

Electronic Supplementary Information

DBU-Catalyzed Transprotection of *N*-Fmoc-Cysteine

Di- and Tripeptides into *S*-Fm-Cysteine Di- and Tripeptides

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***N*-Fmoc-L-Met-L-Cys-OH (3b)**^[1] White microcrystals; yield: 88%; 97.0–99.0 °C. $[\alpha]_{\text{D}}^{23}$ -37.0 (*c* 1.0 in MeOH). ¹H NMR (DMSO-*d*₆) δ 8.22 (d, *J* = 7.5 Hz, 1H), 7.90 (d, *J* = 4.5 Hz, 2H), 7.74 (t, *J* = 5.7 Hz, 2H), 7.65 (d, *J* = 8.1 Hz, 1H), 7.43 (t, *J* = 7.2 Hz, 2H), 7.33 (t, *J* = 6.6 Hz, 2H), 4.44–4.42 (m, 1H), 4.35–4.18 (m, 4H), 2.90–2.79 (m, 2H), 2.47–2.42 (m, 1H), 2.05–1.95 (m, 3H), 1.92–1.85 (m, 2H), 1.24 (br s, 1H), 0.84 (m, 1H). ¹³C NMR (DMSO-*d*₆) δ 173.9, 171.6, 155.7, 143.8, 143.7, 140.6, 138.1, 129.2, 128.0, 127.6, 127.0, 126.2, 125.3, 125.2, 120.0, 65.6, 55.9, 50.3, 46.5, 37.4, 24.2, 22.8, 21.3. Anal. Calcd for C₂₃H₂₆N₂O₅S₂: C, 58.21; H, 5.52; N, 5.90. Found: C, 58.48; H, 5.53; N, 5.64.

***N*-Fmoc-L-Ala-L-Cys-OH (3c)**. White microcrystals (84 %), mp 167.0–169.0 °C. $[\alpha]_{\text{D}}^{23}$ -72.0 (*c* 1.0 in MeOH). ¹H NMR (DMSO-*d*₆) δ 8.16 (d, *J* = 7.4 Hz, 1H), 7.89 (d, *J* = 7.3 Hz, 2H), 7.75–7.70 (m, 2H), 7.61 (d, *J* = 7.3 Hz, 1H), 7.42 (t, *J* = 7.1 Hz, 2H), 7.34 (d, *J* = 7.1 Hz, 2H), 4.44–4.40 (m, 1H), 4.25–4.20 (m, 3H), 4.14 (t, *J* = 6.7 Hz, 1H), 2.86–2.75 (m, 2H), 2.45–2.40 (m, 1H), 1.24 (d, *J* = 6.6 Hz, 3H), 1.09 (t, *J* = 6.9 Hz, 1H). ¹³C NMR (DMSO-*d*₆) δ 172.7, 171.8, 155.6, 143.9, 143.7, 140.7, 135.3, 129.5, 127.6, 127.1, 125.3, 123.9, 121.2, 120.1, 65.7, 51.5, 49.9, 46.6, 18.3. Anal. Calcd for C₂₁H₂₂N₂O₅S: C, 60.85; H, 5.35; N, 6.76. Found: C, 60.59; H, 5.44; N, 6.91.

***N*-Fmoc-L-Leu-L-Cys-OH (3d)**. White microcrystals (86 %), mp 68.0–70.0 °C. $[\alpha]_{\text{D}}^{23}$ -23.0 (*c* 1.0 in MeOH). ¹H NMR (CDCl₃) δ 7.74 (d, *J* = 7.1 Hz, 2H), 7.56 (d, *J* = 7.0 Hz, 2H), 7.38 (t, *J* = 7.3 Hz, 2H), 7.29 (t, *J* = 7.8 Hz, 2H), 5.75 (d, *J* = 8.5 Hz, 1H), 4.82 (s, 1H), 4.36 (d, *J* = 5.4 Hz, 2H), 4.20 (d, *J* = 6.6 Hz, 1H), 3.49 (q, *J* = 7.0 Hz, 1H), 3.10–2.95 (m, 1H), 1.64–1.55 (m, 2H), 1.51 (t, *J* = 9.1 Hz, 1H), 1.21 (t, *J* = 7.0 Hz, 1H), 1.00–0.85 (m, 6H). ¹³C NMR (CDCl₃) δ 172.6, 156.7, 143.7, 141.5, 128.0, 127.3, 125.2, 120.2, 67.6, 54.1, 53.7, 47.3, 41.4, 26.5, 24.9, 23.0, 22.3. Anal. Calcd for C₂₄H₂₈N₂O₅S: C, 63.14; H, 6.18; N, 6.14. Found: C, 63.59; H, 6.48; N, 5.32.

***N*-Fmoc-L-Gly-L-Cys-OH (3e)**^{II}. White microcrystals; yield: 84%; 90.0–91.0 °C. $[\alpha]_D^{23}$ -60.0 (*c* 1.0 in MeOH). ¹H NMR (DMSO-*d*₆) δ 8.18 (d, *J* = 7.5 Hz, 1H) 7.89 (d, *J* = 7.5 Hz, 2H), 7.72 (d, *J* = 7.5 Hz, 2H), 7.61–7.57 (m, 1H), 7.42 (t, *J* = 7.5 Hz, 2H), 7.33 (t, *J* = 7.5 Hz, 2H), 4.50–4.42 (m, 1H), 4.30–4.24 (m, 3H), 3.74–3.70 (m, 2H), 2.88–2.79 (m, 2H), 2.42 (t, *J* = 8.7 Hz, 1H). ¹³C NMR (DMSO-*d*₆) δ 171.4, 169.1, 156.5, 144.0, 140.7, 127.6, 127.0, 125.2, 120.1, 65.8, 54.2, 46.6, 43.2, 25.7. Anal. Calcd for C₂₀H₂₀N₂O₅S: C, 59.99; H, 5.03; N, 7.00. Found: C, 59.65; H, 4.94; N, 7.01.

***N*-Fmoc-L-Lys(N-Boc)-L-Cys-OH (3f)**. White microcrystals (78 %), mp 88.0–90.0 °C. $[\alpha]_D^{23}$ +8.0 (*c* 1.0 in MeOH). ¹H NMR (CDCl₃) δ 7.75 (d, *J* = 7.4 Hz, 2H), 7.65–7.52 (m, 2H), 7.38 (t, *J* = 7.0 Hz, 2H), 7.34–7.24 (m, 4H), 5.95 (br s, 1H), 4.85–4.78 (m, 1H), 4.45–4.25 (m, 3H), 4.20 (d, *J* = 5.6 Hz, 1H), 3.75 (s, 1H), 3.20–2.95 (m, 4H), 1.45–1.25 (m, 15H). ¹³C NMR (CDCl₃) δ 172.6, 156.7, 144.0, 143.8, 141.4, 127.9, 127.2, 125.3, 120.1, 79.8, 67.4, 55.0, 54.2, 47.2, 40.3, 34.8, 32.3, 31.8, 29.7, 29.2, 28.6, 26.6, 25.5, 22.8, 20.9, 14.3, 11.6. Anal. Calcd for C₂₉H₃₇N₃O₇S: C, 60.93; H, 6.52; N, 7.35. Found: C, 60.64; H, 6.73; N, 7.49.

***N*-Fmoc-Gly-L-Leu-L-Cys-OH (7a)**^{II} White microcrystals; yield: 88%; 170.0–172.0 °C. ¹H NMR (DMSO-*d*₆) δ 8.26 (d, *J* = 7.8 Hz, 1H), 8.15 (d, *J* = 8.1 Hz, 1H), 7.90 (d, *J* = 7.5 Hz, 2H), 7.73 (d, *J* = 7.2 Hz, 2H), 7.57 (t, *J* = 5.7 Hz, 1H), 7.43 (t, *J* = 7.2 Hz, 2H), 7.34 (t, *J* = 7.2 Hz, 2H), 4.49–4.37 (m, 2H), 4.32–4.15 (m, 3H), 3.66 (d, *J* = 5.4 Hz, 2H), 2.92–2.72 (m, 2H), 2.48–2.46 (m, 1H), 1.70–1.63 (m, 1H), 1.63–1.31 (m, 2H), 0.89–0.85 (m, 6H). ¹³C NMR (DMSO-*d*₆) δ 172.1, 171.4, 168.8, 156.5, 143.8, 140.7, 127.6, 127.1, 125.2, 120.1, 65.7, 54.4, 50.8, 46.6, 43.3, 41.0, 25.3, 24.0, 23.0, 21.7. Anal. Calcd for C₂₆H₃₁N₃O₆S: C, 60.80; H, 6.08; N, 8.18. Found: C, 60.56; H, 6.21; N, 8.20.

N-Fmoc-Phe-L-Ala-L-Cys-OH (7b). White microcrystals 96%, mp 196.0–198.3 °C. $[\alpha]_D^{23}$ -61.0 (*c* 1.0 in MeOH). ^1H NMR (DMSO- d_6) δ 8.28 (d, *J* = 7.3 Hz, 1H), 8.16 (d, *J* = 7.8 Hz, 1H), 7.88 (d, *J* = 7.4 Hz, 2H), 7.70–7.56 (m, 3H), 7.45–7.15 (m, 10H), 4.50–4.35 (m, 2H), 4.32–4.25 (m, 1H), 4.14 (s, 3H), 3.10–3.00 (m, 1H), 2.90–2.70 (m, 3H), 2.44 (d, *J* = 7.8 Hz, 1H), 1.26 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (DMSO- d_6) δ 172.2, 171.4, 155.8, 143.7, 140.6, 138.2, 129.2, 128.0, 127.6, 127.0, 126., 2125.2, 120.0, 65.6, 56.0, 54.2, 48.2, 46.5, 37.4, 25.6, 18.1. Anal. Calcd for C₃₀H₃₁N₃O₆S: C, 64.15; H, 5.56; N, 7.48. Found: C, 63.86; H, 5.69; N, 7.53.

H-L-Met-L-Cys(S-Fm)-OH (4b). White microcrystals 76 %, mp 205.0–207.0 °C. $[\alpha]_D^{23}$ -8.0 (*c* 1.0 in MeOH). ^1H NMR (DMSO- d_6) δ 8.37 (br s, 1H), 7.85 (d, *J* = 7.5 Hz, 2H), 7.76–7.72 (m, 2H), 7.40–7.28 (m, 4H), 4.29 (br s, 3H), 4.18–4.10 (m, 1H), 3.56 (t, *J* = 6.3 Hz, 2H), 3.16–2.87 (m, 4H), 2.56–2.53 (m, 1H), 2.01 (br s, 3H), 1.90 (t, *J* = 6.3 Hz, 1H), 1.80–1.73 (m, 1H). ^{13}C NMR (DMSO- d_6) δ 172.6, 169.8, 146.3, 140.7, 127.7, 127.3, 125.3, 120.2, 54.1, 52.7, 46.7, 38.0, 36.1, 35.3, 32.4, 28.9, 14.8. Anal. Calcd for C₂₂H₂₆N₂O₃S \cdot 2H₂O: C, 58.90; H, 6.29; N, 6.24. Found: C, 58.63; H, 6.12; N, 5.96

H-L-Ala-L-Cys(S-Fm)-OH (4c). White microcrystals 70 %, mp 202.0–204.0 °C. $[\alpha]_D^{23}$ -104.0 (*c* 1.0 in MeOH). ^1H NMR (D₂O, D₂SO₄) δ 6.88 (br s, 2H), 6.78 (br s, 2H), 6.68–6.58 (m, 4H), 3.95–3.86 (m, 1H), 3.75–3.65 (m, 1H), 3.19–3.15 (m, 1H), 2.30–2.00 (m, 4H), 1.20–1.10 (m, 3H). ^{13}C NMR (D₂O, D₂SO₄) δ 172.9, 170.7, 145.5, 140.5, 127.3, 127.0, 124.7, 119.6, 52.6, 49.0, 46.1, 35.4, 32.8, 16.6. Anal. Calcd for C₂₀H₂₂N₂O₃S: C, 64.84; H, 5.99; N, 7.56. Found: C, 64.86; H, 6.07; N, 7.47.

H-L-Leu-L-Cys(S-Fm)-OH (4d). White microcrystals 87 %, mp 207.0–209.0 °C. $[\alpha]_D^{23}$ -32.0 (*c* 1.0 in MeOH). ^1H NMR (DMSO- d_6) δ 8.32–8.30 (m, 1H), 7.90–7.70 (m, 4H), 7.50–7.25 (m, 4H), 4.34–4.25 (m, 2H), 4.17–4.10 (m, 2H), 3.62–3.58 (m, 1H),

3.12–3.08 (m, 1H), 3.02 (dd, $J = 12.7, 6.0$ Hz, 1H), 2.95–2.80 (m, 1H), 1.69–1.65 (m, 1H), 1.55–1.50 (m, 1H), 1.45–1.35 (m, 1H), 0.95–0.82 (m, 6H). ^{13}C NMR (DMSO- d_6) δ 172.1, 170.7, 146.0, 140.4, 139.4, 128.9, 127.3, 126.9, 125.0, 121.4, 120.0, 109.7, 53.6, 51.7, 46.4, 41.5, 35.9, 34.9, 23.7, 22.9, 21.9. Anal. Calcd for $\text{C}_{23}\text{H}_{28}\text{N}_2\text{O}_3\text{S}\cdot\text{H}_2\text{O}$: C, 64.16; H, 7.02; N, 6.51. Found: C, 64.14; H, 7.05; N, 6.24.

H-Gly-Cys-(S-Fm)-OH (4e) White microcrystals 78 %, mp 230.0–232.0 °C. $[\alpha]_{\text{D}}^{23} -10.0$ (c 1.0 in MeOH). ^1H NMR (DMSO- d_6) δ 8.38 (m, $J = 5.7$ Hz, 1H), 7.84–7.73 (m, 4H), 7.39–7.27 (m, 4H), 4.30–4.00 (br s, 3H), 4.24 (br s, 1H), 4.12 (m, 1H), 3.54 (d, $J = 15$ Hz, 1H), 3.41 (d, $J = 14.9$ Hz, 1H), 3.15–3.00 (m, 2H), 2.95–2.80 (m, 2H). ^{13}C NMR (DMSO- d_6) δ 172.5, 166.8, 146.0, 140.3, 128.8, 127.2, 126.8, 124.9, 121.2, 119.9, 119.7, 53.9, 46.2, 40.9, 35.7, 35.2. Anal. Calcd for $\text{C}_{19}\text{H}_{20}\text{N}_2\text{O}_3\text{S}\cdot 2\text{H}_2\text{O}$: C, 58.15; H, 6.16; N, 7.14. Found: C, 58.00; H, 5.67; N, 6.97.

H-L-Lys(N-Boc)-L-Cys(S-Fm)-OH (4f). White microcrystals (69 %), mp 165.0–167.0 °C. $[\alpha]_{\text{D}}^{23} +96.0$ (c 1.0 in MeOH). ^1H NMR (DMSO- d_6) δ 8.40 (br s, 1H), 7.85–7.70 (m, 4H), 7.50–7.30 (m, 4H), 6.77 (s, 1H), 5.50–4.50 (m, 2H), 4.25–4.12 (m, 2H), 3.55–3.50 (m, 1H), 3.20–2.80 (m, 5H), 1.80–1.20 (m, 16H). ^{13}C NMR (DMSO- d_6) δ 171.6, 168.9, 155.7, 146.0, 140.8, 127.7, 127.3, 125.2, 121.7, 120.3, 77.8, 52.2, 46.7, 36.1, 33.9, 31.0, 29.4, 28.6, 21.5. Anal. Calcd for $\text{C}_{28}\text{H}_{37}\text{N}_3\text{O}_5\text{S}\cdot\text{H}_2\text{O}$: C, 61.63; H, 6.83; N, 7.70. Found: C, 61.59; H, 7.12; N, 7.37.

H-Gly-L-Leu-L-Cys(S-Fm)-OH (8a) White microcrystals 80 %, mp 200.0–202.0 °C. $[\alpha]_{\text{D}}^{23} -34.0$ (c 1.0 in MeOH). ^1H NMR (DMSO- d_6) δ 8.74 (d, $J = 7.2$ Hz, 1H), 7.95 (d, $J = 6.0$ Hz, 1H), 7.84 (d, $J = 7.5$ Hz, 2H), 7.36 (dd, $J = 7.2, 3.6$ Hz, 2H), 7.37 (t, $J = 7.2$ Hz, 2H), 7.29 (t, $J = 7.2$ Hz, 2H), 4.31–4.25 (m, 1H), 4.12–4.09 (m, 2H), 3.54 (q, $J = 16.5$ Hz, 2H), 3.13–2.86 (m, 4H), 1.62–1.55 (m, 1H), 1.50–1.46 (m, 2H), 0.84 (t, $J = 6.9$ Hz, 6H), ^{13}C NMR (DMSO- d_6) δ 172.3, 171.2, 167.5, 146.1, 140.3,

127.3, 126.9, 125.0, 124.9, 119.8, 53.5, 51.8, 46.5, 41.0, 36.0, 34.7, 24.2, 23.1, 21.4.

Anal. Calcd for $C_{25}H_{31}N_3O_4S \cdot 3H_2O$: C, 57.34; H, 7.12; N, 8.02. Found: C, 56.89; H, 6.68; N, 7.82.

H-Phe-L-Ala-L-Cys(S-Fm)-OH (8b). White microcrystals 78 %, mp 218.0–220.0 °C. $[\alpha]_D^{23}$ -12.8 (*c* 1.0 in MeOH). 1H NMR (DMSO- d_6) δ 8.20 (d, *J* = 6.3 Hz, 1H), 7.95–7.80 (m, 4H), 7.73 (t, *J* = 7.2 Hz, 1H), 7.45–7.32 (m, 4H), 7.30–7.20 (m, 5H), 6.30–6.25 (m, 1H), 4.40–4.28 (m, 2H), 4.20–4.10 (m, 1H), 3.12–2.98 (m, 4H), 2.88–2.76 (m, 2H), 2.75–2.70 (m, 1H), 1.30–1.20 (m, 3H). ^{13}C NMR (DMSO- d_6) δ 171.8, 167.5, 145.9, 140.6, 134.9, 129.6, 128.6, 127.4, 127.1, 125.0, 121.5, 120.0, 53.2, 52.3, 48.2, 46.5, 37.0, 35.8, 33.9, 18.7. Anal. Calcd for $C_{29}H_{31}N_3O_4S \cdot H_2O$: C, 65.02; H, 6.21; N, 7.84. Found: C, 65.58; H, 6.27; N, 7.53.

Cbz-Ala-L-Gly-L-Cys(S-Fm)-OH (9b) White microcrystals 70 %, mp 155.0–157.0 °C. $[\alpha]_D^{23}$ -19.0 (*c* 1.0 in MeOH). 1H NMR (DMSO- d_6) δ 12.88 (br s, 1H), 8.21–8.13 (m, 2H), 7.86 (d, *J* = 7.5 Hz, 2H), 7.72 (t, *J* = 7.8 Hz, 2H), 7.52 (d, *J* = 6.3 Hz, 1H), 7.41–7.29 (m, 9H), 5.04 (dd, *J* = 18.0, 12.0 Hz, 2H), 4.49–4.45 (m, 1H), 4.16 (t, *J* = 6.0 Hz, 1H), 4.07 (t, *J* = 6.6 Hz, 1H), 3.76 (d, *J* = 6.0 Hz, 2H), 2.94 (dd, *J* = 13.8, 4.8 Hz, 1H), 2.79 (dd, *J* = 13.8, 7.8 Hz, 1H), 1.22 (m, 3H). ^{13}C NMR (DMSO- d_6) δ 172.6, 171.7, 168.6, 155.6, 145.7, 140.4, 136.8, 128.2, 127.6, 127.2, 126.8, 124.8, 119.8, 65.3, 61.2, 52.0, 50.0, 46.2, 41.6, 35.6, 33.8, 17.9. Anal. Calcd for $C_{30}H_{31}N_3O_6S$: C, 64.15; H, 5.56; N, 7.48. Found: C, 63.72; H, 5.95; N, 7.18.

Cbz-Gly-L-Ala-L-Cys(S-Fm)-OH (9c). White microcrystals 60 %, mp 168.0–170.0 °C. $[\alpha]_D^{23}$ +21.0 (*c* 1.0 in MeOH). 1H NMR (DMSO- d_6) δ 13.00 (s, 1H), 8.34 (d, *J* = 7.5 Hz, 1H), 8.03 (d, *J* = 7.6 Hz, 1H), 7.85 (d, *J* = 7.0 Hz, 2H), 7.73 (t, *J* = 6.4 Hz, 2H), 7.50–7.44 (m, 1H), 7.42–7.25 (m, 9H), 5.03 (s, 2H), 4.45–4.35 (m, 2H), 4.20–4.10 (m, 1H), 3.70–3.60 (m, 1H), 3.45 (br s, 1H), 3.22–3.15 (m, 2H), 2.95 (dd, *J* = 12.0, 9.0

Hz, 1H), 2.85–2.80 (m, 1H), 1.38 (d, $J = 6.4$ Hz, 1H), 1.22 (d, $J = 6.4$ Hz, 2H). ^{13}C NMR (DMSO- d_6) δ 172.2, 171.8, 168.5, 156.4, 145.8, 140.5, 137.0, 128.3, 127.6, 127.3, 126.9, 124.9, 119.9, 65.4, 52.3, 47.8, 46.4, 43.4, 35.7, 33.7, 18.6. Anal. Calcd for $\text{C}_{30}\text{H}_{31}\text{N}_3\text{O}_6\text{S}\cdot\text{H}_2\text{O}$: C, 62.16; H, 5.74; N, 7.25. Found: C, 62.38; H, 5.45; N, 7.86.

Cbz-L-Leu-L-Met-L-Cys-OH (9d). White microcrystals 65 %, mp 131.0–133.0 °C. $[\alpha]_{\text{D}}^{23}$ -18.0 (c 1.0 in MeOH). ^1H NMR (DMSO- d_6) δ 8.25 (d, $J = 7.8$ Hz, 1H), 8.02 (d, $J = 7.8$ Hz, 1H), 7.85 (d, $J = 7.2$ Hz, 2H), 7.72 (d, $J = 7.5$ Hz, 2H), 7.48 (d, $J = 8.4$ Hz, 1H), 7.41–7.28 (m, 9H), 5.02 (s, 2H), 4.44–4.42 (m, 2H), 4.15 (t, $J = 5.7$ Hz, 1H), 4.10–4.00 (m, 1H), 3.15 (d, $J = 6$ Hz, 2H), 2.94 (dd, $J = 12.0, 9.0$ Hz, 1H), 2.80 (dd, $J = 12.0, 6.9$ Hz, 1H), 2.45 (br s, 2H), 2.02 (m, 3H), 1.99–1.87 (m, 1H), 1.84–1.79 (m, 1H), 1.65–1.57 (m, 1H), 1.47–1.40 (m, 2H), 1.26 (d, $J = 6.0$ Hz, 1H), 0.84 (m, $J = 4.2$ Hz, 6H). ^{13}C NMR (DMSO- d_6) δ 172.2, 171.8, 171.0, 155.9, 145.8, 140.5, 137.0, 128.3, 127.6, 127.4, 126.9, 124.9, 119.9, 65.3, 53.1, 52.2, 51.6, 46.4, 35.6, 33.7, 32.3, 29.3, 24.2, 23.0, 21.4, 14.7. Anal. Calcd for $\text{C}_{36}\text{H}_{43}\text{N}_3\text{O}_6\text{S}_2$: C, 63.79; H, 6.39; N, 6.20. Found: C, 63.55; H, 6.57; N, 6.19.

Cbz-L-Leu-Met-Gly-Cys(S-Fm)-OH (10a). White microcrystals 75 %, mp 80.0–82.0 °C. $[\alpha]_{\text{D}}^{23}$ -70.0 (c 1.0 in MeOH). ^1H NMR (DMSO- d_6) δ 12.85 (br s, 1H), 8.25 (t, $J = 6.1$ Hz, 1H), 8.13 (t, $J = 3.0$ Hz, 1H), 7.90–7.81 (m, 2H), 7.72 (t, $J = 7.2$ Hz, 2H), 7.42–7.25 (m, 11 H), 5.01 (s, 2H), 4.52–4.45 (m, 1H), 4.40–4.30 (m, 1H), 4.28–4.24 (m, 1H), 4.20–4.10 (m, 1H), 4.12–4.00 (m, 1H), 3.85–3.60 (m, 1H), 3.14 (d, $J = 6.4$ Hz, 2H), 2.92 (dd, $J = 15.0, 9.0$ Hz, 1H), 2.88–2.75 (m, 1H), 1.65–1.55 (m, 1H), 1.53–1.40 (m, 2H), 1.30–1.20 (m, 4H), 0.90–0.80 (m, 9H). ^{13}C NMR (DMSO- d_6) δ 172.4, 171.9, 171.2, 168.6, 155.9, 145.8, 143.8, 140.5, 137.0, 128.3, 127.6, 127.4, 126.9, 125.2, 124.9, 119.9, 65.4, 62.8, 53.1, 52.2, 51.8, 46.4, 41.7, 35.7, 33.9, 32.1, 31.0, 29.4,

24.2, 23.1, 22.1, 21.4, 14.6, 14.0. Anal. Calcd for C₃₈H₄₆N₄O₇S₂: C, 62.10; H, 6.31.
Found: C, 62.39; H, 6.68.

Cbz-L-Ala-Phe-Ala-Cys(S-Fm)-OH (10b). White microcrystals 72%, mp 149.0–151.0 °C. $[\alpha]_D^{23}$ -103.0 (*c* 1.0 in MeOH). ¹H NMR (DMSO-*d*₆) δ 8.83 (d, *J* = 8.2 Hz, 1H), 8.30–8.20 (m, 2H), 8.13 (d, *J* = 7.7 Hz, 1H), 7.86 (t, *J* = 7.2 Hz, 3H), 7.75–7.70 (m, 2H), 7.45–7.28 (m, 10 H), 7.23–6.90 (m, 3H), 5.00 (s, 1H), 4.50–4.35 (m, 2H), 4.20–4.15 (m, 1H), 3.99 (t, *J* = 6.9 Hz, 1H), 3.70–3.45 (m, 2H), 3.18 (t, *J* = 7.2 Hz, 2H), 3.05–2.92 (m, 2H), 2.90–2.80 (m, 2H), 1.37 (d, *J* = 7.0 Hz, 1H), 1.23 (d, *J* = 7.0 Hz, 2H), 1.11 (d, *J* = 6.7 Hz, 2H), 0.96 (d, *J* = 6.6 Hz, 1H). ¹³C NMR (DMSO-*d*₆) δ 172.0, 171.7, 171.3, 170.3, 169.6, 155.4, 145.7, 140.4, 137.6, 136.8, 129.2, 128.2, 127.8, 127.6, 127.3, 126.8, 126.0, 124.8, 119.8, 65.3, 53.4, 52.5, 52.2, 50.2, 47.9, 46.3, 37.3, 35.6, 33.8, 18.3, 17.1. Anal. Calcd for C₄₀H₄₂N₄O₇S·2H₂O: C, 63.31; H, 6.11; N, 7.38. Found: C, 62.91; H, 5.88; N, 7.31.

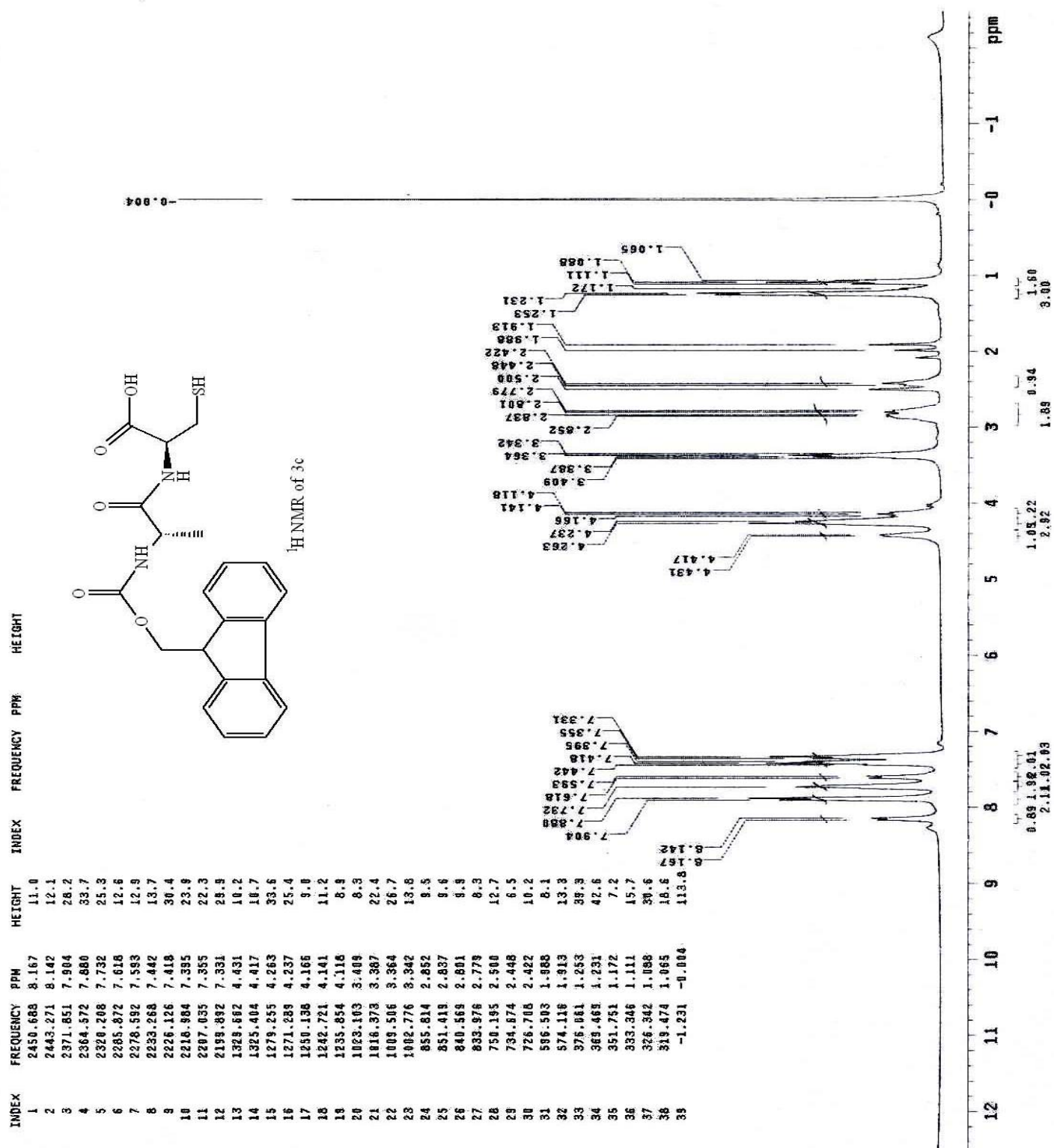
Cbz-L-Ala-Phe-Leu-Cys(S-Fm)-OH (10c). White microcrystals 79 %, mp 129.0–131.0 °C. $[\alpha]_D^{23}$ -118.0 (*c* 1.0 in MeOH). ¹H NMR (DMSO-*d*₆) δ 8.30–8.18 (m, 1H), 7.95–7.90 (m, 1H), 7.85–7.80 (m, 1H), 7.70–7.60 (m, 1H), 7.50–7.15 (m, 16H), 7.10–6.95 (m, 1H), 6.64–6.55 (m, 1H), 5.00 (s, 2H), 4.40 (br s, 1H), 4.30–4.10 (m, 1H), 4.08–3.90 (m, 1H), 3.64 (br s, 2H), 3.40–3.30 (m, 2H), 3.28–3.10 (m, 1H), 3.05–2.90 (m, 2H), 2.75–2.65 (m, 2H), 1.47 (br s, 2H), 1.30–1.10 (m, 3H), 0.84 (s, 5H). ¹³C NMR (DMSO-*d*₆) δ 173.2, 172.7, 171.7, 169.2, 155.6, 148.3, 147.8, 145.8, 141.0, 140.5, 139.7, 137.4, 137.0, 136.5, 131.0, 129.2, 129.0, 128.3, 127.9, 127.7, 127.3, 126.7, 126.4, 125.3, 124.7, 120.3, 120.0, 119.4, 113.9, 65.4, 55.4, 53.5, 49.9, 43.9, 42.8, 36.7, 36.5, 34.9, 23.4, 23.1, 22.8, 21.9, 18.1. Anal. Calcd for C₄₃H₄₈N₄O₇S: C, 67.52; H, 6.32. Found: C, 67.38; H, 6.05.

Fmoc-Gly-L-Leu-Gly-L-Leu-Cys(S-Fm)-OH (11) White microcrystals 69 %, mp 115.0–117.0 °C. $[\alpha]_D^{23}$ -23.0 (*c* 1.0 in MeOH). ^1H NMR (DMSO-*d*₆) δ 8.27 (d, *J* = 7.5 Hz, 1H), 8.18– 8.14 (m, 1H), 7.96 (t, *J* = 7.8 Hz, 1H), 7.85–7.78 (m, 5H), 7.67 (t, *J* = 6.9 Hz, 4H), 7.45 (t, *J* = 6 Hz, 1H), 7.38–7.23 (m, 8H), 4.37–4.31 (m, 2H), 4.27–4.18 (m, 4H), 4.11 (t, *J* = 6.6 Hz, 1H), 3.65 (d, *J* = 5.7 Hz, 2H), 3.60 (d, *J* = 6.0 Hz, 2H), 3.10 (d, *J* = 5.7 Hz, 2H), 2.88 (dd, *J* = 13.8, 5.4 Hz, 1H), 2.74 (dd, *J* = 13.8, 8.1 Hz, 1H), 1.56–1.45 (m, 2H), 1.42–1.39 (m, 4H), 1.19 (br s, 1H), 0.81–0.79 (m, 13 H). ^{13}C NMR (DMSO-*d*₆) δ 172.2, 172.0, 171.8, 168.9, 168.3, 156.4, 145.8, 143.8, 140.7, 140.5, 127.6, 127.3, 127.0, 126.9, 125.2, 124.9, 120.1, 119.9, 65.7, 52.3, 51.0, 50.6, 46.6, 46.4, 35.7, 33.6, 24.1, 23.11, 21.58. Anal. Calcd for C₄₈H₅₅N₅O₈S.H₂O: C, 65.51; H, 6.53; N, 7.96. Found: C, 65.43; H, 6.89; N, 7.68.

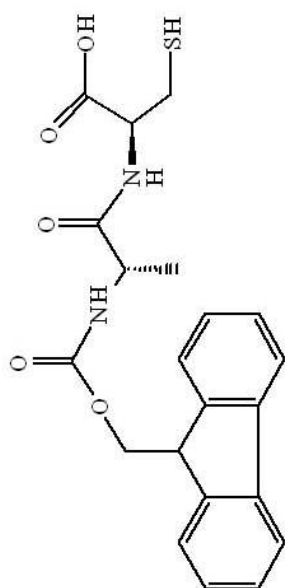
References

1. Katritzky, A. R.; Abo-Dya, Nader E.; Srinivasa R. Tala and Abdel-Samii, Zakaria K. *Org. Biomol. Chem.* **2010**, *8*, 2316-2319.

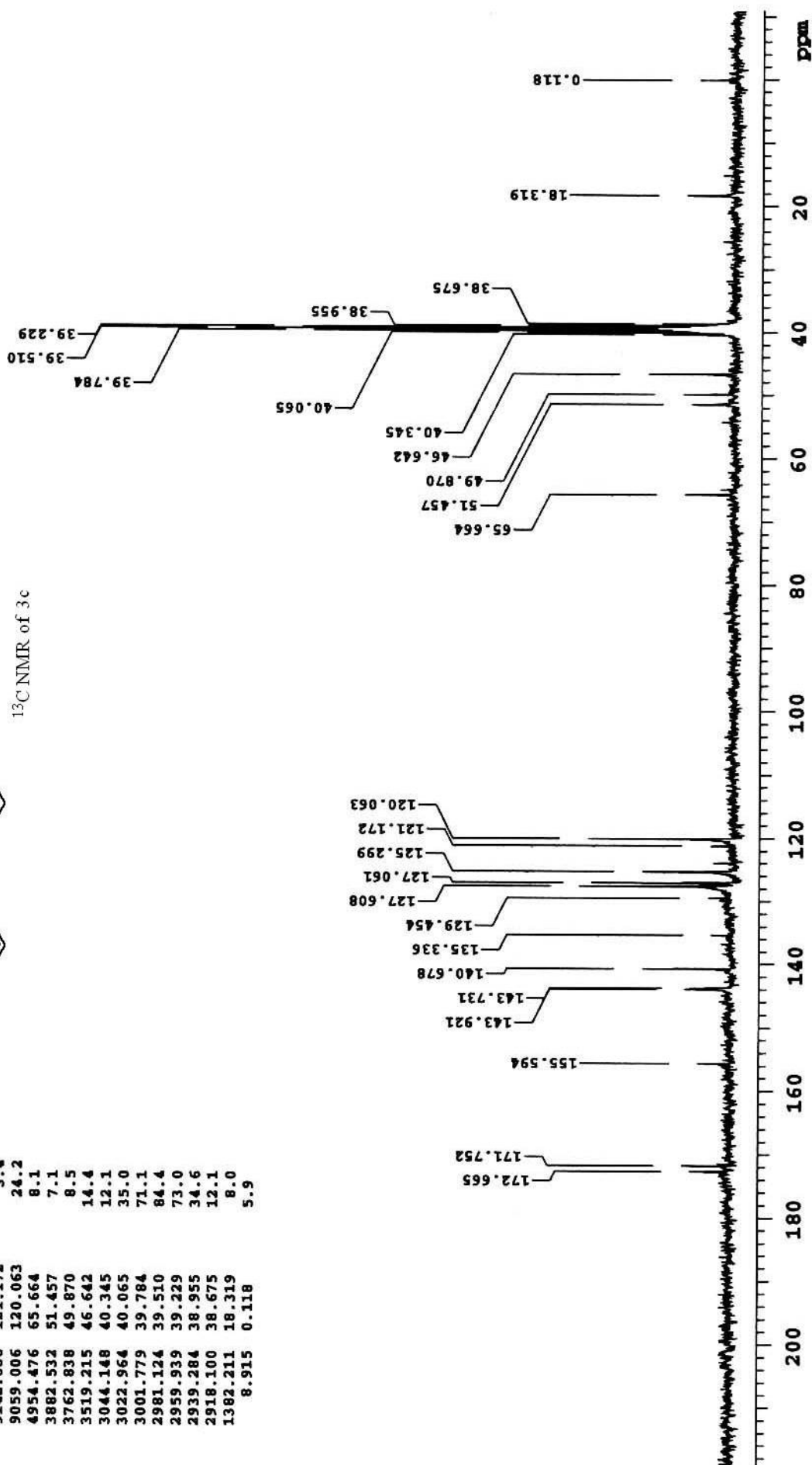
¹H, ¹³C NMR Spectra



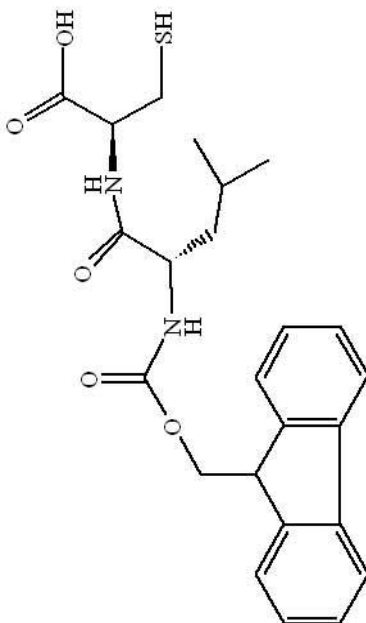
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| 1 | 13027.954 | 172.665 |
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| 3 | 11739.926 | 155.594 |
| 4 | 10859.174 | 143.921 |
| 5 | 10844.874 | 143.731 |
| 6 | 10614.491 | 140.678 |
| 7 | 10211.452 | 135.336 |
| 8 | 9767.633 | 129.454 |
| 9 | 9628.344 | 127.608 |
| 10 | 9587.034 | 127.061 |
| 11 | 9456.100 | 125.299 |
| 12 | 9142.686 | 121.172 |
| 13 | 9059.006 | 120.063 |
| 14 | 4954.476 | 65.664 |
| 15 | 3882.532 | 51.457 |
| 16 | 3762.838 | 49.870 |
| 17 | 3519.215 | 46.642 |
| 18 | 3044.148 | 40.345 |
| 19 | 3022.964 | 40.065 |
| 20 | 3001.779 | 39.784 |
| 21 | 2981.124 | 39.510 |
| 22 | 2959.939 | 39.229 |
| 23 | 2939.284 | 38.955 |
| 24 | 2918.100 | 38.675 |
| 25 | 1382.211 | 18.319 |
| 26 | 8.915 | 0.118 |



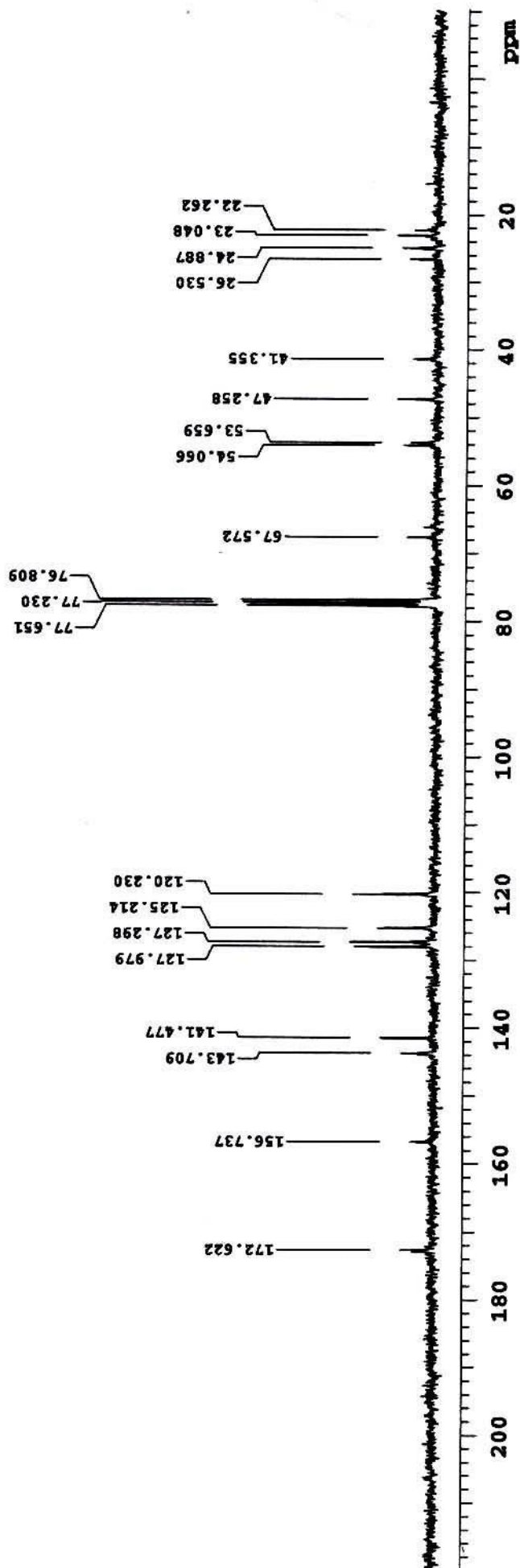
¹³C NMR of 3c



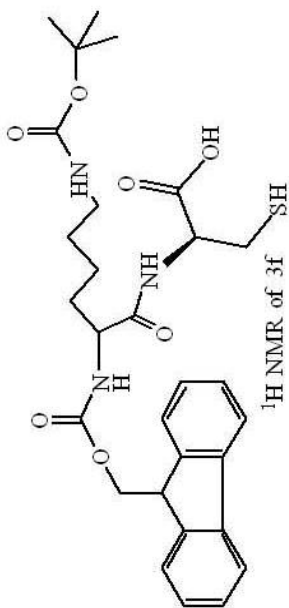
| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|---------|--------|
| 1 | 13024.649 | 172.622 | 5.2 |
| 2 | 11826.126 | 156.737 | 3.7 |
| 3 | 10843.157 | 143.709 | 5.3 |
| 4 | 10674.739 | 141.477 | 8.7 |
| 5 | 9656.286 | 127.979 | 12.9 |
| 6 | 9604.913 | 127.298 | 13.7 |
| 7 | 9447.617 | 125.214 | 9.2 |
| 8 | 9071.589 | 120.230 | 13.3 |
| 9 | 5858.934 | 77.651 | 30.5 |
| 10 | 5827.157 | 77.230 | 31.2 |
| 11 | 5795.380 | 76.809 | 31.5 |
| 12 | 5098.404 | 67.572 | 4.6 |
| 13 | 4079.421 | 54.066 | 5.3 |
| 14 | 4048.703 | 53.659 | 4.2 |
| 15 | 3565.693 | 47.258 | 6.5 |
| 16 | 3120.285 | 41.355 | 3.9 |
| 17 | 2001.734 | 26.530 | 4.5 |
| 18 | 1877.804 | 24.887 | 5.8 |
| 19 | 1739.045 | 23.048 | 6.6 |
| 20 | 1679.727 | 22.262 | 3.7 |



¹³C NMR of 3d



| INDEX | FREQUENCY PPM | HEIGHT |
|-------|---------------|--------|
| 1 | 2327.345 | 24.1 |
| 2 | 2319.925 | 26.1 |
| 3 | 2272.132 | 16.8 |
| 4 | 2221.451 | 14.5 |
| 5 | 2214.721 | 25.4 |
| 6 | 2207.441 | 18.1 |
| 7 | 2192.883 | 20.6 |
| 8 | 2185.603 | 28.2 |
| 9 | 2178.599 | 57.4 |
| 10 | 1313.175 | 18.4 |
| 11 | 1282.769 | 9.7 |
| 12 | 1257.137 | 11.2 |
| 13 | 1124.185 | 21.6 |
| 14 | 919.813 | 23.4 |
| 15 | 429.209 | 94.5 |
| 16 | -0.000 | 68.9 |



1.430

-0.000

7.356
7.308
7.283
7.260

7.756
7.731
7.672
7.403
7.381

4.278
4.208
4.189
3.746
3.065

12 11 10 9 8 7 6 5 4 3 2 1 -0 -1 ppm

2.212, 31
2.208, 08

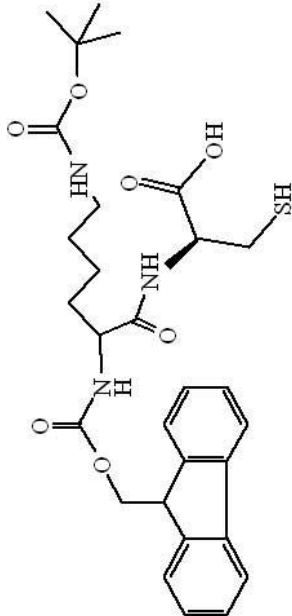
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1.44 1.22 4.00

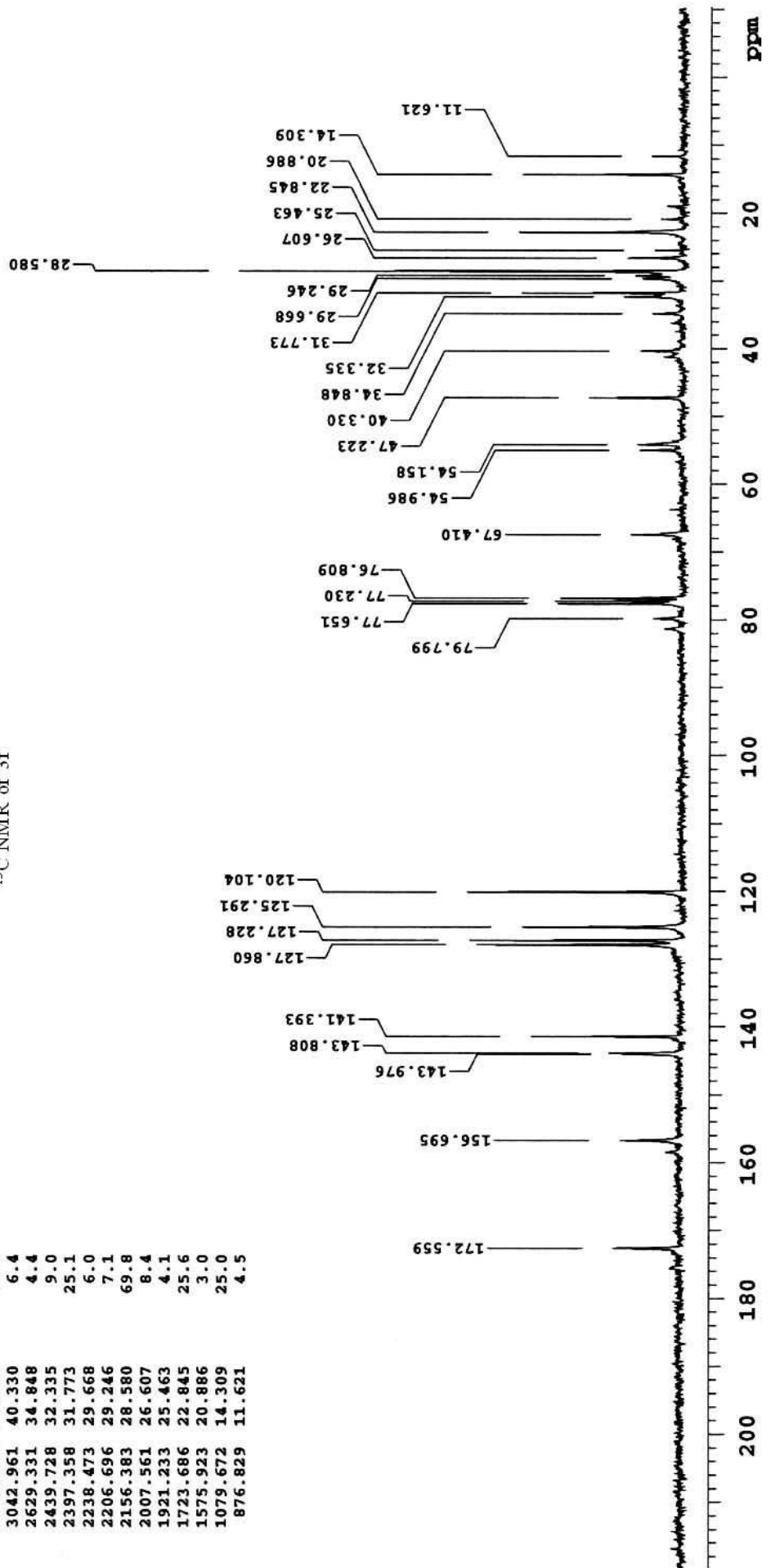
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14.85

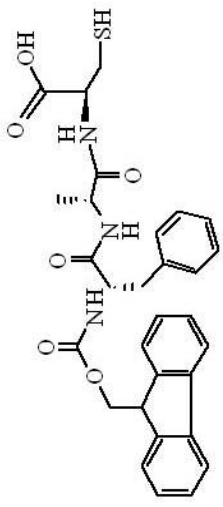
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| 3 | 10863.283 | 9.1 |
| 4 | 10850.572 | 11.1 |
| 5 | 10668.384 | 23.4 |
| 6 | 9647.283 | 32.1 |
| 7 | 9599.617 | 33.2 |
| 8 | 9453.443 | 24.9 |
| 9 | 9062.056 | 33.6 |
| 10 | 6020.997 | 4.1 |
| 11 | 5858.934 | 19.4 |
| 12 | 5827.157 | 19.8 |
| 13 | 5795.380 | 19.1 |
| 14 | 5086.223 | 7.8 |
| 15 | 4148.801 | 6.4 |
| 16 | 4086.306 | 6.7 |
| 17 | 3563.045 | 14.4 |
| 18 | 3042.961 | 6.4 |
| 19 | 2629.331 | 4.4 |
| 20 | 2439.728 | 32.335 |
| 21 | 2397.358 | 31.773 |
| 22 | 2238.473 | 29.668 |
| 23 | 2206.696 | 29.246 |
| 24 | 2156.383 | 28.580 |
| 25 | 2007.561 | 26.607 |
| 26 | 1921.233 | 25.463 |
| 27 | 1723.686 | 22.845 |
| 28 | 1575.923 | 20.886 |
| 29 | 1079.672 | 14.309 |
| 30 | 876.829 | 11.621 |



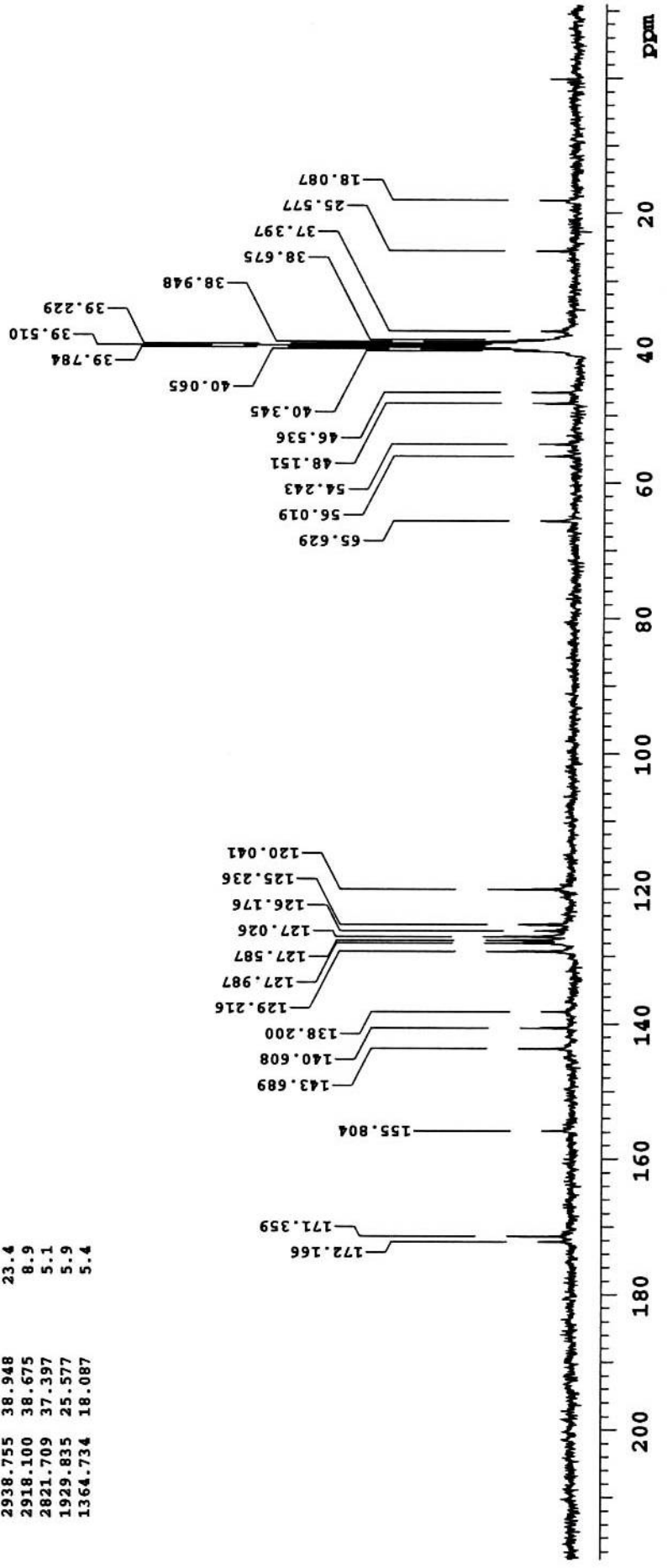
¹³C NMR of 3f

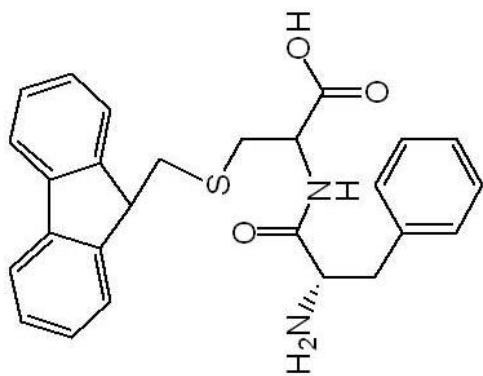


| INDEX | FREQUENCY PPM | HEIGHT |
|-------|---------------|---------|
| 1 | 12990.352 | 172.166 |
| 2 | 12929.446 | 171.359 |
| 3 | 11755.815 | 155.804 |
| 4 | 10841.696 | 143.689 |
| 5 | 10609.194 | 140.608 |
| 6 | 10427.536 | 138.200 |
| 7 | 9749.626 | 129.216 |
| 8 | 9656.944 | 127.987 |
| 9 | 9626.755 | 127.587 |
| 10 | 9584.386 | 127.026 |
| 11 | 9520.302 | 126.176 |
| 12 | 9449.334 | 125.236 |
| 13 | 9057.417 | 120.041 |
| 14 | 4951.828 | 65.629 |
| 15 | 4226.783 | 56.019 |
| 16 | 4092.790 | 54.243 |
| 17 | 3633.082 | 48.151 |
| 18 | 3511.270 | 46.536 |
| 19 | 3044.148 | 40.345 |
| 20 | 3022.964 | 40.065 |
| 21 | 3001.779 | 39.784 |
| 22 | 2981.124 | 39.510 |
| 23 | 2959.939 | 39.229 |
| 24 | 2938.755 | 38.948 |
| 25 | 2918.100 | 38.675 |
| 26 | 2821.709 | 37.397 |
| 27 | 1929.835 | 25.577 |
| 28 | 1366.734 | 18.087 |

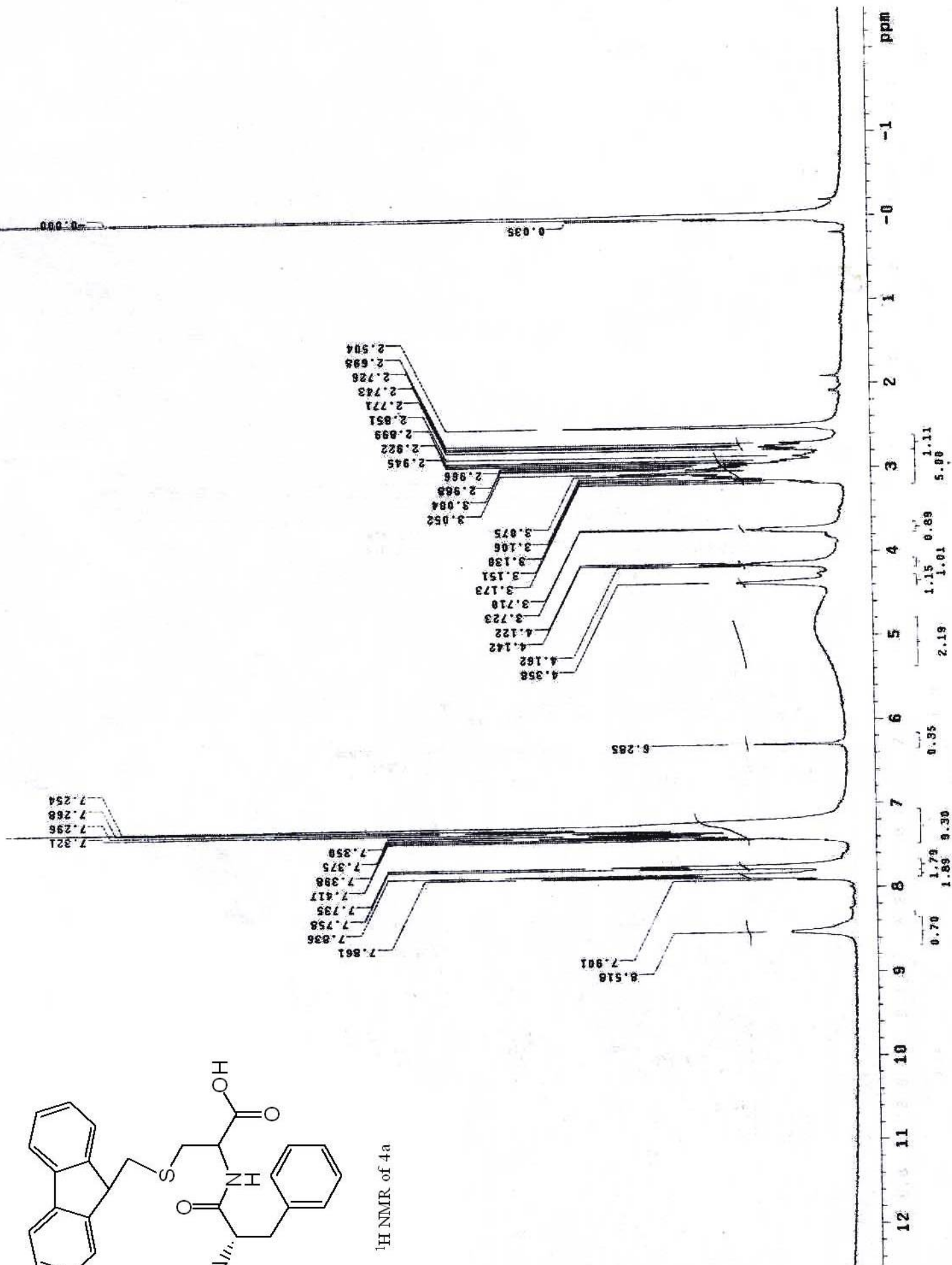


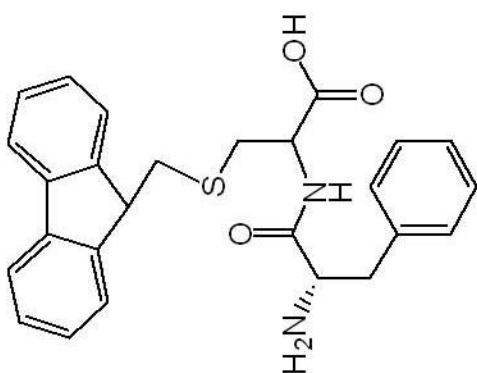
¹³C NMR of 7b



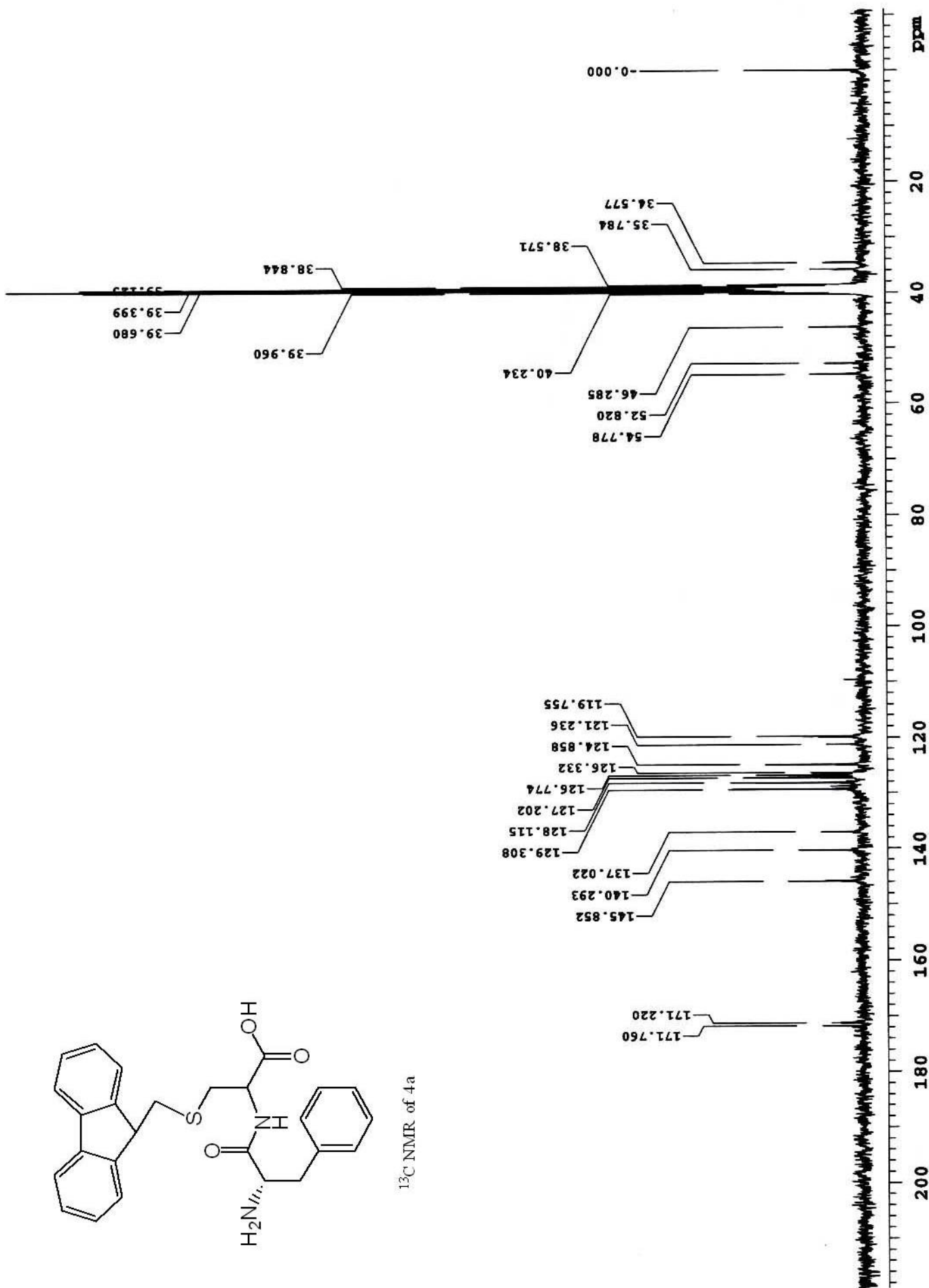


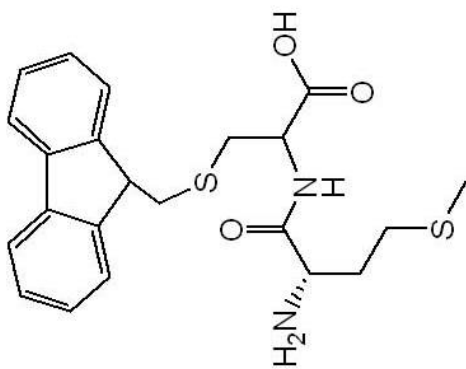
¹H NMR of 4a



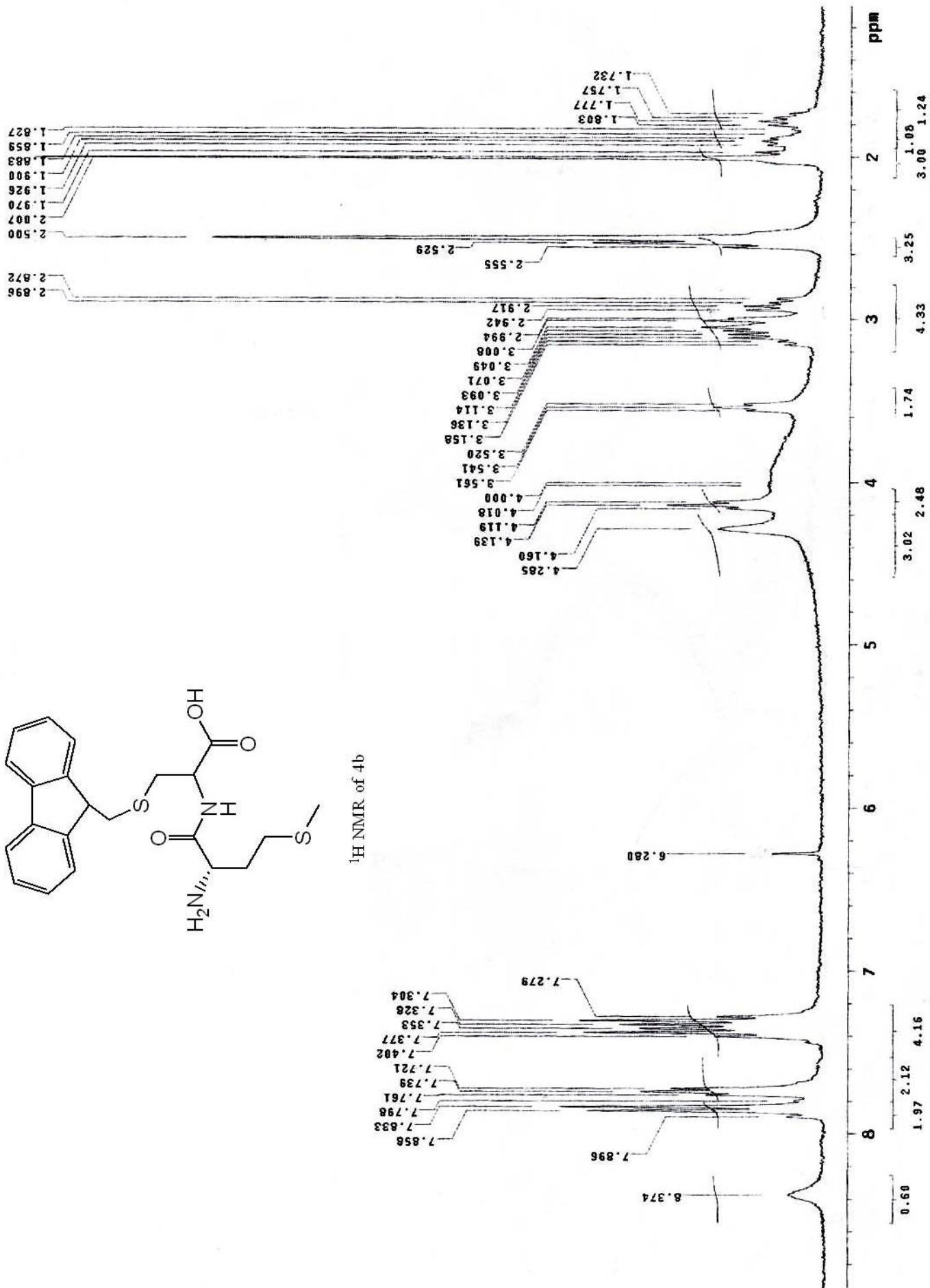


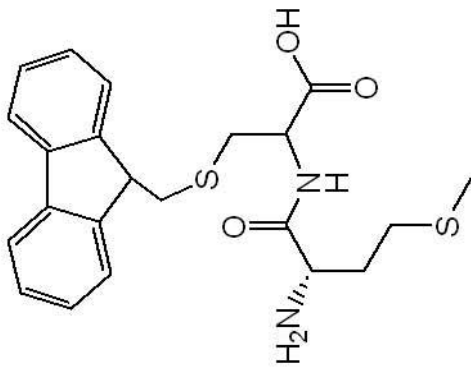
¹³C NMR of 4a





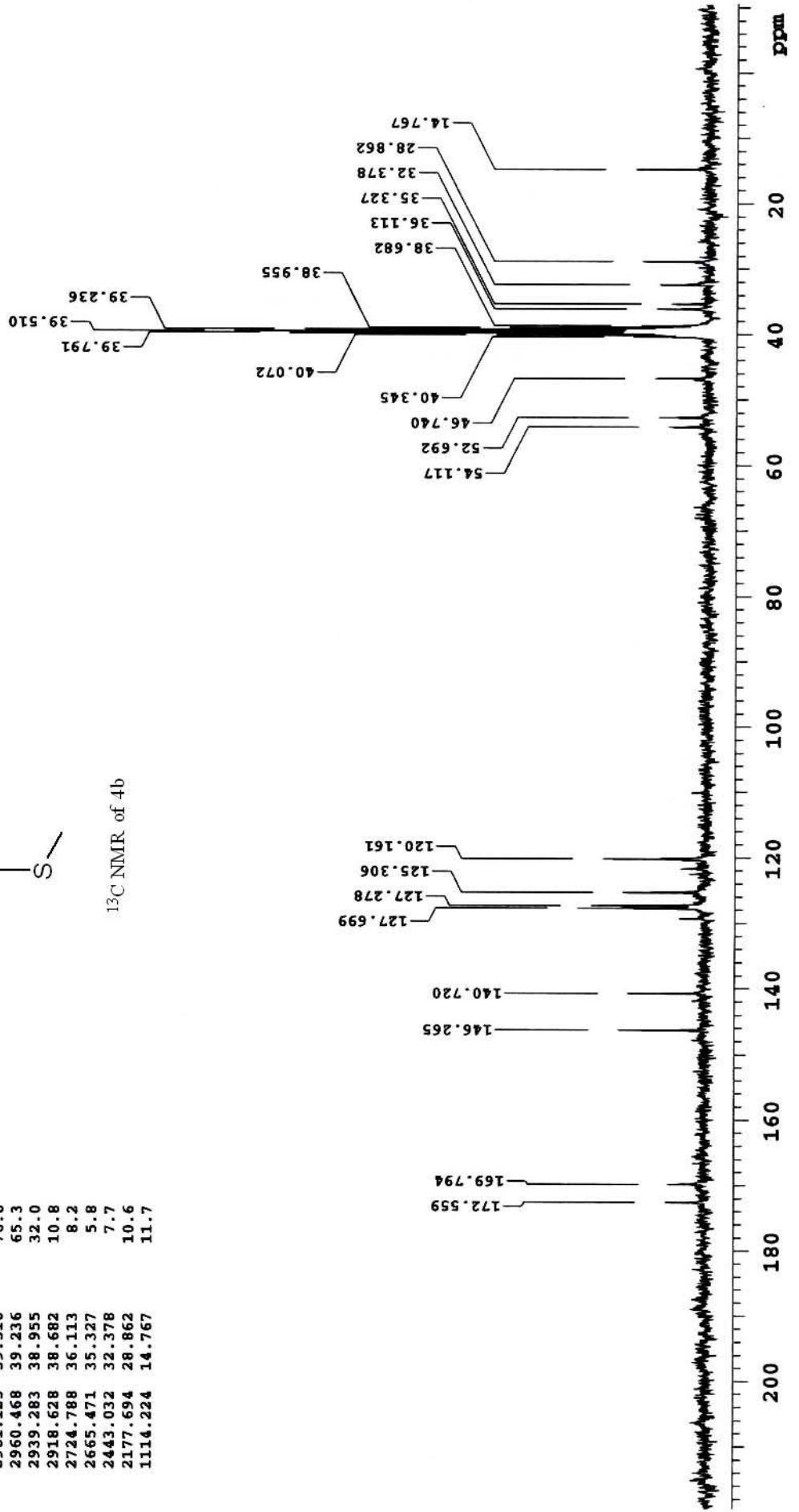
¹H NMR of 4b



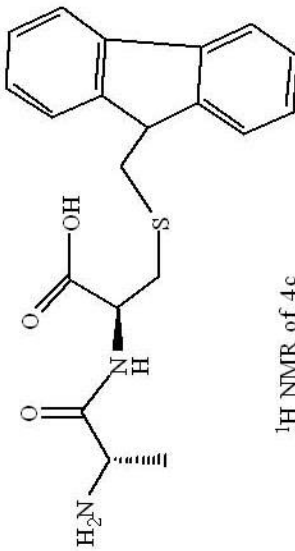


^{13}C NMR of 4b

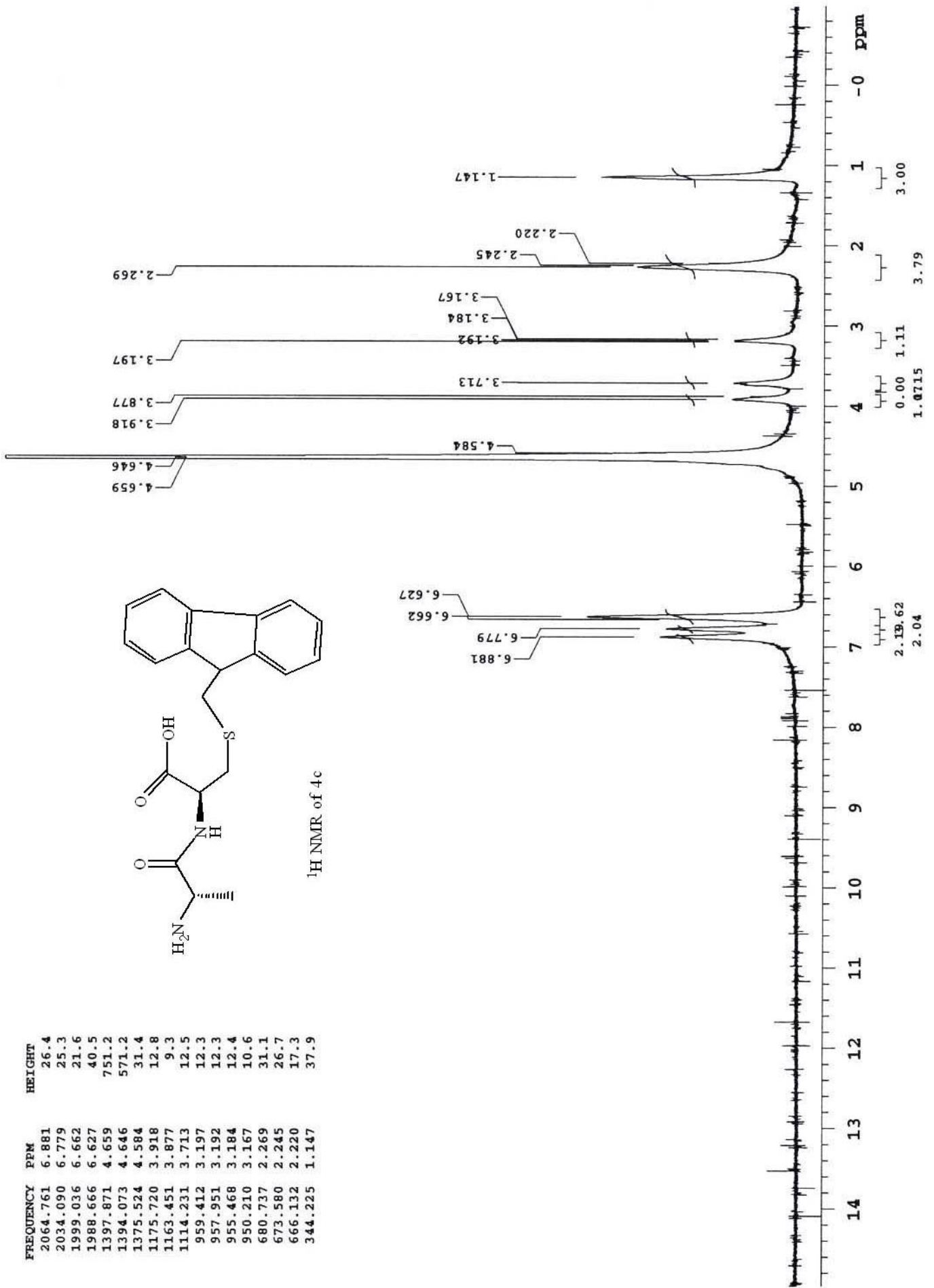
| INDEX | FREQUENCY PPM | HEIGHT |
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| 1 | 13020.009 | 172.559 |
| 2 | 12811.340 | 169.794 |
| 3 | 11036.065 | 146.265 |
| 4 | 10617.667 | 140.720 |
| 5 | 9635.228 | 127.699 |
| 6 | 9603.451 | 127.278 |
| 7 | 9454.629 | 125.306 |
| 8 | 9066.420 | 120.161 |
| 9 | 4083.255 | 54.117 |
| 10 | 3975.743 | 52.692 |
| 11 | 3526.628 | 46.740 |
| 12 | 3044.147 | 40.345 |
| 13 | 3023.492 | 40.072 |
| 14 | 3002.308 | 39.791 |
| 15 | 2981.123 | 39.510 |
| 16 | 2960.468 | 39.236 |
| 17 | 2939.283 | 38.955 |
| 18 | 2918.628 | 38.682 |
| 19 | 2724.788 | 36.113 |
| 20 | 2665.471 | 35.327 |
| 21 | 2443.032 | 32.378 |
| 22 | 2177.694 | 28.862 |
| 23 | 1114.224 | 14.767 |



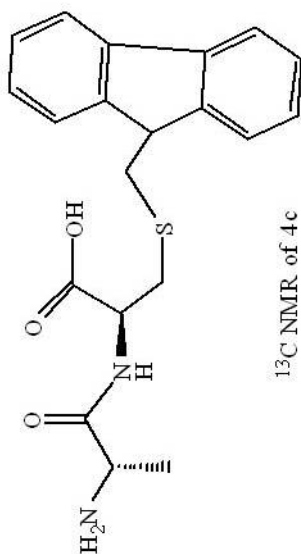
| INDEX | FREQUENCY PPM | HEIGHT |
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| 1 | 2064.761 | 6.881 |
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| 3 | 1999.036 | 6.662 |
| 4 | 1988.666 | 6.627 |
| 5 | 1397.871 | 4.659 |
| 6 | 1394.073 | 4.646 |
| 7 | 1375.524 | 4.584 |
| 8 | 1175.720 | 3.918 |
| 9 | 1163.451 | 3.877 |
| 10 | 1114.231 | 3.713 |
| 11 | 959.412 | 3.197 |
| 12 | 957.951 | 3.192 |
| 13 | 955.468 | 3.184 |
| 14 | 950.210 | 3.167 |
| 15 | 680.737 | 2.269 |
| 16 | 673.580 | 2.245 |
| 17 | 666.132 | 2.220 |
| 18 | 344.225 | 1.147 |



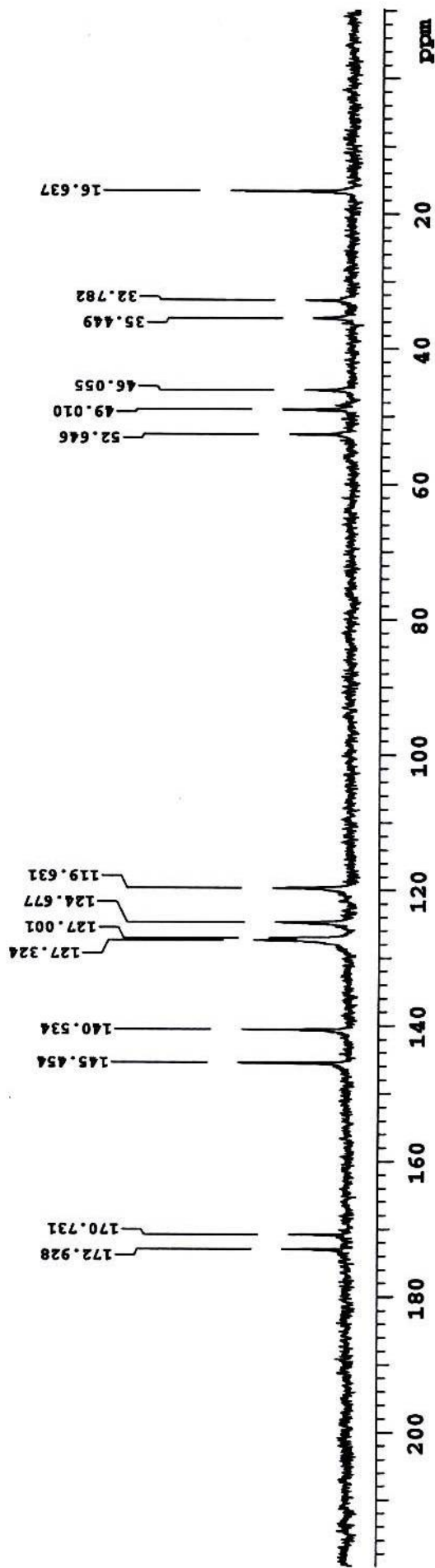
¹H NMR of 4c

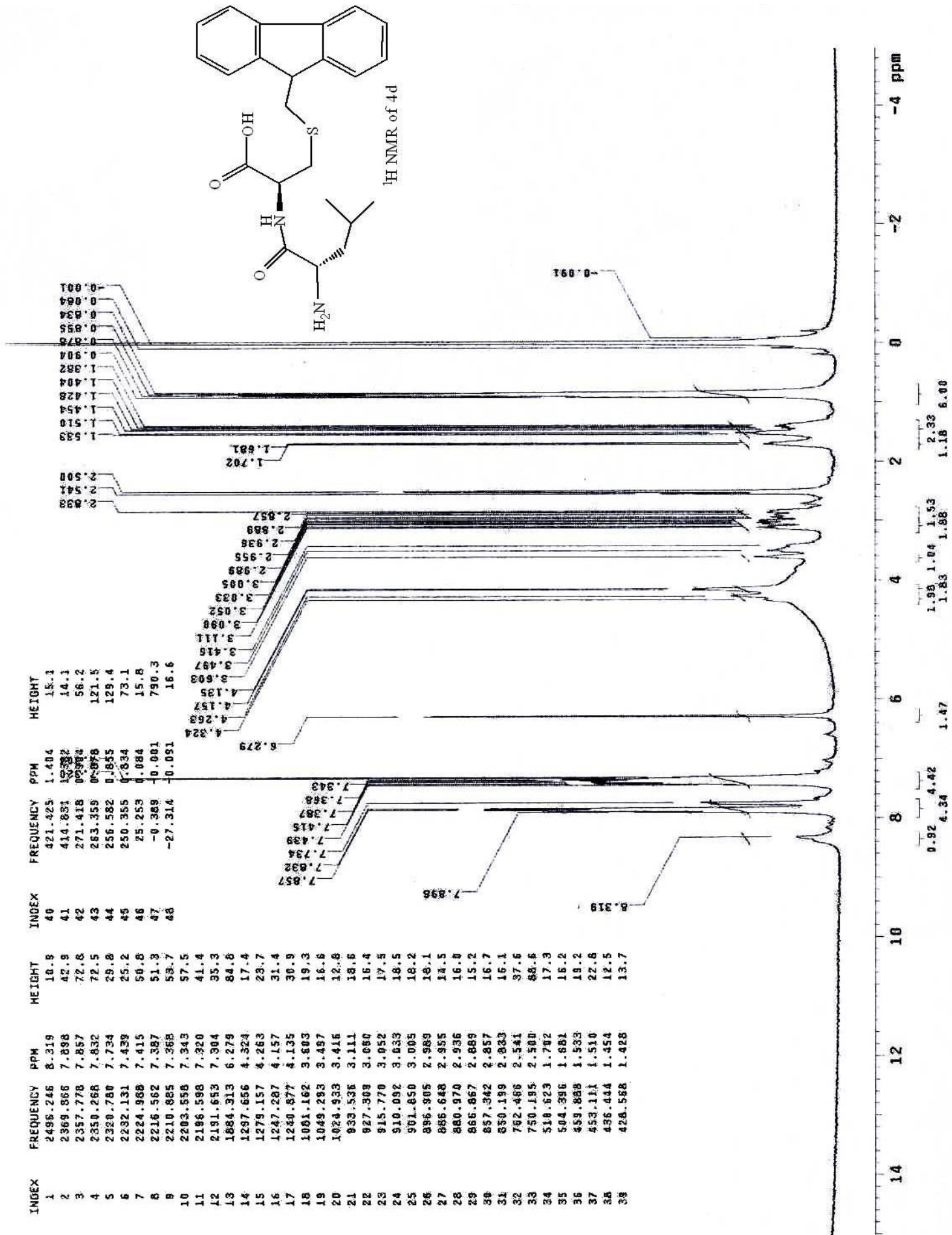


| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|---------|--------|
| 1 | 13047.771 | 172.928 | 10.2 |
| 2 | 12882.001 | 170.731 | 9.2 |
| 3 | 10974.851 | 145.454 | 17.3 |
| 4 | 10603.590 | 140.534 | 16.8 |
| 5 | 9606.851 | 127.324 | 15.0 |
| 6 | 9582.488 | 127.001 | 12.5 |
| 7 | 9407.185 | 124.677 | 11.6 |
| 8 | 9026.391 | 119.631 | 12.1 |
| 9 | 3972.258 | 52.646 | 9.7 |
| 10 | 3697.916 | 49.010 | 10.9 |
| 11 | 3474.948 | 46.055 | 7.4 |
| 12 | 2674.697 | 35.449 | 6.0 |
| 13 | 2473.442 | 32.782 | 7.3 |
| 14 | 1255.324 | 16.637 | 19.2 |

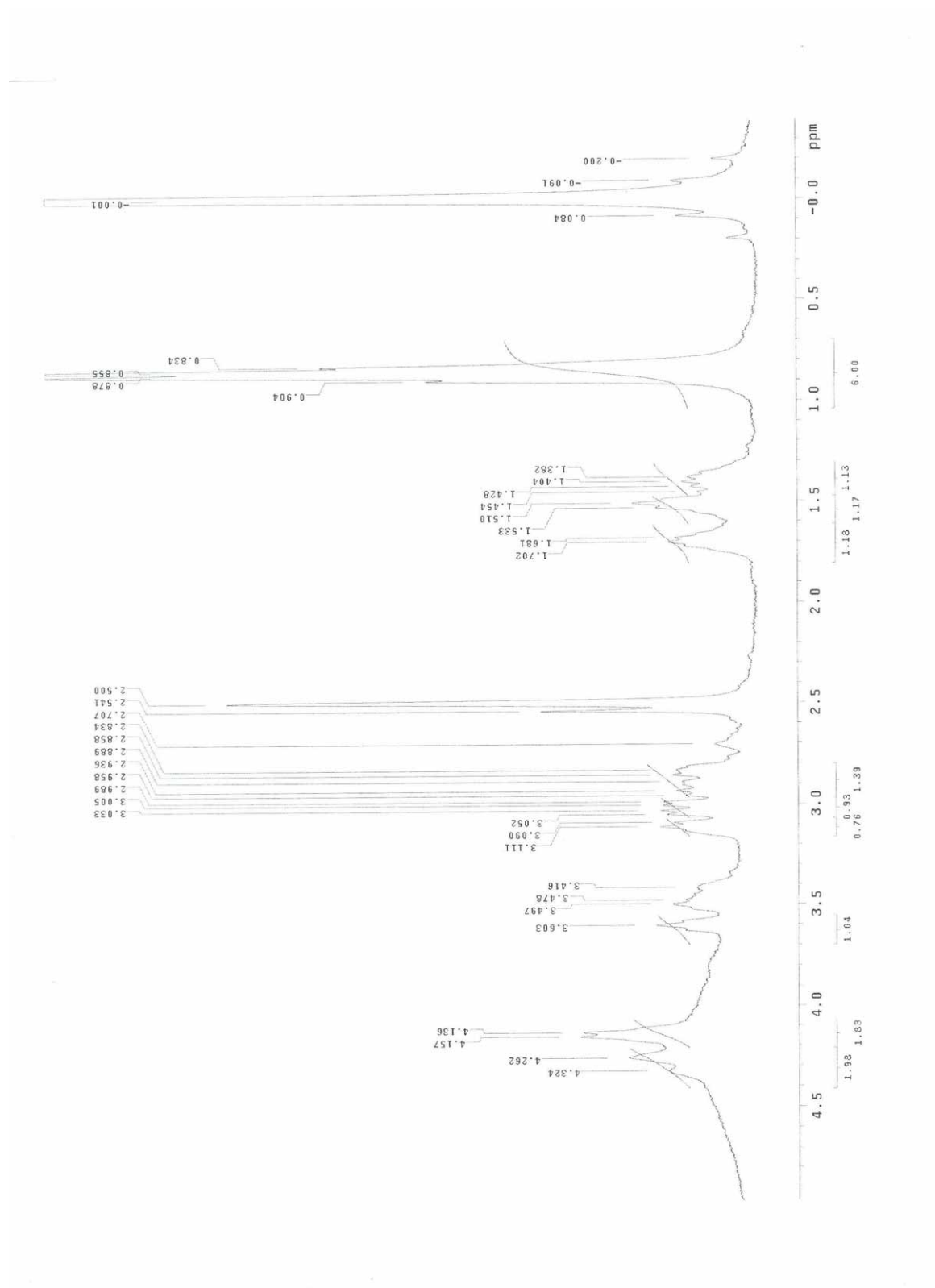


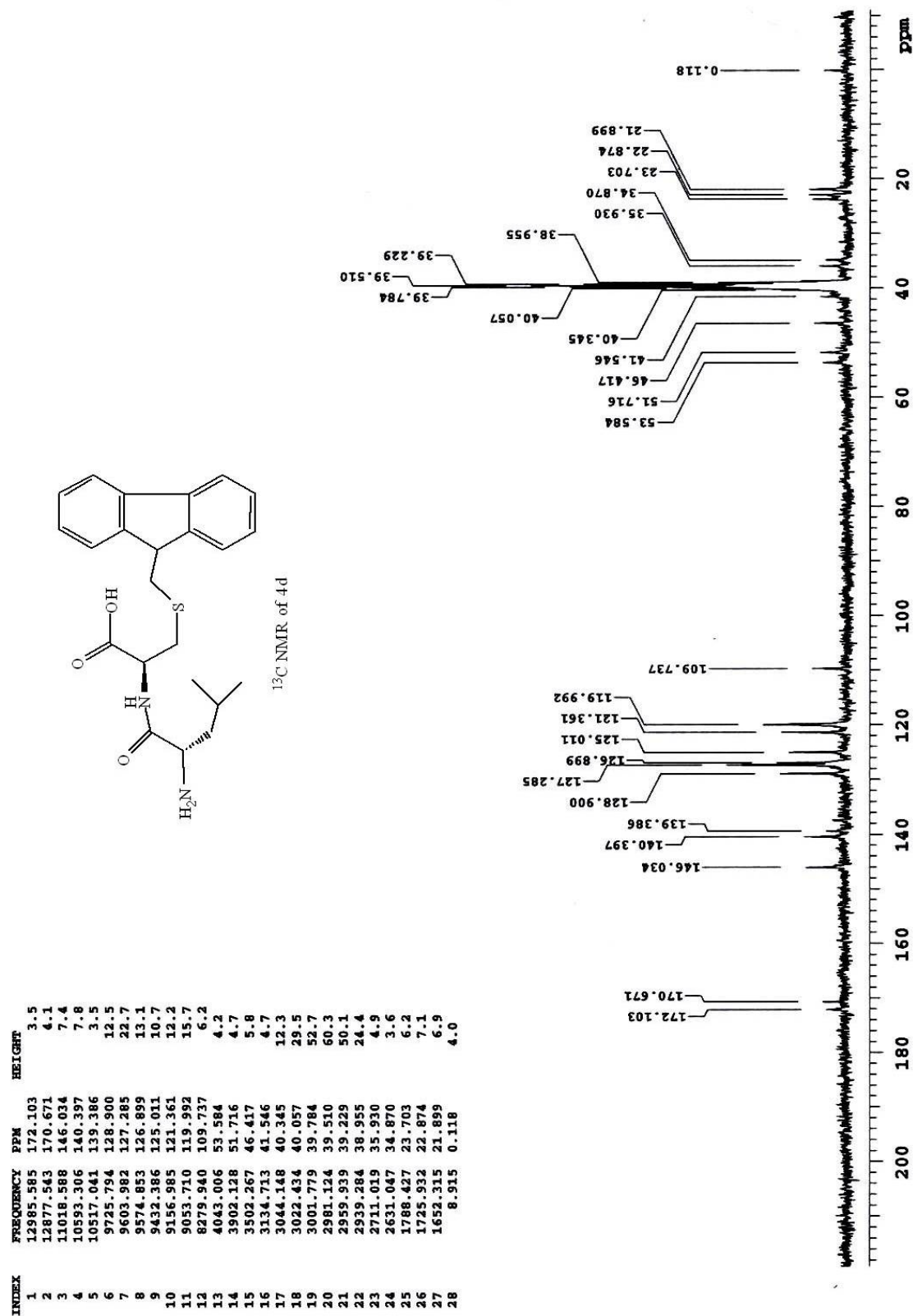
¹³C NMR of 4c

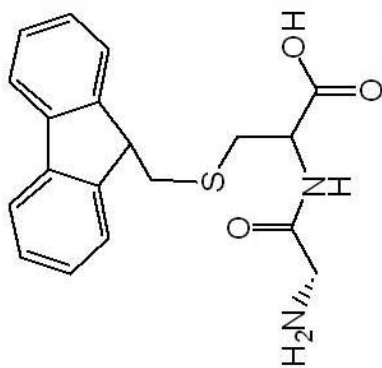




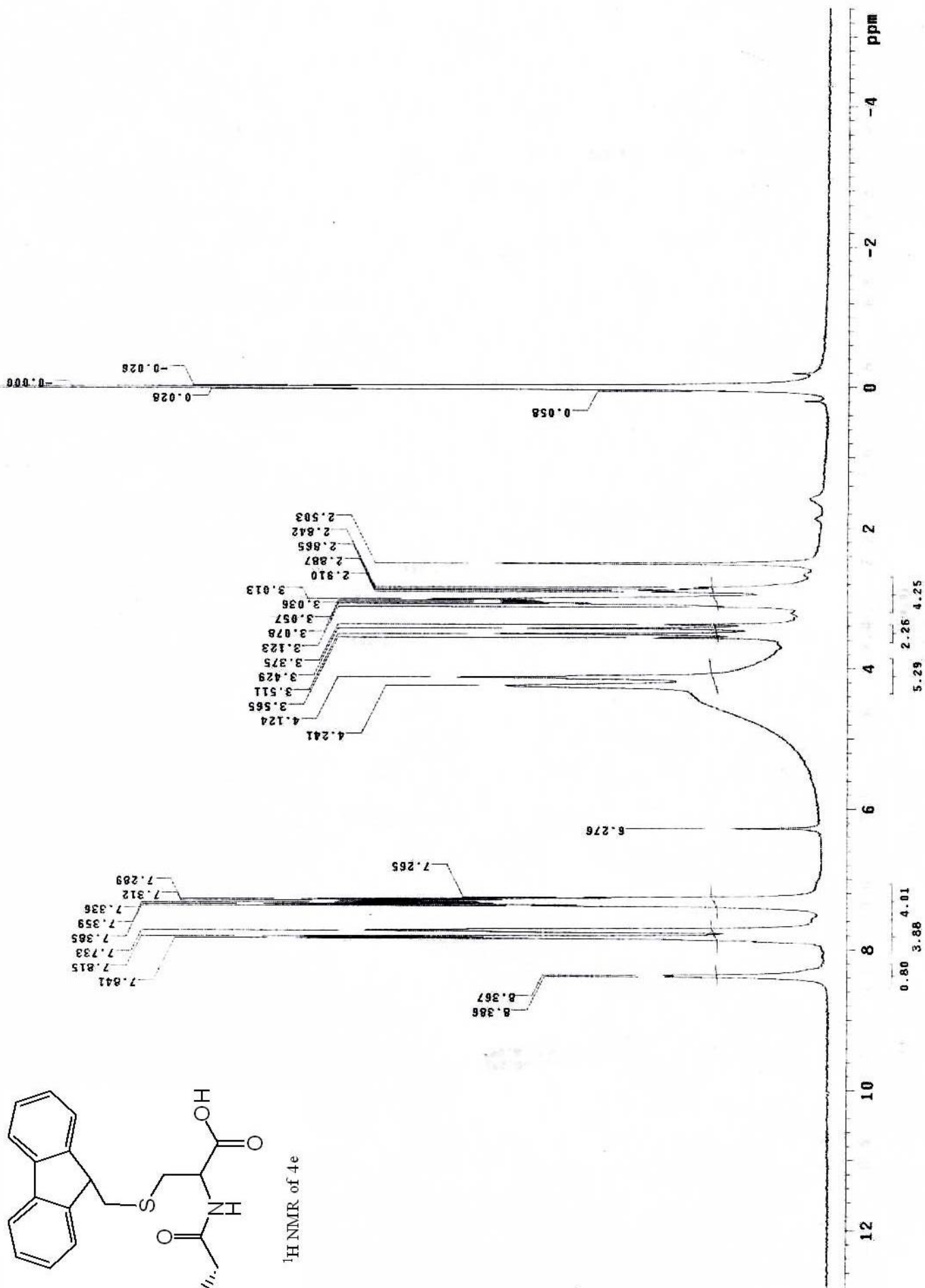
$^1\text{H-NMR}$ spectrum of **4d** in the range of 0-4.5 ppm

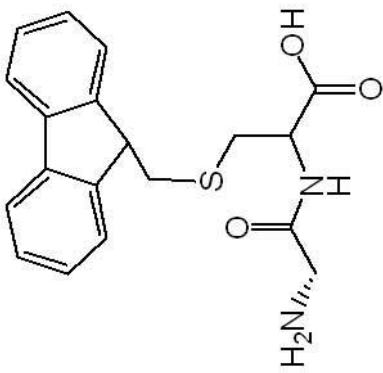




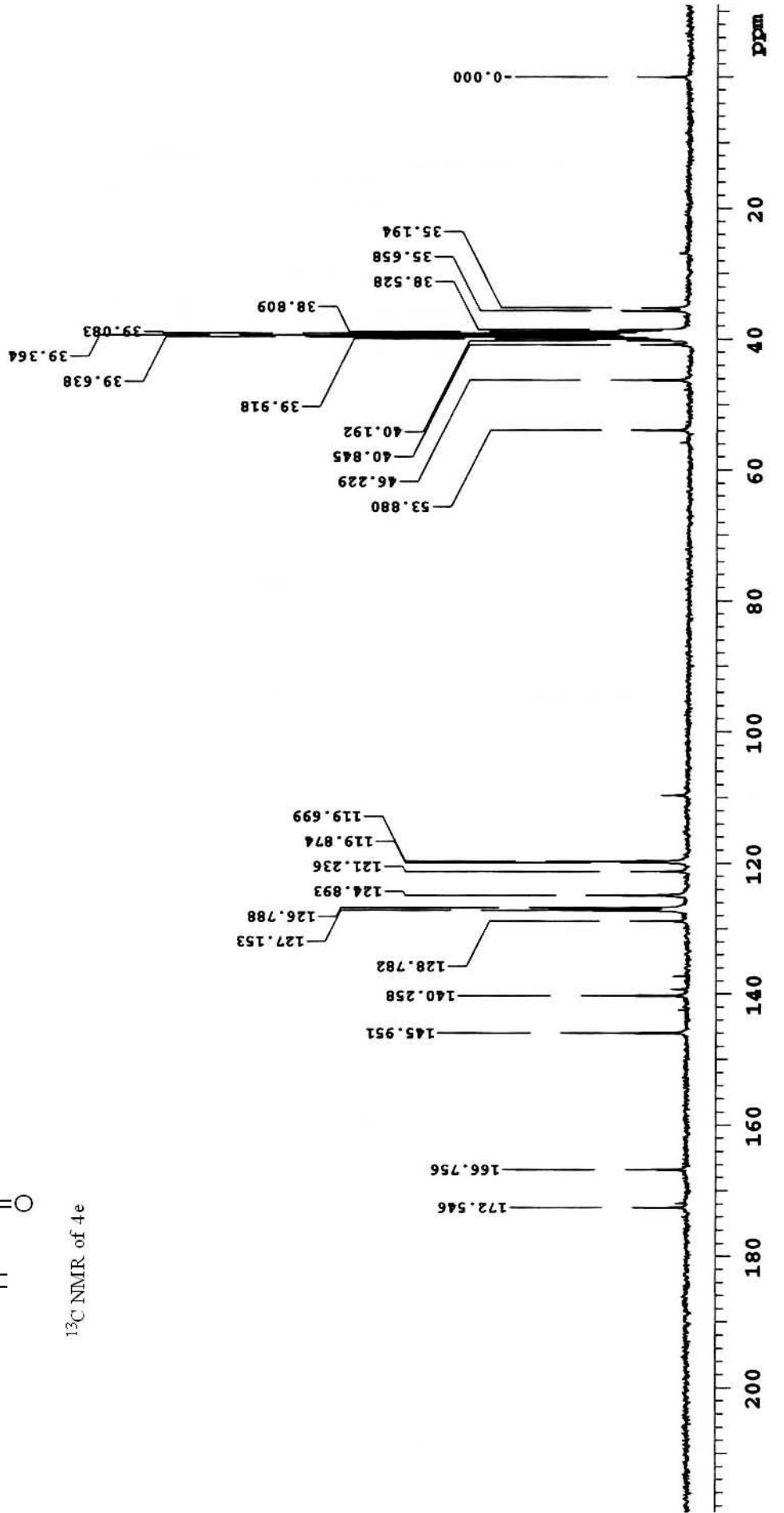


¹H NMR of 4e

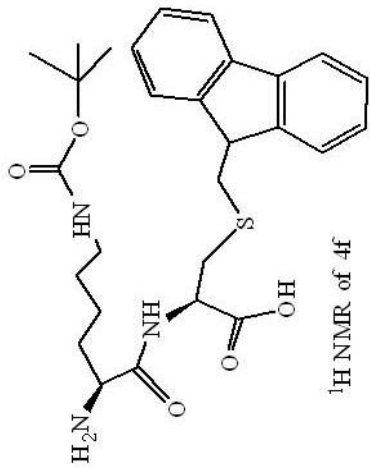




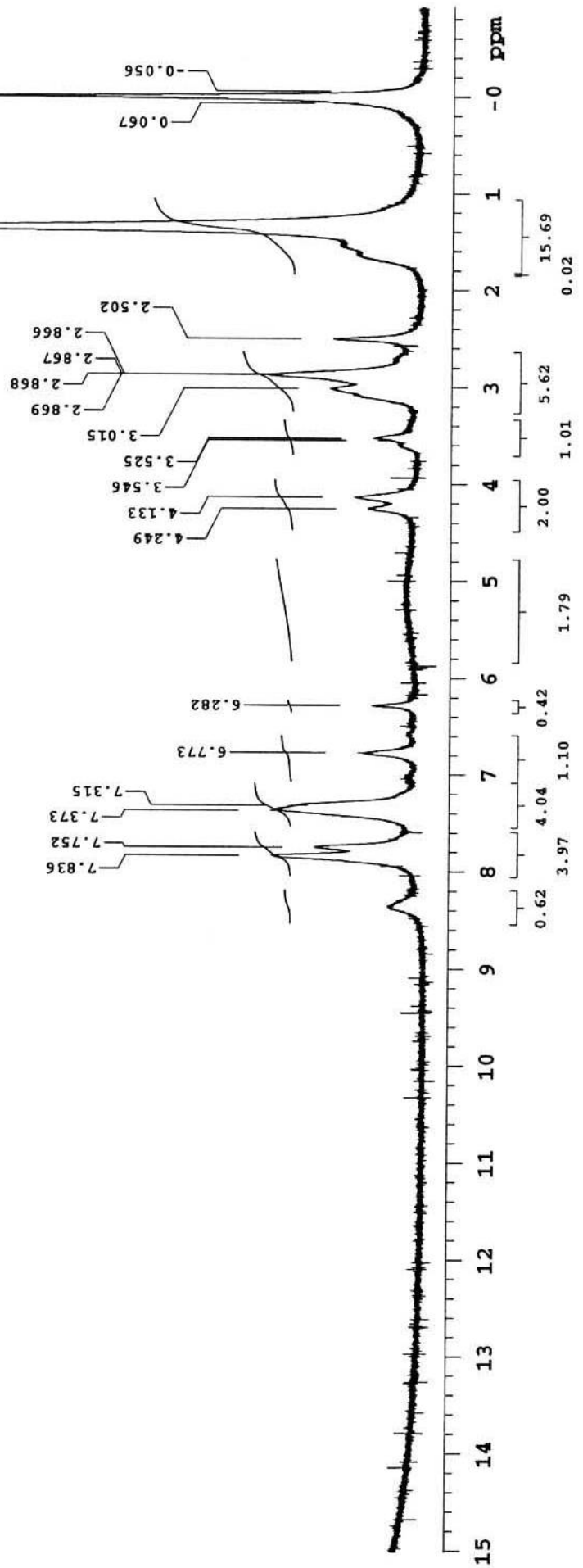
¹³C NMR of 4e



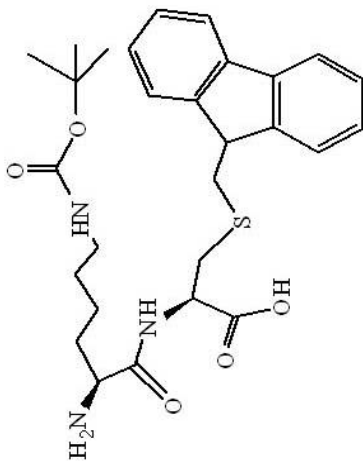
| INDEX | FREQUENCY PPM | HEIGHT |
|-------|---------------|--------|
| 1 | 2351.205 | 7.836 |
| 2 | 2326.230 | 7.752 |
| 3 | 2212.306 | 7.373 |
| 4 | 2195.072 | 7.315 |
| 5 | 2032.219 | 6.773 |
| 6 | 1884.995 | 6.282 |
| 7 | 1275.067 | 4.249 |
| 8 | 1240.305 | 4.133 |
| 9 | 1064.162 | 3.546 |
| 10 | 1057.736 | 3.525 |
| 11 | 904.815 | 3.015 |
| 12 | 860.999 | 2.869 |
| 13 | 860.560 | 2.868 |
| 14 | 860.268 | 2.867 |
| 15 | 859.976 | 2.866 |
| 16 | 750.873 | 2.502 |
| 17 | 408.664 | 1.362 |
| 18 | 20.156 | 0.067 |
| 19 | 0.000 | 0.000 |
| 20 | -16.942 | -0.056 |



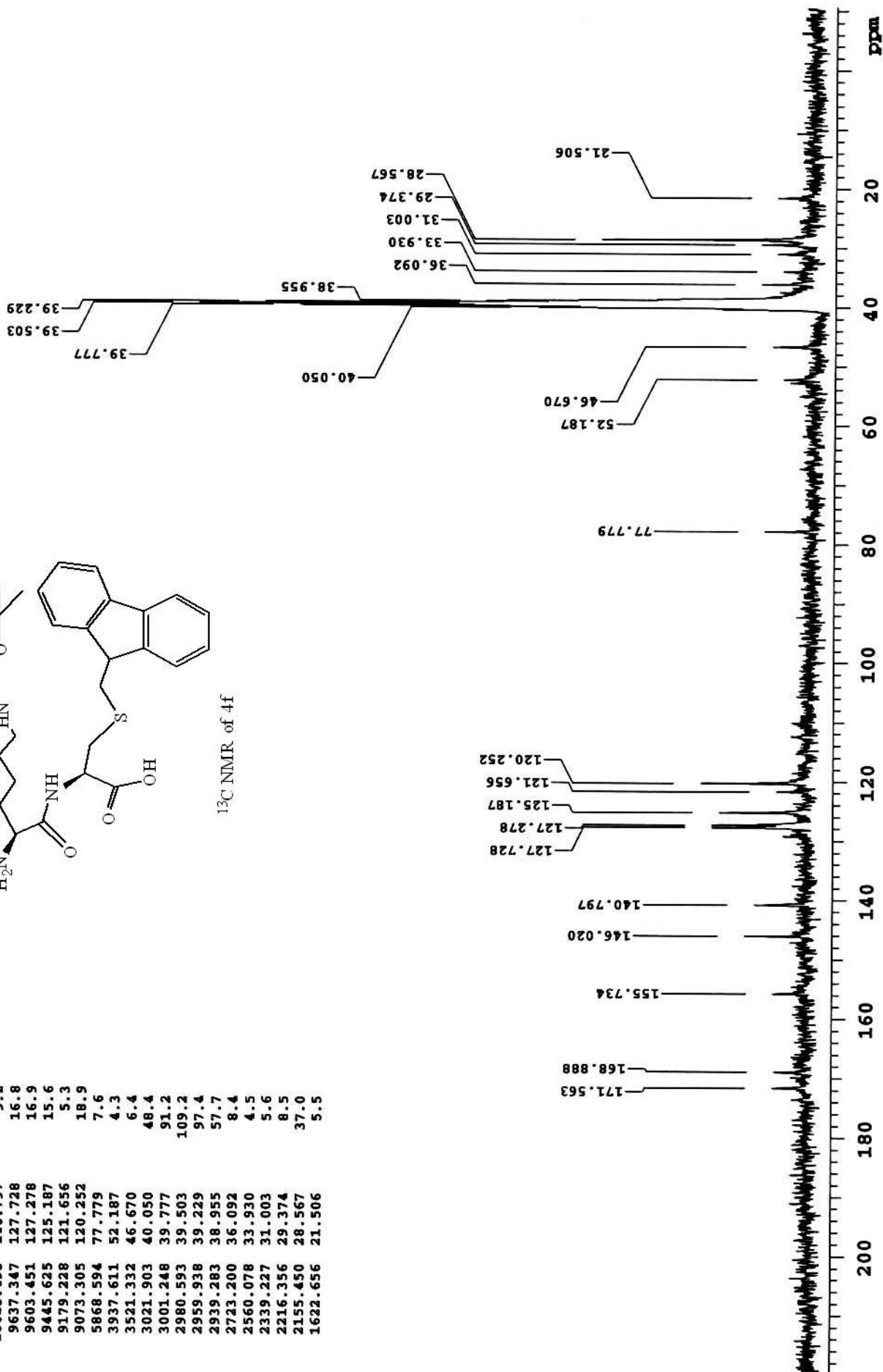
¹H NMR of 4f

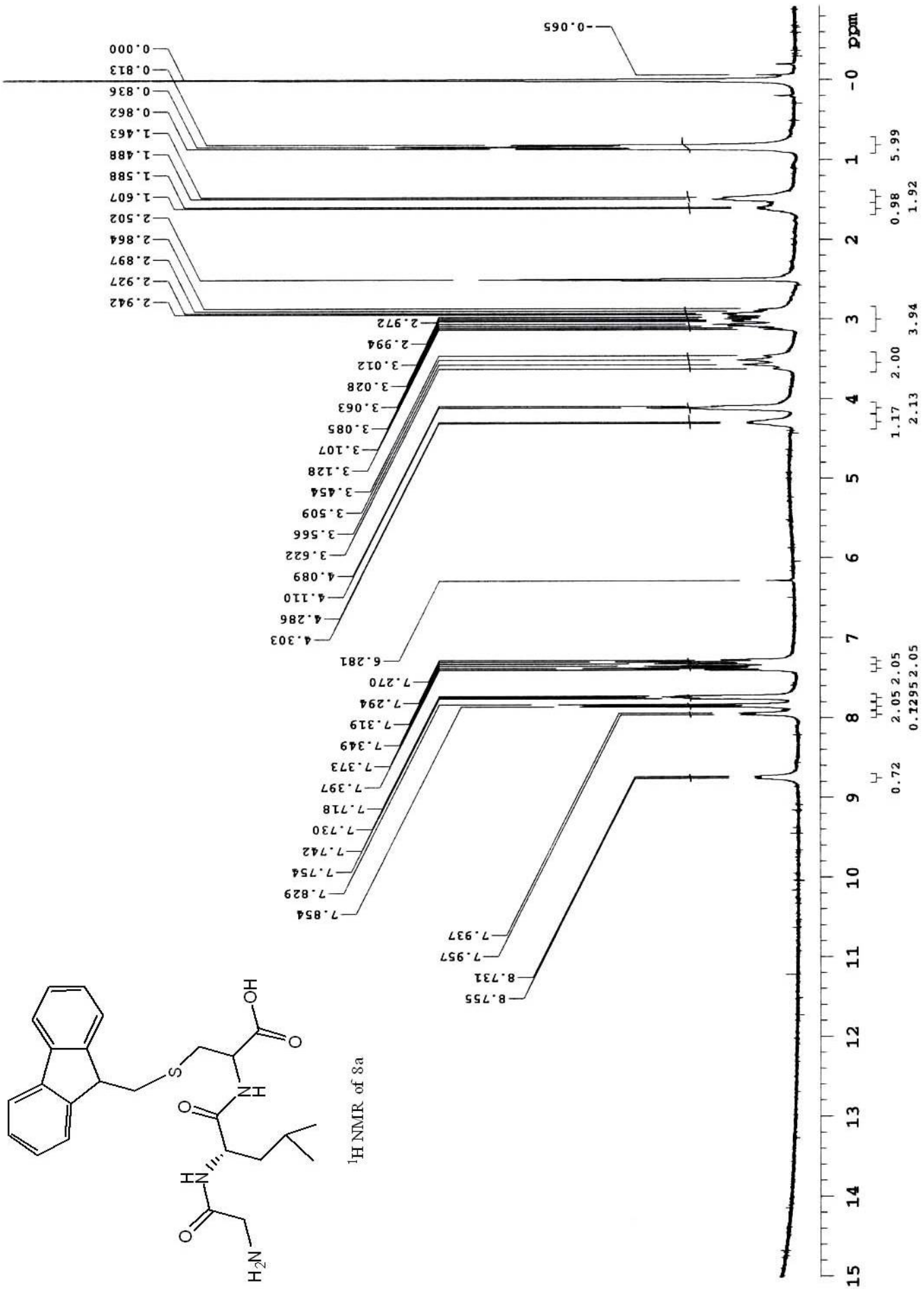


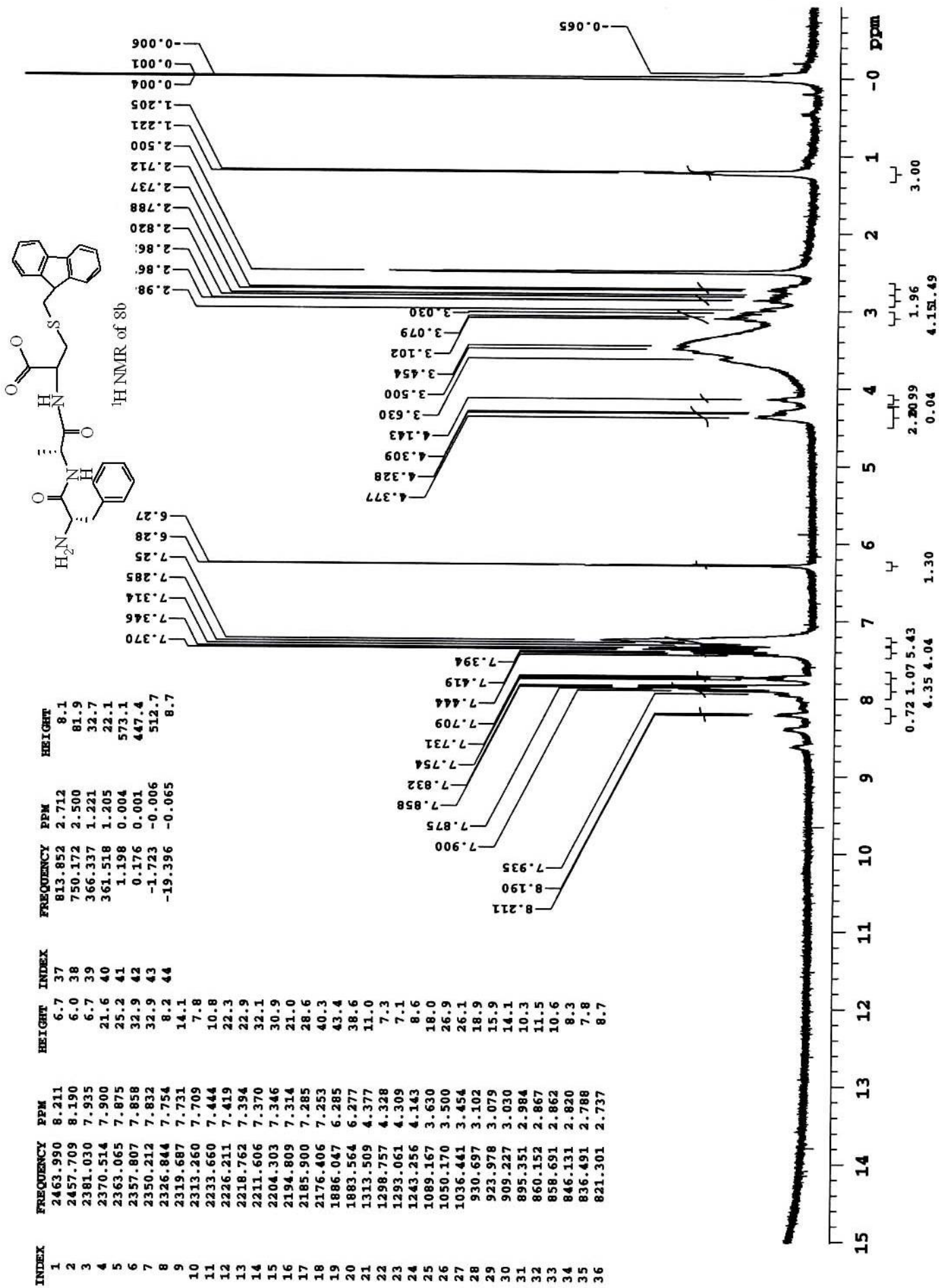
| INDEX | FREQUENCY PPM | HEIGHT |
|-------|---------------|---------|
| 1 | 12944.804 | 171.563 |
| 2 | 12743.020 | 168.888 |
| 3 | 11750.518 | 155.734 |
| 4 | 11017.538 | 146.020 |
| 5 | 10623.493 | 140.797 |
| 6 | 9637.347 | 127.728 |
| 7 | 9603.451 | 127.278 |
| 8 | 9445.625 | 125.187 |
| 9 | 9179.228 | 121.656 |
| 10 | 9073.305 | 120.252 |
| 11 | 5868.594 | 77.779 |
| 12 | 3937.611 | 52.187 |
| 13 | 3521.332 | 46.670 |
| 14 | 3021.903 | 40.050 |
| 15 | 3001.248 | 39.777 |
| 16 | 2980.593 | 39.503 |
| 17 | 2959.938 | 39.229 |
| 18 | 2939.283 | 38.955 |
| 19 | 2723.200 | 36.092 |
| 20 | 2560.078 | 33.930 |
| 21 | 2339.227 | 31.003 |
| 22 | 2216.356 | 29.374 |
| 23 | 2155.450 | 28.567 |
| 24 | 1622.656 | 21.506 |



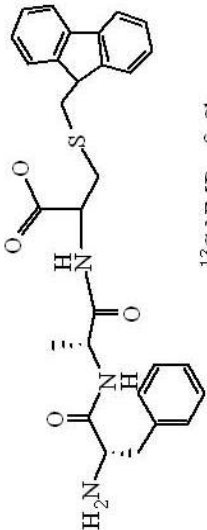
¹³C NMR of 4f



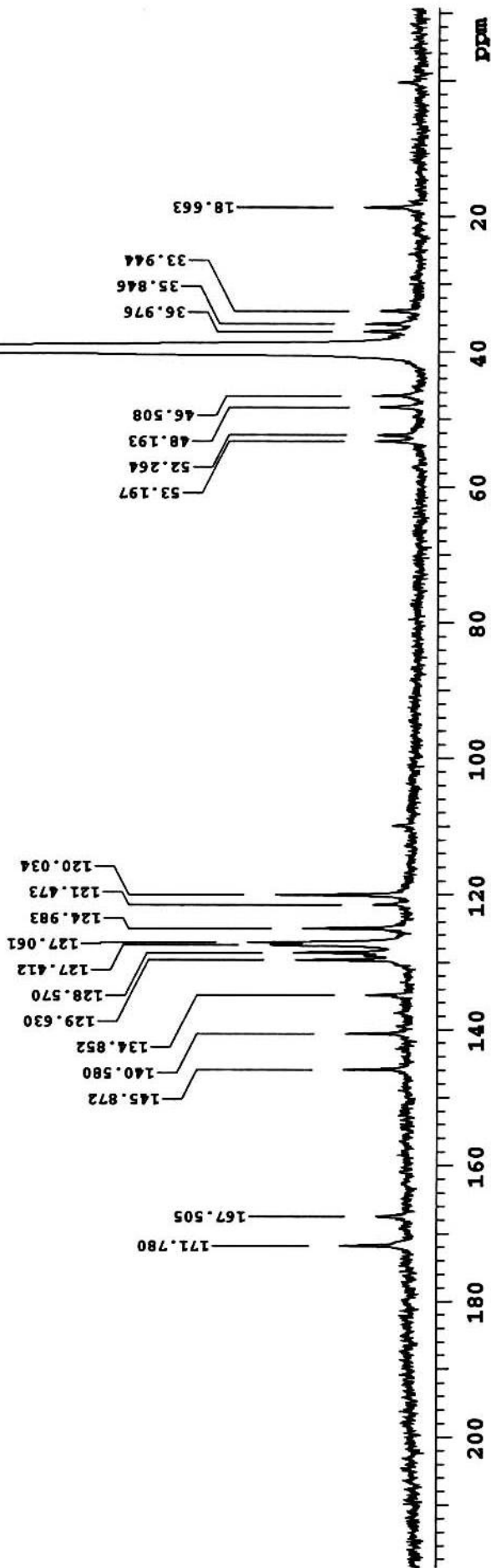


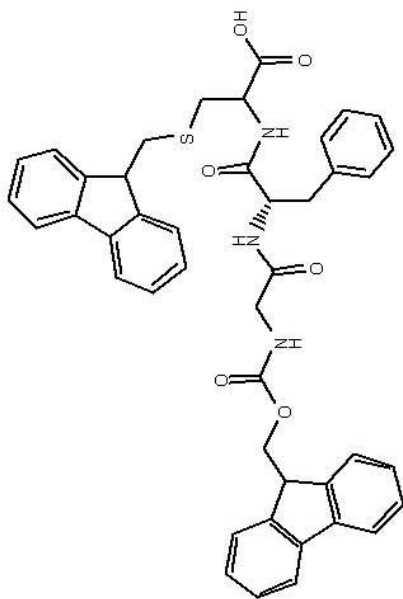


| INDEX | FREQUENCY | PPM | HEIGHT |
|-------|-----------|---------|--------|
| 1 | 12961.222 | 171.780 | 10.4 |
| 2 | 12638.686 | 167.505 | 4.7 |
| 3 | 11006.407 | 145.872 | 9.9 |
| 4 | 10607.076 | 140.580 | 9.6 |
| 5 | 10174.908 | 134.852 | 6.4 |
| 6 | 9780.873 | 129.630 | 17.5 |
| 7 | 9700.901 | 128.570 | 17.8 |
| 8 | 9613.515 | 127.412 | 21.4 |
| 9 | 9587.034 | 127.061 | 24.9 |
| 10 | 9430.267 | 124.983 | 16.3 |
| 11 | 9165.459 | 121.473 | 5.3 |
| 12 | 9056.887 | 120.034 | 20.6 |
| 13 | 4013.876 | 53.197 | 5.1 |
| 14 | 3943.437 | 52.264 | 4.7 |
| 15 | 3636.259 | 48.193 | 4.3 |
| 16 | 3509.151 | 46.508 | 5.6 |
| 17 | 3001.249 | 39.777 | 165.9 |
| 18 | 2980.594 | 39.503 | 191.3 |
| 19 | 2960.468 | 39.236 | 165.9 |
| 20 | 2789.932 | 36.976 | 7.0 |
| 21 | 2704.664 | 35.846 | 6.6 |
| 22 | 2561.137 | 33.944 | 4.4 |
| 23 | 1408.162 | 18.663 | 6.9 |

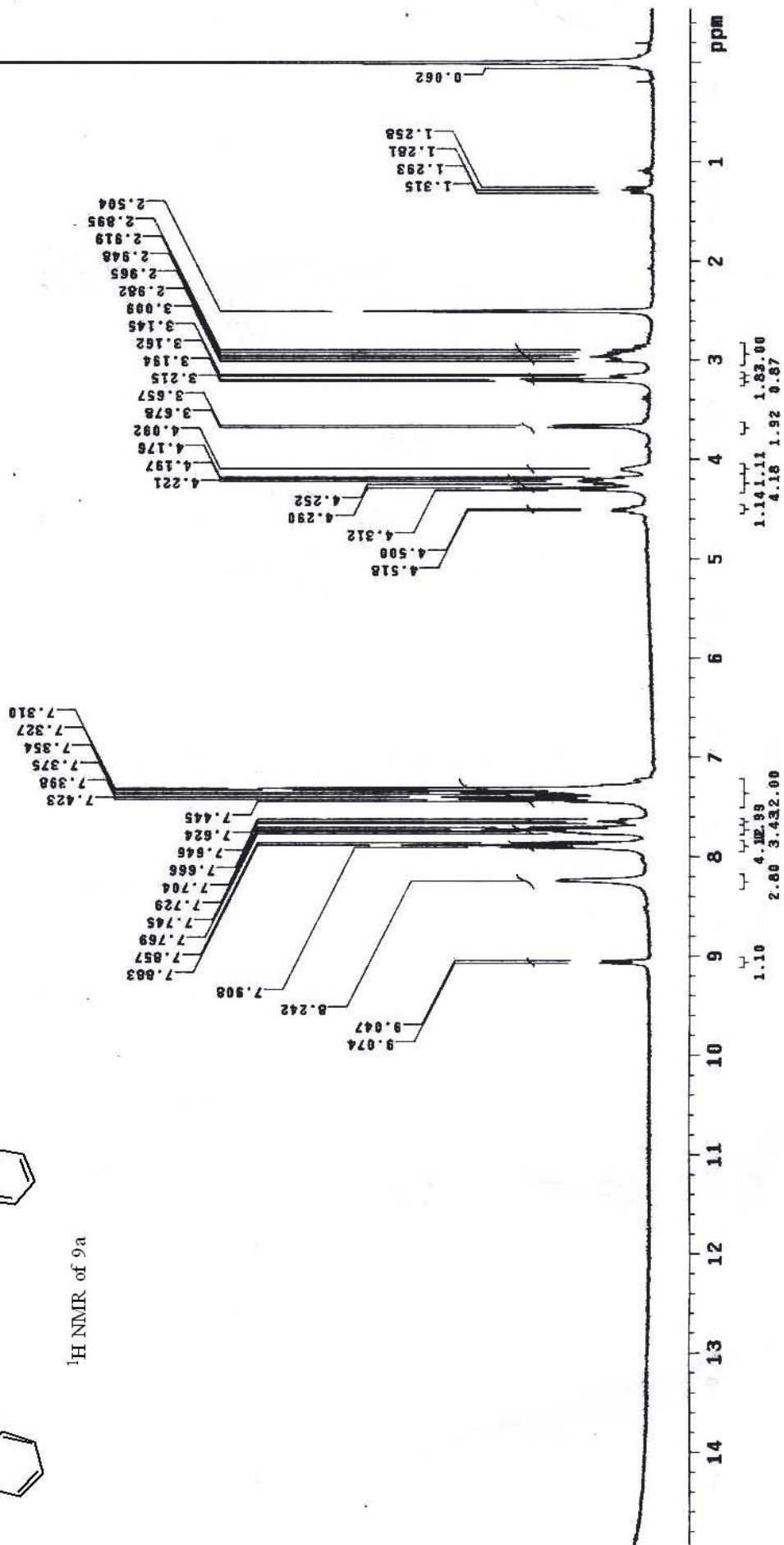


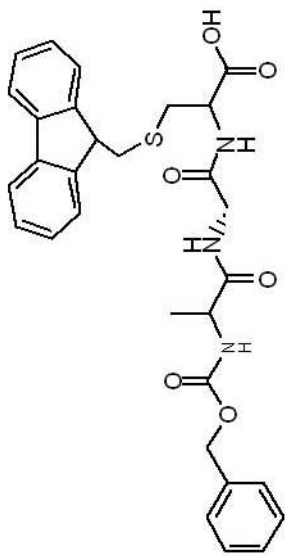
¹³C NMR of 8b



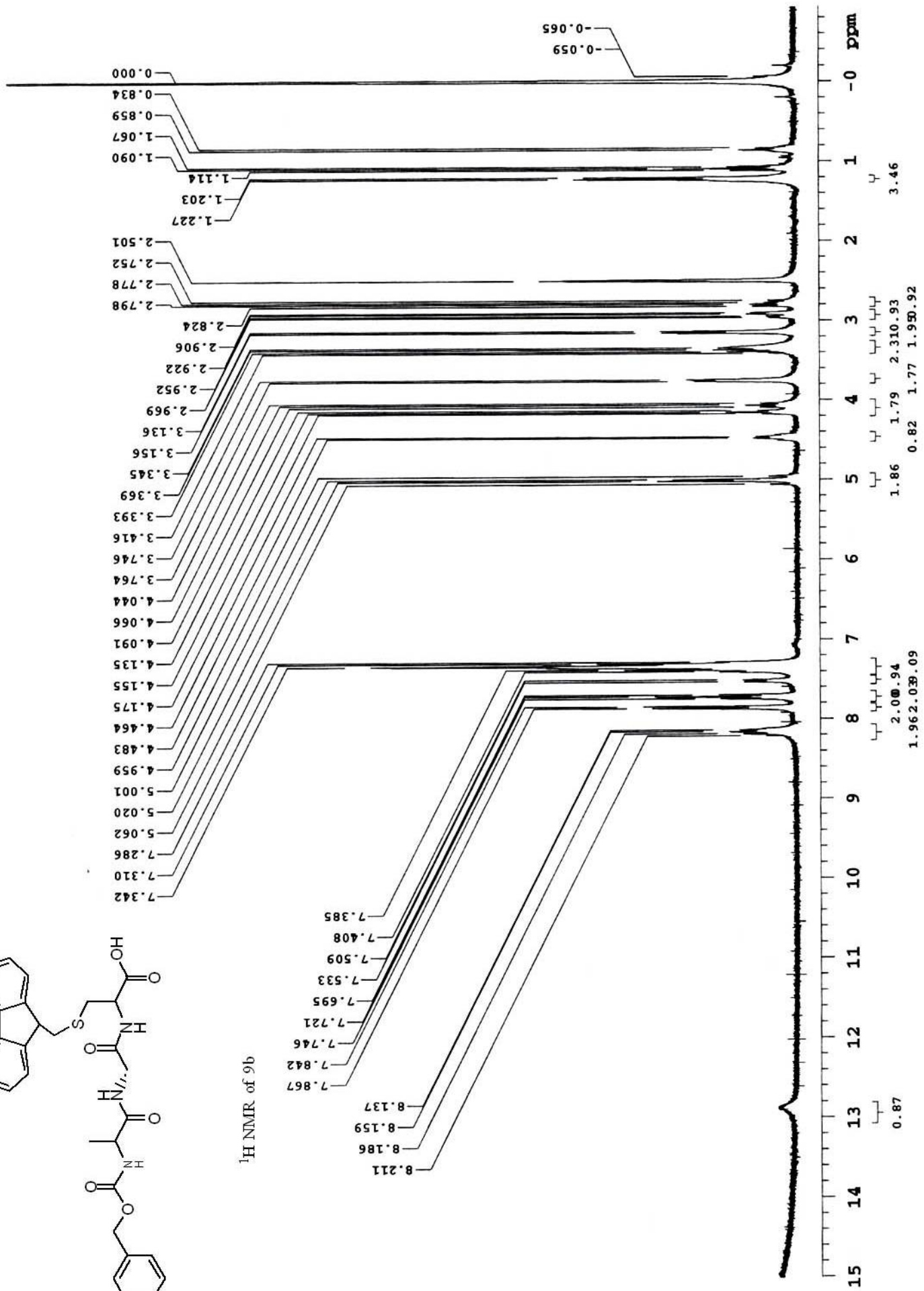


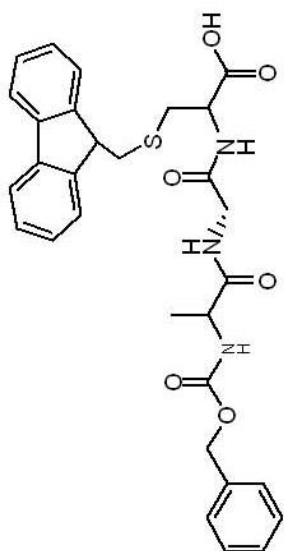
¹H NMR of 9a



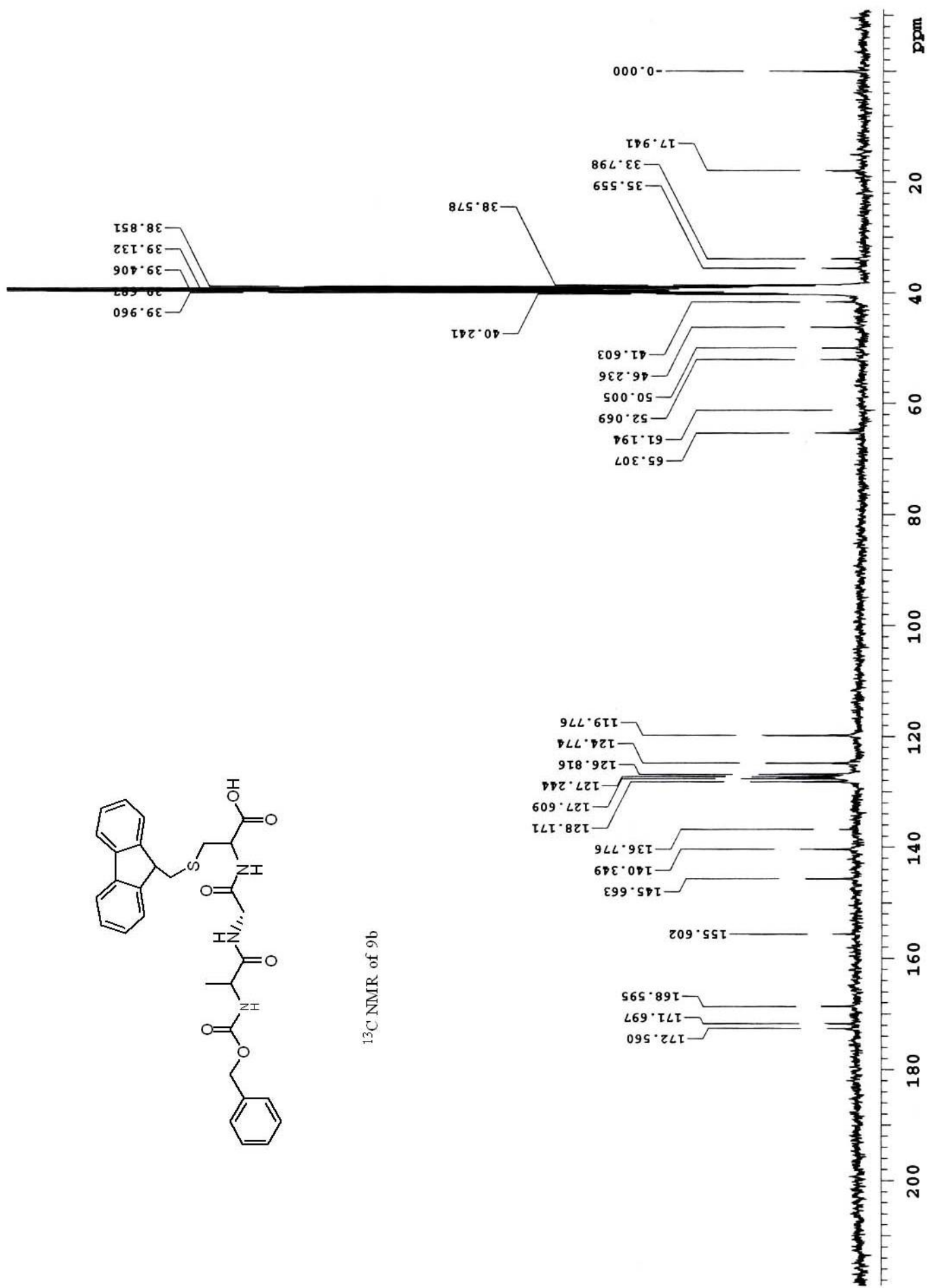


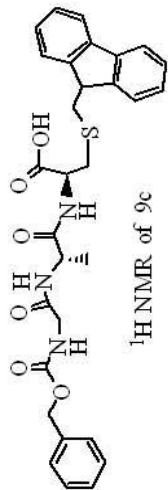
¹H NMR of 9b





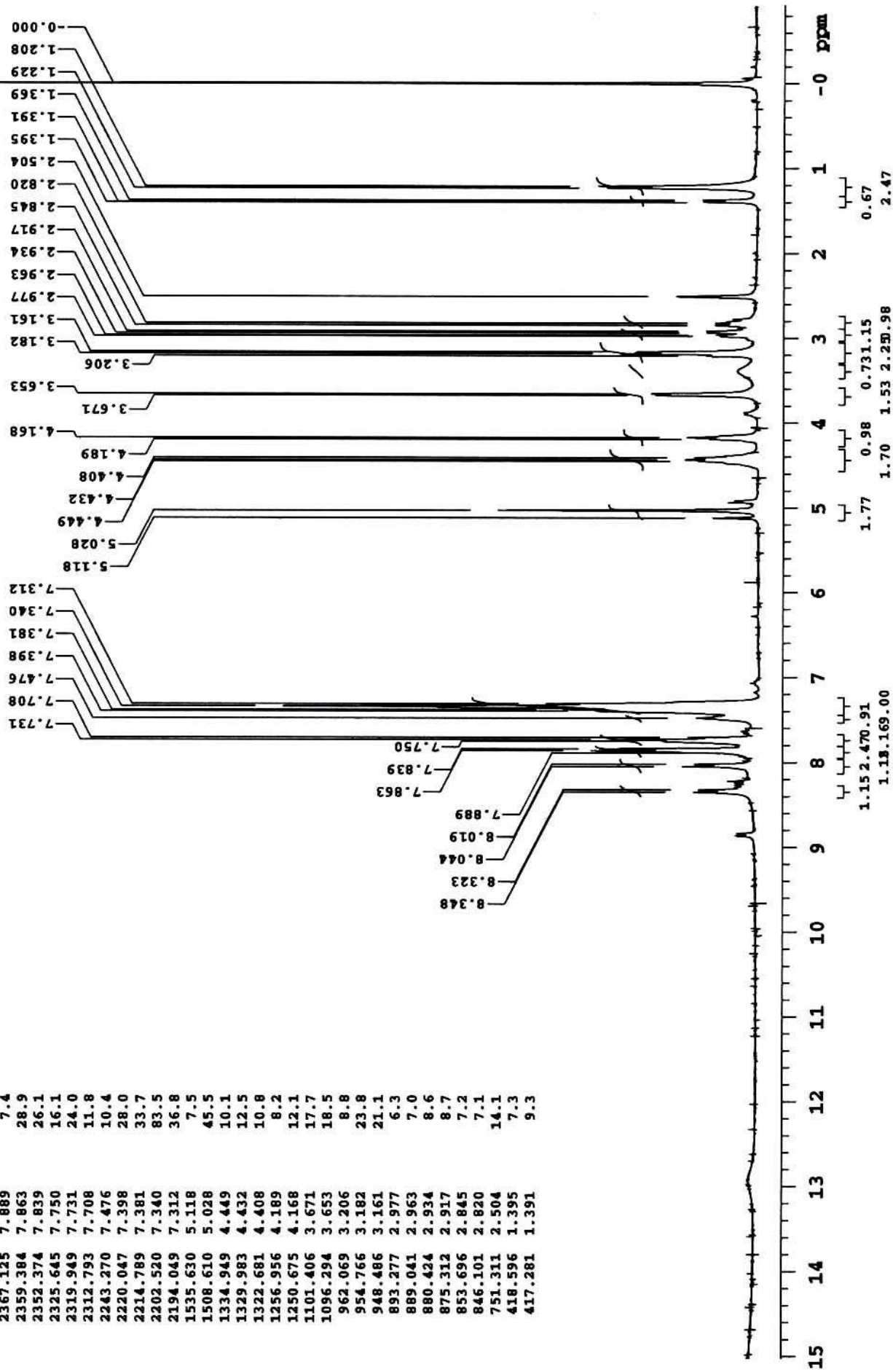
^{13}C NMR of 9b



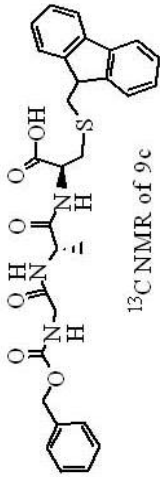


¹H NMR of 9c

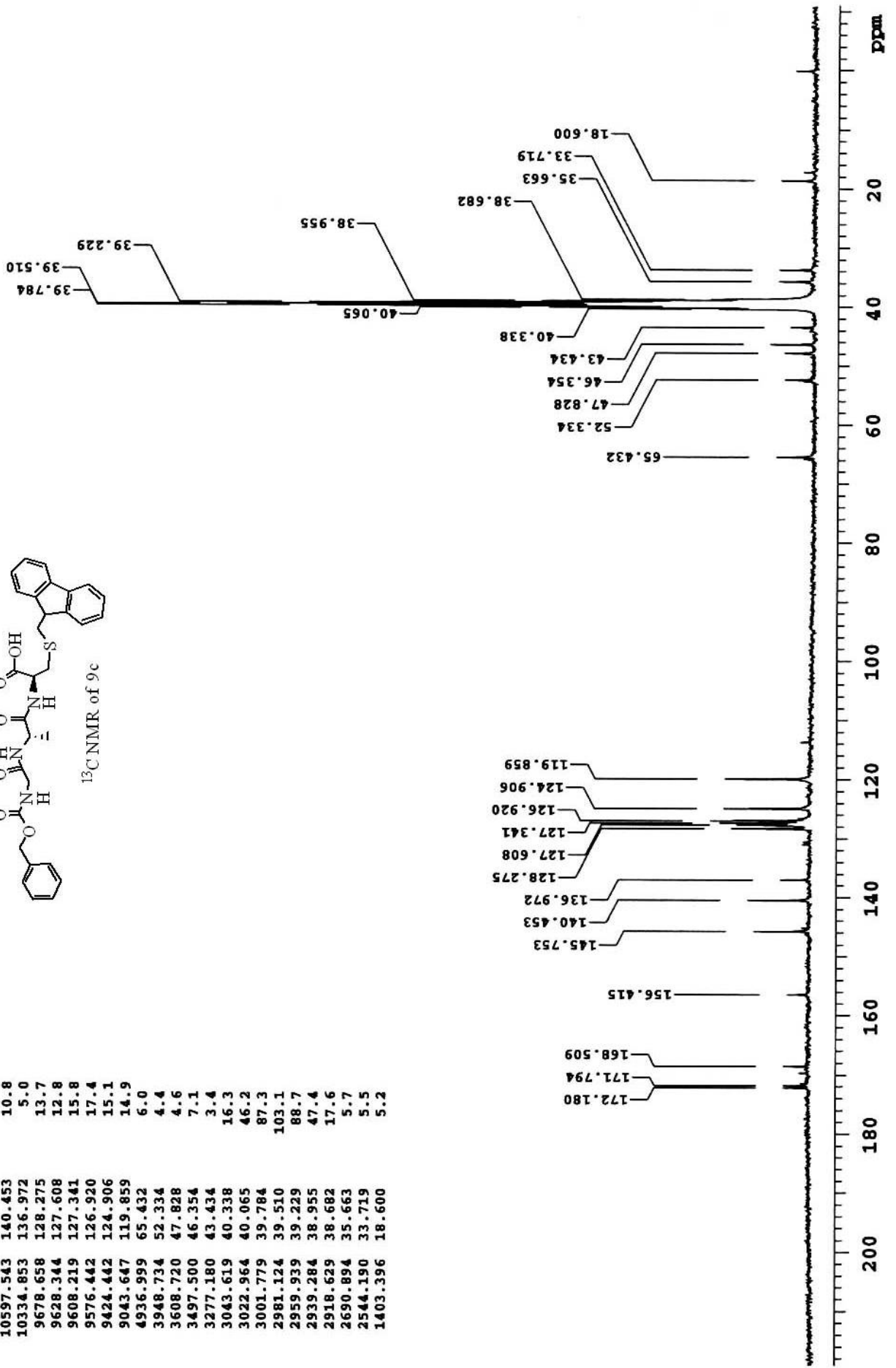
| INDEX | FREQUENCY PPM | HEIGHT | INDEX | FREQUENCY PPM | HEIGHT |
|-------|---------------|--------|-------|---------------|--------|
| 1 | 2505.002 | 8.348 | 37 | 410.855 | 1.369 |
| 2 | 2497.553 | 8.323 | 38 | 368.791 | 1.229 |
| 3 | 2413.863 | 8.044 | 39 | 362.364 | 1.208 |
| 4 | 2406.268 | 8.019 | 40 | -0.146 | -0.000 |
| 5 | 2367.125 | 7.889 | | | |
| 6 | 2359.384 | 7.863 | | | |
| 7 | 2352.374 | 7.839 | | | |
| 8 | 2325.645 | 7.750 | | | |
| 9 | 2319.949 | 7.731 | | | |
| 10 | 2312.793 | 7.708 | | | |
| 11 | 2243.270 | 7.476 | | | |
| 12 | 2220.047 | 7.398 | | | |
| 13 | 2214.789 | 7.381 | | | |
| 14 | 2202.520 | 7.340 | | | |
| 15 | 2194.049 | 7.312 | | | |
| 16 | 1535.630 | 5.118 | | | |
| 17 | 1508.610 | 5.028 | | | |
| 18 | 1334.949 | 4.449 | | | |
| 19 | 1329.983 | 4.432 | | | |
| 20 | 1322.681 | 4.408 | | | |
| 21 | 1256.956 | 4.189 | | | |
| 22 | 1250.675 | 4.168 | | | |
| 23 | 1101.406 | 3.671 | | | |
| 24 | 1096.294 | 3.653 | | | |
| 25 | 962.069 | 3.206 | | | |
| 26 | 954.766 | 3.182 | | | |
| 27 | 948.486 | 3.161 | | | |
| 28 | 892.277 | 2.977 | | | |
| 29 | 889.041 | 2.963 | | | |
| 30 | 880.424 | 2.934 | | | |
| 31 | 875.312 | 2.917 | | | |
| 32 | 853.696 | 2.845 | | | |
| 33 | 846.101 | 2.820 | | | |
| 34 | 751.311 | 2.504 | | | |
| 35 | 418.596 | 1.395 | | | |
| 36 | 417.281 | 1.391 | | | |

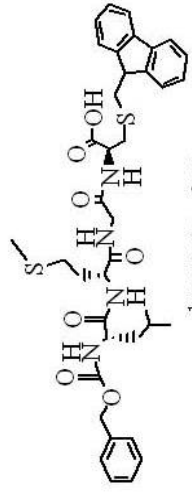


| INDEX | FREQUENCY PPM | HEIGHT |
|-------|---------------|--------|
| 1 | 12991.411 | 4.3 |
| 2 | 12962.282 | 4.3 |
| 3 | 12714.421 | 4.3 |
| 4 | 11801.892 | 3.7 |
| 5 | 10997.404 | 9.7 |
| 6 | 10597.543 | 10.8 |
| 7 | 10334.853 | 5.0 |
| 8 | 9678.658 | 13.7 |
| 9 | 9628.344 | 12.8 |
| 10 | 9608.219 | 15.8 |
| 11 | 9576.442 | 17.4 |
| 12 | 9424.442 | 15.1 |
| 13 | 9043.647 | 14.9 |
| 14 | 4936.999 | 6.0 |
| 15 | 3948.734 | 4.4 |
| 16 | 3608.720 | 4.6 |
| 17 | 3497.500 | 7.1 |
| 18 | 3277.180 | 3.4 |
| 19 | 3043.619 | 16.3 |
| 20 | 3022.964 | 46.2 |
| 21 | 3001.779 | 87.3 |
| 22 | 2981.124 | 103.1 |
| 23 | 2959.939 | 88.7 |
| 24 | 2939.284 | 47.4 |
| 25 | 2918.629 | 17.6 |
| 26 | 2690.894 | 5.7 |
| 27 | 2544.190 | 5.5 |
| 28 | 1403.396 | 5.2 |

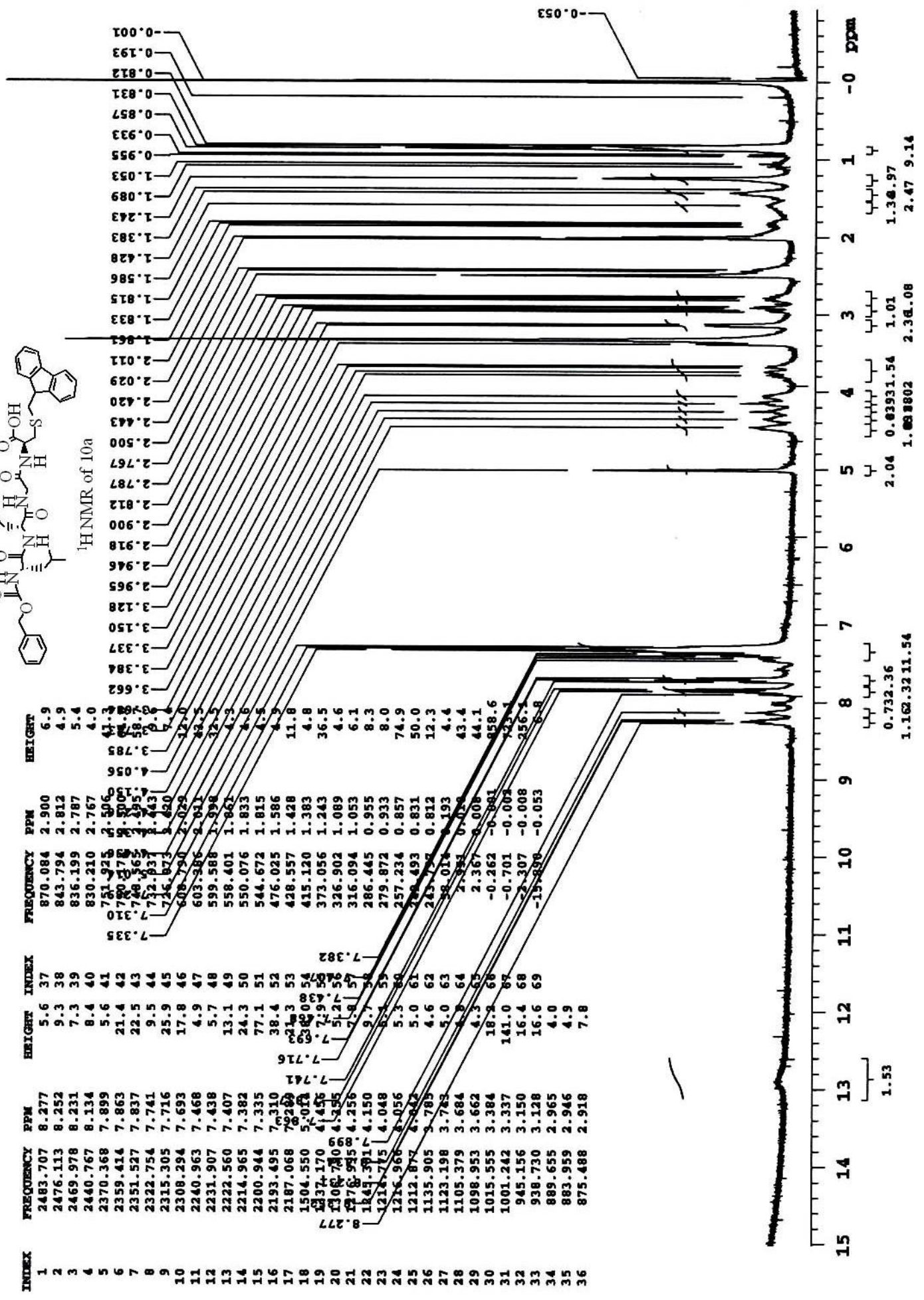


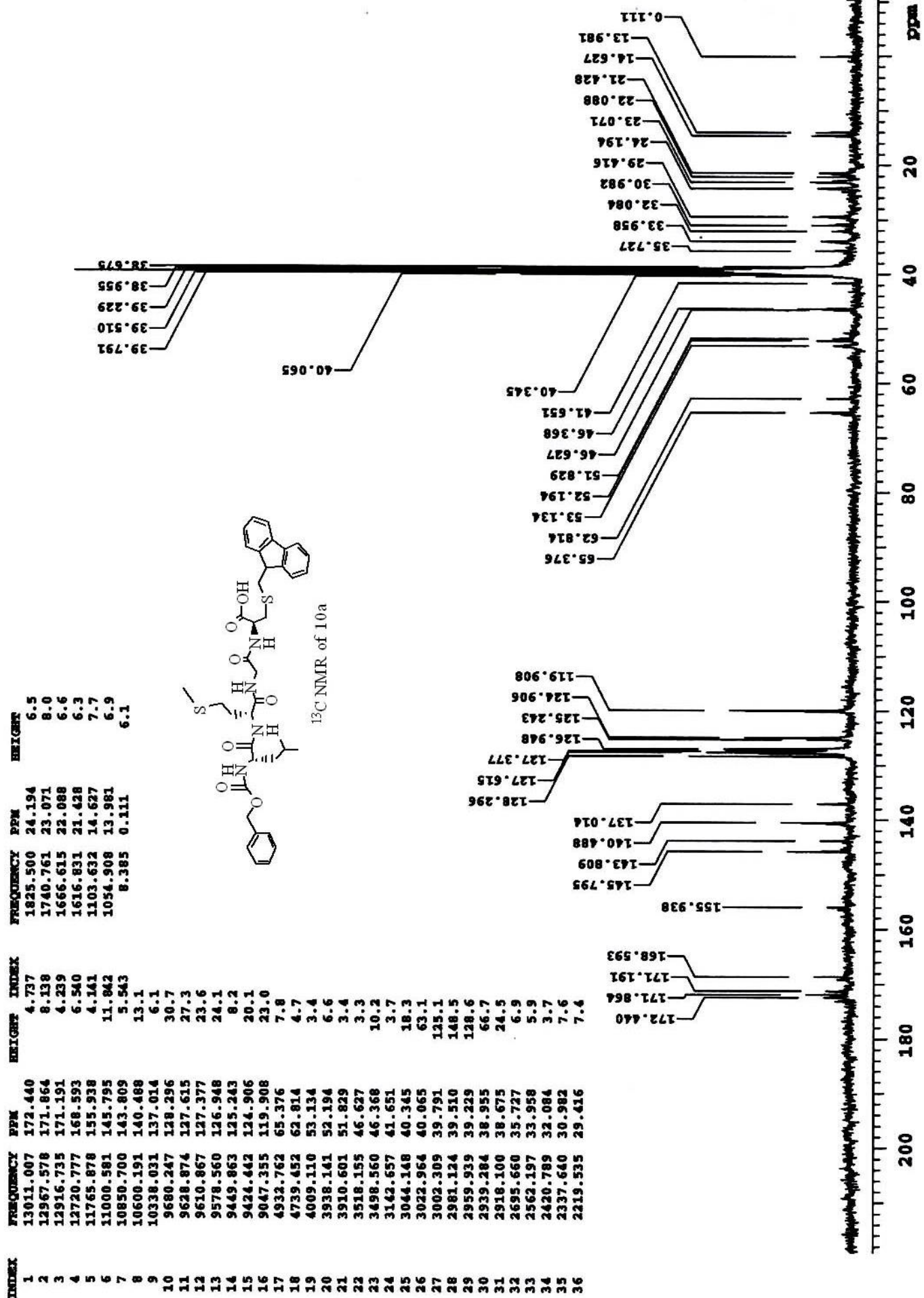
¹³C NMR of 9c

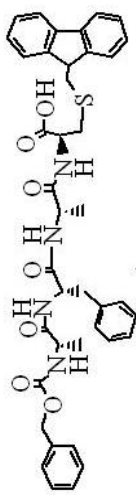




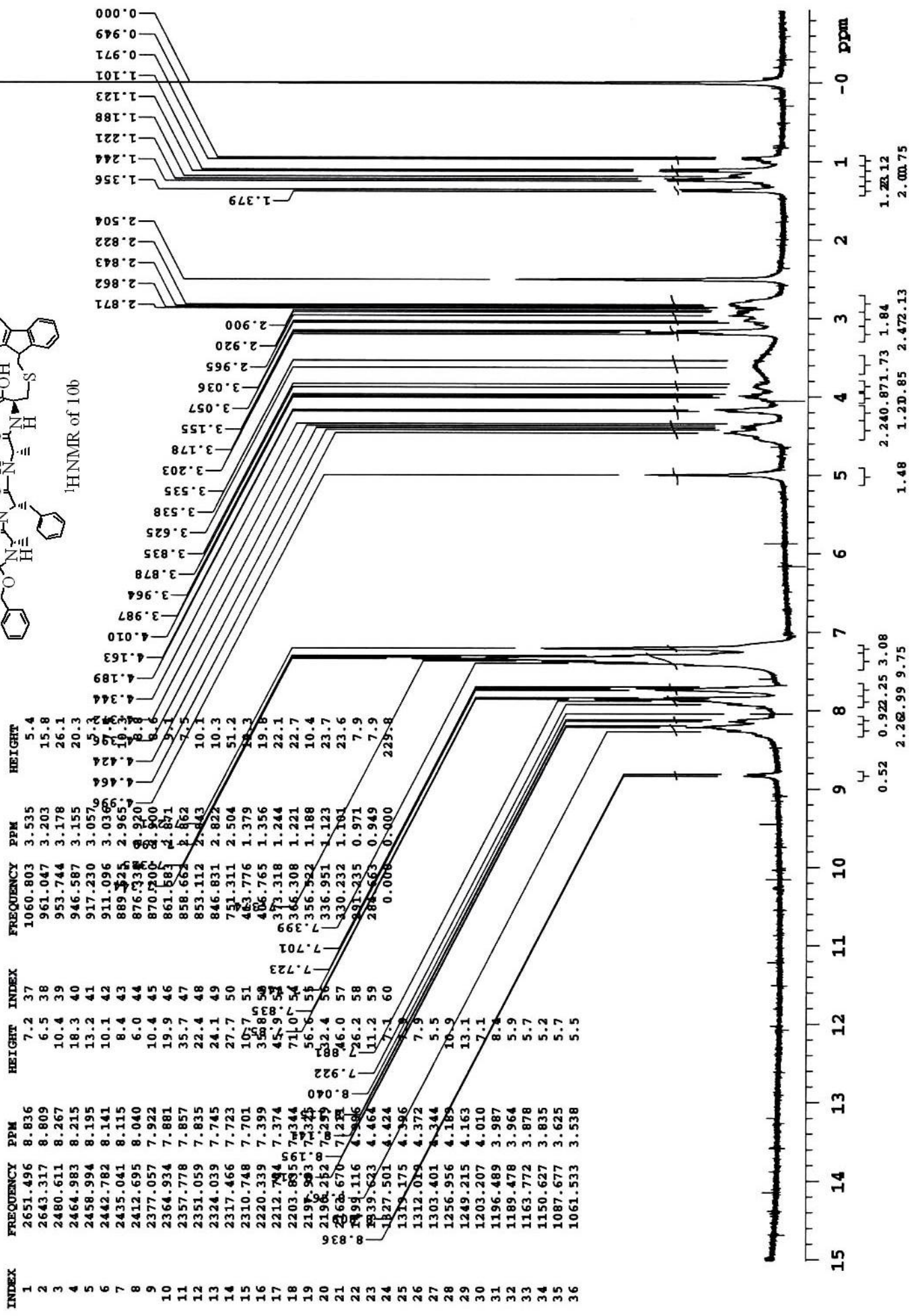
¹H NMR of 10a

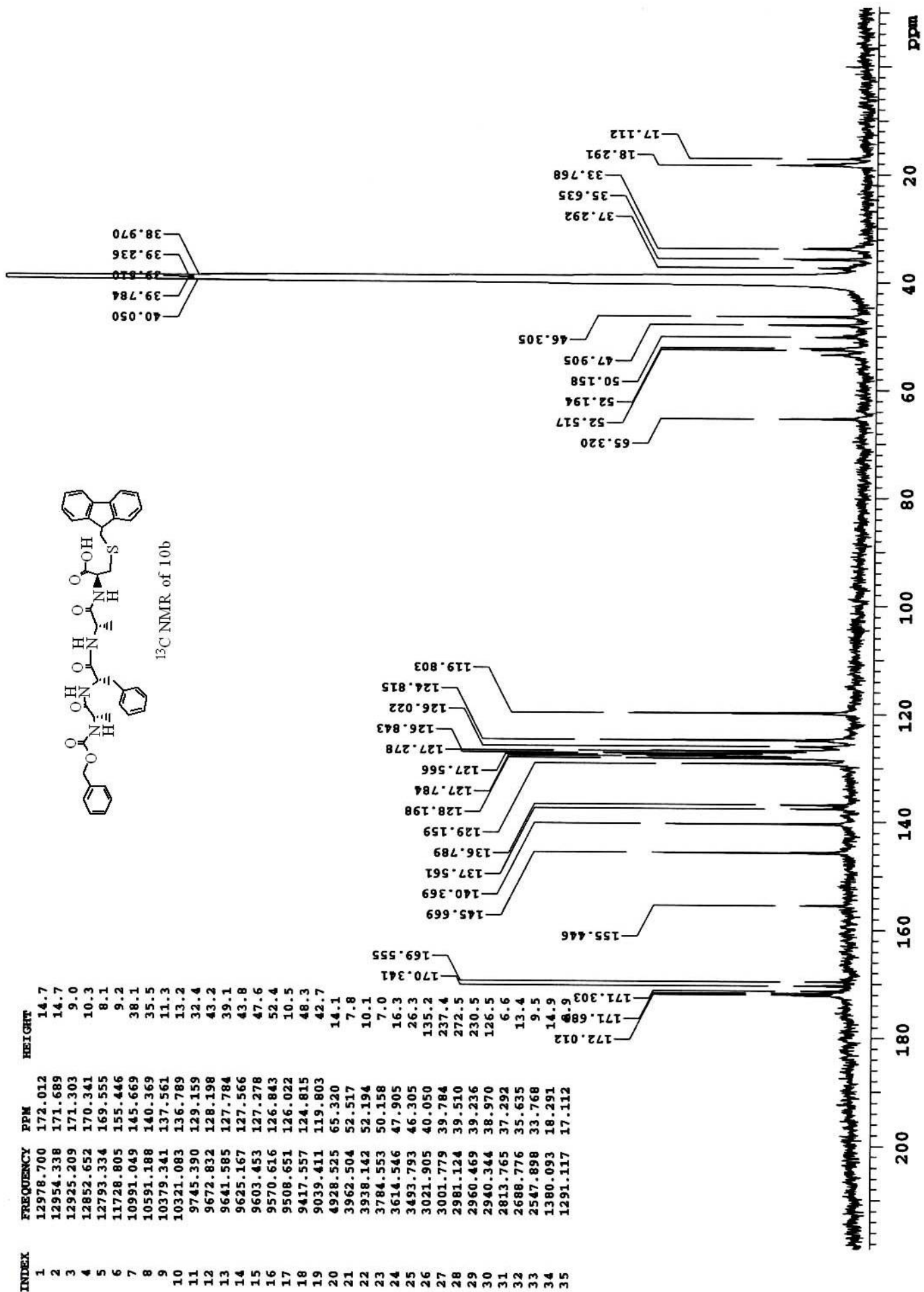


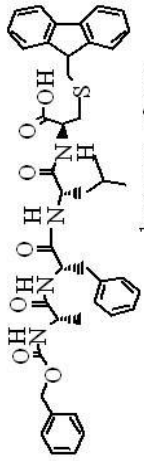




¹H NMR of 10b

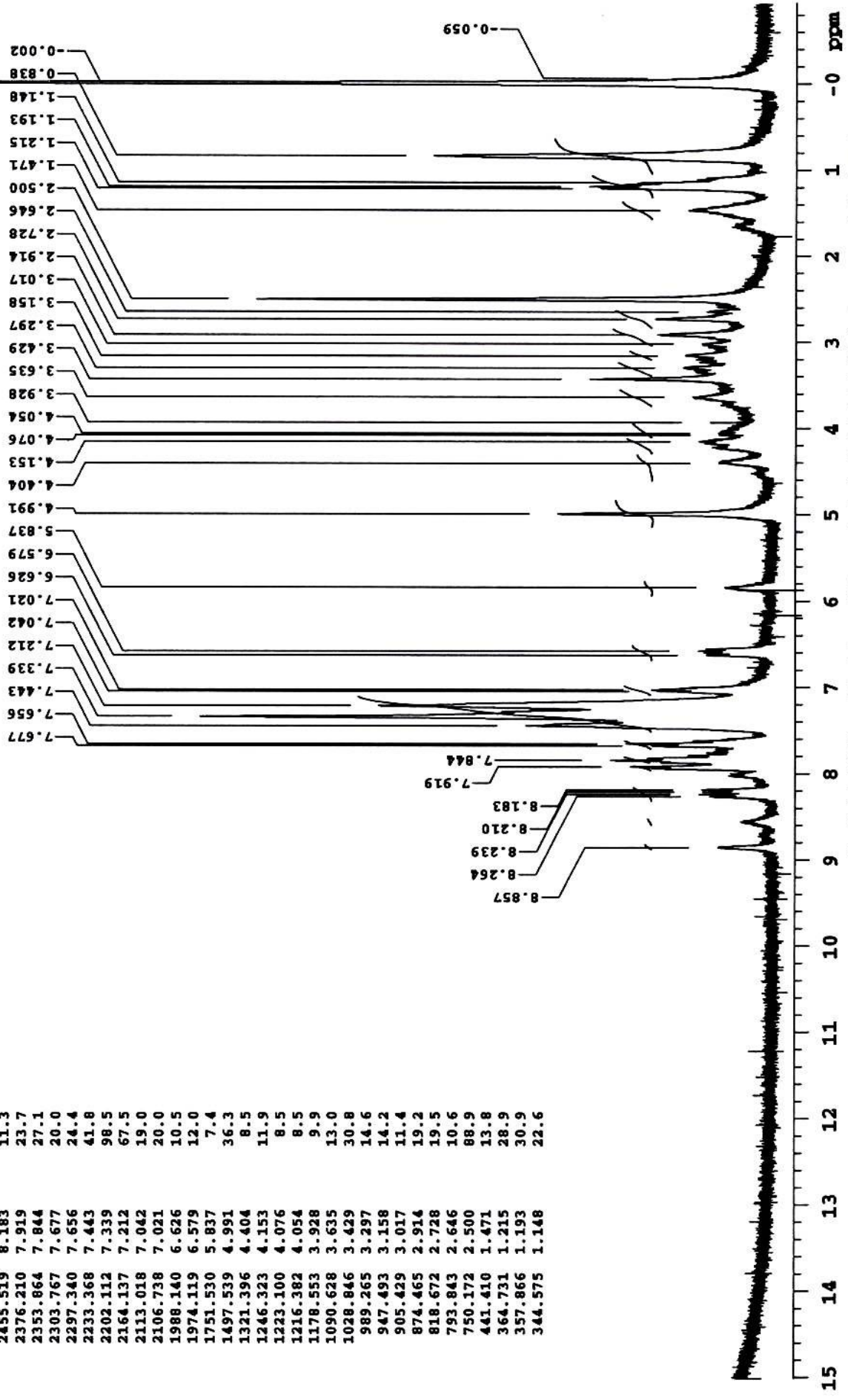




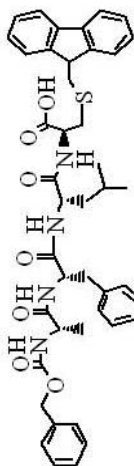


¹H NMR of 10c

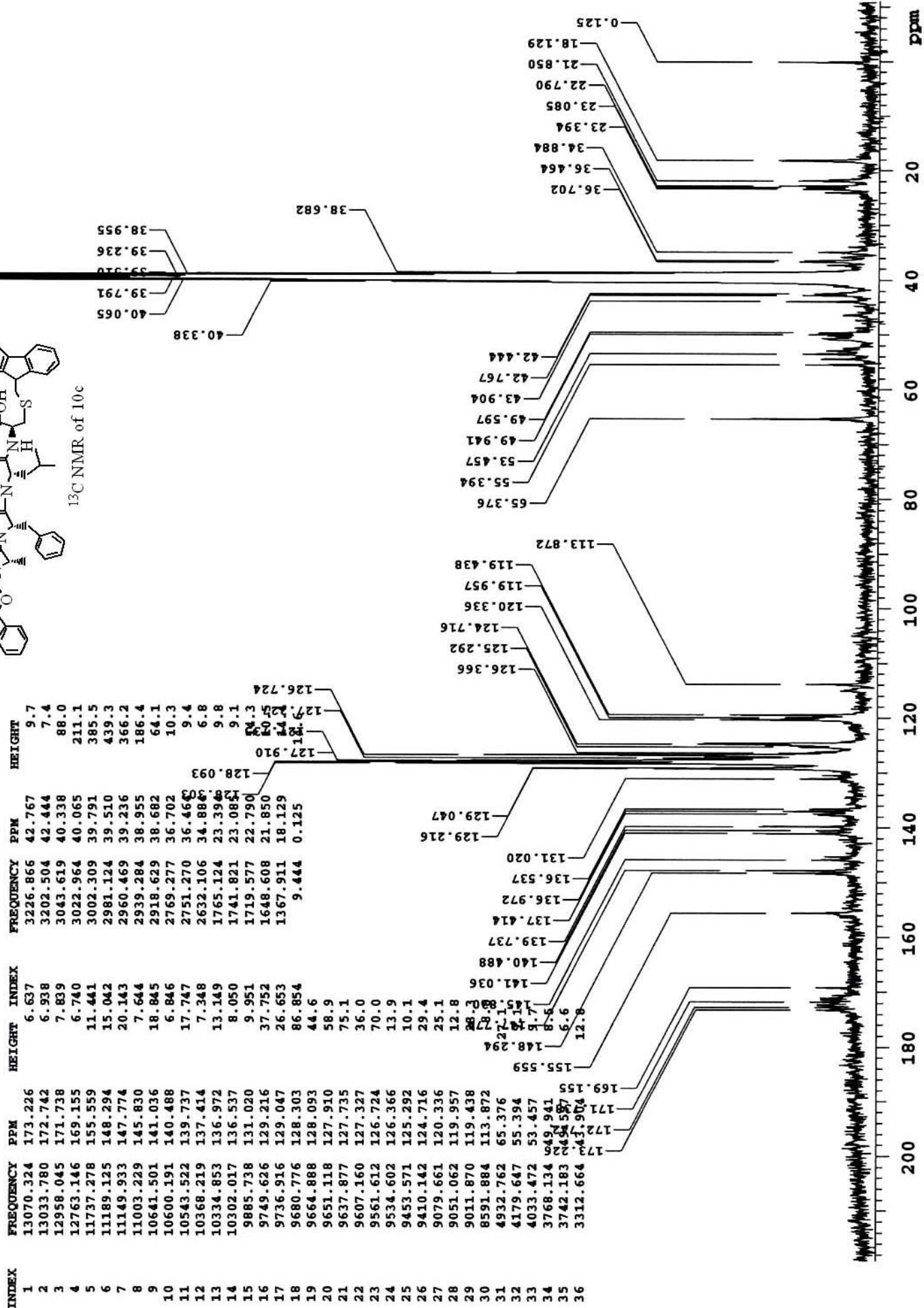
| INDEX | FREQUENCY PPM | HEIGHT | INDEX | FREQUENCY PPM | HEIGHT |
|-------|---------------|--------|-------|---------------|--------|
| 1 | 2657.806 | 8.857 | 8 | 2297.340 | 7.656 |
| 2 | 2479.910 | 8.264 | 9 | 2233.368 | 7.443 |
| 3 | 2472.315 | 8.239 | 10 | 2202.112 | 7.339 |
| 4 | 2463.552 | 8.210 | 11 | 2164.137 | 7.212 |
| 5 | 2455.519 | 8.183 | 12 | 2113.018 | 7.042 |
| 6 | 2376.210 | 7.919 | 13 | 2106.738 | 7.021 |
| 7 | 2353.864 | 7.844 | 14 | 1988.140 | 6.626 |
| 8 | 2303.767 | 7.677 | 15 | 1974.119 | 6.579 |
| 9 | 2297.340 | 7.656 | 16 | 1751.530 | 5.837 |
| 10 | 2233.368 | 7.443 | 17 | 1497.539 | 4.991 |
| 11 | 2202.112 | 7.339 | 18 | 1321.396 | 4.404 |
| 12 | 2164.137 | 7.212 | 19 | 1246.323 | 4.153 |
| 13 | 2113.018 | 7.042 | 20 | 1223.100 | 4.076 |
| 14 | 2106.738 | 7.021 | 21 | 1216.382 | 4.054 |
| 15 | 1988.140 | 6.626 | 22 | 1178.553 | 3.928 |
| 16 | 1974.119 | 6.579 | 23 | 1090.628 | 3.635 |
| 17 | 1751.530 | 5.837 | 24 | 1028.846 | 3.429 |
| 18 | 1497.539 | 4.991 | 25 | 989.265 | 3.297 |
| 19 | 1321.396 | 4.404 | 26 | 947.493 | 3.158 |
| 20 | 1246.323 | 4.153 | 27 | 905.429 | 3.017 |
| 21 | 1223.100 | 4.076 | 28 | 874.465 | 2.914 |
| 22 | 1216.382 | 4.054 | 29 | 818.672 | 2.728 |
| 23 | 1178.553 | 3.928 | 30 | 793.843 | 2.646 |
| 24 | 1090.628 | 3.635 | 31 | 750.172 | 2.500 |
| 25 | 1028.846 | 3.429 | 32 | 441.410 | 1.471 |
| 26 | 989.265 | 3.297 | 33 | 364.731 | 1.215 |
| 27 | 947.493 | 3.158 | 34 | 357.866 | 1.193 |
| 28 | 905.429 | 3.017 | 35 | 344.575 | 1.148 |
| 29 | 874.465 | 2.914 | | | |
| 30 | 818.672 | 2.728 | | | |
| 31 | 793.843 | 2.646 | | | |
| 32 | 750.172 | 2.500 | | | |
| 33 | 441.410 | 1.471 | | | |
| 34 | 364.731 | 1.215 | | | |
| 35 | 357.866 | 1.193 | | | |
| 36 | 344.575 | 1.148 | | | |

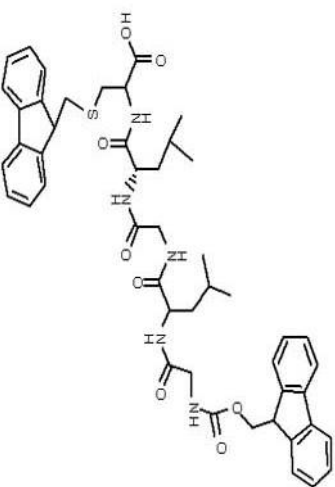


| HEIGHT | INDEX | FREQUENCY PPM | HEIGHT |
|--------|-------|---------------|--------|
| 58.1 | 37 | 251.392 | 0.838 |
| 343.7 | 38 | -0.701 | -0.002 |
| 16.1 | 39 | -17.789 | -0.059 |
| 0.35 | 35 | 357.866 | 1.193 |
| 0.98 | 34 | 364.731 | 1.215 |
| 1.06 | 33 | 441.410 | 1.471 |
| 0.37 | 32 | 750.172 | 2.500 |
| 2.00 | 31 | 818.672 | 2.728 |
| 0.84 | 30 | 874.465 | 2.914 |
| 1.81 | 29 | 905.429 | 3.017 |
| 1.99 | 28 | 947.493 | 3.158 |
| 1.99 | 27 | 989.265 | 3.297 |
| 1.81 | 26 | 1028.846 | 3.429 |
| 1.31 | 25 | 1090.628 | 3.635 |
| 1.81 | 24 | 1178.553 | 3.928 |
| 1.81 | 23 | 1216.382 | 4.054 |
| 1.81 | 22 | 1223.100 | 4.076 |
| 1.81 | 21 | 1246.323 | 4.153 |
| 1.81 | 20 | 1321.396 | 4.404 |
| 1.81 | 19 | 1497.539 | 4.991 |
| 1.81 | 18 | 1751.530 | 5.837 |
| 1.81 | 17 | 1974.119 | 6.579 |
| 1.81 | 16 | 1988.140 | 6.626 |
| 1.81 | 15 | 2106.738 | 7.021 |
| 1.81 | 14 | 2113.018 | 7.042 |
| 1.81 | 13 | 2164.137 | 7.212 |
| 1.81 | 12 | 2202.112 | 7.339 |
| 1.81 | 11 | 2233.368 | 7.443 |
| 1.81 | 10 | 2297.340 | 7.656 |
| 1.81 | 9 | 2455.519 | 8.183 |
| 1.81 | 8 | 2472.315 | 8.239 |
| 1.81 | 7 | 2479.910 | 8.264 |
| 1.81 | 6 | 2657.806 | 8.857 |

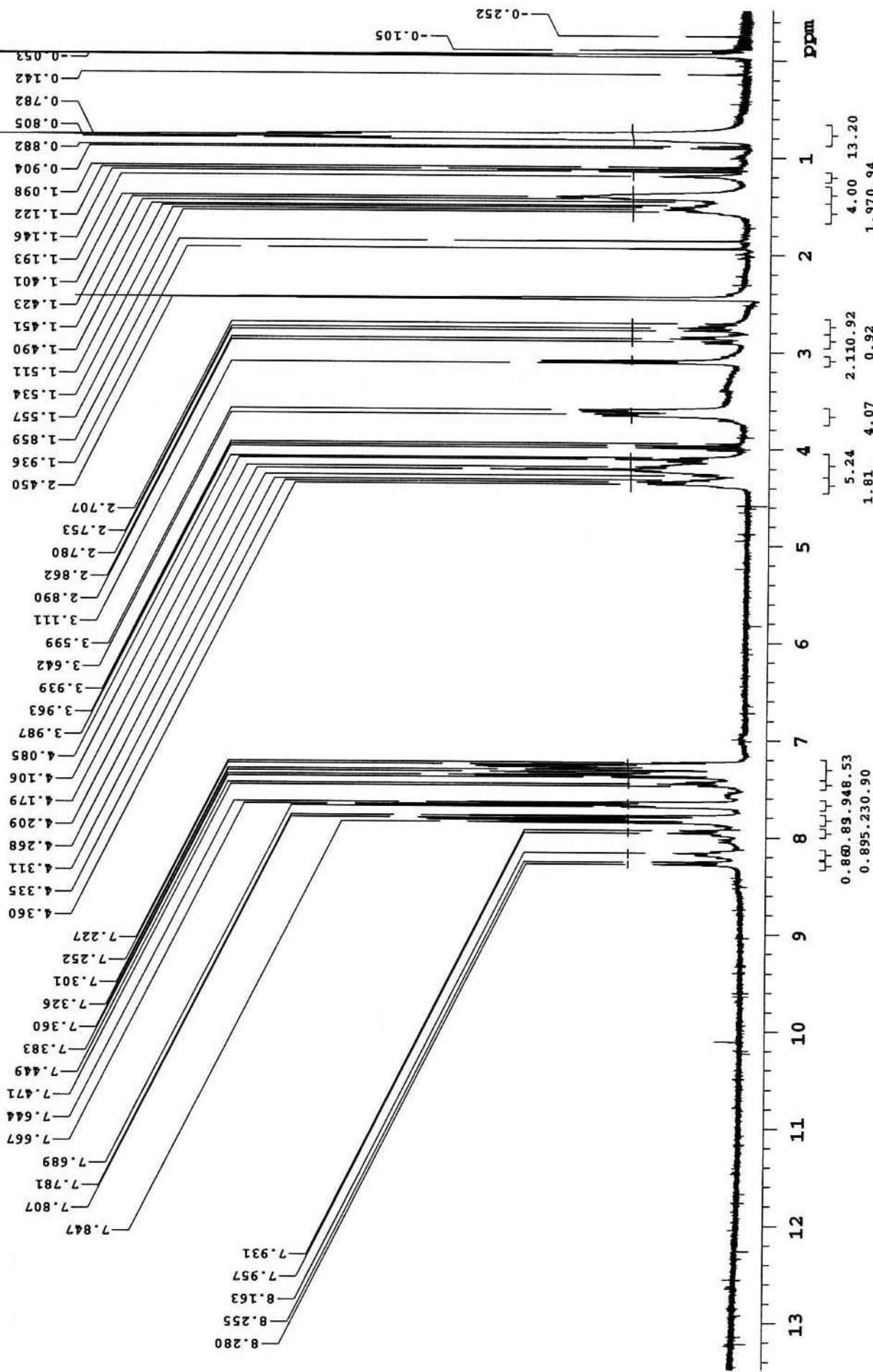


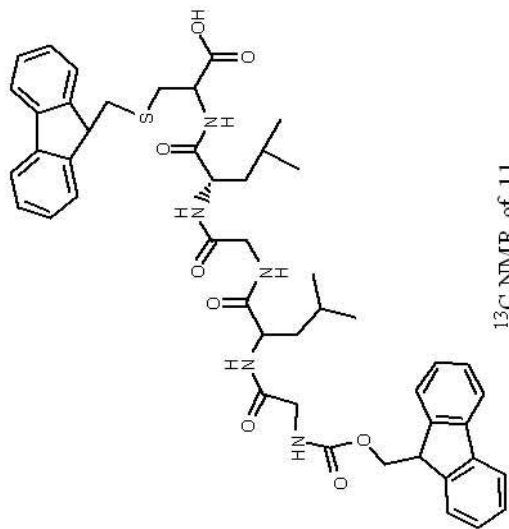
¹³C NMR of 10c





¹H NMR of 11





¹³C NMR of 11

