A Novel Pseudo Four Component Reaction Involving Homoenolate for the Synthesis of γ-Aminobutyric acid (GABA) Derivatives

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Contents

(1) General remarks: All reactions were carried out in oven-dried glassware. Progress of reactions was monitored by Thin Layer Chromatography while purification was effected by column chromatography, using silica gel (100-200 mesh). Melting points were recorded on a Buchi melting point apparatus and are uncorrected. NMR spectra were recorded at 500 MHz (for ^{1}H) and 125 MHz (for ^{13}C) respectively on Brucker Avance DPX-500 MHz. Chemical shifts are reported in δ (ppm) relative to TMS (^{1}H) and $CDCl_3$ (^{13}C) as internal standards. Mass spectra were recorded using JEOL JMS 600H mass spectrometer. IR spectra were recorded on Bomem MB series FT-IR spectrometer; absorbencies are reported in cm^{-1}

(2) General experimental procedures:

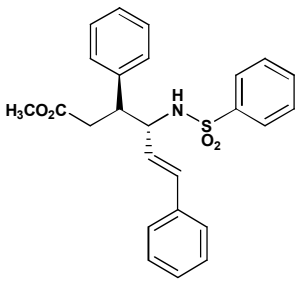
(a) *Synthesis of Methyl 4-(4-methylphenylsulfonamido)-3,6-diphenylhex-5-enoate **4b***:- DBU (7.6 mg, 20 mol %) was added to a suspension of the 1,3-dimesityl imidazolium chloride[1] **3e** (12.7 mg, 15 mol %), cinnamaldehyde **2** (132 mg, 1 mmol) and p- toluene sulfonamide **5** (43 mg, 0.25 mmol) in 3 ml dry methanol under argon atmosphere. This solution was stirred for 3h at room temperature (30 °C). The reaction mixture was then passed through a short pad of Celite®. After the removal of the solvent, the residue was subjected to chromatography on a silica gel (100-200 mesh) column using 90:10 hexane-ethyl acetate solvent mixture as eluent to afford **4b** (78.6 mg, 70% yield). It is then recrystallised from ethanol-chloroform solution & **CCDC** file number for **4b**: CCDC 737837

[1]Arduengo, A. J. III.; Krafczyk, R.; Schmutzler, R. *Tetrahedron* 1999, **55**, 14523.

(b) *Synthesis of 4-(4-methoxyphenyl)-1-(4-methoxyphenylsulfonyl)-5-(4-methoxystyryl)pyrrolidin-2-one* ***8a***:- DBU (7.6 mg, 20 mol %) was added to a suspension of the 1,3-dimesityl imidazolium chloride[1] **3e** (12.7 mg, 15 mol %), 4-methoxy cinnamaldehyde **7** (162 mg, 1 mmol) and 4-methoxy benzene sulfonamide **6** (46.8 mg, 0.25 mmol) in 3 ml dry DCM under argon atmosphere. This solution was stirred for 5h at room temperature (30 °C). The reaction mixture was then passed through a short pad of Celite®. After the removal of the solvent, the residue was subjected to chromatography on a silica gel (100-200 mesh) column using 80:20 hexane-ethyl acetate solvent mixture as eluent to afford **8a** (85 mg, 70% yield). It is then recrystallised from DCM-Hexane solution & **CCDC** file number for **8a**: CCDC 737838

(3) Characterization data for compounds

Compound 4a



White solid, mp.: 127-130 °C

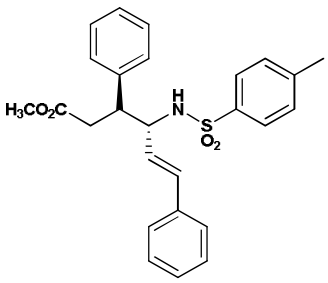
IR (KBr) ν_{max}: 3295, 2917, 1720, 1601, 1451, 1324, 1254, 1220, 1158, 1059 cm^{-1}.

^{1}H NMR: δ 7.72–7.70(m, 2H), 7.41-7.13(m, 11H), 7.03-7.01(m, 2H), 6.09(d, 1H, J = 16 Hz), 5.62(dd, 1H, J_1= 16 Hz, J_2 =8 Hz), 4.49(d, 1H, J = 8 Hz), 4.22-4.18(m,1H), 3.56(s, 3H), 3.35-3.34(m, 1H), 2.96(dd, 1H, J_1 = 16.5 Hz, J_2 =8 Hz), 2.64(dd, 1H, J_1 = 16.5 Hz, J_2 = 6.5 Hz)

^{13}CNMR: 172.4, 140.6, 138.4, 135.9, 132.7, 132.3, 128.9, 128.8, 128.5, 128.4, 127.8, 127.6, 127.3, 126.9, 126.3, 59.4, 53.3, 46.2, 36.5

HRMS-EI Calculated for $C_{25}H_{25}NO_4S$: 435.1504 , Found: 435.1544

Compound 4b



White crystalline solid, mp.: 149-151 °C

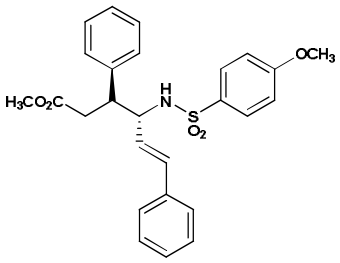
IR (KBr) ν_{max}: 3297, 2913, 1724, 1593, 1437, 1323, 1258, 1205, 1156, 1053 cm^{-1}.

^{1}H NMR: δ 7.59 (d, 2H, J = 8.5 Hz), 7.32-7.00 (m, 12H), 6.06 (d, 1H, J = 16 Hz), 5.57 (dd, 1H, J_1 = 16 Hz, J_2 = 8 Hz), 4.31 (d, 1H, J = 7.5 Hz), 4.18-4.14(m, 1H), 3.57(s, 3H), 3.36-3.32(m, 1H), 2.98(dd, 1H, J_1 = 17 Hz, J_2 = 8.5 Hz), 2.65(dd, 1H, J_1 = 16.5 Hz, J_2 = 6.5 Hz), 2.27(s, 3H)

^{13C}NMR: δ172.4, 143.0, 138.6, 137.6, 135.9, 132.4, 129.3, 128.6, 128.4, 128.2, 128.1, 127.7, 127.5, 127.4, 127.3, 127.1 , 126.8, 126.3, 126.2, 59.4, 51.5, 46.2, 36.5, 21.3

HRMS-EI Calculated for $C_{26}H_{27}NO_4S$: 449.1661, Found: 449.1693

Compound 4c



White solid, mp.: 119-121 °C

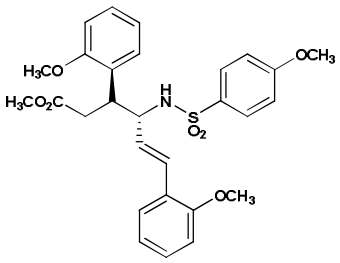
IR (KBr) ν_{max}: 3301, 2920, 1731, 1598, 1447, 1326, 1276, 1256, 1218, 1159, 1055cm^{-1}.

^{1}H NMR: δ 7.62(d, 2H, *J* = 9 Hz), 7.25-7.13(m, 8H), 7.01-6.99(m, 2H), 6.74-6.71(m, 2H), 6.04(d, 1H, *J* = 16 Hz), 5.56(dd, 1H, J_1 = 16 Hz, J_2 = 8.5 Hz), 4.71(d, 1H, *J* = 8 Hz), 4.14-4.10(m, 1H), 3.65(s, 3H), 3.53(s, 3H), 3.35-3.31(m, 1H), 2.95(dd, 1H, J_1 = 16.5 Hz, J_2 = 8 Hz), 2.63(dd, 1H, J_1 = 16.5 Hz, J_2 = 6.5 Hz)

^{13}CNMR: 172.3, 162.5, 138.6, 136.0, 132.5, 132.3, 129.5, 128.7, 128.5, 128.3, 127.7, 127.5, 127.0, 126.3, 113.9, 59.5, 55.2, 51.6, 46.3, 36.6

HRMS-EI Calculated for $C_{26}H_{27}NO_5S$: 465.1610, Found: 465.1661

Compound 4d



White solid, mp.: 132-135 °C

IR (KBr) ν_{max}: 3298, 2911, 1726, 1593, 1432, 1321, 1280, 1251, 1201, 1157, 1049 cm^{-1}.

^{1}H NMR: δ 7.56(d, 2H, *J* = 9 Hz), 7.20-6.70(m, 10H), 6.51(d, 1H, *J* = 16 Hz), 5.72(dd, 1H, J_1 = 16 Hz, J_2 = 8.5 Hz), 4.9(d, 1H, *J* = 7 Hz), 4.17-4.13(m,1H), 3.78(s, 6H), 3.73-3.71(m, 1H), 3.69(s, 3H),3.45(s, 3H), 2.84((dd, 1H, J_1 = 16.5 Hz, J_2 = 7 Hz), 2.64(dd, 1H, J_1 = 16.5 Hz, J_2 = 8 Hz)

^{13}C NMR: 172.6, 162.6, 157.2, 156.5, 134.2, 132.5, 129.3, 129.2, 128.5, 128.4, 128.3, 127.5, 127.1, 127.0, 126.7, 125.2, 120.9, 120.3, 114.0, 113.6, 110.9, 110.6, 60.1, 55.5, 55.4, 55.3, 51.4, 36.0

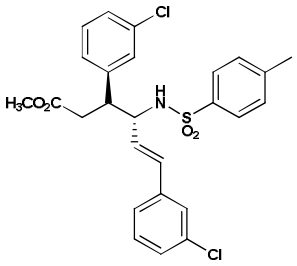
HRMS-EI Calculated for $C_{28}H_{31}NO_7S$: 525.1821, Found : 525.1847

Compound 4e

White solid, mp.: 128-131 °C

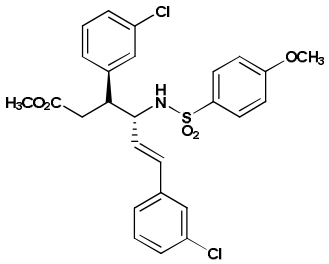
IR (KBr) ν_{max}: 3313, 2962, 1724, 1593, 1466, 1384, 1249, 1180, 1098, 1045 cm^{-1}.

^{1}H NMR: δ 7.58(d, 2H, *J* = 8.5 Hz), 7.23-6.93(m, 10H), 6.00(d, 1H, *J* = 15.5 Hz), 5.6(dd, 1H, J_1 = 16 Hz, J_2 = 8 Hz), 4.58(d, 1H, *J* = 7.5 Hz), 3.68-3.66(m, 1H), 3.57(s, 3H), 3.33-3.29(m, 1H), 2.92(dd, 1H, J_1 = 17 Hz, J_2 = 8 Hz), 2.61(dd,



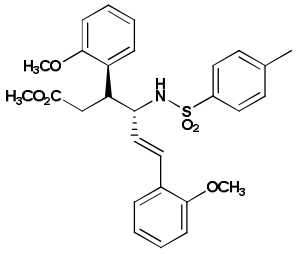
1H, J_1 = 16.5 Hz, J_2 = 6.5 Hz), 2.31(s, 3H)
^{13}CNMR: 172.0, 143.4, 140.7, 137.7, 137.4, 134.7, 134.4, 131.6, 130.0, 129.6, 129.5, 128.5, 128.1, 127.9, 127.3, 126.6, 126.3, 124.5, 59.2, 51.8, 36.4, 31.6, 22.6
HRMS-EI Calculated for $C_{26}H_{25}Cl_2NO_4S$: 517.0881, Found: 517.1008

Compound4f



White solid, mp.: 141-144 °C
IR (KBr) ν_{max}: 3264, 2945, 1732, 1593, 1478, 1433, 1323, 1278, 1239, 1151, 1094, 1029 cm^{-1}.
^{1}H NMR: δ 7.62(d, 2H, J = 9 Hz), 7.21-6.85(m, 10H), 6.03(d, 1H, J = 15.5 Hz), 5.6(dd, 1H, J_1 = 16 Hz, J_2 =8 Hz), 4.68(s, 1H), 3.73(s, 3H), 3.72-3.66(m, 1H), 3.56(s, 3H), 3.33-3.28(m, 1H), 2.91(dd, 1H, J_1 = 17 Hz, J_2 = 7.5 Hz), 2.60(dd, 1H, J_1 = 17 Hz, J_2 = 7 Hz)
^{13}CNMR: 172.0, 162.8, 141.5, 140.8, 137.7, 134.6, 134.4, 133.7, 131.9, 131.6, 129.9, 129.6, 129.4, 128.5, 128.2, 127.8, 126.6, 126.5, 126.3, 124.5, 114.0, 60.3, 55.3, 51.7, 45.9, 36.4
HRMS-EI Calculated for $C_{26}H_{25}Cl_2NO_5S$: 533.0830, Found: 533.0881

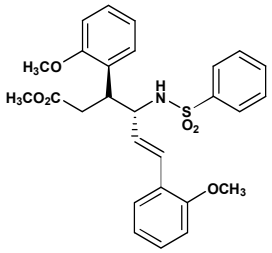
Compound 4g



White solid, mp.: 135-1138 °C
IR (KBr) ν_{max}: 3335, 2916, 1721, 1590, 1442, 1325, 1281, 1256, 1211, 1157, 1045 cm^{-1}.
^{1}H NMR: δ 7.52(d, 2H, J = 8.5 Hz), 7.25-6.76(m, 10H), 6.51(d, 1H, J = 16 Hz), 5.71(dd, 1H, J_1 = 16 Hz, J_2 = 8 Hz), 4.95(d,1H, J = 6.5 Hz), 4.17-4.15(m, 1H), 3.78(s, 3H), 3.77(s, 3H), 3.73-3.70(m, 1H), 3.45(s, 3H), 2.83(dd, 1H, J_1 = 16.5 Hz, J_2 = 7 Hz), 2.63(dd, 1H, J_1 = 16.5 Hz, J_2 = 8 Hz), 2.25(s, 3H)
^{13}C NMR: 172.5, 157.1, 156.5, 142.4, 138.0, 129.5, 129.2, 129.1, 128.6, 128.4, 128.2, 127.4, 127.3, 127.1, 126.7, 126.4, 125.2, 120.9, 120.3, 110.9, 110.6, 60.1, 55.4, 55.2, 51.4, 40.2, 36.0, 21.3
HRMS-EI Calculated for $C_{28}H_{31}NO_6S$: 509.1872, Found : 509.1857

Compound 4h

White solid, mp.: 114-117°C
IR (KBr) ν_{max}: 3268, 2921, 1728, 1601, 1491, 1442, 1319, 1249, 1164, 1094, 1025 cm^{-1}.

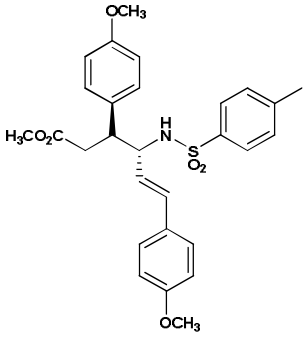


^{1}H NMR: δ 7.65(dd, , 2H, J_1 =8.5 Hz, J_2 = 1.5 Hz), 7.38-7.35(m, 1H), 7.29-7.25(m, 2H), 7.20-7.13(m, 2H), 7.08-7.04(m, 2H), 6.87-6.77(m, 4H), 6.56(d, 1H, J = 16 Hz), 5.75(dd, 1H, J_1 = 16 Hz, J_2 =8 Hz), 4.96(d, 1H, J = 6.5 Hz), 4.22-4.17(m, 1H), 3.78(s, 3H), 3.77(s, 3H), 3.74-3.71(m, 1H), 3.46(s, 3H), 2.85(dd, 1H, J_1 = 16.5 Hz, J_2 = 7 Hz), 2.64(dd, 1H, J_1 = 16.5 Hz, J_2 = 8 Hz)

^{13}CNMR: 172.5, 157.1, 156.6, 140.9, 131.8, 129.2, 128.9, 128.6, 128.5, 128.2, 127.4, 127.3, 127.2, 127.0, 126.8, 126.4, 125.2, 121.0, 120.4, 119.1, 111.0, 60.2, 55.4, 55.2, 51.4, 39.8, 36.0

HRMS-EI Calculated for $C_{27}H_{29}NO_6S$: 495.1716, Found: 495.1742

Compound 4i



White solid, mp.: 120-123 °C

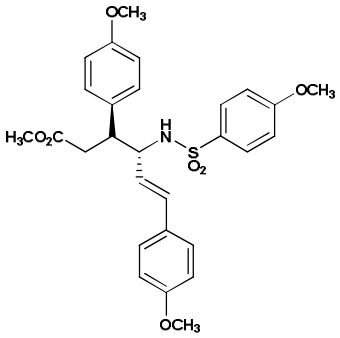
IR (KBr) ν $_{max}$: 3317, 2920, 1724, 1598, 1436, 1321, 1272, 1251, 1218, 1157, 1051 cm^{-1}.

^{1}H NMR: δ 7.57(d, 2H, J = 8 Hz), 7.28-6.73(m, 10H), 5.99(d, 1H, J = 15.5 Hz), 5.43(dd, 1H, J_1 = 15.5 Hz, J_2 = 8 Hz), 4.59(d, 1H, J = 8 Hz), 3.78(s, 3H), 3.77(s, 3H), 3.73-3.72(m, 1H), 3.53(s, 3H), 3.28-3.24(m, 1H), 2.90(dd, 1H, J_1 = 16.5 Hz, J_2 = 8 Hz), 2.59(dd, 1H, J_1 = 16.5 Hz, J_2 = 7.5 Hz), 2.27(s, 3H)

^{13}CNMR: 172.5, 159.3, 158.8, 142.9, 137.7, 132.0, 130.4, 129.5, 129.4, 129.3, 128.8, 127.5, 127.3, 126.4, 124.8, 114.0, 113.7, 60.3, 55.1, 55.0, 51.5, 45.6, 36.9, 20.9

HRMS-EI Calculated for $C_{28}H_{31}NO_6S$: 509.1872, Found: 509.1888

Compound 4j



White solid, mp.: 127-130 °C

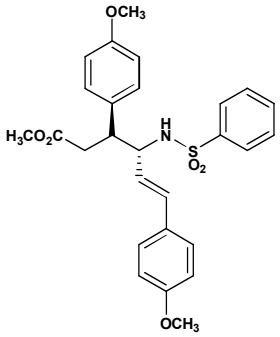
IR (KBr) ν $_{max}$: 3298, 2924, 1723, 1596, 1440, 1321, 1281, 1259, 1224, 1154, 1048 cm^{-1}.

^{1}H NMR: δ 7.60(d, 2H, J = 9 Hz), 7.07-6.74(m, 10H), 6.04(d, 1H, J = 16 Hz), 5.42(dd, 1H, J_1 = 16 Hz, J_2 = 8.5 Hz), 4.26(d, 1H, J = 7 Hz), 4.06-4.01(m, 1H), 3.79(s, 3H), 3.78(s, 3H), 3.71(s, 3H), 3.54(s, 3H), 3.27-3.22(m, 1H), 2.90(dd, 1H, J_1 = 16.5 Hz, J_2 = 8 Hz), 2.59(dd, 1H, J_1 = 16.5 Hz, J_2 = 7 Hz)

^{13}CNMR: 172.4, 162.5, 159.3, 158.8, 132.3, 132.0, 130.5, 1 29.4, 128.8, 128.5, 128.2, 127.5, 124.9, 114.0, 113.9, 113.8, 113.7, 113.0, 112.9, 60.3, 55.3, 55.1, 55.0, 51.5, 45.6, 37.0

HRMS-EI Calculated for $C_{28}H_{31}NO_7S$: 525.1821, Found: 525.1853

Compound 4k



White solid, mp.: 131-133 °C

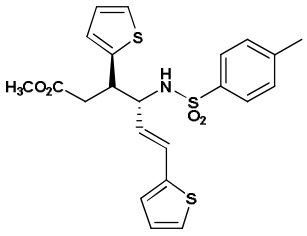
IR (KBr) ν_{max}: 3297, 2918, 1731, 1596, 1433, 1322, 1282, 1246, 1196, 1158, 1051 cm^{-1}.

^{1}H NMR: δ 7.93(d, 2H, J = 9.5 Hz), 7.71-6.73(m, 11H), 6.04(d, 1H, J = 16 Hz), 5.45(dd, 1H, J_1 = 16 Hz, J_2 = 8 Hz), 4.41(d, 1H, J = 7 Hz), 4.12-4.08(m, 1H), 3.79(s, 3H), 3.78(s, 3H), 3.54(s, 3H), 3.28-3.24(m, 1H), 2.90(dd, 1H, J_1 = 16.5 Hz, J_2 = 8 Hz), 2.59(dd, 1H, J_1 = 16.5 Hz, J_2 = 6.5 Hz)

^{13}CNMR: 172.4, 159.4, 158.9, 140.7, 132.6, 132.3, 132.2, 130.3, 129.4, 129.0, 128.7, 127.5, 127.3, 126.4, 124.8, 114.2, 113.8, 59.8, 55.1, 55.0, 51.6, 45.5, 36.9

HRMS-EI Calculated for $C_{27}H_{29}NO_6S$: 495.1716, Found: 495.1729

Compound 4l



White solid, mp.: 128-130 °C

IR (KBr) ν_{max}: 3295, 2916, 1722, 1611, 1457, 1324, 1249, 1220, 1157, 1047 cm^{-1}.

^{1}H NMR: δ 7.66(d, 2H, J = 8 Hz), 7.20-6.71(m, 8H), 6.15(d, 1H, J = 15.5 Hz), 5.52(dd, 1H, J_1 = 16 Hz, J_2 = 7.5 Hz), 4.50(d, 1H, J = 8.5 Hz), 4.22-4.18(m, 1H), 3.67-3.66(m, 4H), 3.00(dd, 1H, J_1 = 17 Hz, J_2 = 8.5 Hz), 2.72(dd, 1H, J_1 = 17 Hz, J_2 = 6.5 Hz), 2.32(s, 3H)

^{13}CNMR: 171.9**,** 143.2, 140.9, 140.8, 137.6, 129.5, 127.3, 127.2, 127.0, 126.2, 126.0, 125.9, 125.7, 124.8, 124.6, 58.5, 51.8, 42.0, 37.5, 21.4

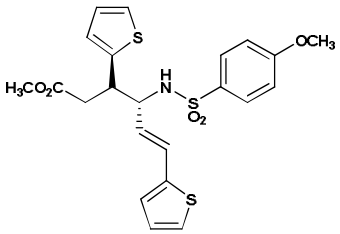
HRMS-EI Calculated for $C_{22}H_{23}NO_4S_3$: 461.0789, Found: 461.0815

Compound 4m

White solid, mp.: 121-124 °C

IR (KBr) ν_{max}: 3304, 2923, 1718, 1621, 1453, 1331, 1281, 1252, 1230, 1159, 1061 cm^{-1}.

^{1}H NMR: δ 7.03(d, 2H, J = 8.5 Hz), 7.21(d, 1H, J = 5 Hz),

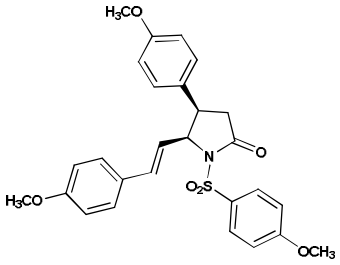


7.11(d, 1H, J = 5 Hz), 6.98-6.73(m, 6H), 6.19(d, 1H, J = 18 Hz), 5.51(dd, 1H, J_1 = 15.5 Hz, J_2 = 7.5 Hz), 4.44(d, 1H, J = 8.5 Hz), 4.20-4.16(m, 1H), 3.76(s, 3H), 3.68-3.64(m, 4H), 3.00(dd, 1H, J_1 = 17 Hz, J_2 = 8.5 Hz), 2.72(dd, 1H, J_1 = 17 Hz, J_2 = 6 Hz)

^{13}CNMR: 171.9, 162.8, 140.9, 140.8, 132.3, 129.4, 127.2, 127.0, 126.4, 126.0, 125.9, 125.8, 124.8, 124.5, 114.0, 58.6, 55.3, 51.7, 42.0, 37.5

HRMS-EI Calculated for $C_{22}H_{23}NO_5S_3$: 477.0738 Found: 477.0761

Compound 8a



White crystalline solid, mp.: 140-142 °C

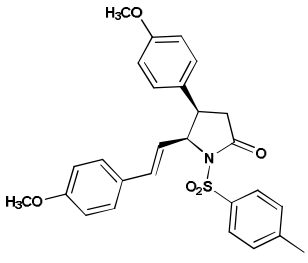
IR (KBr) ν_{max}: 3058, 2923, 1729, 1607, 1493, 1351, 1311, 1287, 1154, 1076 cm^{-1}.

^{1}H NMR: δ 7.93(dd, 2H, J_1 = 7 Hz, J_2 = 2 Hz), 7.03-6.99(m, 4H), 6.86-6.75(m, 6H), 6.30(d, 1H, J = 15.5 Hz), 5.42(dd, 1H, J_1 = 16 Hz, J_2 = 8 Hz), 5.20(t, 1H, J = 7.5 Hz), 3.89-3.85(m, 1H), 3.83(s, 3H), 3.78(s, 3H), 3.75(s, 3H), 2.85(dd, 1H, J_1 = 17 Hz, J_2 = 13.5 Hz), 2.67(dd, 1H, J_1 = 17 Hz, J_2 = 7.5 Hz)

^{13}CNMR: 171.8, 163.7, 159.6, 158.9, 133.8, 131.1, 130.3, 128.9, 128.4, 127.8, 127.6, 120.7, 114.0, 113.9, 113.7, 65.8, 55.5, 55.1, 55.0, 42.7, 35.4

HRMS-EI Calculated for $C_{27}H_{27}NO_6S$: 493.1559, Found: 493.1572

Compound 8b



White solid, mp.: 157-159 °C

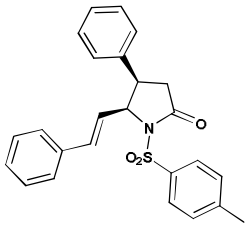
IR (KBr) ν_{max}: 3061, 2924, 1733, 1591, 1493, 1349, 1329, 1278, 1153, 1079 cm^{-1}.

^{1}H NMR: δ 7.88(d, 2H, J = 8.5 Hz), 7.20(d, 2H, J = 8 Hz), 7.03-6.97(m, 4H), 6.80-6.75(m, 4H), 6.30(d, 1H, J = 15.5 Hz), 5.42(dd, 1H, J_1 = 16 Hz, J_2 = 8 Hz), 5.22(t, 1H, J = 7.5 Hz), 3.91-3.85(m, 1H), 3.78(s, 3H), 3.75(s, 3H), 2.88(dd, 1H, J_1 = 17 Hz, J_2 = 13.5 Hz), 2.66(dd, 1H, J_1 = 17 Hz, J_2 = 7.5 Hz), 2.40(s, 3H)

^{13}CNMR: 171.8, 159.5, 158.9, 144.7, 135.9, 133.9, 129.2, 129.0, 128.9, 128.4, 127.8, 127.5, 120.6, 113.9, 113.8, 65.8, 55.1, 55.0, 42.8, 35.4, 21.6

HRMS-EI Calculated for $C_{27}H_{27}NO_5S$: 477.1610, Found: 477.1618

Compound 8c



White solid, mp.: 148-151 °C

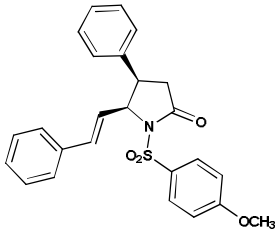
IR (KBr) ν_{max}: 3060, 2921, 1736, 1597, 1491, 1356, 1307, 1229, 1168, 1119, 1082 cm^{-1}.

^{1}H NMR: δ 7.89(d, 2H, J = 8.5 Hz), 7.33-7.00(m, 12H), 6.63(d, 1H, J = 15.5 Hz), 5.55(dd, 1H, J_1 = 16 Hz, J_2 = 8 Hz), 5.29(t, 1H, J = 8 Hz), 3.97-3.95(m, 1H), 2.95(dd, 1H, J_1 = 17 Hz, J_2 = 13.5 Hz), 2.70(dd, 1H, J_1 = 17 Hz, J_2 = 7.5 Hz), 2.40(s, 3H)

^{13}CNMR: 171.6, 144.8, 135.9, 135.8, 135.6, 135.5, 134.4, 133.2, 129.3, 128.9, 128.6, 128.4, 128.1, 127.9, 127.7, 126.7, 126.5, 126.4, 123.3, 65.4, 43.4, 37.9, 35.0, 21.6

HRMS-EI Calculated for $C_{25}H_{23}NO_3S$: 417.1399, Found: 417.1418

Compound 8d



White solid, mp.: 114-117 °C

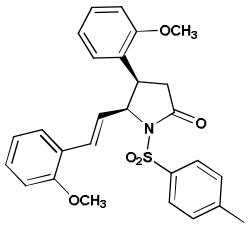
IR (KBr) ν_{max}: 3062, 2921, 1738, 1593, 1496, 1351, 1307, 1285, 1166, 1059 cm^{-1}.

^{1}H NMR: δ 7.94(d, 2H, J = 9 Hz), 7.28-7.21(m, 6H), 7.12(d, 2H, J = 7 Hz), 7.03-7.02(m, 2H), 6.84(d, 2H, J = 9 Hz), 6.35(d, 1H, J = 16 Hz), 5.55(dd, 1H, J_1 = 16 Hz, J_2 = 8 Hz), 5.28(t, 1H, J = 7.5 Hz), 3.99-3.93(m, 1H), 3.82(s, 3H), 2.95(dd, 1H, J_1 = 17 Hz, J_2 = 13.5 Hz), 2.70(dd, 1H, J_1 = 17 Hz, J_2 = 7.5 Hz)

^{13}CNMR: δ 171.7, 163.8, 135.6, 134.4, 131.1, 130.9, 130.2, 129.1, 128.6, 128.5, 128.1, 127.9, 127.7, 126.7, 126.5, 126.3, 123.0, 113.8, 65.4, 55.5, 43.3, 35.0

HRMS-EI Calculated for $C_{25}H_{23}NO_4S$: 433.1348, Found: 433.1325

Compound 8e



White solid, mp.: 127-129 °C

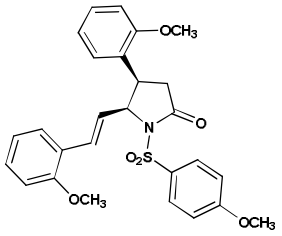
IR (KBr) ν_{max}: 3043, 2970, 1736, 1593, 1491, 1437, 1356, 1315, 1245, 1168, 1086, 1021 cm^{-1}.

^{1}H NMR: δ 7.88(d, 2H, J = 8.5 Hz), 7.24-6.69(m, 11H), 5.52-5.42(m, 2H), 4.14-4.12(m, 1H), 3.90(s, 3H), 3.72(s, 3H), 2.96(dd, 1H, J_1 = 17 Hz, J_2 = 14 Hz), 2.57(dd, 1H, J_1 = 17 Hz, J_2 = 7.5 Hz), 2.37(s, 3H)

^{13}CNMR: δ 171.4, 157.5, 156.7, 144.0, 136.4, 129.3, 129.0, 128.8, 128.6, 127.0, 126.5, 125.2, 124.5, 123.9, 120.4, 120.3, 110.6, 110.1, 64.5, 55.2, 55.1, 38.4, 34.1, 21.6

HRMS-EI Calculated for $C_{27}H_{27}NO_5S$: 477.1610, Found: 477.1643

Compound 8f



White solid, mp.: 191-193 °C

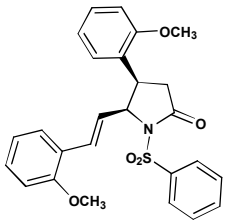
IR (KBr) ν_{max}: 3056, 2947, 1719, 1589, 1495, 1462, 1356, 1311, 1254, 1176, 1086, 1008 cm^{-1}.

^{1}H NMR: δ 7.93(dd, 2H, J_1 = 7 Hz, J_2 = 2 Hz), 7.16-7.09(m, 2H), 6.92(d, 1H, J = 7 Hz), 6.83-6.79(m, 4H), 6.74-6.66(m, 3H), 5.51-5.42(m, 2H), 4.17-4.12(m, 1H), 3.91(s, 3H), 3.85-3.84(m, 1H), 3.80(s, 3H), 3.72(s, 3H), 2.97(dd, 1H, J_1 = 16.5 Hz, J_2 = 13.5 Hz), 2.58(dd, 1H, J_1 = 16.5 Hz, J_2 = 7.5 Hz)

^{13}CNMR: δ 171.4, 163.4, 156.7, 155.2, 131.2, 130.8, 129.0, 128.8, 128.6, 127.0, 125.2, 123.9, 120.3, 120.2, 113.5, 110.6, 110.0, 64.3, 55.3, 55.2, 55.1, 38.3, 34.1

HRMS-EI Calculated for $C_{27}H_{27}NO_6S$: 493.1559, Found: 493.1547

Compound 8g



White solid, mp.: 187-189 °C

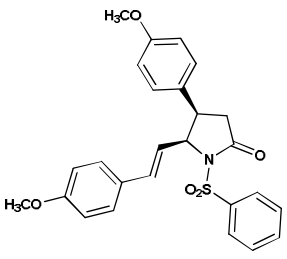
IR (KBr) ν_{max}: 3056, 2921, 1744, 1597, 1491, 1437, 1356, 1311, 1245, 1176, 1086, 1029 cm^{-1}.

^{1}H NMR: δ 8.04-8.02(m, 2H), 7.53(t, 1H, J = 7.25 Hz), 7.39-7.36(m, 2H), 7.25-7.11(m, 2H), 6.93(d, 1H, J = 7.5 Hz), 6.84-6.74(m, 6H), 5.55(t, 1H, J = 8.25 Hz), 5.47(dd, 1H, J_1 = 15.5 Hz, J_2 = 8.5 Hz), 4.17-4.14(m, 1H), 3.89(s, 3H), 3.72(s, 3H), 3.00(dd, 1H, J_1 = 17 Hz, J_2 = 13.5 Hz), 2.61(dd, 1H, J_1 = 17 Hz, J_2 = 7.5 Hz)

^{13}CNMR: δ 171.8, 157.5, 156.8, 139.1, 133.4, 129.5, 128.9, 128.7, 128.4, 127.1, 127.0, 124.3, 123.6, 120.4, 120.3, 110.7, 110.1, 64.7, 55.2, 55.1, 38.4, 34.2

HRMS-EI Calculated for $C_{26}H_{25}NO_5S$: 463.1453, Found: 463.1477

Compound 8h



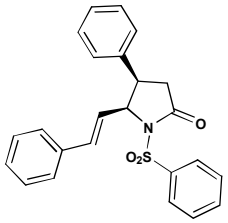
White solid, mp.: 116-118 °C

IR (KBr) ν_{max}: 3068, 2933, 1740, 1609, 1515, 1450, 1368, 1315, 1258, 1172, 1086, 1025 cm^{-1}.

^{1}H NMR: δ 8.02-8.00(m, 2H), 7.60-7.56(m, 1H), 7.43-7.39(m, 2H), 7.02(d, 2H, J = 8.5 Hz), 6.98-6.96(m, 2H), 6.80-6.74(m, 4H), 6.29(d, 1H, J = 16 Hz), 5.42(dd, 1H, J_1 = 16 Hz, J_2 = 8 Hz), 5.25-5.22(m, 1H), 3.91-3.88(m, 1H), 3.78(s, 3H), 3.75(s, 3H), 2.89(dd, 1H, J_1 = 17 Hz, J_2 = 13.5 Hz), 2.68(dd, 1H, J_1 = 17 Hz, J_2 = 8 Hz)

^{13}CNMR: δ 171.7, 159.6, 158.9, 139.0, 133.9, 133.6, 128.9, 128.8, 128.6, 128.4, 127.7, 127.5, 120.7, 114.0, 113.9, 65.8, 55.1, 55.0, 42.8, 35.4
HRMS-EI Calculated for $C_{26}H_{25}NO_5S$: 463.1453, Found: 463.1421

Compound 8i



White solid, mp.: 120-122 °C
IR (KBr) ν_{max}: 3051, 2941, 1738, 1595, 1494, 1359, 1321, 1278, 1159, 1037 cm^{-1}.
^{1}H NMR: δ 8.03(dd, 2H, J_1 = 8.5 Hz, J_2 = 1 Hz), 7.60-7.57(m, 1H), 7.44-7.41(m, 2H), 7.31-7.20(m, 6H), 7.13(d, 2H, J = 7 Hz), 7.01-6.99(m, 2H), 6.36(d, 1H, J = 16 Hz), 5.55(dd, 1H, J_1 = 16 Hz, J_2 = 8 Hz), 5.33-5.29(m, 1H), 4.01-3.96(m, 1H), 2.97(dd, 1H, J_1 = 17 Hz, J_2 = 13.5 Hz), 2.73(dd, 1H, J_1 = 17 Hz, J_2 = 7.5 Hz)
^{13}CNMR:
δ 171.6, 138.9, 135.5, 134.5, 133.7, 128.8, 128.6, 128.5, 128.1, 127.8, 127.7, 126.5, 122.9, 65.4, 43.4, 35.0
HRMS-EI Calculated for $C_{24}H_{21}NO_3S$: 403.1242, Found: 403.1267

Figure 1: X-ray crystal structure of 4b:

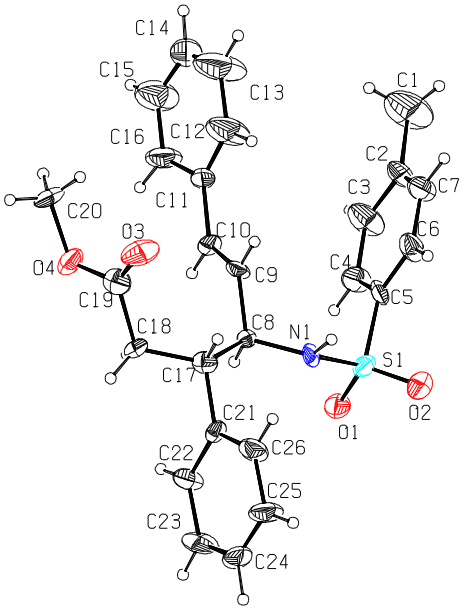
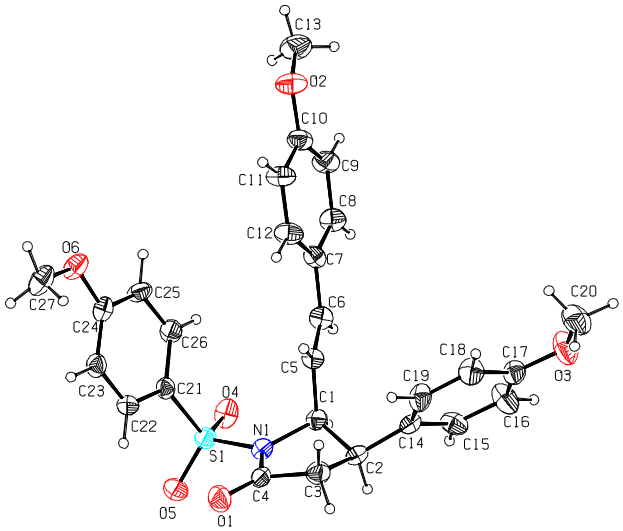
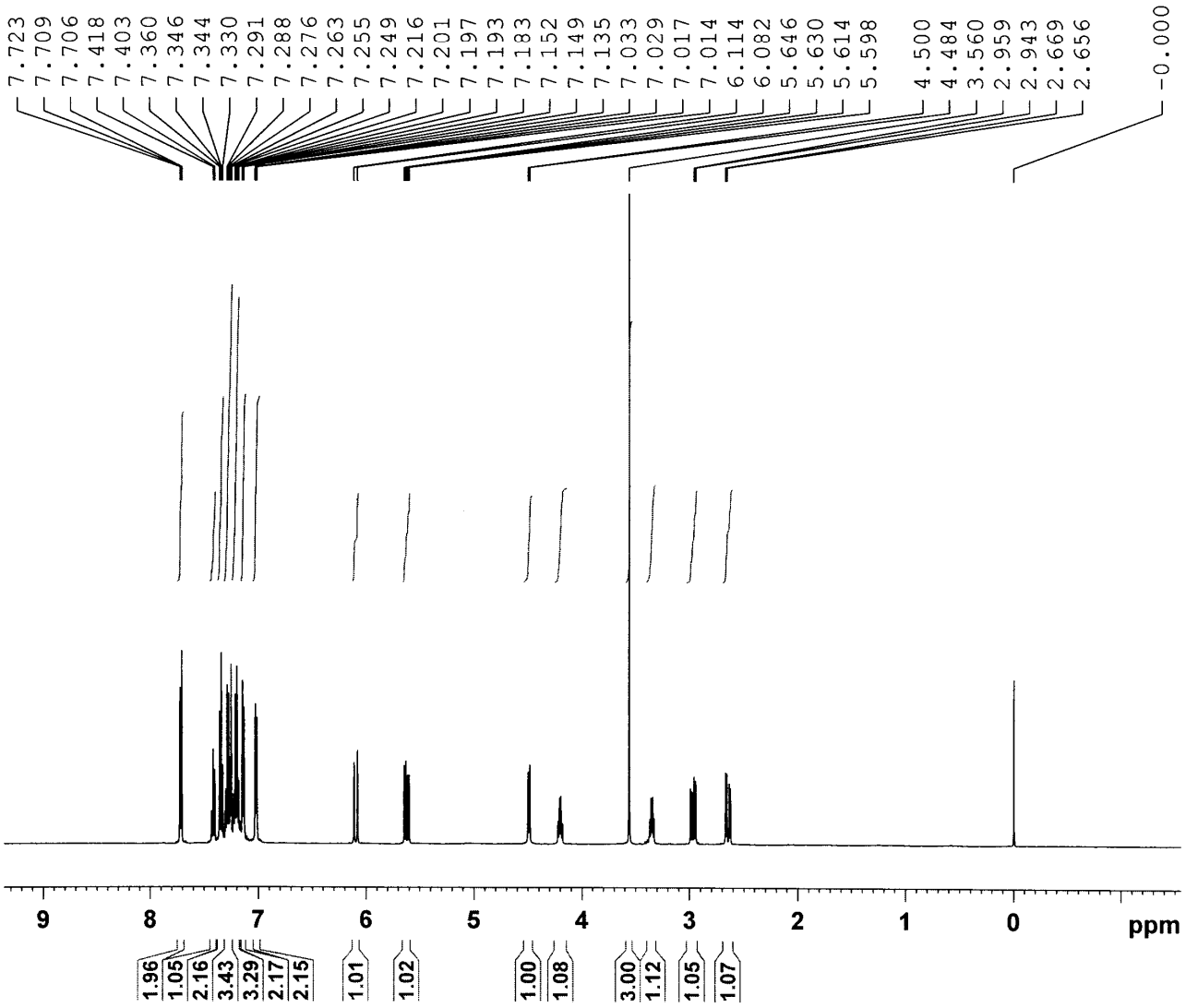


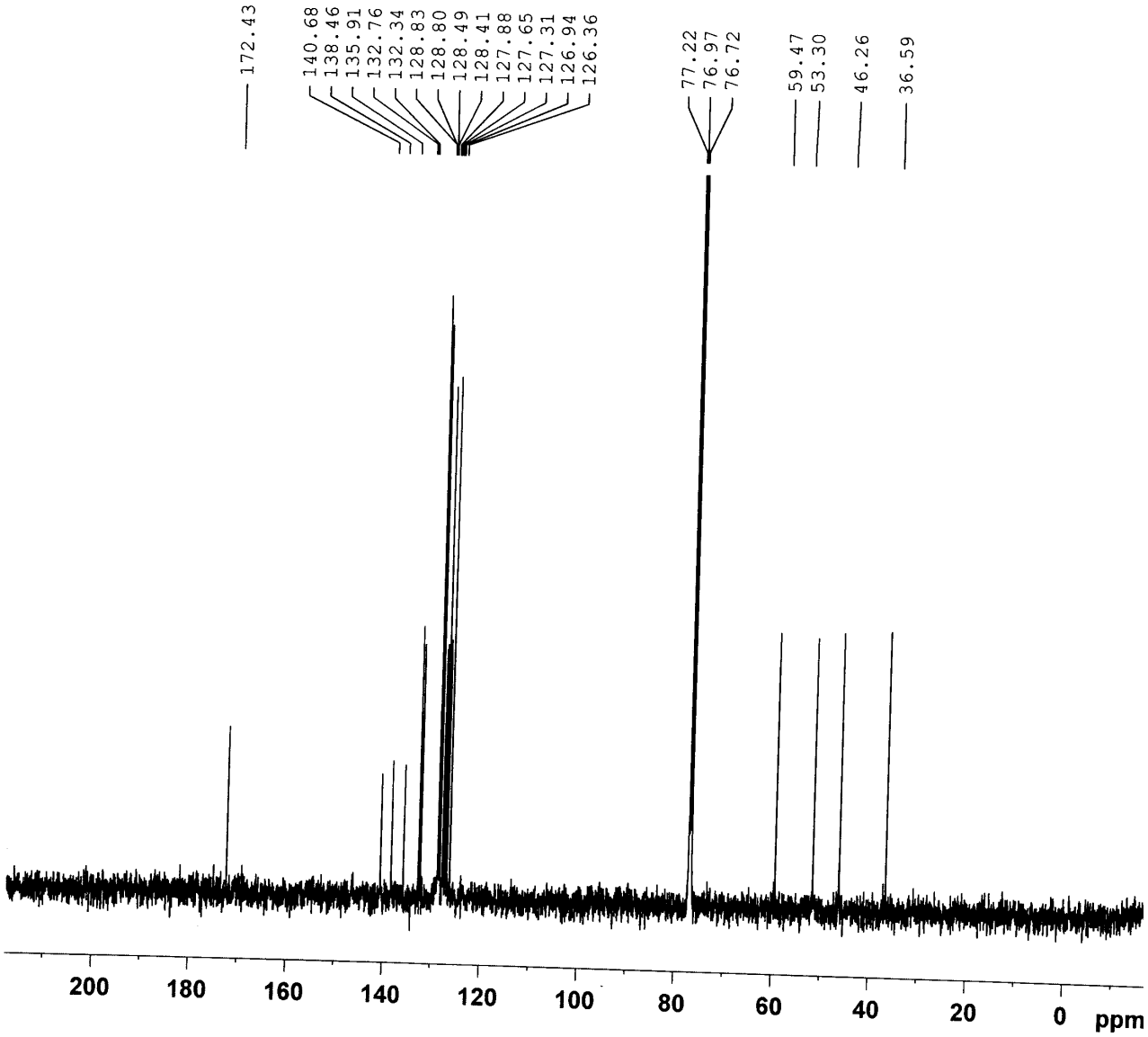
Figure 2: X-ray crystal structure of 8a:



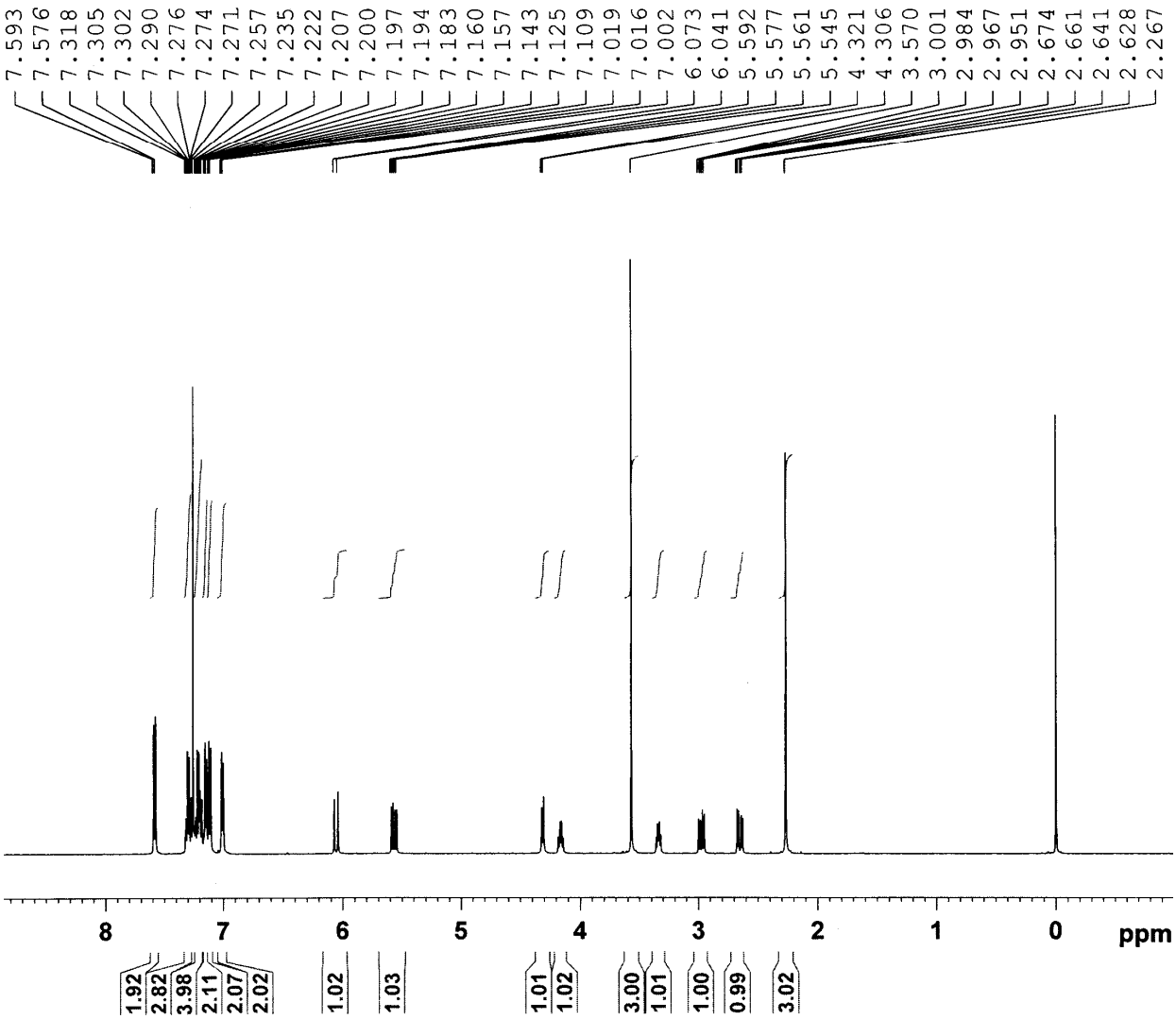
Compound 4a – ^{1}H NMR (500 MHz)



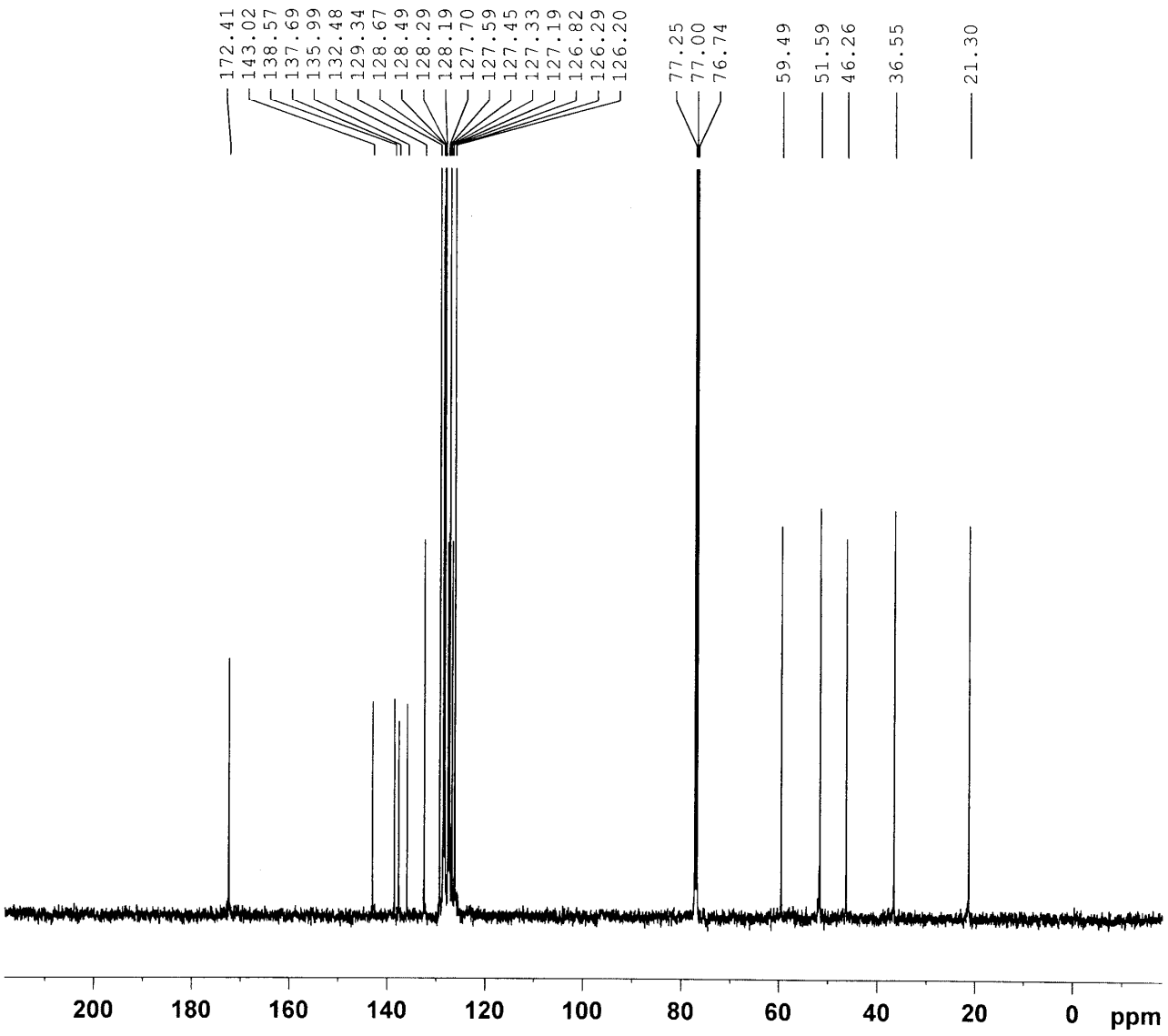
Compound 4a – ^{13}C NMR (125 MHz)



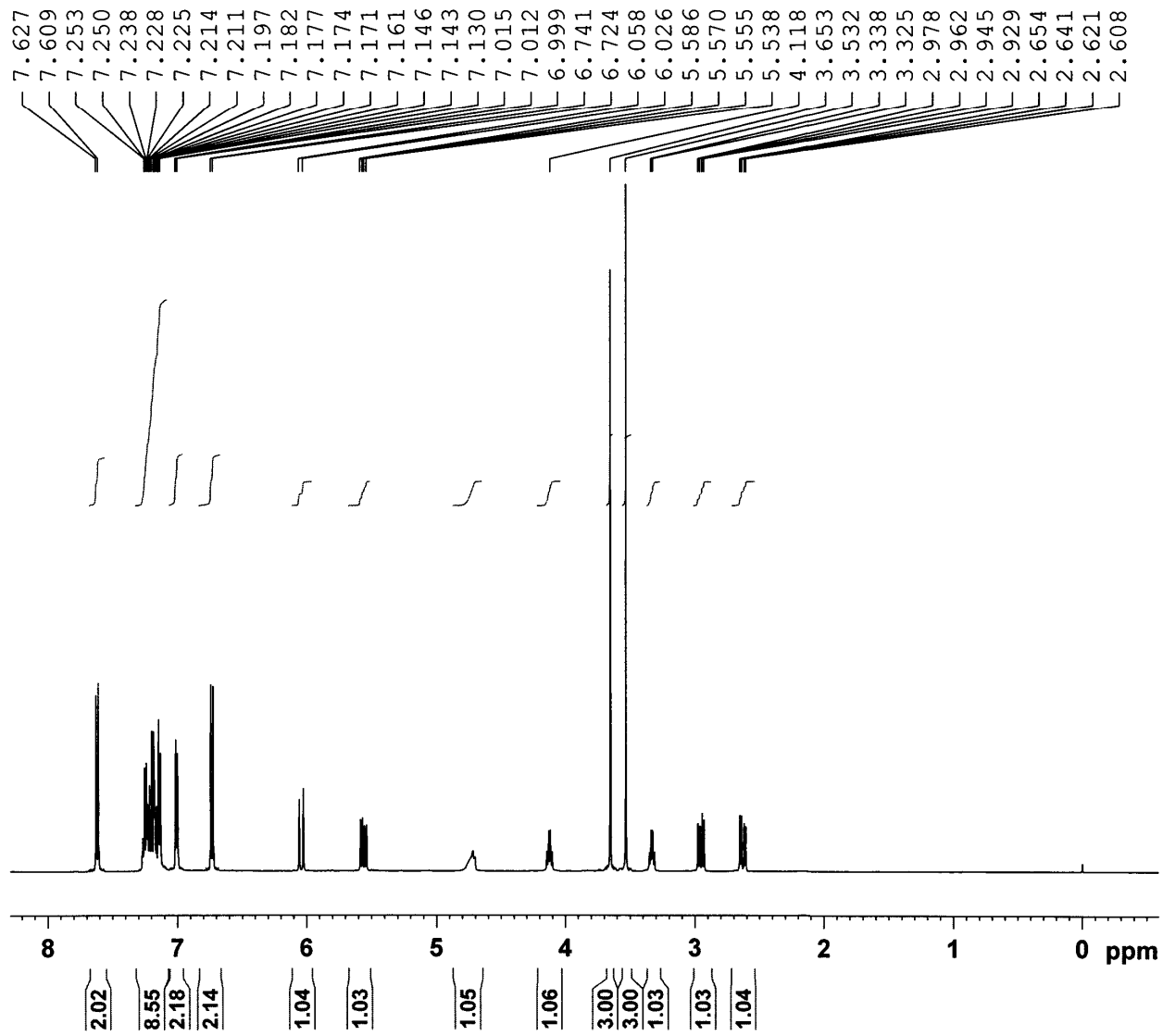
Compound 4b – ^{1}H (500 MHz)



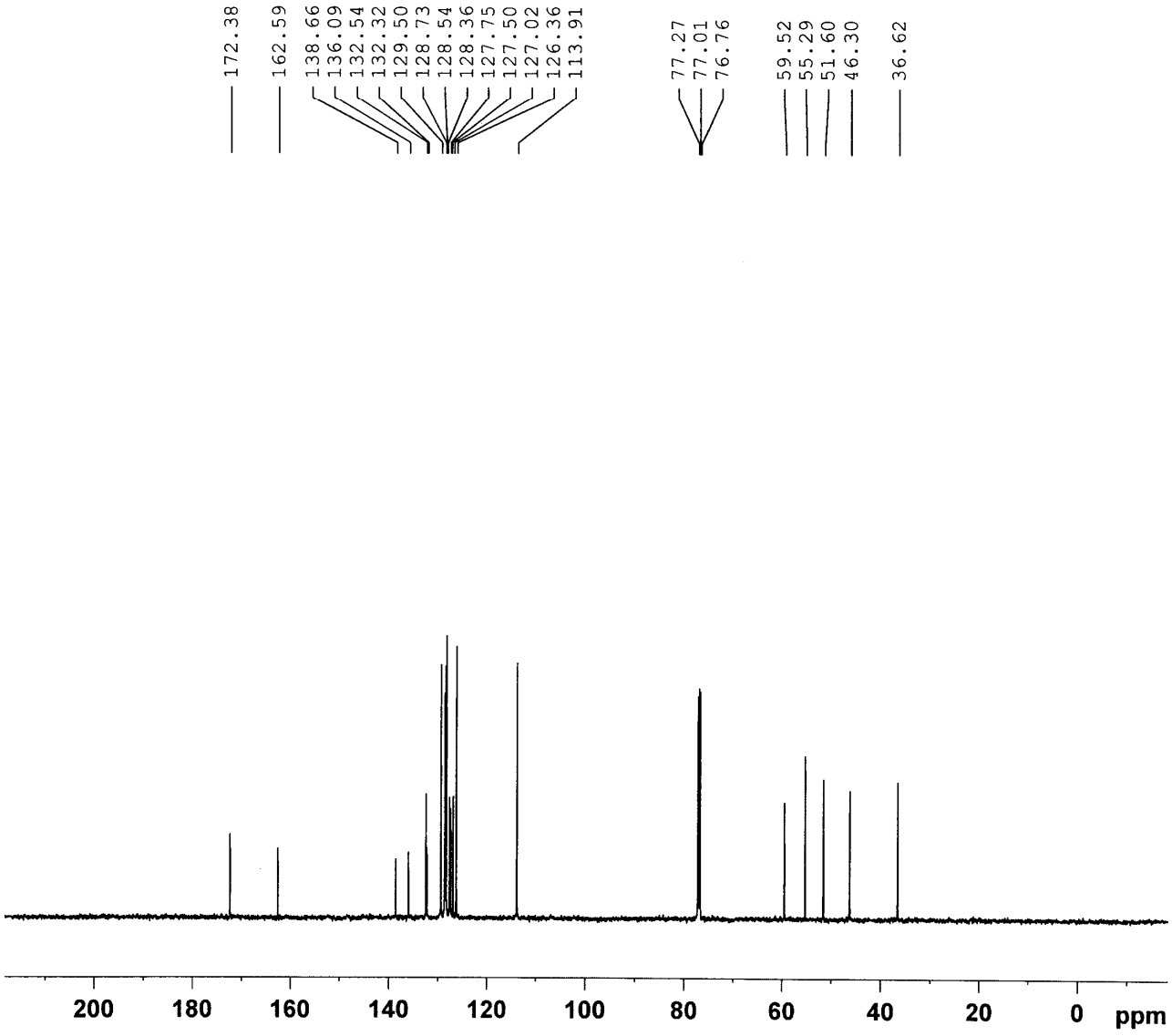
Compound 4b – ^{13}C (125 MHz)



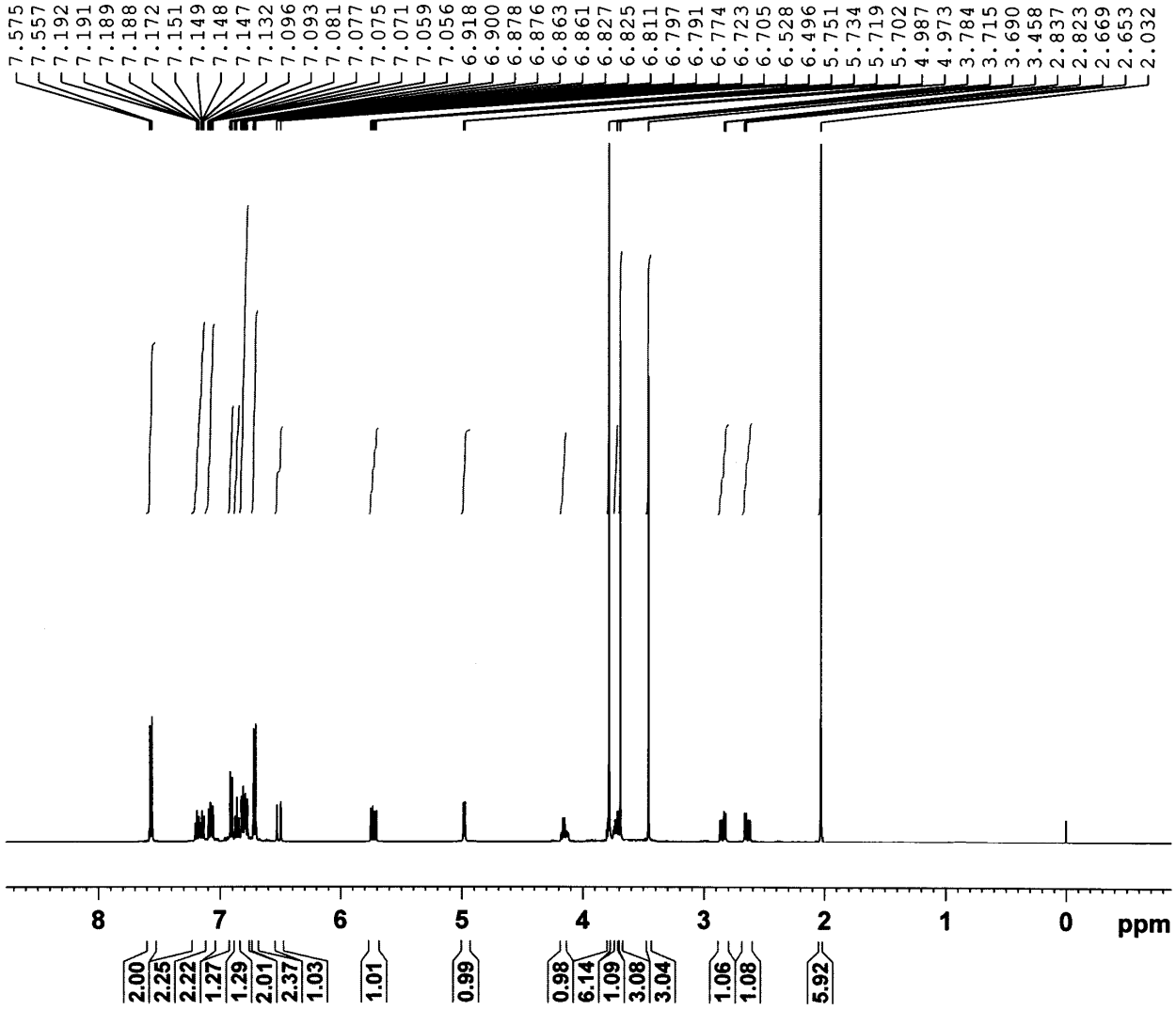
Compound 4c – ^{1}H (500 MHz)



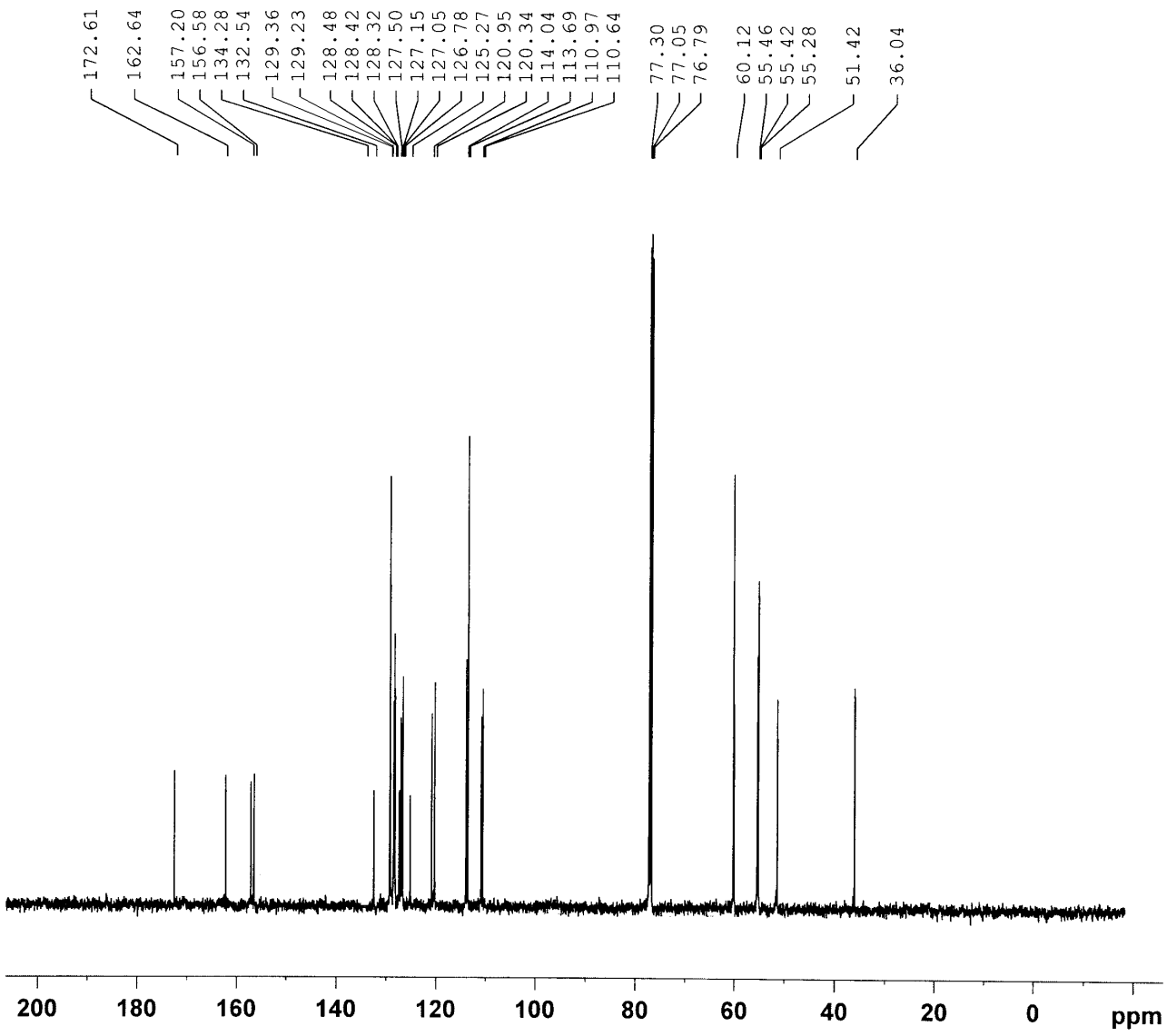
Compound 4c – ^{13}C (125 MHz)



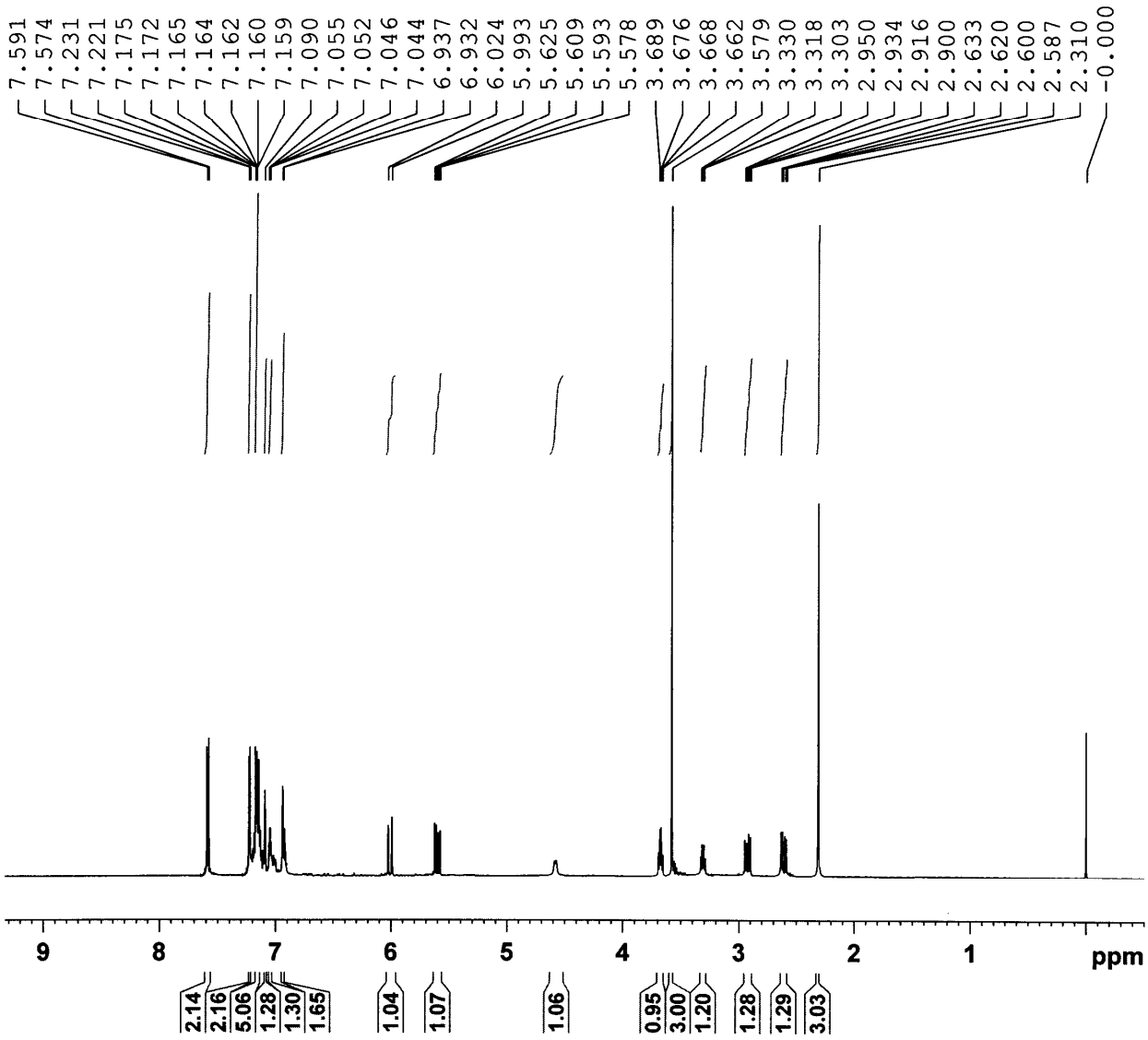
Compound 4d – ^{1}H (500 MHz)



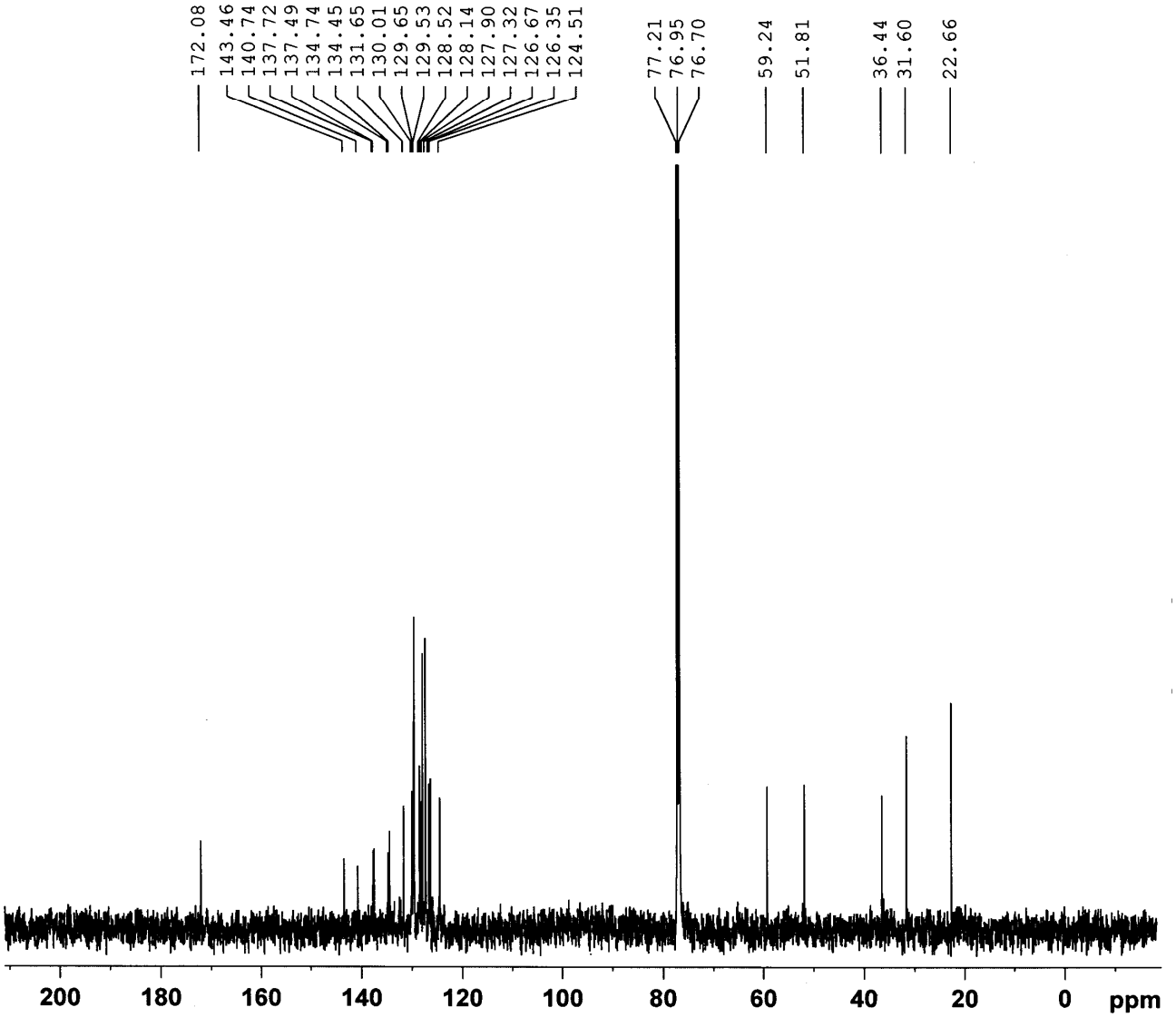
Compound 4d - ^{13}C (125 MHz)



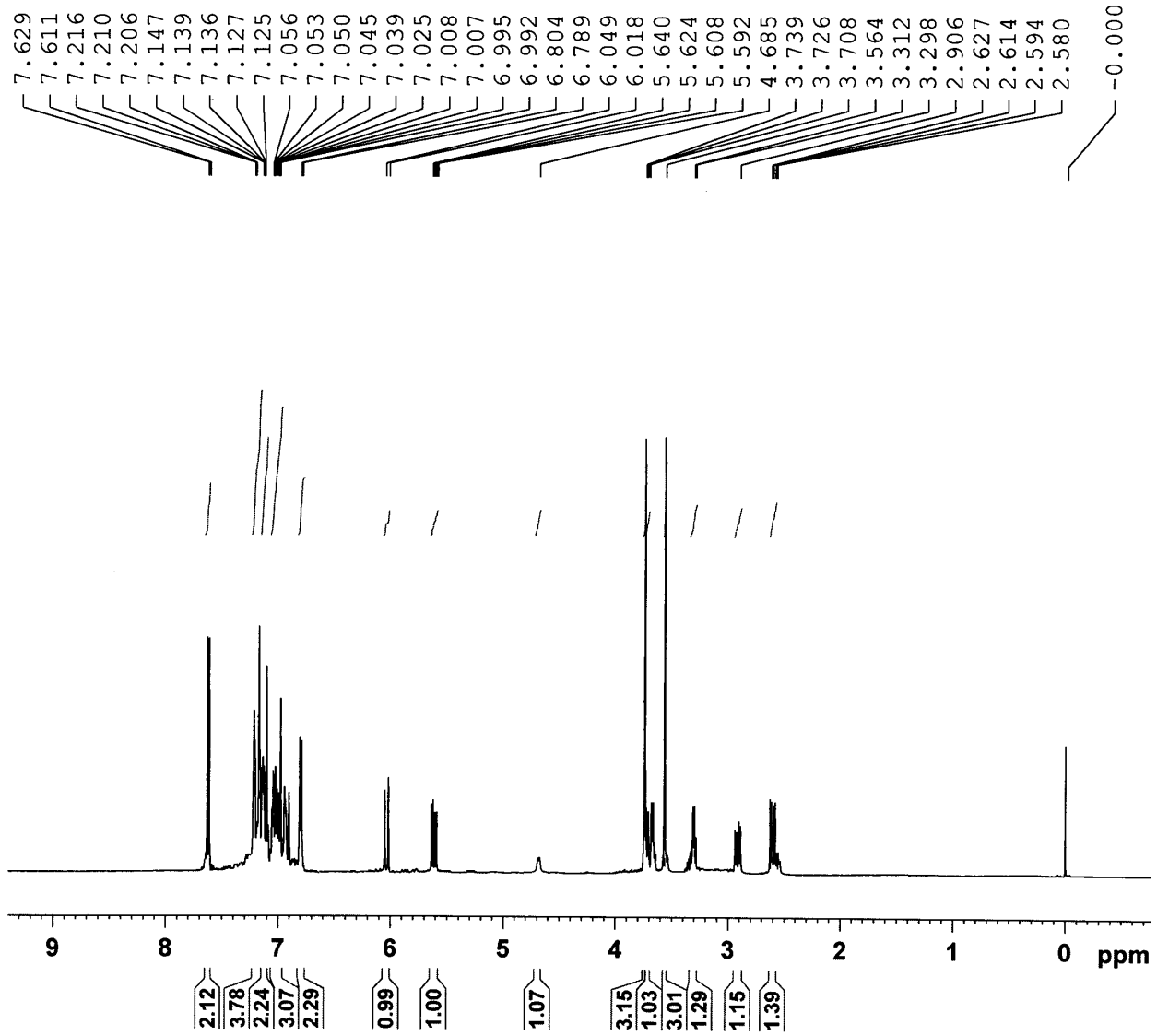
Compound 4e – ^{1}H (500 MHz)



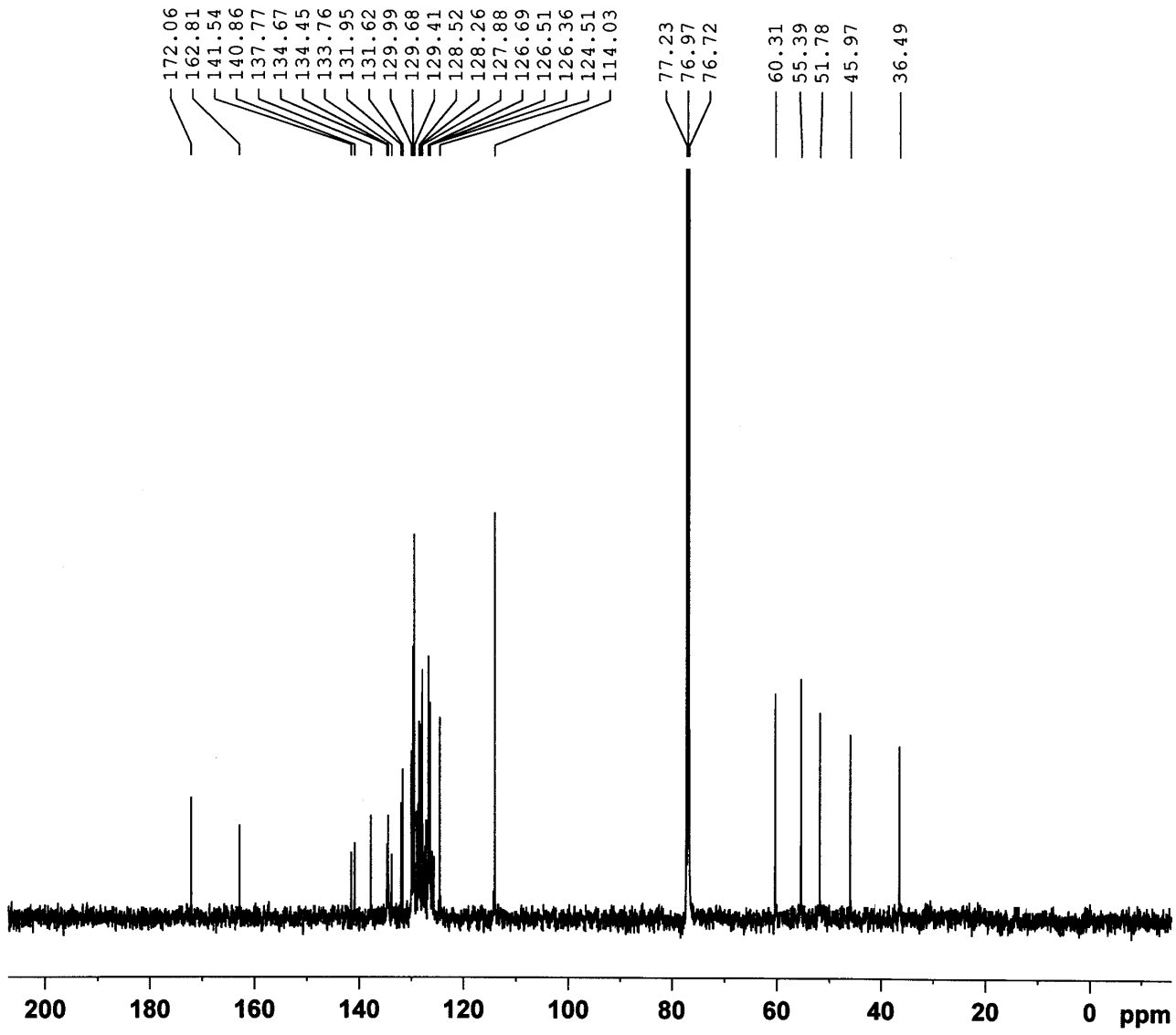
Compound 4e – ^{13}C (125 MHz)



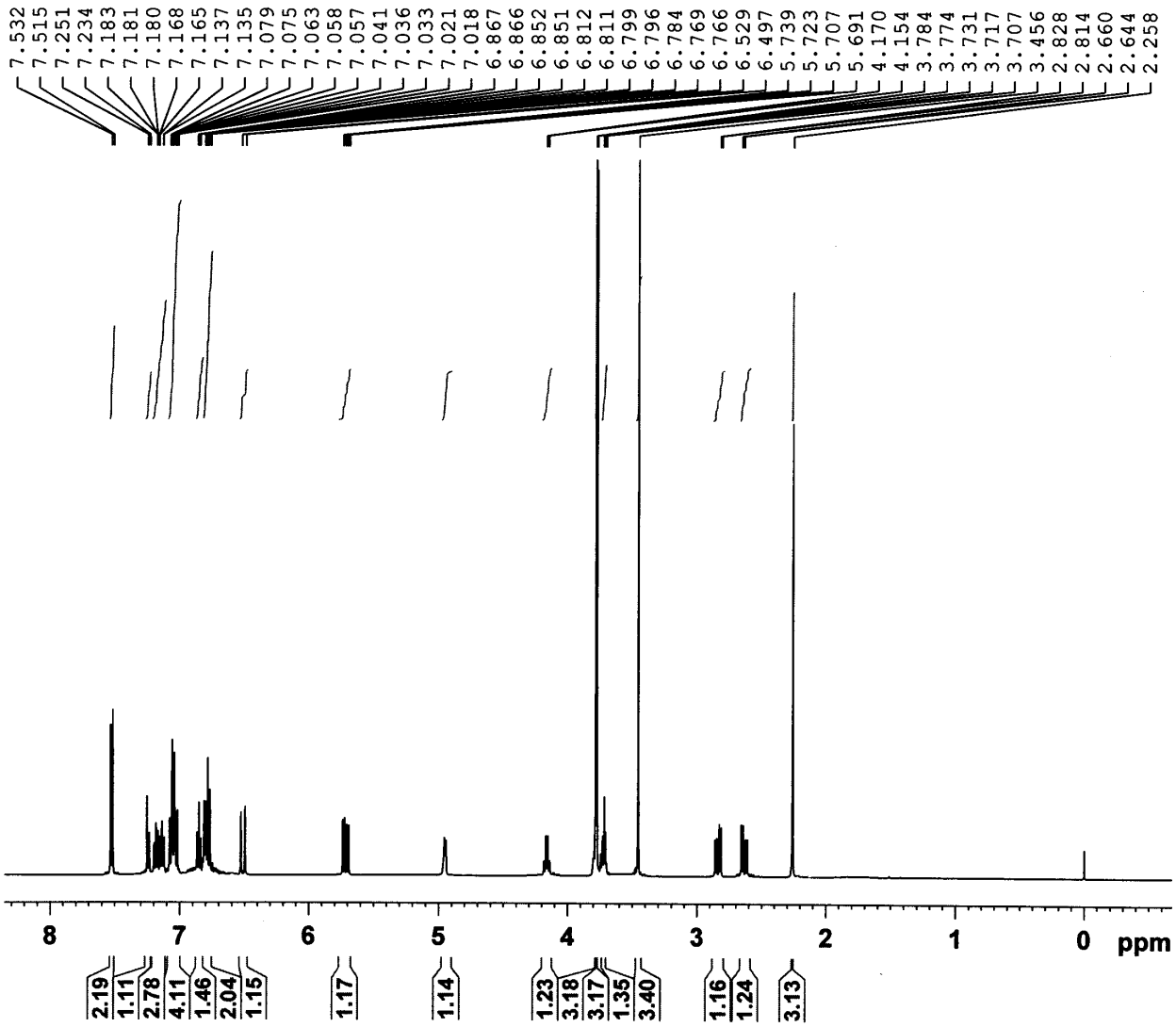
Compound 4f – ^{1}H (500 MHz)



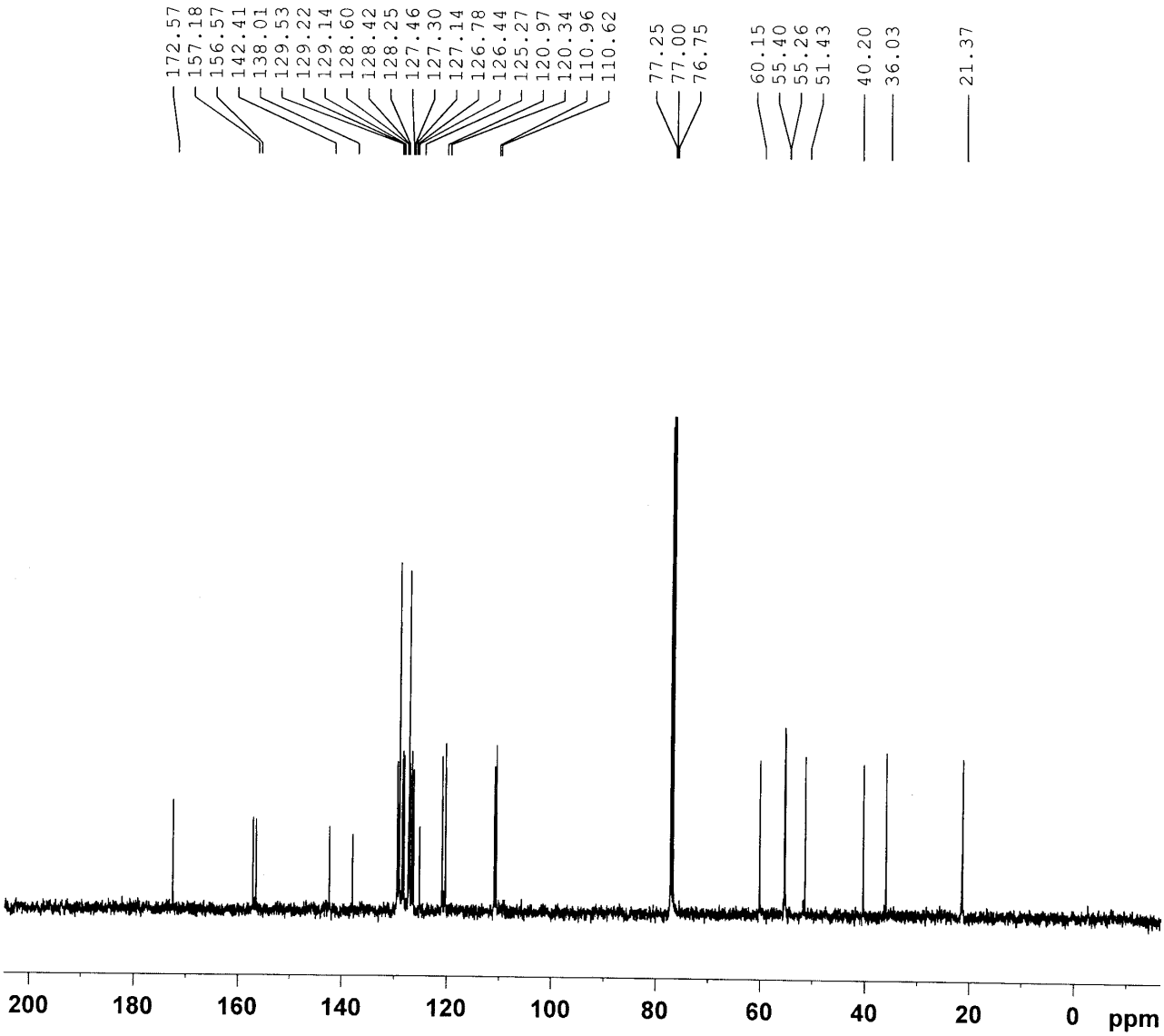
Compound4f – ^{13}C (125 MHz)



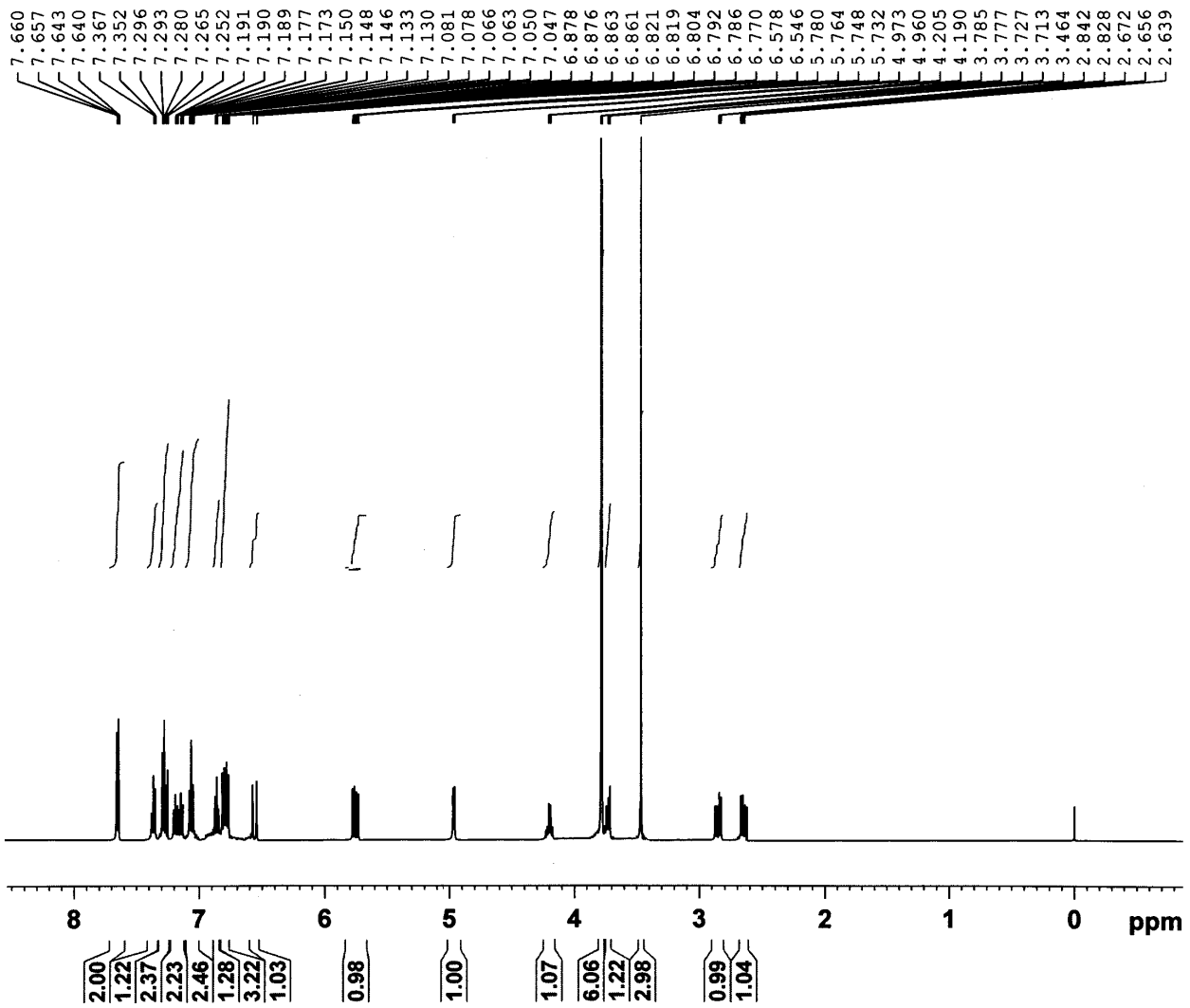
Compound 4g – ^{1}H (500 MHz)



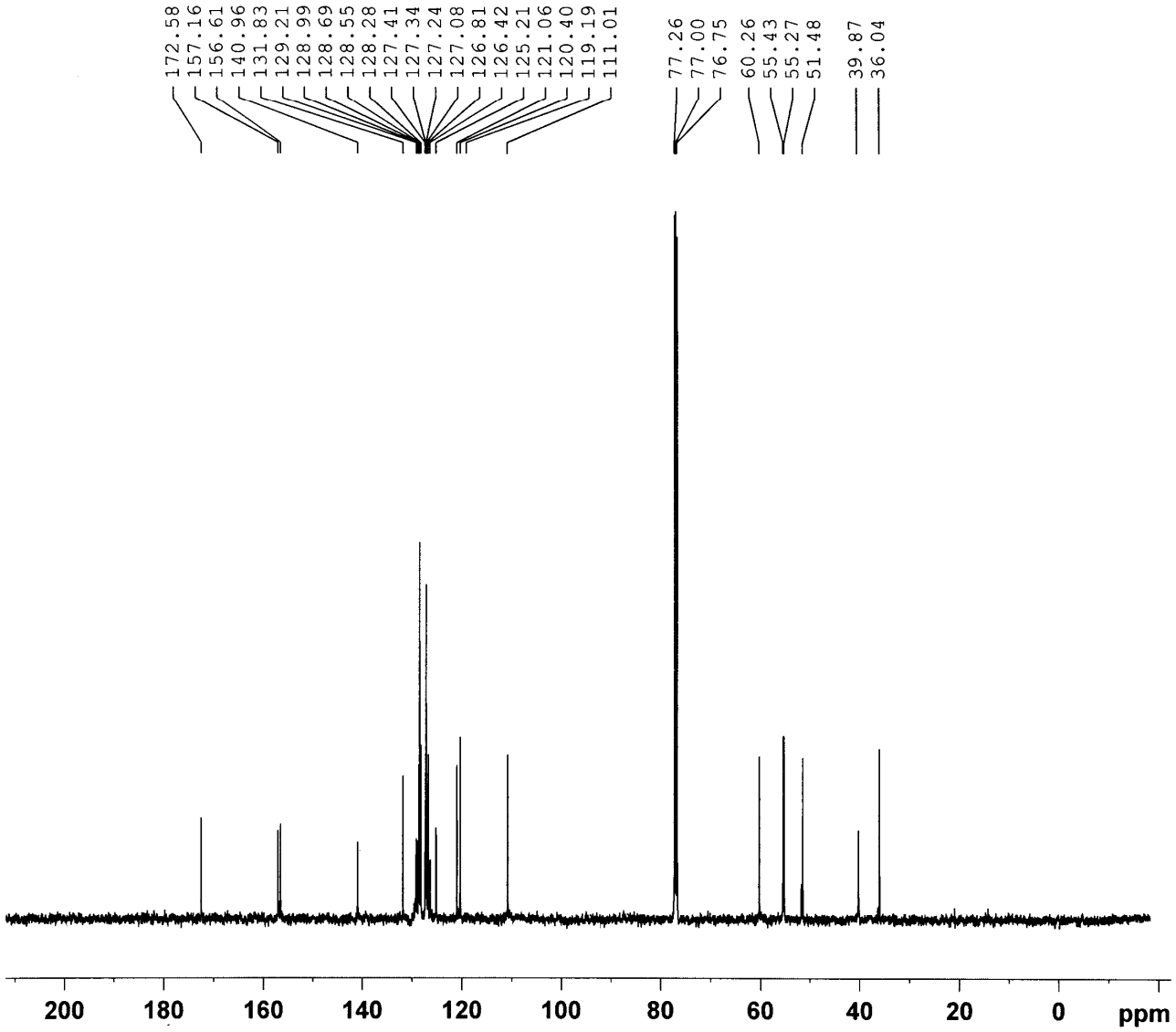
Compound 4g – ^{13}C (125 MHz)



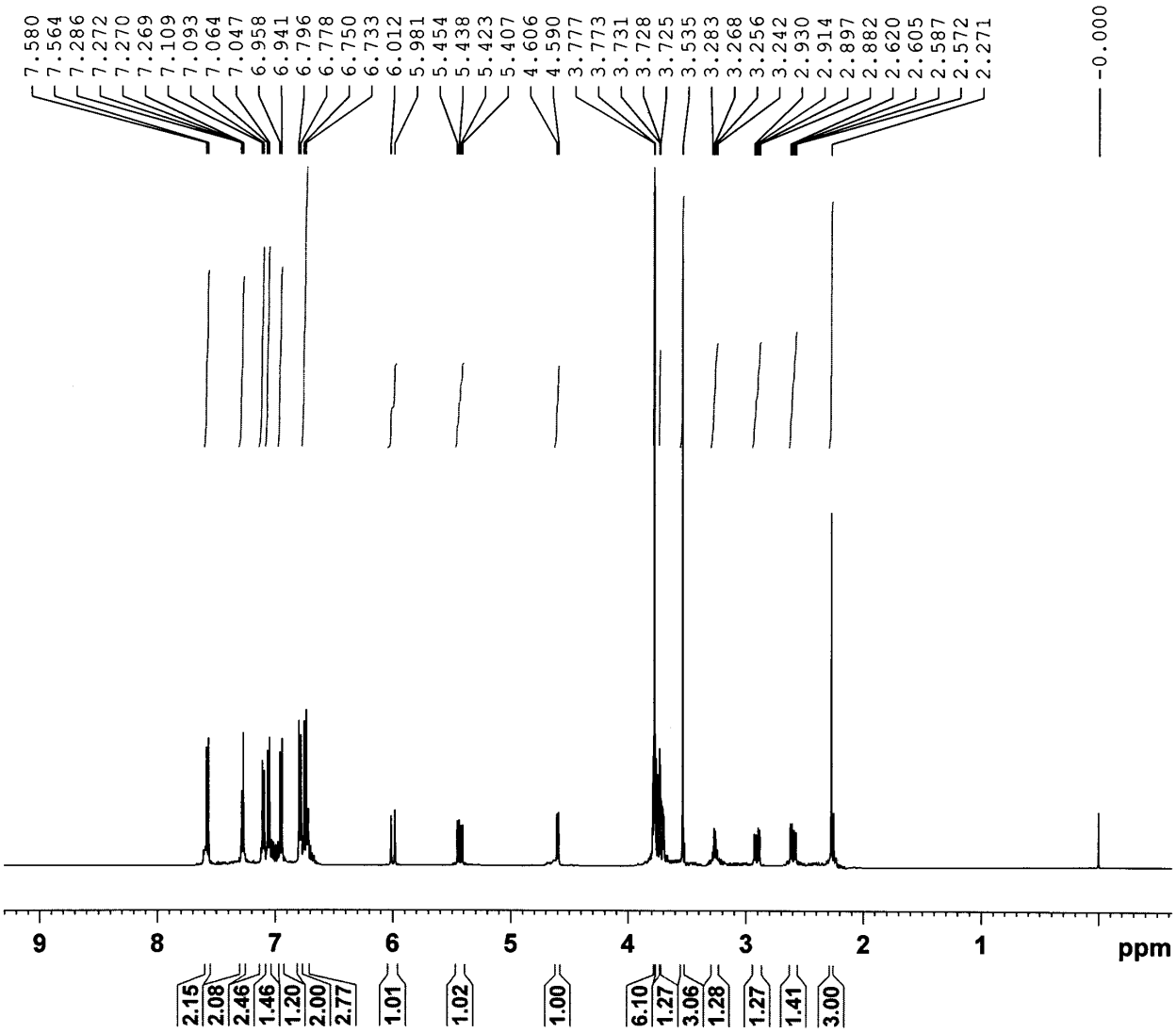
Compound 4h – ^{1}H (500 MHz)



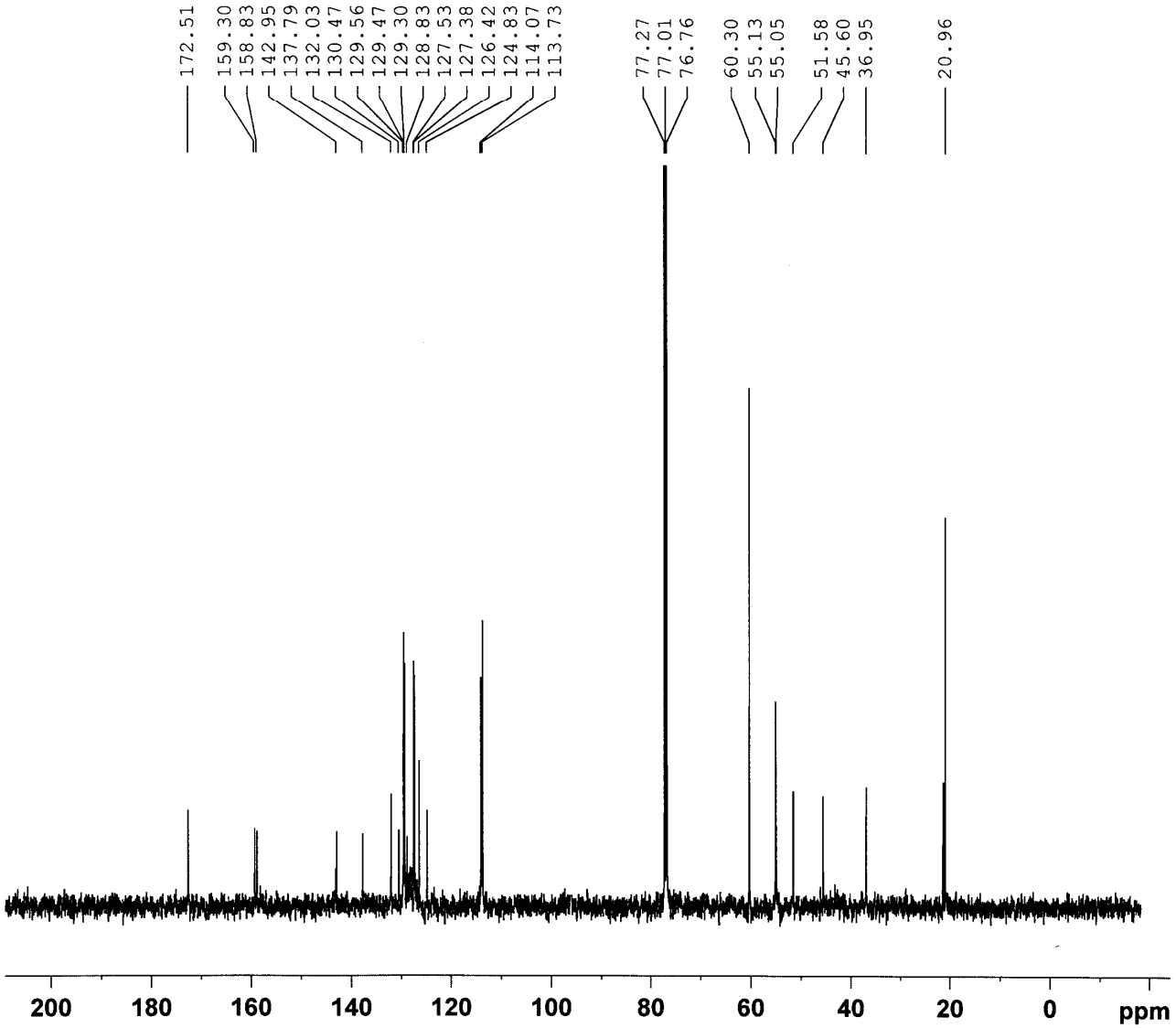
Compound 4h – ^{13}C (125 MHz)



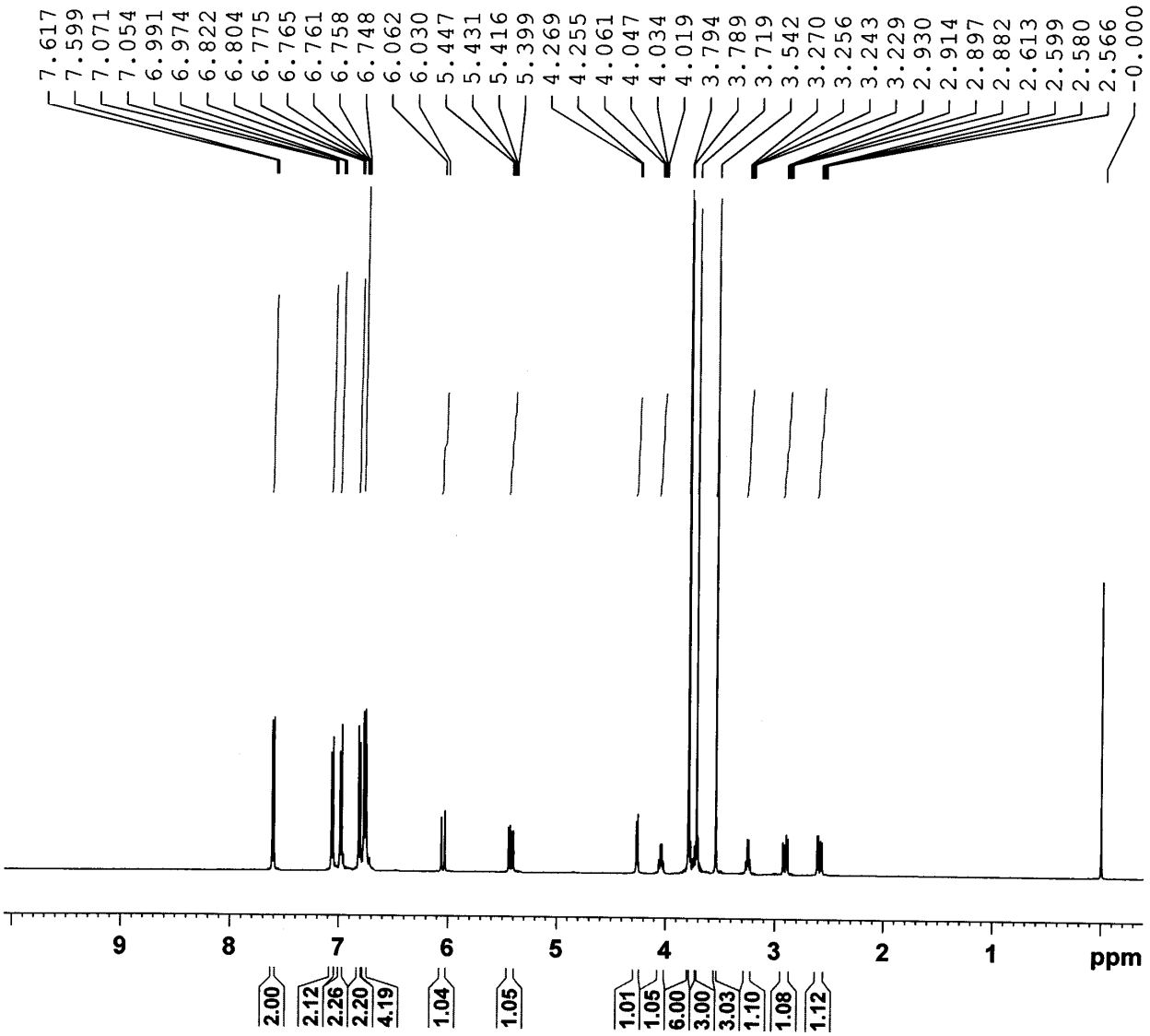
Compound 4i – ^{1}H (500 MHz)



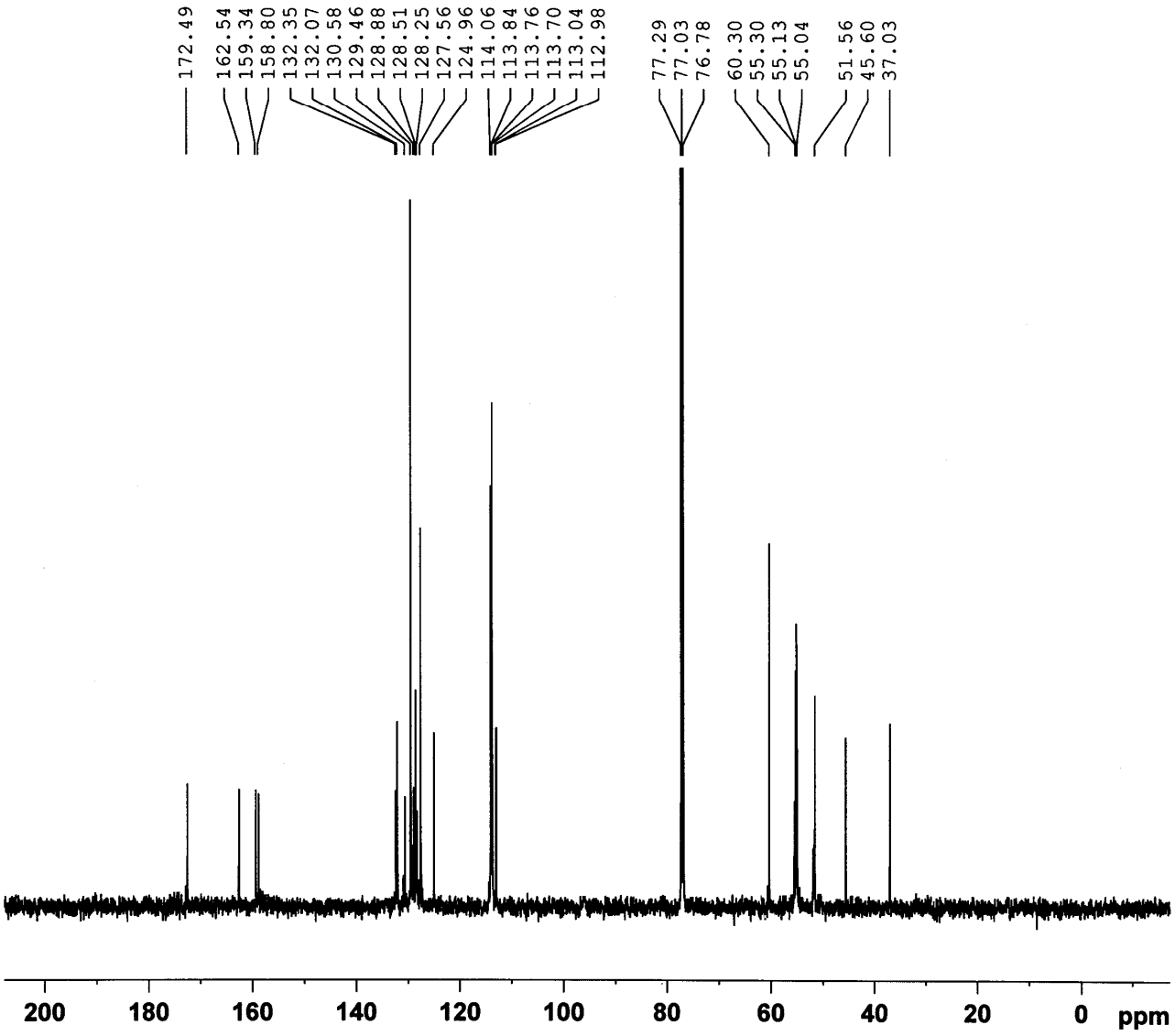
Compound 4i – ^{13}C (125 MHz)



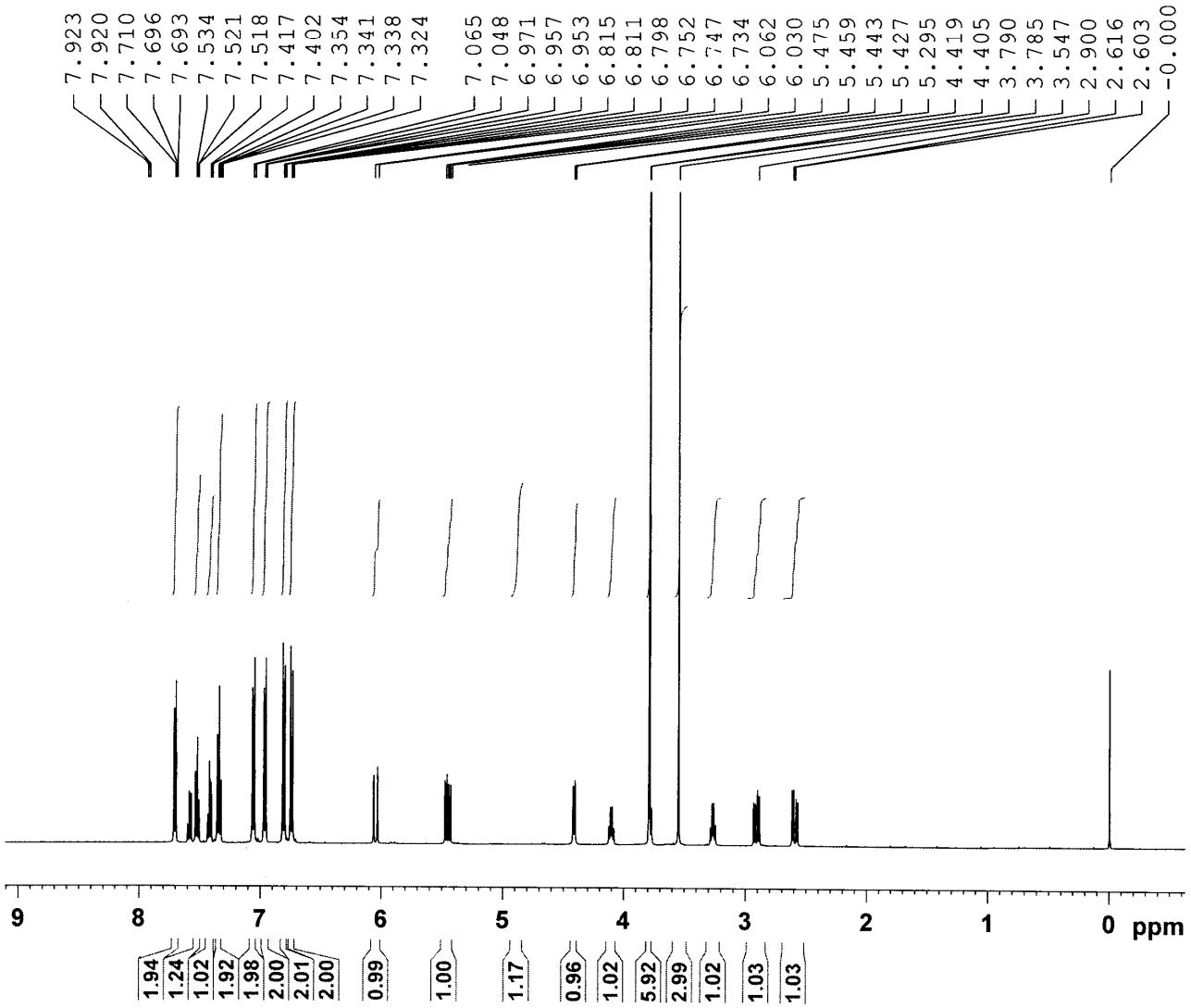
Compound4j - ^{1}H (500 MHz)



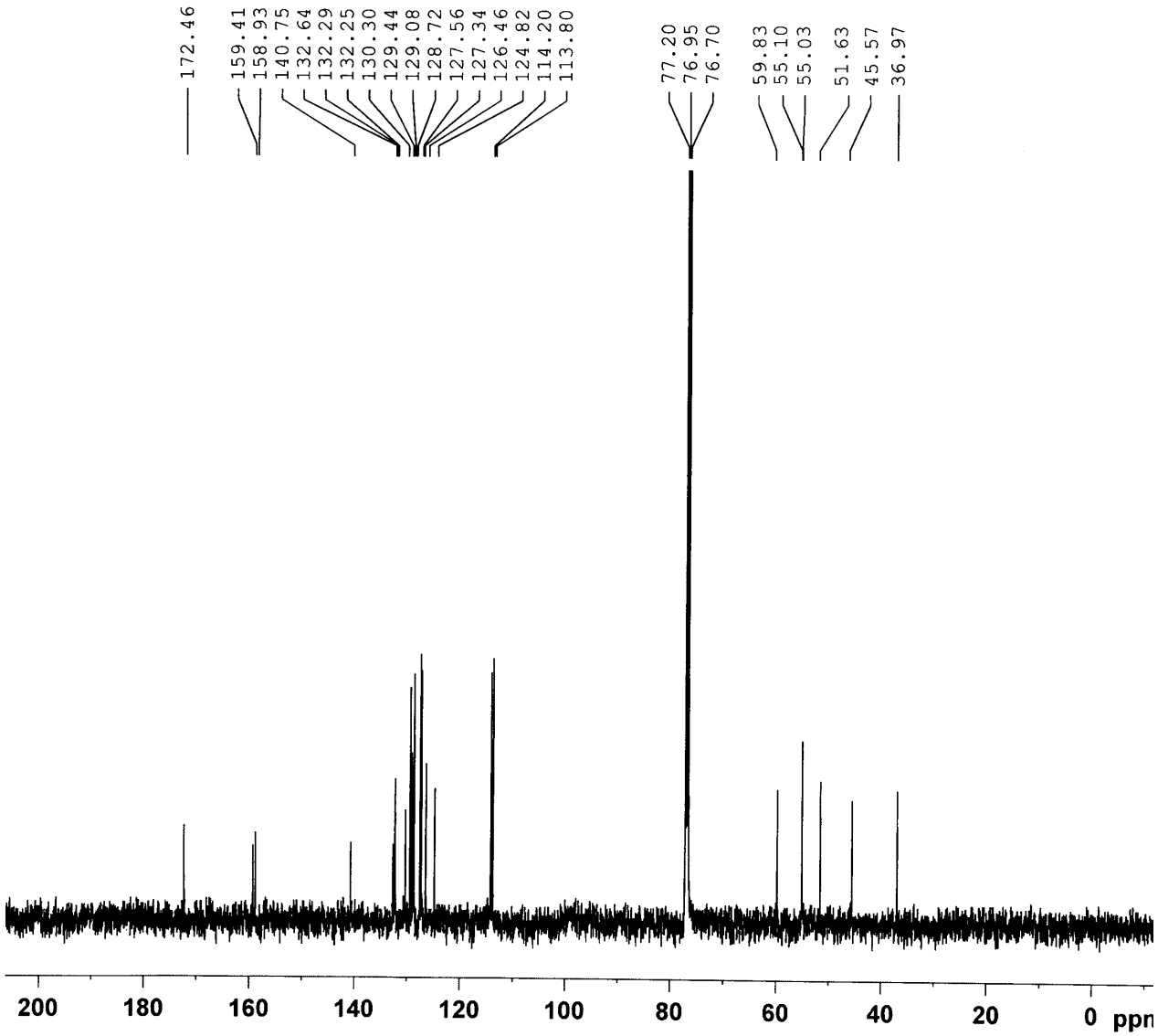
Compound 4j – ^{13}C (125 MHz)



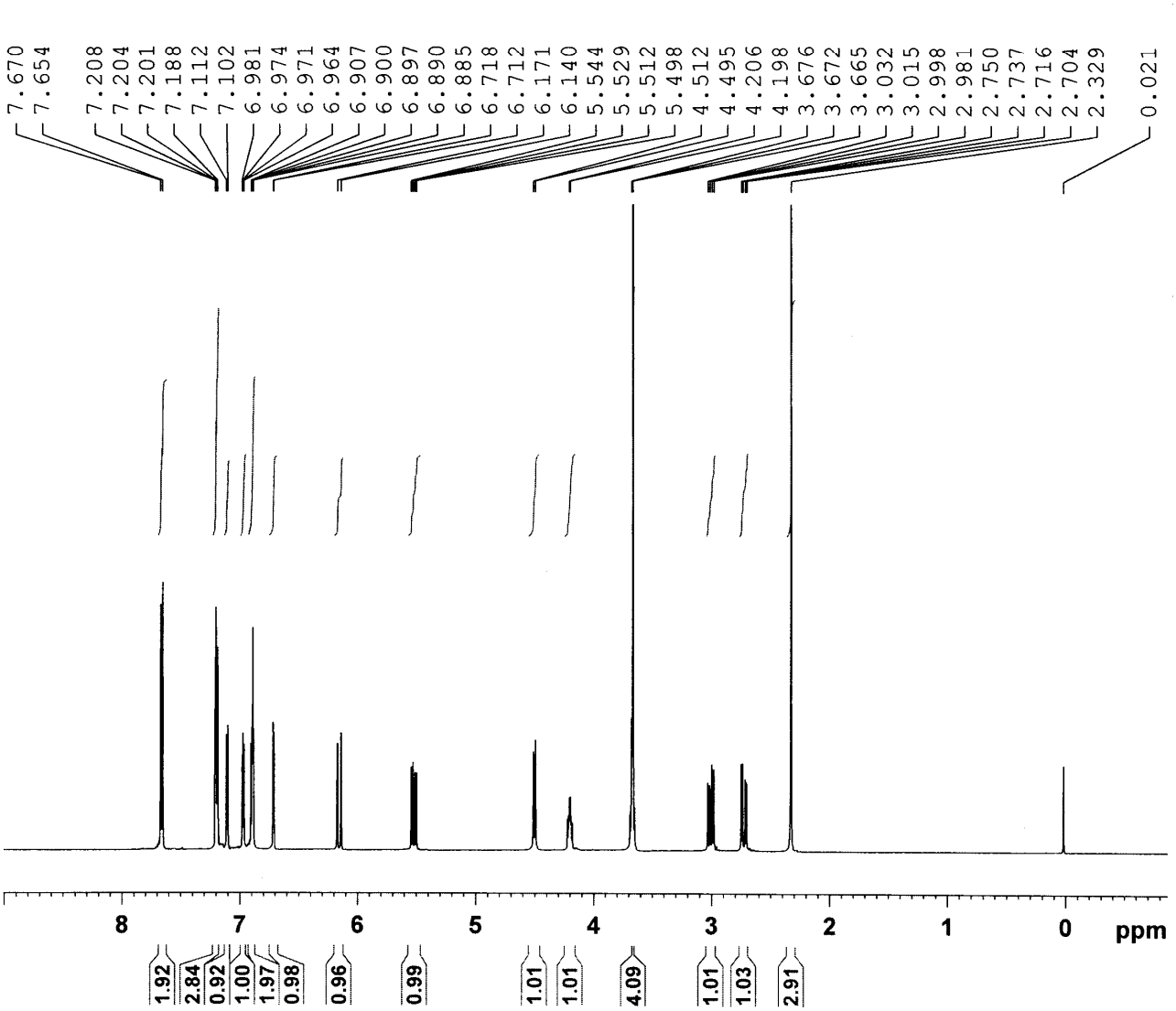
Compound 4k – ^{1}H (500 MHz)



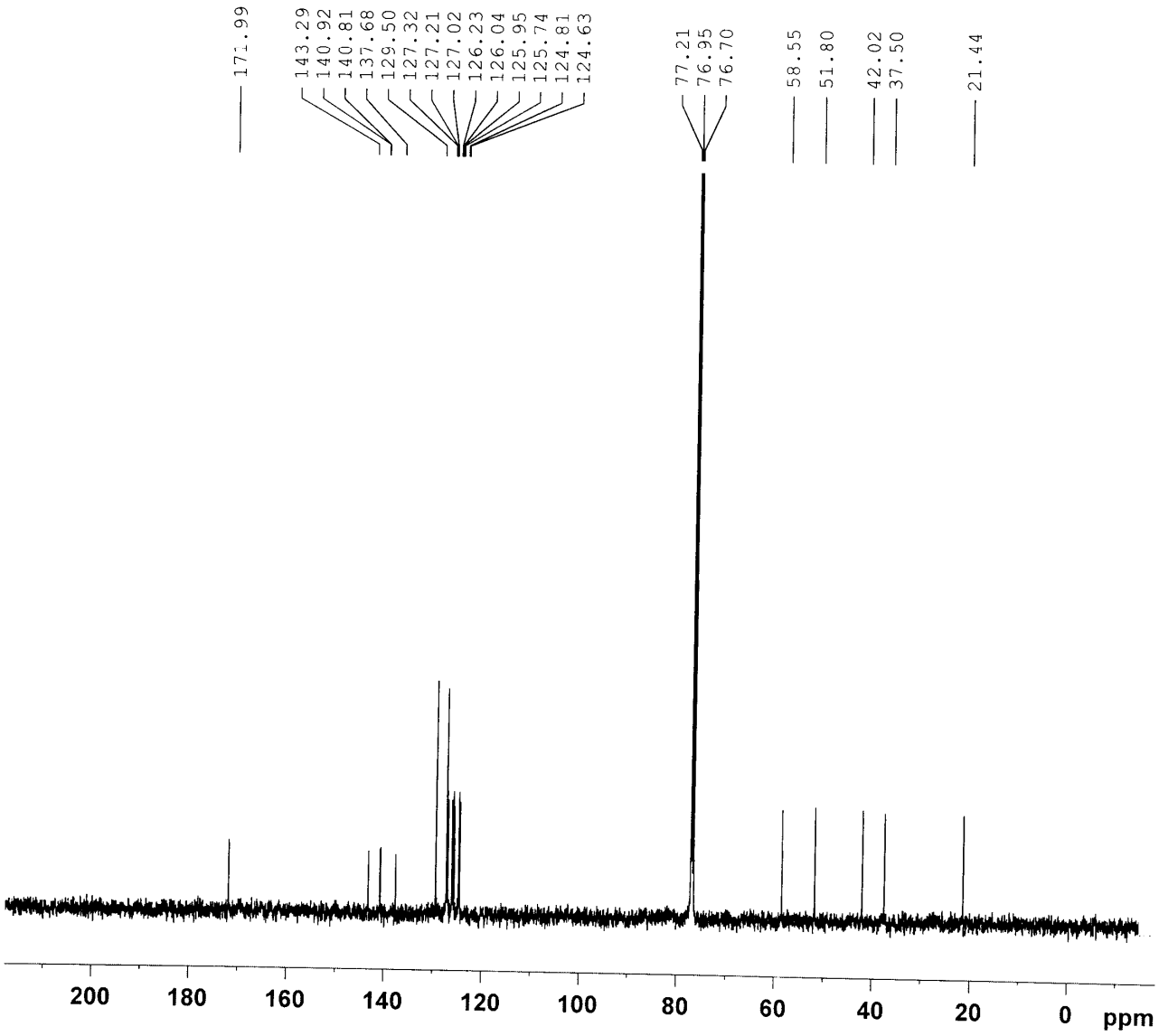
Compound 4k – ^{13}C (125 MHz)



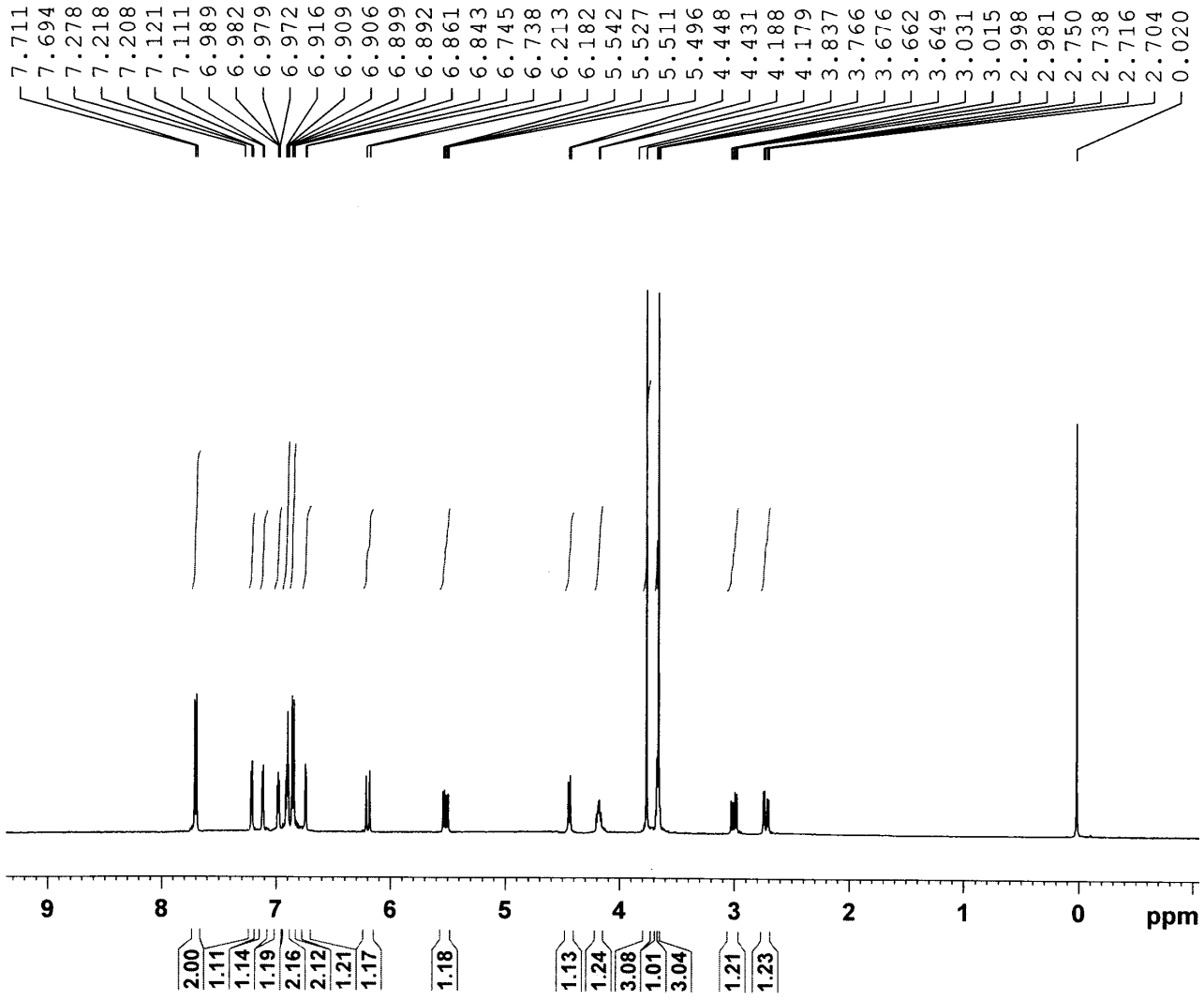
Compound 4l – ^{1}H (500 MHz)



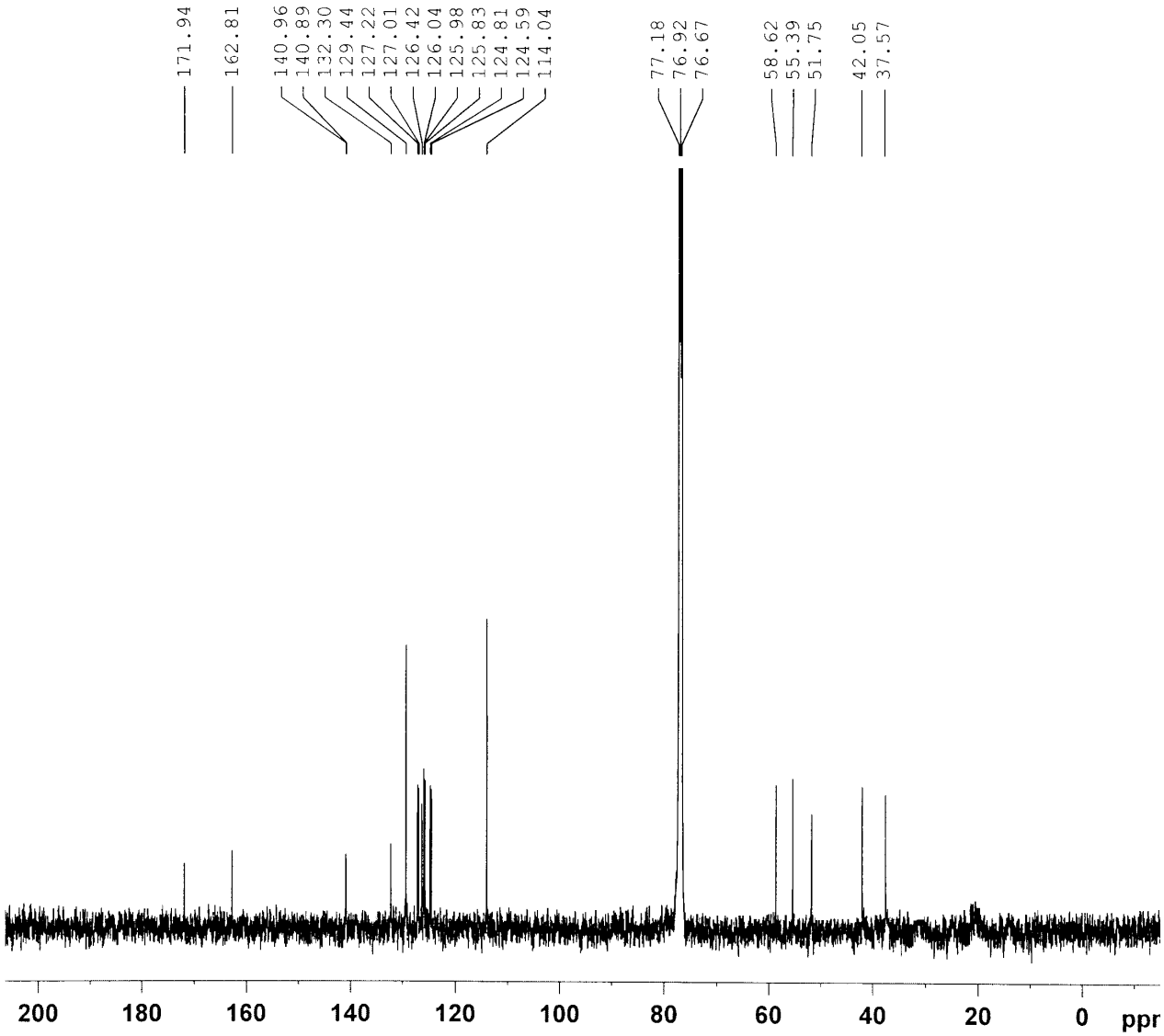
Compound 4l – ^{13}C (125 MHz)



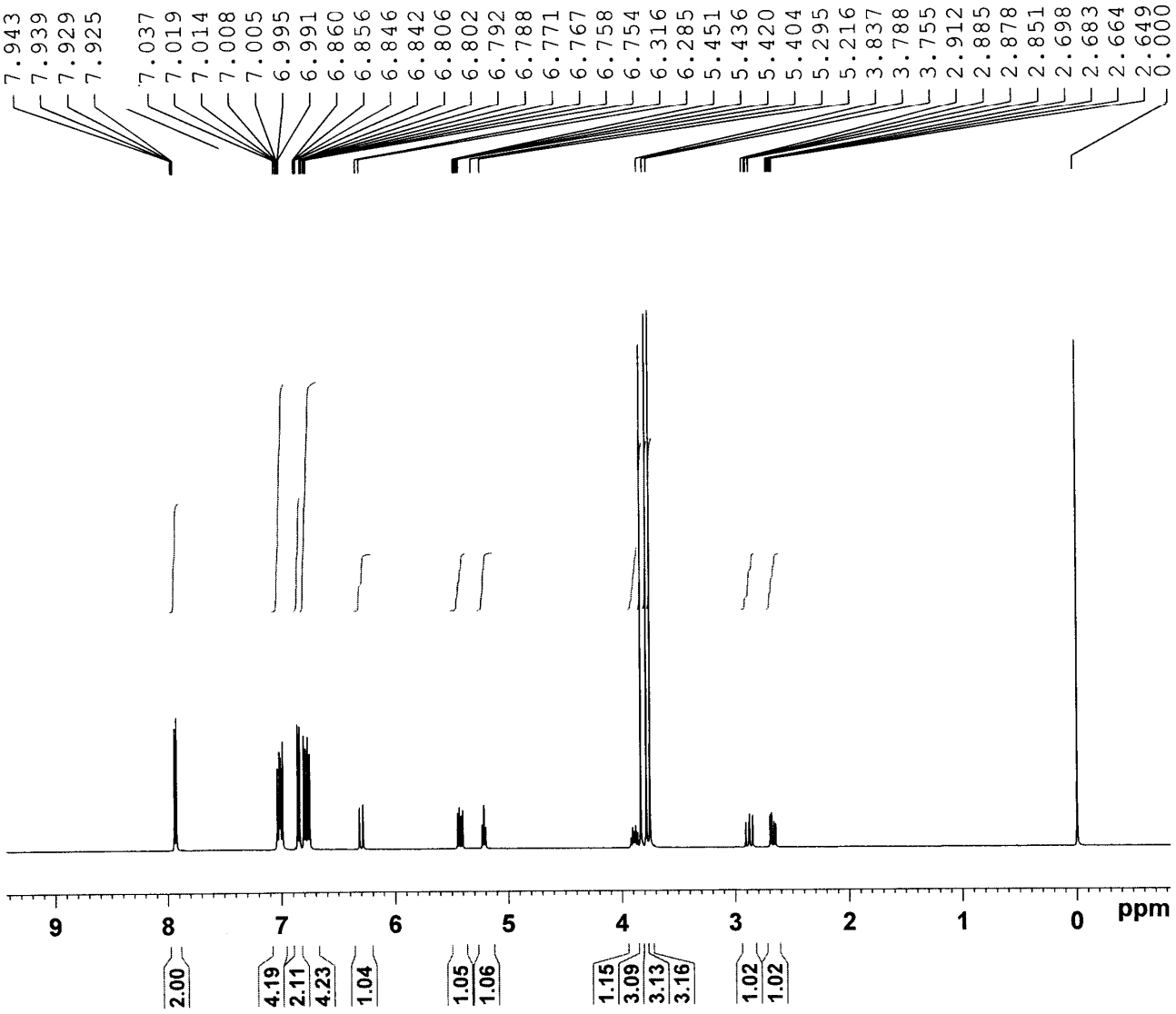
Compound 4m – ^{1}H (500 MHz)



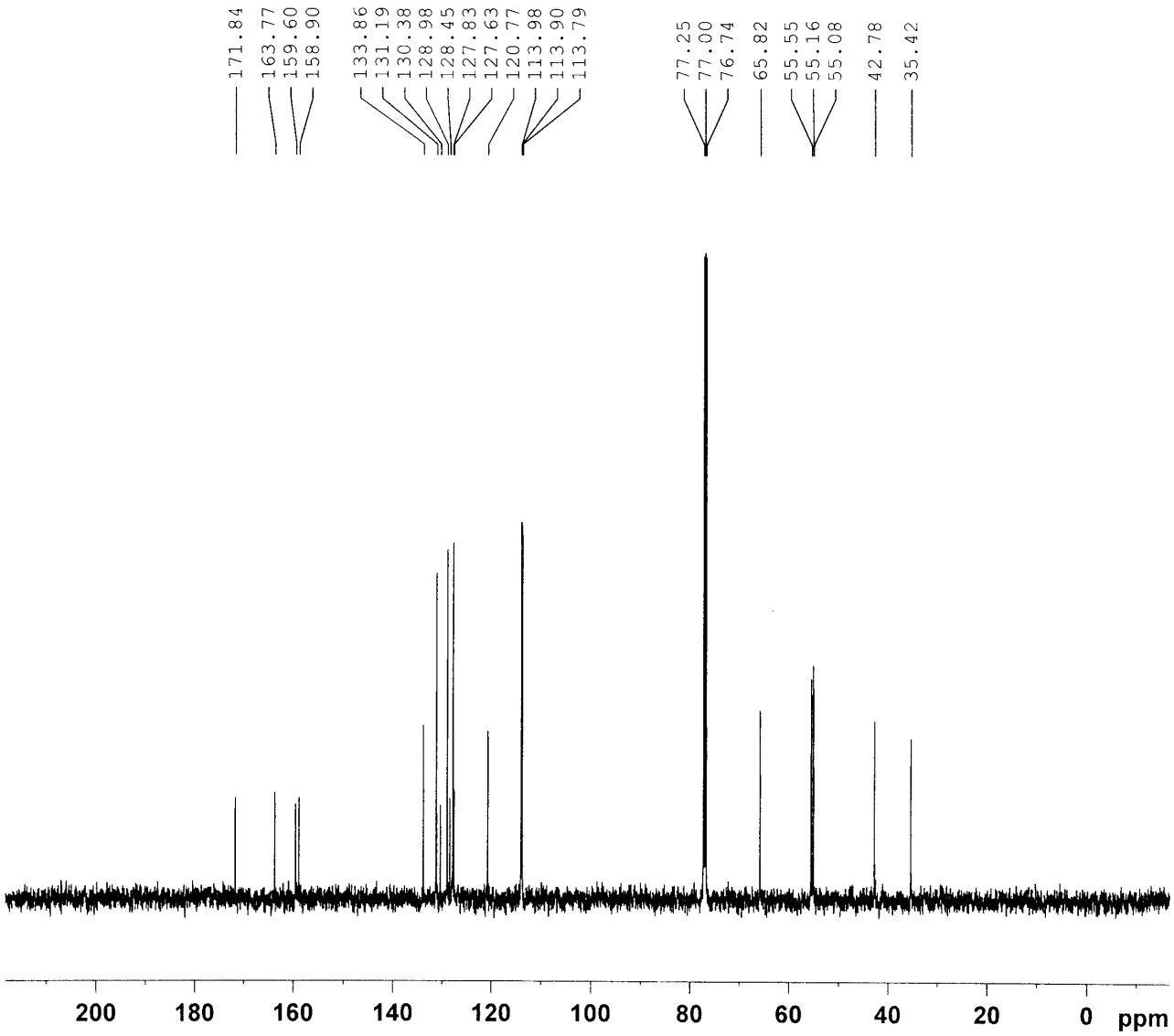
Compound 4m – ^{13}C (125 MHz)



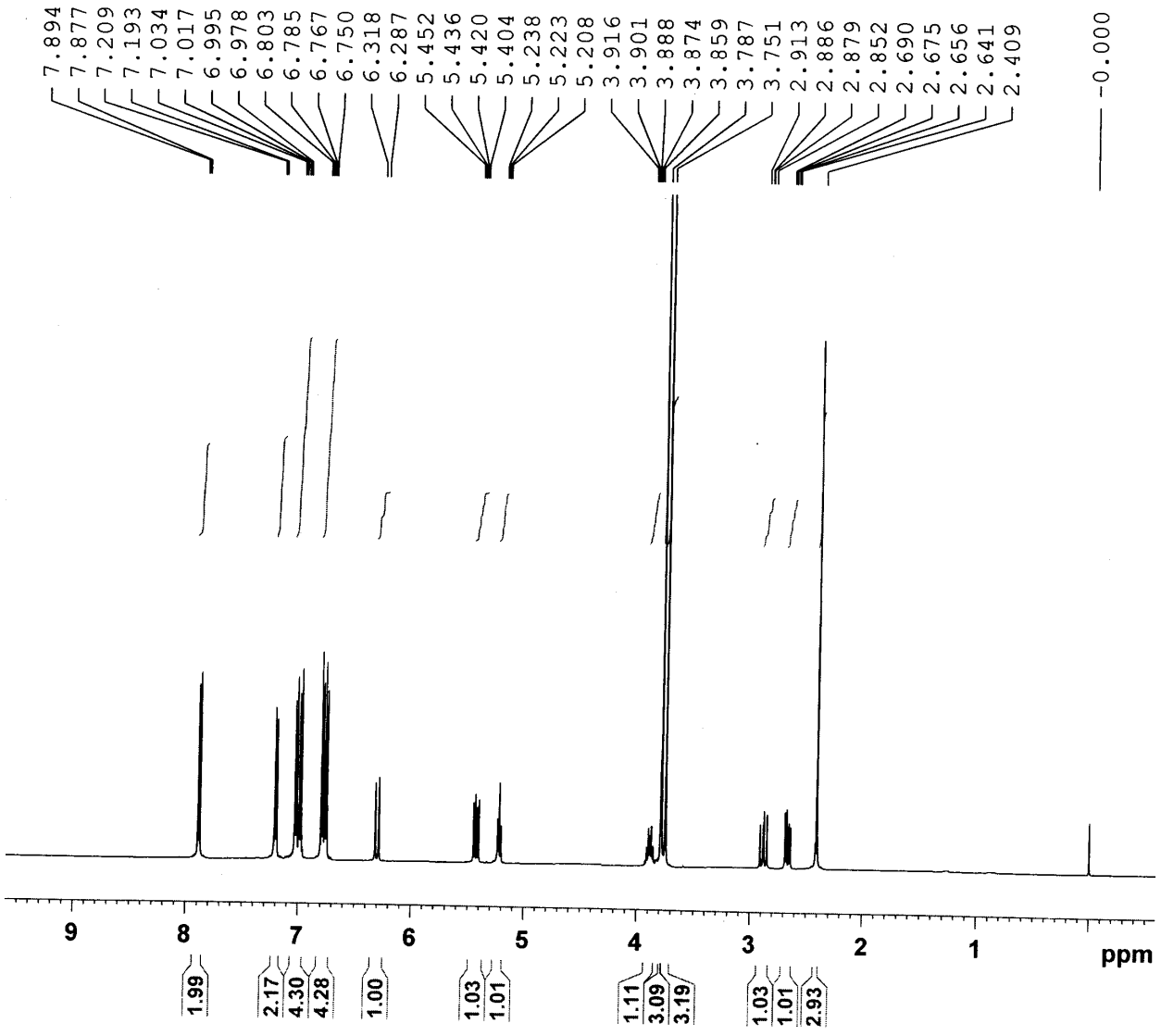
Compound 8a – ^{1}H (500 MHz)



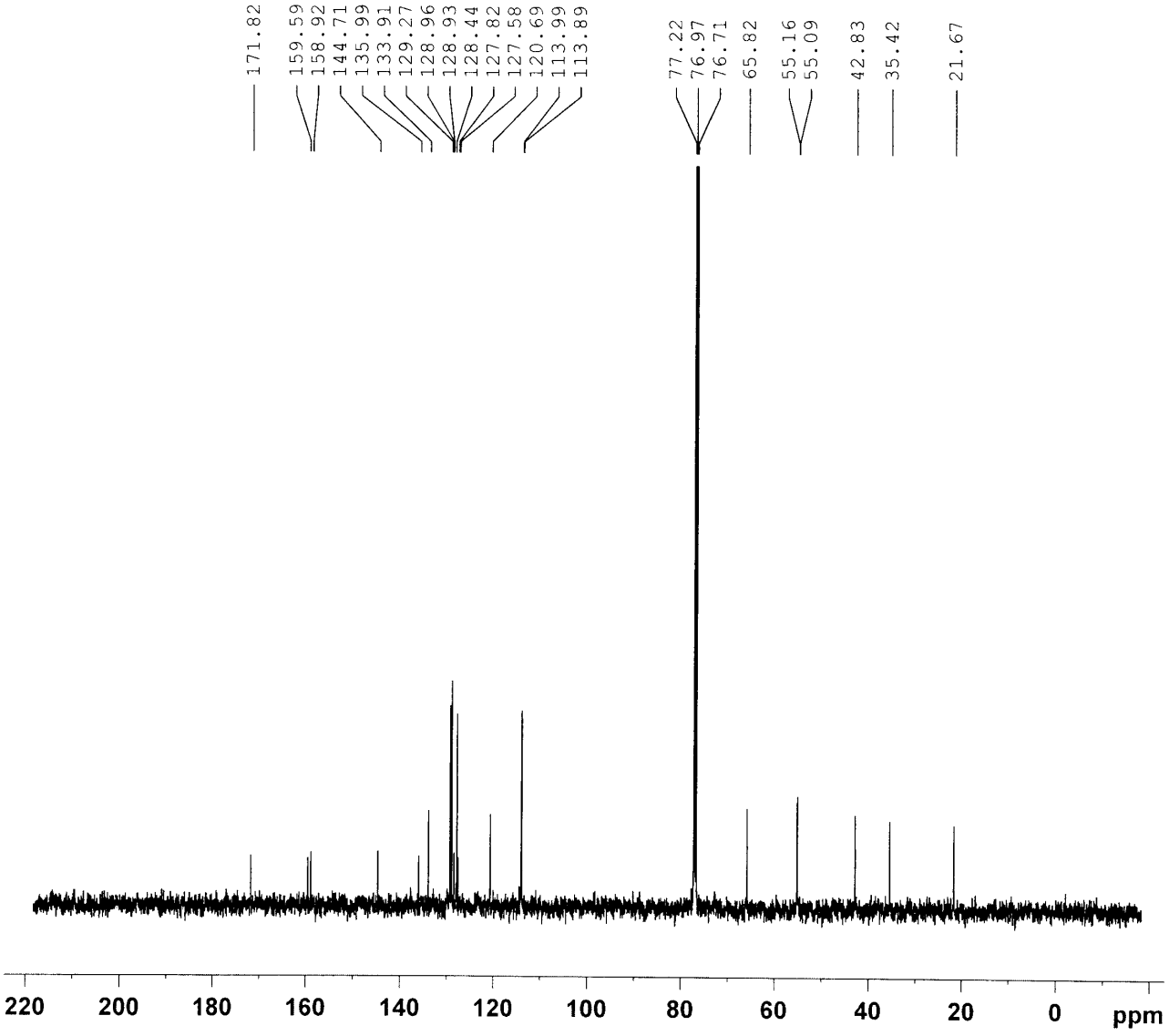
Compound 8a – ^{13}C (125 MHz)



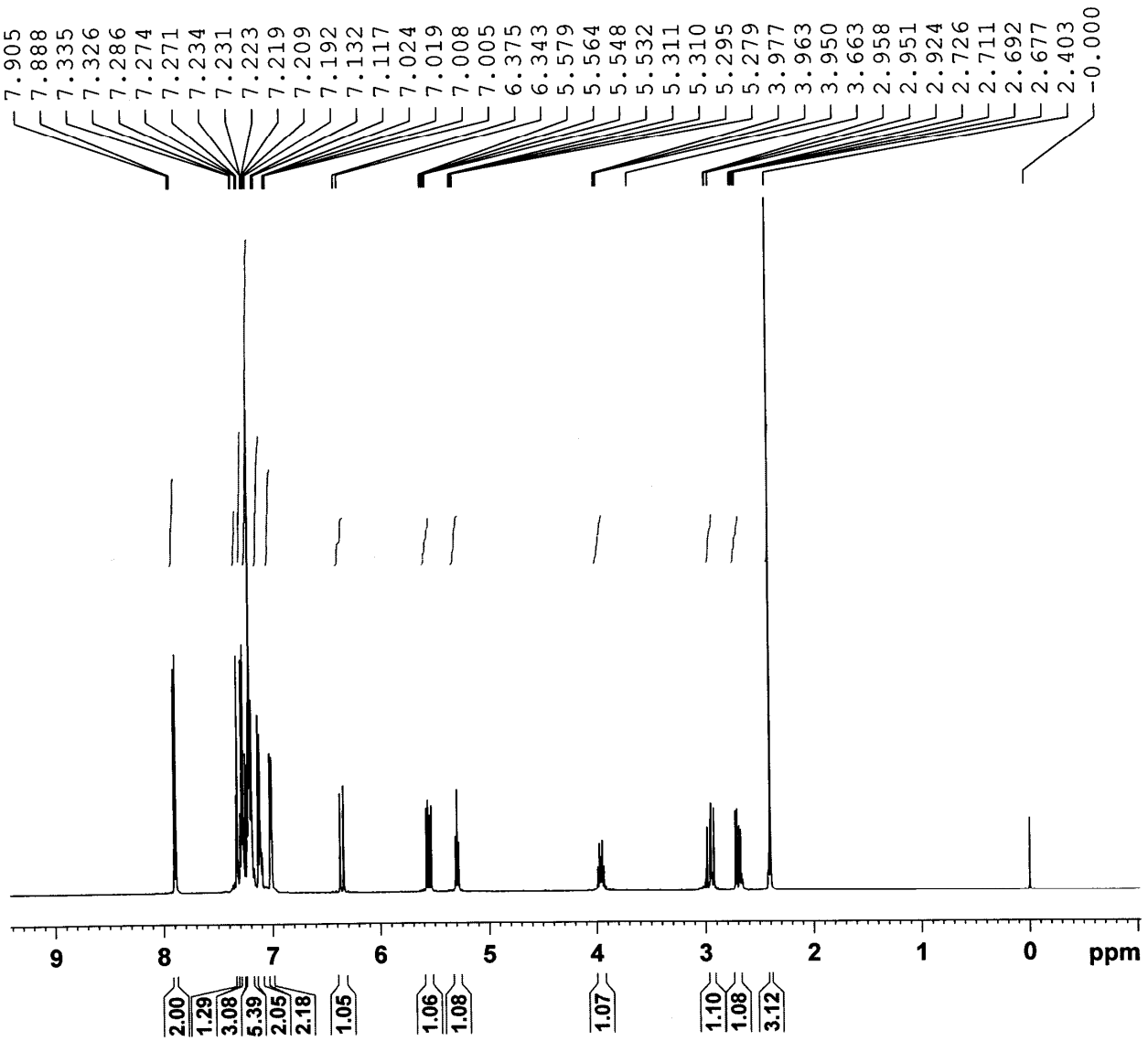
Compound 8b – ^{1}H (500 MHz)



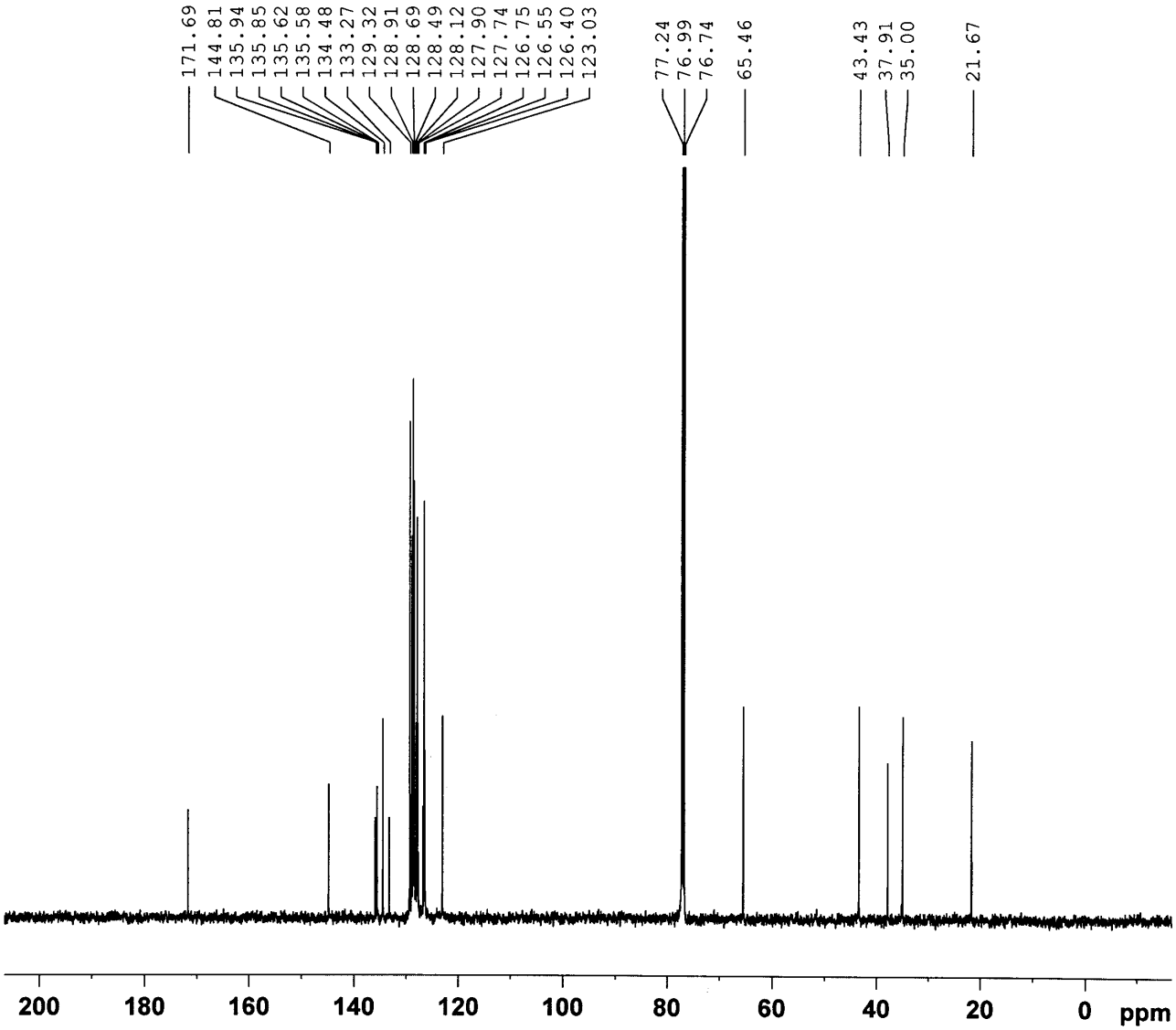
Compound 8b – ^{13}C (125 MHz)



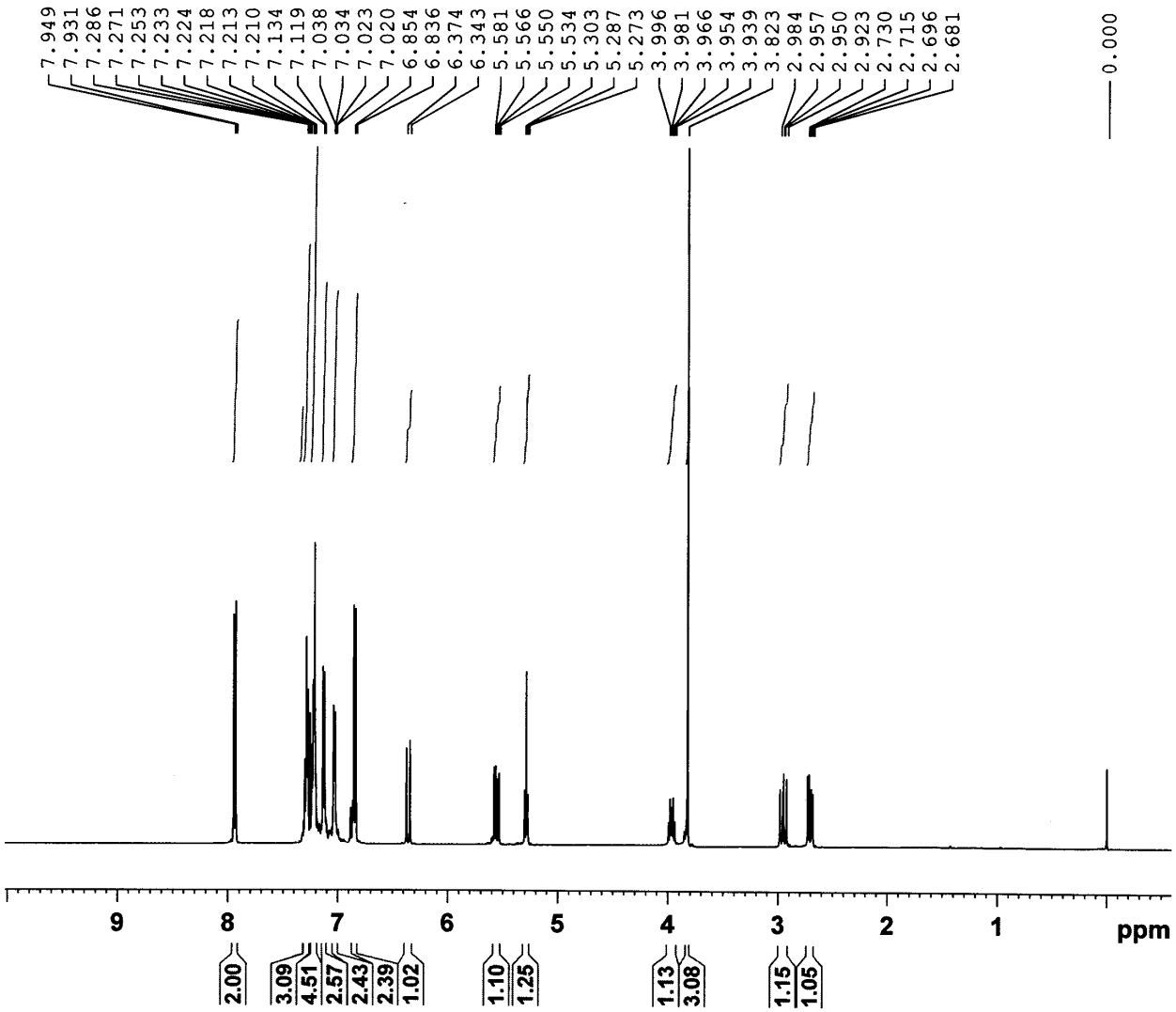
Compound 8c – ^{1}H (500 MHz)



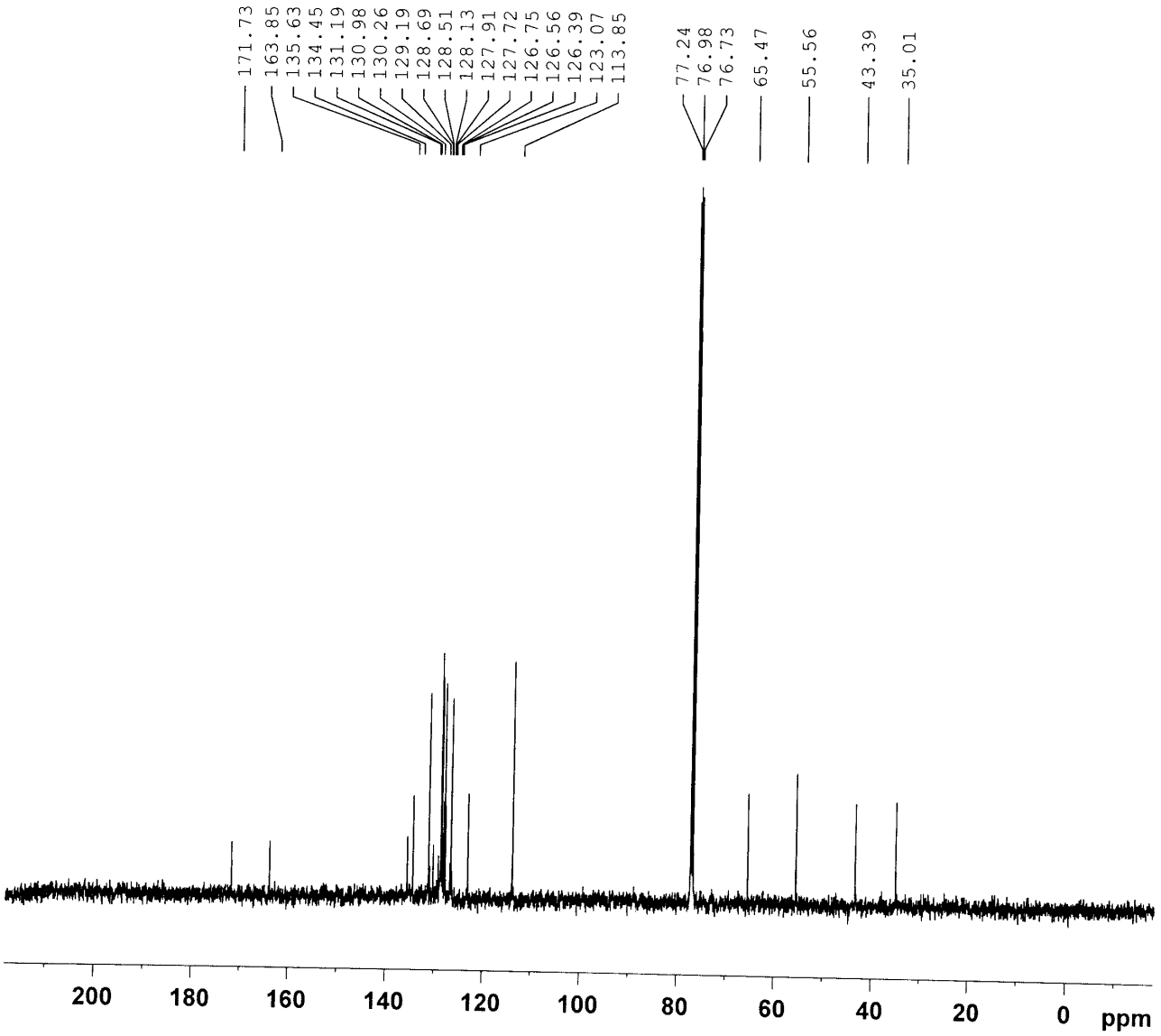
Compound 8c – ^{13}C (125 MHz)



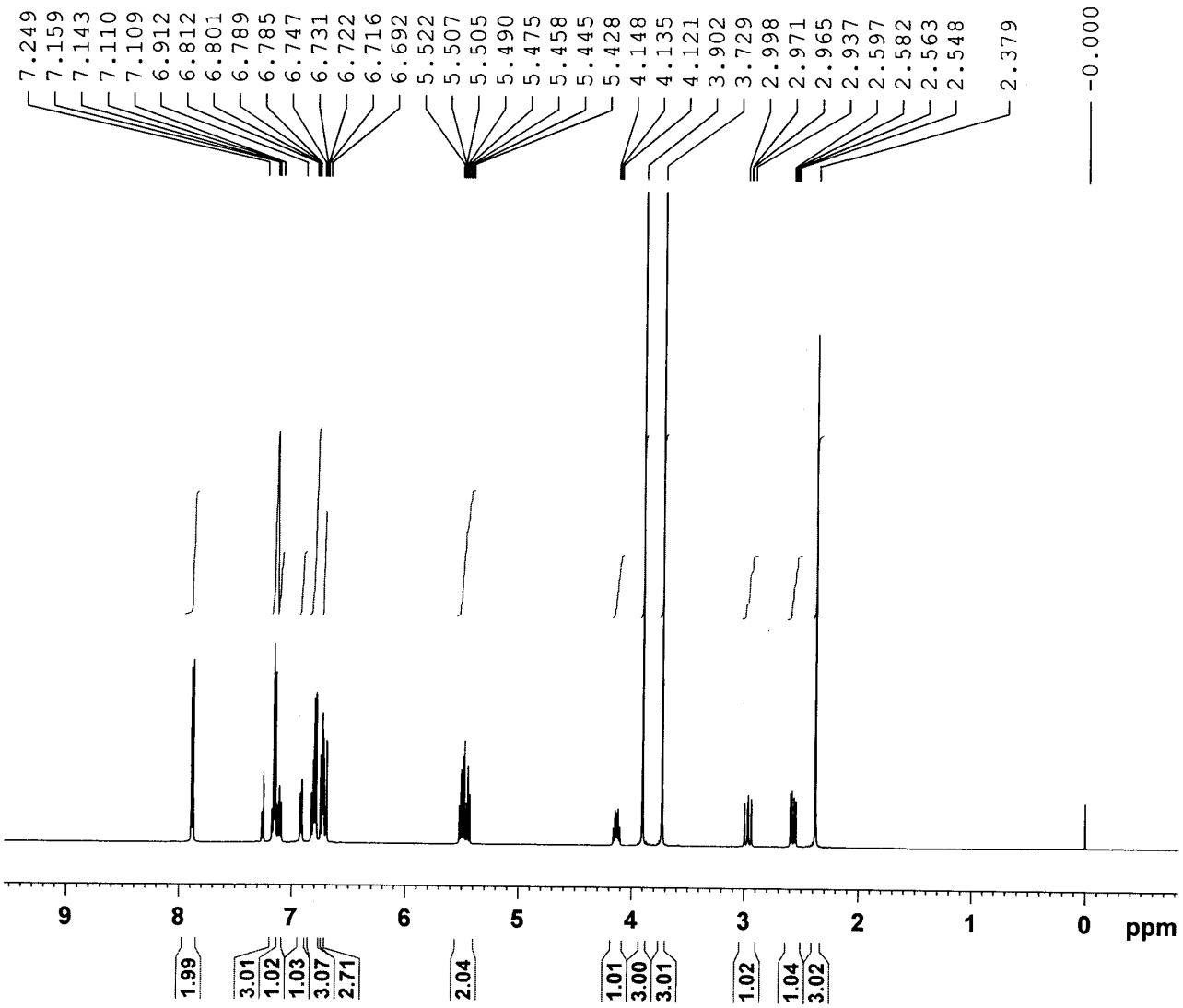
Compound 8d - ^{1}H (500 MHz)



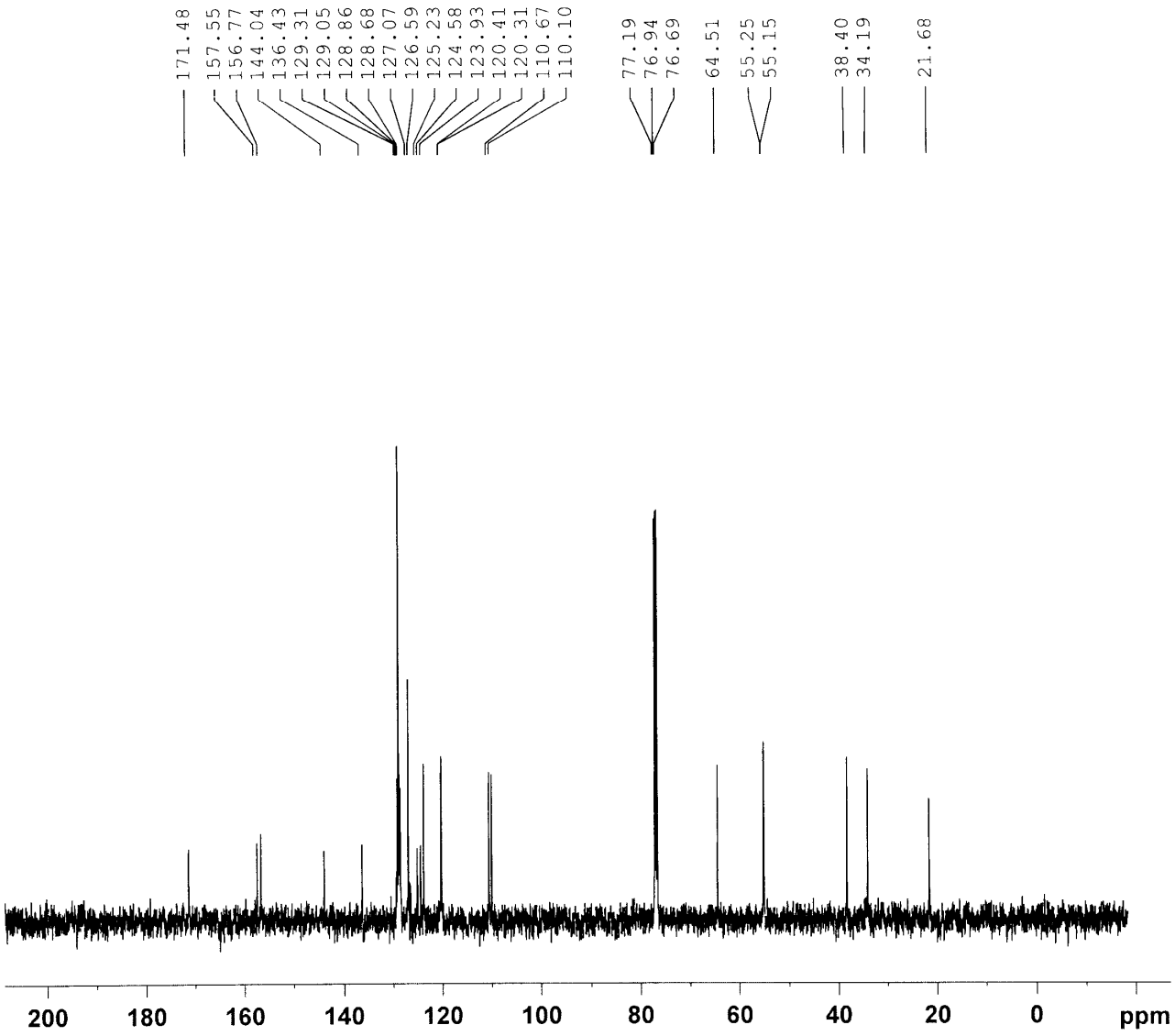
Compound 8d -^{13}C (125 MHz)



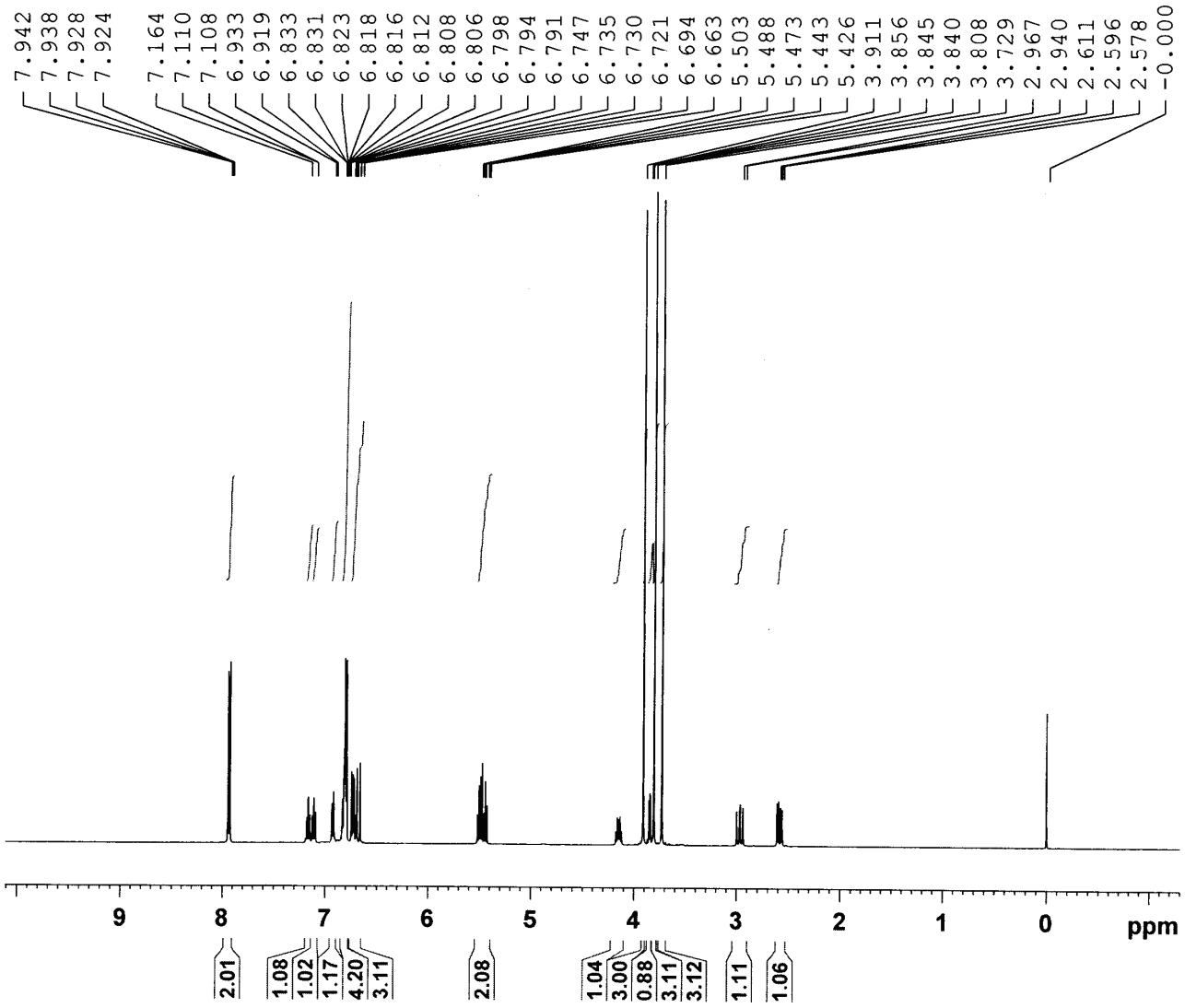
Compound 8e – ^{1}H (500 MHz)



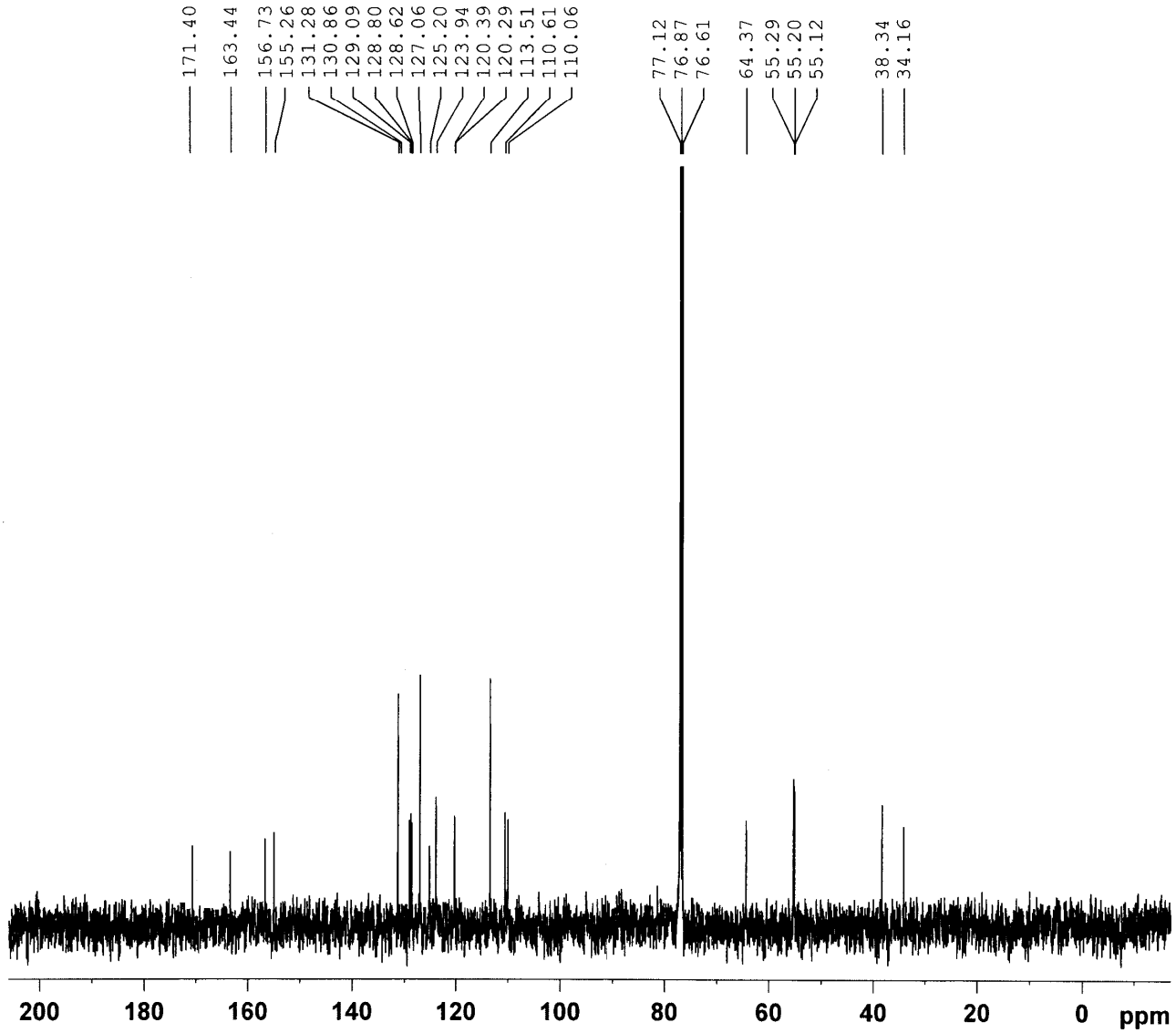
Compound 8e- ^{13}C (125 MHz)



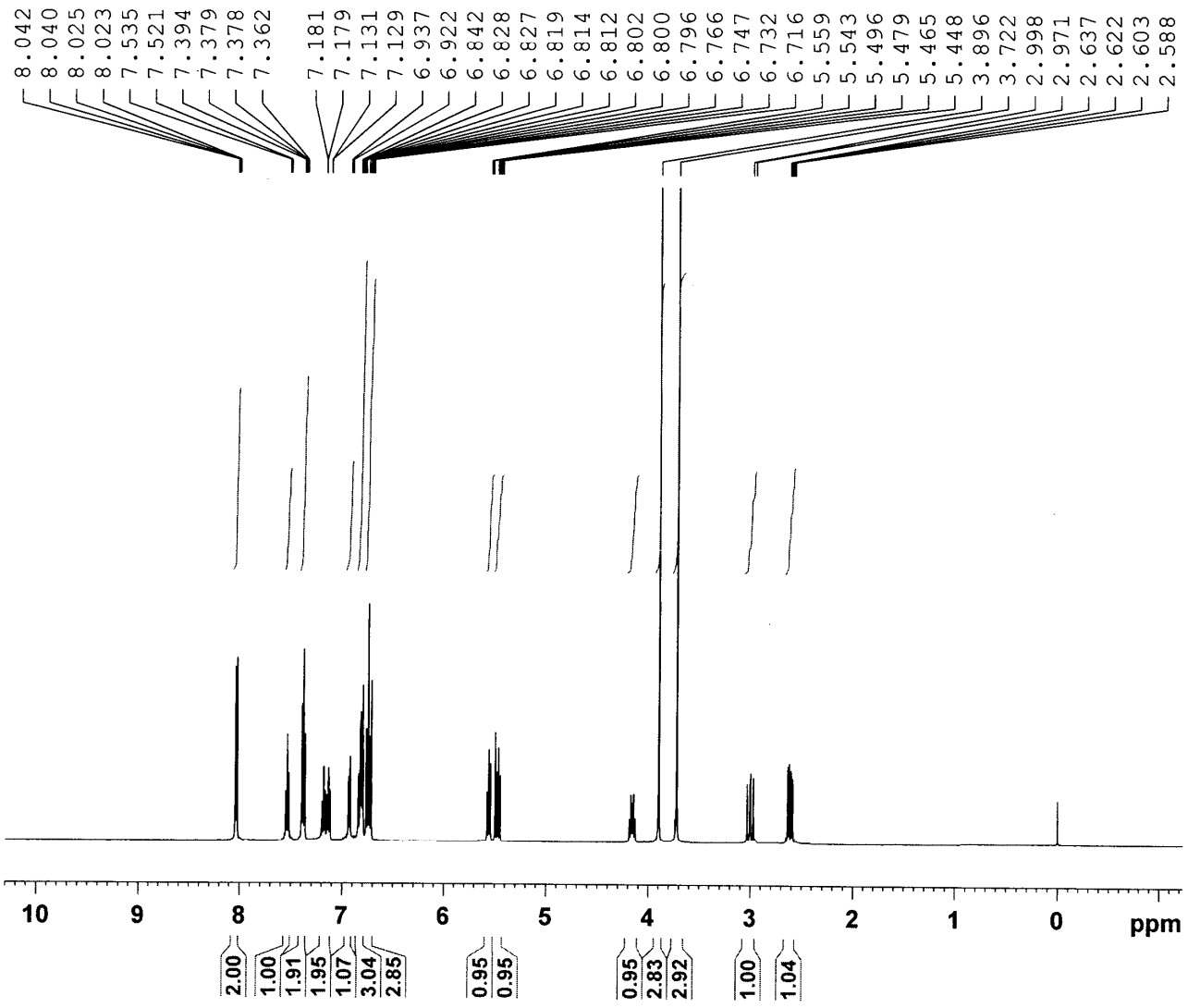
Compound 8f – ^{1}H (500 MHz)



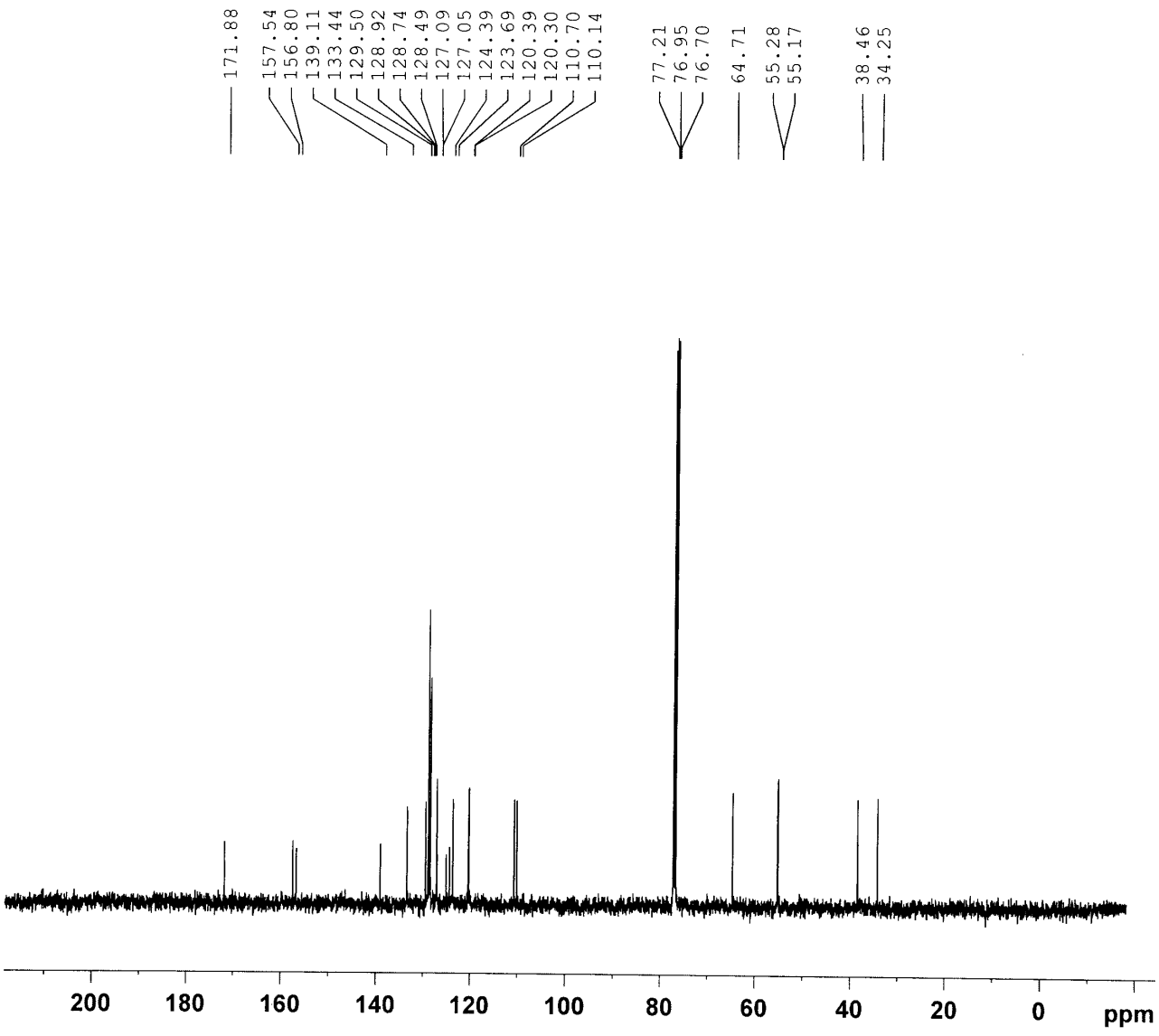
Compound 8f – ^{13}C (125 MHz)



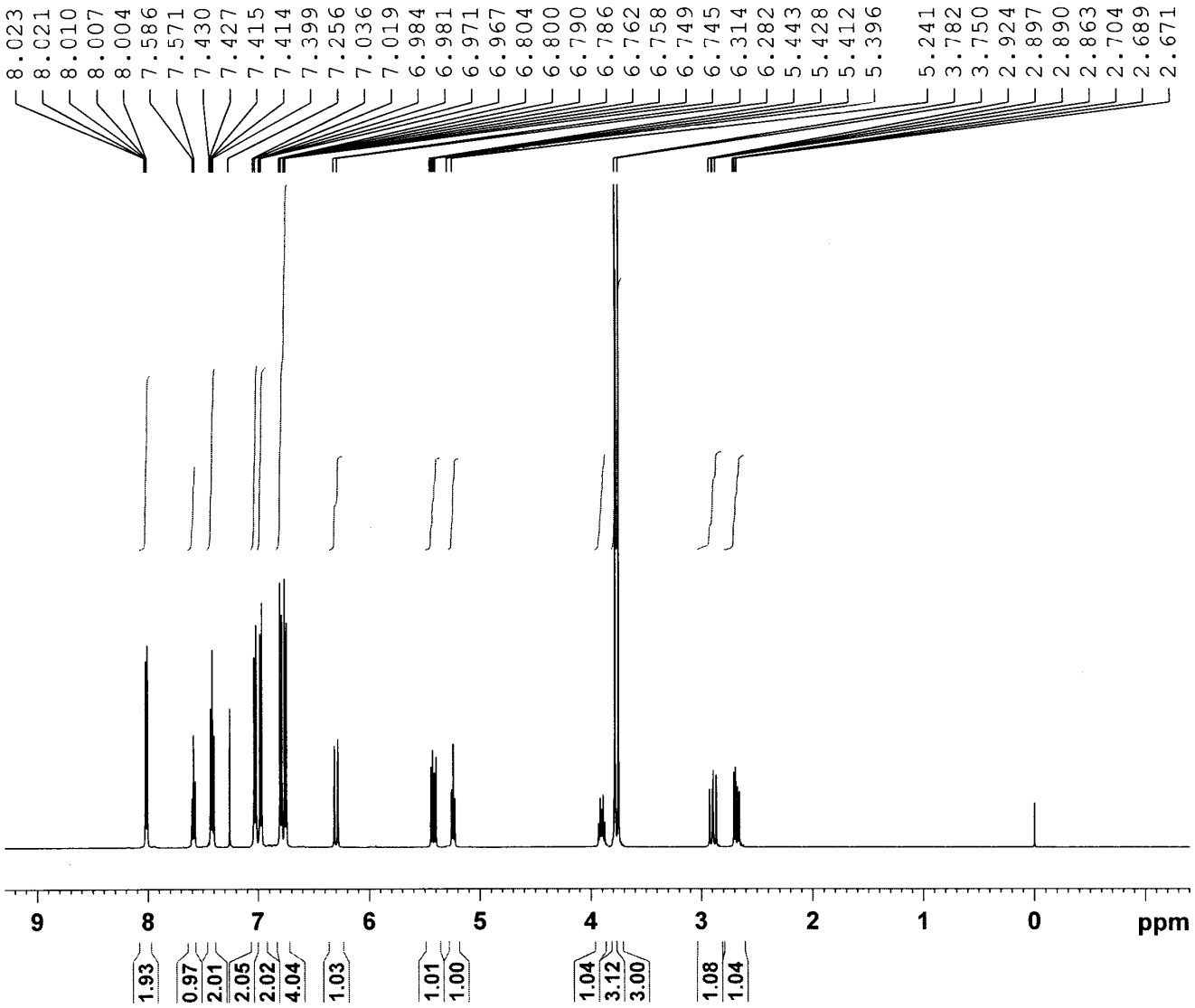
Compound 8g – ^{1}H (500 MHz)



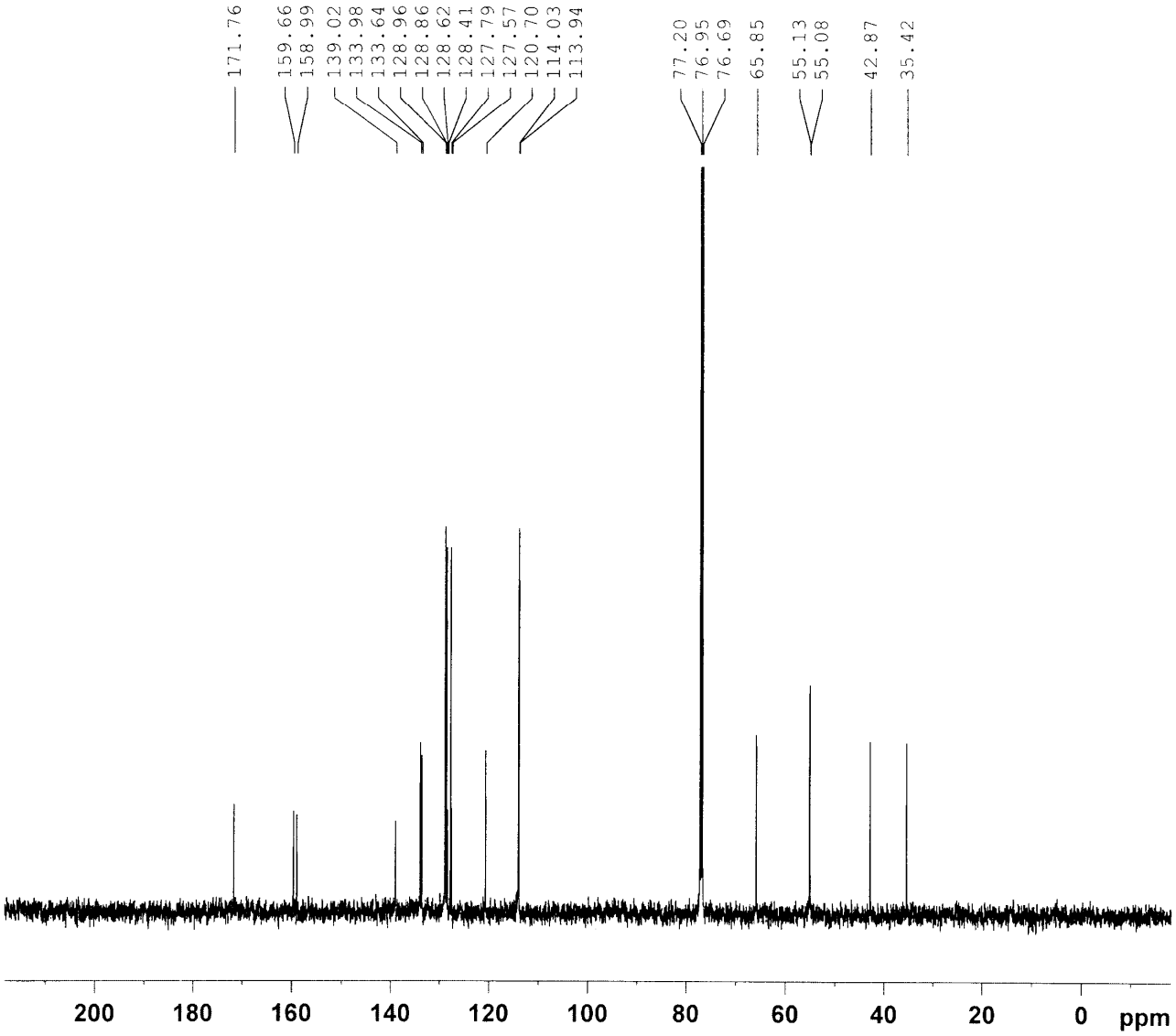
Compound 8g – ^{13}C (125 MHz)



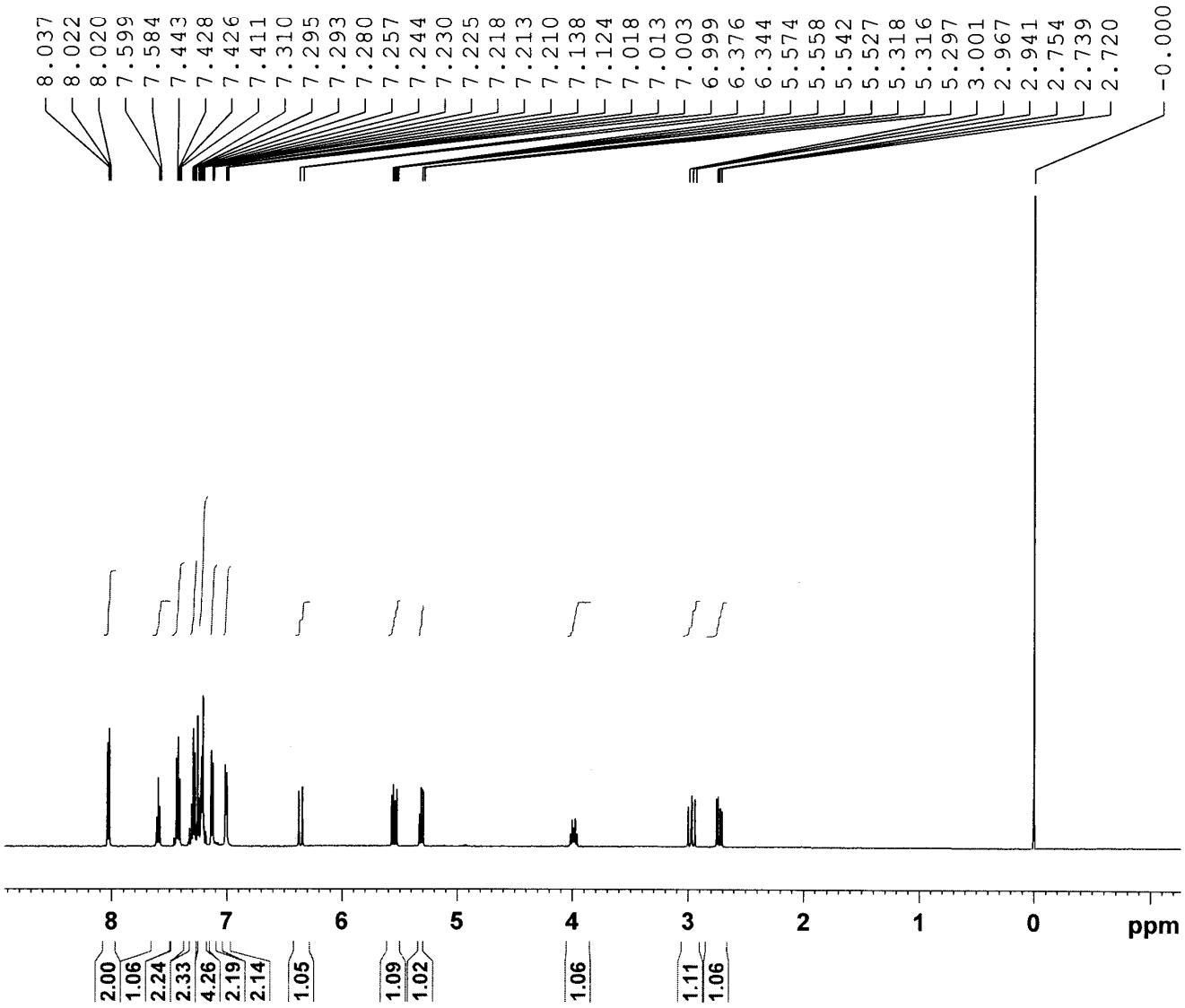
Compound 8h – ^{1}H (500 MHz)



Compound 8h – ^{13}C (125 MHz)



Compound 8i – ^{1}H (500 MHz)



Compound 8i – ^{13}C (125 MHz)