

α -Vinylolation of Amides with Arylacetylenes: Synthesis of Allylamines in Metal-Free condition

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Supporting Information

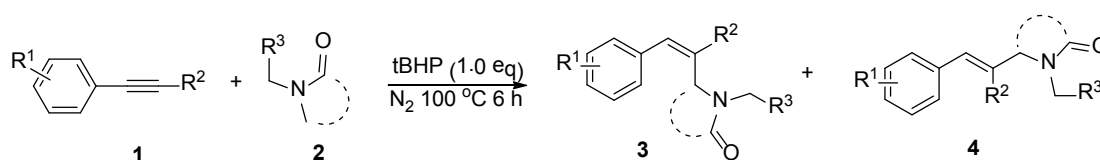
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General Remarks.

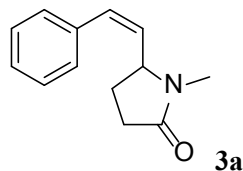
All reactions were carried out in Schlenk tubes. All the reagents were commercially available. Merck 60 silica gel was used for chromatography, and Whatman silica gel plates with fluorescence F254 were used for thin-layer chromatography (TLC) analysis. All products were confirmed by ^1H NMR, ^{13}C NMR, HRMS and IR. Tetramethylsilane (TMS) or CDCl_3 (7.26 ppm for ^1H NMR, 77.0 ppm for ^{13}C NMR) was used as a reference in ^1H NMR and ^{13}C NMR spectra. Data for ^1H were reported as follows: chemical shift (ppm), and multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet). Data for ^{13}C NMR were reported as ppm. Melting points were uncorrected.

Experimental Procedure



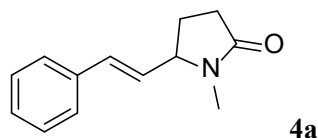
A dry Schlenk tube was charged with **1** (0.5 mmol) and **2** (2 mL) under nitrogen at room temperature. *t*BHP (91 μL , 0.5 mmol, 5-6 M in water) was added dropwise into the mixture. The resulting mixture was stirred at 100 °C for 6 h. Then the cooled reaction mixture was dissolved in water (15 mL) and extracted with ethyl acetate (3 x 15 mL). The combined organic layer was washed with water, dried with anhydrous MgSO_4 . After evaporating the solvent under reduced pressure, the residue was purified by silica gel column chromatography with ethanol and dichloromethane (10 : 1) as the eluent to give the pure product **3** and **4**.

Date



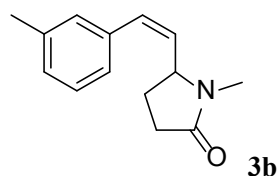
(Z)-1-methyl-5-styrylpyrrolidin-2-one (3a)

Brown liquid; yield 50 % (50 mg) (there is a little conformational isomer); ^1H NMR (400M Hz, CDCl_3) δ 7.37 (t, J = 7.2 Hz, 2H), 7.30 (t, J = 7.2 Hz, 1H), 7.21 (d, J = 7.2 Hz, 2H), 6.73 (d, J = 11.6 Hz, 1H), 5.54 (t, J = 10.4 Hz, 1H), 4.52-4.47 (m, 1H), 2.72 (s, 3H), 2.53-2.29 (m, 3H), 1.88-1.83 (m, 1H); ^{13}C NMR (100M Hz, CDCl_3) δ 175.5, 136.7, 133.6, 132.8, 129.2, 129.1, 128.2, 58.0, 30.9, 28.5, 26.5; IR (neat) ν = 2923, 1679, 1421, 1394, 1265, 1149, 1113, 982, 923, 806, 772, 699, 672; HRMS (TOF MS EI^+) $[\text{M}]^+$ calculated for $\text{C}_{13}\text{H}_{15}\text{NO}$ 201.1154, found 201.1153.



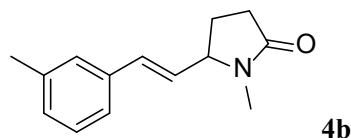
(E)-1-methyl-5-styrylpyrrolidin-2-one (4a)

Brown liquid; yield 25 % (25 mg); ^1H NMR (400M Hz, CDCl_3) δ 7.40-7.27 (m, 5H), 6.57 (d, $J = 16.0$ Hz, 1H), 6.02 (dd, $J_1 = 8.8$ Hz, $J_2 = 16.0$ Hz, 1H), 4.13-4.08 (m, 1H), 2.79 (s, 3H), 2.54-2.27 (m, 3H), 1.88-1.80 (m, 1H); ^{13}C NMR (100M Hz, CDCl_3) δ 75.7, 136.5, 133.7, 129.5, 129.4, 128.8, 127.2, 63.6, 30.7, 28.6, 26.4; IR (neat) $\nu = 2927, 1667, 1447, 1397, 1268, 1158, 1113, 971, 749, 695, 663$; HRMS (TOF MS EI^+) $[\text{M}]^+$ calculated for $\text{C}_{13}\text{H}_{15}\text{NO}$ 201.1154, found 201.1157.



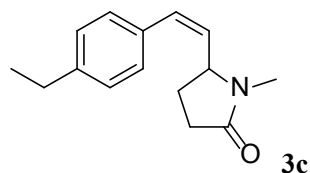
(Z)-1-methyl-5-(3-methylstyryl)pyrrolidin-2-one (3b)

Brown liquid; yield 31 % (33 mg) (there is a little conformational isomer); ^1H NMR (400M Hz, CDCl_3) δ 7.25 (t, $J = 8.0$ Hz, 1H), 7.11 (d, $J = 8.0$ Hz, 1H), 7.01-7.00 (m, 2H), 6.69 (d, $J = 11.6$ Hz, 1H), 5.51 (dd, $J_1 = 11.6$ Hz, $J_2 = 10.0$ Hz, 1H), 4.53-4.47 (m, 1H), 2.73 (s, 3H), 2.51-2.28 (m, 6H), 1.88-1.82 (m, 1H); ^{13}C NMR (100M Hz, CDCl_3) δ 174.9, 138.1, 136.0, 133.1, 131.9, 129.3, 128.33, 128.29, 125.5, 57.4, 30.2, 27.9, 25.9, 21.5; IR (neat) $\nu = 2922, 1688, 1603, 1422, 1395, 1276, 1115, 983, 909, 806, 701$; HRMS (TOF MS EI^+) $[\text{M}]^+$ calculated for $\text{C}_{14}\text{H}_{17}\text{NO}$ 215.1310, found 215.1311.



(E)-1-methyl-5-(3-methylstyryl)pyrrolidin-2-one (4b)

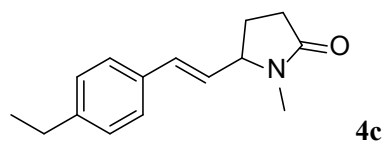
Brown liquid; yield 27 % (29 mg) (there is a little conformational isomer); ^1H NMR (400M Hz, CDCl_3) δ 7.25-7.18 (m, 3H), 7.10 (d, $J = 7.2$ Hz, 1H), 6.54 (d, $J = 15.6$ Hz, 1H), 6.01 (dd, $J_1 = 15.6$ Hz, $J_2 = 8.8$ Hz, 1H), 4.11-4.06 (m, 1H), 2.78 (s, 3H), 2.53-2.25 (m, 6H), 1.88-1.80 (m, 1H); ^{13}C NMR (100M Hz, CDCl_3) δ 175.0, 138.3, 135.9, 133.1, 129.0, 128.7, 128.6, 127.2, 123.7, 63.0, 30.0, 27.9, 25.8, 21.4; IR (neat) $\nu = 2922, 1681, 1423, 1395, 1270, 1157, 1113, 973, 783, 696, 663$; HRMS (TOF MS EI^+) $[\text{M}]^+$ calculated for $\text{C}_{14}\text{H}_{17}\text{NO}$ 215.1310, found 215.1312.



(Z)-5-(4-ethylstyryl)-1-methylpyrrolidin-2-one (3c)

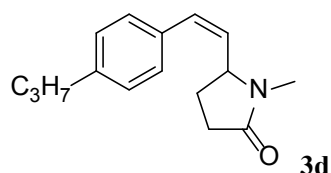
Brown liquid; yield 33 % (38 mg) (there is a little conformational isomer); ^1H NMR (400M Hz, CDCl_3) δ 7.20 (d, $J = 8.0$ Hz, 2H), 7.13 (d, $J = 8.0$ Hz, 2H), 6.69 (d, $J = 11.2$ Hz, 1H), 5.49 (dd, $J_1 = 11.2$ Hz, $J_2 = 9.6$ Hz, 1H), 4.55-4.49 (m, 1H), 2.73 (s, 3H), 2.69-2.63 (q, $J = 7.6$ Hz, 2H), 2.53-2.29 (m, 3H), 1.91-1.81 (m, 1H), 1.25 (t, $J = 7.6$ Hz, 3H); ^{13}C NMR (100M Hz, CDCl_3) δ

174.9, 143.7, 133.4, 132.9, 131.5, 128.6, 128.0, 57.4, 30.3, 28.6, 27.9, 25.9, 15.5; IR (neat) ν = 2964, 1684, 1509, 1421, 1394, 1267, 1114, 1057, 981, 843, 649; HRMS (TOF MS EI^+) $[M]^+$ calculated for $C_{15}H_{19}NO$ 229.1467, found 229.1466.



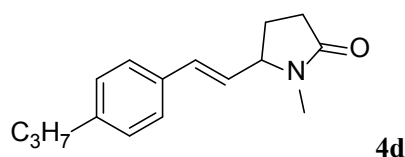
(E)-5-(4-ethylstyryl)-1-methylpyrrolidin-2-one (4c)

Brown liquid; yield 24 % (27 mg) (there is a little conformational isomer); 1H NMR (400M Hz, $CDCl_3$) δ 7.31 (d, J = 8.0 Hz, 2H), 7.17 (d, J = 8.4 Hz, 2H), 6.54 (d, J = 15.6 Hz, 1H), 5.97 (dd, J_1 = 16.0 Hz, J_2 = 8.8 Hz, 1H), 4.11-4.06 (m, 1H), 2.78 (s, 3H), 2.67-2.62 (q, J = 7.6 Hz, 2H), 2.53-2.26 (m, 3H), 1.87-1.79 (m, 1H), 1.23 (t, J = 7.6 Hz, 3H); ^{13}C NMR (100M Hz, $CDCl_3$) δ 175.0, 144.6, 133.4, 132.9, 128.2, 127.9, 126.6, 63.1, 30.1, 28.6, 27.9, 25.8, 15.6; IR (neat) ν = 2964, 1679, 1511, 1421, 1395, 1267, 1156, 1112, 971, 818, 662; HRMS (TOF MS EI^+) $[M]^+$ calculated for $C_{15}H_{19}NO$ 229.1467, found 229.1468.



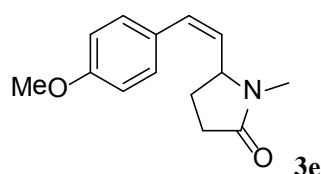
(Z)-1-methyl-5-(4-propylstyryl)pyrrolidin-2-one (3d)

Brown liquid; yield 32 % (39 mg) (there is a little conformational isomer); 1H NMR (400M Hz, $CDCl_3$) δ 7.19-7.12 (m, 4H), 6.68 (d, J = 11.6 Hz, 1H), 5.49 (t, J = 11.2 Hz, 1H), 4.55-4.49 (m, 1H), 2.73 (s, 3H), 2.59 (t, J = 7.6 Hz, 2H), 2.51-2.31 (m, 3H), 1.87-1.82 (m, 1H), 1.70-1.60 (m, 2H), 0.95 (t, J = 7.6 Hz, 3H); ^{13}C NMR (100M Hz, $CDCl_3$) δ 174.9, 142.2, 133.4, 132.9, 131.5, 128.6, 128.5, 57.4, 37.7, 30.3, 27.9, 25.9, 24.5, 13.8; IR (neat) ν = 2958, 2928, 2870, 1686, 1509, 1421, 1394, 1309, 1267, 1148, 1114, 982, 919, 864, 749, 651; HRMS (TOF MS EI^+) $[M]^+$ calculated for $C_{16}H_{21}NO$ 243.1623, found 243.1622.



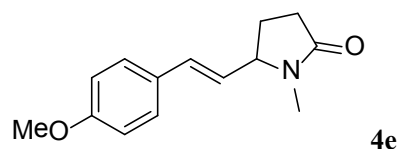
(E)-1-methyl-5-(4-propylstyryl)pyrrolidin-2-one (4d)

Brown liquid; yield 20 % (24 mg) (there is a little conformational isomer); 1H NMR (400M Hz, $CDCl_3$) δ 7.31 (d, J = 8.0 Hz, 2H), 7.15 (d, J = 8.0 Hz, 2H), 6.54 (d, J = 16.0 Hz, 1H), 5.97 (dd, J_1 = 16.0 Hz, J_2 = 8.8 Hz, 1H), 4.11-4.06 (m, 1H), 2.78 (s, 3H), 2.58 (t, J = 7.6 Hz, 2H), 2.51-2.26 (m, 3H), 1.87-1.79 (m, 1H), 1.68-1.59 (m, 2H), 0.94 (t, J = 7.6 Hz, 3H); ^{13}C NMR (100M Hz, $CDCl_3$) δ 175.0, 143.0, 133.4, 133.0, 128.8, 127.9, 126.5, 63.1, 37.8, 30.1, 27.9, 25.8, 24.5, 13.8; IR (neat) ν = 2958, 2928, 2868, 1680, 1511, 1421, 1395, 1267, 1157, 1112, 972, 814, 793, 663; HRMS (TOF MS EI^+) $[M]^+$ calculated for $C_{16}H_{21}NO$ 243.1623, found 243.1625.



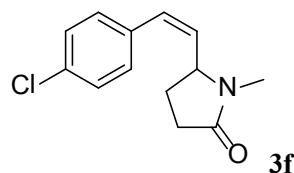
(Z)-5-(4-methoxystyryl)-1-methylpyrrolidin-2-one (3e)

Light yellow liquid; yield 42 % (48 mg) (there is a little conformational isomer); ^1H NMR (400M Hz, CDCl_3) δ 7.15 (d, $J = 8.4$ Hz, 2H), 6.90 (d, $J = 8.0$ Hz, 2H), 6.65 (d, $J = 11.6$ Hz, 1H), 5.44 (t, $J = 11.6$ Hz, 1H), 4.54-4.48 (m, 1H), 3.82 (s, 3H), 2.72 (s, 3H), 2.51-2.29 (m, 3H), 1.87-1.80 (m, 1H); ^{13}C NMR (100M Hz, CDCl_3) δ 174.9, 159.0, 132.4, 130.8, 129.8, 128.5, 113.9, 57.4, 55.3, 30.3, 27.9, 25.9; IR (neat) $\nu = 2950, 2838, 1671, 1605, 1509, 1460, 1423, 1397, 1300, 1249, 1176, 1151, 1113, 1029, 982, 920, 840, 706, 670$; HRMS (TOF MS EI^+) $[\text{M}]^+$ calculated for $\text{C}_{14}\text{H}_{17}\text{NO}_2$ 231.1259, found 231.1260.



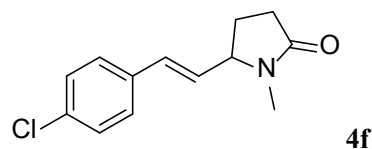
(E)-5-(4-methoxystyryl)-1-methylpyrrolidin-2-one (4e)

Light yellow solid; yield 26 % (30 mg), mp 71-72 °C (there is a little conformational isomer); ^1H NMR (400M Hz, CDCl_3) δ 7.32 (d, $J = 8.8$ Hz, 2H), 6.87 (d, $J = 8.8$ Hz, 2H), 6.51 (d, $J = 15.6$ Hz, 1H), 5.87 (dd, $J_1 = 15.6$ Hz, $J_2 = 8.8$ Hz, 1H), 4.10-4.04 (m, 1H), 3.82 (s, 3H), 2.78 (s, 3H), 2.53-2.26 (m, 3H), 1.87-1.78 (m, 1H); ^{13}C NMR (100M Hz, CDCl_3) δ 175.0, 159.6, 132.5, 128.7, 127.8, 126.6, 114.1, 63.1, 55.3, 30.1, 27.9, 25.9; IR (neat) $\nu = 2948, 2839, 1685, 1607, 1577, 1513, 1463, 1423, 1397, 1302, 1251, 1178, 1114, 1032, 973, 853, 825, 736, 666$; HRMS (TOF MS EI^+) $[\text{M}]^+$ calculated for $\text{C}_{14}\text{H}_{17}\text{NO}_2$ 231.1259, found 231.1262.



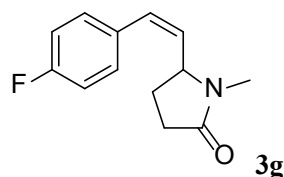
(Z)-5-(4-chlorostyryl)-1-methylpyrrolidin-2-one (3f)

Brown liquid; yield 35 % (41 mg) (there is a little conformational isomer); ^1H NMR (400M Hz, CDCl_3) δ 7.34 (d, $J = 8.4$ Hz, 2H), 7.14 (d, $J = 8.4$ Hz, 2H), 6.67 (d, $J = 11.6$ Hz, 1H), 5.56 (dd, $J_1 = 11.6$ Hz, $J_2 = 9.6$ Hz, 1H), 4.46-4.06 (m, 1H), 2.71 (s, 3H), 2.52-2.28 (m, 3H), 1.88-1.81 (m, 1H); ^{13}C NMR (100M Hz, CDCl_3) δ 174.8, 134.4, 133.5, 132.9, 131.7, 129.8, 128.7, 57.2, 30.1, 27.9, 25.7; IR (neat) $\nu = 2945, 1678, 1592, 1487, 1421, 1394, 1311, 1271, 1183, 1149, 1113, 1090, 1010, 982, 920, 841, 799, 779, 752, 715, 656$; HRMS (TOF MS EI^+) $[\text{M}]^+$ calculated for $\text{C}_{13}\text{H}_{14}\text{ClNO}$ 235.0764, found 235.0763.



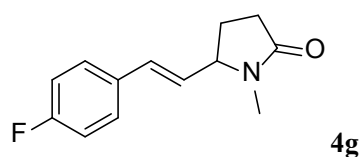
(E)-5-(4-chlorostyryl)-1-methylpyrrolidin-2-one (4f)

Brown liquid; yield 26 % (30 mg) (there is a little conformational isomer); ^1H NMR (400M Hz, CDCl_3) δ 7.31-7.28 (m, 4H), 6.52 (d, $J = 16.0$ Hz, 1H), 6.00 (dd, $J_1 = 15.6$ Hz, $J_2 = 8.4$ Hz, 1H), 4.12-4.06 (m, 1H), 2.78 (s, 3H), 2.49-2.27 (m, 3H), 1.88-1.79 (m, 1H); ^{13}C NMR (100M Hz, CDCl_3) δ 175.0, 134.4, 133.9, 131.7, 129.6, 128.9, 127.8, 62.8, 30.0, 28.0, 25.7; IR (neat) $\nu = 2943, 1672, 1593, 1489, 1423, 1397, 1364, 1266, 1157, 1088, 1010, 971, 856, 811, 759, 662$; HRMS (TOF MS EI^+) $[\text{M}]^+$ calculated for $\text{C}_{13}\text{H}_{14}\text{ClNO}$ 235.0764, found 235.0766.



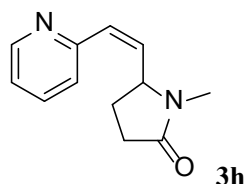
(Z)-5-(4-fluorostyryl)-1-methylpyrrolidin-2-one (3g)

Brown liquid; yield 36 % (39 mg) (there is a little conformational isomer); ^1H NMR (400M Hz, CDCl_3) δ 7.18 (dd, $J_1 = 8.4$ Hz, $J_2 = 5.6$ Hz, 2H), 7.06 (t, $J = 8.4$ Hz, 2H), 6.68 (d, $J = 11.6$ Hz, 1H), 5.54 (t, $J = 10.4$ Hz, 1H), 4.47-4.41 (m, 1H), 2.72 (s, 3H), 2.52-2.28 (m, 3H), 1.91-1.81 (m, 1H); ^{13}C NMR (100M Hz, CDCl_3) δ 174.8, 162.0 (d, $J_{\text{C-F}} = 245.9$ Hz), 132.04 (d, $J_{\text{C-F}} = 46$ Hz), 132.01 (d, $J_{\text{C-F}} = 3.9$ Hz), 130.2 (d, $J_{\text{C-F}} = 8.2$ Hz), 115.6, 115.3, 57.2, 30.2, 27.9, 25.7; IR (neat) $\nu = 2924, 1679, 1600, 1506, 1477, 1422, 1395, 1222, 1157, 1113, 982, 920, 844, 757, 734, 704, 669$; HRMS (TOF MS EI^+) $[\text{M}]^+$ calculated for $\text{C}_{13}\text{H}_{14}\text{FNO}$ 219.1059, found 219.1061.



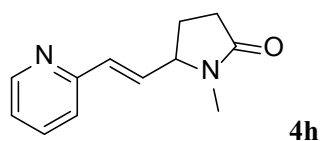
(E)-5-(4-fluorostyryl)-1-methylpyrrolidin-2-one (4g)

Brown liquid; yield 36 % (39 mg) (there is a little conformational isomer); ^1H NMR (400M Hz, CDCl_3) δ 7.36 (dd, $J_1 = 8.8$ Hz, $J_2 = 5.6$ Hz, 2H), 7.03 (t, $J = 8.8$ Hz, 2H), 6.52 (d, $J = 16.0$ Hz, 1H), 5.94 (dd, $J_1 = 16.0$ Hz, $J_2 = 8.8$ Hz, 1H), 4.11-4.06 (m, 1H), 2.78 (s, 3H), 2.53-2.27 (m, 3H), 1.88-1.79 (m, 1H); ^{13}C NMR (100M Hz, CDCl_3) δ 175.0, 163.8, 161.4, 132.12, 132.09, 131.8, 128.70, 128.68, 128.2, 128.1, 115.8, 115.5, 62.9, 30.0, 28.0, 25.7; IR (neat) $\nu = 2923, 1680, 1600, 1508, 1421, 1395, 1268, 1226, 1158, 1113, 972, 858, 821, 783, 663$; HRMS (TOF MS EI^+) $[\text{M}]^+$ calculated for $\text{C}_{13}\text{H}_{14}\text{FNO}$ 219.1059, found 219.1062.



(Z)-1-methyl-5-(2-(pyridin-2-yl)vinyl)pyrrolidin-2-one (3h)

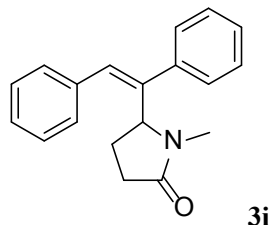
Brown liquid; yield 20 % (20 mg); ^1H NMR (400M Hz, CDCl_3) δ 8.60 (d, $J = 4.0$ Hz, 1H), 7.67 (td, $J_1 = 7.6$ Hz, $J_2 = 2.0$ Hz, 1H), 7.21-7.15 (m, 2H), 6.57 (d, $J = 11.6$ Hz, 1H), 5.73 (dd, $J_1 = 12.0$ Hz, $J_2 = 9.2$ Hz, 1H), 5.54-5.49 (m, 1H), 2.79 (s, 3H), 2.51-2.38 (m, 3H), 1.82-1.75 (m, 1H); ^{13}C NMR (100M Hz, CDCl_3) δ 175.2, 155.3, 149.3, 136.6, 136.3, 130.8, 124.5, 122.0, 57.5, 30.3, 28.0, 25.4; IR (neat) $\nu = 2926, 1669, 1585, 1563, 1472, 1437, 1395, 1306, 1272, 1249, 1203, 1151, 1113, 1047, 993, 921, 817, 747, 656$; HRMS (TOF MS EI^+) $[\text{M}]^+$ calculated for $\text{C}_{12}\text{H}_{14}\text{N}_2\text{O}$ 202.1106, found 202.1107.



(E)-1-methyl-5-(2-(pyridin-2-yl)vinyl)pyrrolidin-2-one (4h)

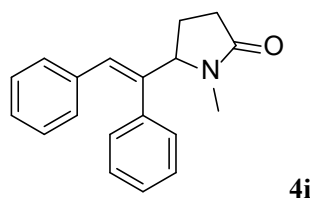
Brown liquid; yield 8 % (8 mg); ^1H NMR (400M Hz, CDCl_3) δ 8.58 (d, $J = 4.0$ Hz, 1H), 7.68 (td,

$J_1 = 8.0$ Hz, $J_2 = 1.6$ Hz, 1H), 7.29 (d, $J = 8.0$ Hz, 1H), 7.21-7.18 (m, 1H), 6.67-6.57 (m, 2H), 4.20-4.15 (m, 1H), 2.81 (s, 3H), 2.55-2.29 (m, 3H), 1.94-1.86 (m, 1H); ^{13}C NMR (100M Hz, CDCl_3) δ 175.1, 154.2, 149.5, 136.9, 133.5, 132.2, 122.8, 122.1, 62.5, 29.9, 28.1, 25.5; IR (neat) $\nu = 2924, 1677, 1587, 1469, 1435, 1398, 1278, 1246, 1157, 1049, 981, 777$; HRMS (TOF MS EI^+) $[\text{M}]^+$ calculated for $\text{C}_{12}\text{H}_{14}\text{N}_2\text{O}$ 202.1106, found 202.1105.



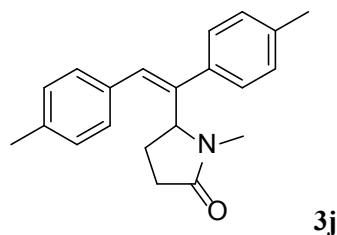
(Z)-5-(1,2-diphenylvinyl)-1-methylpyrrolidin-2-one (3i)

Brown liquid; yield 31 % (43 mg); ^1H NMR (400M Hz, CDCl_3) δ 7.41-7.24 (m, 10H), 6.90 (s, 1H), 4.90-4.87 (m, 1H), 2.76 (s, 3H), 2.28-2.17 (m, 2H), 2.10-1.92 (m, 2H); ^{13}C NMR (100M Hz, CDCl_3) δ 175.3, 141.0, 139.7, 136.5, 134.4, 128.7, 128.6, 128.5, 128.1, 127.8, 127.4, 58.9, 30.1, 28.2, 23.4; IR (neat) $\nu = 2947, 1680, 1598, 1488, 1442, 1423, 1395, 1277, 1154, 1114, 1075, 1029, 992, 939, 875, 763, 699, 634$; HRMS (TOF MS EI^+) $[\text{M}]^+$ calculated for $\text{C}_{19}\text{H}_{19}\text{NO}$ 277.1467, found 277.1469.



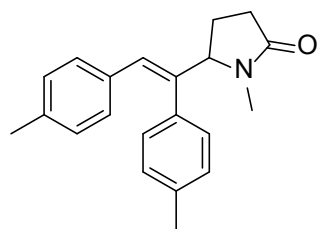
(E)-5-(1,2-diphenylvinyl)-1-methylpyrrolidin-2-one (4i)

Brown liquid; yield 31 % (43 mg) (there is a little conformational isomer); ^1H NMR (400M Hz, CDCl_3) δ 7.35-7.32 (m, 3H), 7.11-7.07 (m, 5H), 6.94-6.92 (m, 2H), 6.47 (s, 1H), 4.32-4.29 (m, 1H), 2.97 (s, 3H), 2.29-2.16 (m, 2H), 2.02-1.86 (m, 2H); ^{13}C NMR (100M Hz, CDCl_3) δ 175.7, 140.1, 137.5, 135.7, 129.3, 129.1, 129.1, 129.0, 128.1, 127.8, 127.3, 68.2, 29.6, 28.4, 23.7; IR (neat) $\nu = 3054, 2919, 1679, 1599, 1491, 1442, 1395, 1277, 1157, 1110, 1072, 1028, 985, 920, 759, 731, 697$; HRMS (TOF MS EI^+) $[\text{M}]^+$ calculated for $\text{C}_{19}\text{H}_{19}\text{NO}$ 277.1467, found 277.1466.



(Z)-5-(1,2-di-p-tolylvinyl)-1-methylpyrrolidin-2-one (3j)

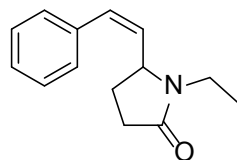
Lighe yellow solid; yield 31 % (47 mg); mp 104-106 °C (there is a little conformational isomer); ^1H NMR (400M Hz, CDCl_3) δ 7.21-7.13 (m, 8H), 6.84 (s, 1H), 4.91-4.87 (m, 1H), 2.74 (s, 3H), 2.38 (s, 3H), 2.35 (s, 3H), 2.29-1.90 (m, 4H); ^{13}C NMR (100M Hz, CDCl_3) δ 175.4, 140.4, 137.5, 137.2, 136.9, 134.0, 133.7, 129.2, 128.7, 128.0, 59.0, 30.2, 28.2, 23.4, 21.2, 21.1; IR (neat) $\nu = 2921, 1688, 1512, 1422, 1396, 1278, 1185, 1154, 1115, 1042, 885, 817, 726, 648$; HRMS (TOF MS EI^+) $[\text{M}]^+$ calculated for $\text{C}_{21}\text{H}_{23}\text{NO}$ 305.1780, found 305.1781.



4j

(E)-5-(1,2-di-p-tolylvinyl)-1-methylpyrrolidin-2-one (4j)

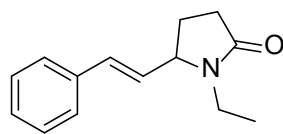
Brown liquid; yield 28 % (43 mg) (there is a little conformational isomer); ^1H NMR (400M Hz, CDCl_3) δ 7.15 (d, $J = 7.6$ Hz, 2H), 6.96-6.92 (m, 4H), 6.84 (d, $J = 8.0$ Hz, 2H), 6.41 (s, 1H), 4.28-4.25 (m, 1H), 2.94 (s, 3H), 2.36 (s, 3H), 2.24 (s, 3H), 2.23-2.14 (m, 2H), 1.98-1.86 (m, 2H); ^{13}C NMR (100M Hz, CDCl_3) δ 175.7, 139.1, 137.4, 137.1, 134.5, 133.0, 129.8, 129.2, 128.9, 128.80, 128.78, 68.4, 29.7, 28.3, 23.7, 21.3, 21.1; IR (neat) $\nu = 2920, 1680, 1511, 1421, 1396, 1309, 1276, 1183, 1111, 1040, 984, 898, 813, 788, 731, 650$; HRMS (TOF MS EI^+) $[\text{M}]^+$ calculated for $\text{C}_{21}\text{H}_{23}\text{NO}$ 305.1780, found 305.1782.



3k

(Z)-1-ethyl-5-styrylpyrrolidin-2-one (3k)

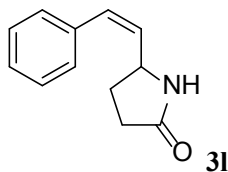
Brown liquid; yield 32 % (34 mg); the ratio of two conformational isomers is about 0.77 : 0.23; ^1H NMR (400M Hz, CDCl_3) δ 7.39-7.20 (m, 5H), 6.71 (d, $J = 11.2$ Hz, 0.77H), 6.52 (d, $J = 12.0$ Hz, 0.23H), 5.55 (t, $J = 11.2$ Hz, 0.77H), 5.59 (dd, $J_1 = 12.0$ Hz, $J_2 = 8.4$ Hz, 0.23H), 5.29-5.22 (m, 0.23H), 4.62-4.56 (m, 0.77H), 3.57-3.49 (m, 0.77H), 3.38-3.26 (m, 0.46H), 3.00-2.92 (m, 0.77H), 2.52-2.25 (m, 3H), 1.92-1.82 (m, 1H), 1.23 (d, $J = 6.8$ Hz, 0.69H), 0.90 (t, $J = 7.2$ Hz, 2.31H); ^{13}C NMR (100M Hz, CDCl_3) δ 174.4, 174.0, 136.1, 132.6, 132.4, 131.6, 129.9, 128.7, 128.4, 128.3, 127.5, 127.4, 55.0, 45.0, 42.7, 35.5, 31.3, 30.5, 25.9, 19.4, 18.1, 12.7; IR (neat) $\nu = 2975, 2973, 1679, 1492, 1448, 1416, 1377, 1355, 1311, 1262, 1217, 1148, 1126, 1078, 1027, 984, 920, 871, 773, 699, 664$; HRMS (TOF MS EI^+) $[\text{M}]^+$ calculated for $\text{C}_{14}\text{H}_{17}\text{NO}$ 215.1310, found 215.1311.



4k

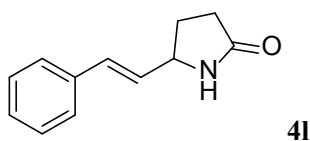
(E)-1-ethyl-5-styrylpyrrolidin-2-one (4k)

Brown liquid; yield 30 % (32 mg); the ratio of two conformational isomers is about 0.81 : 0.19; ^1H NMR (400M Hz, CDCl_3) δ 7.40-7.26 (m, 5H), 6.57 (d, $J = 16.0$ Hz, 0.81H), 6.50 (d, $J = 16.0$ Hz, 0.19H), 6.16 (dd, $J_1 = 16.0$ Hz, $J_2 = 6.0$ Hz, 0.19H), 6.02 (dd, $J_1 = 16.0$ Hz, $J_2 = 8.4$ Hz, 0.81H), 5.03-4.96 (m, 0.19H), 4.27-4.21 (m, 0.81H), 3.65-3.57 (m, 0.81H), 3.38-3.33 (m, 0.38H), 3.07-2.99 (m, 0.81H), 2.53-2.25 (m, 3H), 2.05-1.97 (m, 0.19H), 1.88-1.79 (m, 0.81H), 1.34 (d, $J = 7.2$ Hz, 0.57H), 1.09 (t, $J = 7.2$ Hz, 2.43H); ^{13}C NMR (100M Hz, CDCl_3) δ 174.6, 174.4, 136.0, 132.7, 131.1, 129.2, 128.9, 128.7, 128.6, 128.2, 127.7, 126.5, 126.4, 60.7, 47.7, 42.5, 35.5, 31.5, 30.4, 25.9, 18.1, 17.0, 12.8; IR (neat) $\nu = 2977, 2931, 1685, 1495, 1448, 1419, 1374, 1280, 1218, 1157, 1074, 973, 914, 755, 697, 669$; HRMS (TOF MS EI^+) $[\text{M}]^+$ calculated for $\text{C}_{14}\text{H}_{17}\text{NO}$ 215.1310, found 215.1312.



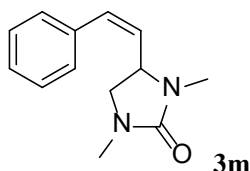
(Z)-5-styrylpyrrolidin-2-one (31)

Brown solid; yield 15 % (14 mg); mp 79-81 °C (there is a little conformational isomer); ¹H NMR (400M Hz, CDCl₃) δ 7.36 (t, *J* = 7.2 Hz, 2H), 7.28 (t, *J* = 7.2 Hz, 1H), 7.19 (d, *J* = 7.2 Hz, 2H), 6.60 (d, *J* = 11.2 Hz, 1H), 6.39 (br, 1H), 5.62 (dd, *J*₁ = 11.2 Hz, *J*₂ = 9.2 Hz, 1H), 4.64-4.59 (m, 1H), 2.44-2.32 (m, 3H), 1.99-1.92 (m, 1H); ¹³C NMR (100M Hz, CDCl₃) δ 178.2, 136.1, 132.6, 131.8, 128.6, 128.4, 127.5, 51.7, 30.4, 28.9; IR (neat) ν = 3220, 2977, 1691, 1600, 1493, 1447, 1367, 1263, 1203, 1073, 922, 771, 700, 647; HRMS (TOF MS EI⁺) [M]⁺ calculated for C₁₂H₁₃NO 187.0997, found 187.0999.



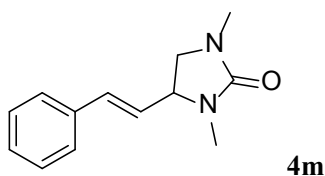
(E)-5-styrylpyrrolidin-2-one (41)

Brown solid; yield 13 % (12 mg); mp 92-93 °C (there is a little conformational isomer); ¹H NMR (400M Hz, CDCl₃) δ 7.38-7.24 (m, 5H), 6.54 (d, *J* = 15.6 Hz, 1H), 6.13 (dd, *J*₁ = 15.6 Hz, *J*₂ = 7.6 Hz, 1H), 6.10 (br, 1H), 4.36-4.31 (m, 1H), 2.45-2.34 (m, 3H), 1.97-1.91 (m, 1H); ¹³C NMR (100M Hz, CDCl₃) δ 178.3, 136.1, 131.1, 129.9, 128.7, 128.0, 126.5, 56.5, 30.0, 28.5; IR (neat) ν = 3243, 2925, 1684, 1494, 1451, 1419, 1348, 1258, 1182, 1072, 969, 914, 844, 751, 696, 645; HRMS (TOF MS EI⁺) [M]⁺ calculated for C₁₂H₁₃NO 187.0997, found 187.0996.



(Z)-1,3-dimethyl-4-styrylimidazolidin-2-one (3m)

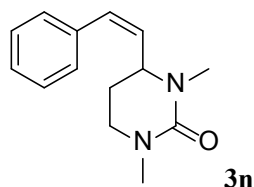
Light yellow liquid; yield 35 % (38 mg) (there is a little conformational isomer); ¹H NMR (400M Hz, CDCl₃) δ 7.33 (t, *J* = 7.2 Hz, 2H), 7.29 (t, *J* = 7.2 Hz, 1H), 7.19 (d, *J* = 7.2 Hz, 2H), 6.77 (d, *J* = 11.6 Hz, 1H), 5.60 (dd, *J*₁ = 9.6 Hz, *J*₂ = 11.6 Hz, 1H), 4.40-4.33 (m, 1H), 3.53 (t, *J* = 8.4 Hz, 1H), 3.04 (t, *J* = 8.4 Hz, 1H), 2.80 (s, 3H), 2.67 (s, 3H); ¹³C NMR (100M Hz, CDCl₃) δ 161.5, 136.0, 134.2, 130.5, 128.5, 128.4, 127.6, 53.3, 51.6, 31.4, 29.4; IR (neat) ν = 2869, 1694, 1601, 1496, 1443, 1397, 1364, 1317, 1243, 1127, 1076, 1029, 1001, 921, 892, 820, 758, 701, 649; HRMS (TOF MS EI⁺) [M]⁺ calculated for C₁₃H₁₆N₂O 216.1263, found 216.1261.



(E)-1,3-dimethyl-4-styrylimidazolidin-2-one (4m)

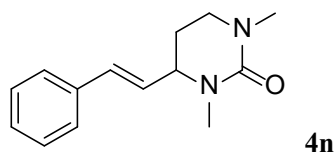
Light yellow liquid; yield 32 % (35 mg) (there is a little conformational isomer); ¹H NMR (400M

Hz, CDCl₃) δ 7.41-7.26 (m, 5H), 6.62 (d, $J = 16.0$ Hz, 1H), 6.06 (dd, $J_1 = 8.8$ Hz, $J_2 = 16.0$ Hz, 1H), 3.40-3.94 (m, 1H), 3.51 (t, $J = 8.4$ Hz, 1H), 3.03 (t, $J = 8.4$ Hz, 1H), 2.81 (s, 3H), 2.72 (s, 3H); ¹³C NMR (100M Hz, CDCl₃) δ 161.6, 136.0, 134.3, 128.7, 128.3, 127.4, 126.6, 59.0, 51.7, 31.3, 29.5; IR (neat) $\nu = 2923, 2863, 1693, 1497, 1444, 1397, 1370, 1254, 1127, 1075, 1030, 971, 897, 845, 753, 696$; HRMS (TOF MS EI⁺) [M]⁺ calculated for C₁₃H₁₆N₂O 216.1263, found 216.1262.



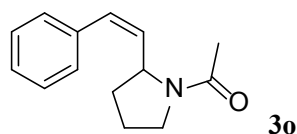
(Z)-1,3-dimethyl-4-styryltetrahydropyrimidin-2(1H)-one (3n)

Brown liquid; yield 44 % (51 mg) (there is a little conformational isomer); ¹H NMR (400M Hz, CDCl₃) δ 7.38-7.19 (m, 5H), 6.64 (d, $J = 12.0$ Hz, 1H), 5.68 (dd, $J_1 = 11.6$ Hz, $J_2 = 10.0$ Hz, 1H), 4.31-4.28 (m, 1H), 3.42-3.37 (m, 0.88H), 3.23-3.18 (m, 1.12H), 2.96-2.94 (m, 3.35H), 2.79 (s, 2.65H), 2.26-2.17 (m, 0.88H), 1.98-1.89 (m, 1.12H); ¹³C NMR (100M Hz, CDCl₃) δ 156.4, 156.3, 136.8, 136.3, 132.1, 131.5, 131.2, 129.8, 128.9, 128.52, 128.46, 128.2, 127.3, 126.9, 54.3, 47.9, 45.7, 45.3, 45.2, 35.8, 35.7, 33.8, 28.1, 22.3; IR (neat) $\nu = 3015, 2927, 2862, 1628, 1509, 1446, 1404, 1319, 1252, 1214, 1107, 1054, 972, 941, 918, 845, 804, 754, 700, 667$; HRMS (TOF MS EI⁺) [M]⁺ calculated for C₁₄H₁₈N₂O 230.1419, found 231.1370.



(E)-1,3-dimethyl-4-styryltetrahydropyrimidin-2(1H)-one (4n)

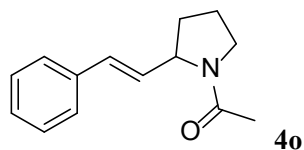
Brown liquid; yield 22 % (25 mg); ¹H NMR (400M Hz, CDCl₃) δ 7.39-7.23 (m, 5H), 6.45 (d, $J = 15.6$ Hz, 1H), 6.10 (dd, $J_1 = 15.6$ Hz, $J_2 = 6.4$ Hz, 1H), 4.01-3.98 (m, 1H), 3.40-3.34 (m, 1H), 3.14-3.09 (m, 1H), 2.97 (s, 3H), 2.96 (s, 3H), 2.26-2.17 (m, 1H), 1.89-1.82 (m, 1H); ¹³C NMR (100M Hz, CDCl₃) δ 156.4, 136.3, 131.4, 128.7, 128.5, 127.8, 126.5, 59.1, 44.4, 35.8, 34.6, 27.3; IR (neat) $\nu = 2928, 1628, 1512, 1450, 1405, 1330, 1231, 1108, 1051, 972, 755, 696, 669$; HRMS (TOF MS EI⁺) [M]⁺ calculated for C₁₄H₁₈N₂O 230.1419, found 231.1371.



(Z)-1-(2-styrylpyrrolidin-1-yl)ethanone (3o)

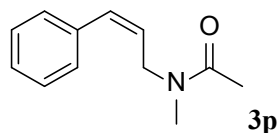
Brown liquid; yield 43 % (46 mg); the ratio of two conformational isomers is about 0.77 : 0.23; ¹H NMR (400M Hz, CDCl₃) δ 7.45-7.20 (m, 5H), 6.55 (d, $J = 11.6$ Hz, 0.77H), 6.45 (d, $J = 11.6$ Hz, 0.23H), 5.65 (dd, $J_1 = 11.6$ Hz, $J_2 = 9.6$ Hz, 0.77H), 5.56 (dd, $J_1 = 11.6$ Hz, $J_2 = 8.8$ Hz, 0.23H), 5.05-5.01 (m, 0.23H), 4.70-4.65 (m, 0.77H), 3.64-3.52 (m, 1.77H), 3.48-3.42 (m, 0.23H), 2.40-2.30 (m, 0.77H), 2.04-1.86 (m, 3.92H), 1.75 (s, 2.31H); ¹³C NMR (100M Hz, CDCl₃) δ 170.0, 169.2, 137.1, 136.1, 133.4, 132.8, 129.6, 129.0, 128.9, 128.6, 128.4, 128.2, 127.5, 126.7, 55.8, 55.1, 47.9, 46.2, 34.2, 32.7, 24.7, 23.3, 23.0, 22.1; IR (neat) $\nu = 2973, 2876, 1642, 1493, 1414, 1354, 1245, 1187, 1072, 1033, 1002, 917, 831, 794, 770, 736, 701, 671$; HRMS (TOF MS

EI^+ $[\text{M}]^+$ calculated for $\text{C}_{14}\text{H}_{17}\text{NO}$ 215.1310, found 215.1312.



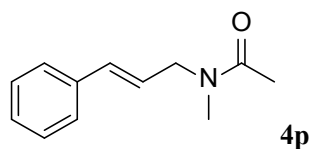
(E)-1-(2-styrylpyrrolidin-1-yl)ethanone (4o)

Light yellow liquid; yield 29 % (31 mg); the ratio of two conformational isomers is about 0.70 : 0.30; ^1H NMR (400M Hz, CDCl_3) δ 7.38-7.19 (m, 5H), 6.44-6.37 (m, 1H), 6.14 (dd, $J_1 = 16.0$ Hz, $J_2 = 6.0$ Hz, 1H), 4.85-4.82 (m, 0.30H), 4.50-4.47 (m, 0.70H), 3.65-3.53 (m, 1.70H), 3.50-3.43 (m, 0.30H), 2.22-2.15 (m, 0.70H), 2.11 (s, 0.90H), 2.07 (s, 2.10H), 2.06-1.88 (m, 3.30H); ^{13}C NMR (100M Hz, CDCl_3) δ 170.0, 169.2, 136.9, 136.2, 130.1, 129.53, 129.49, 129.3, 128.7, 128.4, 127.9, 127.3, 126.44, 126.41, 60.2, 57.9, 47.7, 46.2, 33.2, 31.0, 23.9, 22.9, 22.5, 22.1; IR (neat) $\nu = 2970$, 2876, 1634, 1415, 1355, 1253, 1189, 1072, 1033, 970, 916, 748, 696, 672, 649; HRMS (TOF MS EI^+) $[\text{M}]^+$ calculated for $\text{C}_{14}\text{H}_{17}\text{NO}$ 215.1310, found 215.1311.



(Z)-N-methyl-N-(3-phenylallyl)acetamide (3p)

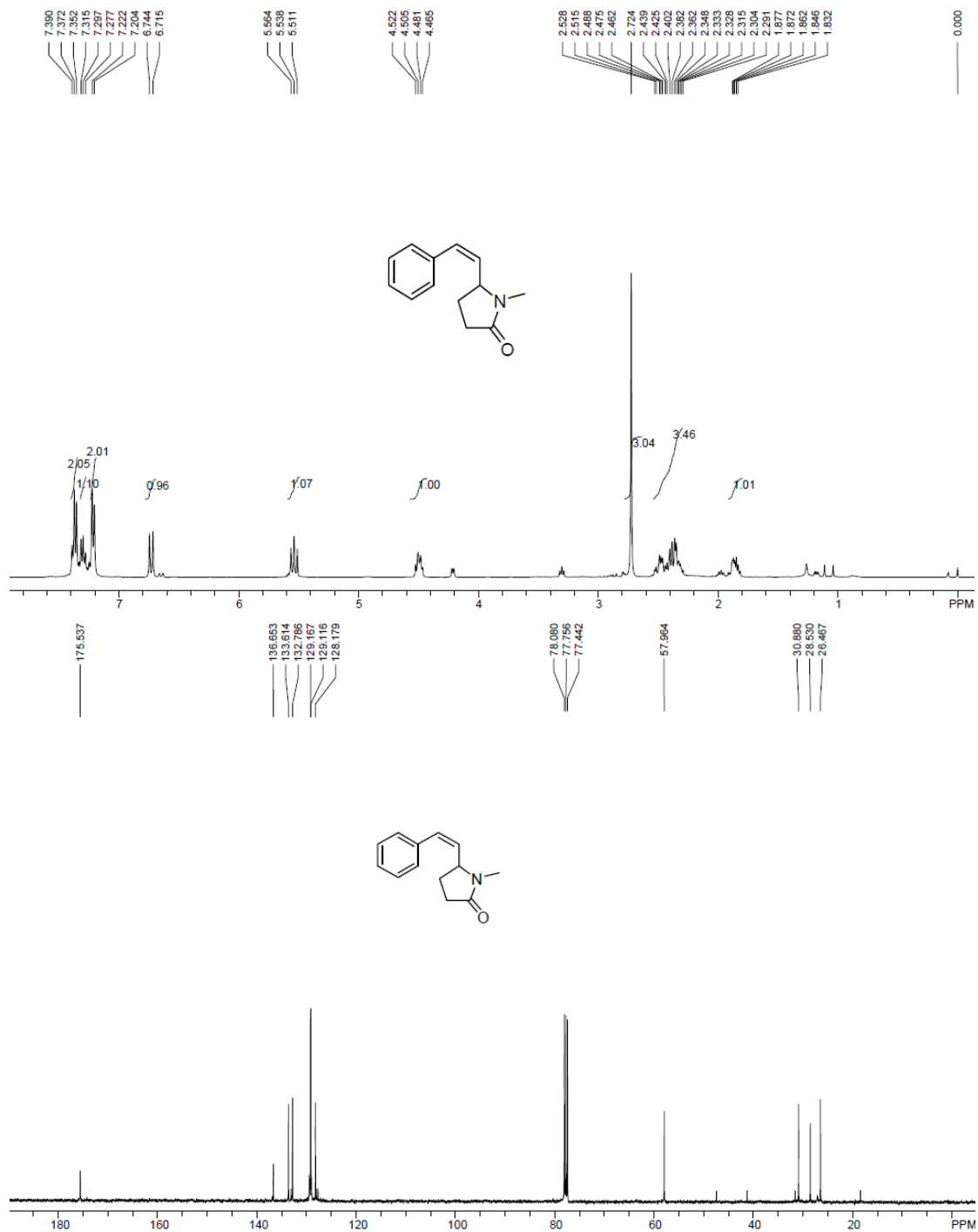
Light yellow liquid; yield 23 % (22 mg); the ratio of two conformational isomers is about 0.50 : 0.50; ^1H NMR (400M Hz, CDCl_3) δ 7.40-7.19 (m, 5H), 6.69-6.64 (m, 1H), 5.66-5.55 (m, 1H), 4.33 (d, $J = 6.0$ Hz, 1H), 4.17 (d, $J = 5.6$ Hz, 1H), 2.91 (s, 1.5H), 2.88 (s, 1.5H), 2.09 (s, 1.5H), 1.99 (s, 1.5H); ^{13}C NMR (100M Hz, CDCl_3) δ 170.55, 170.46, 136.5, 136.0, 132.3, 132.1, 128.8, 128.7, 128.5, 128.3, 128.2, 127.5, 127.3, 127.1, 48.9, 45.0, 35.5, 33.4, 21.8, 21.4; IR (neat) $\nu = 3020$, 2928, 1643, 1487, 1400, 1361, 1329, 1281, 1240, 1135, 1076, 1018, 929, 810, 769, 699; HRMS (TOF MS EI^+) $[\text{M}]^+$ calculated for $\text{C}_{12}\text{H}_{15}\text{NO}$ 189.1154, found 189.1152.



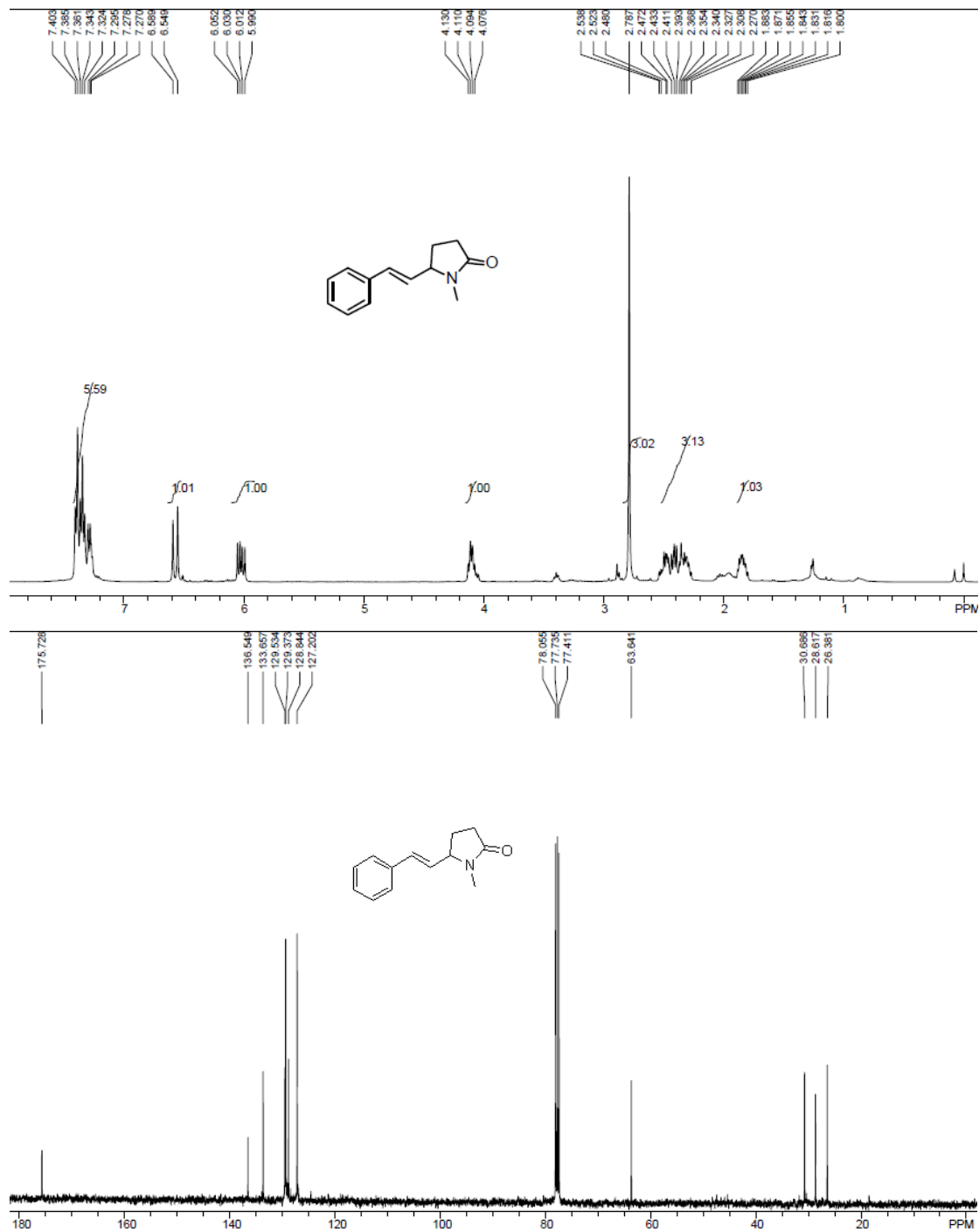
(E)-N-methyl-N-(3-phenylallyl)acetamide (4p)

Light yellow liquid; yield 17 % (16 mg); the ratio of two conformational isomers is about 0.50 : 0.50; ^1H NMR (400M Hz, CDCl_3) δ 7.39-7.23 (m, 5H), 6.52-6.46 (m, 1H), 6.19-6.11 (m, 1H), 4.16 (d, $J = 6.4$ Hz, 1H), 4.07 (d, $J = 5.2$ Hz, 1H), 3.00 (s, 1.5H), 2.99 (s, 1.5H), 2.16 (s, 1.5H), 2.14 (s, 1.5H); ^{13}C NMR (100M Hz, CDCl_3) δ 170.8, 170.5, 136.6, 136.1, 132.8, 131.8, 128.7, 128.6, 128.0, 127.7, 126.41, 126.40, 124.6, 123.7, 52.6, 49.3, 35.5, 33.5, 21.9, 21.4; IR (neat) $\nu = 2927$, 1635, 1488, 1401, 1358, 1287, 1234, 1136, 1018, 969, 760, 733, 694; HRMS (TOF MS EI^+) $[\text{M}]^+$ calculated for $\text{C}_{12}\text{H}_{15}\text{NO}$ 189.1154, found 189.1155.

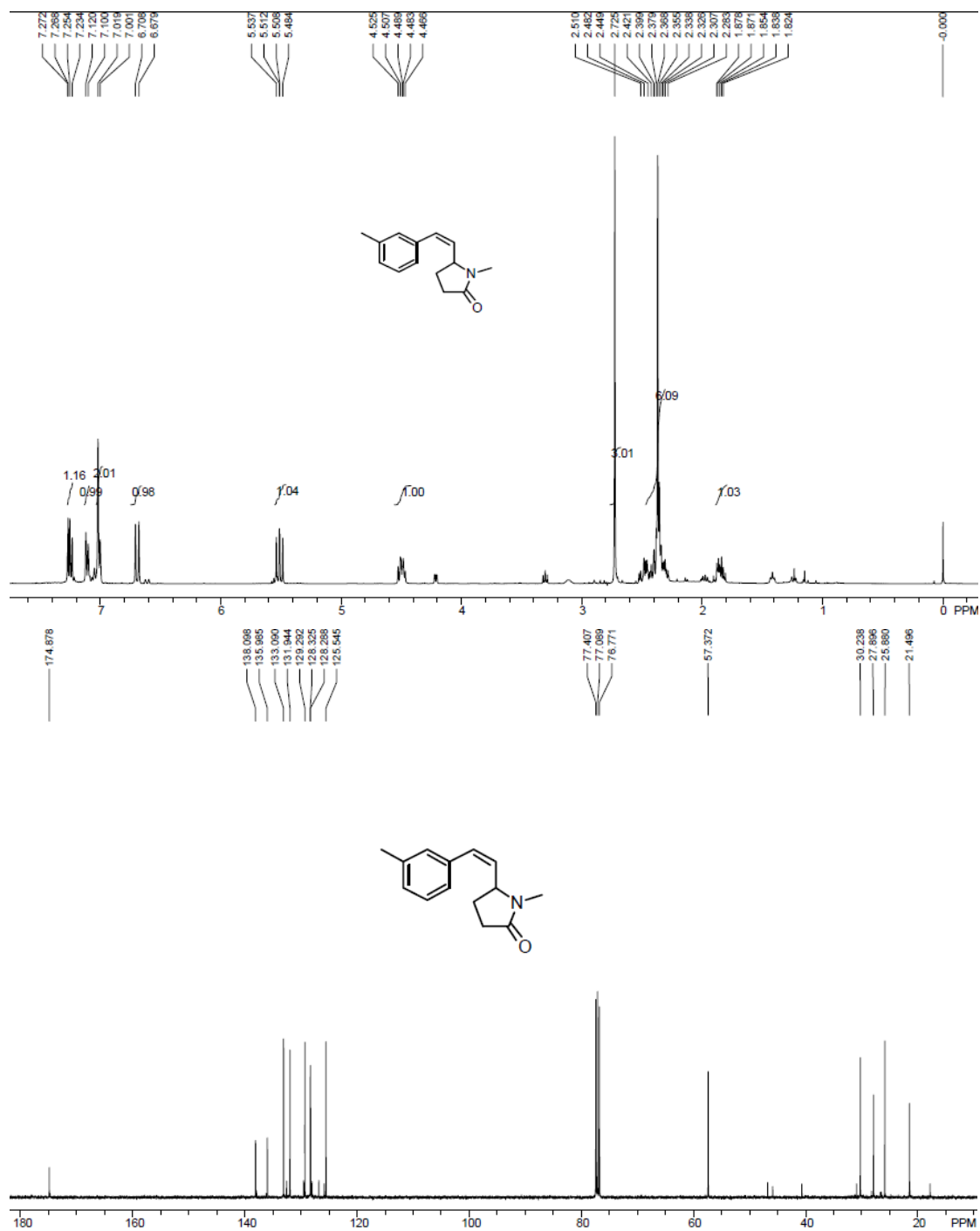
(Z)-1-methyl-5-styrylpyrrolidin-2-one (3a)



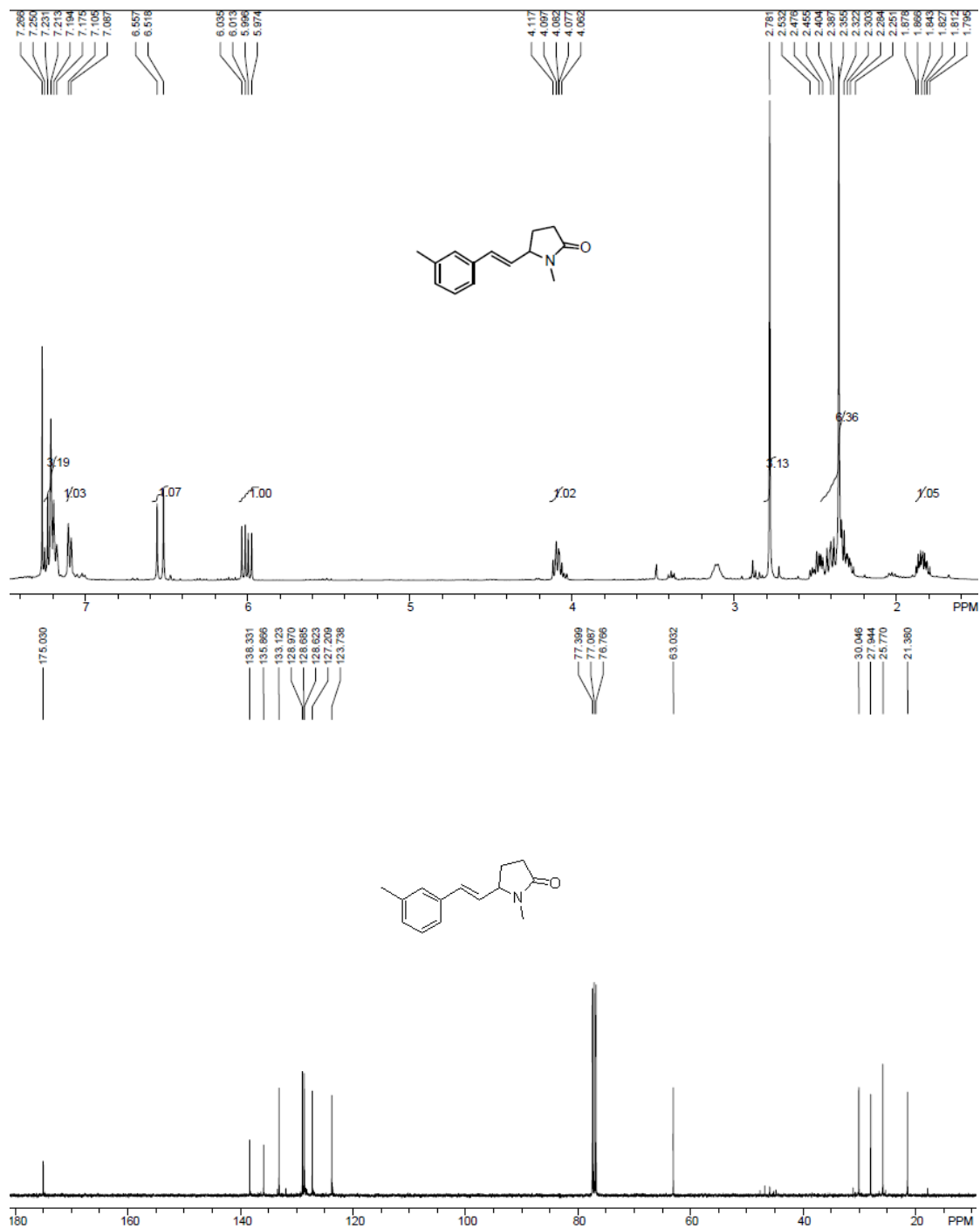
(E)-1-methyl-5-styrylpyrrolidin-2-one (4a)



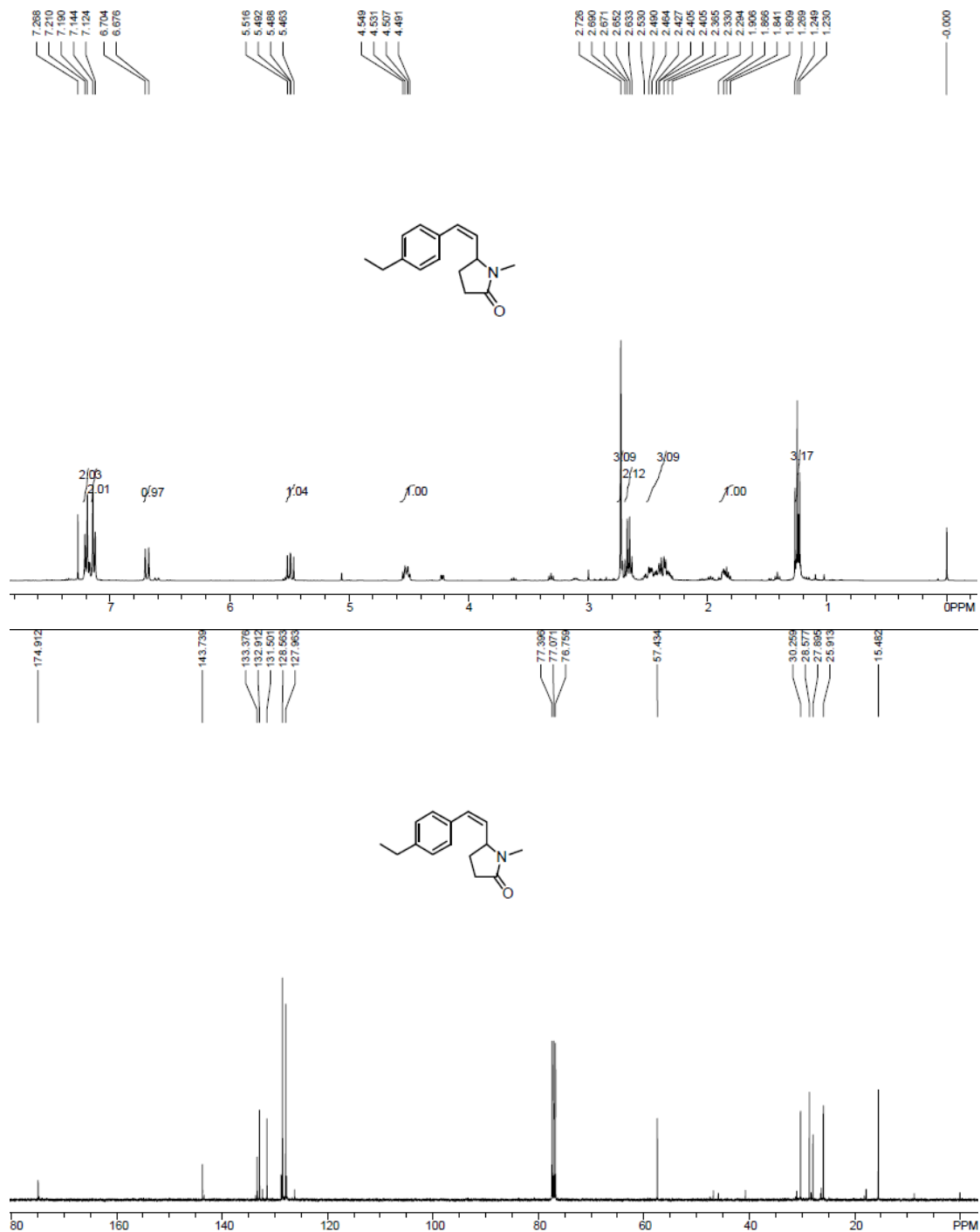
(Z)-1-methyl-5-(3-methylstyryl)pyrrolidin-2-one (3b)



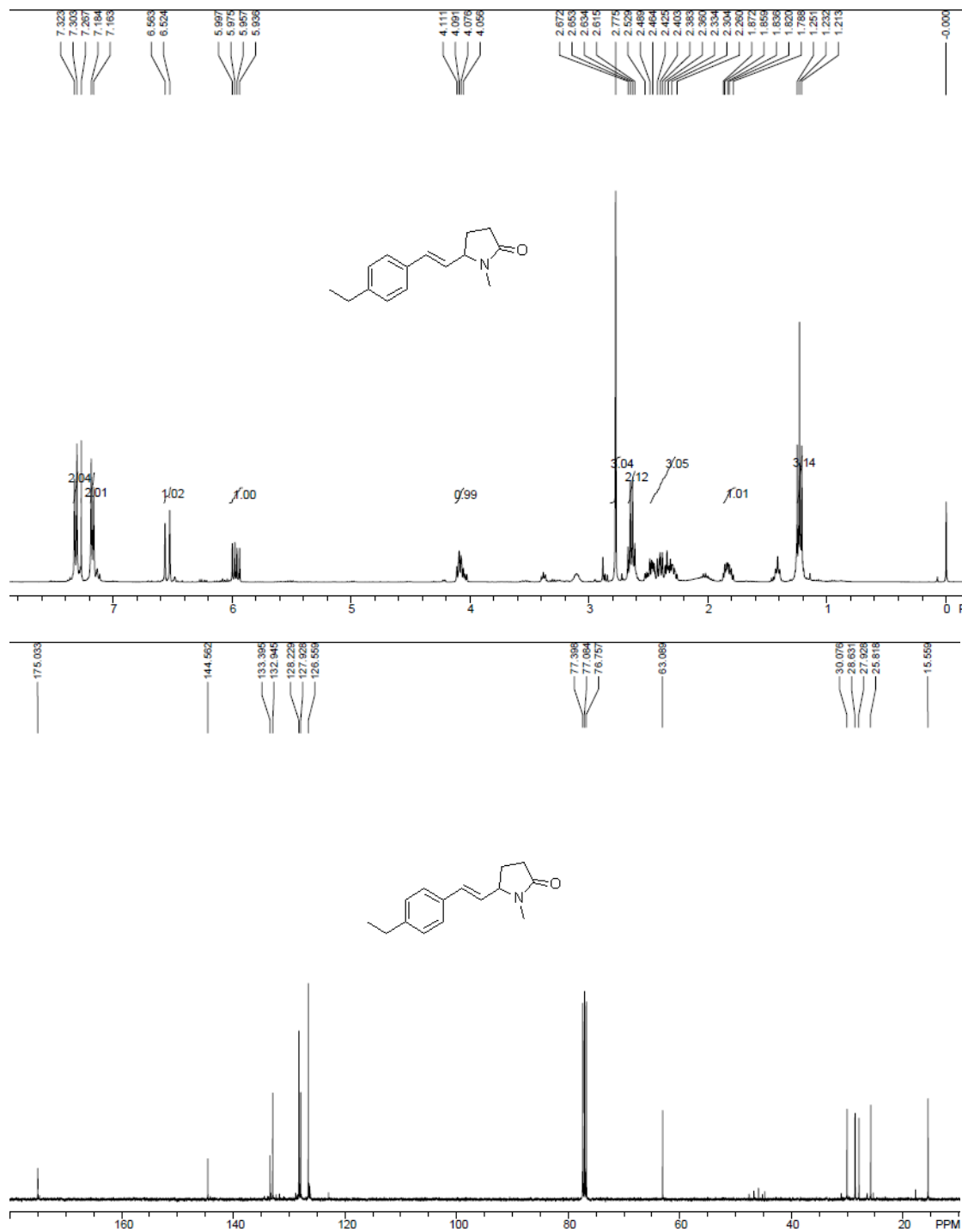
(E)-1-methyl-5-(3-methylstyryl)pyrrolidin-2-one (4b)



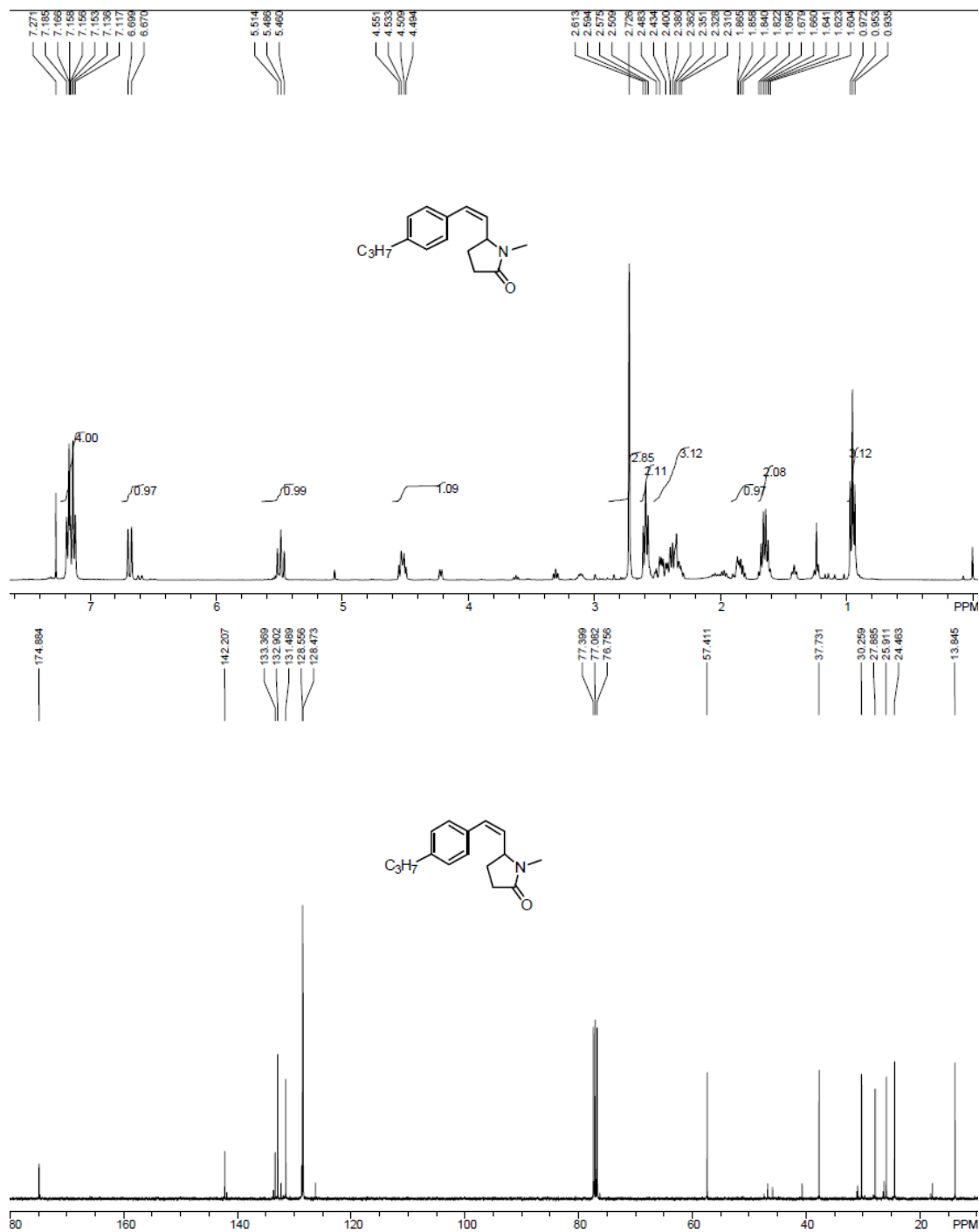
(Z)-5-(4-ethylstyryl)-1-methylpyrrolidin-2-one (3c)



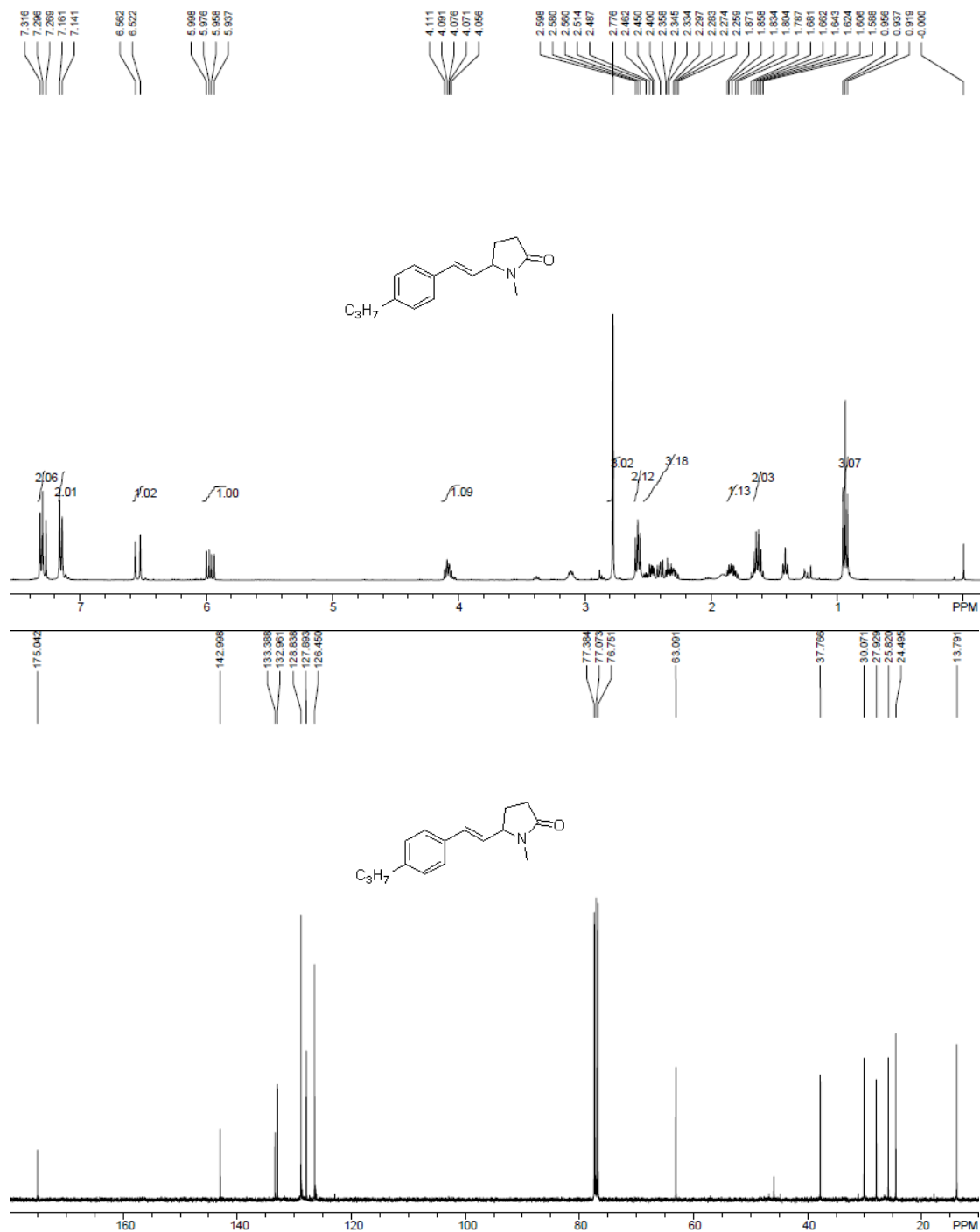
(E)-5-(4-ethylstyryl)-1-methylpyrrolidin-2-one (4c)



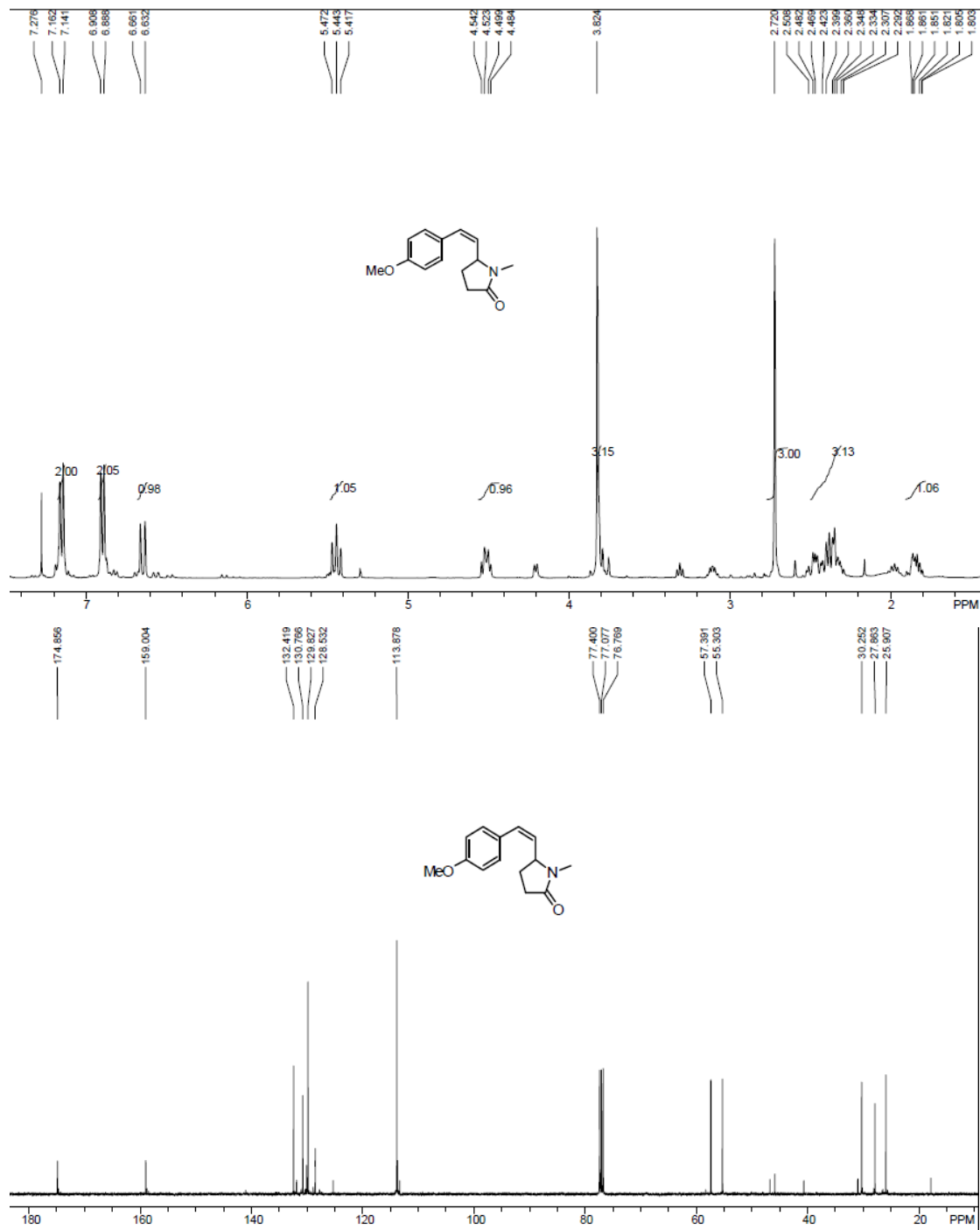
(Z)-1-methyl-5-(4-propylstyryl)pyrrolidin-2-one (3d)



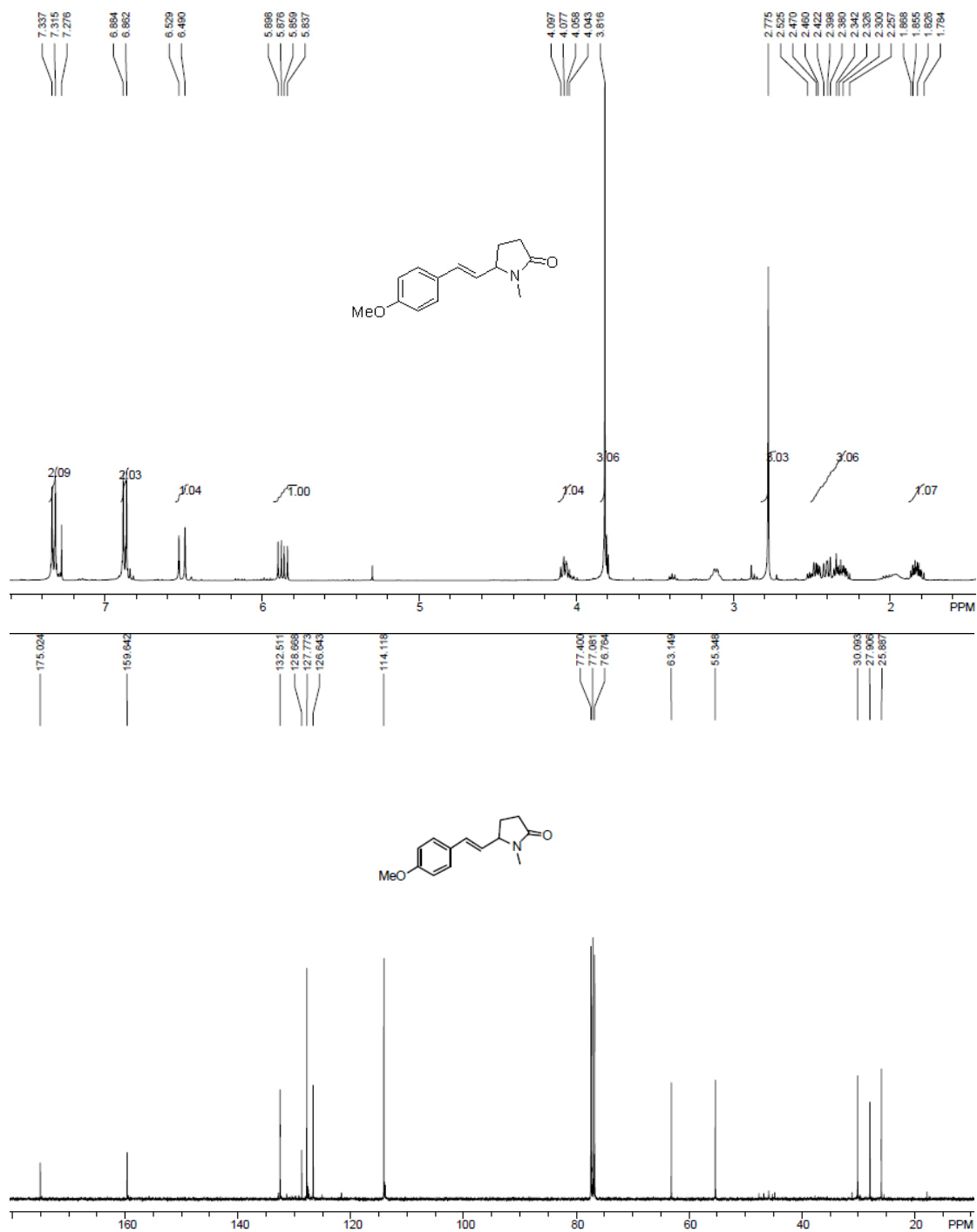
(E)-1-methyl-5-(4-propylstyryl)pyrrolidin-2-one (4d)



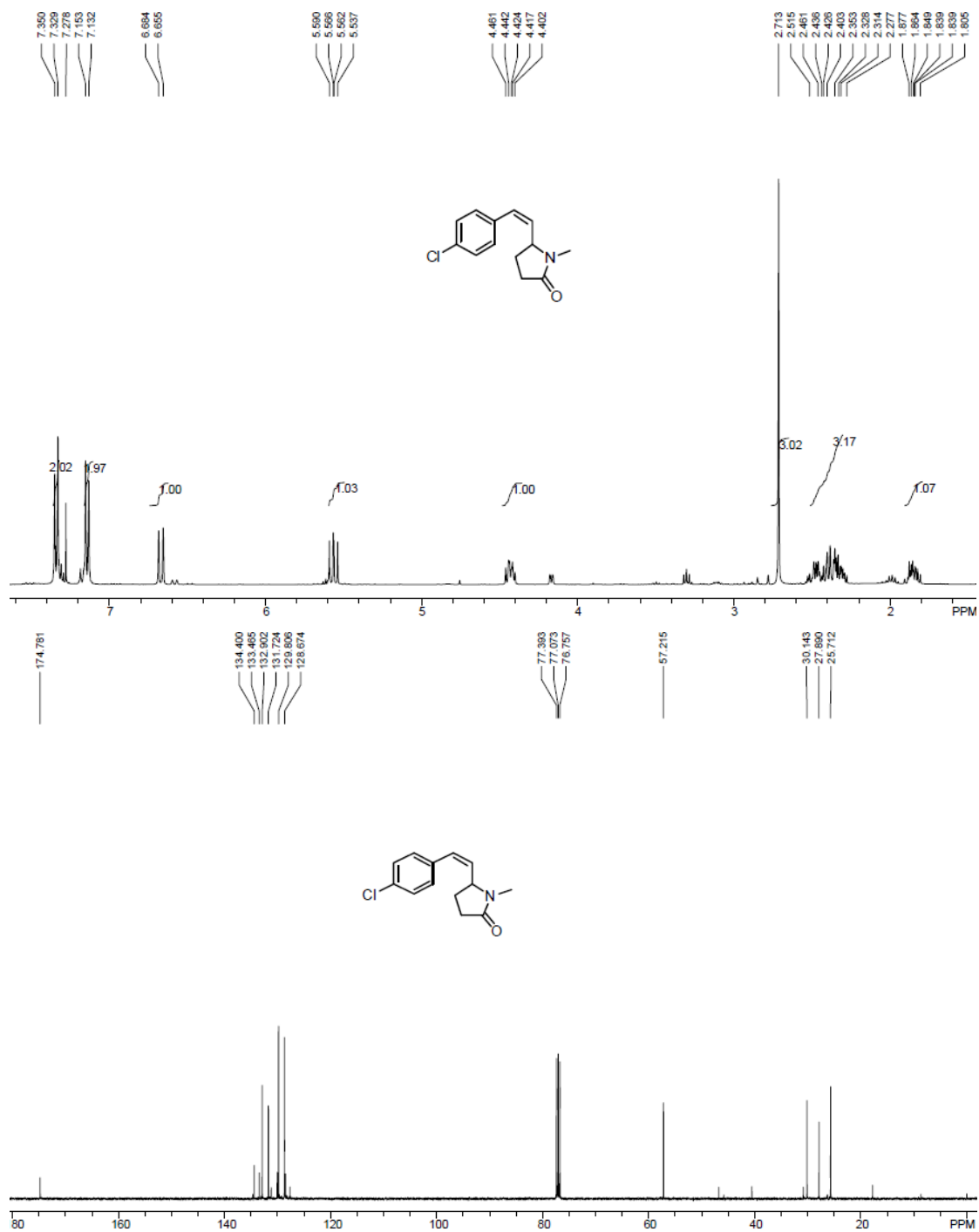
(Z)-5-(4-methoxystyryl)-1-methylpyrrolidin-2-one (3e)



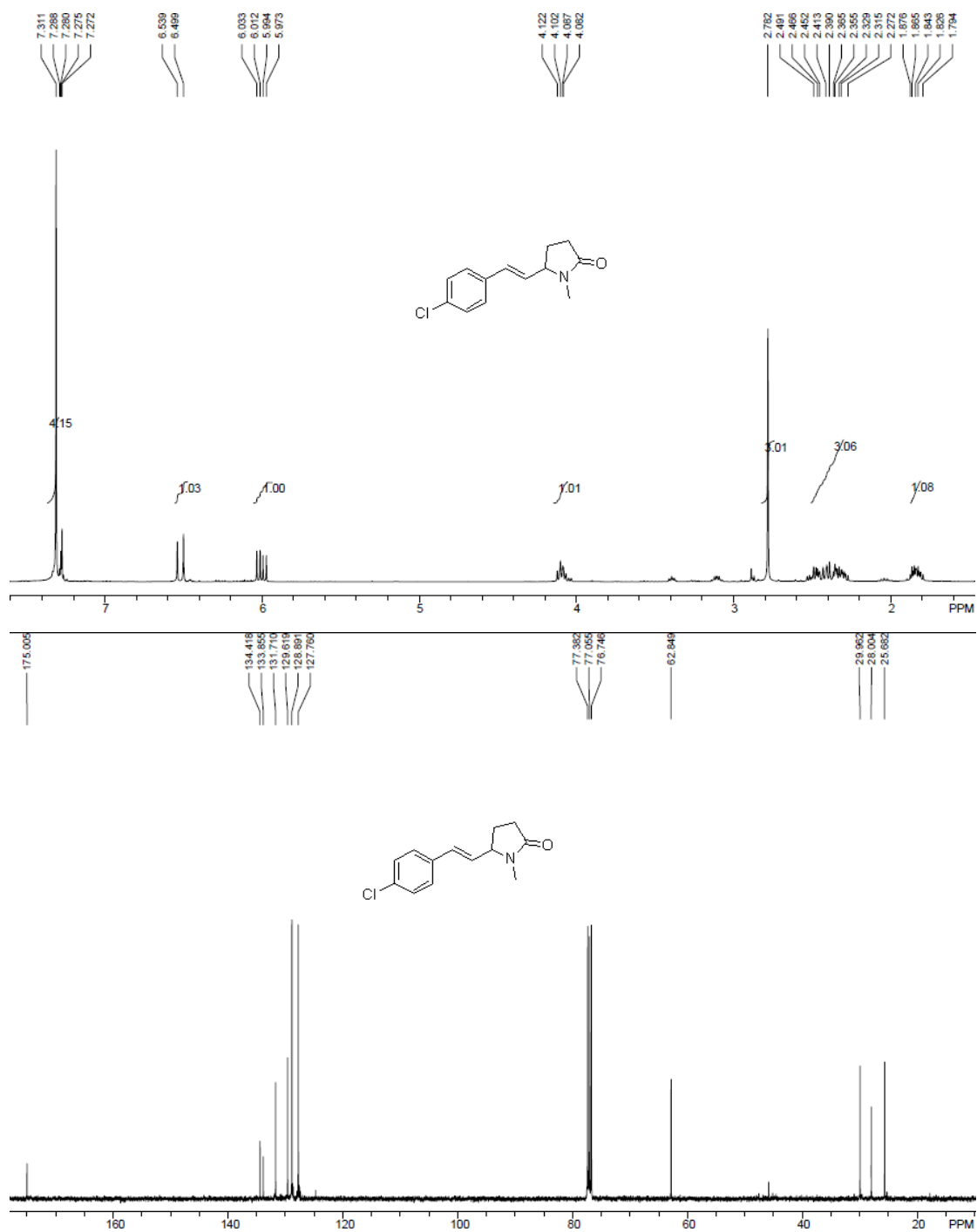
(E)-5-(4-methoxystyryl)-1-methylpyrrolidin-2-one (4e)



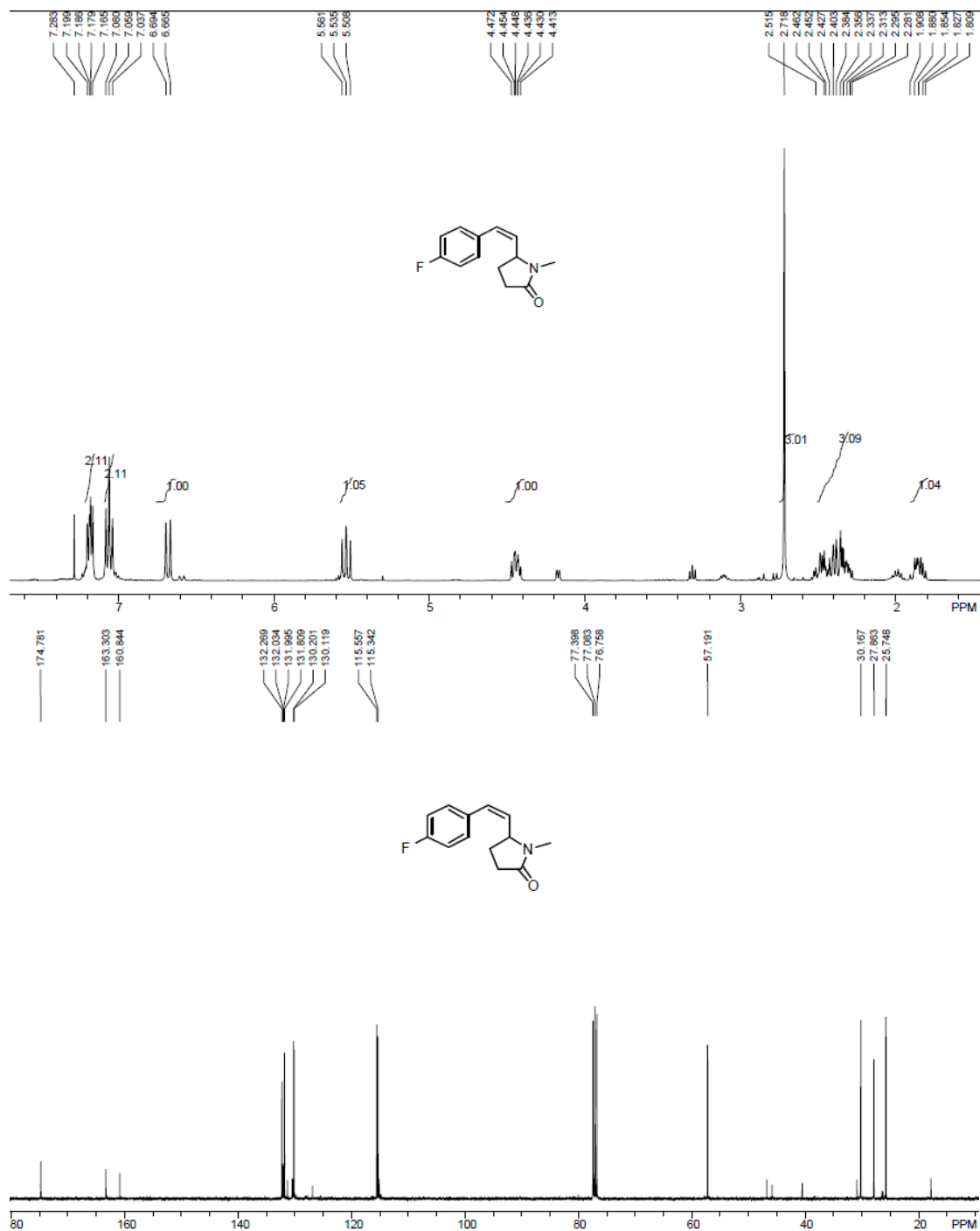
(Z)-5-(4-chlorostyryl)-1-methylpyrrolidin-2-one (3f)



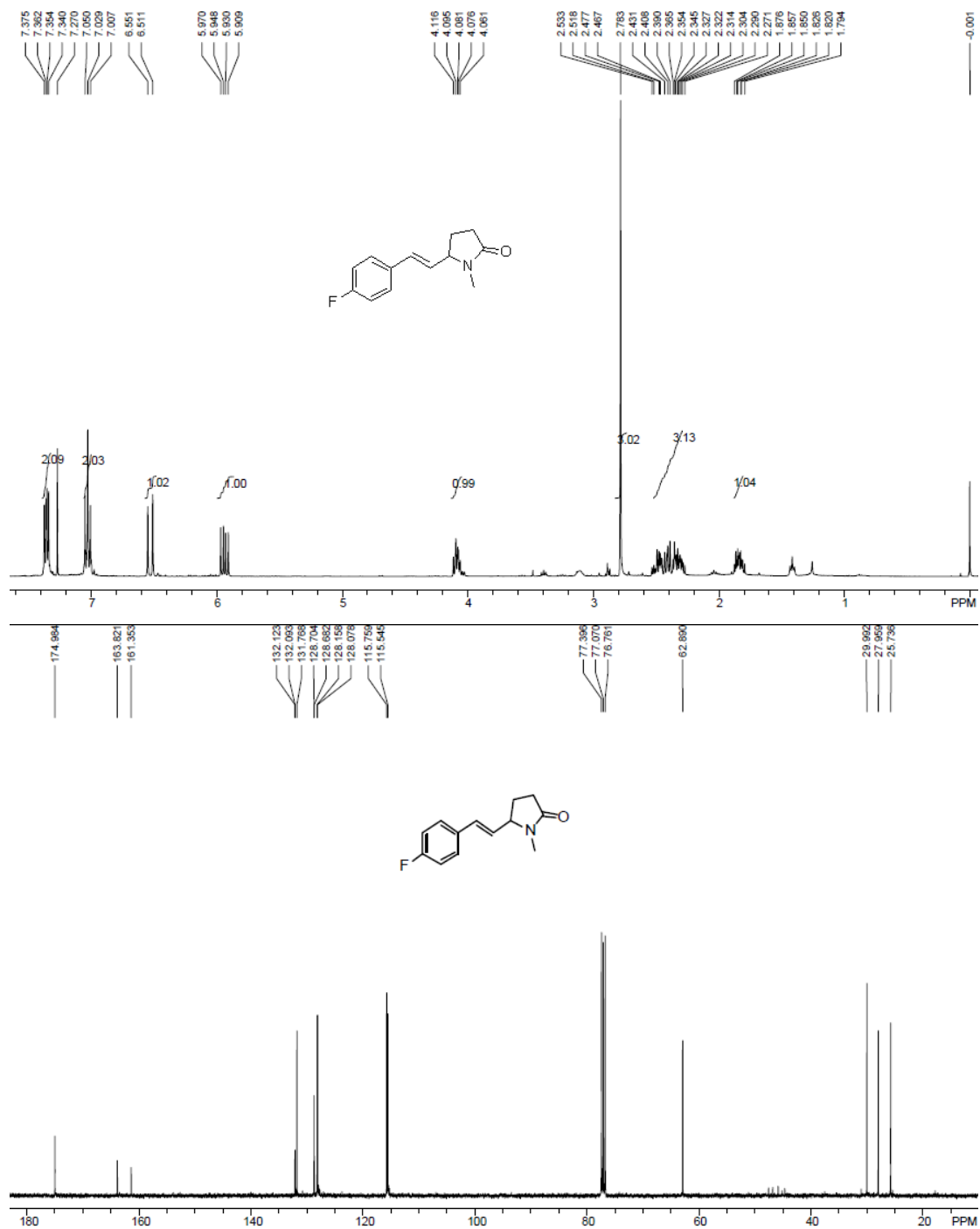
(E)-5-(4-chlorostyryl)-1-methylpyrrolidin-2-one (4f)



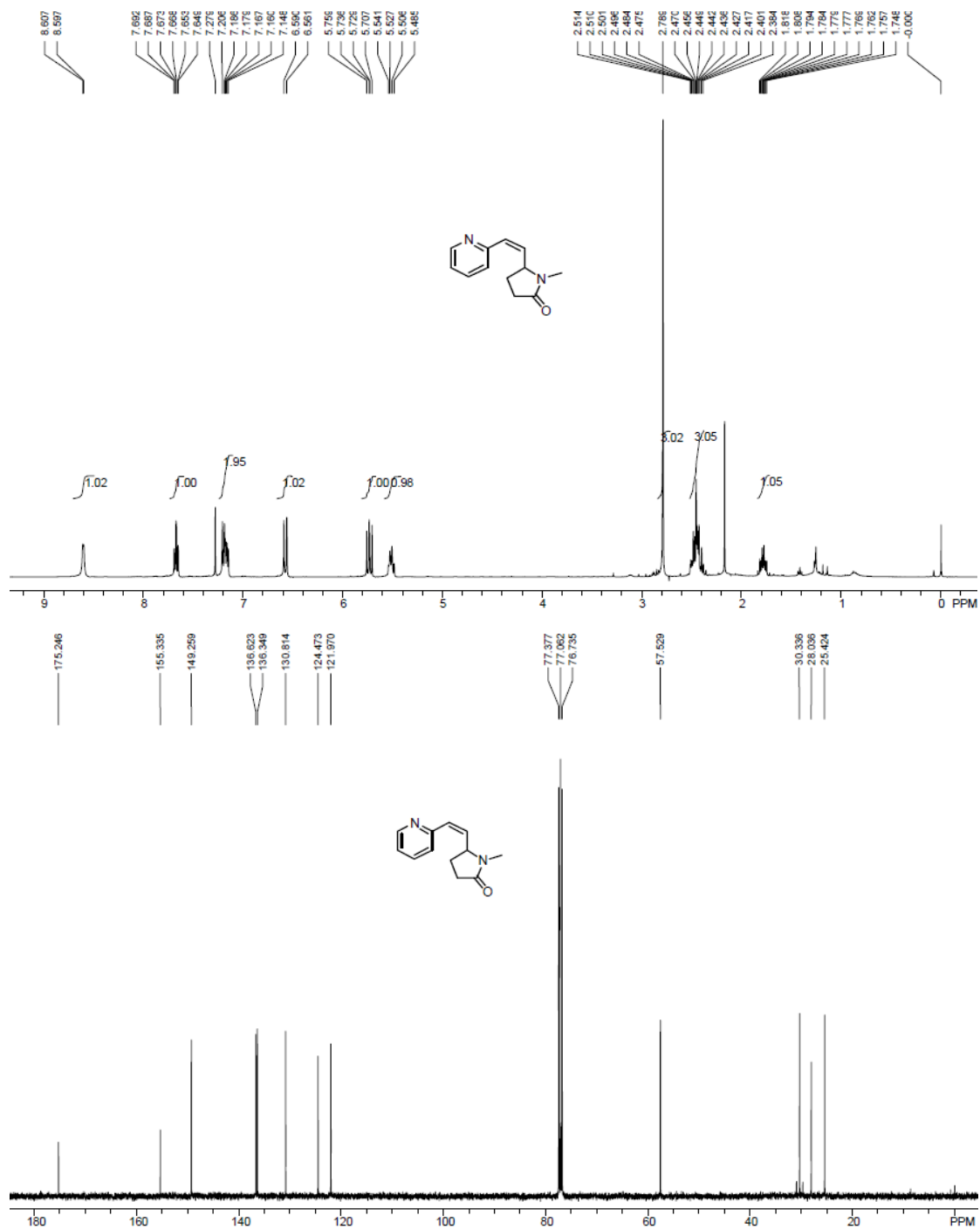
(Z)-5-(4-fluorostyryl)-1-methylpyrrolidin-2-one (3g)



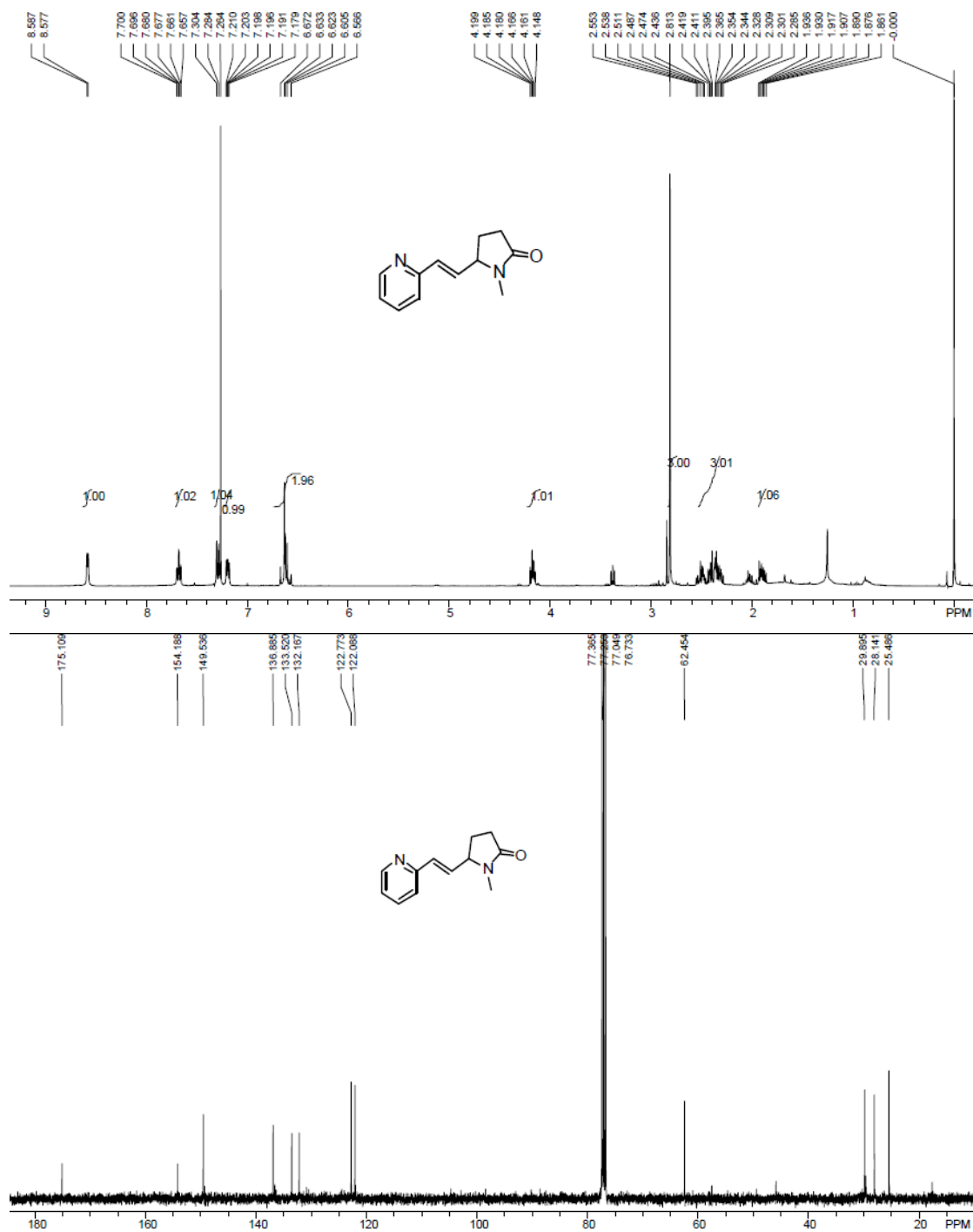
(E)-5-(4-fluorostyryl)-1-methylpyrrolidin-2-one (4g)



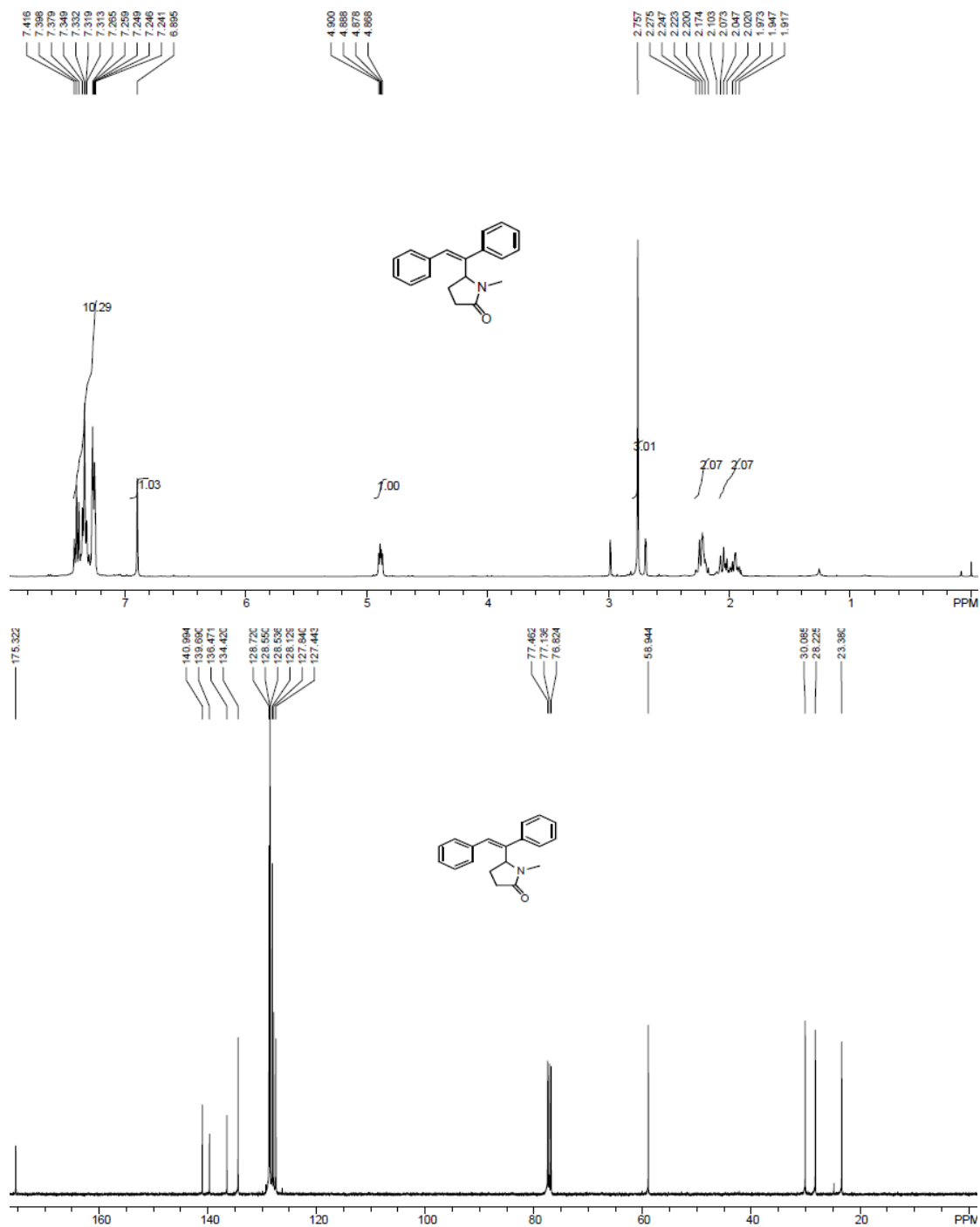
(Z)-1-methyl-5-(2-(pyridin-2-yl)vinyl)pyrrolidin-2-one (3h)



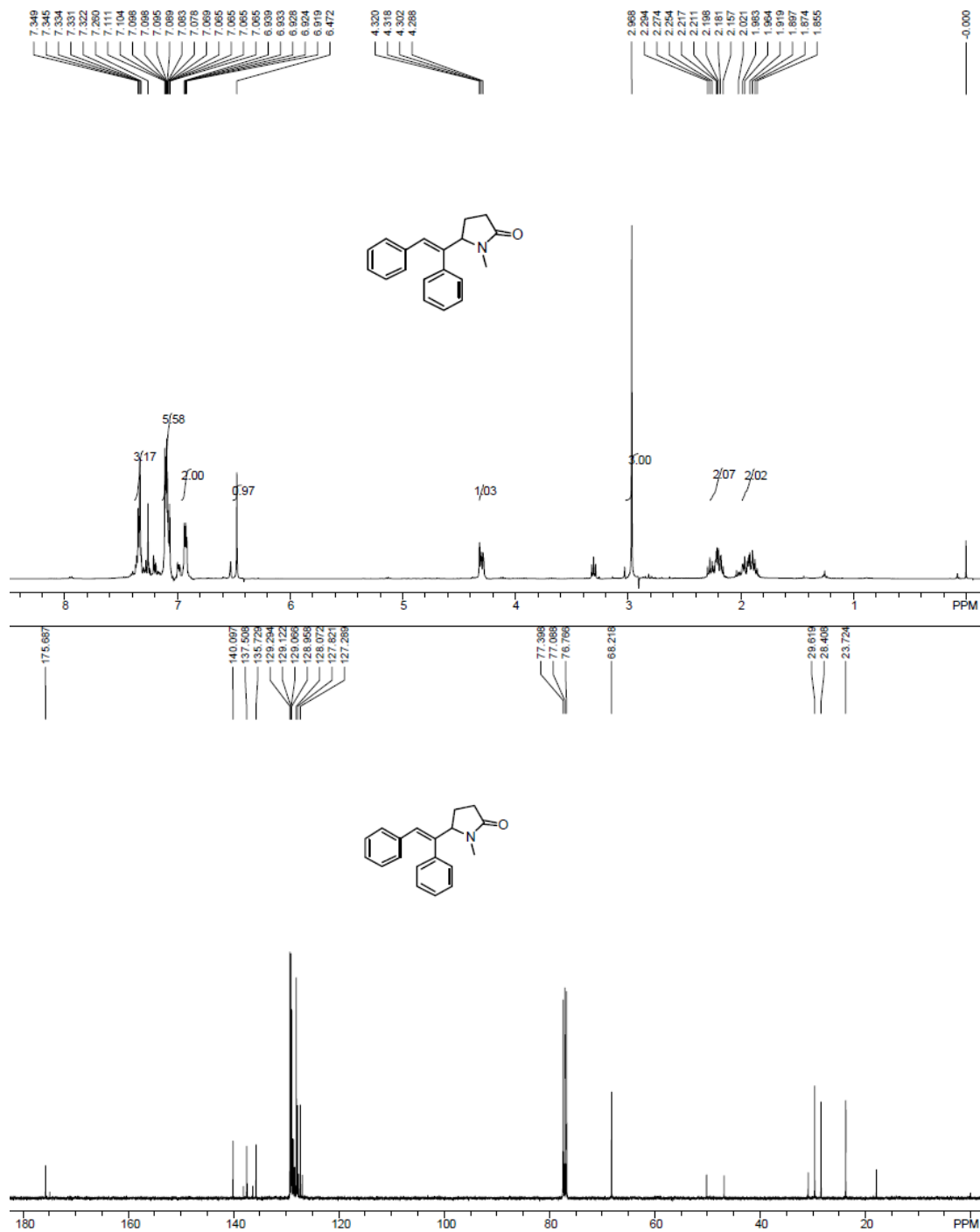
(E)-1-methyl-5-(2-(pyridin-2-yl)vinyl)pyrrolidin-2-one (4h)



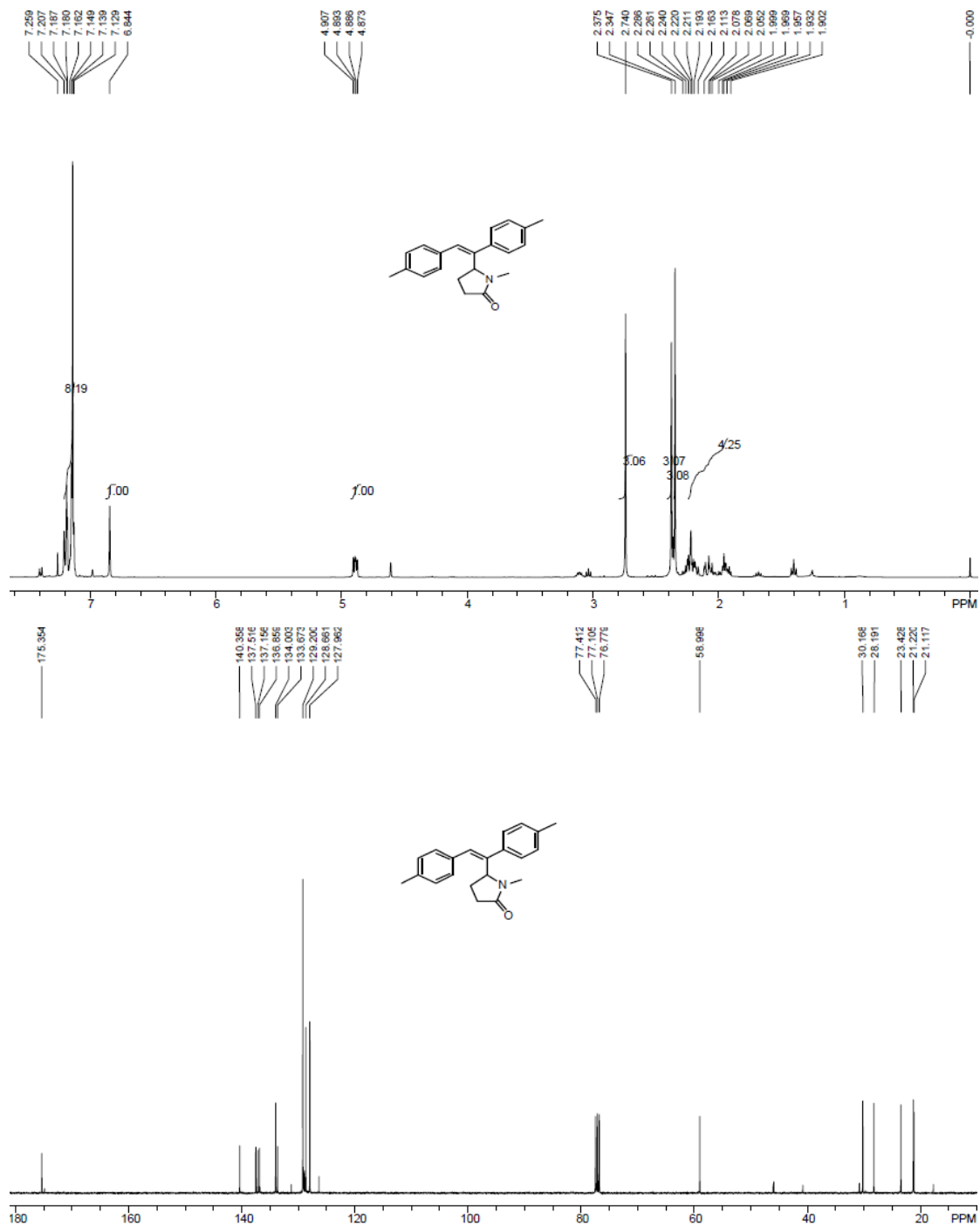
(Z)-5-(1,2-diphenylvinyl)-1-methylpyrrolidin-2-one (3i)



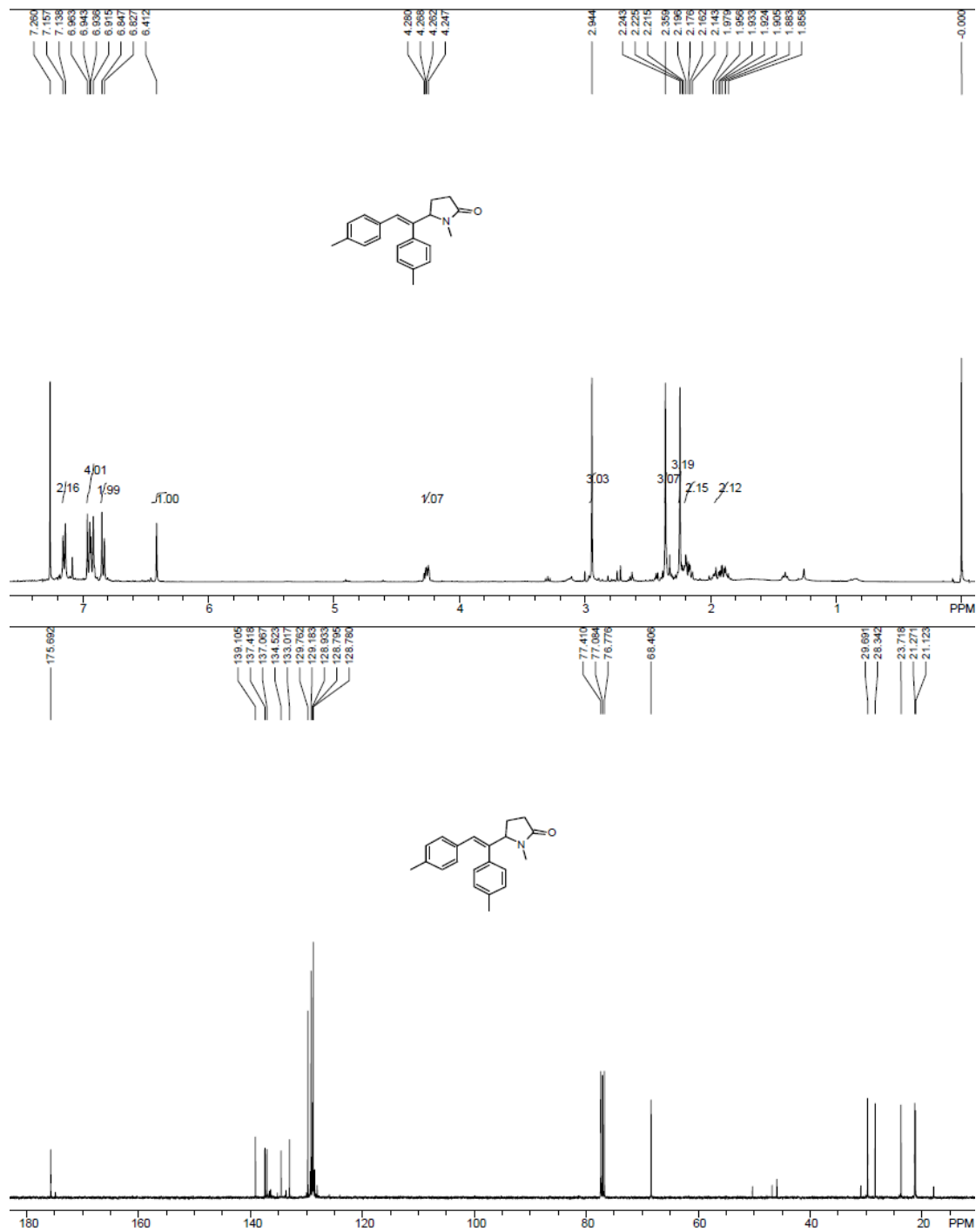
(E)-5-(1,2-diphenylvinyl)-1-methylpyrrolidin-2-one (4i)



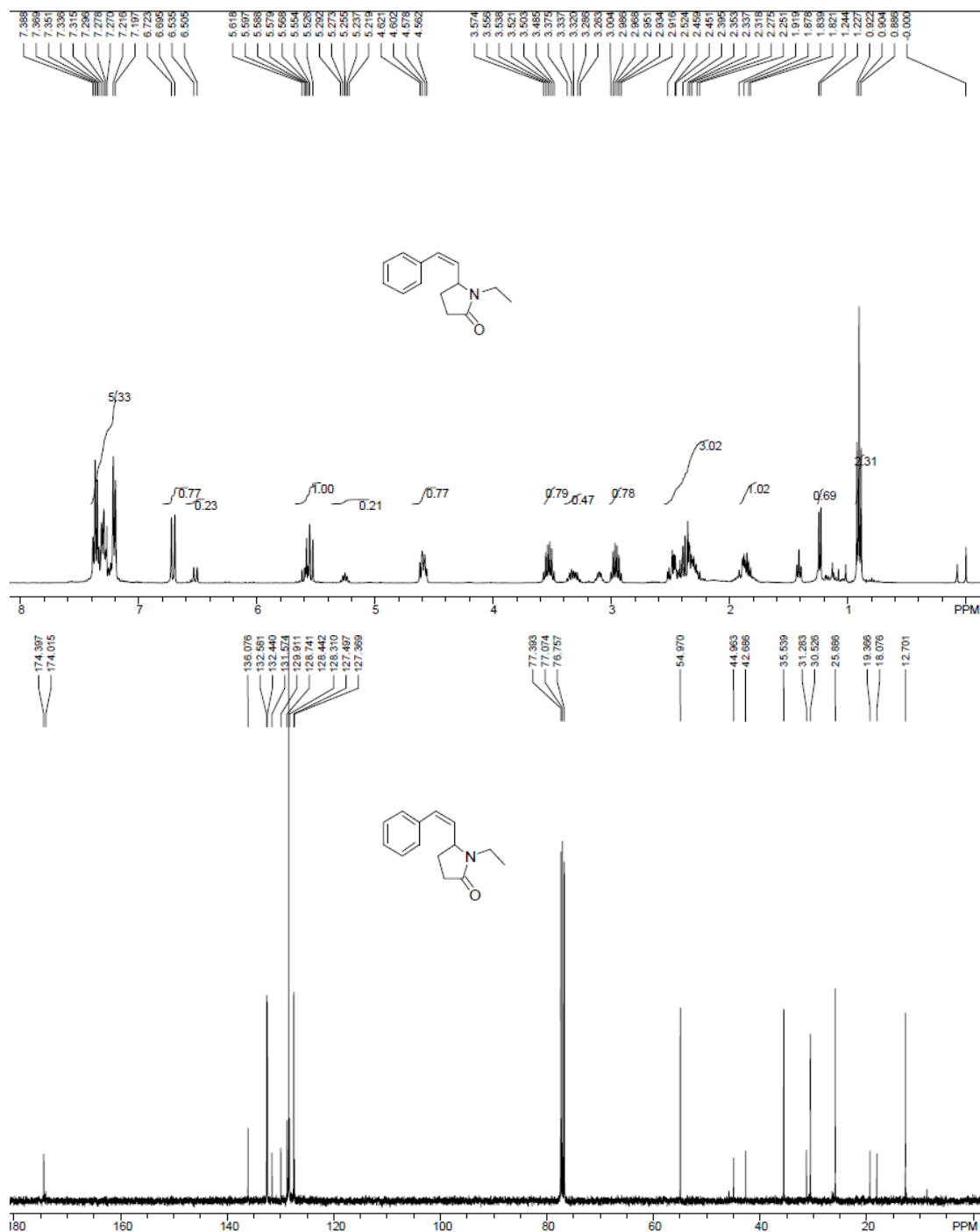
(Z)-5-(1,2-di-p-tolylvinyl)-1-methylpyrrolidin-2-one (3j)



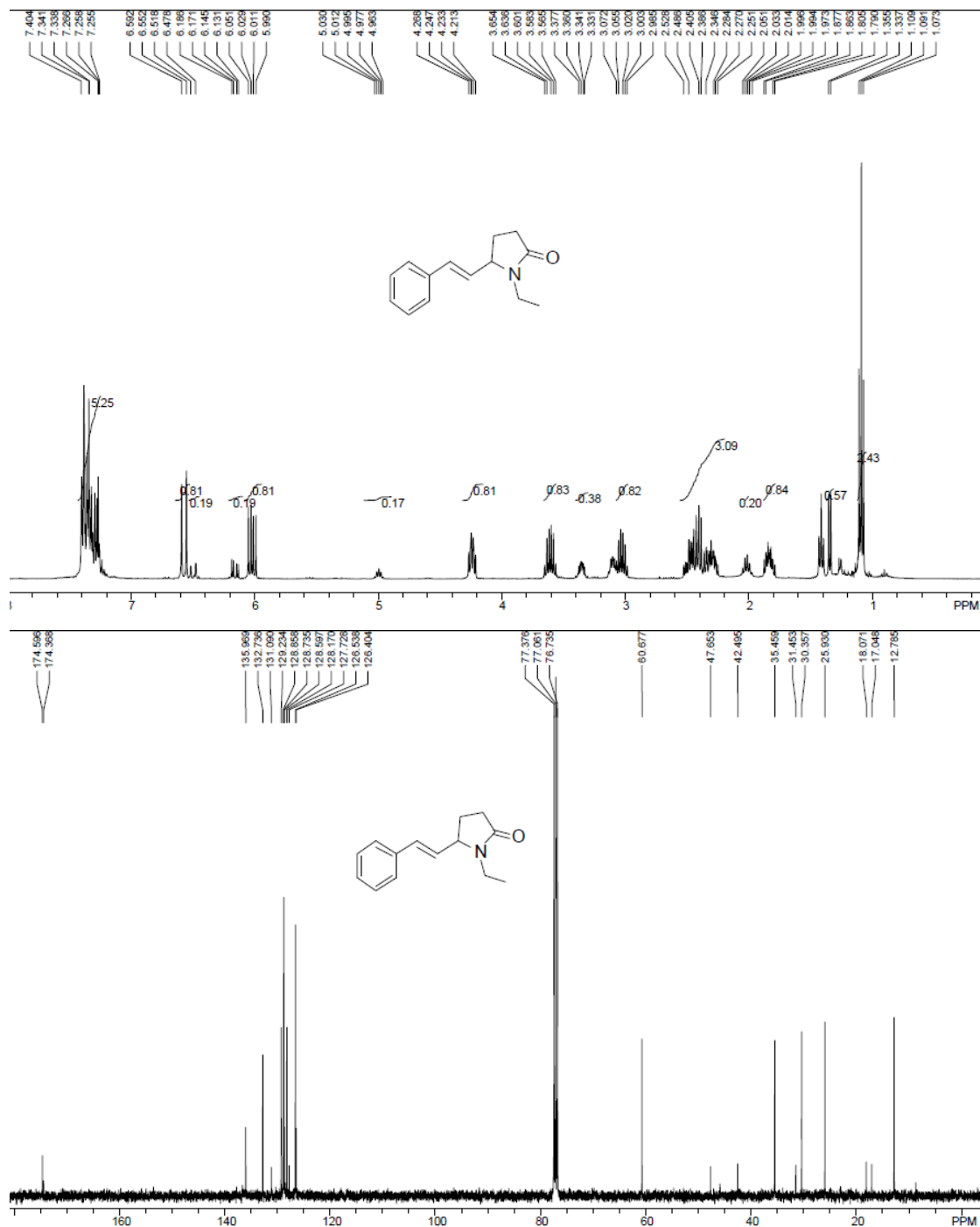
(E)-5-(1,2-di-p-tolylvinyl)-1-methylpyrrolidin-2-one (4j)



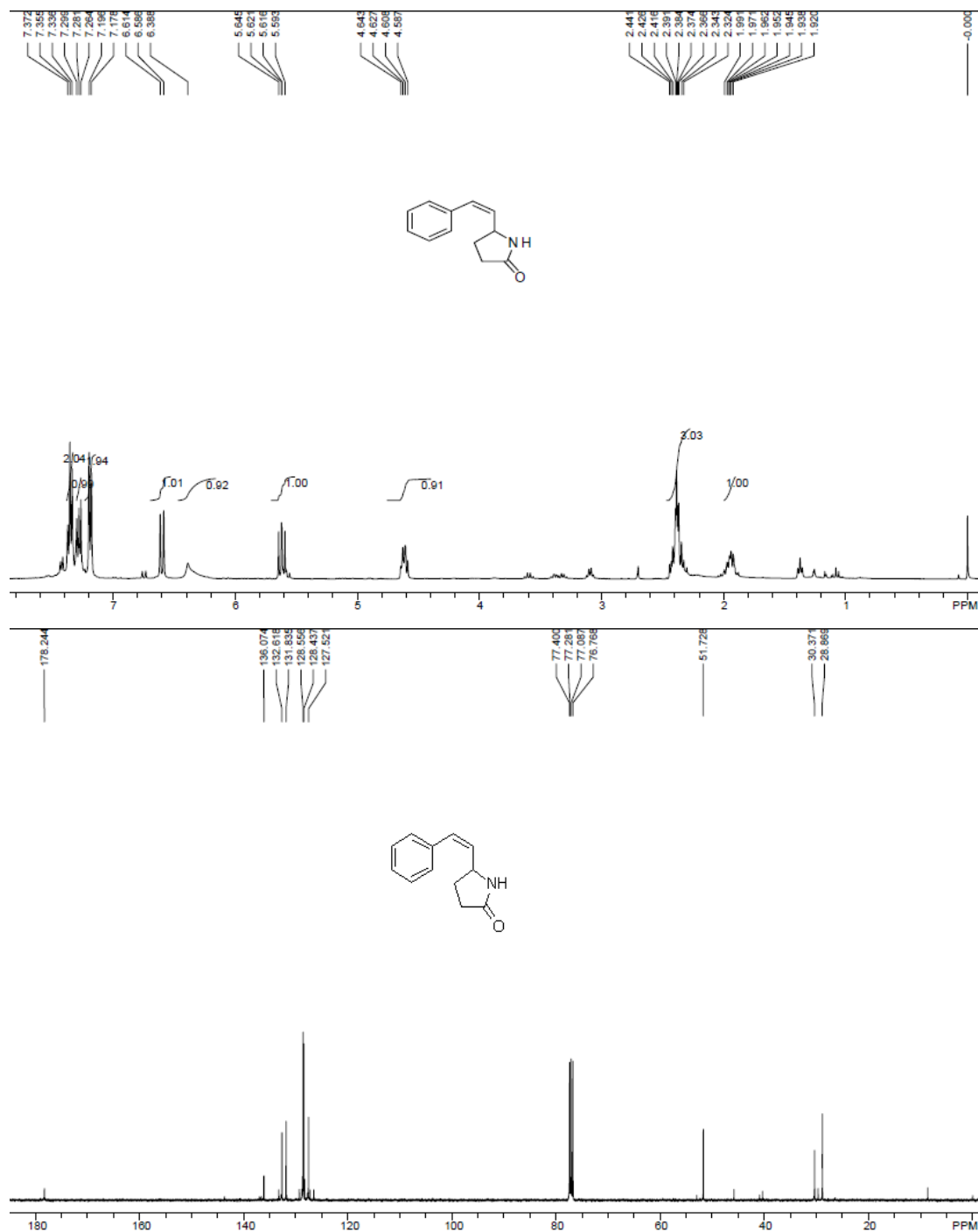
(Z)-1-ethyl-5-styrylpyrrolidin-2-one (3k)



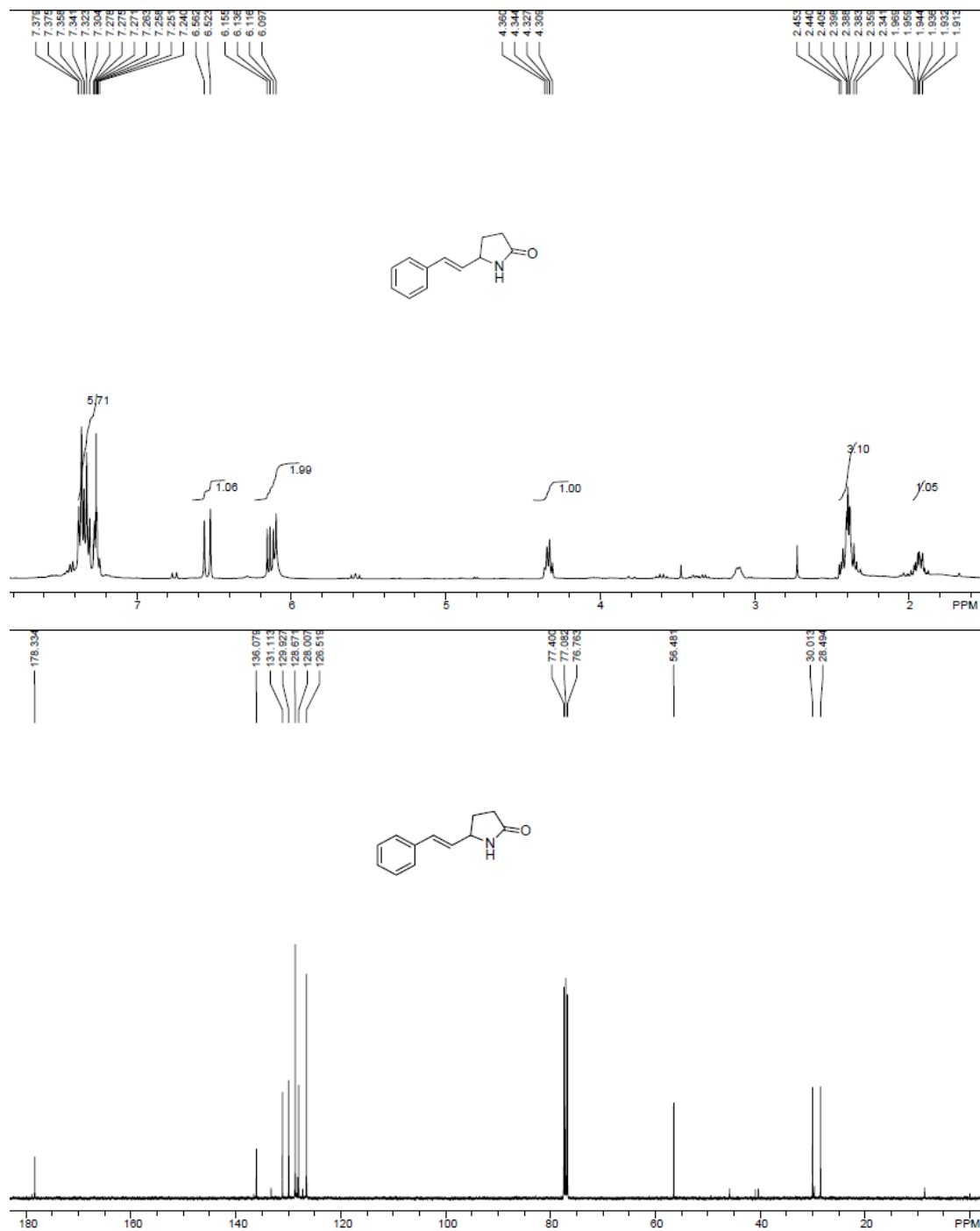
(E)-1-ethyl-5-styrylpyrrolidin-2-one (4k)



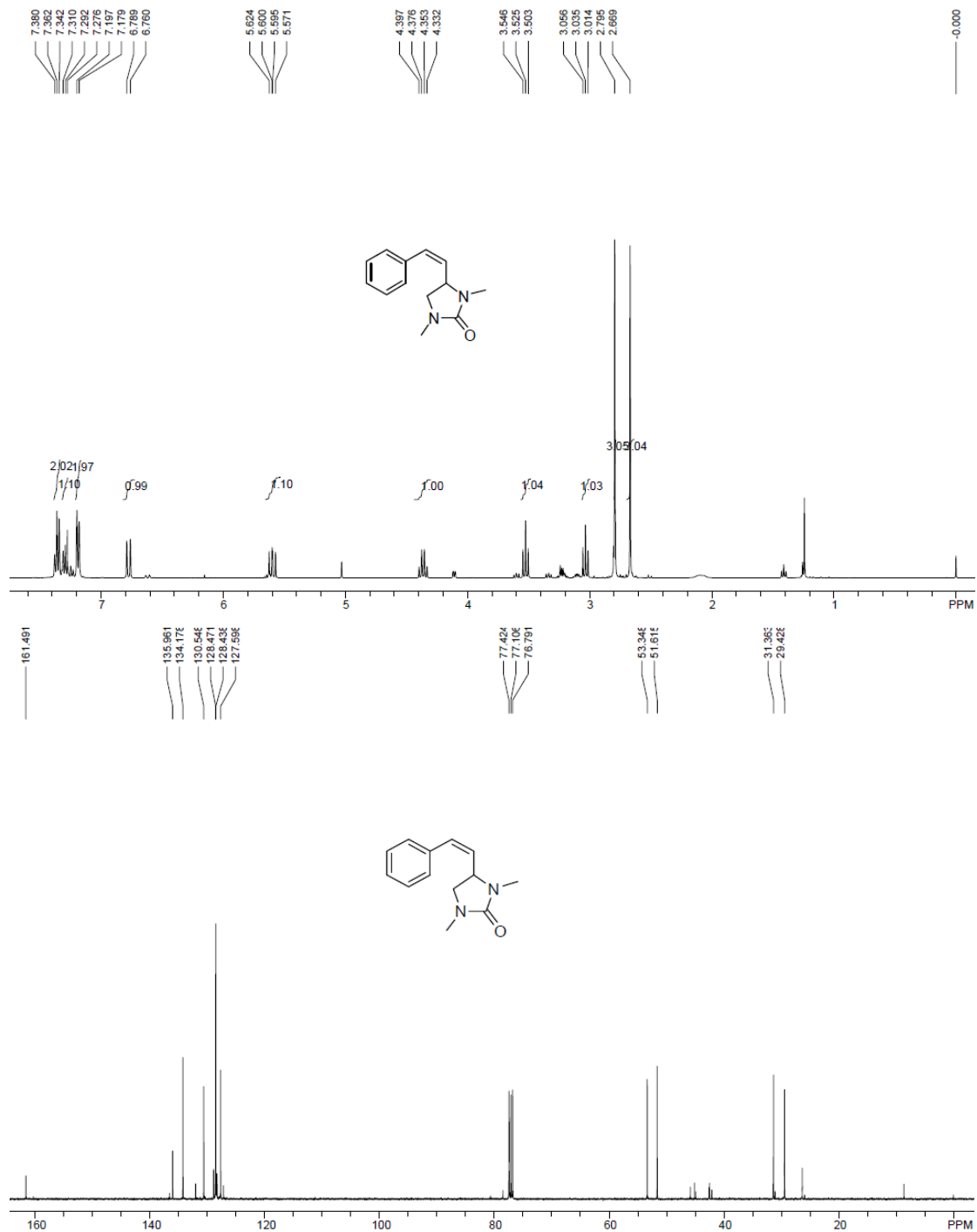
(Z)-5-styrylpyrrolidin-2-one (3l)



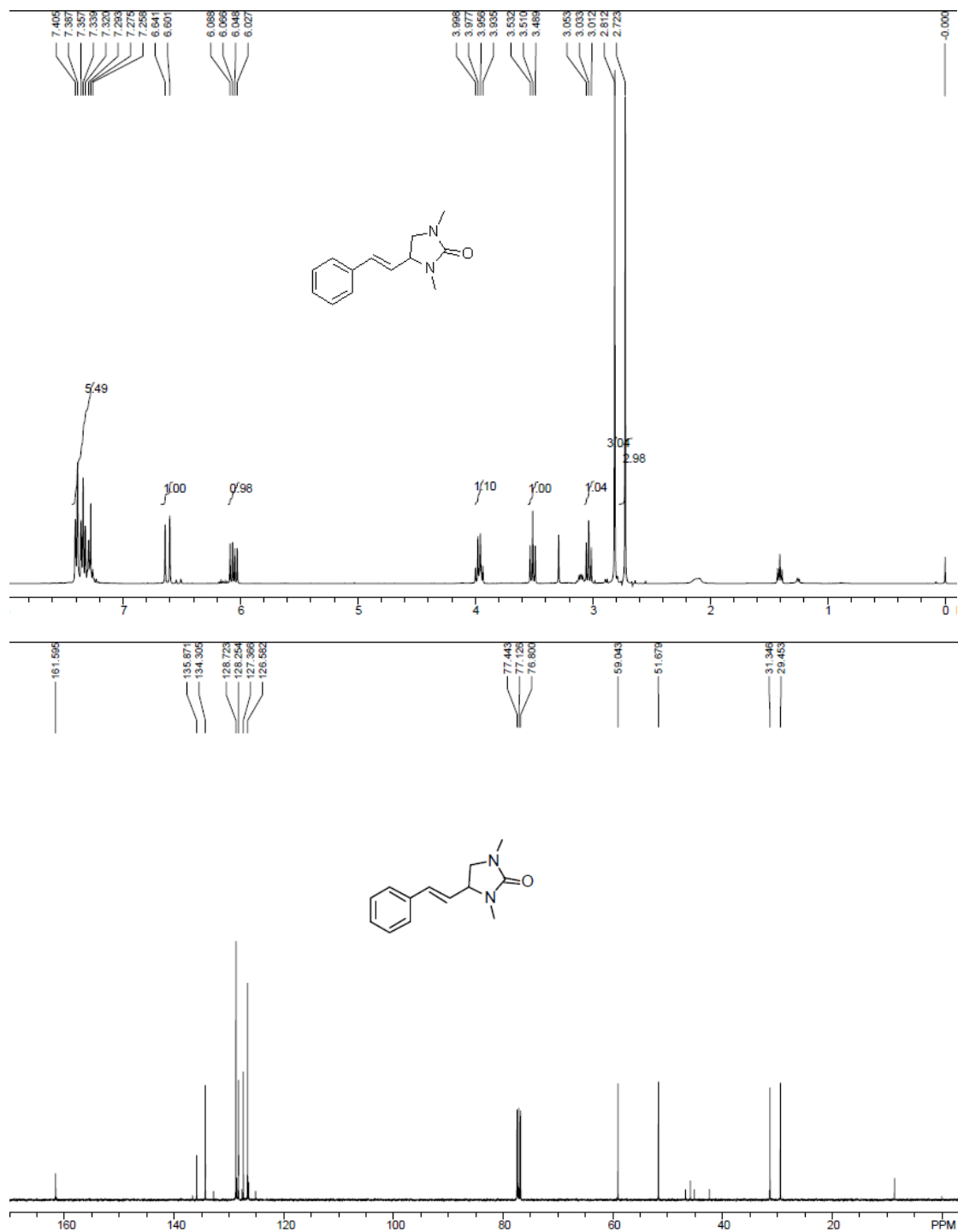
(E)-5-styrylpyrrolidin-2-one (4l)



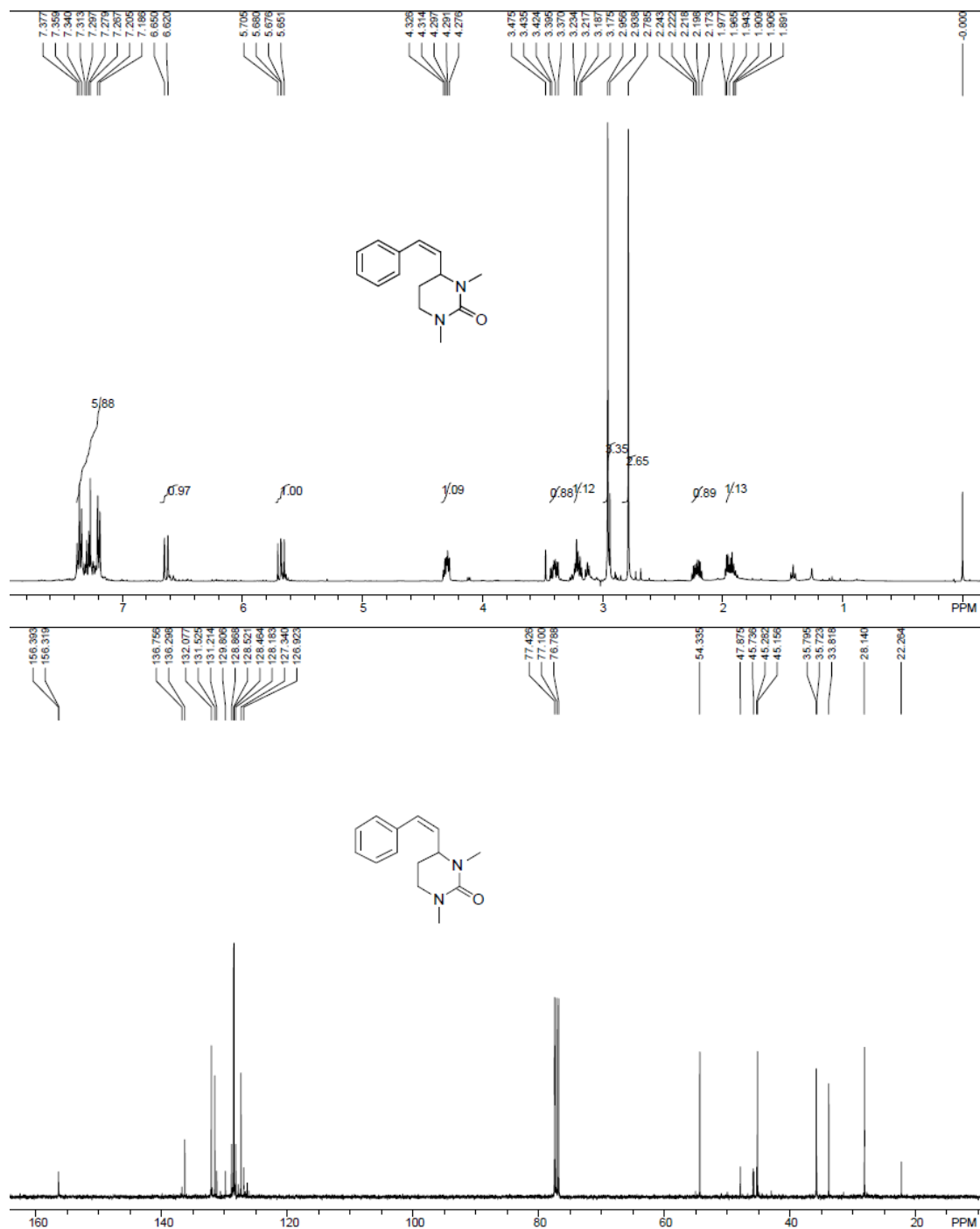
(Z)-1,3-dimethyl-4-styrylimidazolidin-2-one (3m)



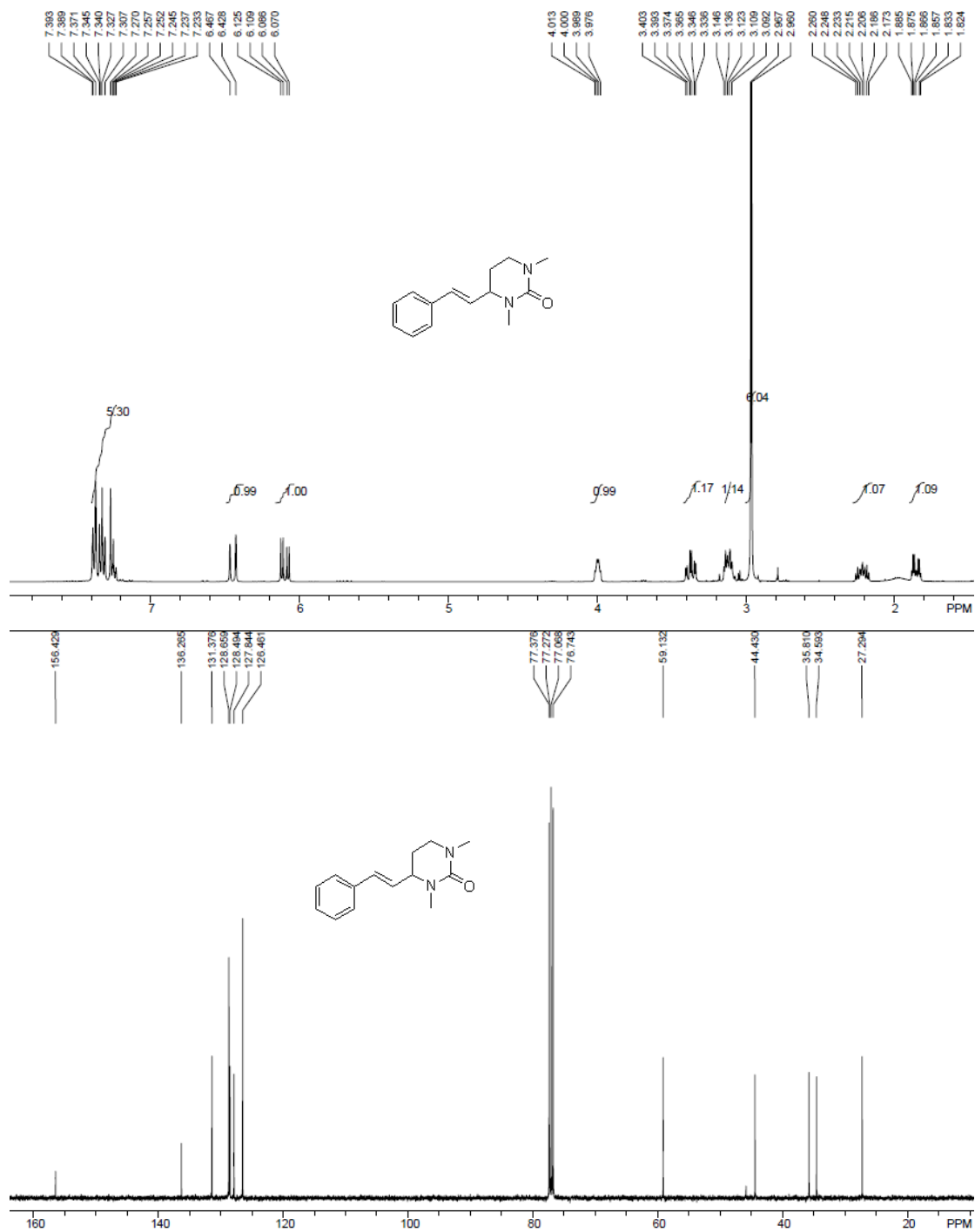
(E)-1,3-dimethyl-4-styrylimidazolidin-2-one (4m)



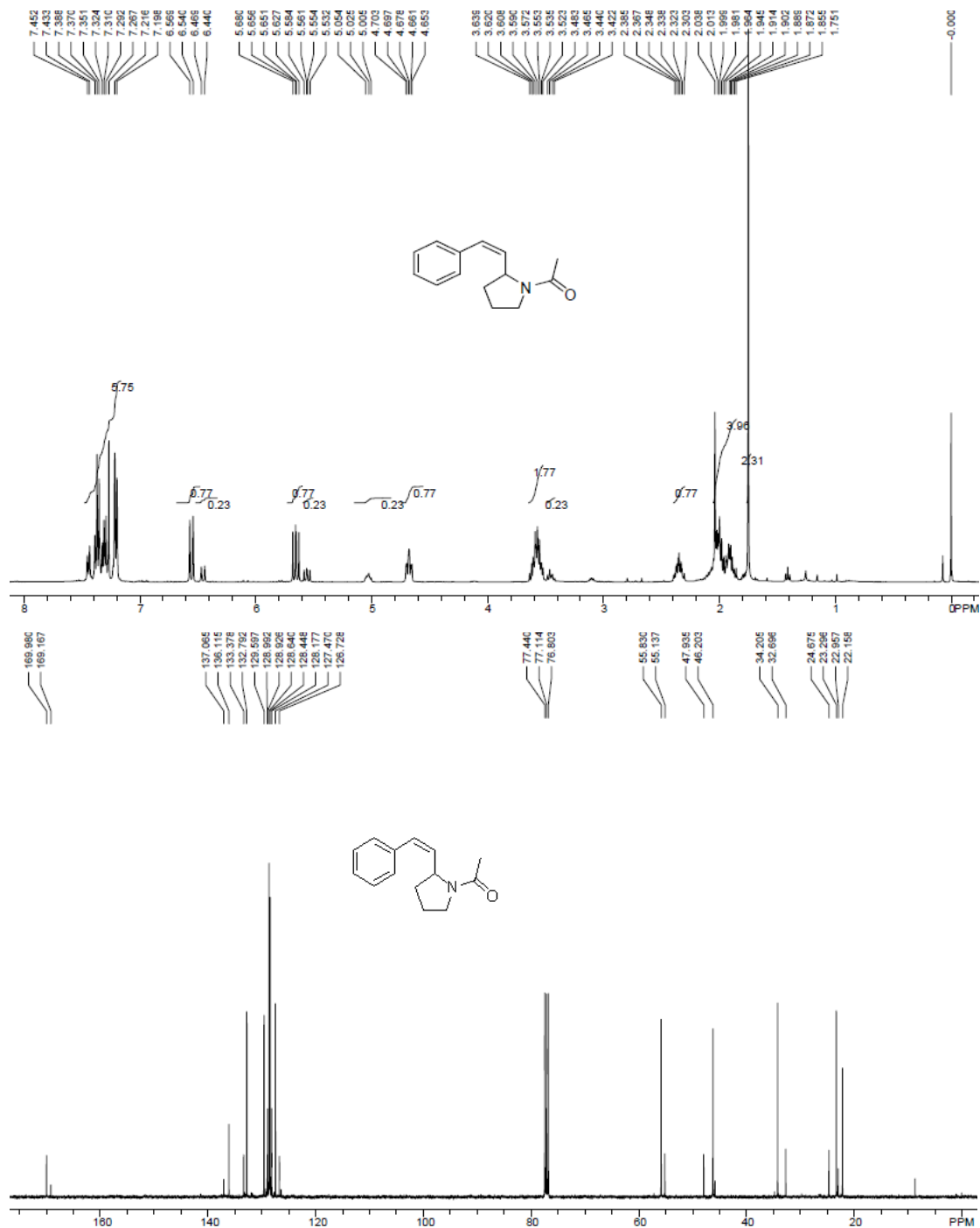
(Z)-1,3-dimethyl-4-styryltetrahydropyrimidin-2(1H)-one (3n)



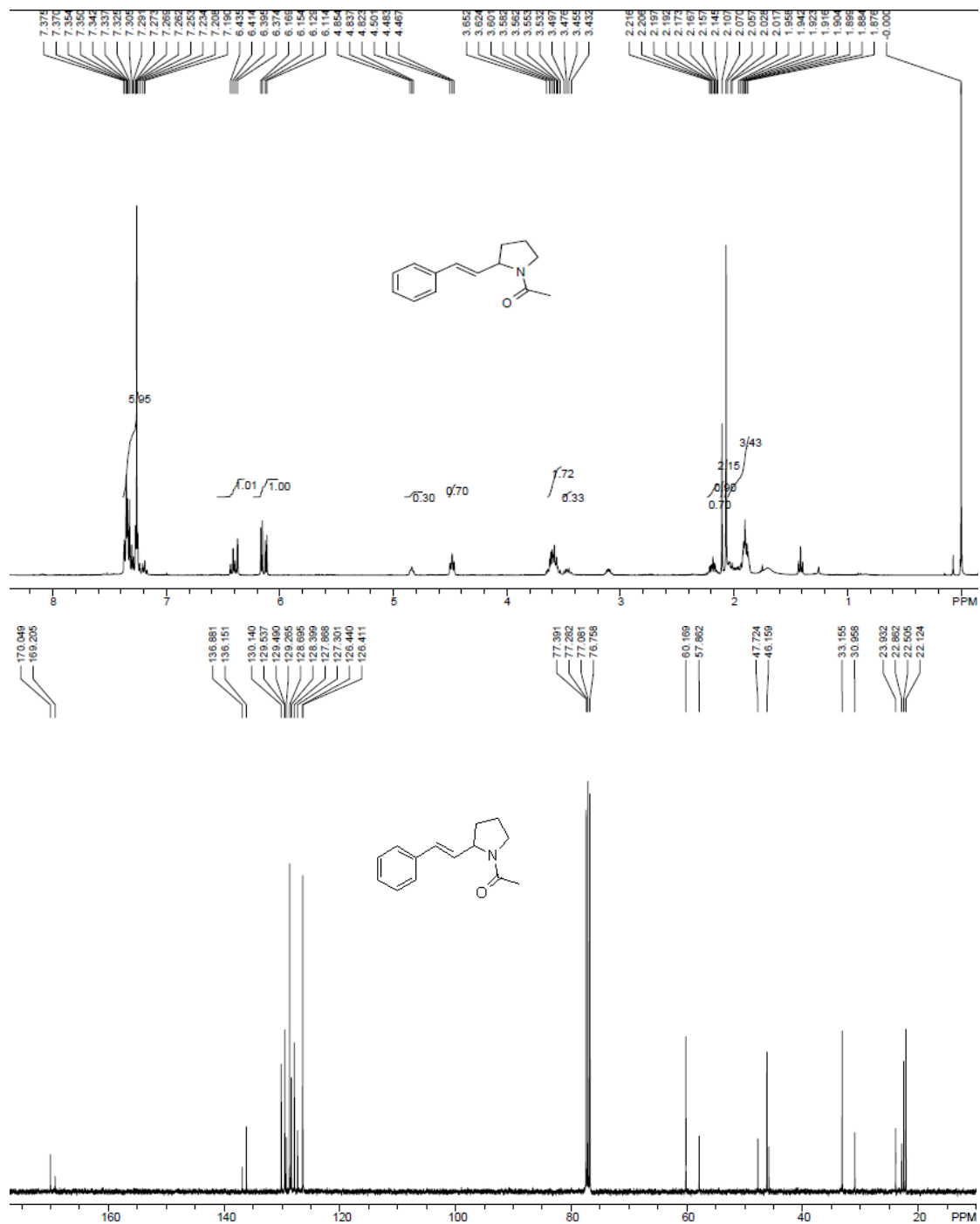
(E)-1,3-dimethyl-4-styryltetrahydropyrimidin-2(1H)-one (4n)



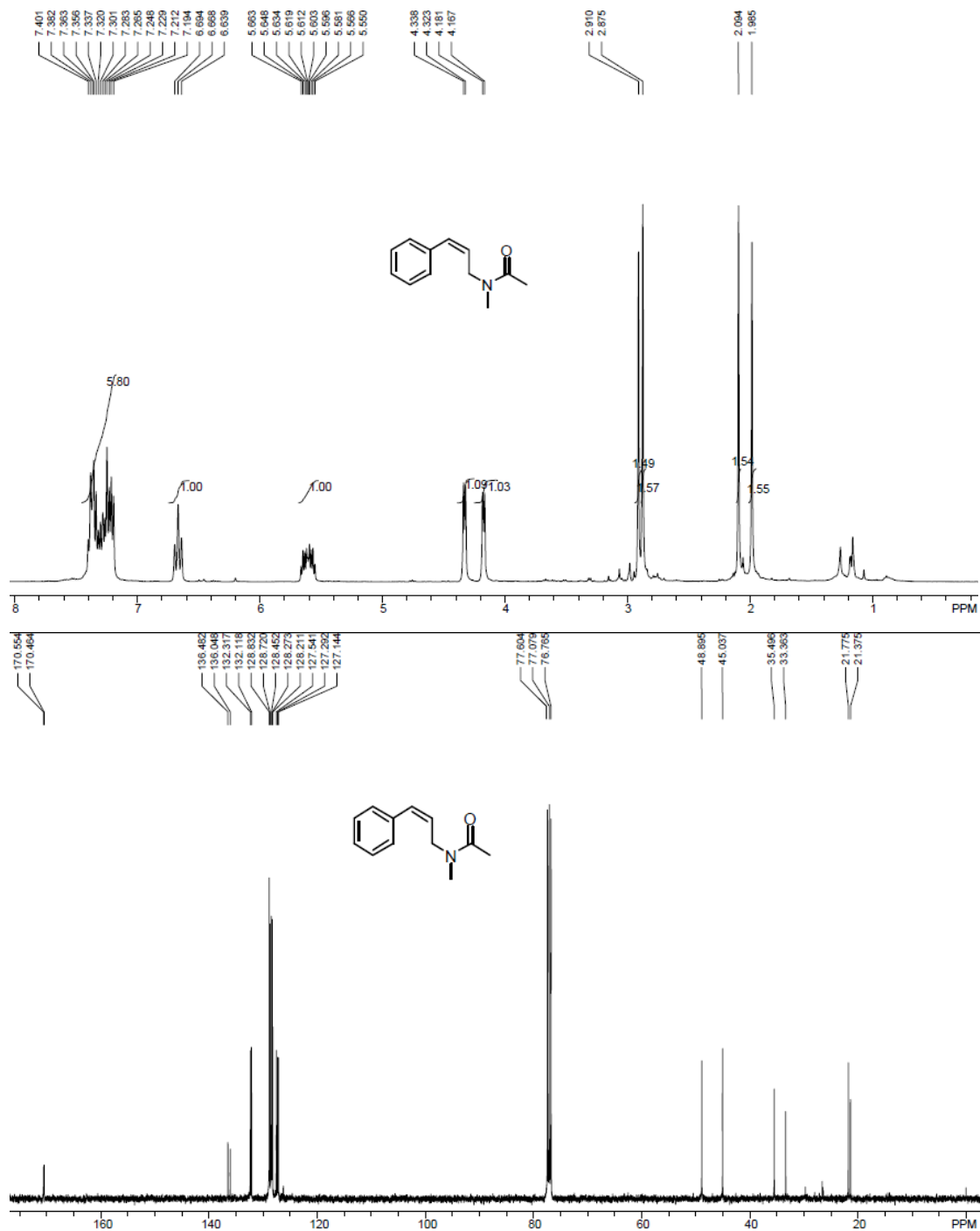
(Z)-1-(2-styrylpyrrolidin-1-yl)ethanone (3o)



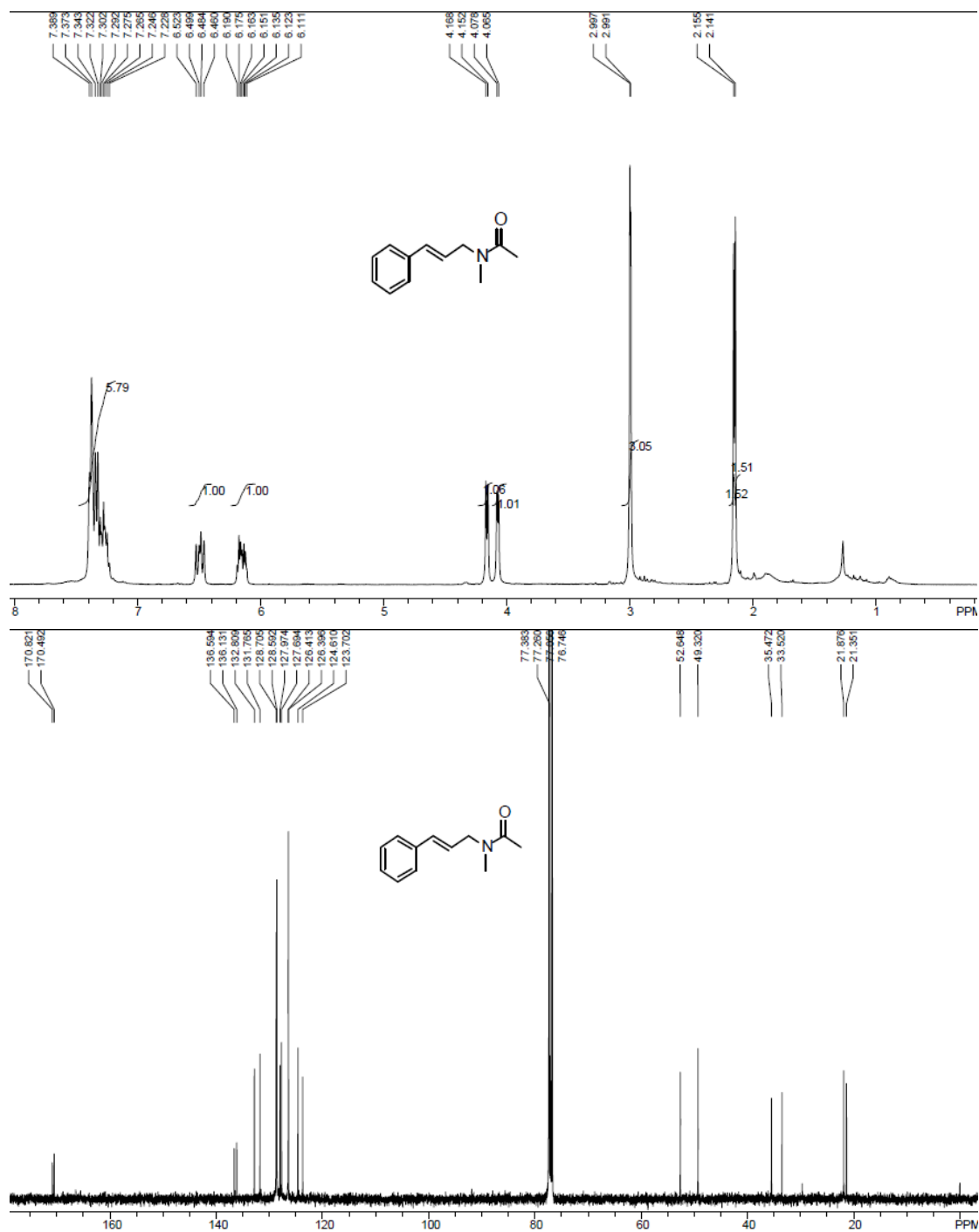
(E)-1-(2-styrylpyrrolidin-1-yl)ethanone (4o)



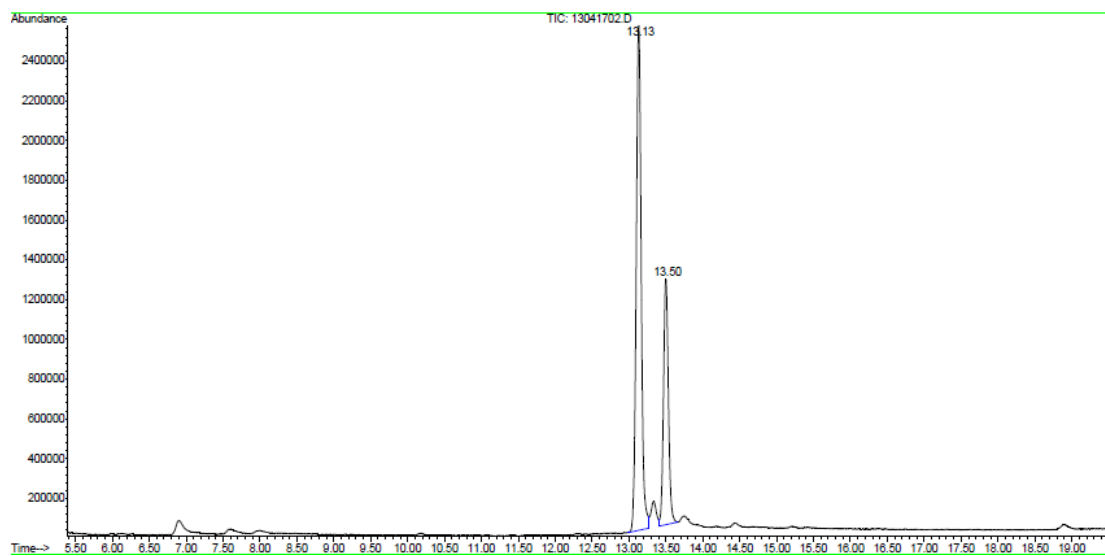
(Z)-N-methyl-N-(3-phenylallyl)acetamide (3p)



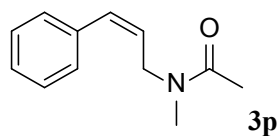
(E)-N-methyl-N-(3-phenylallyl)acetamide (4p)



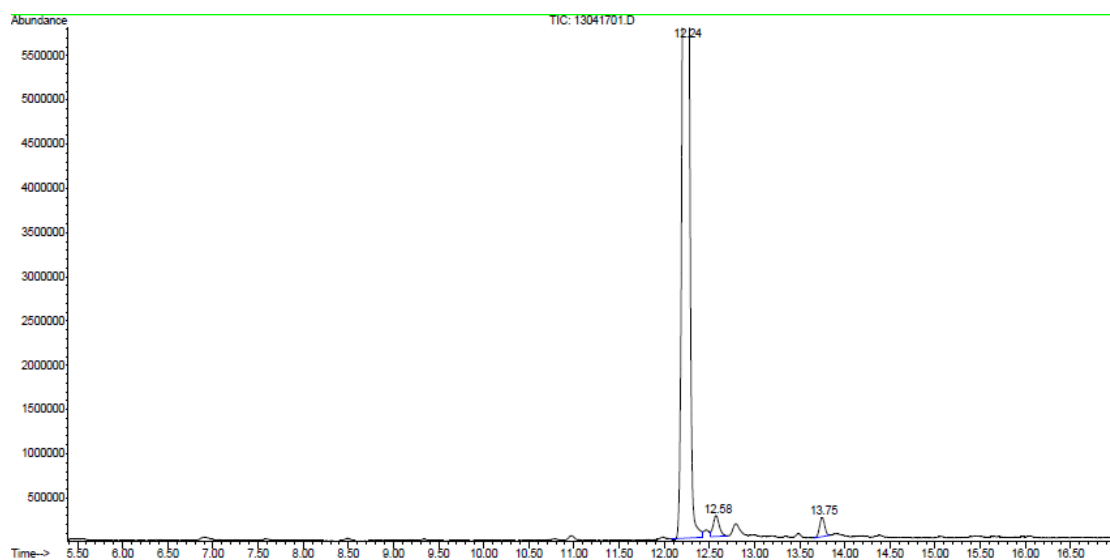
reaction with 1.0 equivalent of *t*BHP



peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	13.136	1139	1161	1182	BV 2	2541398	119175937	100.00%	67.917%
2	13.503	1202	1216	1239	VV	1235378	56296200	47.24%	32.083%



(Z)-N-methyl-N-(3-phenylallyl)acetamide (3p)



peak #	R.T. min	first scan	max scan	last scan	PK TY	peak height	corr. area	corr. % max.	% of total
1	12.240	1002	1027	1055	BV	12580099	590843363	100.00%	96.768%
2	12.574	1067	1077	1096	VV 2	236303	11308408	1.91%	1.852%
3	13.750	1240	1253	1265	BV 2	214527	8426662	1.43%	1.380%