Supporting Material

Facile *N*-Derivatization of α-Amino Esters and Amides *via* Benzotriazolylmethyl Derivatives

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Methyl (2*S*)-2-[(1*H*-1,2,3-benzotriazol-1-ylmethyl)amino]-3-phenylpropanoate (2a). White needles (from $CHCl_3$/Hexanes); yield 81%; mp 97– 98 °C (lit.[12] 96– 97 °C); $[\alpha]^{25}_D$ = +25 (*c* 2.17, EtOH) (lit.[12] $[\alpha]^{25}_D$ = +18 (*c* 2.17, EtOH)); ^{1}H NMR δ 2.62–2.73 (m, 1H), 2.79 (dd, *J* = 13.7, 8.2 Hz, 1H), 2.97 (dd, *J* = 13.7, 5.2 Hz, 1H), 3.47 (s, 3H), 3.60–3.70 (m, 1H), 5.40–5.62 (m, 2H), 6.80–7.05 (m, 2H), 7.12–7.22 (m, 3H), 7.31–7.46 (m, 3H), 8.04 (d, *J* = 8.2 Hz, 1H); ^{13}C NMR δ 39.2, 52.0, 59.4, 61.5, 109.4, 119.9, 123.9, 126.8, 127.4, 128.4, 129.0, 132.5, 136.4, 146.1, 173.7.

Methyl (2*S*)-3-phenyl-2-{[(phenylsulfanyl)methyl]amino}propanoate (3a). Colorless oil; yield 85% (according to crude NMR); ^{1}H NMR δ 2.03 (br s, 1H), 2.81 (dd, *J* = 13.6, 8.8 Hz, 1H), 3.06 (dd, *J* = 13.6, 5.1 Hz, 1H), 3.69 (s, 3H), 3.97 (dd, *J* = 8.8, 5.1 Hz, 1H), 4.22 (d, *J* = 0.7 Hz, 2H), 7.10–7.29 (m, 10H); ^{13}C NMR δ 39.0, 52.0, 56.0, 59.2, 126.8, 127.0, 128.5, 128.9, 128.9, 129.1, 132.0, 136.8, 173.9.

Methyl (2*S*)-2-{[(diethoxyphosphoryl)methyl]amino}-3-phenylpropanoate (3b). Colorless oil; yield 62%; $[\alpha]^{25}_D$ = -3 (*c* 1.54, $CHCl_3$); ^{1}H NMR δ 1.27 (t, *J* = 7.0 Hz, 6H), 1.75 (brs, 1H), 2.72–3.12 (m, 4H), 3.58–3.66 (m, 1H), 3.67 (s, 3H), 3.98–4.14 (m, 4H), 7.15–7.35 (m, 5H).; ^{13}C NMR δ 16.3 (d, *J* = 5.7 Hz, $(CH_3CH_2O)_2$), 39.2 ($PhCH_2$), 43.3 (d, *J* = 158.6 Hz, $(CH_3CH_2O)_2PCH_2$), 51.7 (CH_3O), 62.2 (d, *J* = 6.8 Hz, $(CH_3CH_2O)_2P$), 63.4 (d, *J* = 15.5 Hz, CHNH), 126.6, 128.3, 129.1, 136.9, 174.0. HRMS Calcd for $C_{15}H_{24}NO_5P$: 330.1472; Found: 330.1472.

Methyl (2*S*)-2-(3-butenylamino)-3-phenylpropanoate (3c). Colorless oil; yield 68%; $[\alpha]^{25}_D$ = +20 (*c* 1.54, $CHCl_3$); ^{1}H NMR δ 1.56 (br s, 1H), 2.14–2.24 (m, 2H), 2.46–2.60 (m, 1H), 2.60–2.71 (m, 1H), 2.94 (d, *J* = 6.9 Hz, 2H), 3.52 (t, *J* = 7.0 Hz, 1H), 3.63 (s,

3H), 4.94–5.07 (m, 2H), 5.62–5.80 (m, 1H), 7.11–7.32 (m, 5H); ^{13}C NMR δ 34.2, 39.6, 47.1, 51.5, 63.0, 116.3, 126.7, 128.4, 129.1, 135.9, 137.2, 174.9. Anal. Calcd for $C_{14}H_{19}NO_2$: C, 72.07; H, 8.21; N, 6.00. Found: C, 71.98; H, 8.14; N, 6.35.

Methyl (2*S*)-2-(3-butenylamino)-3-phenylpropanoate (3c'). Colorless oil; yield 68%; $[\alpha]^{25}_D$ = -20 (c 1.54, CHCl3); ^{1}H NMR δ 1.57 (br s, 1H), 2.15-2.23 (m, 2H), 2.48-2.57 (m, 1H), 2.61-2.70 (m, 1H), 2.94 (d, J = 7.0 Hz, 2H), 3.52 (t, J = 7.0 Hz, 1H), 3.63 (s, 3H), 4.96-5.06 (m, 2H), 5.64-5.79 (m, 1H), 7.15-7.31 (m, 5H). ^{13}C NMR δ 34.2, 39.6, 47.0, 51.5, 63.0, 116.3, 126.6, 128.3, 129.1, 135.9, 137.2, 174.9. Anal. Calcd for $C_{14}H_{19}NO_2$: C, 72.07; H, 8.21; N, 6.00. Found: C, 704.98; H, 8.44; N, 6.36.

Methyl-3-{[(1*S*)-1-benzyl-2-methoxy-2-oxoethyl]amino}-2,2-dimethylpropanoate (3d). Colorless oil; yield 75%; $[\alpha]^{25}_D$ = +10 (*c* 1.54, $CHCl_3$) ^{1}H NMR δ 1.12 (s, 3H), 1.13 (s, 3H), 1.62 (br s, 1H), 2.46 (d, *J* = 11.8 Hz, 1H), 2.73 (d, *J* = 11.8 Hz, 1H), 2.84–2.92 (m, 2H), 3.43 (t, *J* = 7.0 Hz, 1H), 3.58 (s, 3H), 3.65 (s, 3H), 7.10–7.38 (m, 5H); ^{13}C NMR δ 23.0, 23.5, 39.6, 43.6, 51.5, 51.6, 56.8, 63.8, 126.5, 128.2, 129.2, 137.6, 175.0, 177.5. Anal. Calcd for $C_{16}H_{23}NO_4$: C, 65.51; H, 7.90; N, 4.77. Found: C, 65.62; H, 8.28; N, 5.03.

Methyl (2*S*)-2-[(cyanomethyl)amino]-3-phenylpropanoate (3e). Colorless oil; yield 98%; $[\alpha]^{28}_D$ = +7.4 (*c* 0.82, $CHCl_3$) (lit. ref.[14]); ^{1}H NMR δ 1.86 (br s, 1H), 2.91 (dd, *J* = 13.7, 7.8 Hz, 1H), 3.09 (dd, *J* = 13.7, 5.4 Hz, 1H), 3.50–3.54 (m, 2H), 3.62–3.72 (m, 1H), 3.73 (s, 3H), 7.15–7.35 (m, 5H); ^{13}C NMR δ 35.9, 39.1, 52.1, 61.0, 117.2, 127.1, 128.6, 129.1, 136.1, 173.2. Anal. Calcd for $C_{12}H_{14}N_2O_2$: C, 66.04; H, 6.47. Found: C, 66.08; H, 6.76.

Methyl (2*S*)-2-(3-butenylamino)-2-phenylethanoate (3f). Colorless oil; yield 42%; $[\alpha]^{25}_{D}$ = +71 (*c* 1.54, $CHCl_3$); ^{1}H NMR δ 1.99 (br s, 1H), 2.23–2.32 (m, 2H), 2.54–2.69 (m, 2H), 3.69 (s, 3H), 4.38 (s, 1H), 5.01–5.13 (m, 2H), 5.70–5.88 (m, 1H), 7.20–7.45 (m, 5H); ^{13}C NMR δ 34.1, 46.7, 52.2, 65.4, 116.5, 127.4, 128.1, 128.7, 136.0, 138.1, 173.4. Anal. Calcd for $C_{13}H_{17}NO_2$: C, 71.21; H, 7.81; N, 6.39. Found: C, 71.46; H, 8.11; N, 6.41.

Methyl 3-{[(1*S*)-2-methoxy-2-oxo-1-phenylethyl]amino}-2,2-dimethylpropanoate (3g). Colorless oil; yield 51%; $[\alpha]^{25}_{D}$ = +35 (*c* 1.54, $CHCl_3$); (lit. ref.[15]); ^{1}H NMR δ 1.18 (s, 3H), 1.19 (s, 3H), 2.18 (br s, 1H), 2.50 (d, *J* = 11.7 Hz, 1H), 2.68 (d, *J* = 11.7 Hz, 1H), 3.66 (s, 3H), 3.68 (s, 3H), 4.34 (s, 1H), 7.20–7.42 (m, 5H); ^{13}C NMR δ 23.5, 23.6, 43.5, 51.7, 52.1, 56.1, 66.0, 127.4, 127.9, 128.5, 138.2, 173.3, 177.6. Anal. Calcd for $C_{15}H_{21}NO_4$: C, 64.50; H, 7.58; N, 5.01. Found: C, 64.78; H, 7.88; N, 5.22.

Methyl (2*S*)-2-[(3-oxo-3-phenylpropyl)amino]-2-phenylethanoate (3h). Yellow microcrystals; yield 40%, mp 90– 91 °C; $[\alpha]^{25}_{D}$ = +47 (*c* 1.54, $CHCl_3$); ^{1}H NMR δ 2.60 (br s, 1H), 2.85–3.05 (m, 2H), 3.21 (t, *J* = 6.3 Hz, 2H), 3.68 (s, 3H), 4.44 (s, 1H), 7.25–7.38 (m, 3H), 7.39–7.48 (m, 4H), 7.50–7.59 (m, 1H), 7.93 (d, *J* = 7.3 Hz, 2H); ^{13}C NMR δ 38.8, 42.4, 52.2, 65.8, 127.4, 127.9, 128.1, 128.5, 128.6, 133.1, 136.7, 137.9, 173.1, 199.1. Anal. Calcd for $C_{18}H_{19}NO_3$: C, 72.71; H, 6.44. Found: C, 72.71; H, 6.44.

Methyl (2*S*)-2-[(cyanomethyl)amino]-2-phenylethanoate (3i). Colorless oil; yield 62%; $[\alpha]^{25}_{D}$ = +1 (*c* 1.54, $CHCl_3$); ^{1}H NMR δ 2.50 (br s, 1H), 3.36 (dd, *J* = 17.5, 8.8 Hz, 1H), 3.62 (d, *J* = 4.7 Hz, 1H), 3.69 (s, 3H), 4.56 (d, *J* = 2.6 Hz, 1H), 7.27–7.44 (m, 5H);

^{13}C NMR δ 34.7, 52.6, 63.5, 117.0, 127.9, 128.8, 128.9, 135.8, 171.8. Anal. Calcd for $C_{11}H_{12}N_2O_2$: C, 64.69; H, 5.92; N, 13.72. Found: C, 64.73; H, 6.25; N, 14.03.

Methyl (2*S*)-2-[(3-oxo-3-phenylpropyl)amino]propanoate (3j). Colorless oil; yield 55%; $[\alpha]^{25}_D$ = +20 (*c* 1.54, $CHCl_3$); (lit. ref.[16]); ^{1}H NMR δ 1.32 (d, *J* = 7.0 Hz, 3H), 1.98 (br s, 1H), 2.84–2.98 (m, 1H), 2.99–3.10 (m, 1H), 3.14–3.24 (m, 2H), 3.41 (q, *J* = 7.0 Hz, 1H), 3.73 (s, 3H), 7.41–7.50 (m, 2H), 7.52–7.60 (m, 1H), 7.95 (d, *J* = 7.1 Hz, 2H); ^{13}C NMR δ 19.0, 39.0, 42.7, 51.8, 56.9, 127.9, 128.6, 133.1, 136.8, 175.9, 199.1. Anal. Calcd for $C_{13}H_{17}NO_3$: N, 5.95. Found: N, 5.82.

(2*S*)-2-[(1*H*-Benzotriazol-1-ylmethyl)amino]-*N*-(4-methylphenyl)-3-phenylpropanamide (7a). White microcrystals (from EtOAc); yield 80%; mp 143–144 °C; $[\alpha]^{25}_D$ = -110 (*c* 1.54, $CHCl_3$); ^{1}H NMR δ 2.32 (s, 3H), 2.64–2.75 (m, 1H), 2.79 (dd, *J* = 14.0 Hz, 8.9 Hz, 1H), 3.01 (dd, *J* = 14.0 Hz, 4.8 Hz, 1H), 3.55–3.68 (m, 1H), 5.35–5.57 (m, 2H), 6.83–6.92 (m, 2H), 7.01–7.19 (m, 5H), 7.30–7.50 (m, 5H), 8.02 (d, *J* = 8.7 Hz, 1H), 8.71 (br s, 1H); ^{13}C NMR δ 20.9, 39.0, 60.9, 61.3, 108.7, 119.6, 120.1, 124.1, 127.1, 127.9, 128.7, 128.7, 129.4, 132.5, 134.1, 134.6, 135.9, 146.0, 170.2. Anal. Calcd for $C_{23}H_{23}N_5O$: C, 71.67; H, 6.01; N, 18.17. Found: C, 71.78; H, 6.28; N, 18.15.

(2*S*)-2-[(1*H*-Benzotriazol-1-ylmethyl)amino]-*N*-benzylpropanamide (7b). White microcrystals (from $CHCl_3$/Et_2O); yield 86%; mp 111–112 °C; $[\alpha]^{25}_D$ = -37 (*c* 1.54, $CHCl_3$); ^{1}H NMR δ 1.22 (d, *J* = 7.0 Hz, 3H), 2.50–2.65 (m, 1H), 3.22–3.40 (m, 1H), 4.38 (d, *J* = 5.8 Hz, 2H), 5.40 (dd, *J* = 14.0 Hz, 9.6 Hz, 1H), 5.57 (dd, *J* = 14.0 Hz, 5.0 Hz, 1H), 7.02 (br s, 1H), 7.20–7.59 (m, 8H), 8.07 (d, *J* = 8.2 Hz, 1H); ^{13}C NMR δ 19.7, 43.2,

55.3, 60.8, 109.1, 120.0, 124.2, 127.5, 127.6, 127.9, 128.7, 132.8, 138.1, 145.9, 173.5. Anal. Calcd for $C_{17}H_{19}N_5O$: C, 66.00; H, 6.19; N, 22.64. Found: C, 66.39; H, 6.44; N, 22.77.

(2*S*)-2-[(1*H*-Benzotriazol-1-ylmethyl)amino]-4-methyl-*N*-(4-methylphenyl)-pentanamide (7c). White microcrystals (from EtOAc); yield 93%; mp 153–154 °C; $[\alpha]^{25}_D$ = -85 (*c* 1.54, $CHCl_3$); ^{1}H NMR δ 0.49 (d, *J* = 6.0 Hz, 3H), 0.80 (d, *J* = 6.0 Hz, 3H), 1.40–1.57 (m, 3H), 2.32 (s, 3H), 2.75–2.78 (m, 1H), 3.26–3.31 (m, 1H), 5.44 (dd, *J* = 14.1, 10.2 Hz, 1H), 5.66 (dd, *J* = 14.1, 5.0 Hz, 1H), 7.12 (d, *J* = 8.5 Hz, 2H), 7.36–7.40 (m, 3H), 7.52 (t, *J* = 7.5 Hz, 1H), 7.59 (d, *J* = 8.4 Hz, 1H), 8.05 (d, *J* = 8.4 Hz, 1H), 8.66 (s, 1H); ^{13}C NMR δ 20.8, 21.0, 23.0, 24.6, 42.7, 58.9, 60.8, 109.1, 119.5, 119.9, 124.2, 128.0, 129.4, 133.0, 133.8, 134.8, 145.8, 171.6. Anal. Calcd for $C_{20}H_{25}N_5O$: C, 68.35; H, 7.17; N, 19.93. Found: C, 67.97; H, 7.37; N, 19.87.

(2*S*)-2-[(4,4-Dimethyl-3-oxopentyl)amino]-*N*-(4-methylphenyl)-3-phenylpropanamide (8a). Colorless oil; yield 50%; $[\alpha]^{25}_D$ = -103 (*c* 1.54, $CHCl_3$); ^{1}H NMR δ 1.09 (s, 9H), 1.52 (br s, 1H), 2.32 (s, 3H), 2.40–2.55 (m, 2H), 2.59–2.68 (m, 1H), 2.69–2.78 (m, 1H), 2.80–2.92 (m, 1H), 3.27–3.43 (m, 2H), 7.15 (d, *J* = 8.2 Hz, 2H), 7.20–7.38 (m, 5H), 7.62 (d, *J* = 8.3 Hz, 2H), 9.41 (br s, 1H); ^{13}C NMR δ 20.9, 26.3, 35.8, 39.4, 43.0, 44.0, 64.4, 119.6, 126.9, 128.8, 129.0, 129.4, 133.4, 135.5, 137.6, 171.6, 214.8. Anal. Calcd for $C_{23}H_{30}N_2O_2$: C, 75.37; H, 8.25; N, 7.64. Found: C, 75.23; H, 8.49; N, 7.86.

5-Benzyl-3-(4-methylphenyl)tetrahydro-4*H*-imidazol-4-one (9a). White microcrystals; yield 40%; mp 125-126 °C; $[\alpha]^{25}_D$ = -3 (*c* 1.54, $CHCl_3$); ^{1}H NMR (acetone-d_6) δ 2.29 (s, 3H), 2.80-2.87 (m, 1H), 2.91 (dd, *J* = 14.0, 8.1 Hz, 1H), 3.12 (dd, *J* = 14.0, 3.8 Hz, 1H),

3.73-3.84 (m, 1H), 4.50-4.61 (m, 1H), 4.65-4.73 (m, 1H), 7.10–7.37 (m, 7H), 7.51 (d, *J* = 8.5 Hz, 2H); ^{13}C NMR (acetone-d_6) δ 20.9, 38.0, 63.1, 64.9, 119.3, 127.2, 129.1, 130.1, 130.4, 134.3, 137.1, 139.5, 174.3. Anal. Calcd for $C_{17}H_{18}N_2O$: C, 76.66; H, 6.81; N, 10.52. Found: C, 77.01; H, 7.15; N, 10.37.

(2*S*)-2-[(Cyanomethyl)amino]-*N*-(4-methylphenyl)-3-phenylpropanamide (8b). White prisms; yield 92%; mp 116-117°C; $[\alpha]^{25}_D$ = -166 (*c* 1.54, $CHCl_3$); ^{1}H NMR δ 1.93 (br s, 1H), 2.30 (s, 3H), 2.88 (dd, *J* = 14.0, 8.9 Hz, 1H), 3.27 (dd, *J* = 14.0, 4.7 Hz, 1H), 3.38 (dd, *J* = 17.3, 7.8 Hz, 1H), 3.54 (d, *J* = 17.3 Hz, 1H), 3.63 (dd, *J* = 8.9, 4.7 Hz, 1H), 7.11 (d, *J* = 8.2 Hz, 2H), 7.19–7.36 (m, 5H), 7.40 (d, *J* = 8.3 Hz, 2H) 8.62 (s, 1H); ^{13}C NMR δ 20.7, 36.1, 39.0, 63.2, 116.8, 119.8, 127.3, 128.9, 129.0, 129.4, 134.2, 134.4, 135.8, 169.7. Anal. Calcd for $C_{18}H_{19}N_3O$: C, 73.69; H, 6.53; N, 14.32. Found: C, 73.34; H, 6.83; N, 14.35.

Methyl 3-{[(1*S*)-2-(benzylamino)-1-methyl-2-oxoethyl]amino}-2,2-dimethylpropanoate (8c). Colorless oil; yield 82%; $[\alpha]^{25}_D$ = -3 (*c* 1.69, $CHCl_3$); ^{1}H NMR δ 1.12 (s, 3H), 1.14 (s, 3H), 1.33 (d, *J* = 7.0 Hz, 3H), 2.52 (d, *J* = 11.5 Hz, 1H), 2.73 (d, *J* = 11.5 Hz, 1H), 3.17 (q, *J* = 7.0 Hz, 1H), 3.54 (s, 3H), 4.40–4.50 (m, 2H), 7.19–7.40 (m, 5H), 7.58 (br s, 1H); ^{13}C NMR δ 19.8, 23.3, 23.8, 42.9, 43.0, 51.7, 57.3, 58.6, 127.2, 127.6, 128.5, 138.5, 174.6, 177.4. Anal. Calcd for $C_{16}H_{24}N_2O_3$: C, 65.73; H, 8.27; N, 9.58. Found: C, 65.93; H, 8.37; N, 9.81.

(2*S*)-*N*-Benzyl-2-(3-butenylamino)propanamide (8d). Colorless oil; yield 61%; $[\alpha]^{25}_D$ = +5 (*c* 1.88, $CHCl_3$); ^{1}H NMR δ 1.22–1.32 (m, 1H), 1.36 (d, *J* = 7.0 Hz, 3H), 2.18–2.28 (m, 2H), 2.57–2.80 (m, 2H), 3.25 (q, *J* = 7.0 Hz, 1H), 4.45–4.54 (m, 2H), 5.02–5.12 (m, 2H), 5.66–5.81 (m, 1H), 7.28–7.41 (m, 5H), 7.68 (br s, 1H); ^{13}C NMR δ 19.8, 34.2, 42.8,

47.5, 58.2, 116.7, 127.3, 127.5, 128.6, 135.9, 138.5, 175.0. Anal. Calcd for $C_{14}H_{20}N_2O$: N, 12.06. Found: N, 12.02.

(2*S*)-4-Methyl-*N*-(4-methylphenyl)-2-[(3-oxo-3-phenylpropyl)amino]pentanamide (8e). Colorless oil; yield 64%; $[\alpha]^{25}_D$ = +1 (*c* 1.54, $CHCl_3$); ^{1}H NMR δ 0.96 (d, *J* = 6.2 Hz, 3H), 0.97 (d, *J* = 6.2 Hz, 3H), 1.39–1.52 (m, 1H), 1.62–1.70 (m, 1H), 1.71–1.76 (m, 1H), 1.77 (br s, 1H), 2.31 (s, 3H), 2.98–3.12 (m, 2H), 3.14–3.30 (m, 3H), 7.13 (d, *J* = 8.2 Hz, 2H), 7.42–7.51 (m, 2H), 7.58 (d, *J* = 8.4 Hz, 3H), 7.98 (d, *J* = 7.1 Hz, 2H), 9.46 (s, 1H); ^{13}C NMR δ 20.8, 21.7, 23.3, 25.1, 38.2, 42.8, 43.3, 62.0, 119.4, 128.0, 128.7, 129.3, 133.2, 133.4, 135.6, 136.6, 172.9, 199.1. HRMS Calcd for $C_{22}H_{28}N_2O_2$: 353.2151; Found: 353.2222.

(2*S*)-2-[(Cyanomethyl)amino]-4-methyl-*N*-(4-methylphenyl)pentanamide (8f). Colorless oil; yield 89%; ; $[\alpha]^{25}_D$ = -122 (*c* 1.54, $CHCl_3$); ^{1}H NMR δ 0.98 (d, *J* = 6.3 Hz, 6H), 1.43–1.58 (m, 1H), 1.60–1.84 (m, 2H), 2.00 (br s, 1H), 2.31 (s, 3H), 3.36–3.52 (m, 2H), 3.68 (d, *J* = 17.4 Hz, 1H), 7.12 (d, *J* = 8.2 Hz, 2H), 7.43 (*J* = 8.4 Hz, 2H); ^{13}C NMR δ 20.7, 21.7, 23.0, 24.9, 36.1, 42.7, 61.1, 117.4, 119.7, 129.4, 134.0, 134.7, 171.1. HRMS Calcd for $C_{15}H_{21}N_3O$: 259.1684; Found: 259.1687.

Methyl (2*R*)-2-(3-butenyl{[(2*S*)-1-(2,2,2-trifluoroacetyl)tetrahydro-1*H*-pyrrol-2-yl] carbonyl} amino)-3-phenylpropanoate (10). Colorless oil. ^{1}H NMR δ 1.73-1.78 (m, 1H), 1.91-2.21 (m, 5H), 2.77-2.87 (m, 1H), 3.24-3.43 (m, 3H), 3.69-3.75 (m, 4H), 3.83-3.90 (m, 1H), 4.15 (dd, *J* = 10.2, 4.7 Hz, 1H), 4.72 (dd, *J* = 8.0, 4.0 Hz, 1H), 4.95-5.02 (m, 2H), 5.57-5.70 (m, 1H), 7.20-7.34 (m, 5H). ^{13}C NMR δ 24.8, 28.2, 32.9, 35.0, 47.4, 48.9, 52.3, 58.3, 63.2, 116.3 (q, *Jc-f* = 287 Hz), 117.3, 126.7, 128.5, 129.6, 134.1, 137.9, 155.6 (q, *Jc-f* = 37 Hz), 170.6, 170.7. ^{19}F NMR δ -72.9.

Methyl (2*S*)-2-(3-butenyl{[(2*S*)-1-(2,2,2-trifluoroacetyl)tetrahydro-1*H*-pyrrol-2-yl] carbonyl} amino)-3-phenylpropanoate (10'). Colorless oil. ^{1}H NMR δ 1.85-1.92 (m, 1H), 2.00-2.41 (m, 5H), 2.72-2.81 (m, 1H), 3.13-3.42 (m, 3H), 3.68-3.78 (m, 4H), 3.88-3.96 (m, 1H), 4.07 (dd, *J* = 10.2, 5.4 Hz, 1H), 4.72 (dd, *J* = 8.4, 3.7 Hz, 1H), 4.97-5.06 (m, 2H), 5.64-5.78 (m, 1H), 7.15-7.38 (m, 5H). ^{13}C NMR δ 24.8, 28.4, 32.7, 34.8, 47.4, 49.4, 52.1, 58.4, 62.8, 116.0 (q, *Jc-f* = 252 Hz), 117.2, 126.8, 128.5, 129.2, 134.3, 138.0, 155.5 (q, *Jc-f* = 37 Hz), 170.4, 170.7. ^{19}F NMR: δ -73.1

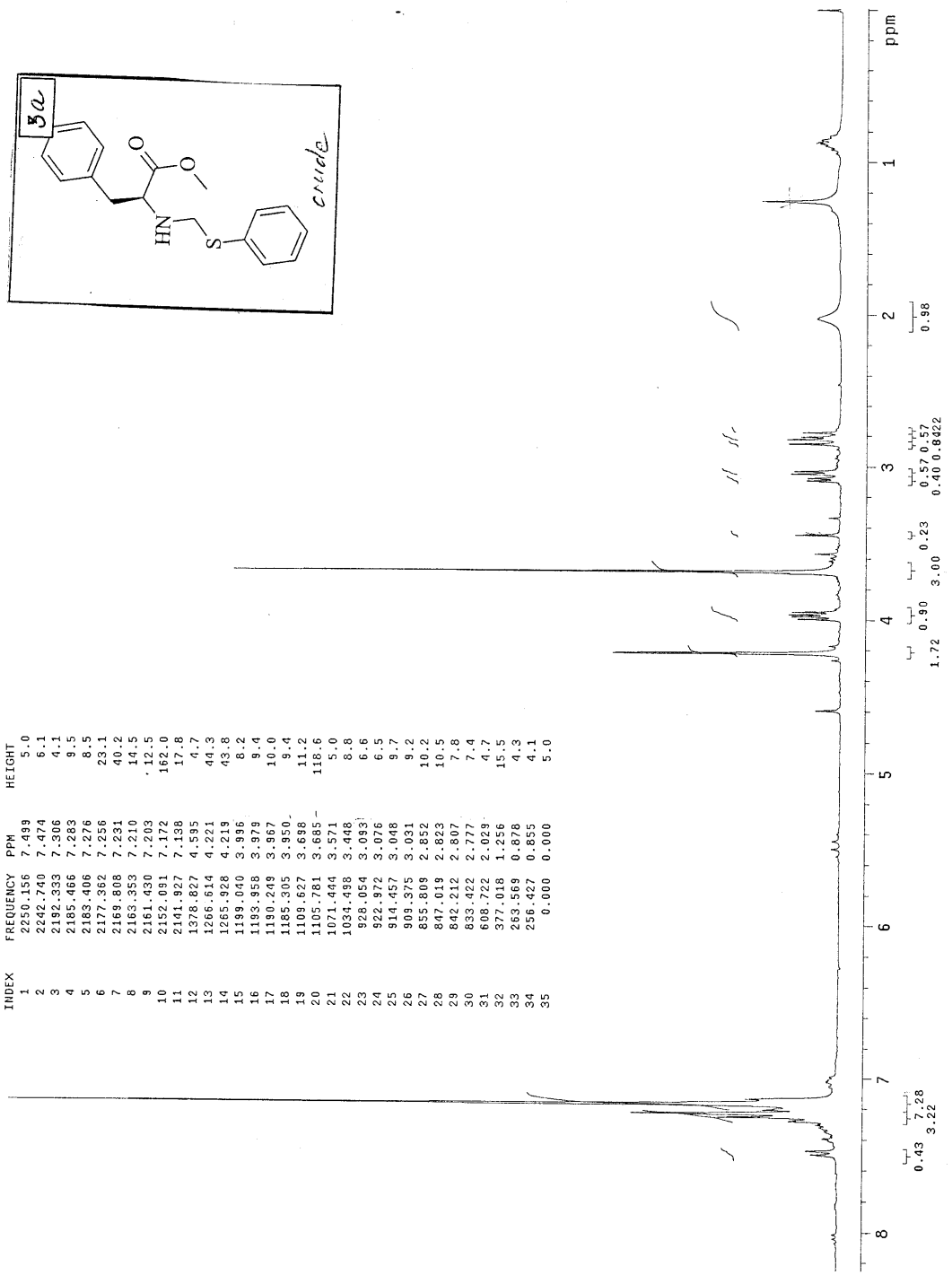


Figure 1. ^{1}H MNR spectra for compound **3a**.

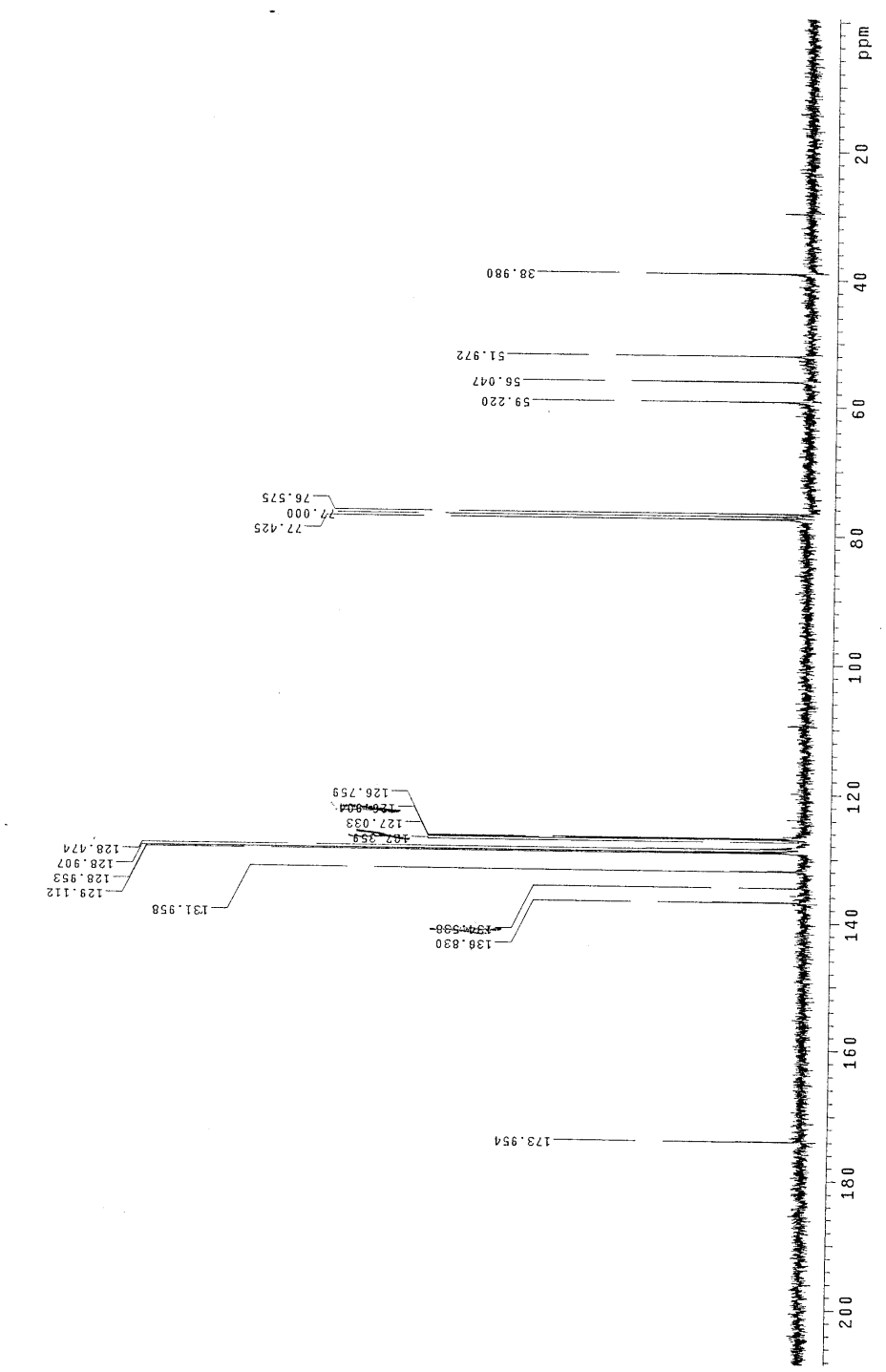
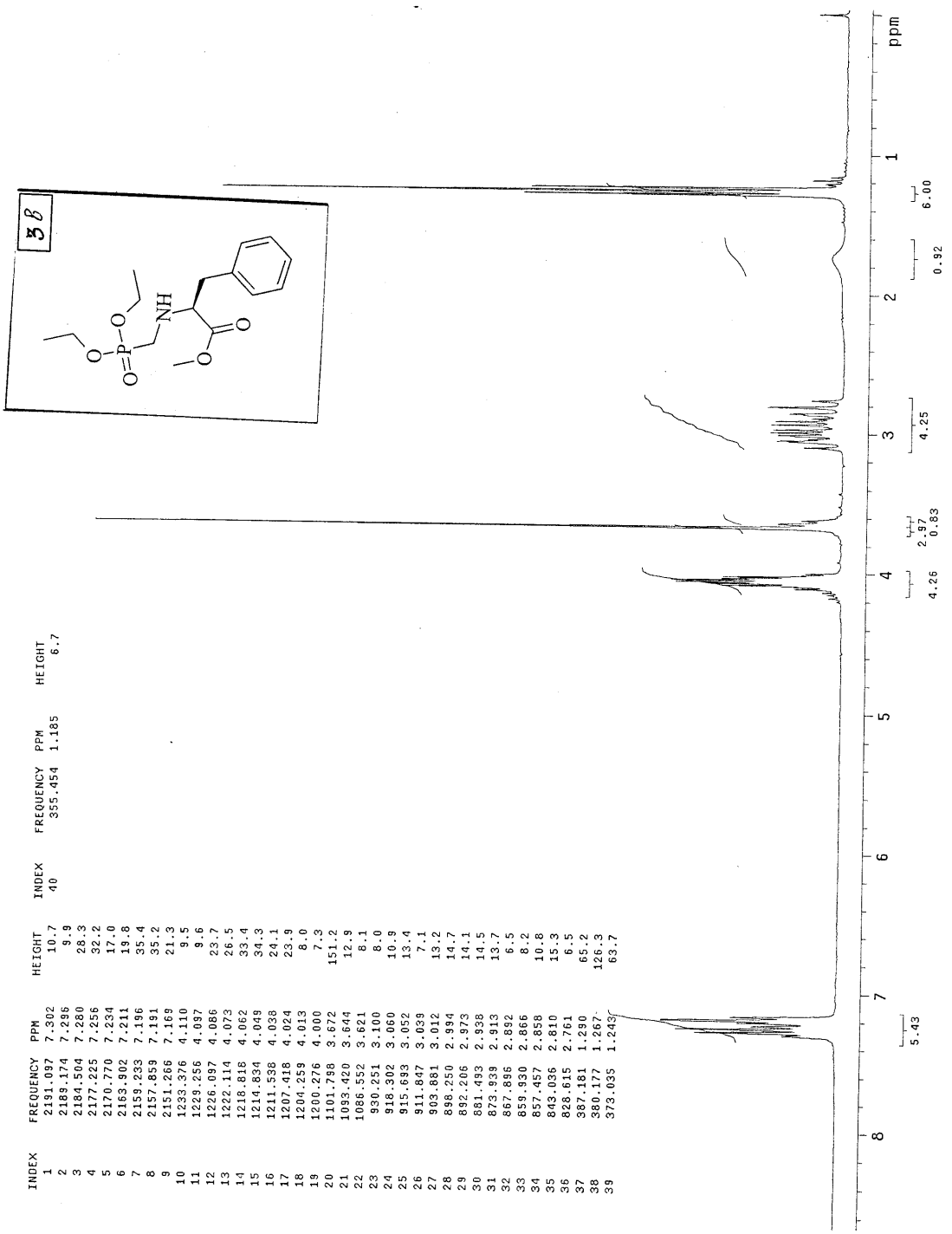


Figure 2. ^{13}C NMR spectra for compound **3a**.



INDEX	FREQUENCY	PPM	HEIGHT
1	2191.097	7.302	10.7
2	2189.174	7.296	9.9
3	2184.504	7.280	28.3
4	2177.225	7.256	32.2
5	2170.770	7.234	17.0
6	2163.902	7.211	19.8
7	2159.233	7.196	35.4
8	2157.859	7.191	35.2
9	2151.266	7.169	21.3
10	1233.376	4.110	9.5
11	1229.256	4.097	9.6
12	1226.097	4.086	23.7
13	1222.114	4.073	26.5
14	1218.816	4.062	33.4
15	1214.834	4.049	34.3
16	1211.538	4.038	24.1
17	1207.418	4.024	23.9
18	1204.259	4.013	8.0
19	1200.276	4.000	7.3
20	1101.798	3.672	151.2
21	1093.420	3.644	12.9
22	1086.552	3.621	8.1
23	930.251	3.100	8.0
24	918.302	3.060	10.9
25	915.693	3.052	13.4
26	911.847	3.039	7.1
27	903.881	3.012	13.2
28	898.250	2.994	14.7
29	892.206	2.973	14.1
30	881.493	2.938	14.5
31	873.939	2.913	13.7
32	867.896	2.892	6.5
33	859.930	2.866	8.2
34	857.457	2.858	10.8
35	843.036	2.810	15.3
36	828.615	2.761	6.5
37	387.181	1.290	65.2
38	380.177	1.267	126.3
39	373.035	1.243	63.7

INDEX	FREQUENCY	PPM	HEIGHT
40	355.454	1.185	6.7

Figure 3. ^{1}H NMR Spectra for compound **3b**.

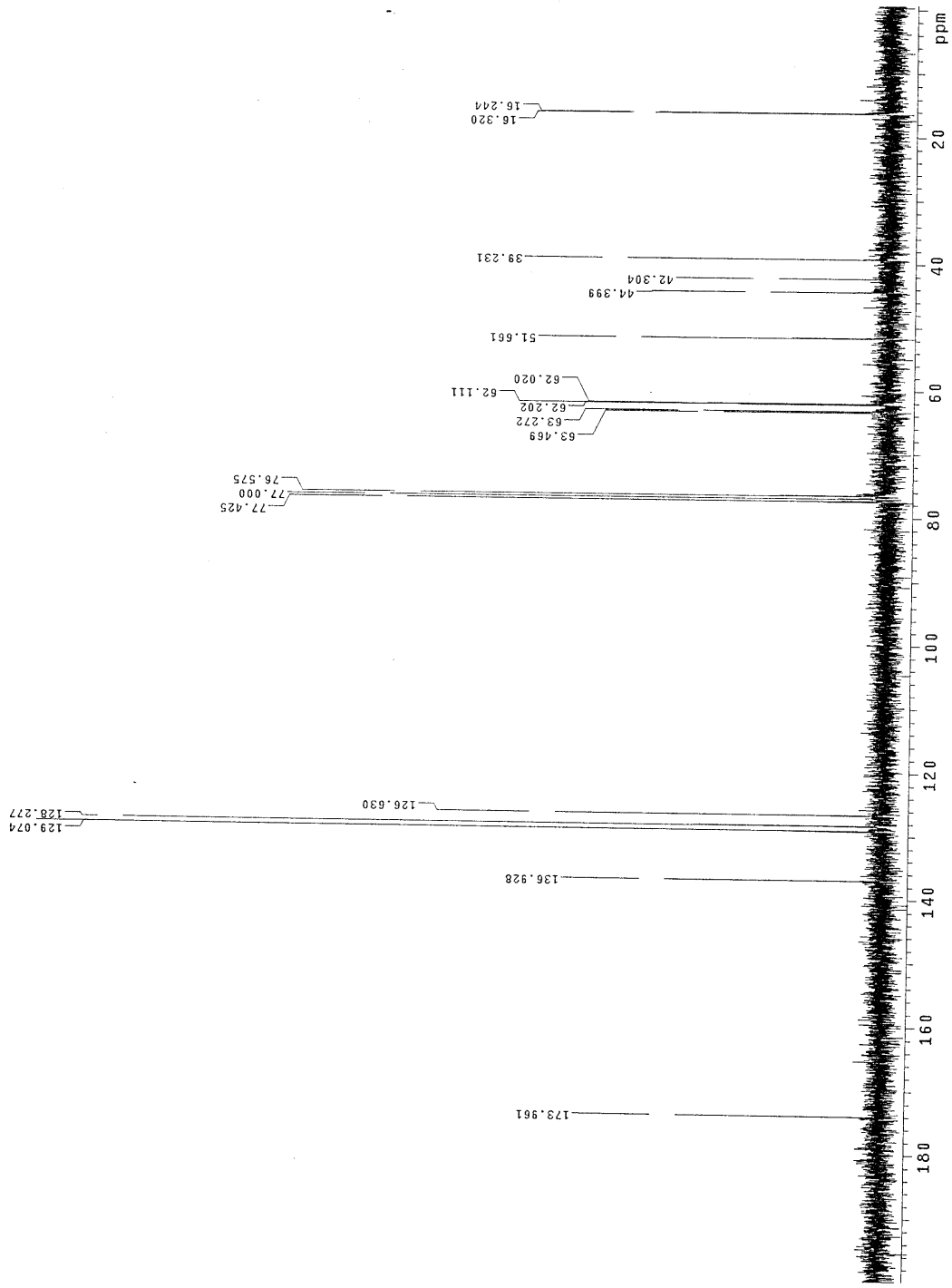
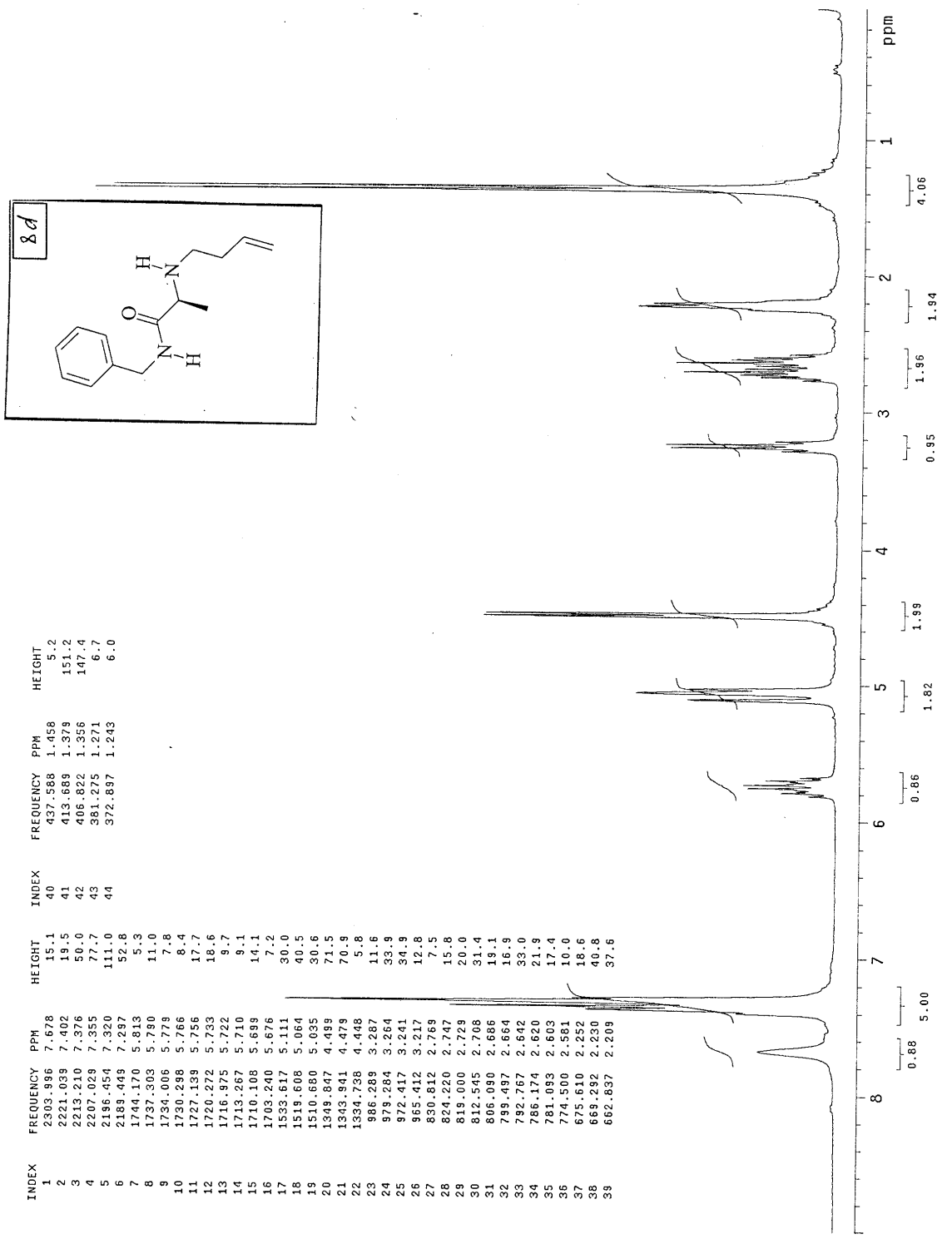


Figure 4. ^{13}C NMR spectra for compound **3b**.



INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	2303.996	7.678	15.1	40	437.588	1.458	5.2
2	2221.039	7.402	19.5	41	413.689	1.379	151.2
3	2213.210	7.376	50.0	42	406.822	1.356	147.4
4	2207.029	7.355	77.7	43	381.275	1.271	6.7
5	2196.454	7.320	111.0	44	372.897	1.243	6.0
6	2189.449	7.297	52.8				
7	1744.170	5.813	5.3				
8	1737.303	5.790	11.0				
9	1734.006	5.779	7.8				
10	1730.298	5.766	8.4				
11	1727.139	5.756	17.7				
12	1720.272	5.733	18.6				
13	1716.975	5.722	9.7				
14	1713.267	5.710	9.1				
15	1710.108	5.699	14.1				
16	1703.240	5.676	7.2				
17	1533.617	5.111	30.0				
18	1519.608	5.064	40.5				
19	1510.680	5.035	30.6				
20	1349.847	4.499	71.5				
21	1343.941	4.479	70.9				
22	1334.738	4.448	5.8				
23	986.289	3.287	11.6				
24	979.284	3.264	33.9				
25	972.417	3.241	34.9				
26	965.412	3.217	12.8				
27	830.812	2.769	7.5				
28	824.220	2.747	15.8				
29	819.000	2.729	20.0				
30	812.545	2.708	31.4				
31	806.090	2.686	19.1				
32	799.497	2.664	16.9				
33	792.767	2.642	33.0				
34	786.174	2.620	21.9				
35	781.093	2.603	17.4				
36	774.500	2.581	10.0				
37	675.610	2.252	18.6				
38	669.292	2.230	40.8				
39	662.837	2.209	37.6				

Figure 5. ^{1}H NMR Spectra for compound **8d**.

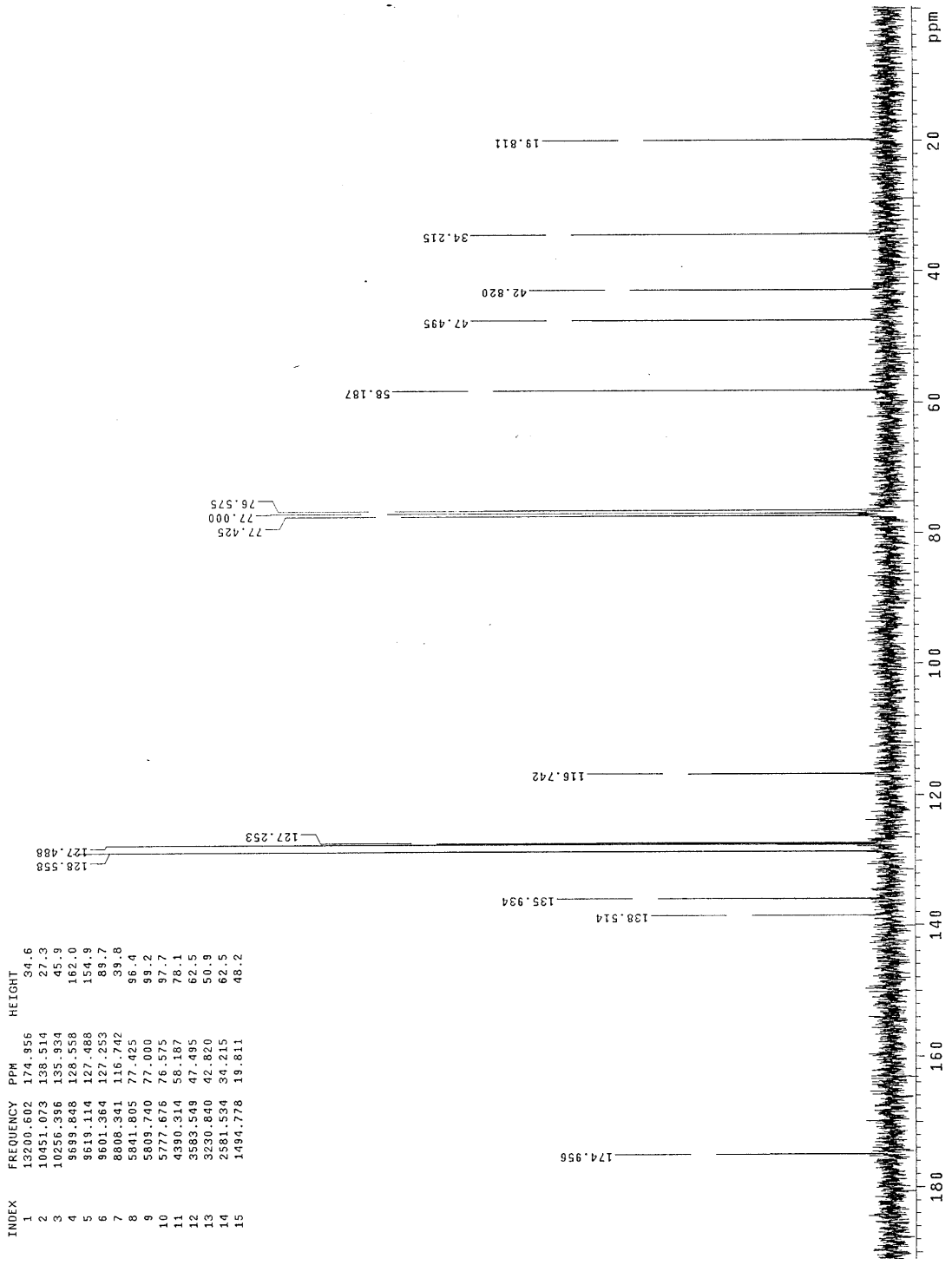
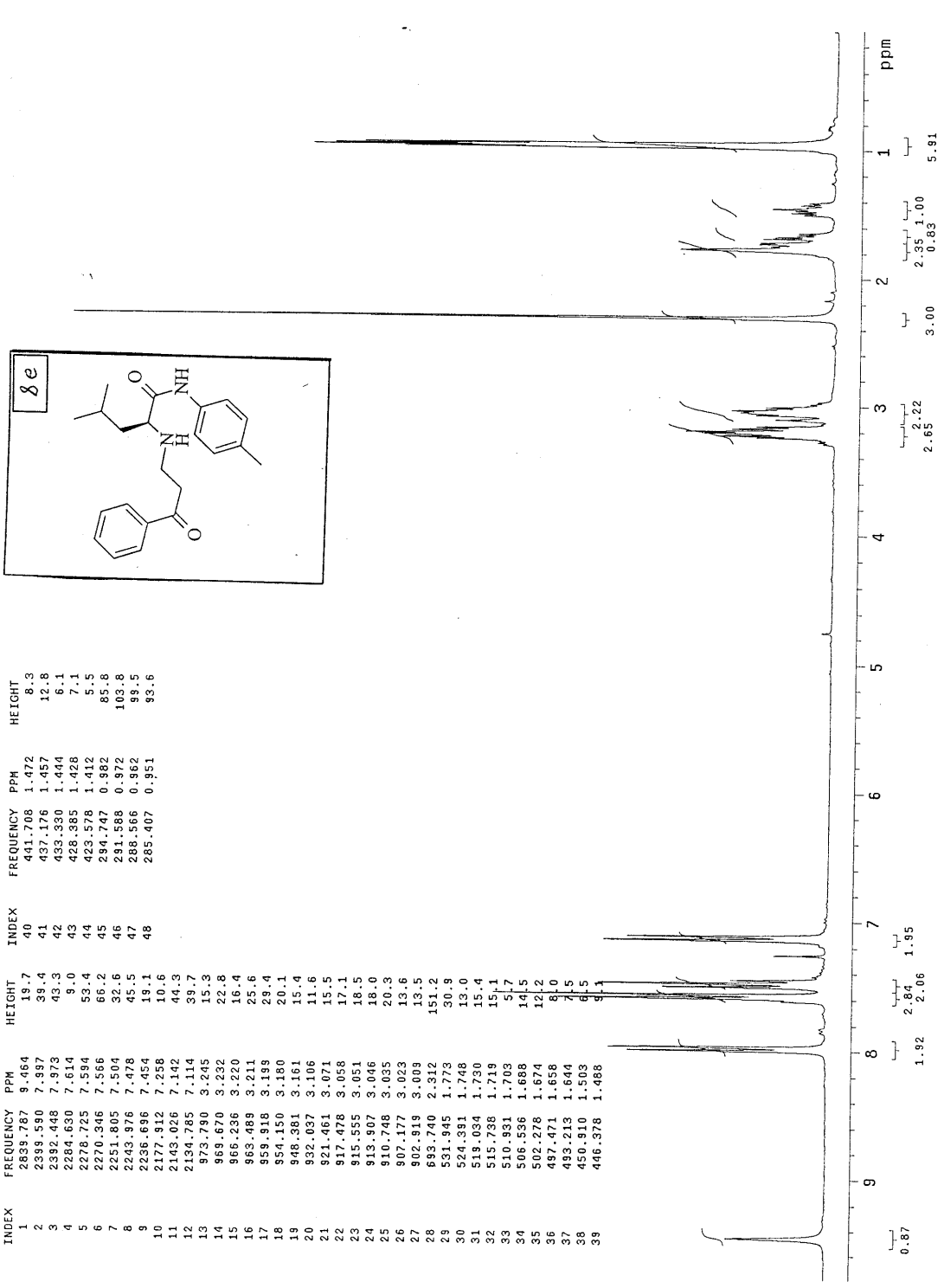


Figure 6. ^{13}C NMR spectra for compound **8d**.



INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	2839.787	9.464	19.7	40	441.708	1.472	8.3
2	2399.590	7.997	39.4	41	437.176	1.457	12.8
3	2392.448	7.973	43.3	42	433.330	1.444	6.1
4	2284.630	7.614	9.0	43	428.385	1.428	7.1
5	2278.725	7.594	53.4	44	423.578	1.412	5.5
6	2270.346	7.566	66.2	45	294.747	0.982	85.8
7	2251.805	7.504	32.6	46	291.588	0.972	103.8
8	2243.976	7.478	45.5	47	288.566	0.962	99.5
9	2236.696	7.454	19.1	48	285.407	0.951	93.6
10	2177.912	7.258	10.6				
11	2143.026	7.142	44.3				
12	2134.785	7.114	39.7				
13	973.790	3.245	15.3				
14	969.670	3.232	22.8				
15	966.236	3.220	16.4				
16	963.489	3.211	25.6				
17	959.918	3.199	29.4				
18	954.150	3.180	20.1				
19	948.381	3.161	15.4				
20	932.037	3.106	11.6				
21	921.461	3.071	15.5				
22	917.478	3.058	17.1				
23	915.555	3.051	18.5				
24	913.907	3.046	18.0				
25	910.748	3.035	20.3				
26	907.177	3.023	13.6				
27	902.919	3.009	13.5				
28	693.740	2.312	151.2				
29	531.945	1.773	30.9				
30	524.391	1.748	13.0				
31	519.034	1.730	15.4				
32	515.738	1.719	15.1				
33	510.931	1.703	5.7				
34	506.536	1.688	14.5				
35	502.278	1.674	12.2				
36	497.471	1.658	8.0				
37	493.213	1.644	7.5				
38	450.910	1.503	6.5				
39	446.378	1.488	9.1				

Figure 7. ^{1}H NMR Spectra for compound **8e**.

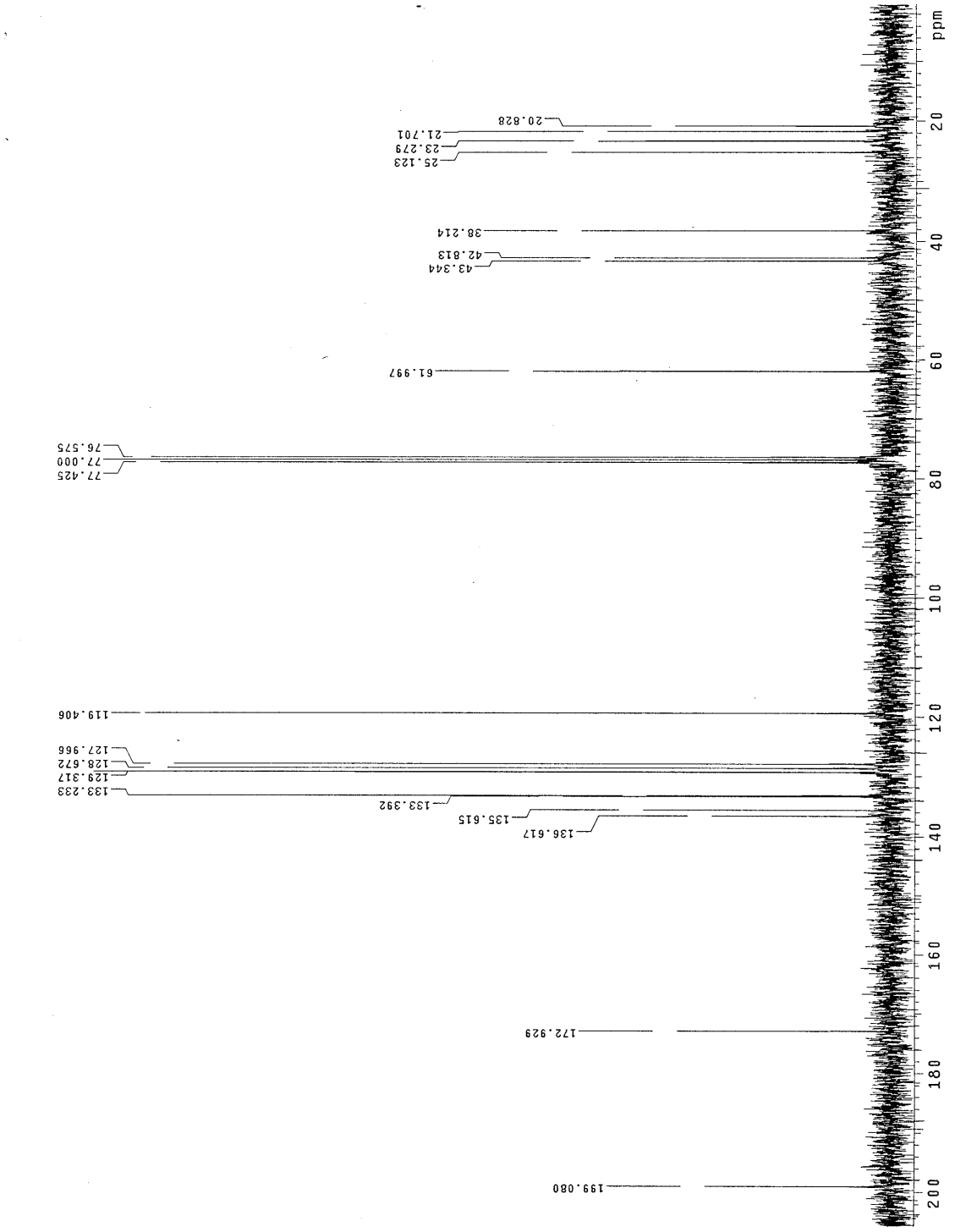
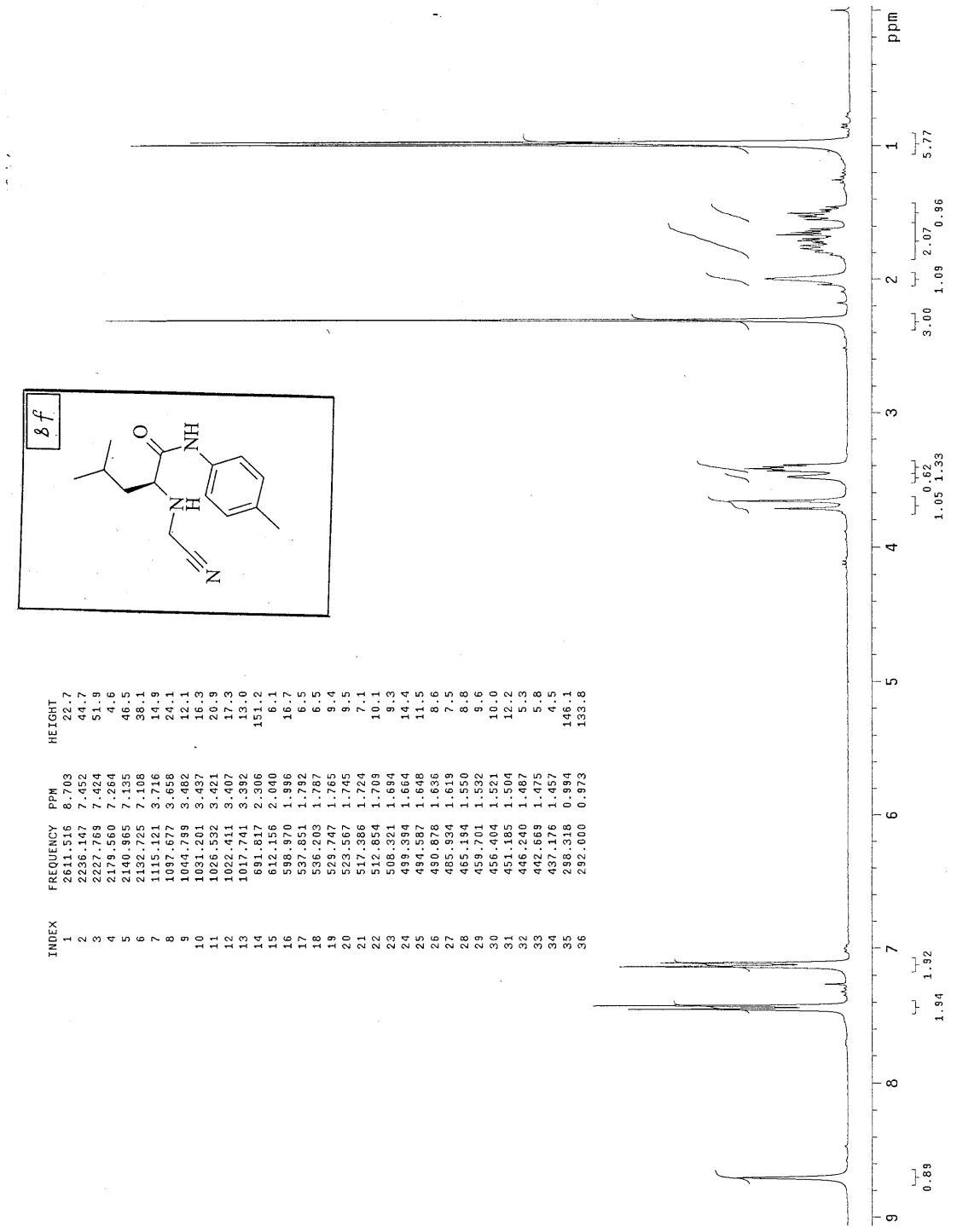


Figure 8. ^{13}C NMR spectra for compound **8e**.



INDEX	FREQUENCY	PPM	HEIGHT
1	2611.516	8.703	22.7
2	2236.147	7.452	44.7
3	2227.769	7.424	51.9
4	2179.560	7.264	4.6
5	2140.965	7.135	46.5
6	2132.725	7.108	38.1
7	1115.121	3.716	14.9
8	1097.677	3.658	24.1
9	1044.739	3.482	12.1
10	1031.201	3.437	16.3
11	1026.532	3.421	20.9
12	1022.411	3.407	17.3
13	1017.741	3.392	13.0
14	691.817	2.306	151.2
15	612.156	2.040	6.1
16	598.970	1.996	16.7
17	537.851	1.792	6.5
18	536.203	1.787	6.5
19	529.747	1.765	9.4
20	523.567	1.745	9.5
21	517.386	1.724	7.1
22	512.854	1.709	10.1
23	508.321	1.694	9.3
24	499.394	1.664	14.4
25	494.587	1.648	11.5
26	490.878	1.636	8.6
27	485.934	1.619	7.5
28	465.194	1.550	8.8
29	459.701	1.532	9.6
30	456.404	1.521	10.0
31	451.185	1.504	12.2
32	446.240	1.487	5.3
33	442.669	1.475	5.8
34	437.176	1.457	4.5
35	298.318	0.994	146.1
36	292.000	0.973	133.8

Figure 9. ^{1}H NMR Spectra for compound **8f**.

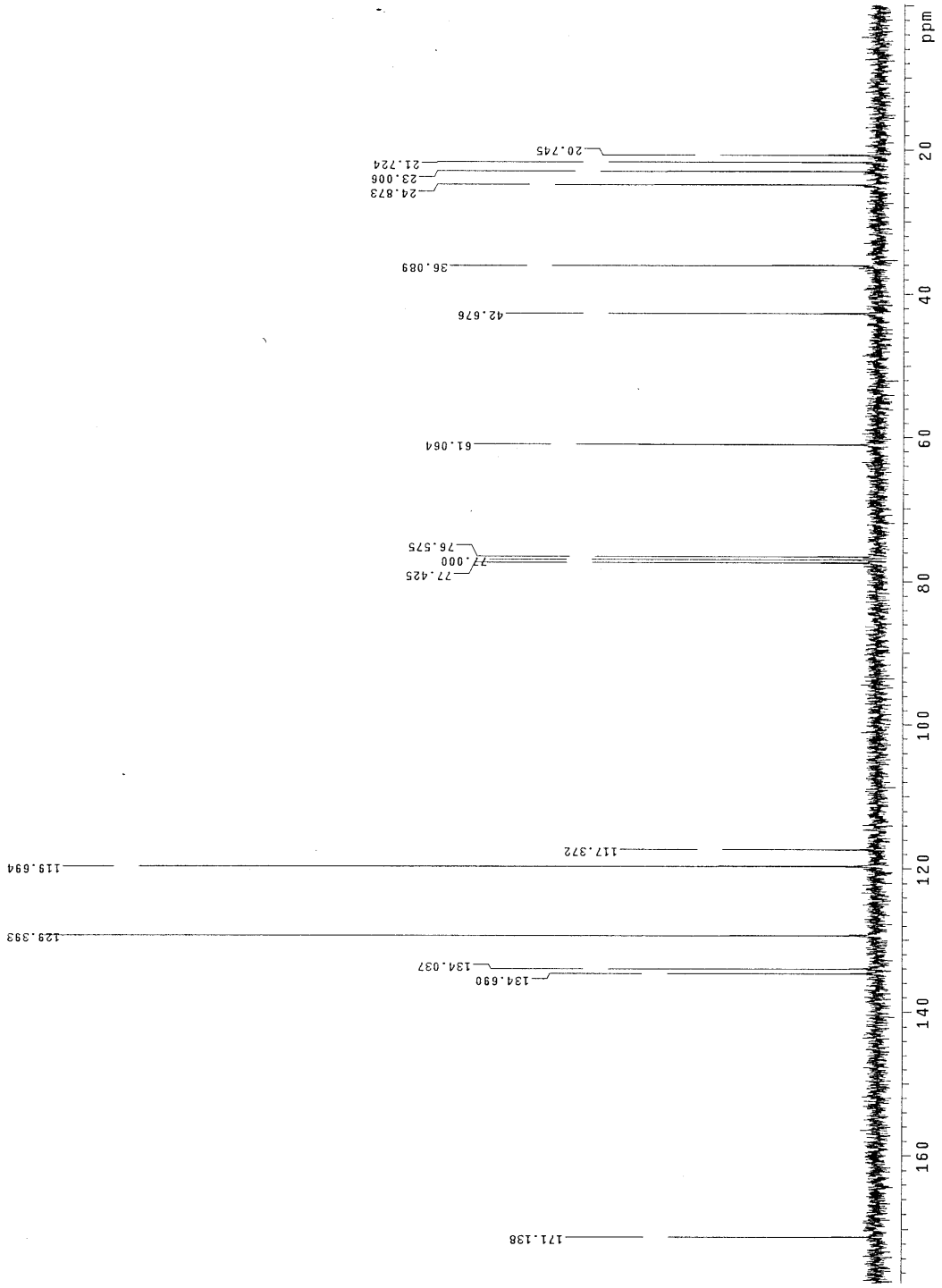


Figure 10. ^{13}C NMR spectra for compound **8f**.

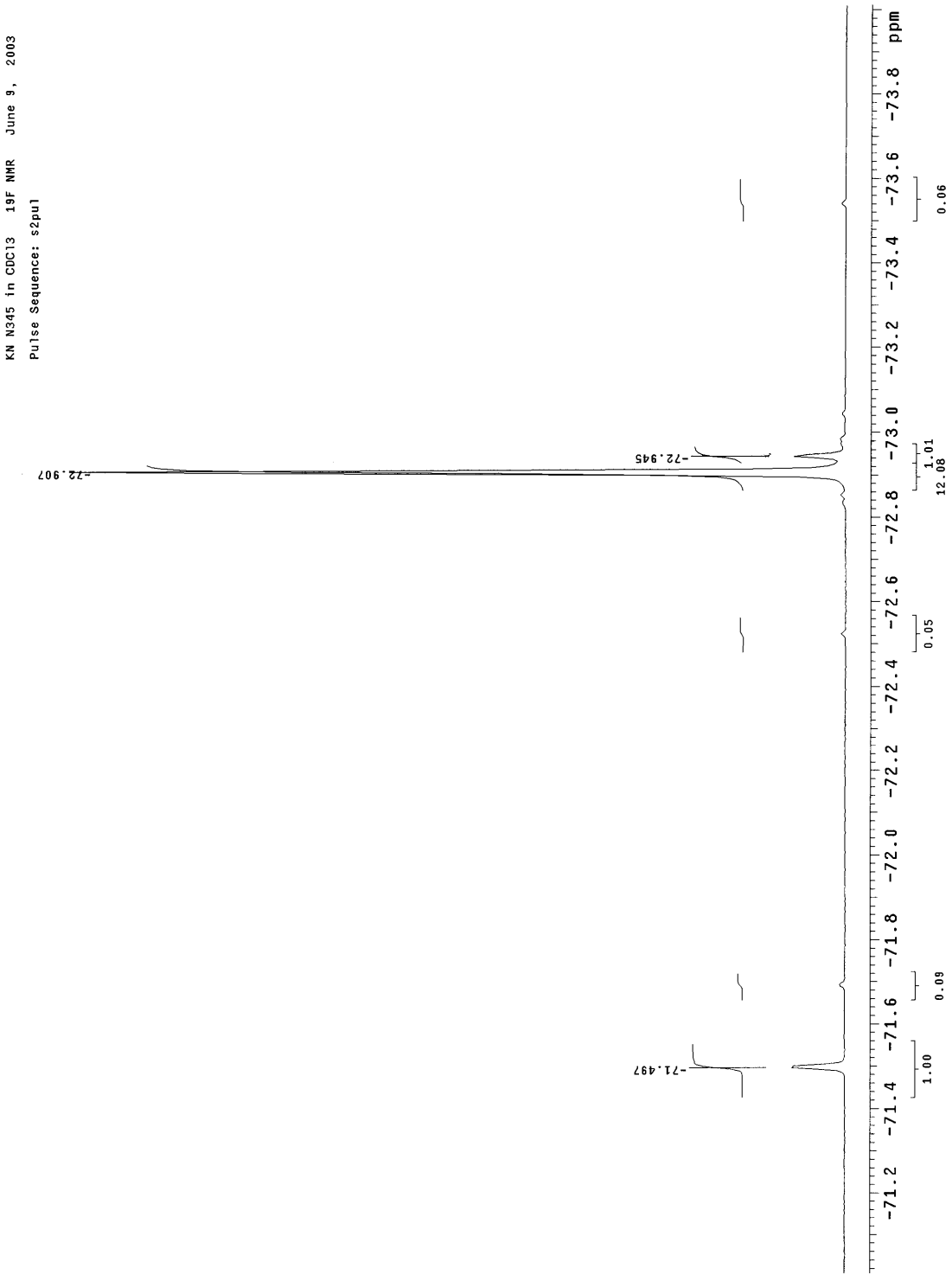


Figure 11. ^{19}F NMR of methyl (2R)-2-(3-butenyl{[(2S)-1-(2,2,2-trifluoroacetyl) tetrahydro-1H-pyrrol-2-yl]carbonyl}amino)-3-phenylpropanoate **10**.

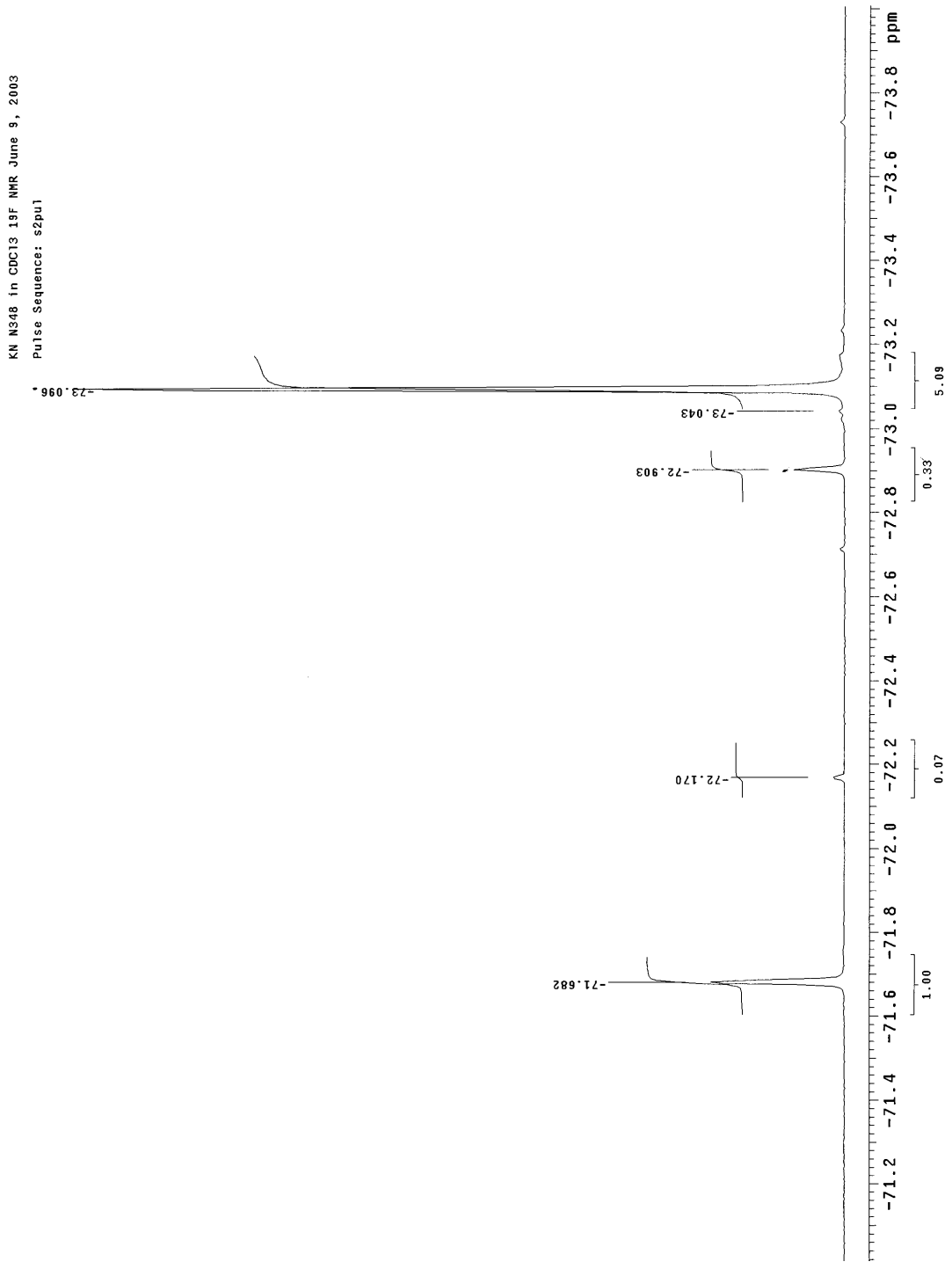


Figure 12. ^{19}F NMR of methyl (2S)-2-(3-butenyl{[(2S)-1-(2,2,2-trifluoroacetyl) tetrahydro-1H-pyrrol-2-yl]carbonyl}amino)-3-phenylpropanoate **10'**.

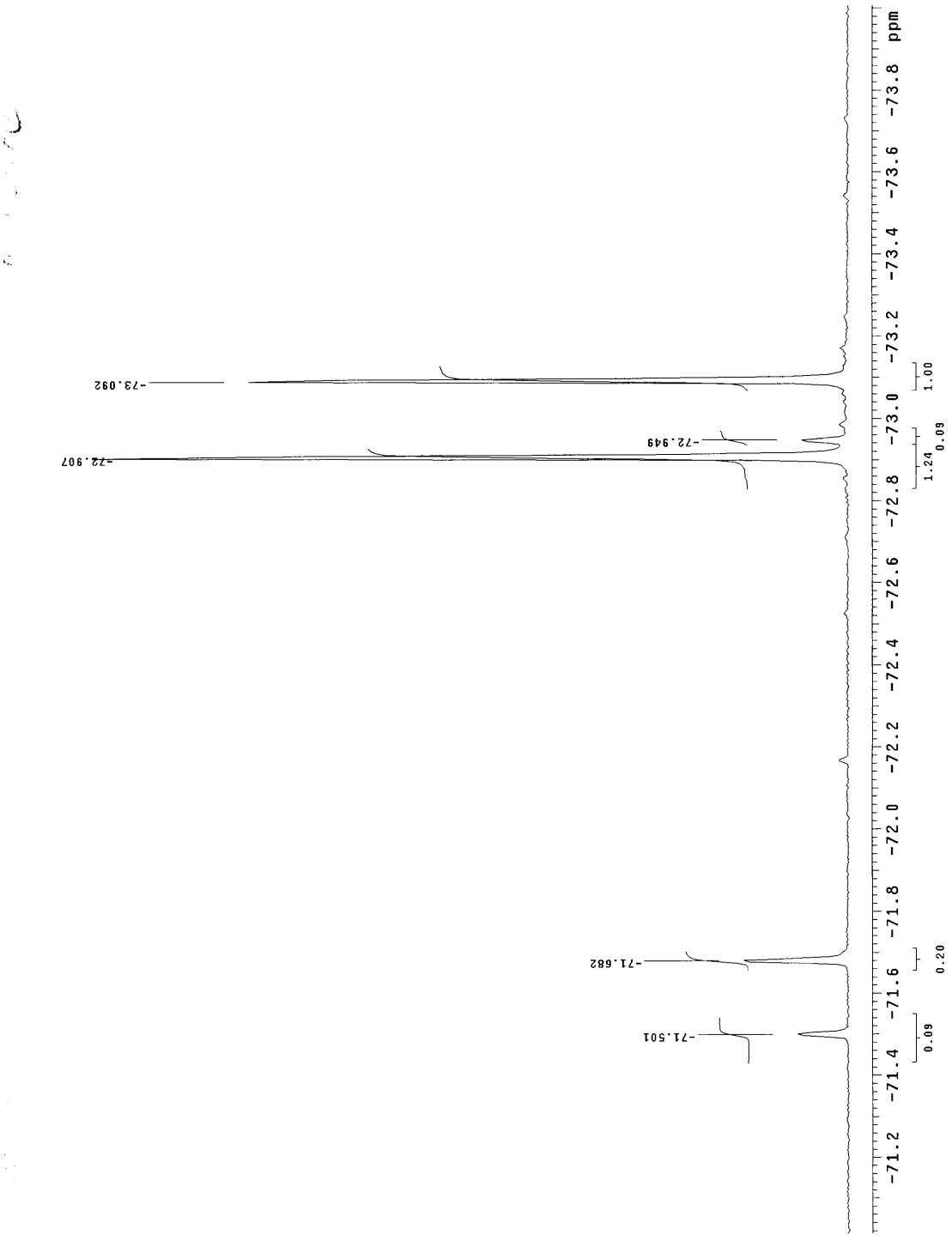


Figure 13. ^{19}F NMR for mixture of methyl (2R)-2-(3-butenyl{[(2S)-1-(2,2,2-trifluoroacetyl)tetrahydro-1H-pyrrol-2-yl]carbonyl}amino)-3-phenylpropanoate **10** and methyl (2S)-2-(3-butenyl{[(2S)-1-(2,2,2-trifluoroacetyl)tetrahydro-1H-pyrrol-2-yl]carbonyl}amino)-3-phenylpropanoate **10'**.

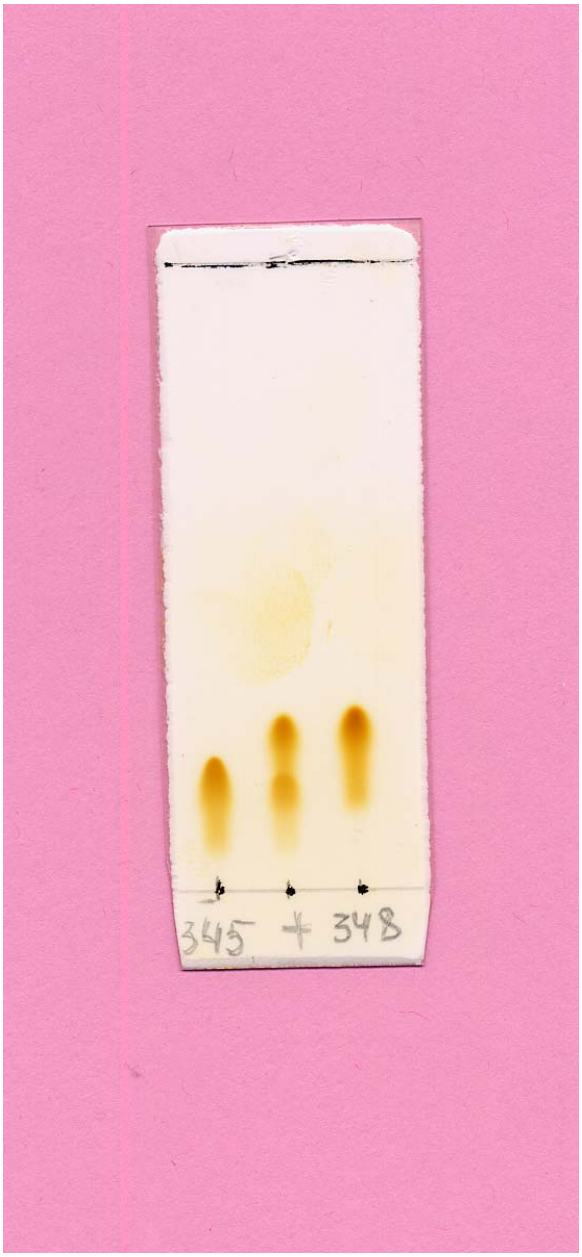


Figure 14. TLC comparison of diastereomers **10** and **10'**.

345- methyl (2R)-2-(3-butenyl{[(2S)-1-(2,2,2-trifluoroacetyl)tetrahydro-1H-pyrrol-2-yl]carbonyl}amino)-3-phenylpropanoate **10**

348- (2S)-2-(3-butenyl{[(2S)-1-(2,2,2-trifluoroacetyl)tetrahydro-1H-pyrrol-2-yl]carbonyl}amino)-3-phenylpropanoate **10'**

+ - mixture of 101+100