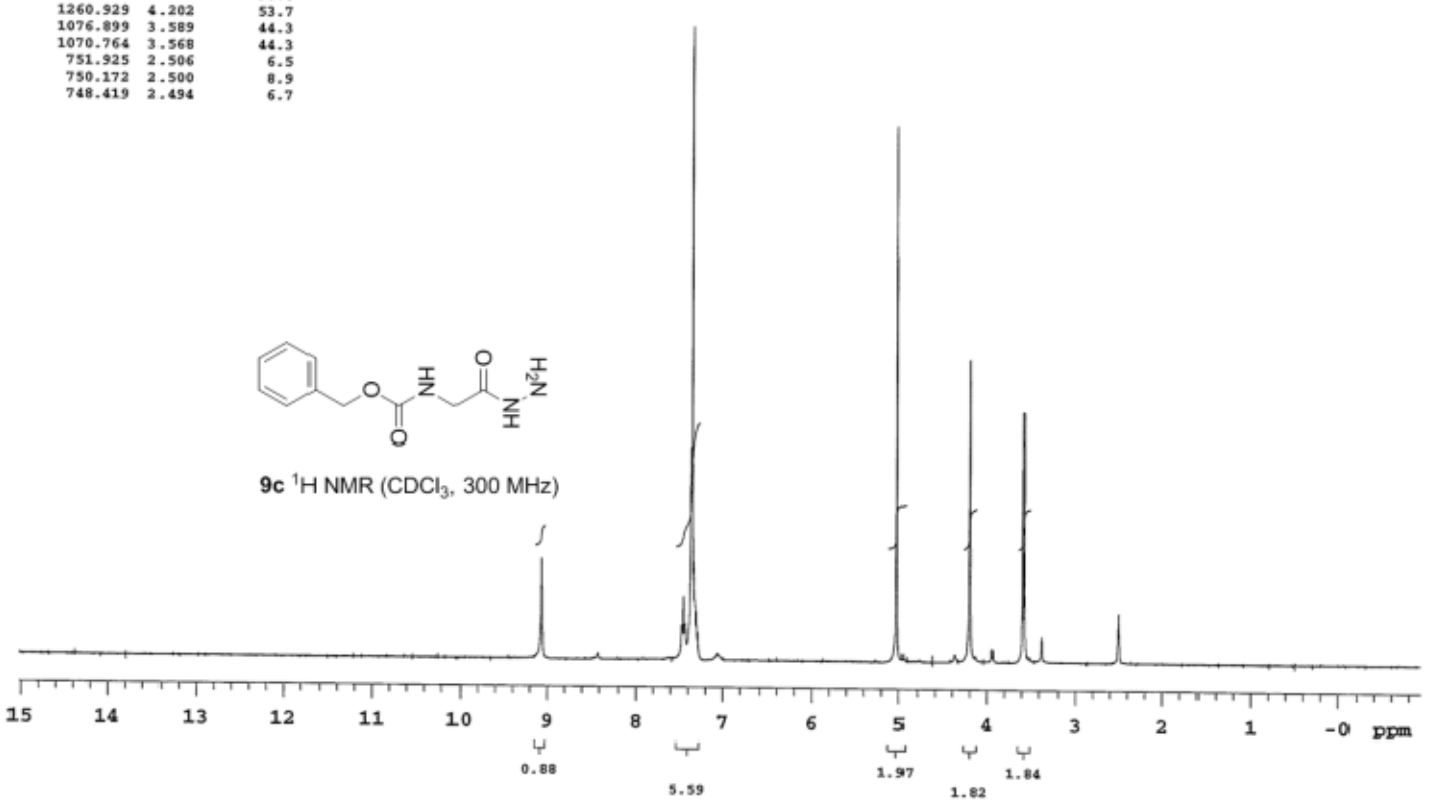

Z-Phe-NH2
INDEX FREQUENCY PPM HEIGHT
1 2780.785 9.267 26.3
2 2273.533 7.577 16.0
3 2265.062 7.548 16.9
4 2208.831 7.361 5.8
5 2206.494 7.353 9.7
6 2200.359 7.333 36.2
7 2192.910 7.308 72.2
8 2185.170 7.282 141.9
9 2182.248 7.272 85.0
10 2179.619 7.264 129.8
11 2171.586 7.237 46.9
12 2165.452 7.217 22.9
13 2159.756 7.198 17.7
14 2156.104 7.185 8.8
15 1482.495 4.941 113.6
16 1278.894 4.262 68.1
17 1268.816 4.228 13.7
18 1266.333 4.220 11.5
19 1260.053 4.199 7.3
20 1015.993 3.386 12.5
21 890.239 2.967 8.3
22 885.858 2.952 8.9
23 876.510 2.921 14.9
24 872.421 2.907 12.9
25 847.883 2.826 13.1
26 837.659 2.792 13.2
27 823.930 2.746 6.4
28 751.925 2.506 8.6
29 750.172 2.500 10.6
9b ^{1}H NMR ($CDCl_3$, 300 MHz)
0.92
10.20
0.86
2.82
2.00
1.79
ppm



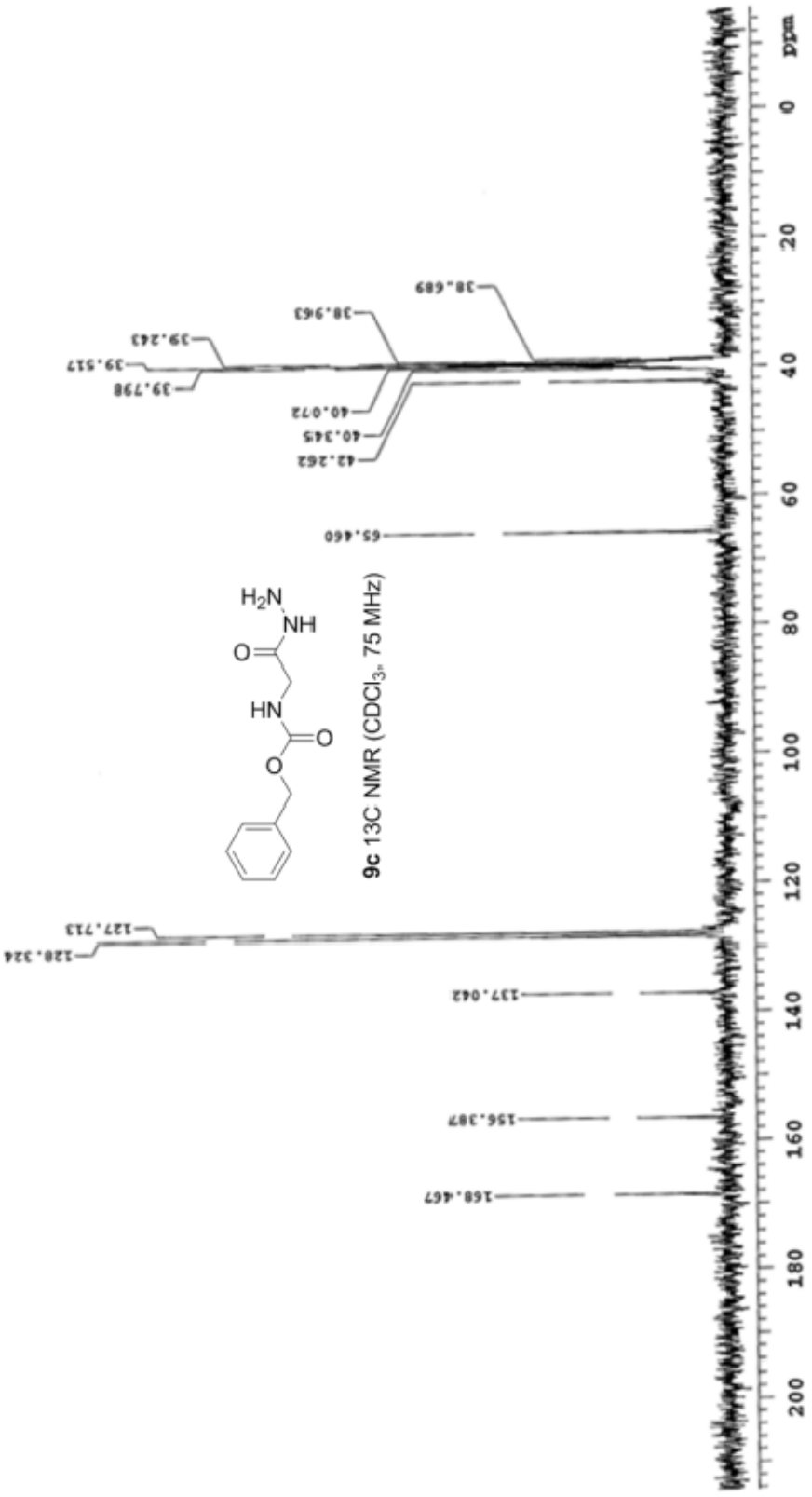
INDEX	FREQUENCY	PPM	HEIGHT
1	12884.958	170.769	23.8
2	11750.519	155.734	20.2
3	10418.003	138.074	26.6
4	10339.619	137.035	20.0
5	9748.567	129.202	72.3
6	9678.658	128.275	81.0
7	9662.769	128.064	78.0
8	9633.111	127.671	35.1
9	9618.281	127.475	70.0
10	9527.187	126.268	34.3
11	4920.581	65.214	37.3
12	4151.047	55.015	27.7
13	3023.493	40.072	30.1
14	3002.309	39.791	58.4
15	2981.653	39.517	60.3
16	2960.469	39.236	58.1
17	2939.814	38.963	29.6
18	2853.486	37.818	24.5

Z-GlyN$_2$H$_3$

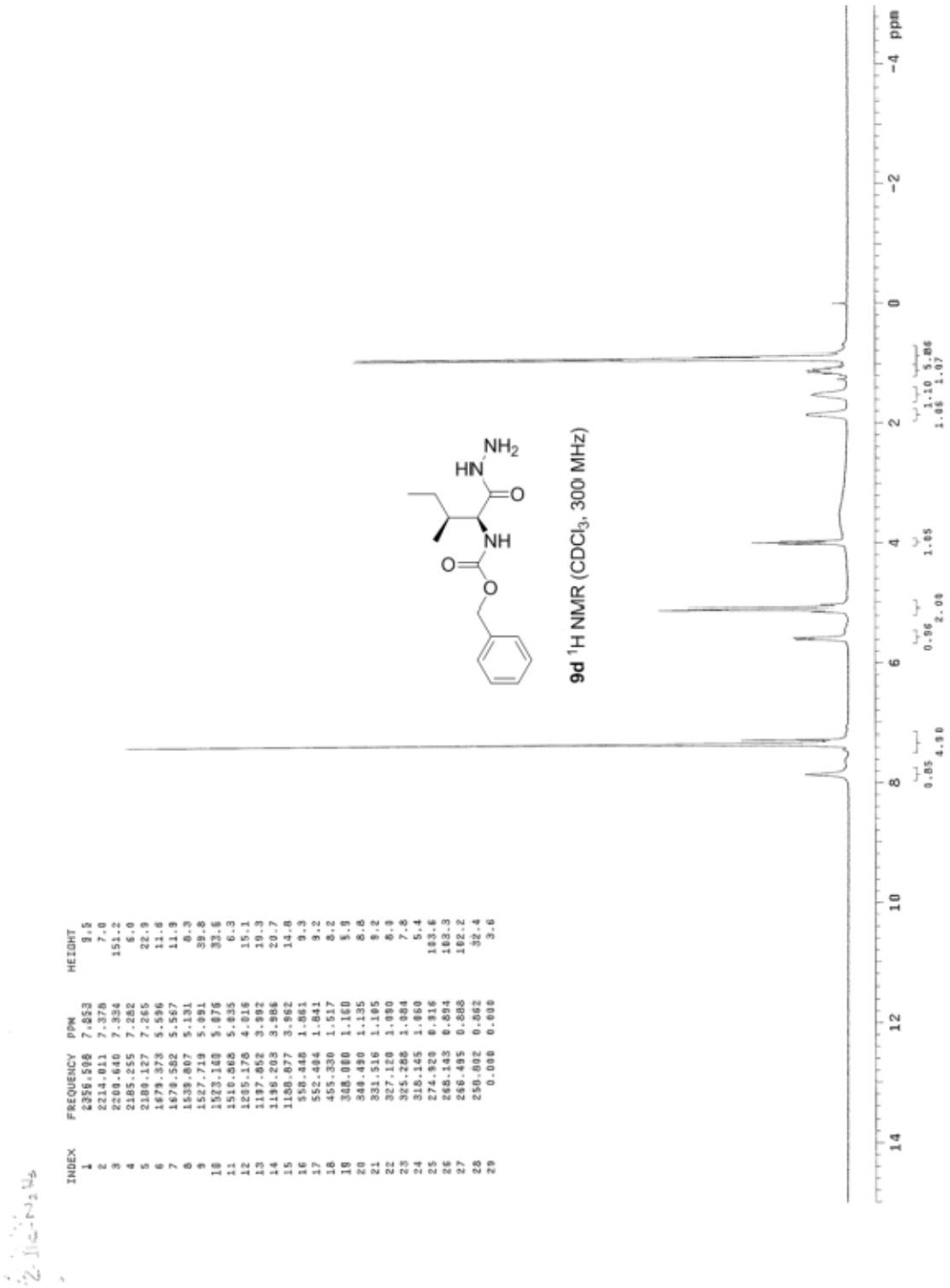
INDEX	FREQUENCY	PPM	HEIGHT
1	2720.756	9.067	17.7
2	2242.277	7.473	6.3
3	2236.289	7.453	11.3
4	2230.155	7.432	6.5
5	2209.999	7.365	37.8
6	2206.348	7.353	112.5
7	2200.067	7.332	17.4
8	2197.584	7.324	12.0
9	2191.450	7.303	8.6
10	1507.617	5.024	95.0
11	1260.929	4.202	53.7
12	1076.899	3.589	44.3
13	1070.764	3.568	44.3
14	751.925	2.506	6.5
15	750.172	2.500	8.9
16	748.419	2.494	6.7



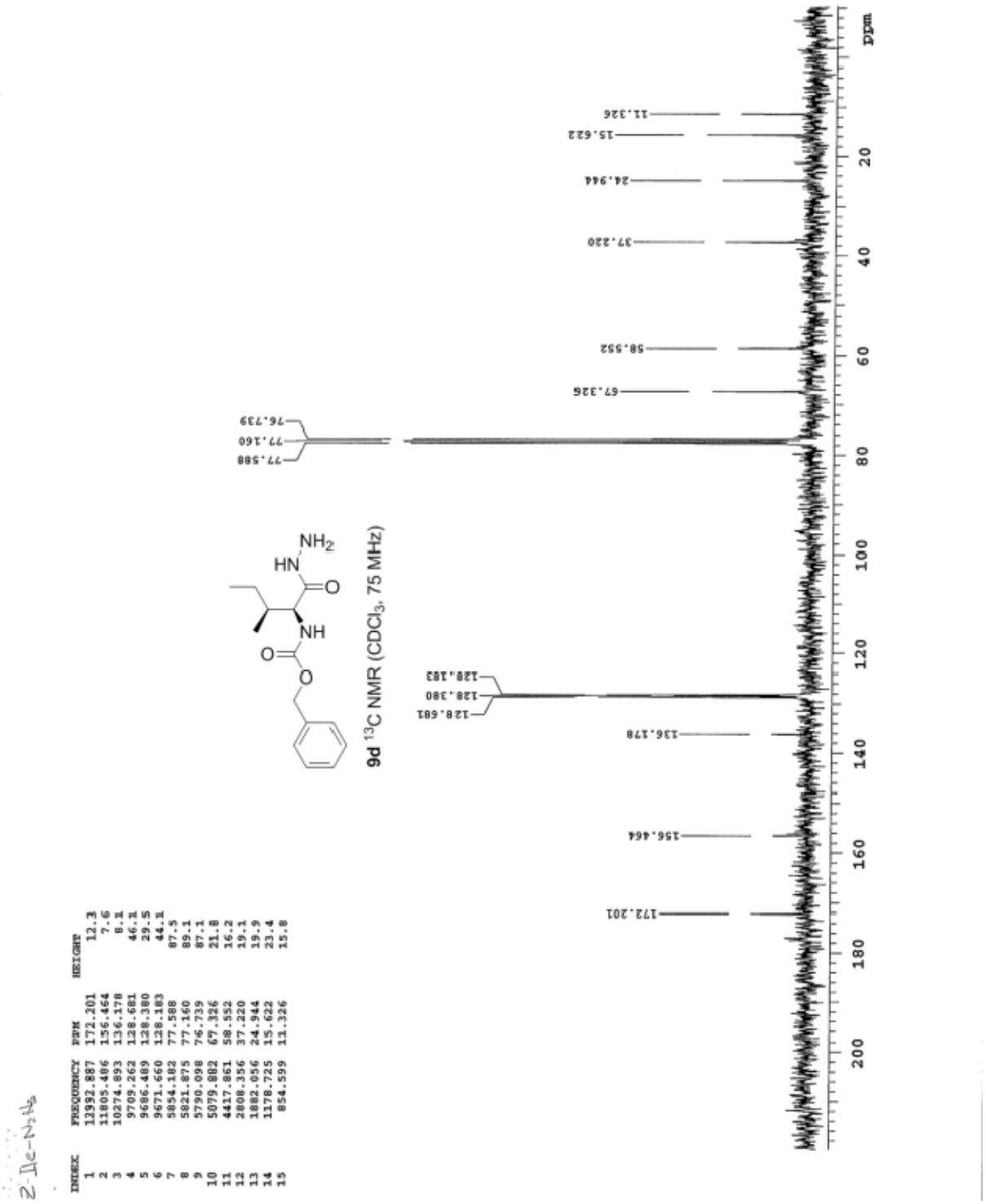
9c ^{1}H NMR ($CDCl_3$, 300 MHz)



9c 13C NMR ($CDCl_3$, 75 MHz)

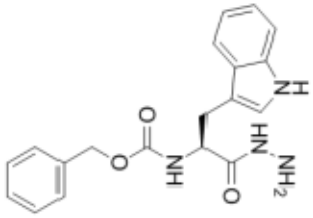


INDEX	FREQUENCY	PPM	HEIGHT
1	2356.508	7.853	9.5
2	2214.011	7.378	7.0
3	2208.640	7.334	151.2
4	2185.255	7.282	6.0
5	2180.127	7.265	22.9
6	1679.373	5.596	11.6
7	1670.582	5.567	11.9
8	1538.807	5.131	8.3
9	1527.719	5.091	39.8
10	1523.140	5.076	33.6
11	1510.868	5.035	6.3
12	1205.178	4.016	15.1
13	1197.852	3.992	10.3
14	1196.203	3.986	20.7
15	1188.877	3.962	14.8
16	558.448	1.861	9.3
17	552.404	1.841	9.2
18	455.330	1.517	8.2
19	348.080	1.160	5.9
20	340.490	1.135	8.8
21	331.516	1.105	9.2
22	327.120	1.090	8.3
23	325.288	1.084	7.8
24	318.145	1.060	5.4
25	274.920	0.916	183.6
26	268.143	0.894	183.3
27	266.495	0.888	182.2
28	250.002	0.662	32.4
29	0.000	0.000	3.6

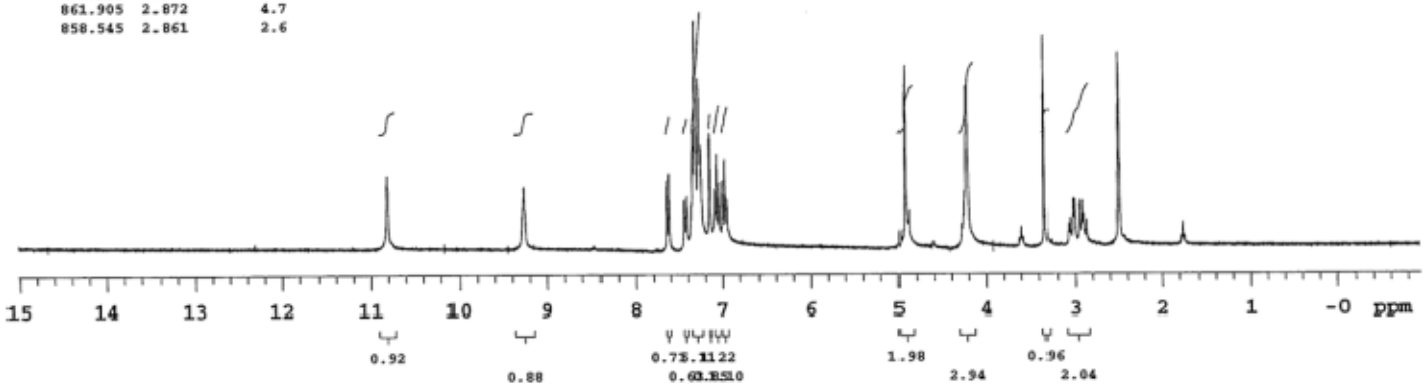


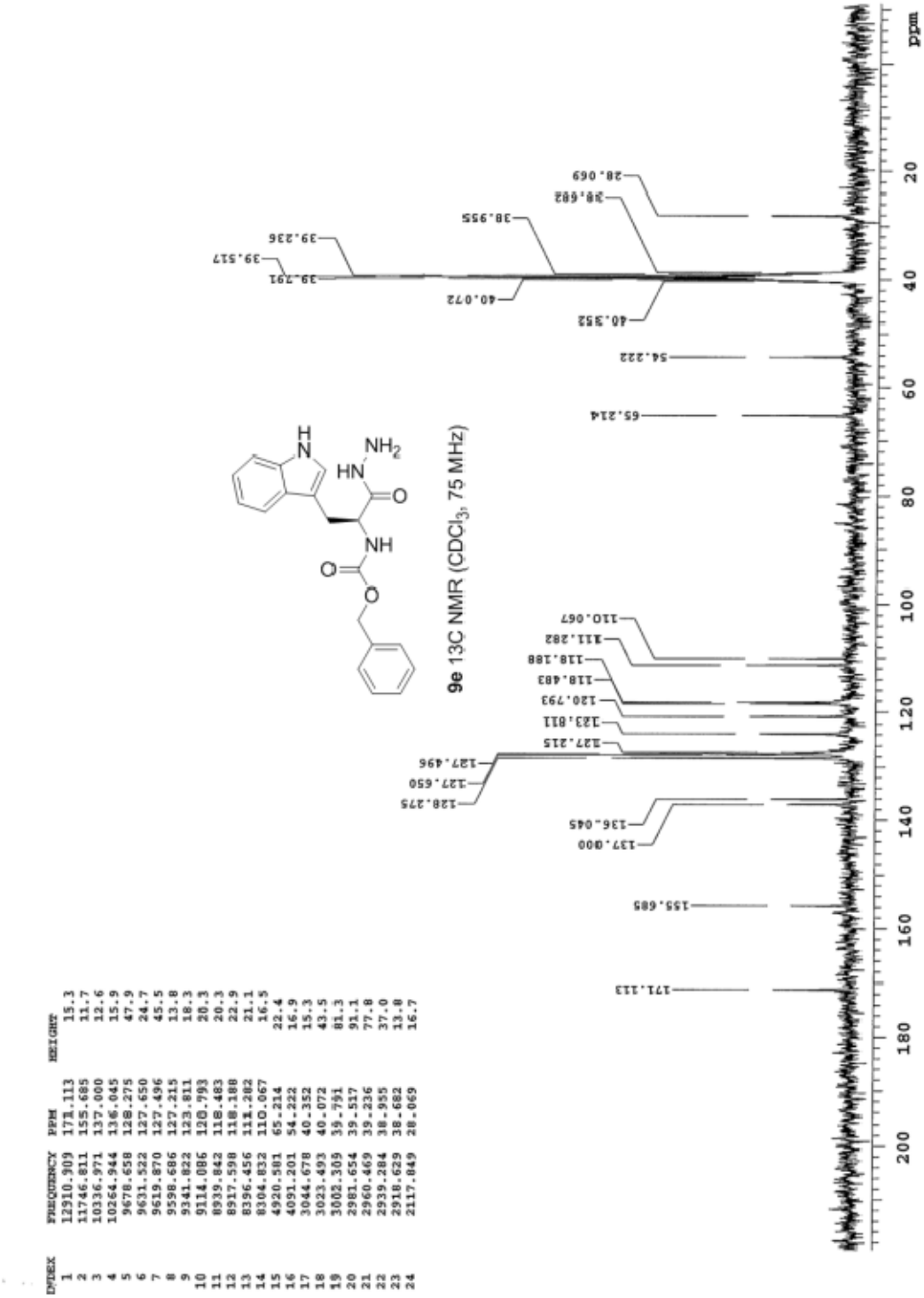
INDEX	FREQUENCY	PPM	HEIGHT
1	12992.887	172.201	12.3
2	11805.406	156.464	7.6
3	10274.893	136.178	8.1
4	9709.262	128.681	46.1
5	9686.489	128.380	29.5
6	9671.660	128.183	44.1
7	5854.182	77.588	87.5
8	5821.875	77.160	89.1
9	5790.098	76.739	87.1
10	5079.882	67.326	21.8
11	4417.861	58.552	16.2
12	2808.356	37.220	19.1
13	1882.056	24.944	19.9
14	1178.725	15.622	23.4
15	854.599	11.326	15.8

INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	3243.781	10.810	12.9	37	751.779	2.505	25.1
2	2779.616	9.263	10.9	38	750.172	2.500	34.3
3	2291.936	7.638	11.9	39	748.419	2.494	26.9
4	2284.487	7.613	13.2	40	526.853	1.756	4.3
5	2232.492	7.440	8.5				
6	2224.166	7.412	9.2				
7	2202.404	7.340	21.1				
8	2193.787	7.311	40.1				
9	2182.687	7.274	29.8				
10	2175.092	7.249	18.0				
11	2144.712	7.147	20.1				
12	2142.959	7.142	19.7				
13	2125.725	7.084	10.4				
14	2118.568	7.060	16.3				
15	2110.827	7.034	11.5				
16	2098.997	6.995	11.8				
17	2091.256	6.969	15.4				
18	2084.245	6.946	8.6				
19	1494.034	4.979	2.8				
20	1481.327	4.937	28.6				
21	1479.136	4.929	32.0				
22	1466.721	4.888	6.6				
23	1274.366	4.247	10.0				
24	1269.254	4.230	28.4				
25	1265.603	4.218	28.7				
26	1079.820	3.599	3.5				
27	1001.972	3.339	37.4				
28	922.080	3.073	4.1				
29	916.968	3.056	5.0				
30	907.620	3.025	8.6				
31	902.946	3.009	8.6				
32	885.858	2.952	8.3				
33	876.364	2.921	8.2				
34	871.252	2.904	5.9				
35	861.905	2.872	4.7				
36	858.545	2.861	2.6				

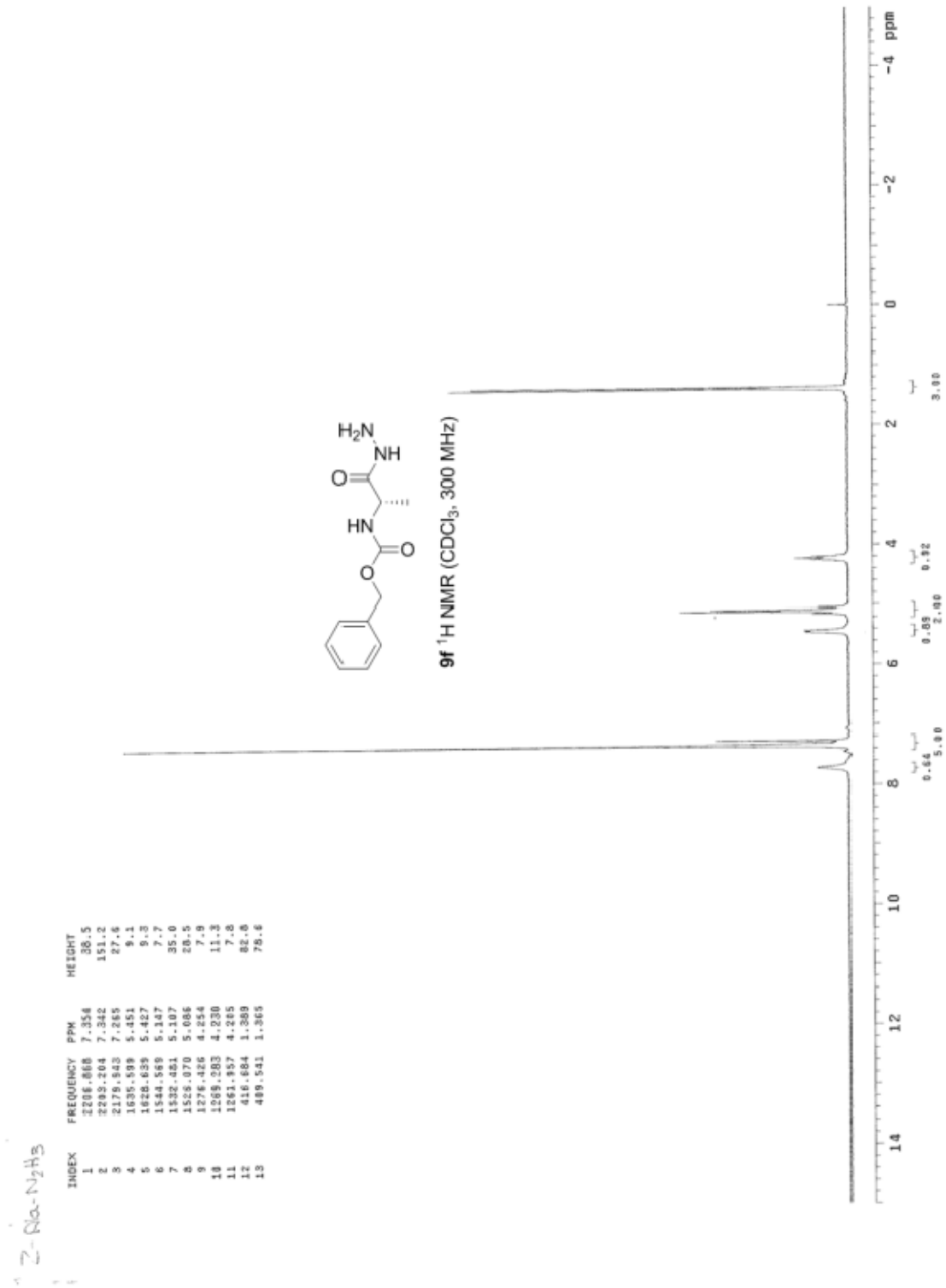


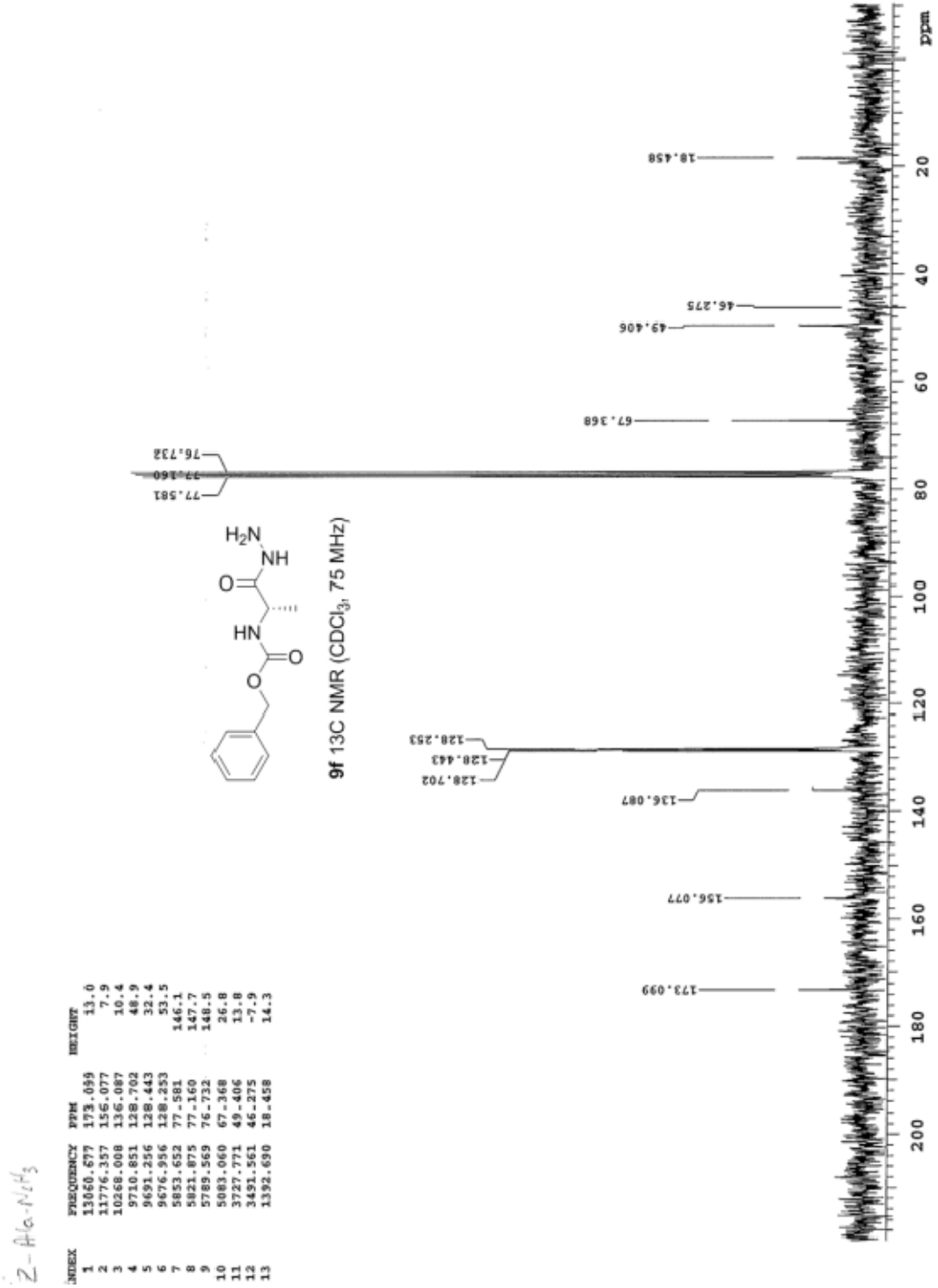
9e ^{1}H NMR ($CDCl_3$, 300 MHz)



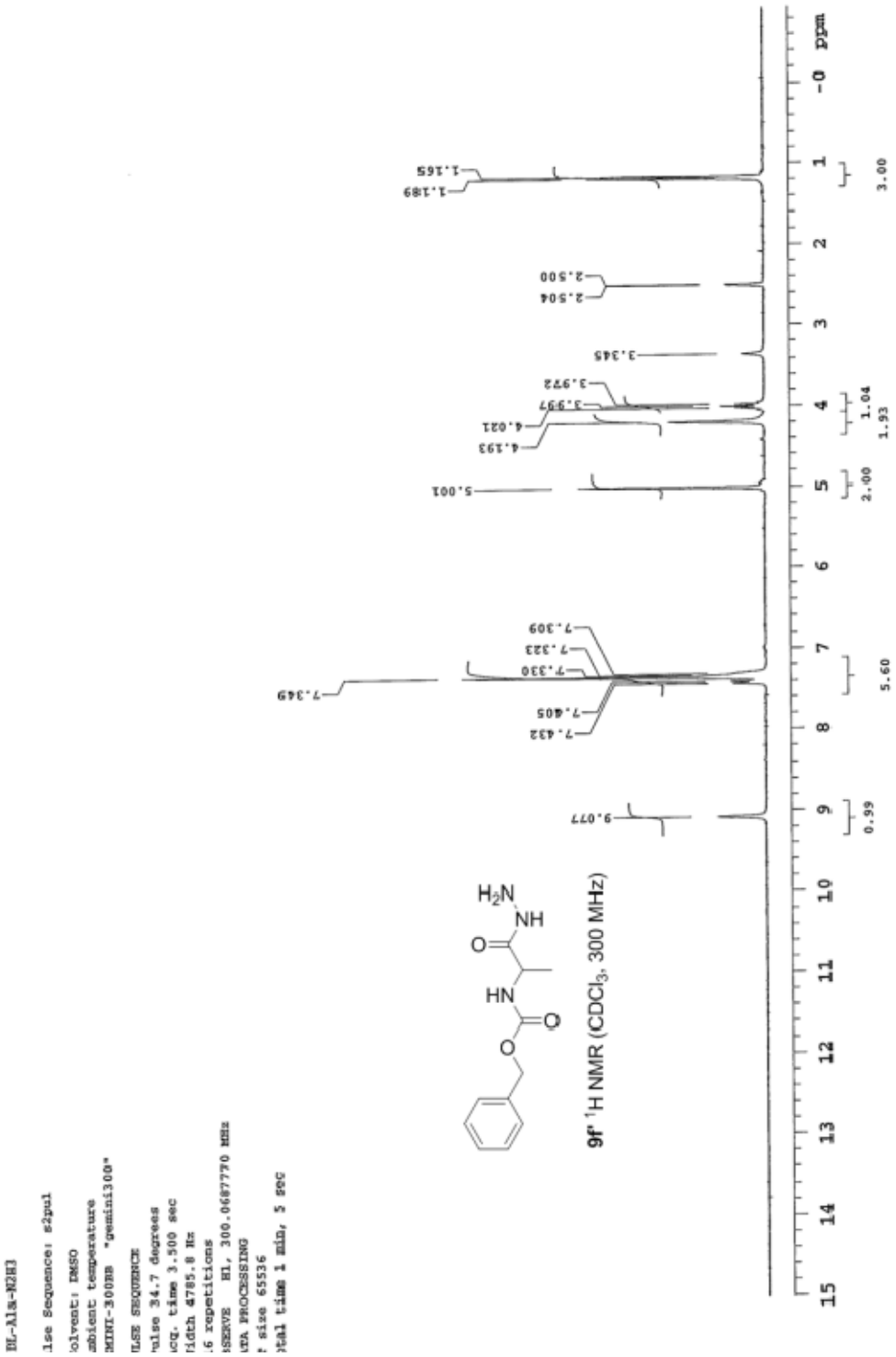


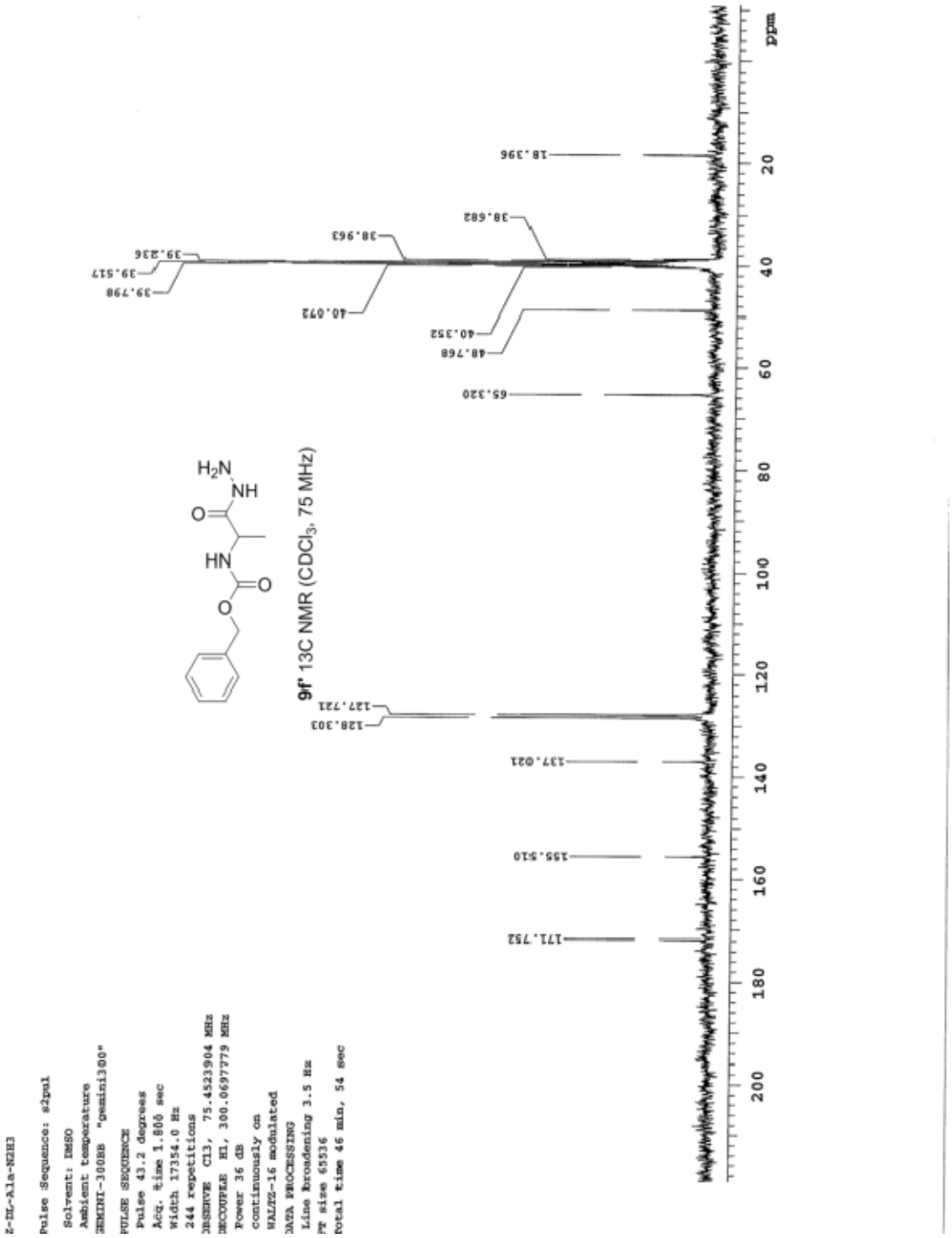
INDEX	FREQUENCY	PPM	HEIGHT
1	12910.909	171.113	15.3
2	11746.811	155.685	11.7
3	10336.971	137.000	12.6
4	10264.944	136.045	15.9
5	9678.658	128.275	47.9
6	9631.522	127.650	24.7
7	9619.870	127.496	45.5
8	9598.686	127.215	13.8
9	9341.822	123.811	18.3
10	9114.086	120.793	20.3
11	8939.842	118.483	20.3
12	8917.598	118.188	22.9
13	8396.456	118.282	21.1
14	8304.832	110.067	16.5
15	4920.501	65.214	22.4
16	4091.201	54.222	16.9
17	3044.678	40.352	15.3
18	3023.493	40.072	43.5
19	3002.309	39.791	81.3
20	2981.654	39.517	91.1
21	2960.469	39.236	77.8
22	2939.284	38.955	37.0
23	2918.629	38.682	13.8
24	2117.849	28.069	16.7

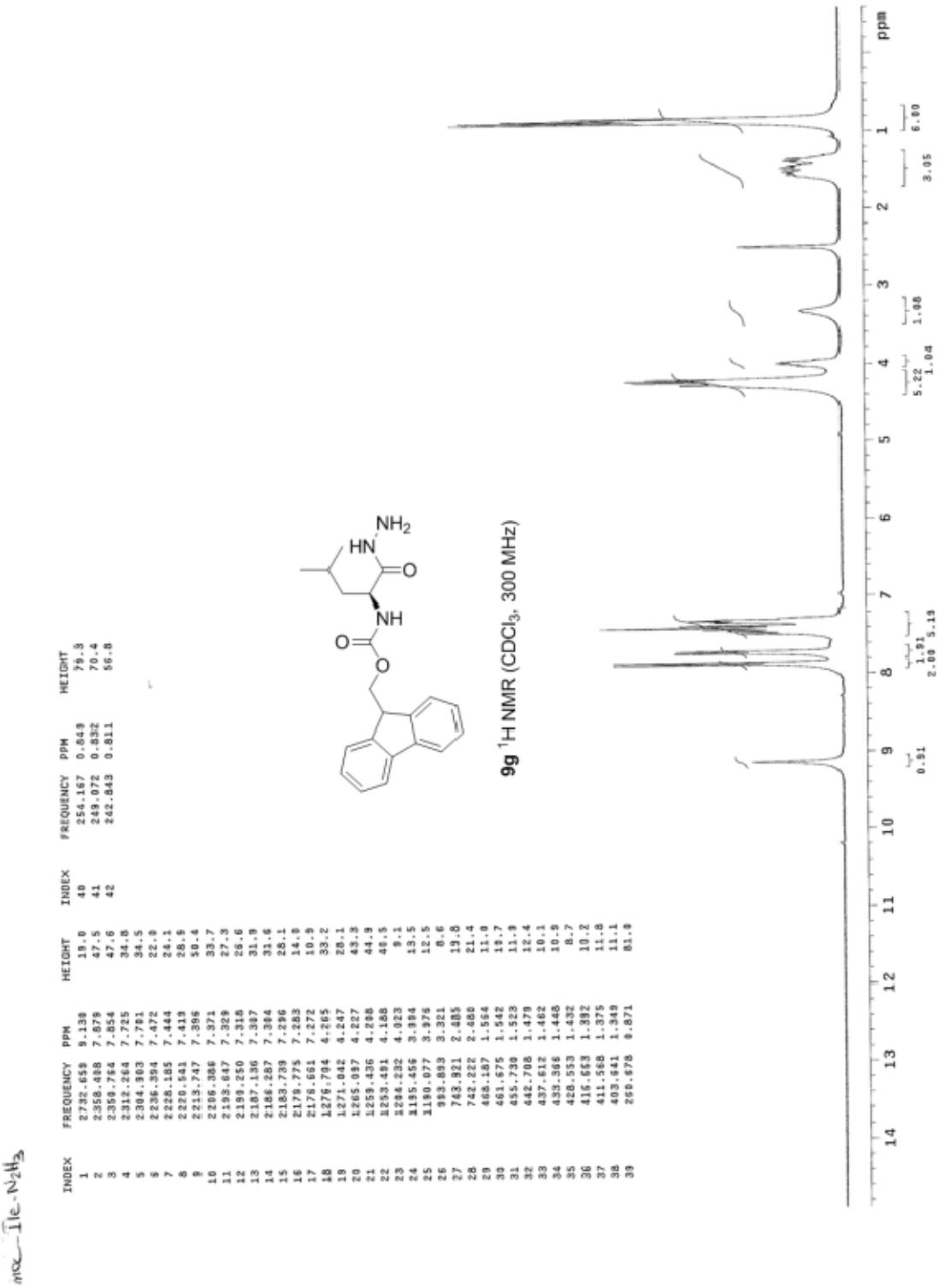

Z-Ala-NHNH2
INDEX FREQUENCY PPM HEIGHT
1 2206.868 7.356 38.5
2 2209.204 7.342 151.2
3 2179.943 7.265 27.6
4 1635.599 5.451 9.1
5 1628.639 5.427 9.3
6 1544.569 5.147 7.7
7 1532.481 5.107 35.0
8 1526.070 5.086 28.5
9 1276.426 4.254 7.9
10 1269.083 4.230 11.3
11 1261.957 4.205 7.8
12 416.684 1.389 82.6
13 409.541 1.365 78.6
9f ^{1}H NMR (CDCl$_3$, 300 MHz)
H2N
NH
O
HN
O
O
14
12
10
8
6
4
2
0
-2
-4 ppm
0.64
5.00
0.89
2.00
0.92
3.00

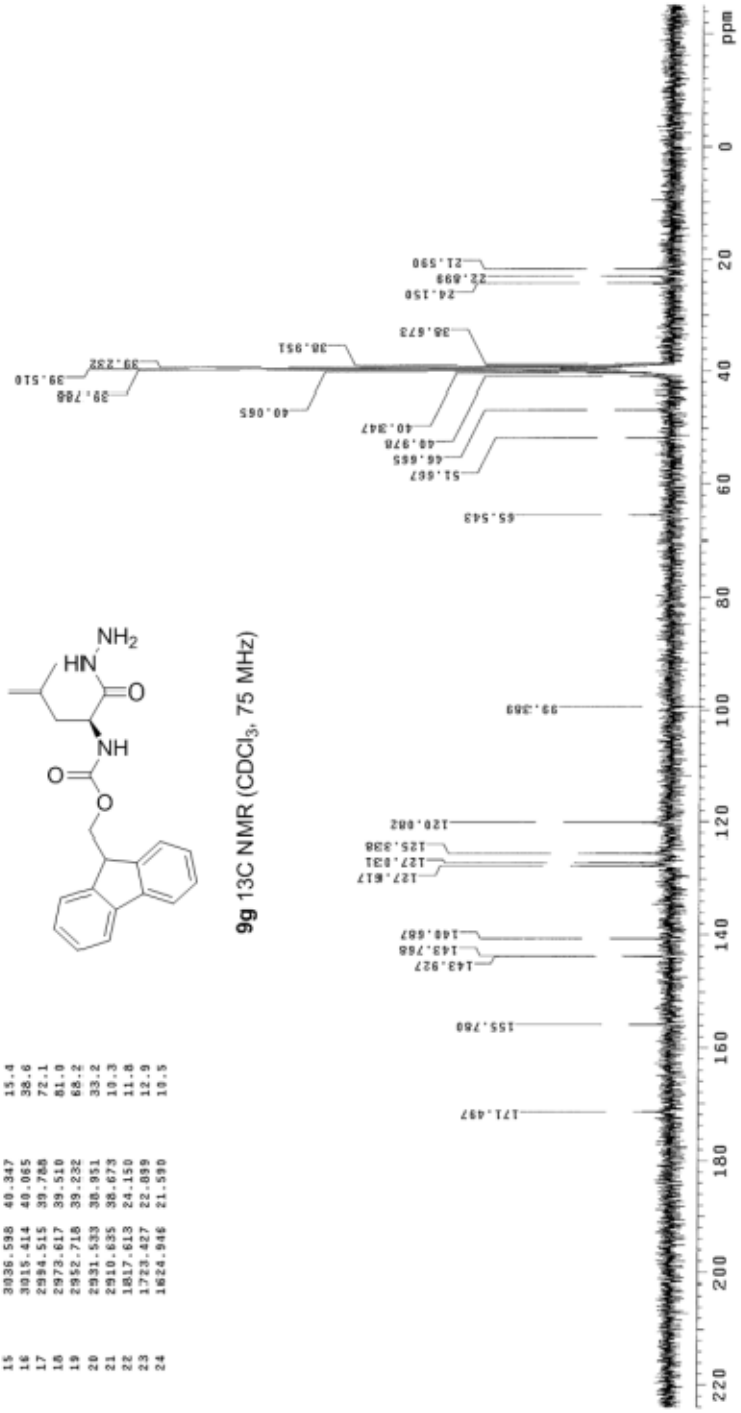


INDEX	FREQUENCY	PPM	HEIGHT
1	13060.677	173.099	13.0
2	11776.357	156.077	7.9
3	10268.008	136.087	10.4
4	9710.851	128.702	48.9
5	9691.256	128.443	32.4
6	9676.956	128.253	53.5
7	5853.652	77.581	146.1
8	5821.875	77.160	147.7
9	5789.569	76.732	148.5
10	5083.060	67.368	26.8
11	3727.771	49.406	13.8
12	3491.561	46.275	-7.9
13	1392.690	18.458	14.3


BL-Ala-N2H3
lse Sequence: s2pul
olvent: DMSO
mbient temperature
MINI-300BB "gemini300"
ULSE SEQUENCE
ulse 34.7 degrees
cq. time 3.500 sec
idth 4785.8 Hz
6 repetitions
SERVE H1, 300.0687770 MHz
TA PROCESSING
t size 65536
otal time 1 min, 5 sec
9f' ^{1}H NMR ($CDCl_3$, 300 MHz)
H_2N
NH
O
HN
O
O
1.165
1.189
2.500
2.504
3.345
3.972
3.997
4.021
4.193
5.001
7.309
7.323
7.330
7.349
7.405
7.432
9.077
ppm
3.00
1.04
1.93
2.00
5.60
0.99

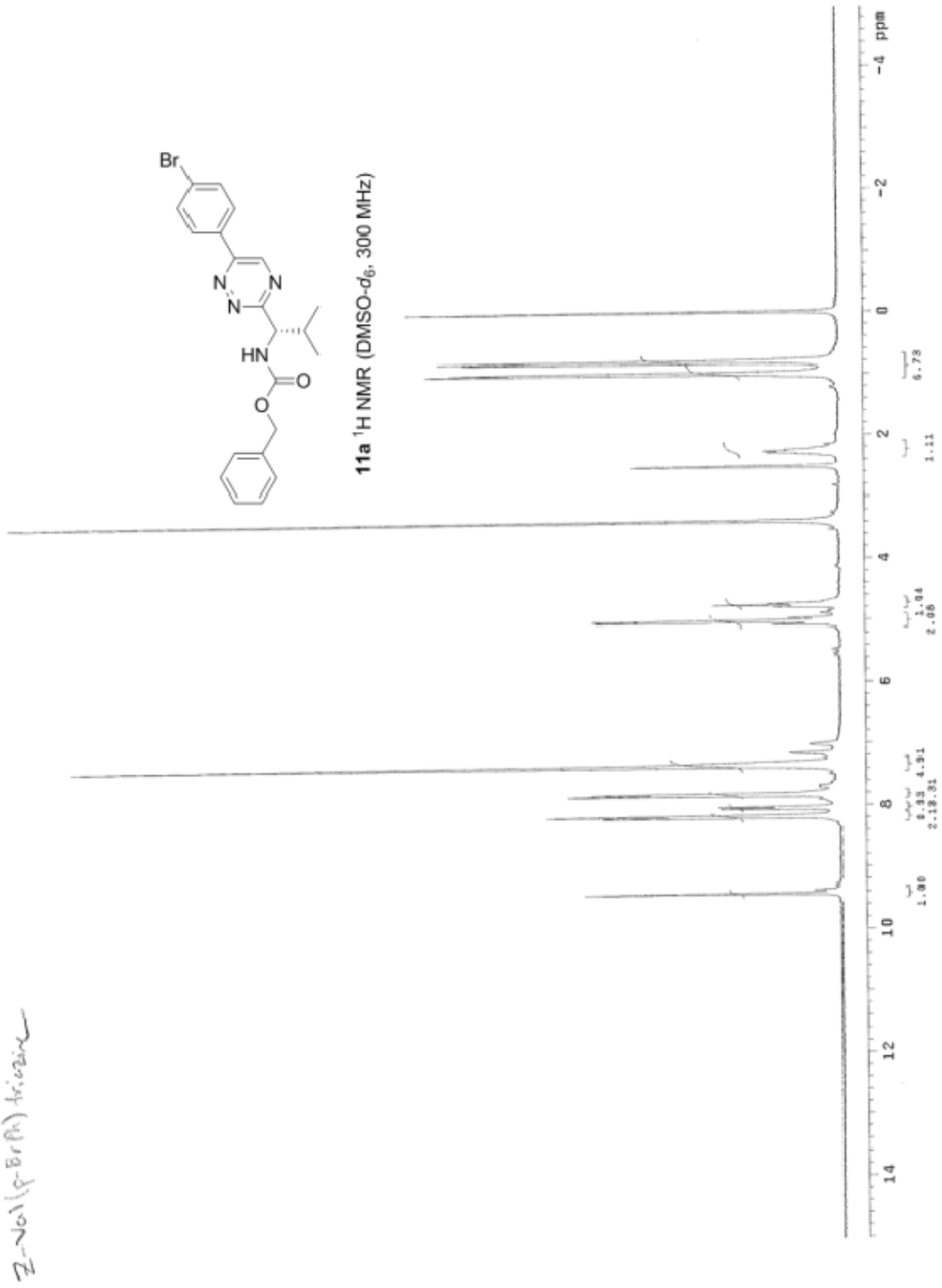

9f 13C NMR (CDCl3, 75 MHz)
H2N
NH
O
HN
O
O
18.396
38.682
38.963
39.236
39.517
39.798
40.072
40.352
48.768
65.320
127.731
128.303
137.021
155.510
171.752
ppm
20
40
60
80
100
120
140
160
180
200


mor-Ile-NH2Me
INDEX FREQUENCY PPM HEIGHT INDEX FREQUENCY PPM HEIGHT
1 2732.659 9.138 19.0 40 254.167 0.849 79.3
2 2358.488 7.879 47.5 41 249.072 0.832 70.4
3 2350.764 7.854 47.6 42 242.843 0.811 56.8
4 2312.264 7.725 34.8
5 2304.303 7.701 34.5
6 2236.394 7.472 22.0
7 2228.185 7.444 24.1
8 2220.541 7.419 28.9
9 2213.747 7.396 50.4
10 2206.386 7.371 33.7
11 2193.647 7.329 27.3
12 2190.250 7.318 26.6
13 2187.136 7.307 31.9
14 2186.287 7.304 31.6
15 2183.739 7.296 28.1
16 2178.775 7.283 14.0
17 2176.661 7.272 10.9
18 1276.794 4.265 33.2
19 1271.842 4.247 28.1
20 1265.097 4.227 43.3
21 1259.436 4.208 44.9
22 1253.491 4.188 40.5
23 1234.232 4.023 9.1
24 1195.456 3.994 13.5
25 1190.077 3.976 12.5
26 993.893 3.321 0.6
27 743.321 2.485 13.8
28 742.222 2.480 21.4
29 468.187 1.564 11.0
30 461.675 1.542 19.7
31 455.730 1.523 11.9
32 442.708 1.479 12.4
33 437.612 1.462 10.1
34 433.366 1.448 10.9
35 428.553 1.432 8.7
36 416.663 1.392 10.2
37 411.568 1.375 11.8
38 403.641 1.349 11.1
39 269.478 0.871 81.0
NH2
HN
O
NH
O
O
9g 1H NMR (CDCl3, 300 MHz)
ppm
0.91
1.91
2.00 5.13
5.22
1.04
1.08
3.05
6.80



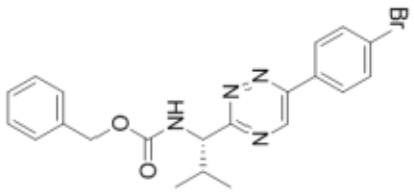
9g 13C NMR ($CDCl_3$, 75 MHz)

INDEX	FREQUENCY	PPM	HEIGHT
1	12307.288	171.497	6.2
2	11724.375	155.780	7.1
3	10832.322	143.927	8.1
4	10820.239	143.768	8.5
5	10588.411	148.687	10.6
6	9484.749	127.817	17.8
7	9560.661	127.831	17.1
8	9433.266	125.338	16.4
9	9037.625	120.882	19.1
10	7480.256	99.389	-5.8
11	4932.925	65.543	7.6
12	3888.571	51.687	8.4
13	3512.112	46.655	10.3
14	3084.121	40.976	7.6
15	3036.598	40.347	15.4
16	3015.414	40.055	38.6
17	2994.515	39.788	72.1
18	2973.617	39.510	81.0
19	2952.718	39.232	68.2
20	2931.533	38.951	33.2
21	2910.635	38.673	10.3
22	1817.613	24.150	11.8
23	1723.427	22.899	12.9
24	1624.946	21.590	10.5

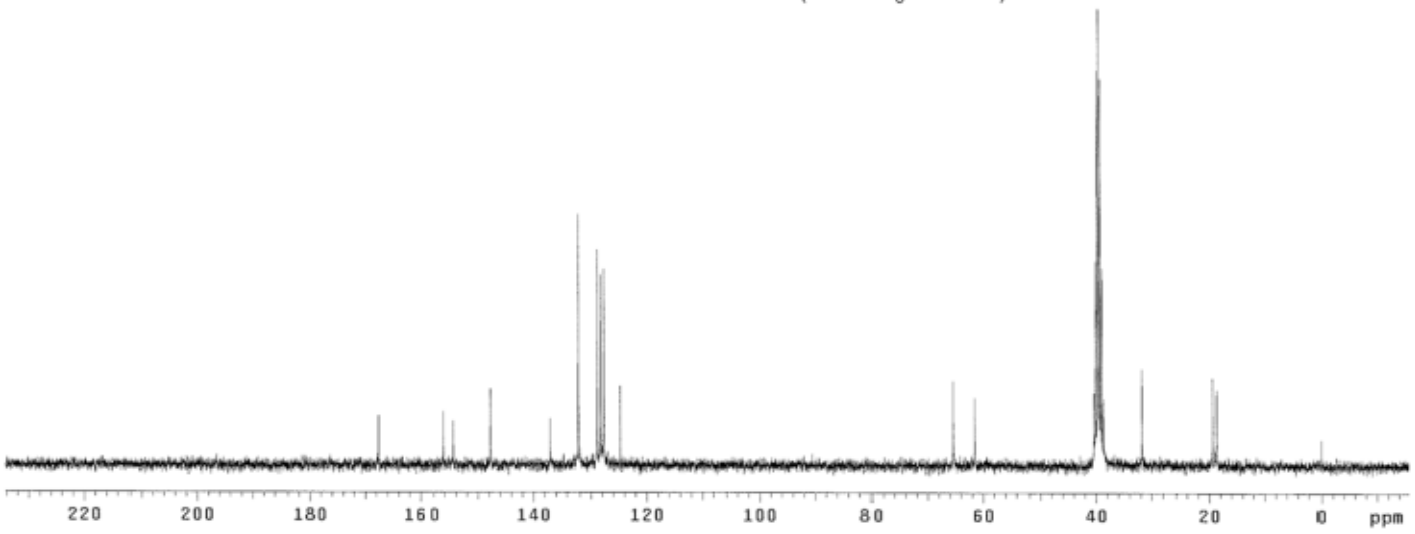

Br
N
N
N
HN
O
O
11a ^{1}H NMR (DMSO-d_6, 300 MHz)
Z-Val(p-Br-Ph) triazine
ppm
14
12
10
8
6
4
2
0
-2
-4

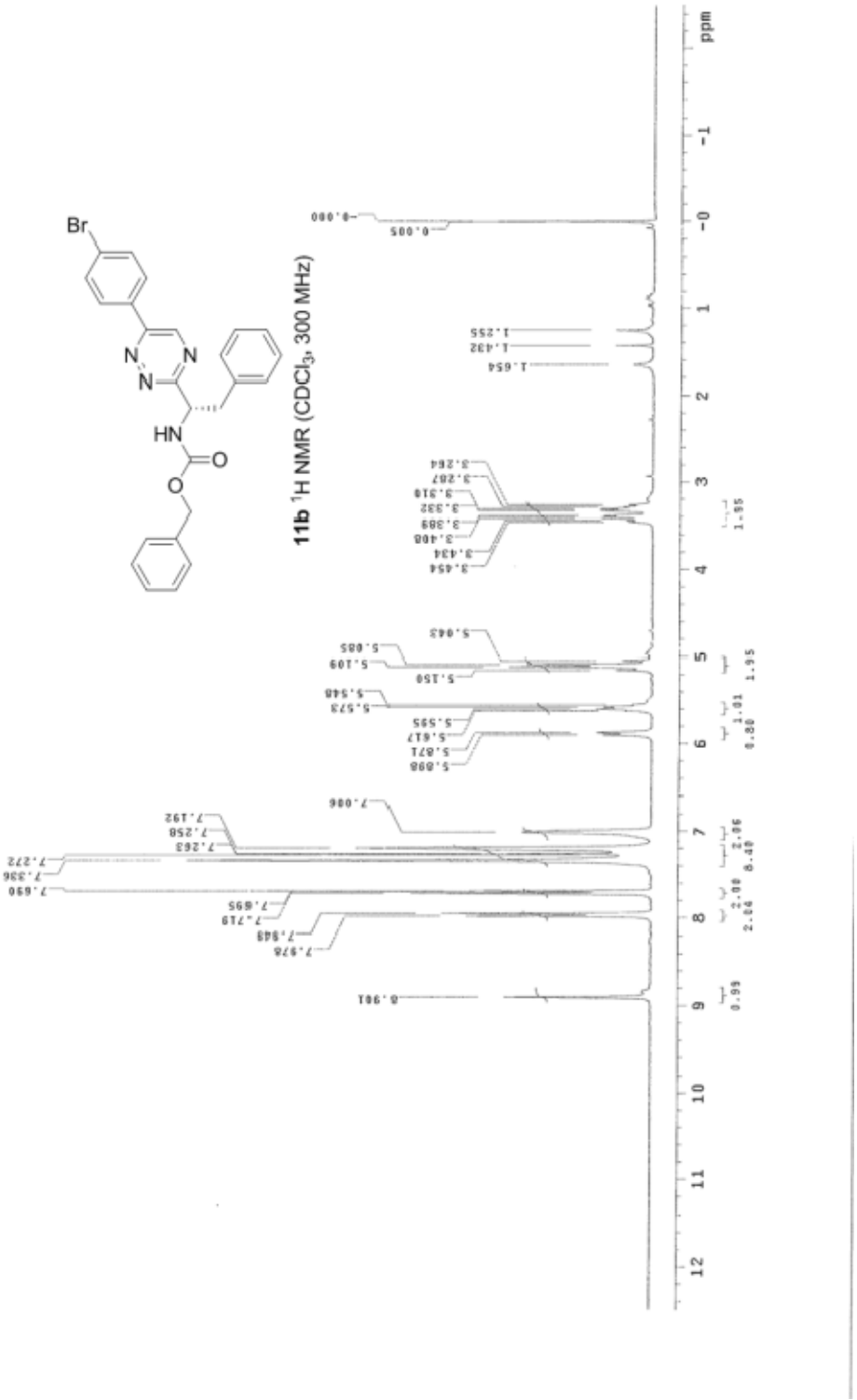
2-Val triazine (p-Br/Ph)

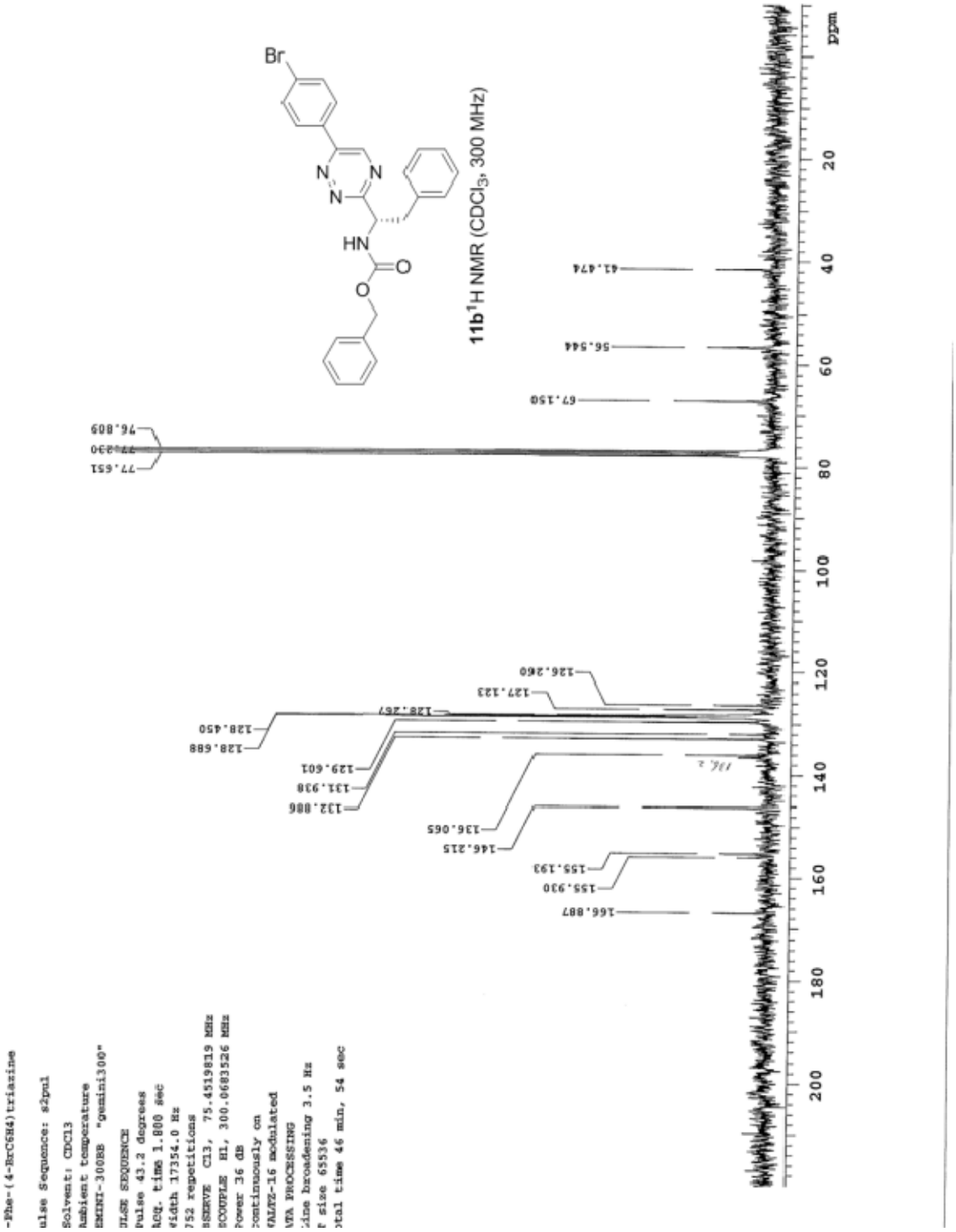
INDEX	FREQUENCY	PPM	HEIGHT
1	12648.405	167.587	8.8
2	11793.036	156.254	9.4
3	11658.872	154.476	7.9
4	11150.430	147.748	13.4
5	10339.399	136.994	8.2
6	9985.562	132.305	17.8
7	9980.380	132.237	44.2
8	9725.583	128.861	37.8
9	9684.412	128.315	33.8
10	9643.530	127.774	19.8
11	9638.347	127.705	34.8
12	9417.235	124.775	13.8
13	4939.144	65.442	14.8
14	4645.768	61.555	11.7
15	3044.722	40.342	12.7
16	3023.785	40.063	36.3
17	3002.976	39.788	69.8
18	2981.959	39.510	81.9
19	2960.942	39.232	68.4
20	2939.925	38.953	35.9
21	2919.195	38.678	11.5
22	2396.358	31.751	16.9
23	1463.830	19.395	15.4
24	1407.688	18.651	13.3

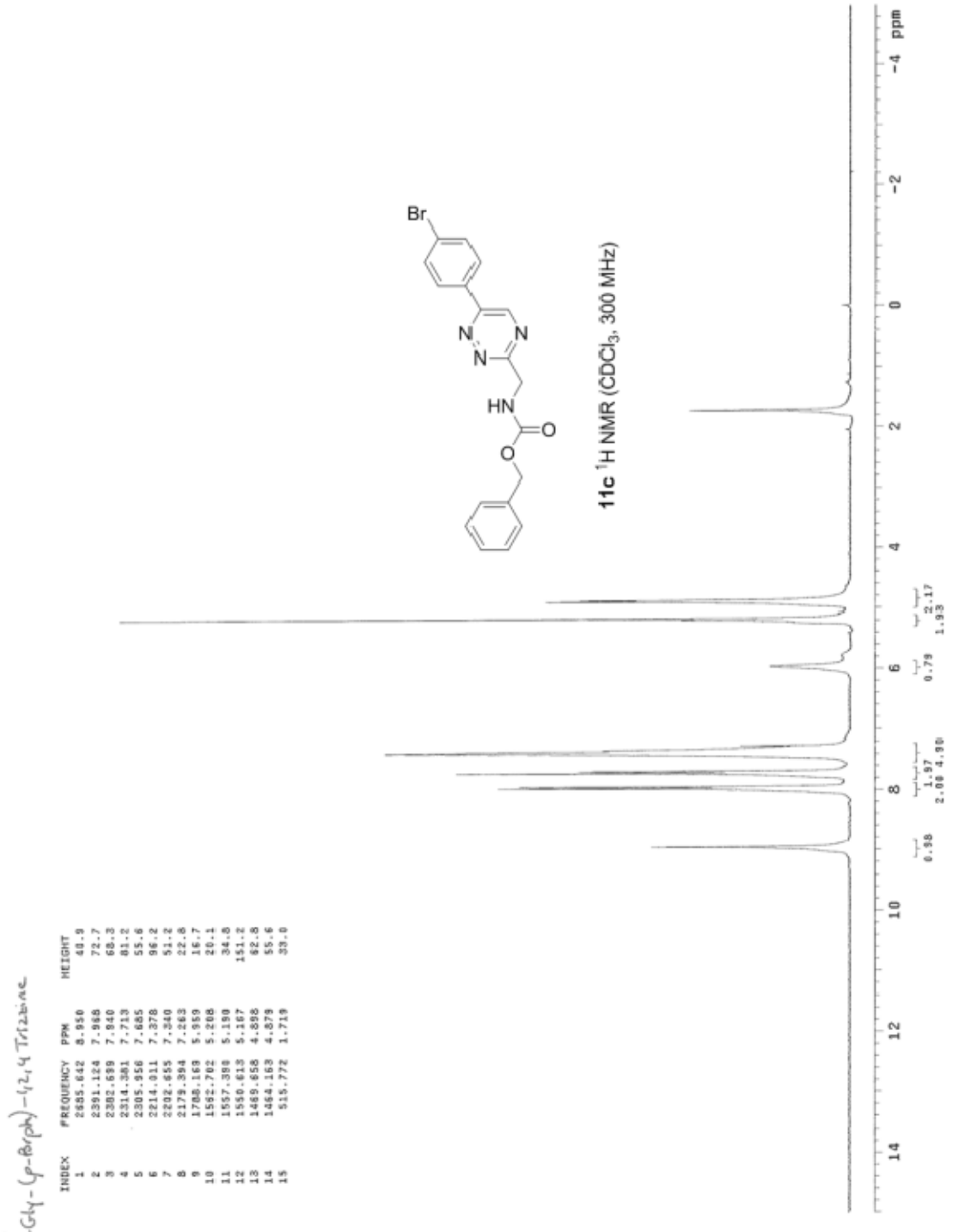


11a ^{1}H NMR (DMSO-d_6 300 MHz)



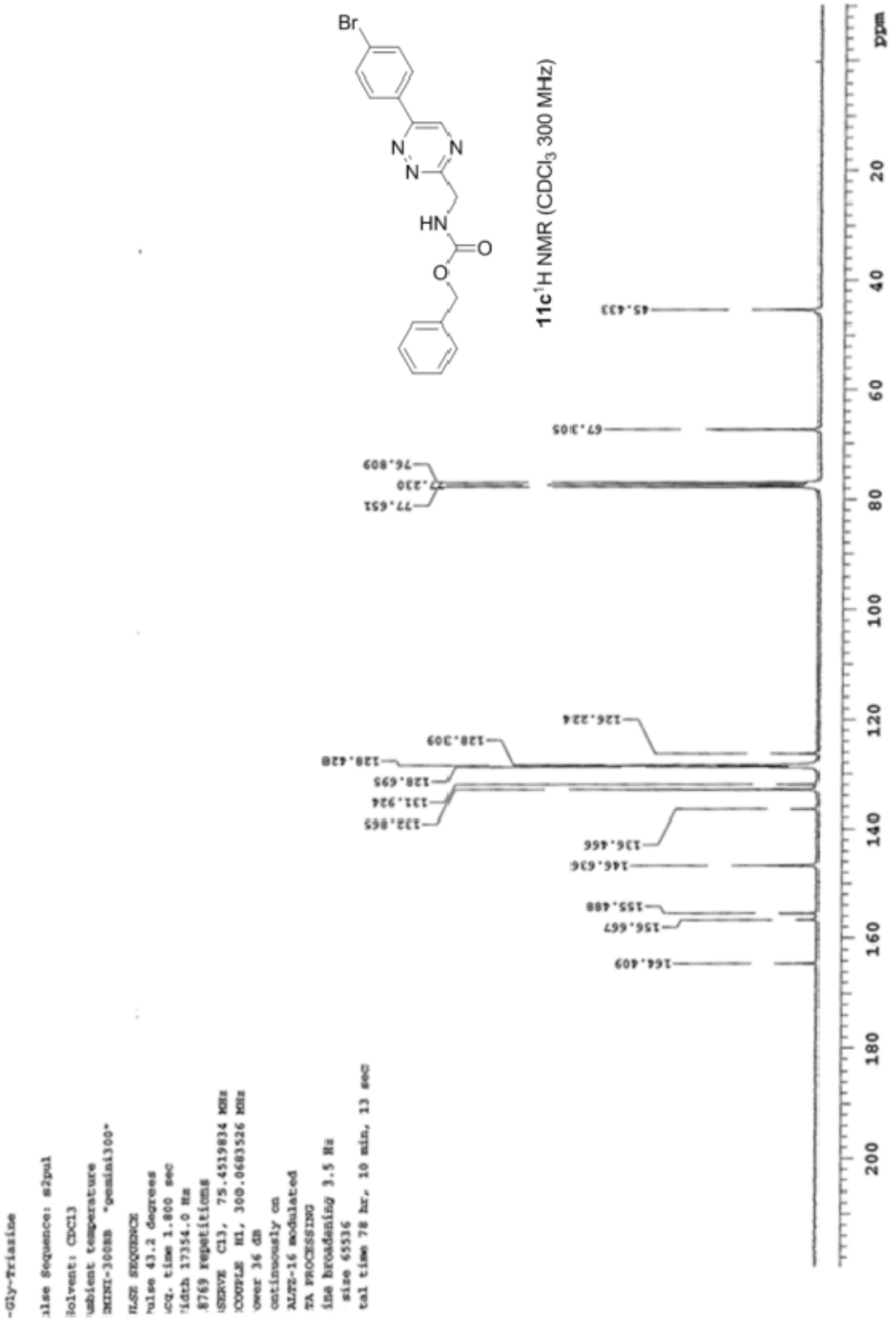

Br
N
N
N
HN
O
O
11b ^{1}H NMR (CDCl$_3$, 300 MHz)
ppm

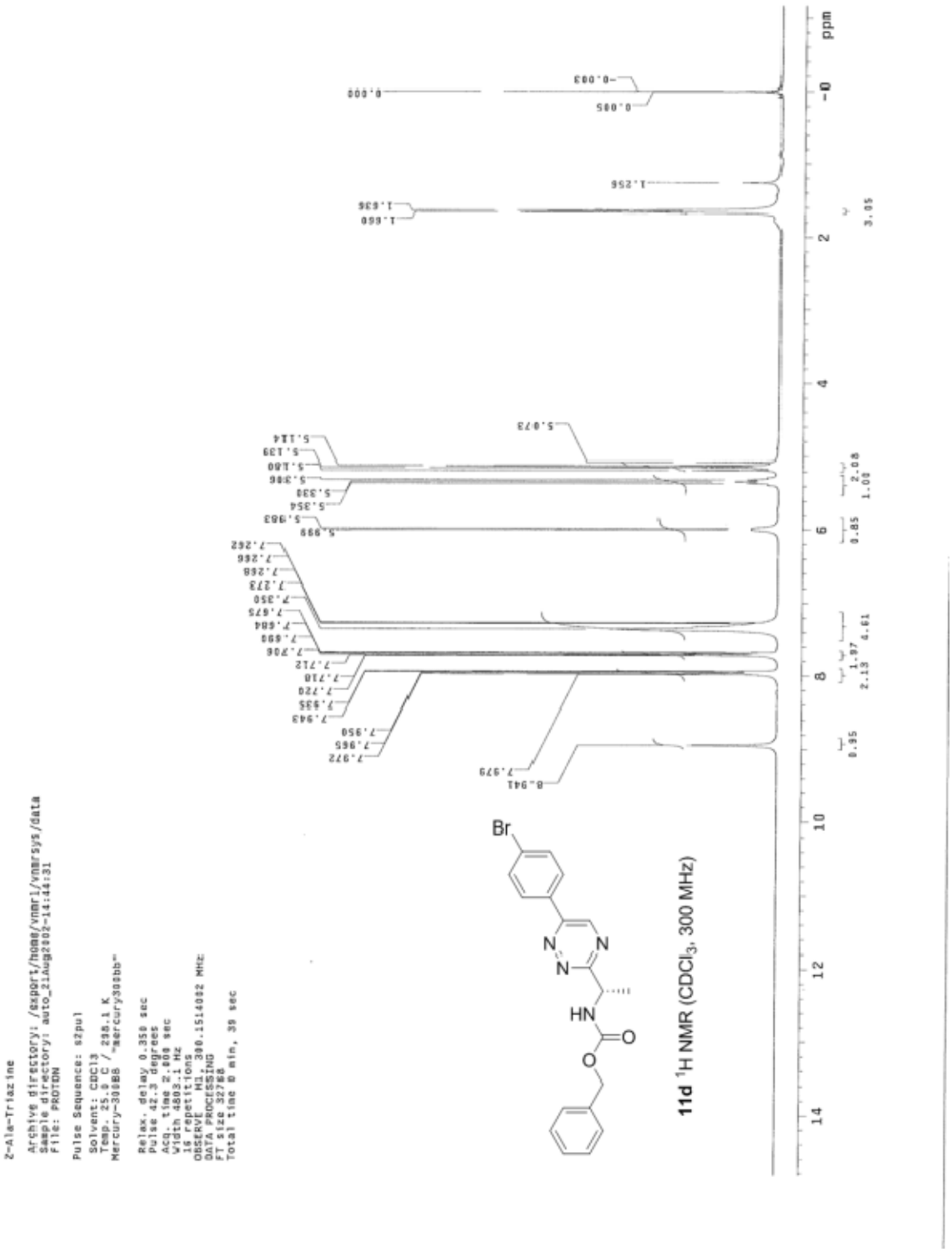

11b ^{1}H NMR ($CDCl_3$, 300 MHz)
Br
N
N
N
HN
O
O
ppm
20
40
60
80
100
120
140
160
180
200
41.474
56.544
67.150
76.809
77.230
77.651
126.260
127.123
128.267
128.450
128.688
129.601
131.938
132.086
136.065
146.215
155.193
155.930
166.887



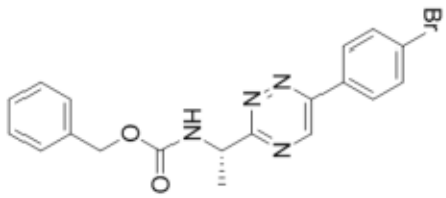
Z-Gly-(p-Brph)-1,2,4 Triazine

INDEX	FREQUENCY	PPM	HEIGHT
1	2565.642	8.950	40.9
2	2391.124	7.968	72.7
3	2302.699	7.840	68.3
4	2314.381	7.713	81.2
5	2305.956	7.685	55.6
6	2214.011	7.378	96.2
7	2202.655	7.340	51.2
8	2179.394	7.263	22.8
9	1708.168	5.959	16.7
10	1562.702	5.208	20.1
11	1557.398	5.190	34.8
12	1550.613	5.167	151.2
13	1469.658	4.898	62.8
14	1464.163	4.879	55.6
15	515.772	1.719	39.0

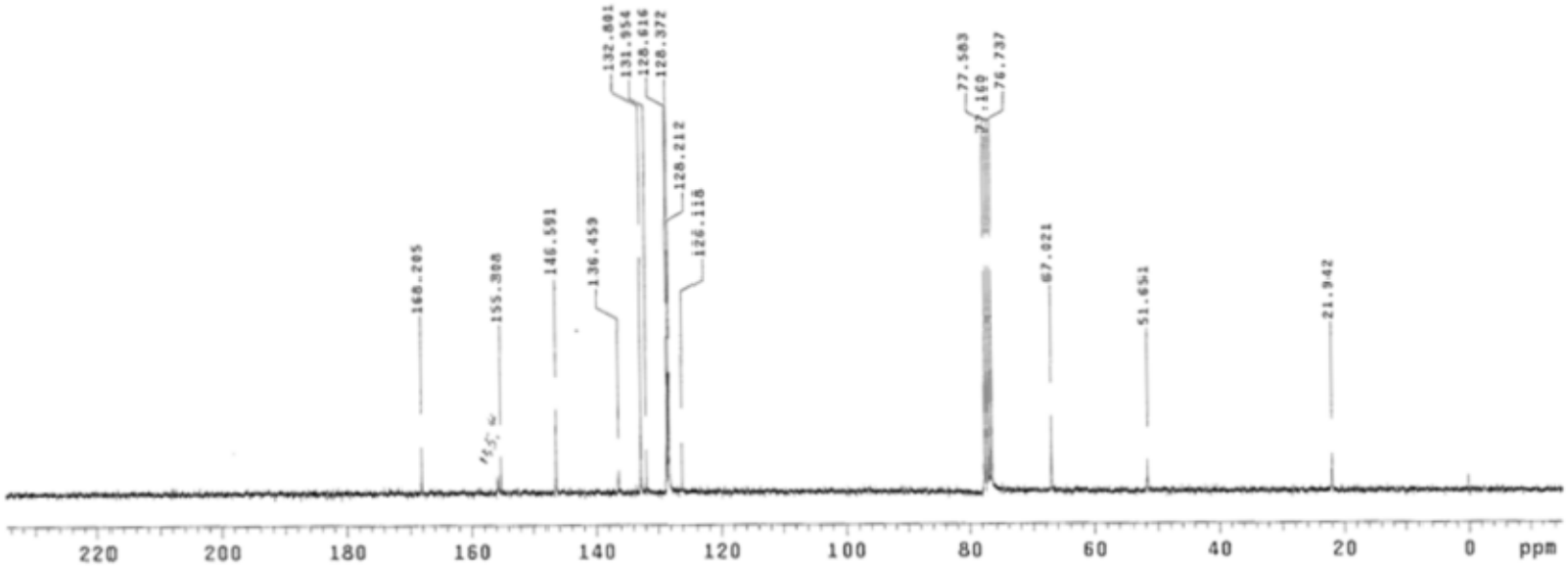

11c ^{1}H NMR ($CDCl_3$ 300 MHz)
Br
N
N
N
HN
O
O
ppm
200
180
160
140
120
100
80
60
40
20
164.409
156.667
155.488
146.636
136.466
132.865
131.924
128.695
128.428
128.309
126.324
77.651
77.230
76.809
67.305
45.433
-Gly-Triazine
ulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-300BB "gemini300"
PLSE SEQUENCE
Pulse 43.2 degrees
Acq. time 1.800 sec
Width 17354.0 Hz
8769 repetitions
OBSERVE C13, 75.4519834 MHz
COUPLE H1, 300.0603526 MHz
Power 36 dB
continuously on
ALTZ-16 modulated
DA PROCESSING
Line broadening 3.5 Hz
size 65536
tal time 78 hr, 10 min, 13 sec

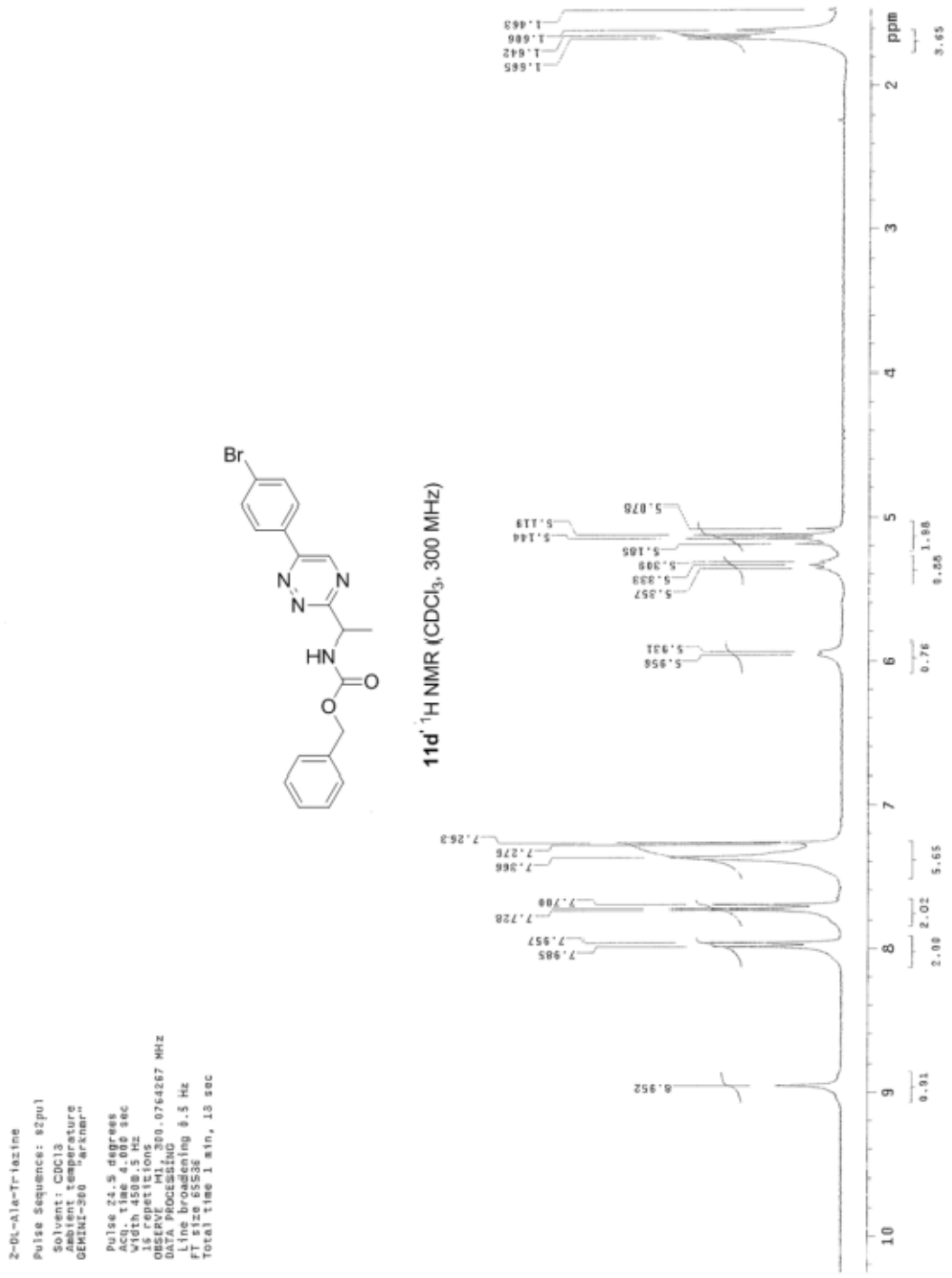

Z-Ala-Triazine
Archive directory: /export/home/vnmrl/vnmrsys/data
Sample directory: auto_21Aug2002-14:44:31
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Mercury-300BB "mercury300bb"
Relax. delay 0.350 sec
Pulse 42.3 degrees
Acq. time 2.800 sec
Width 4808.1 Hz
16 repetitions
OBSERVE H1, 300.1514002 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 39 sec
11d ^{1}H NMR ($CDCl_3$, 300 MHz)
Br
N
N
N
HN
O
O
ppm

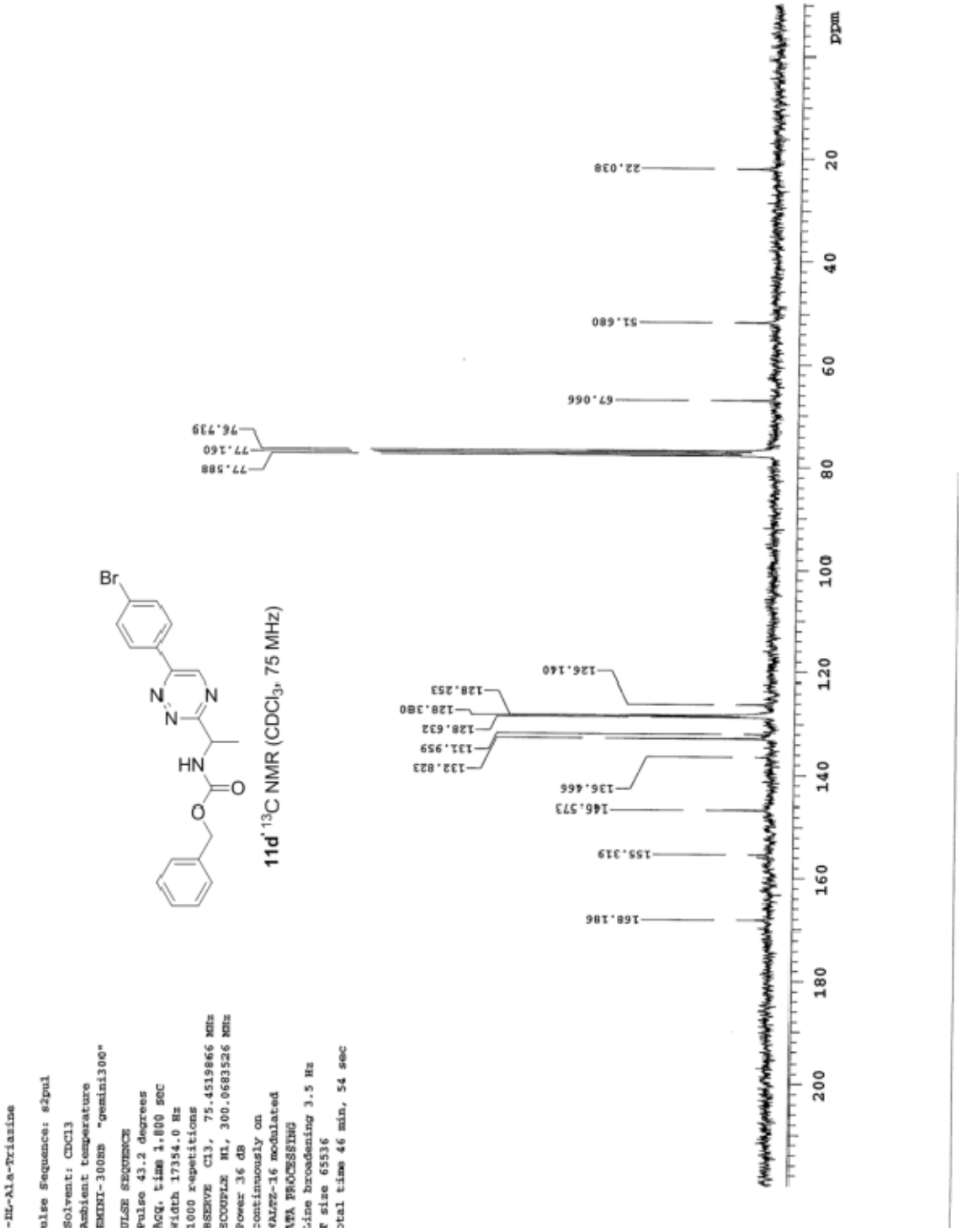
```
Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory: auto_12Nov2003
File: CARBON

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
Mercury-300BB "mercury300bb"

Relax. delay 1.000 sec
Pulse 35.2 degrees
Acq. time 1.815 sec
Width 18867.9 Hz
868 repetitions
OBSERVE C13, 75.4731210 MHz
DECOUPLE H1, 300.1528521 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 10 hr, 51 min, 48 sec
```



11d ^{1}H NMR ($CDCl_3$, 300 MHz)




11d' ^{1}H NMR (CDCl$_3$, 300 MHz)
Br
N
N
HN
O
O
ppm
1.463
1.686
1.642
1.665
5.078
5.119
5.144
5.185
5.309
5.333
5.357
5.931
5.956
7.263
7.276
7.366
7.708
7.728
7.957
7.985
8.952
3.65
1.88
0.88
0.76
5.65
2.02
2.08
0.91
Z-DL-Ala-Triazine
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300 "Varian"
Pulse 24.5 degree
Acq. time 4.000 sec
Width 4500.5 Hz
16 repetitions
OBSERVE H1, 300.0764267 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 min, 18 sec

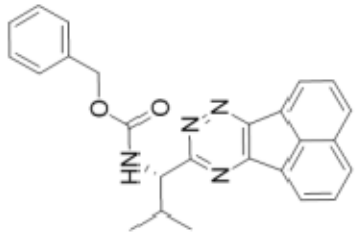

11d ^{13}C NMR (CDCl$_3$, 75 MHz)
Br
N
N
HN
O
O
22.038
51.680
67.066
76.739
77.160
77.588
128.140
128.253
128.380
128.632
131.959
132.823
136.466
146.573
155.319
168.186
ppm
20
40
60
80
100
120
140
160
180
200

1937-38

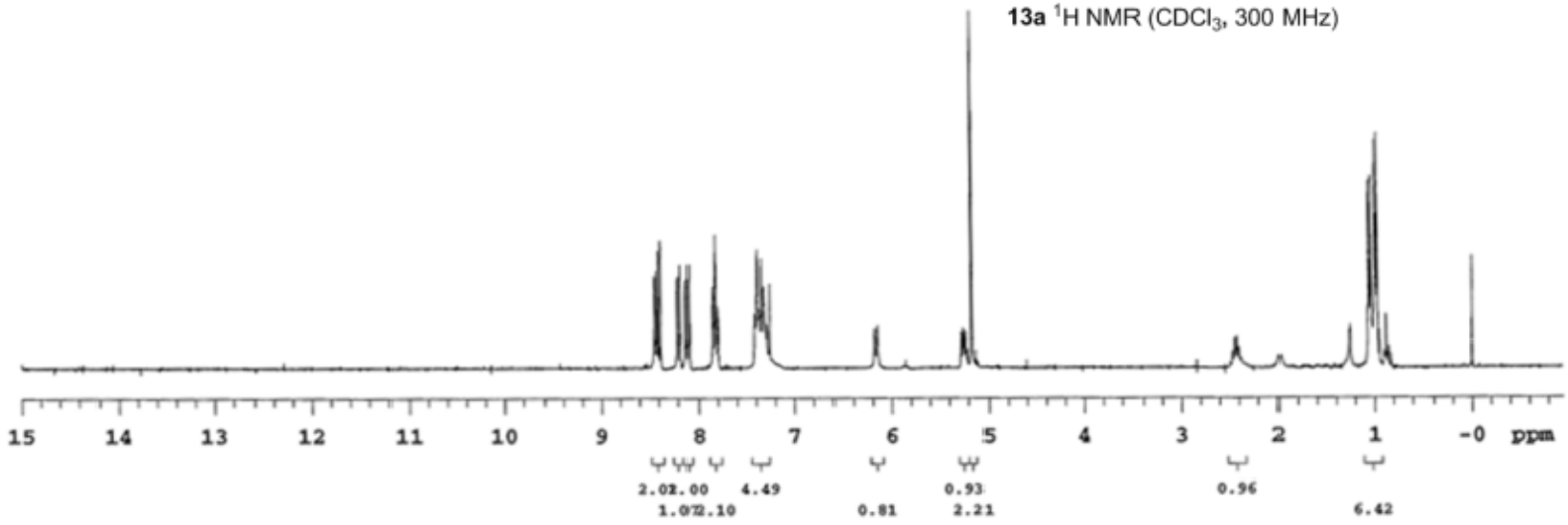
Pulse Sequence: s2pul

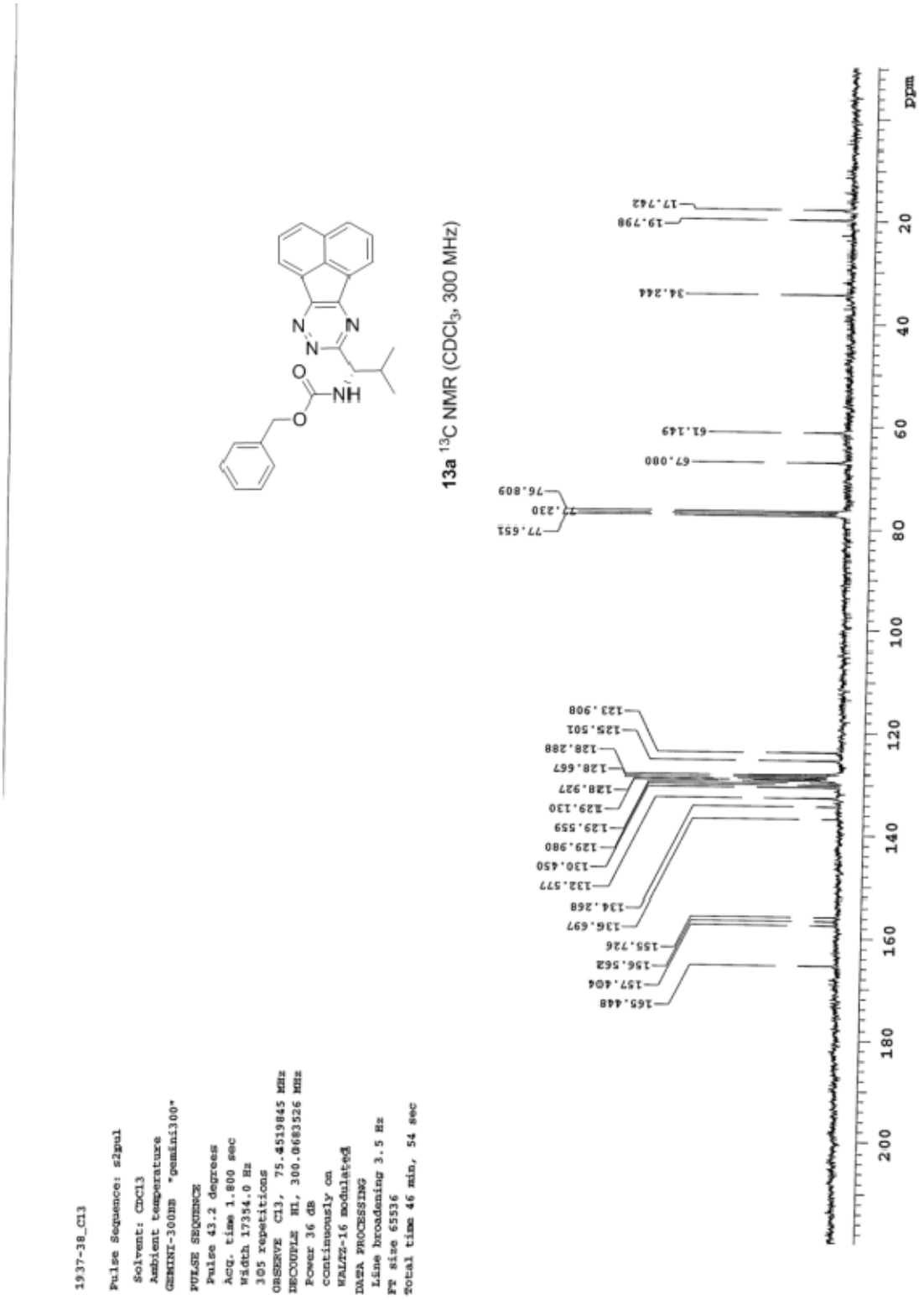
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "gemini300"

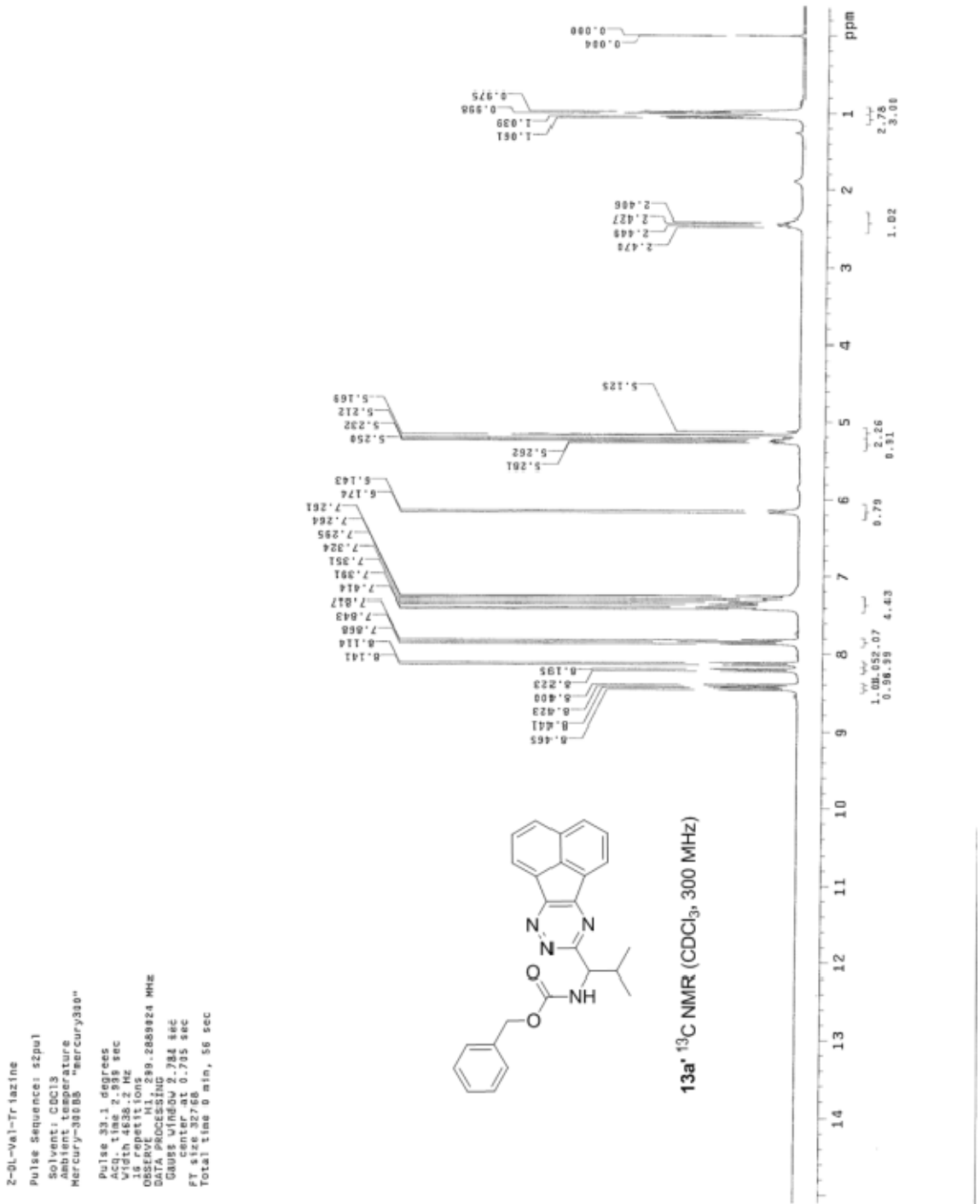
PULSE SEQUENCE
Pulse 34.7 degrees
Acq. time 3.500 sec
Width 4785.8 Hz
16 repetitions
OBSERVE H1, 300.0673569 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 5 sec

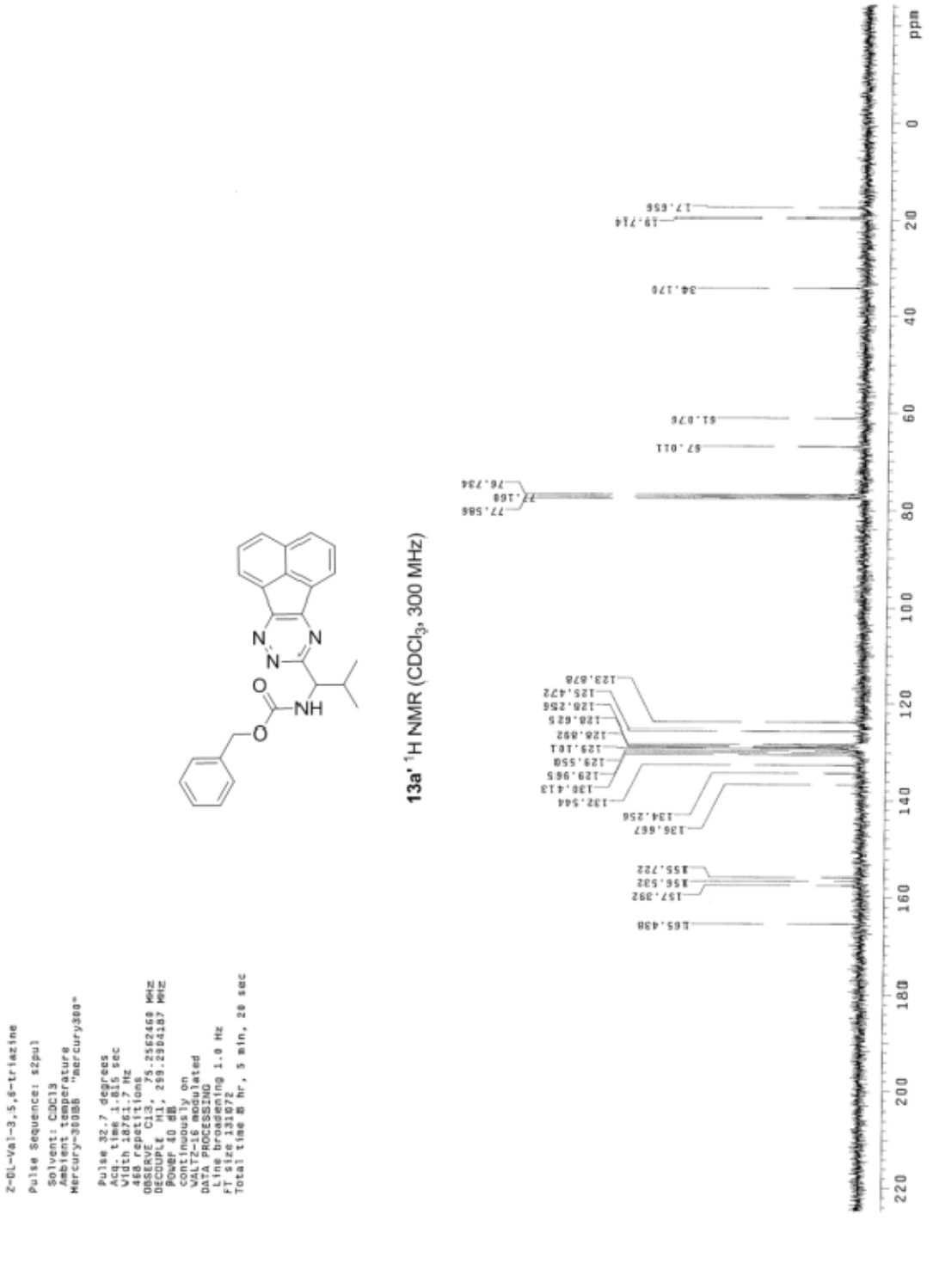


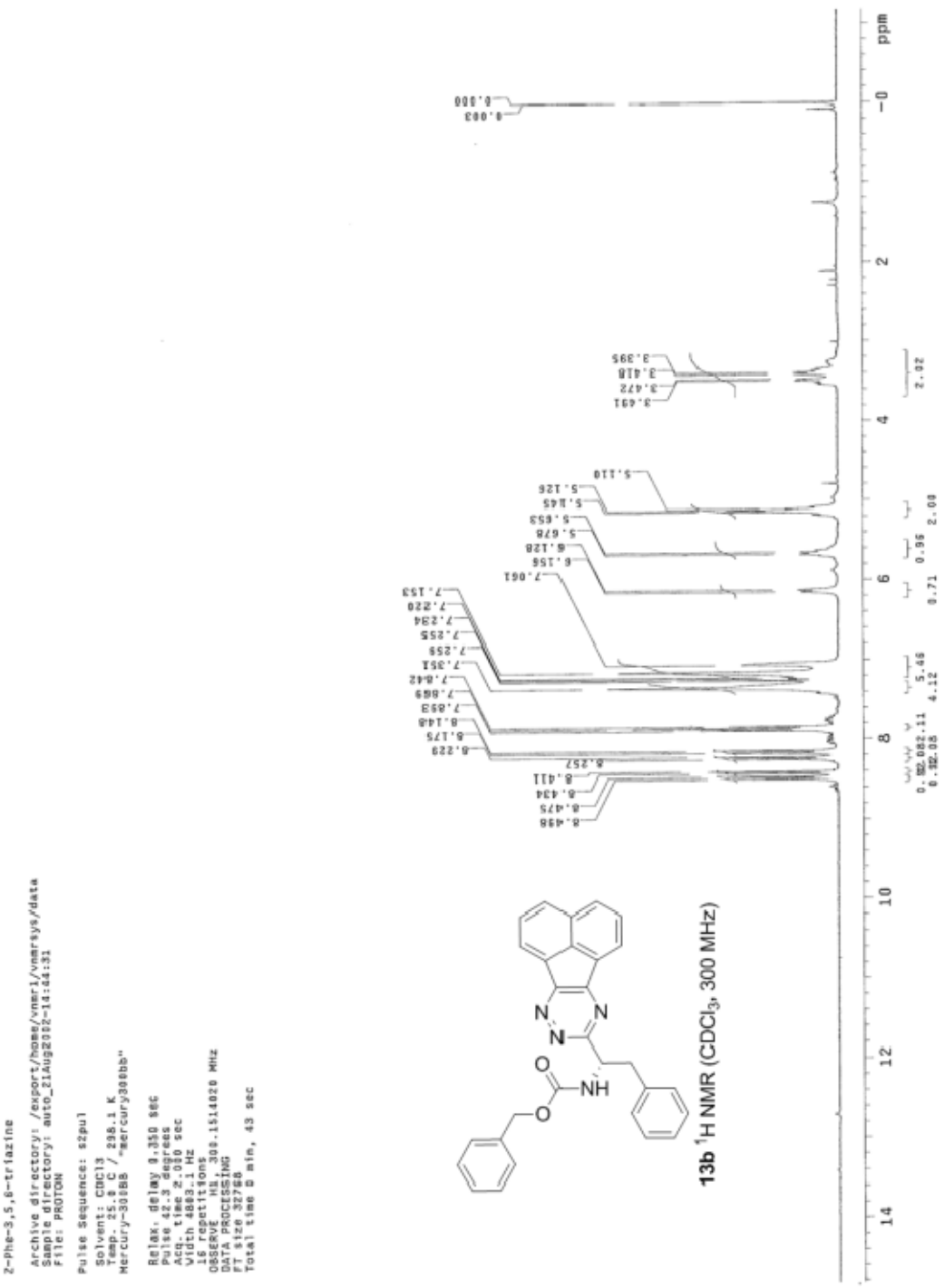
13a ^{1}H NMR ($CDCl_3$, 300 MHz)

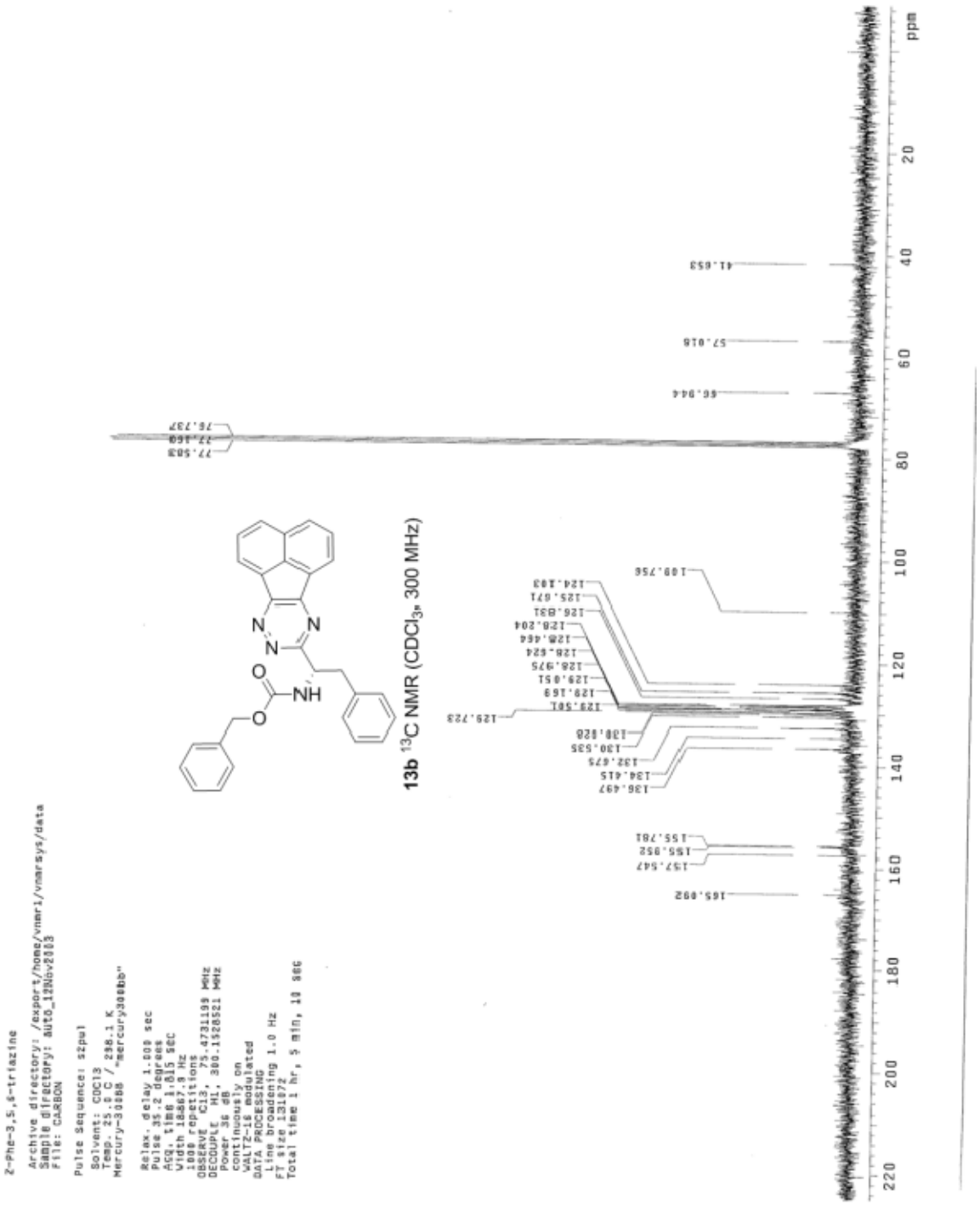



13a ^{13}C NMR (CDCl$_3$, 300 MHz)
ppm
17.742
19.798
34.344
61.149
67.080
76.809
77.230
77.651
123.908
125.501
128.288
128.667
128.927
129.130
129.559
129.980
130.450
132.577
134.268
136.697
155.726
156.562
157.404
165.448
1937-38_C13
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300BB "gemini300"
PULSE SEQUENCE
Pulse 43.2 degrees
Acq. time 1.800 sec
Width 17354.0 Hz
305 repetitions
OBSERVE C13, 75.4519845 MHz
DECOUPLE H1, 300.0683526 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 3.5 Hz
FT size 65536
Total time 46 min, 54 sec


2-DL-Val-Triazine
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Pulse 33.1 degrees
Acq. time 2.995 sec
Width 4638.2 Hz
16 repetitions
OBSERVE H1, 299.2889824 MHz
DATA PROCESSING
Gauss Window 2.784 sec
center at 0.735 sec
FT size 32768
Total time 0 min, 56 sec
13a' ^{13}C NMR ($CDCl_3$, 300 MHz)
ppm


13a' ^{1}H NMR ($CDCl_3$, 300 MHz)
N
N
N
O
NH
O
ppm
0
20
40
60
80
100
120
140
160
180
200
220
17.656
19.714
34.170
61.076
67.011
76.734
77.168
77.586
123.878
125.472
126.256
126.625
126.892
129.161
129.550
129.965
130.413
132.544
134.256
136.667
155.722
156.522
157.392
165.438
Z-DL-Val-3,5,6-triazine
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Pulse 32.7 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
468 repetitions
OBSERVE C13, 75.2562468 MHz
DECOUPLE H1, 299.2304187 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 8 hr, 5 min, 26 sec


2-Phe-3,5,6-triazine
Archive directory: /export/home/vnmrj/vnmrsys/data
Sample directory: auto_21Aug2012-14:44:31
File: PROTON
Pulse Sequence: s2pul
Solvent: CDCl3
Temp: 25.0 C / 298.1 K
Mercury-300BB "mercury300bb"
Relax. delay 8.350 sec
Pulse 42.3 degrees
Acq. time 2.010 sec
Width 4803.1 Hz
16 repetitions
OBSERVE H1, 300.1514029 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 43 sec
13b ^{1}H NMR ($CDCl_3$, 300 MHz)
ppm


13b ^{13}C NMR ($CDCl_3$, 300 MHz)
N
N
N
O
NH
O
ppm