Supplementary Information

Nickel(0)-Catalyzed [2+2+2] Cycloaddition of Diynes and 3,4-Pyridynes: Novel Synthesis of Isoquinoline Derivatives

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Typical procedure

[A] Typical Procedure for [2+2+2] Cycloaddition of Diyne 1a and 3,4-Pyridyne (Table 1, run 5)

An CH_3CN solution of Ni $(cod)_2$ (7.0 mg, 0.025 mmol, 10 mol% to **1a**), PPh_3 (26.2 mg, 0.10 mmol, 40 mol% to **1a**), and CsF (464.8 mg, 3.06 mmol, 3 equiv. to **2a**) was stirred at room temperature for 5 min, and to the mixture was added a solution of 3,4-pyridyne precursor **2a**[2] (346.5 mg, 1.02 mmol, 4 equiv. to **1a**) in CH_3CN (0.5 mL). To the mixture was added a solution of diyne **1a**[1] (53.0 mg, 0.25 mmol) in CH_3CN (2.0 mL) over a period of 3 h by a syringe pump at room temperature. After finishing addition of **1a**, disappearance of the **1a** was observed on TLC. Then the reaction mixture was quenched with 10% aqueous HCl solution, and the mixture was basified by the addition of 10% aqueous NaOH solution. The mixture was extracted with AcOEt, and the organic layer was washed with brine, and dried over Na_2SO_4. After removal of the solvent, the residue was purified by column chromatography (AcOEt) on silica gel to give isoquinoline derivative **3a** (44.9 mg, 0.157 mmol, 63%) along with dimer **4aa**[3] (3.7 mg, 0.009 mmol, 7%).

[B] Typical Procedure for Intramolecular [2+2+2] Cycloaddition of 5a (Table 3, run 1)

An CH_3CN solution of Ni $(cod)_2$ (5.5 mg, 0.02 mmol, 10 mol% to **5a**), PPh_3 (10.5 mg, 0.04 mmol, 20 mol% to **5a**), and CsF (91.1 mg, 0.60 mmol, 3 equiv. to **5a**) was stirred at room temperature for 5 min, and to the mixture was added a solution of substrate **5a** (141.1 mg, 0.20 mmol) in CH_3CN (1.5 mL) over a period of 8 h by a syringe pump at 0 ºC. After finishing addition of **5a**, the reaction mixture was stirred at the same temperature for 12 h. The reaction mixture was quenched with 10% aqueous HCl solution, and the mixture was basified by the addition of 10% aqueous NaOH solution. The mixture was extracted with AcOEt, and the organic layer was washed with brine, and dried over Na_2SO_4. After removal of the solvent, the residue was purified by column chromatography ($AcOEt/Et_2O$ = 1/1) on silica gel to give isoquinoline derivative **6a** (66.2 mg, 0.15 mmol, 75%).

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[1] **1a** [CAS Registry No. 63104-44-9]: R. S. Atkinson and M. J. Grimshire, *J. Chem. Soc., Perkin Trans. 1*, 1986, 1215-1224.

[2] **2a** [CAS Registry No. 204459-37-0]: M. Tsukazaki and V. Snieckus, *Heterocycles*, 1992, **33**, 533-536.

[3] **4aa** [CAS Registry No. 206008-32-4]: T. Sugihara, A. Wakabayashi, Y. Nagai, H. Takao, H. Imagawa and M. Nishizawa, *Chem. Commun.*, 2002, 576-577.

Spectral Data of Substrates

4-(Triethylsilyl)-6-methoxypyridin-3-yl trifluoromethanesulfonate (2b)

IR (neat) 2959, 2879, 2359, 1596, 1536, 1464, 1422, 1249, 1213, 1146 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 8.13 (s, 1 H), 6.82 (s, 1 H), 3.94 (s, 3 H), 1.01-0.84 (m, 15 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 162.2, 146.9, 143.8, 137.8, 118.5, 118.4 (q, J_{C-F} = 318.4 Hz), 54.0, 7.1, 2.8; LRMS (EI) *m/z* 371 (M^+), 342, 314, 209, 172; HRMS (EI) calcd for $C_{13}H_{20}NO_4F_3SSi$ (M^+) 371.0834, found 371.0838.

4-(Triethylsilyl)-2-methoxypyridin-3-yl trifluoromethanesulfonate (2c)

IR (neat) 2958, 2879, 2342, 1595, 1529, 1468, 1416, 1207, 1130 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.09 (d, *J* = 4.6 Hz, 1 H), 6.95 (d, *J* = 4.6 Hz, 1 H), 4.01 (s, 3 H), 0.97-0.93 (m, 15 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 155.3, 144.9, 143.1, 138.5, 122.9, 118.9 (q, J_{C-F} = 319.5 Hz), 53.6, 7.1, 3.0; LRMS (EI) *m/z* 371 (M^+), 342, 273, 238, 209, 138, 122, 87.; HRMS (EI) calcd for $C_{13}H_{20}NO_4F_3SSi$ (M^+) 371.0834, found 371.0828.

2-(Diethylcarbamoyl)-4-(triethylsilyl)pyridin-3-yl trifluoromethanesulfonate (2d)

IR (neat) 2960, 2878, 2250, 1640, 1577, 1520, 1465, 1405, 1316, 1216, 1104 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.51 (d, *J* = 4.8 Hz, 1 H), 7.45 (d, *J* = 4.8 Hz, 1 H), 3.54 (q, *J* = 7.2 Hz, 2 H), 3.29 (q, *J* = 7.2 Hz, 2 H), 1.27 (t, *J* = 7.2 Hz, 3 H), 1.25 (t, *J* = 7.2 Hz, 3 H), 0.94 (m, 15 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.1, 149.1, 146.4, 146.3, 144.2, 131.7, 118.2 (q, J_{C-F} = 318.4 Hz), 43.4, 39.3, 13.4, 12.1, 7.2, 3.2; LRMS (EI) *m/z* 439 (M^+-H), 307, 263, 206, 178, 150, 72; HRMS (EI) calcd for $C_{17}H_{27}N_2O_4F_3SiS$ (M^+) 440.141, found 440.1413.

2-(Methoxycarbonyl)-4-(triethylsilyl)pyridin-3-yl trifluoromethanesulfonate (2e)

IR (neat) 2959, 2879, 2359, 1746, 1609, 1523, 1455, 1299, 1214, 1172, 1137, 1112 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.64 (d, *J* = 4.4 Hz, 1 H), 7.59 (d, *J* = 4.4 Hz, 1 H), 3.99 (s, 3 H), 0.97 (m, 15 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.1, 147.34, 147.27, 144.8, 143.2, 134.0, 118.3 (q, J_{C-F} = 322.9 Hz), 53.2, 7.1, 3.2; LRMS (EI) *m/z* 370 (M^+-Et), 310, 282, 212, 179, 59; HRMS (EI) calcd for $C_{14}H_{20}F_3NO_5SSi$ (M^+-Et) 370.0394, found 370.0392.

2-(2,2,7,7-Tetra(methoxycarbonyl)deca-4,9-diynyl)-4-(triethylsilyl)pyridin-3-yl trifluoromethanesulfonate (5a)

IR (neat) 3303, 2956, 2878, 2360, 2260, 1741, 1580, 1522, 1436, 1405, 1329, 1294, 1217, 1139, 1073 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.37 (d, *J* = 4.5 Hz, 1 H), 7.25 (d, *J* = 4.5 Hz, 1 H), 3.74 (s, 6 H), 3.73 (s, 6 H), 2.91 (d, *J* = 2.2 Hz, 2 H), 2.88 (s, 4 H), 1.99 (t, *J* = 2.2 Hz, 1 H), 0.95 (m, 15 H).; ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.0, 169.1, 150.5, 148.0, 146.8, 142.3, 129.5, 118.1 (q, J_{C-F} = 294.6 Hz), 78.4, 78.2, 77.2, 71.5, 56.5, 56.3, 53.0, 52.9, 33.4, 23.2, 22.7, 22.5, 7.2, 3.3; LRMS (EI) *m/z* 706 (M^++H), 676, 646, 572, 512, 484, 355, 351, 222, 193; HRMS (ESI) calcd for $C_{30}H_{38}F_3NNaO_{11}SSi$ (M^++Na) 728.17846, found 728.17612.

Dimethyl

2-(4-(4-methyl-*N*-((4-(triethylsilyl)-3-(trifluoromethylsulfonyloxy)pyridin-2-yl)methyl)phenylsulfonamido)but-2-ynyl)-2-(prop-2-ynyl)malonate (5b)

IR (neat) 3307, 2957, 2878, 2259, 1741, 1598, 1522, 1494, 1402, 1349, 1216, 1162, 1120 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.44 (d, *J* = 4.4 Hz, 1 H), 7.67 (d, *J* = 8.4 Hz, 2 H), 7.32 (d, *J* = 4.4 Hz, 1 H), 7.27 (d, *J* = 8.4 Hz, 2 H), 4.64 (s, 2 H), 4.42 (s, 2 H), 3.70 (s, 6 H), 2.81 (s, 2 H), 2.74 (d, *J* = 2.8 Hz, 2 H), 2.43 (s, 3 H), 1.99 (t, *J* = 2.8 Hz, 1 H), 0.90-0.88 (m, 15 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.9, 150.2, 147.7, 146.0, 143.3, 142.7, 136.5, 130.5, 129.4, 127.3, 118.4 (q, J_{C-F} = 318.3 Hz), 79.8, 78.2, 77.2, 71.5, 56.2, 53.0, 45.4, 38.6, 22.7, 22.5, 21.5, 7.2, 3.3; LRMS (FAB) *m/z* 745 (M^++H), 715, 613, 589, 537, 460, 307, 289, 219, 192, 154, 136; HRMS (ESI) calcd for $C_{32}H_{40}F_3N_2O_9S_2Si$ (M^++H) 745.1897, found 745.1907.

2-(2,2-Di(methoxycarbonyl)-6-(*N*-(prop-2-ynyl)-*N*-tosylamino)hex-4-ynyl)-4-(triethylsilyl)pyridin-3-yl trifluoromethanesulfonate (5c)

IR (neat) 3306, 2956, 2878, 2360, 2260, 1740, 1597, 1521, 1495, 1405, 1354, 1219, 1165, 1138 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.38 (d, *J* = 4.4 Hz, 1 H), 7.67 (d, *J* = 8.4 Hz, 2 H), 7.27 (d, *J* = 8.4 Hz, 2 H), 7.26 (d, *J* = 4.4 Hz, 1 H), 4.10 (d, *J* = 2.4 Hz, 2 H), 4.06 (s, 2 H), 3.71 (s, 6 H), 3.70 (s, 2 H), 2.82 (s, 2 H), 2.40 (s, 3 H), 2.07 (t, *J* = 2.4 Hz, 1 H), 0.94 (m, 15 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 169.9, 150.2, 147.8, 146.7, 143.7, 142.4, 135.1, 129.6, 129.4, 127.7, 118.4 (q, J_{C-F} = 318.4 Hz) 80.8, 76.0, 75.6, 73.7, 56.0, 52.8, 36.3, 35.8, 33.5, 23.2, 21.4, 7.1, 3.2; LRMS (ESI) *m/z* 767 (M^++Na), 709, 414, 360, 282; HRMS (ESI) calcd for $C_{32}H_{39}F_3N_2NaO_9S_2Si$ (M^++Na) 767.17160, found 767.17289.

2-((4-Methyl-*N*-(4-(4-methyl-*N*-(prop-2-ynyl)phenylsulfonamido)but-2-ynyl)phenylsulfonamido)methyl)-4-(triethylsilyl)pyridin-3-yl trifluoromethanesulfonate (5d)

IR (neat) 3306, 2959, 2878, 2257, 1597, 1495, 1403, 1353, 1221, 1164, 1138 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.44 (d, *J* = 4.4 Hz, 1 H), 7.65 (d, *J* = 8.3 Hz, 2 H), 7.63 (d, *J* = 8.3 Hz, 2 H), 7.33 (d, *J* = 4.4 Hz, 1 H), 7.27-7.25 (m, 4 H), 4.58 (s, 2 H), 4.31 (s, 2 H), 3.94 (s, 2 H), 3.92 (d, *J* = 2.4 Hz, 2 H), 2.43 (s, 3 H), 2.39 (s, 3 H), 2.06 (t, *J* = 2.4 Hz, 1 H), 0.96-0.88 (m, 15 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 149.9, 147.7, 145.9, 144.0, 143.6, 142.9, 136.2, 135.0, 130.7, 129.5, 129.4, 127.7, 127.3, 118.4 (q, J_{C-F} = 318.4 Hz), 79.0, 78.0, 75.9, 73.9, 45.6, 38.4, 36.2, 36.0, 21.5, 21.4, 7.2, 3.3; LRMS (FAB) *m/z* 784 (M^++H), 652, 630, 525, 341, 289, 222, 154, 136; HRMS (FAB) calcd for $C_{34}H_{41}F_3N_3O_7S_3Si$ (M^++H) 784.1891, found 784.1821.

2-((5,5-Di(methoxycarbonyl)octa-2,7-diynyloxy)methyl)-4-(triethylsilyl)pyridin-3-yl trifluoromethanesulfonate (5e)

IR (neat) 3305, 2957, 2878, 2386, 2288, 1743, 1580, 1404, 1217, 1139, 1072 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.60 (d, *J* = 4.4 Hz, 1 H), 7.38 (d, *J* = 4.4 Hz, 1 H), 4.79 (s, 2 H), 4.27 (t, *J* = 1.2 Hz, 2 H), 3.75 (s, 6 H), 3.04 (t, *J* = 1.2 Hz, 2 H), 2.98 (d, *J* = 2.8 Hz, 2 H), 2.02 (t, *J* = 2.8 Hz, 1 H), 0.94 (m, 15 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.1, 150.5, 147.9, 146.9, 143.0, 131.0, 118.4 (q, J_{C-F} = 320.9 Hz), 81.4, 78.7, 78.3, 71.7, 67.6, 58.7, 56.5, 53.1, 22.9, 22.7, 7.2, 3.3; LRMS (FAB) *m/z* 592 (M^++H), 534, 460, 370, 307, 289, 222, 154, 136, 107; HRMS (FAB) calcd for $C_{25}H_{33}F_3NO_8SSi$ (M^++H) 592.1729, found 592.1639.

2-(2,2-Di(methoxycarbonyl)-6-(prop-2-ynyloxy)hex-4-ynyl)-4-(triethylsilyl)pyridin-3-yl trifluoromethanesulfonate (5f)

IR (neat) 3303, 2956, 2878, 2360, 2228, 1741, 1581, 1523, 1405, 1217, 1139, 1078 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.38 (d, *J* = 4.8 Hz, 1 H), 7.26 (d, *J* = 4.8 Hz, 1 H), 4.19 (s, 2 H), 4.18 (d, *J* = 2.8 Hz, 2 H), 3.78 (s, 2 H), 3.76 (s, 6 H), 2.94 (s, 2 H), 2.44 (t, *J* = 2.8 Hz, 1 H), 0.94 (m, 15 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.1, 150.3, 148.0, 146.8, 142.4, 129.6, 1118.6 (q, J_{C-F} = 316.0 Hz), 81.9, 78.9, 78.4, 74.7, 56.6, 56.2, 56.1, 53.0, 33.6, 23.3, 7.2, 3.3; LRMS (FAB) *m/z* 592 (M^++H), 534, 460, 307, 289, 154, 136, 107; HRMS (FAB) calcd for $C_{25}H_{33}F_3NO_8SSi$ (M^++H) 592.1729, found 592.1627.

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[4] **1b** [CAS Registry No. 2396-63-6] and **1d** [CAS Registry No. 6921-27-3] were obtained from Aldrich.
[5] **1c** [CAS Registry No. 18773-54-1]: B. M. Trost and M. T. Rudd, *J. Am. Chem. Soc.*, 2005, **127**, 4763-4776.

Spectral Data of Isoquinoline Derivatives

6*H*-Cyclopenta[*g*]isoquinoline-7,7(8*H*)-dicarboxylate (Table 1, run 5, 3aa)

Mp 114-115 °C; IR (film) 2960, 2928, 2901, 2850, 1731, 1638, 1592, 1437, 1275, 1201, 1163, 1053 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 9.15 (s, 1 H), 8.44 (d, *J* = 5.6 Hz, 1 H), 7.77 (s, 1 H), 7.63 (s, 1 H), 7.57 (d, *J* = 5.6 Hz, 1 H), 3.77 (s, 6 H), 3.76-3.75 (m, 4 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.5, 152.0, 143.8, 142.7, 140.4, 135.6, 128.5, 122.3, 121.2, 120.3, 60.6, 53.1, 40.4, 40.1; LRMS (EI) *m/z* 285 (M^+), 225, 166, 59; HRMS (EI) calcd for $C_{16}H_{15}NO_4$ (M^+) 285.1003, found 285.1001.

7,8-Dihydro-6*H*-cyclopenta[*g*]isoquinoline (Table 2, run 1, 3ba)

Mp 110 °C; IR (film) 2954, 2842, 1632, 1593, 1456 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 9.13 (s, 1 H), 8.41 (d, *J* = 6.0 Hz, 1 H), 7.75 (s, 1 H), 7.62 (s, 1 H), 7.55 (d, *J* = 6.0 Hz, 1 H), 3.08 (t, *J* = 7.6 Hz, 4 H), 2.16 (quin, *J* = 7.6 Hz, 2 H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 150.4, 150.1, 145.9, 139.8, 136.0, 128.0, 122.2, 121.2, 121.0, 120.9, 33.0, 32.6, 25.9; LRMS (EI) *m/z* 169 (M^+), 168, 115; HRMS (EI) calcd for $C_{12}H_{11}N$ (M^+) 169.0895, found 169.0891.

2,3-Dihydro-2-tosyl-1*H*-pyrrolo[3,4-*g*]isoquinoline (Table 2, run 2, 3ca)

Mp 203 °C; IR (film) 2927, 2856, 1644, 1598, 1456, 1337, 1215, 1162 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 9.19 (s, 1 H), 8.49 (d, *J* = 5.9 Hz, 1 H), 7.82 (s, 1 H), 7.78 (d, *J* = 7.9 Hz, 2 H), 7.62 (s, 1 H), 7.59 (d, *J* = 5.9 Hz, 1 H), 7.33 (d, *J* = 7.9 Hz, 2 H), 4.77 (s, 4 H), 2.40 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.3, 144.0, 143.2, 139.7, 136.4, 135.5, 133.2, 129.93, 129.85, 127.7, 127.6, 121.2, 120.0, 53.1, 53.0, 21.5; LRMS (EI) *m/z* 324 (M^+), 169, 155, 91, 65; HRMS (EI) calcd for $C_{18}H_{16}N_2O_2S$ (M^+) 324.0922, found 324.0932.

1,3-Dihydrofuro[3,4-*g*]isoquinoline (Table 2, run 3, 3da)

Mp 95 °C; IR (film) 2929, 2854, 1644, 1598, 1454, 1242, 1051 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 9.23 (s, 1 H), 8.50 (d, *J* = 6.0 Hz, 1 H), 7.81 (s, 1 H), 7.63 (s, 1 H), 7.56 (d, *J* = 6.0 Hz, 1 H), 5.25 (s, 4 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.4, 143.1, 142.9, 139.8, 135.8, 128.5, 120.7, 119.2, 118.0, 72.74, 72.67; LRMS (EI) *m/z* 171 (M^+), 141, 115, 57; HRMS (EI) calcd for $C_{11}H_9NO$ (M^+) 171.0676, found 171.0684.

Dimethyl 3-methoxy-6*H*-cyclopenta[*g*]isoquinoline-7,7(8*H*)-dicarboxylate (Table 2, run 4, 3ab)

Mp 128-129 °C; IR (film) 2954, 2925, 2851, 1733, 1644, 1604, 1441, 1273, 1203, 1158, 1072 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 8.85 (s, 1 H), 7.68 (s, 1 H), 7.50 (s, 1 H), 6.93 (s, 1 H), 4.01 (s, 3 H), 3.76 (s, 6 H), 3.69 (s, 4 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.7, 161.2, 150.0, 144.1, 139.2, 137.5, 125.1, 122.1, 120.1, 101.0, 60.7, 54.2, 53.1, 40.3, 39.8; LRMS (EI) *m/z* 315 (M^+), 284, 255, 196, 59; HRMS (EI) calcd for $C_{17}H_{17}NO_5$ (M^+) 315.1106, found 315.1108.

Dimethyl 1-methoxy-6*H*-cyclopenta[*g*]isoquinoline-7,7(8*H*)-dicarboxylate (Table 2, run 5, 3ac)

Mp 98 °C; IR (film) 2954, 2900, 2844, 1731, 1639, 1600, 1455, 1256, 1182, 1134 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 8.06 (s, 1 H), 7.92 (d, *J* = 5.9 Hz, 1 H), 7.55 (s, 1 H), 7.14 (d, *J* = 5.9 Hz, 1 H), 4.11 (s, 3 H), 3.76 (s, 6

H), 3.73 (s, 4 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.7, 160.7, 143.6, 139.6, 139.1, 137.6, 121.0, 119.2, 119.0, 114.9, 60.7, 53.6, 53.1, 40.3, 40.2; LRMS (EI) *m/z* 315 (M^+), 284, 255, 196, 59; HRMS (EI) calcd for $C_{17}H_{17}NO_5$ (M^+) 315.1106, found 315.1096.

Dimethyl 1-(diethylcarbamoyl)-6*H*-cyclopenta[*g*]isoquinoline-7,7(8*H*)-dicarboxylate (Table 2, run 6, 3ad)

Mp 100 °C; IR (film) 2975, 2876, 1731, 1633, 1590, 1436, 1348, 1281, 1201, 1141, 1072 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.42 (d, *J* = 5.6 Hz, 1 H), 7.78 (s, 1 H), 7.59 (s, 1 H), 7.57 (d, *J* = 5.6 Hz, 1 H), 3.76 (s, 6 H), 3.74 (s, 4 H), 3.72 (q, *J* = 7.2 Hz, 2 H), 3.15 (q, *J* = 7.2 Hz, 2 H), 1.39 (t, *J* = 7.2 Hz, 3 H), 1.07 (t, *J* = 7.2 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.4, 168.1, 155.4, 144.3, 141.29, 141.26, 136.4, 125.1, 121.6, 120.9, 120.6, 60.6, 53.1, 43.0, 40.3, 40.2, 39.4, 14.2, 13.1; LRMS (EI) *m/z* 384 (M^+), 369, 325, 313, 285, 224, 166, 72; HRMS (EI) calcd for $C_{21}H_{24}N_2O_5$ (M^+) 384.1684, found 384.1685.

Tetramethyl indeno[6,5,4-*de*]quinoline-2,2,9,9(1*H*,3*H*,8*H*,10*H*)-tetracarboxylate (Table 3, run 1, 6a)

Mp 149 °C; IR (film) 2955, 2924, 2851, 1734, 1633, 1594, 1578, 1435, 1200, 1069 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.35 (d, *J* = 5.5 Hz, 1 H), 7.50 (s, 1 H), 7.45 (d, *J* = 5.5 Hz, 1 H), 3.77 (s, 6 H), 3.76 (s, 2 H), 3.75 (s, 2 H), 3.72 (s, 2 H), 3.69 (s, 6 H), 3.56 (s, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.6, 170.8, 155.4, 143.7, 142.1, 137.2, 135.8, 128.6, 123.2, 119.5, 119.3, 60.2, 54.4, 53.15, 53.07, 40.6, 38.8, 38.7, 32.9; LRMS (EI) *m/z* 441 (M^+), 382, 322, 262, 204, 59; HRMS (EI) calcd for $C_{23}H_{23}O_8N$ (M^+) 441.1423, found 441.1412.

Dimethyl 2-tosyl-2,3,8,10-tetrahydroindeno[6,5,4-*de*][1,7]naphthyridine-9,9(1*H*)-dicarboxylate (Table 3, run 2, 6b)

Mp 163-164 °C; IR (film) 2955, 2925, 2853, 1734, 1634, 1596, 1436, 1344, 1258, 1215, 1162, 1055 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.27 (d, *J* = 6.0 Hz, 1 H), 7.50 (d, *J* = 8.4 Hz, 2 H), 7.42 (s, 1 H), 7.33 (d, *J* = 6.0 Hz, 1 H), 6.96 (d, *J* = 8.4 Hz, 2 H), 4.74 (s, 2 H), 4.67 (s, 2 H), 3.81 (s, 6 H), 3.72 (s, 2 H), 3.66 (s, 2 H), 2.24 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.4, 152.6, 143.9, 143.5, 141.9, 135.4, 133.3, 129.1, 128.2, 127.4, 126.0, 122.1, 119.6, 119.2, 60.3, 59.1, 53.3, 51.1, 46.3, 40.4, 38.2, 21.3; LRMS (EI) *m/z* 324 (M^+-Ts-H), 207, 124, 91; HRMS (EI) calcd for $C_{18}H_{16}O_4N_2$ (M^+-Ts-H) 324.1110, found 342.1098.

Dimethyl 9-tosyl-3,8,9,10-tetrahydroisoindolo[6,5,4-*de*]quinoline-2,2(1*H*)-dicarboxylate (Table 3, run 3, 6c)

Mp 189 °C; IR (film) 2956, 2925, 2854, 1736, 1637, 1598, 1438, 1348, 1264, 1215, 1162, 1061 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.38 (d, *J* = 6.0 Hz, 1 H), 7.82 (d, *J* = 8.0 Hz, 2 H), 7.54 (d, *J* = 6.0 Hz, 1 H), 7.52 (s, 1 H), 7.35 (d, *J* = 8.4 Hz, 2 H), 4.78 (s, 2 H), 4.74 (s, 2 H), 3.82 (s, 2 H), 3.69 (s, 6 H), 3.48 (s, 2 H), 2.41 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.2, 155.5, 144.1, 141.0, 140.9, 136.1, 134.1, 133.3, 130.0, 128.6, 127.7, 123.0, 120.0, 118.5, 54.1, 53.5, 53.3, 52.1, 38.0, 32.6, 21.6; LRMS (EI) *m/z* 480 (M^+), 421, 363, 325, 155, 91; HRMS (EI) calcd for $C_{25}H_{24}O_6N_2S$ (M^+) 480.1354, found 483.1360.

2,9-Ditosyl-1,2,3,8,9,10-hexahydroisoindolo[6,5,4-*de*][1,7]naphthyridine (Table 3, run 4, 6d)

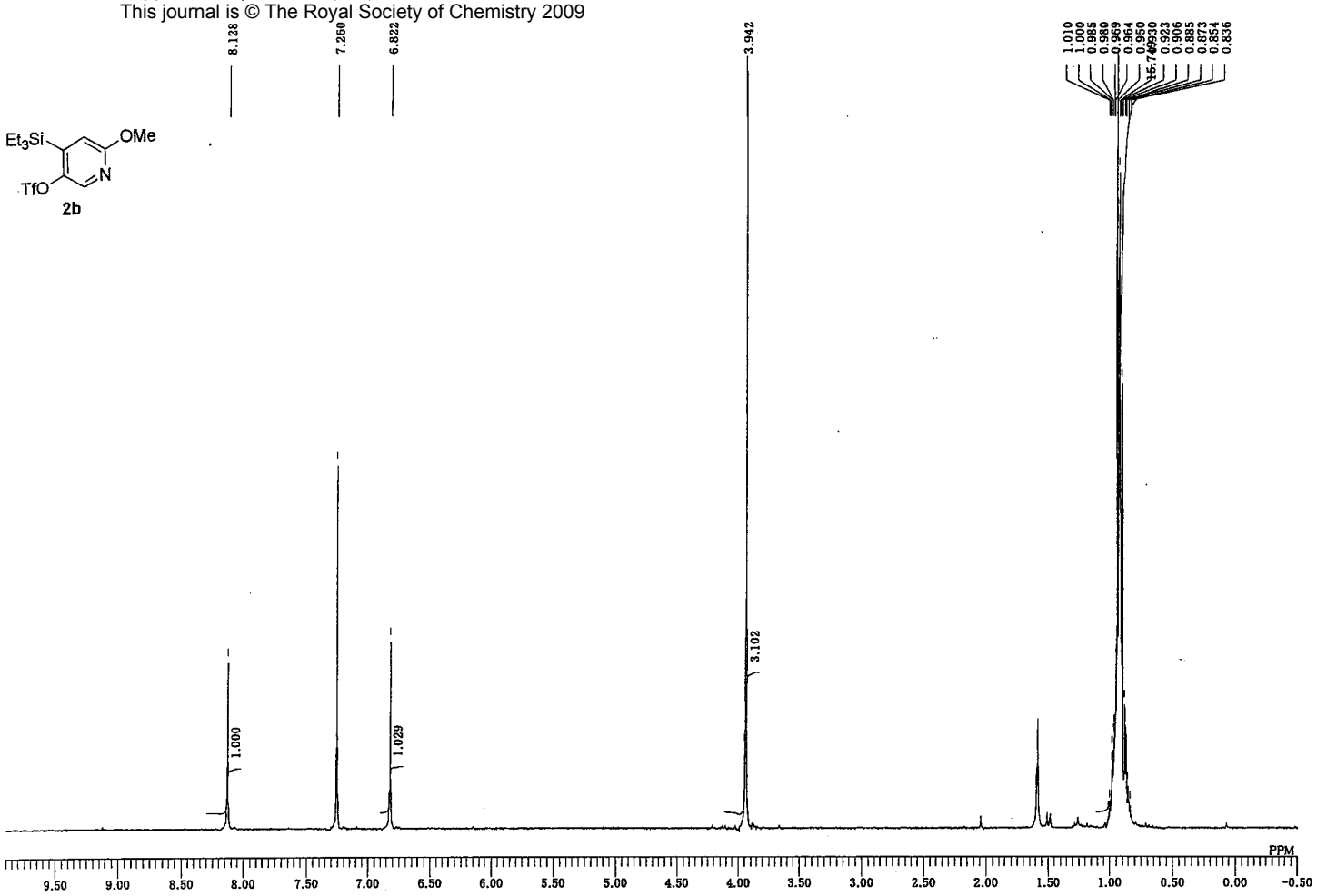
Mp 190 °C (dec.); IR (film) 2926, 2855, 1637, 1596, 1450, 1342, 1216, 1164 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.35 (d, *J* = 5.2 Hz, 1 H), 7.84 (d, *J* = 8.4 Hz, 2 H), 7.57 (d, *J* = 8.4 Hz, 2 H), 7.44 (s, 1 H), 7.40 (d, *J* = 5.2 Hz, 1 H), 7.38 (d, *J* = 8.4 Hz, 2 H), 7.07 (d, *J* = 8.4 Hz, 2 H), 4.73 (s, 2 H), 4.68 (s, 2 H), 4.66 (s, 2 H), 4.49 (s, 2 H), 2.42 (s, 3 H), 2.30 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.1, 144.3, 144.0, 142.6, 139.7, 135.4, 133.0, 132.8, 131.4, 130.1, 129.4, 127.8, 127.6, 125.8, 122.0, 119.4, 118.5, 53.2, 51.6, 50.9, 45.8, 21.6, 21.4; LRMS (EI) *m/z* 363 (M^+-Ts-H), 324, 207, 124, 91; HRMS (EI) calcd for $C_{20}H_{17}O_2N_3S$ (M^+-Ts-H) 363.1041, found 363.1037.

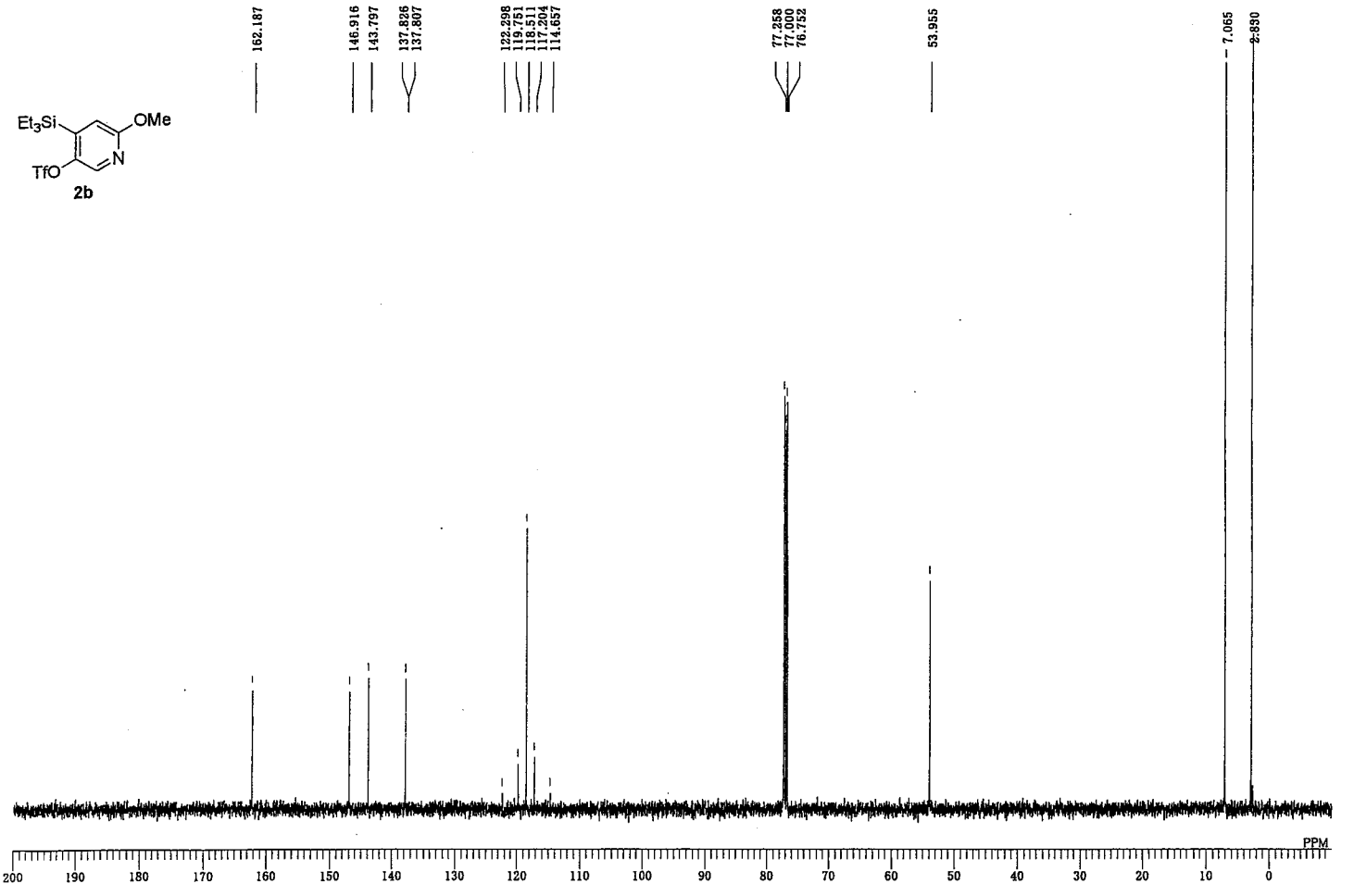
Dimethyl 8,10-dihydro-1*H*-cyclopenta[*g*]pyrano[3,4,5-*ij*]isoquinoline-9,9(3*H*)-dicarboxylate (Table 3, run 5, 6e)

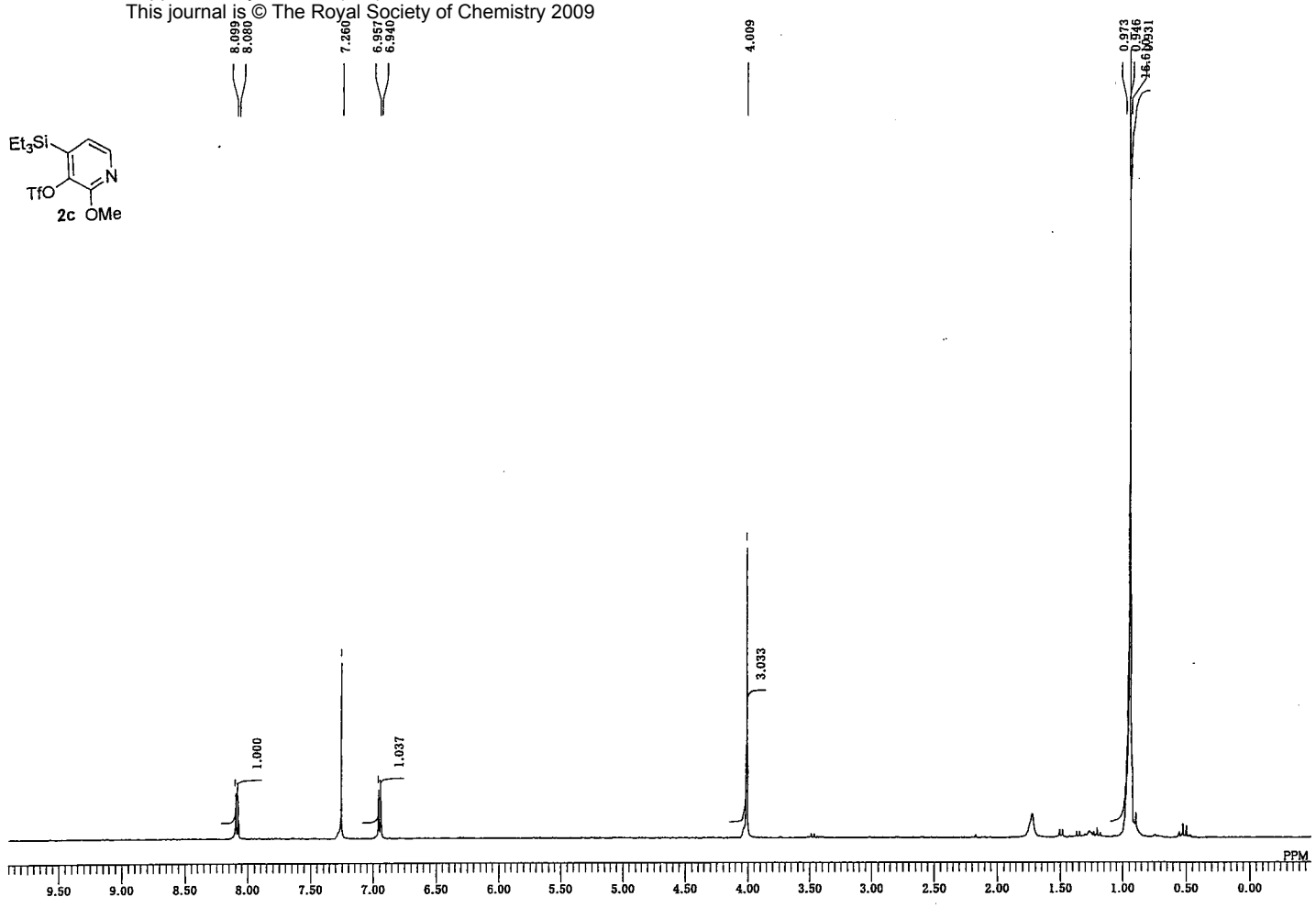
Mp 168 °C; IR (film) 2955, 2926, 2854, 1735, 1635, 1584, 1437, 1251, 1215, 1162, 1054 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.35 (d, *J* = 6.0 Hz, 1 H), 7.55 (s, 1 H), 7.47 (d, *J* = 6.0 Hz, 1 H), 5.07 (s, 2 H), 5.04 (s, 2 H), 3.77 (s, 6 H), 3.75 (s, 2 H), 3.60 (s, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.5, 155.6, 144.1, 141.8, 135.3, 133.3, 129.2, 121.8, 119.5, 119.3, 70.5, 66.3, 60.5, 53.2, 40.4, 37.9; LRMS (EI) *m/z* 327 (M^+), 296, 267, 238, 208, 127, 59; HRMS (EI) calcd for $C_{18}H_{17}O_5N$ (M^+) 327.1107, found 327.1116.

Dimethyl 8,10-dihydro-1*H*-isobenzofuro[6,5,4-*de*]quinoline-2,2(3*H*)-dicarboxylate (Table 3, run 6, 6f)

Mp 166 °C; IR (film) 2955, 2926, 2854, 1731, 1639, 1596, 1579, 1442, 1259, 1229, 1172, 1046 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.41 (d, *J* = 6.0 Hz, 1 H), 7.51 (s, 1 H), 7.48 (d, *J* = 6.0 Hz, 1 H), 5.26 (s, 4 H), 3.80 (s, 2 H), 3.69 (s, 6 H), 3.50 (s, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.7, 155.5, 142.8, 142.4, 136.5, 135.9, 126.3, 123.3, 119.5, 116.3, 73.4, 72.1, 54.4, 53.2, 38.9, 32.9; LRMS (EI) *m/z* 327 (M^+), 296, 268, 238, 208, 127, 59; HRMS (EI) calcd for $C_{18}H_{17}O_5N$ (M^+) 327.1107, found 327.1113.



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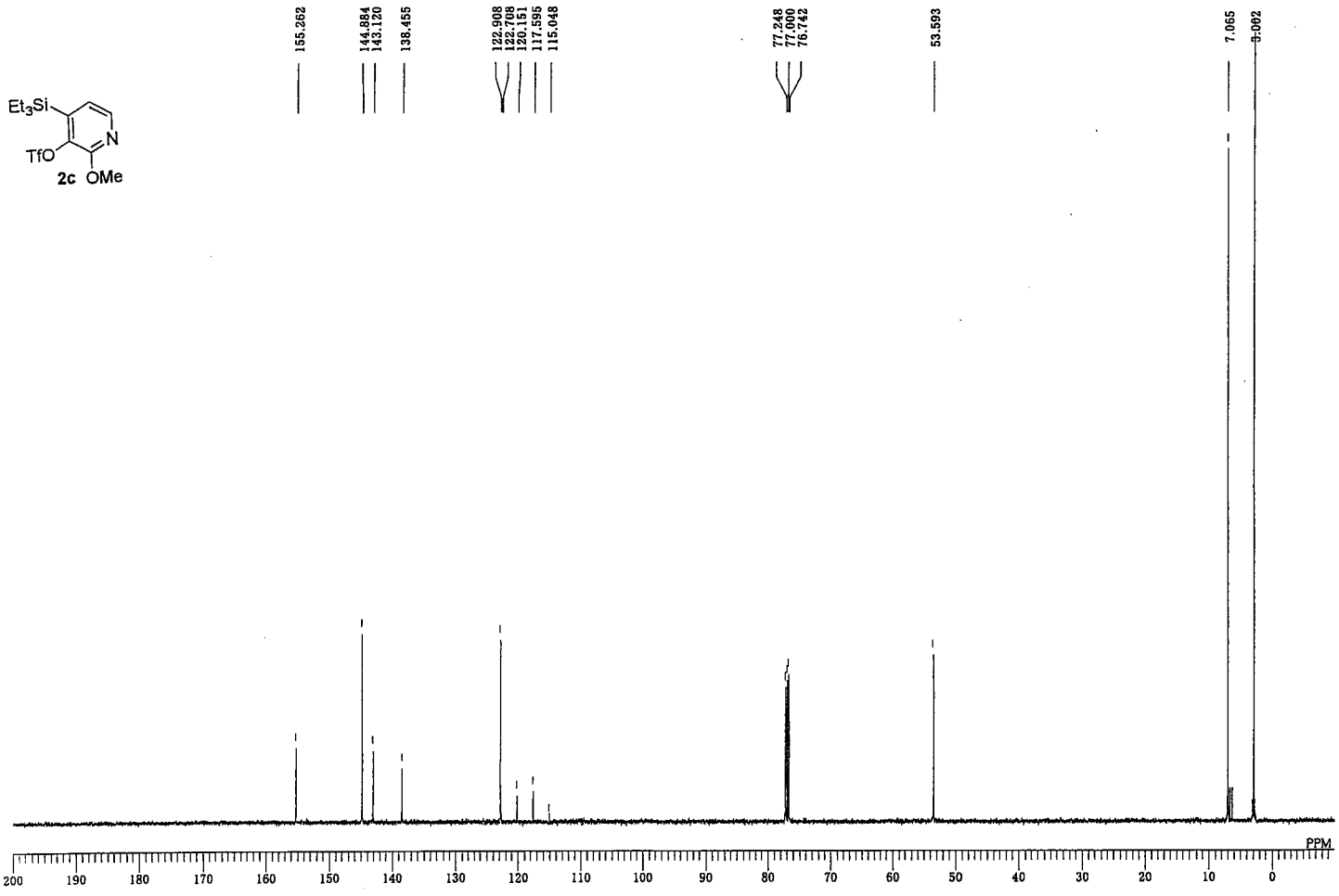
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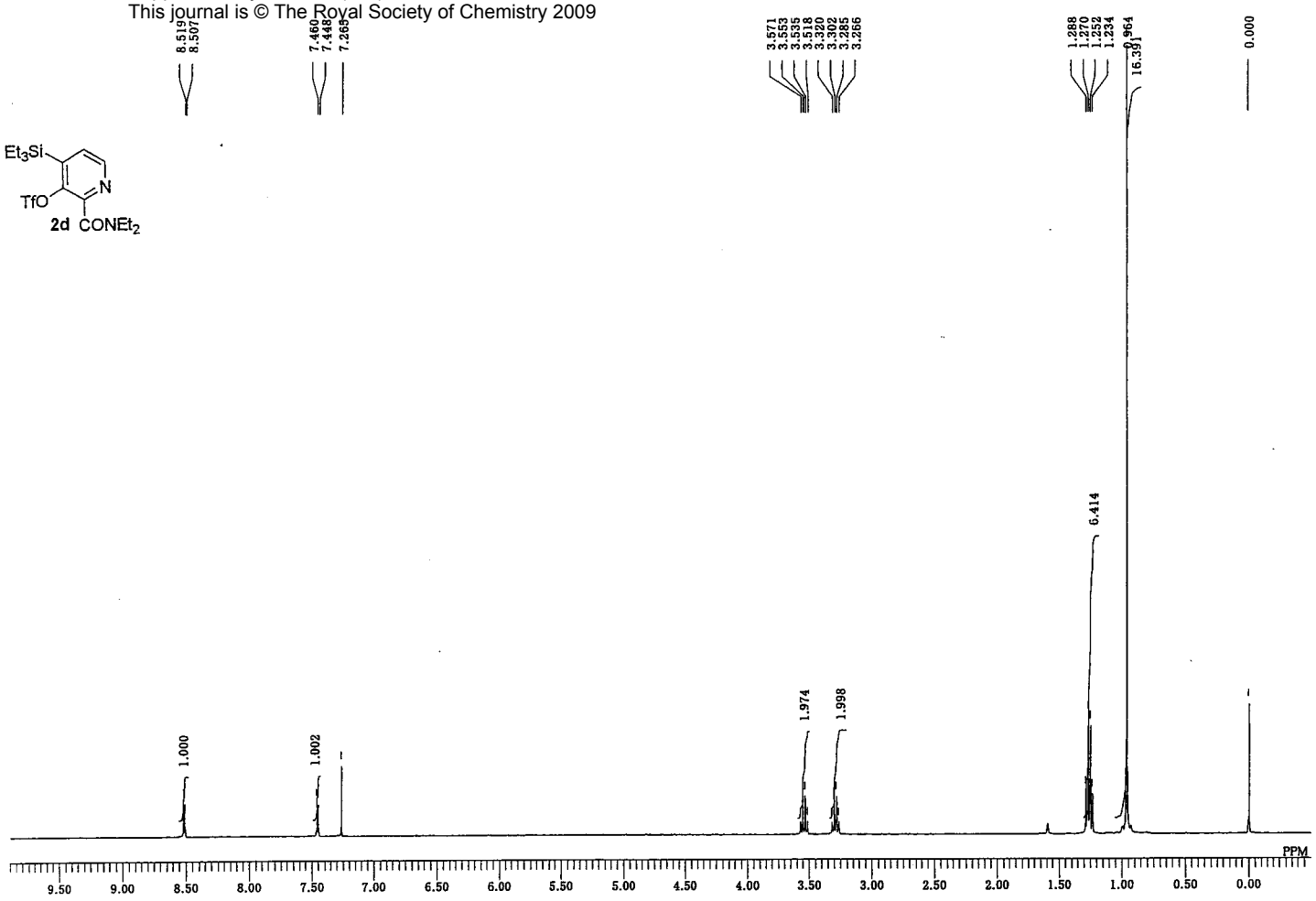

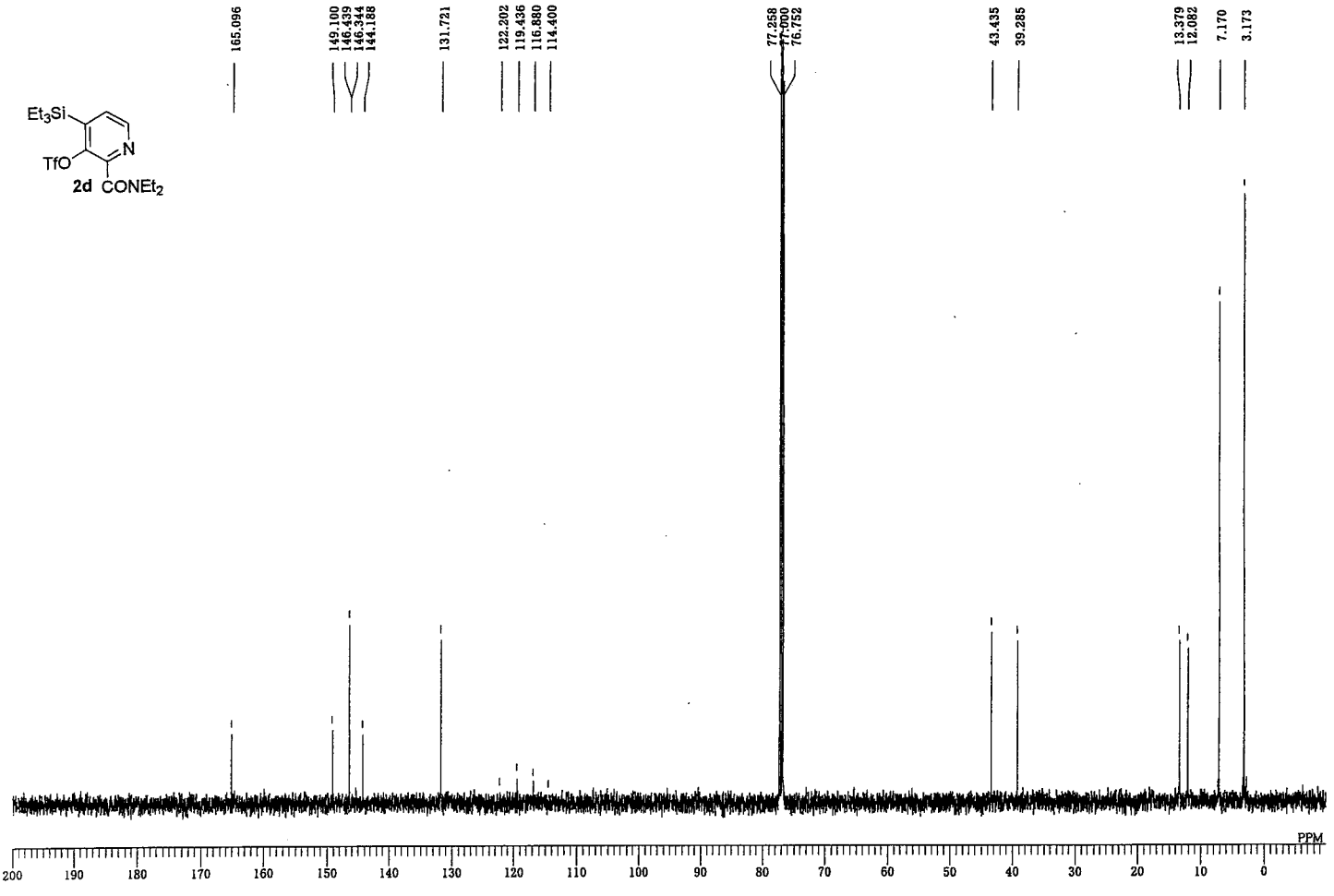

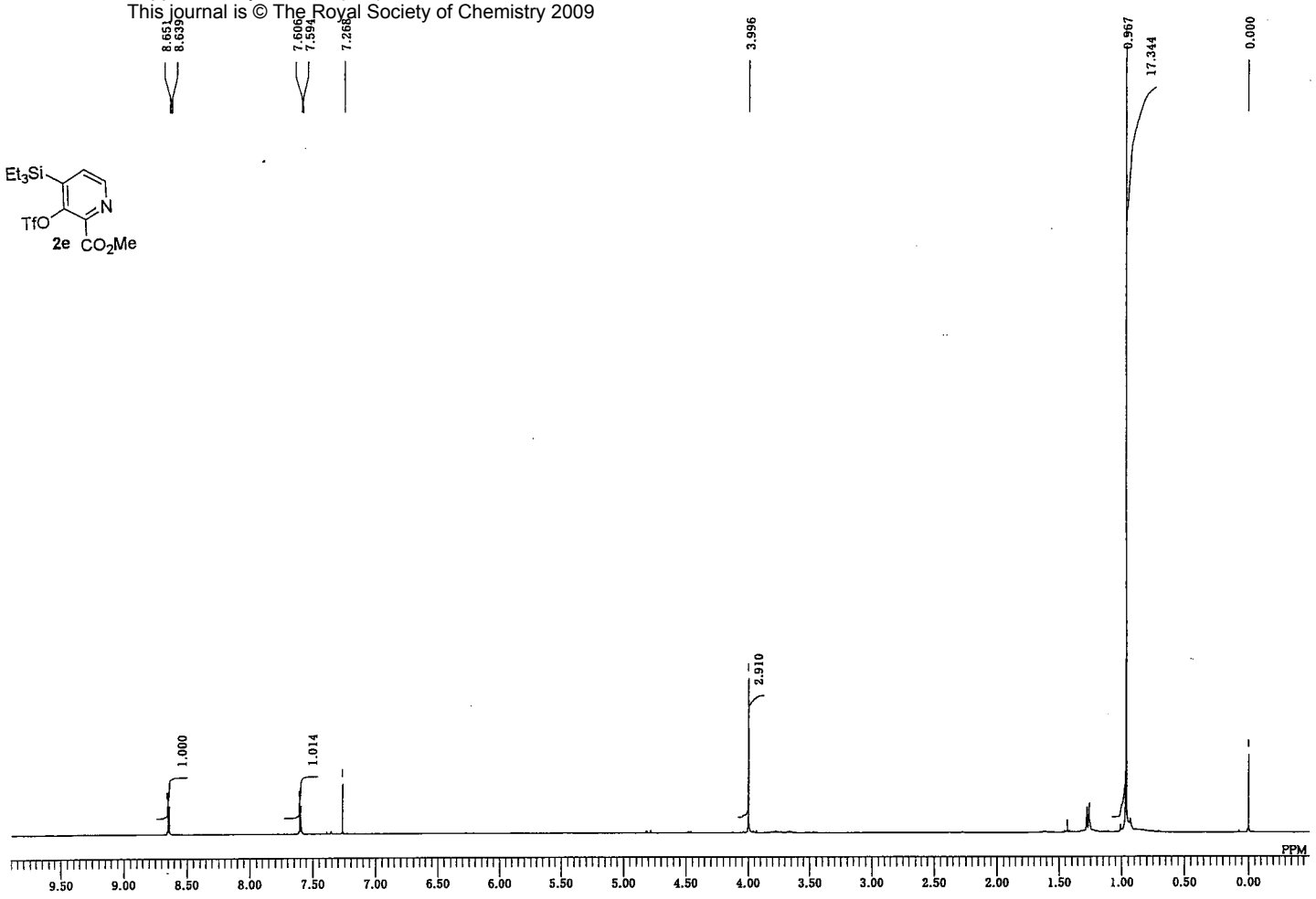
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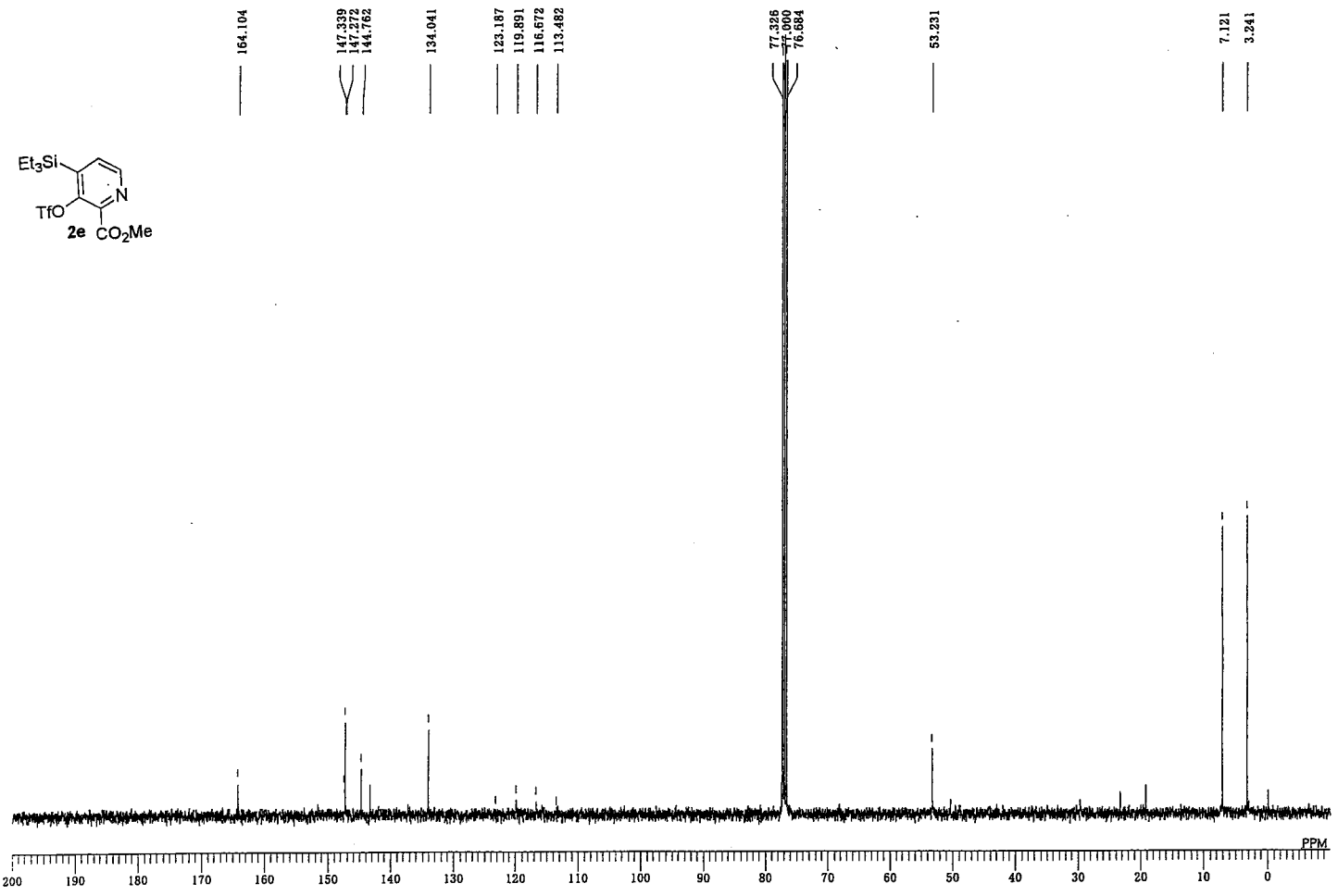
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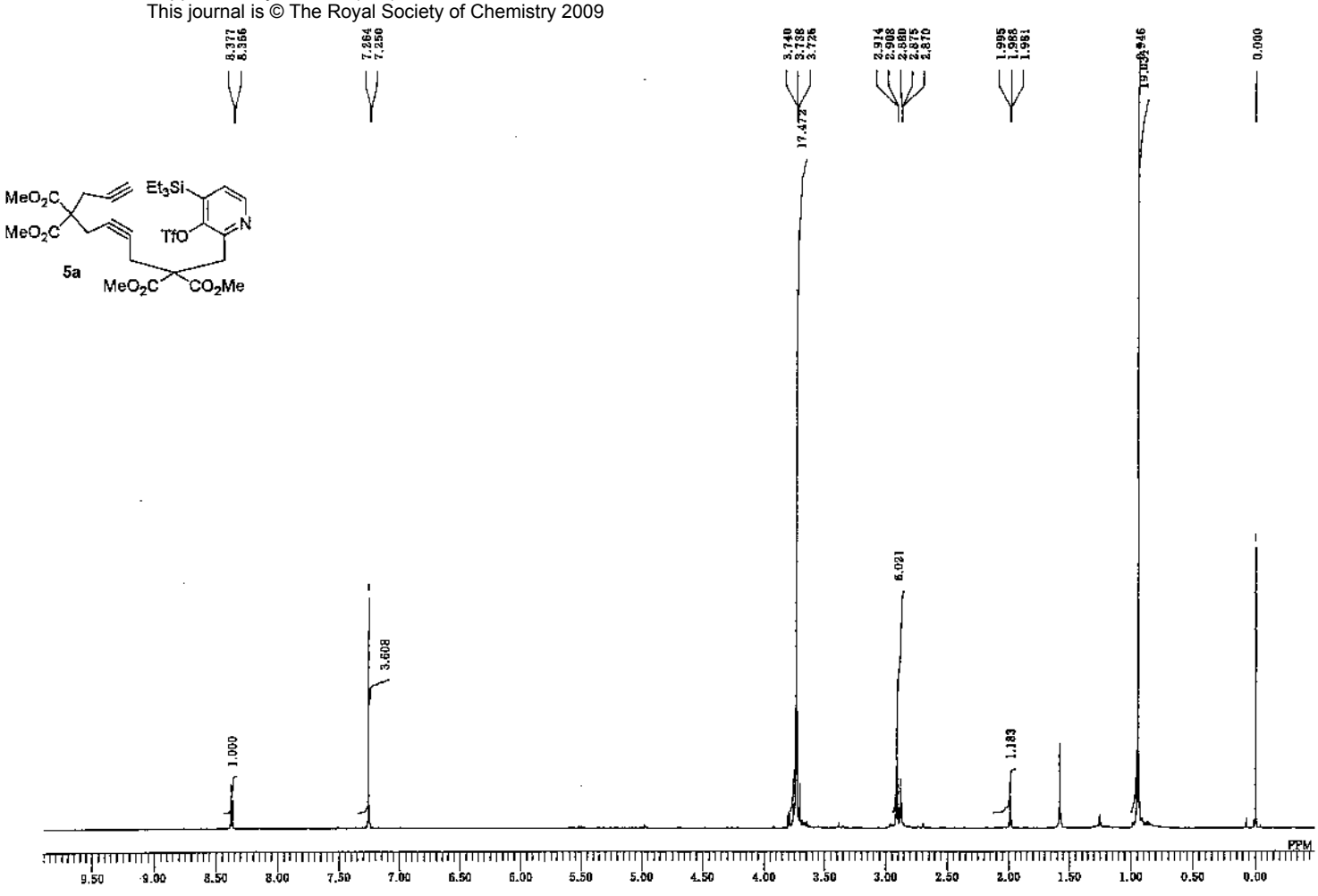
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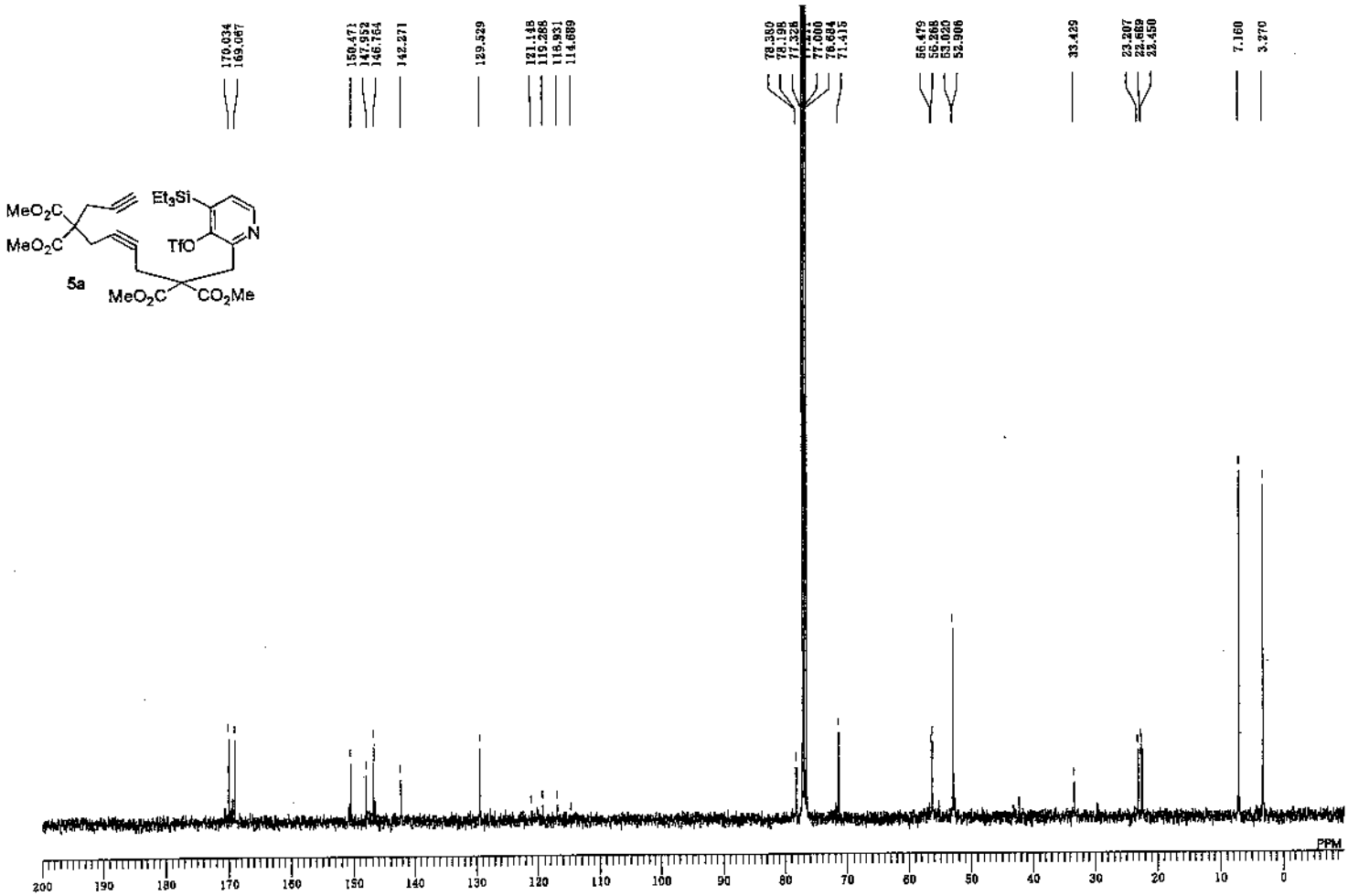
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N
5a
MeO2C
CO2Me
PPM
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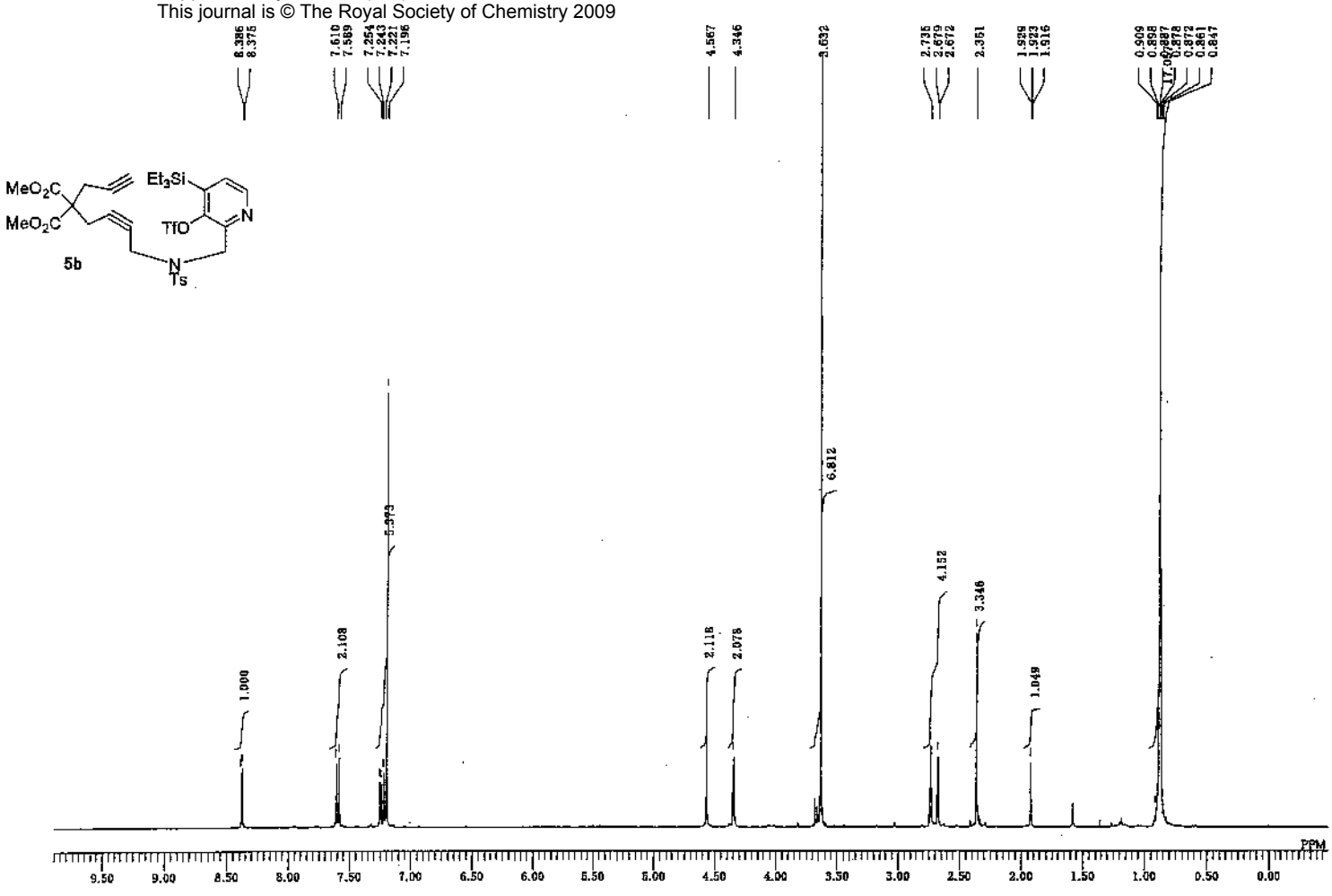
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MeO2C
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N
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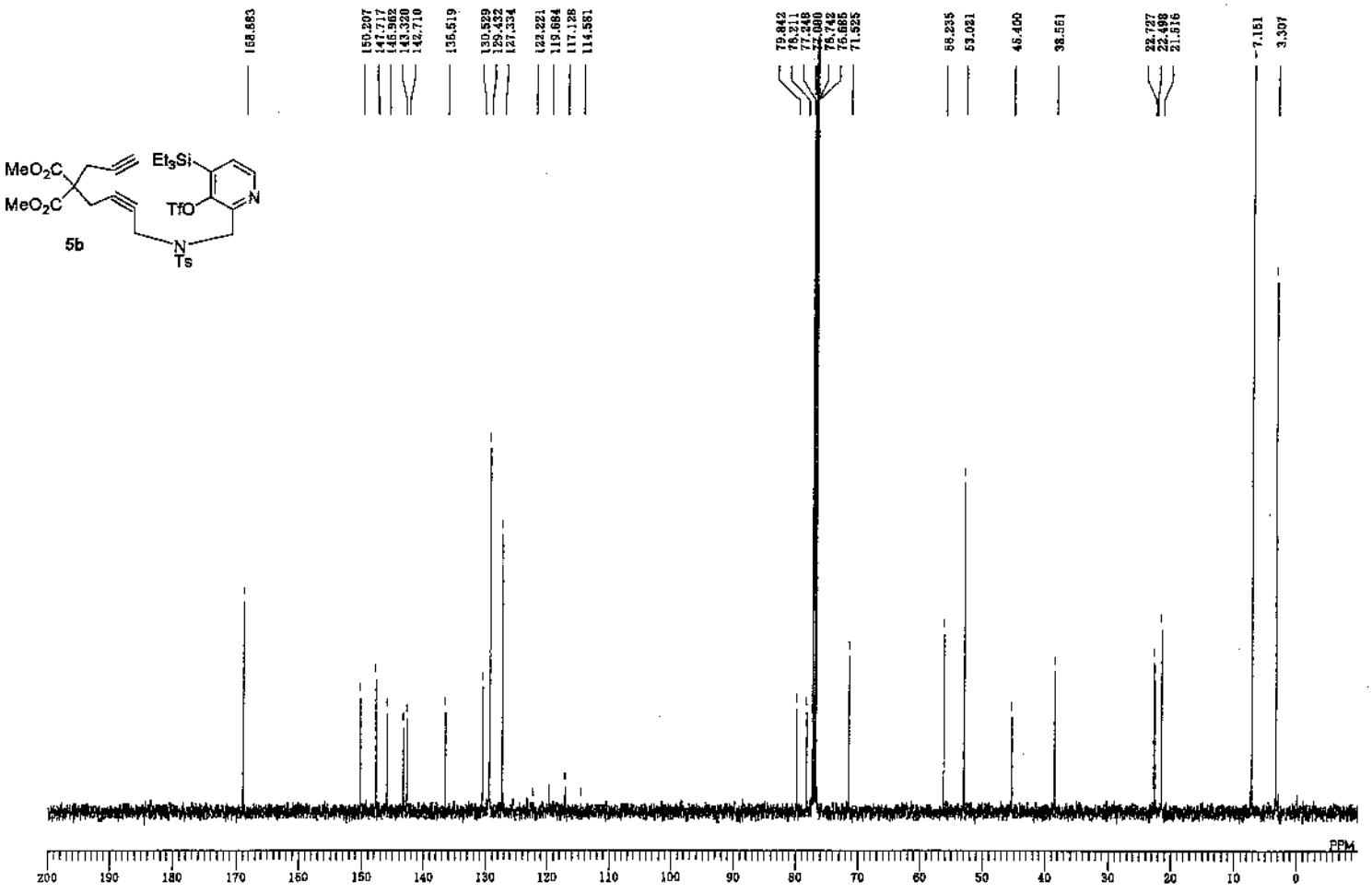
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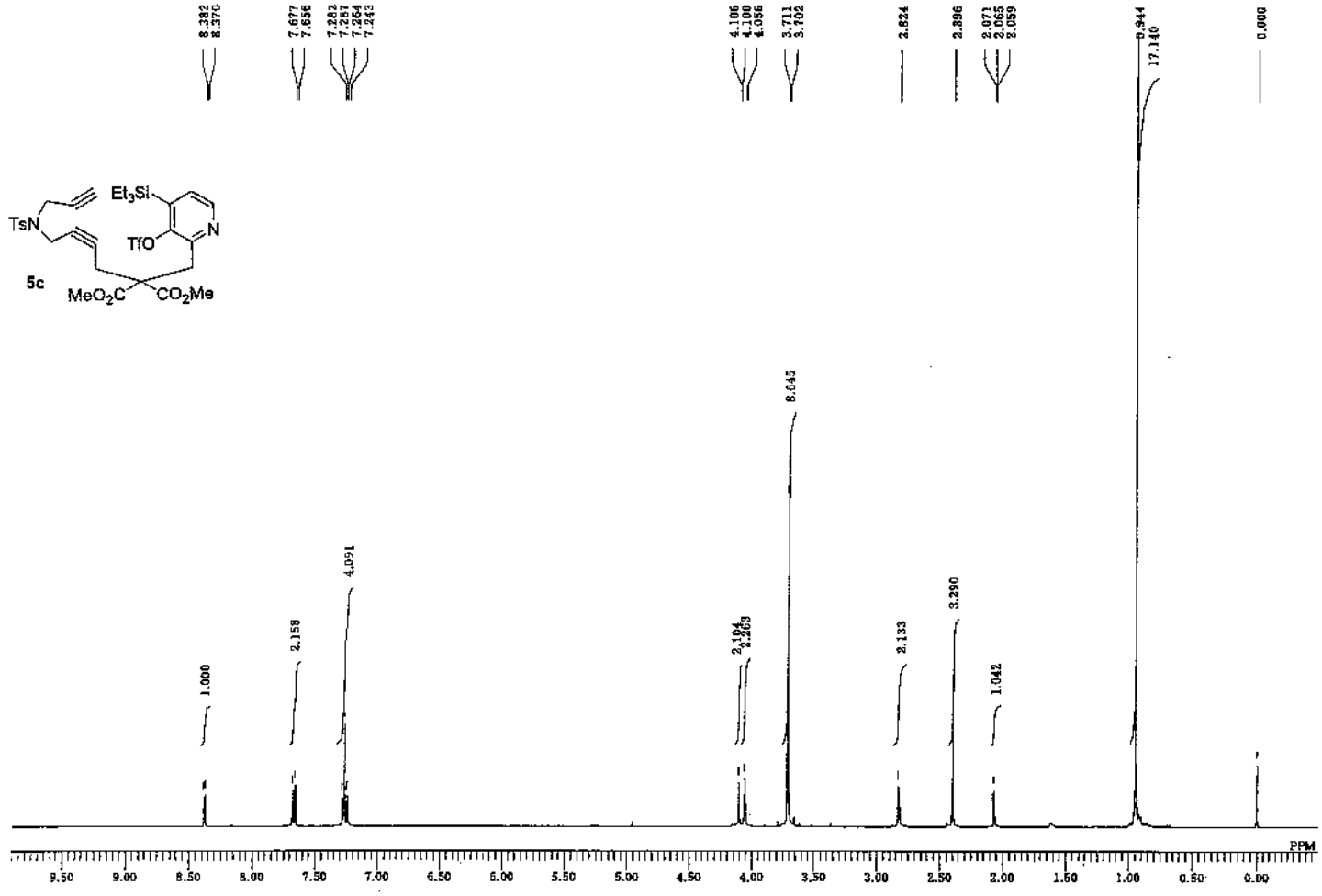
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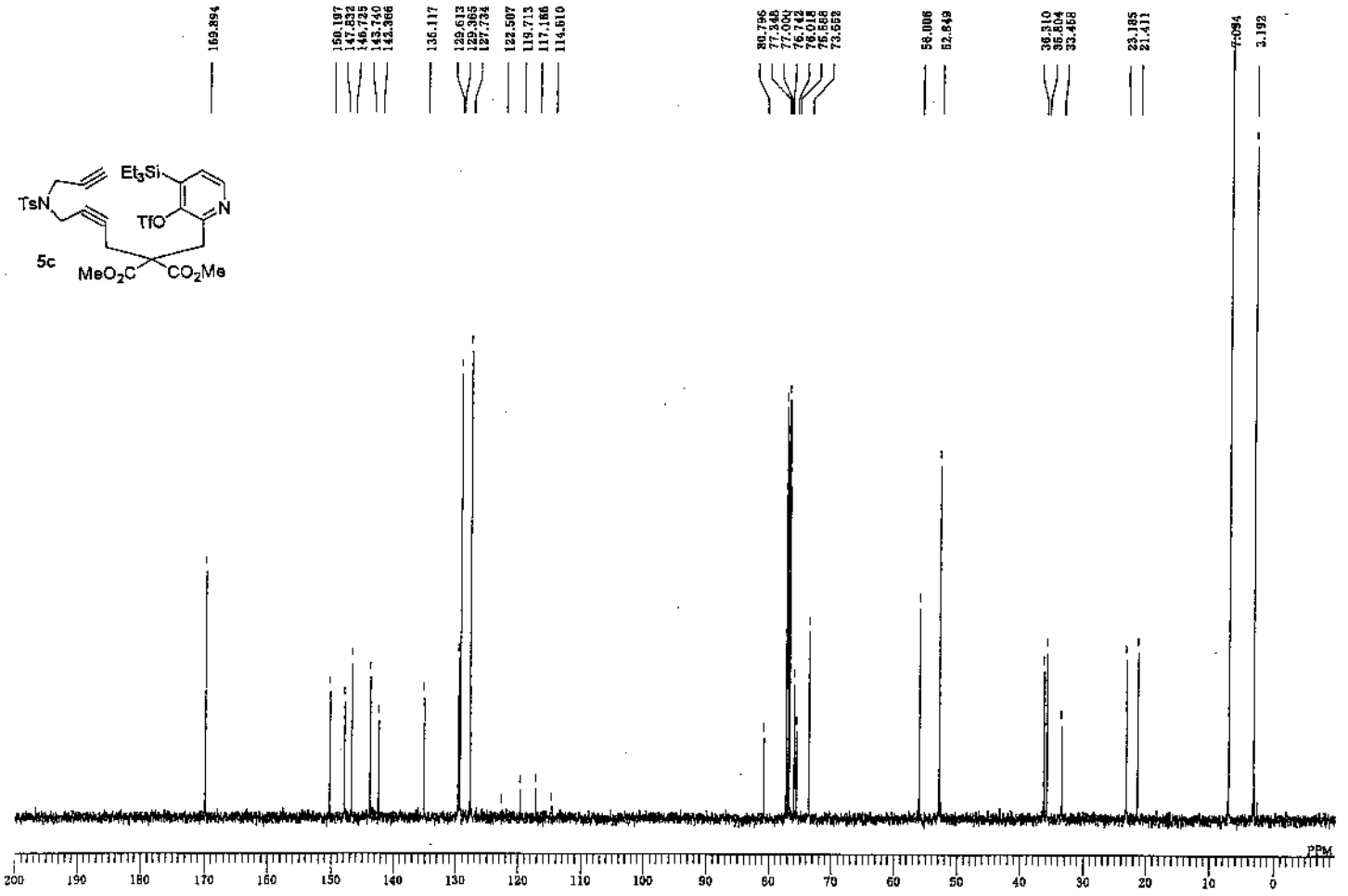
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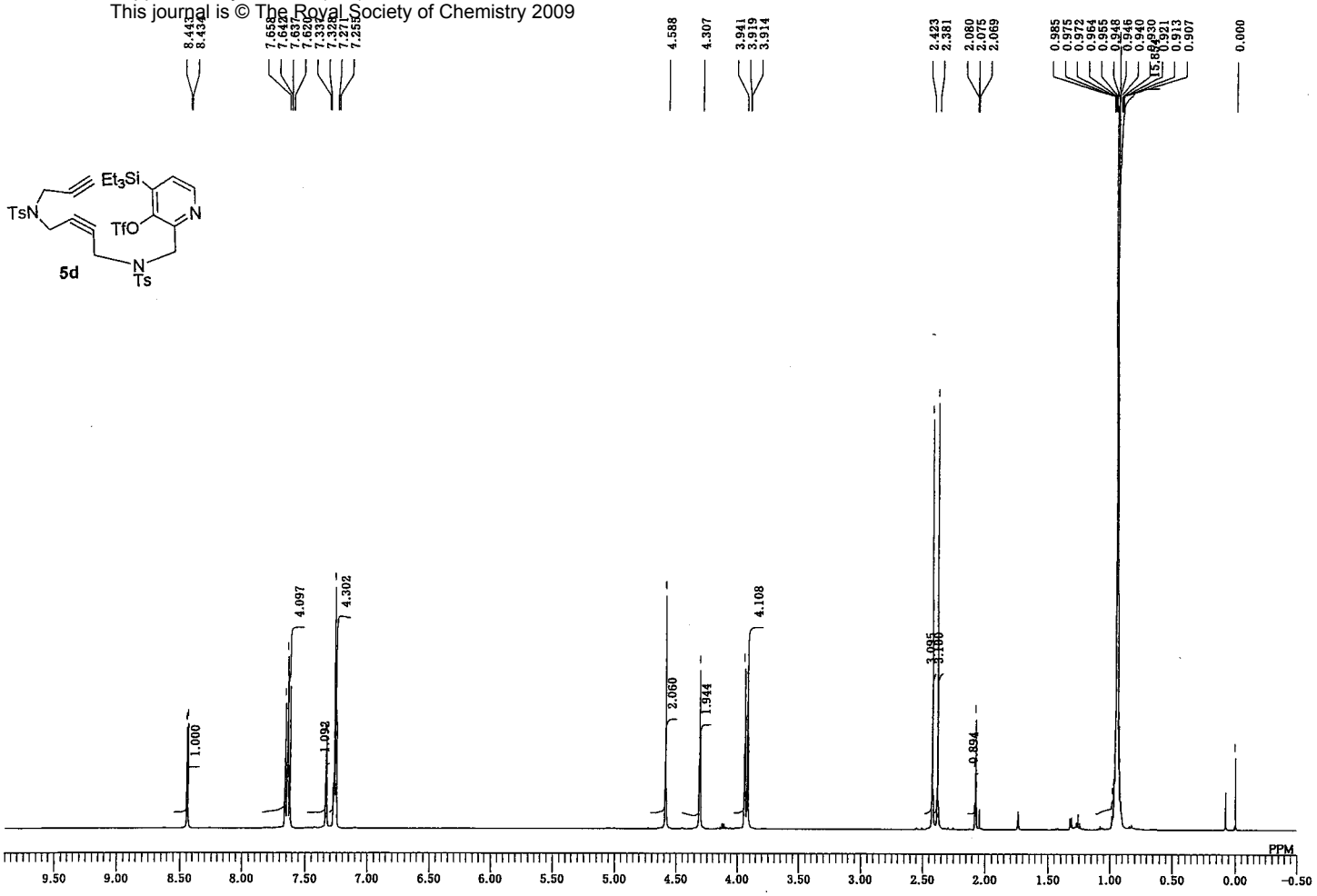

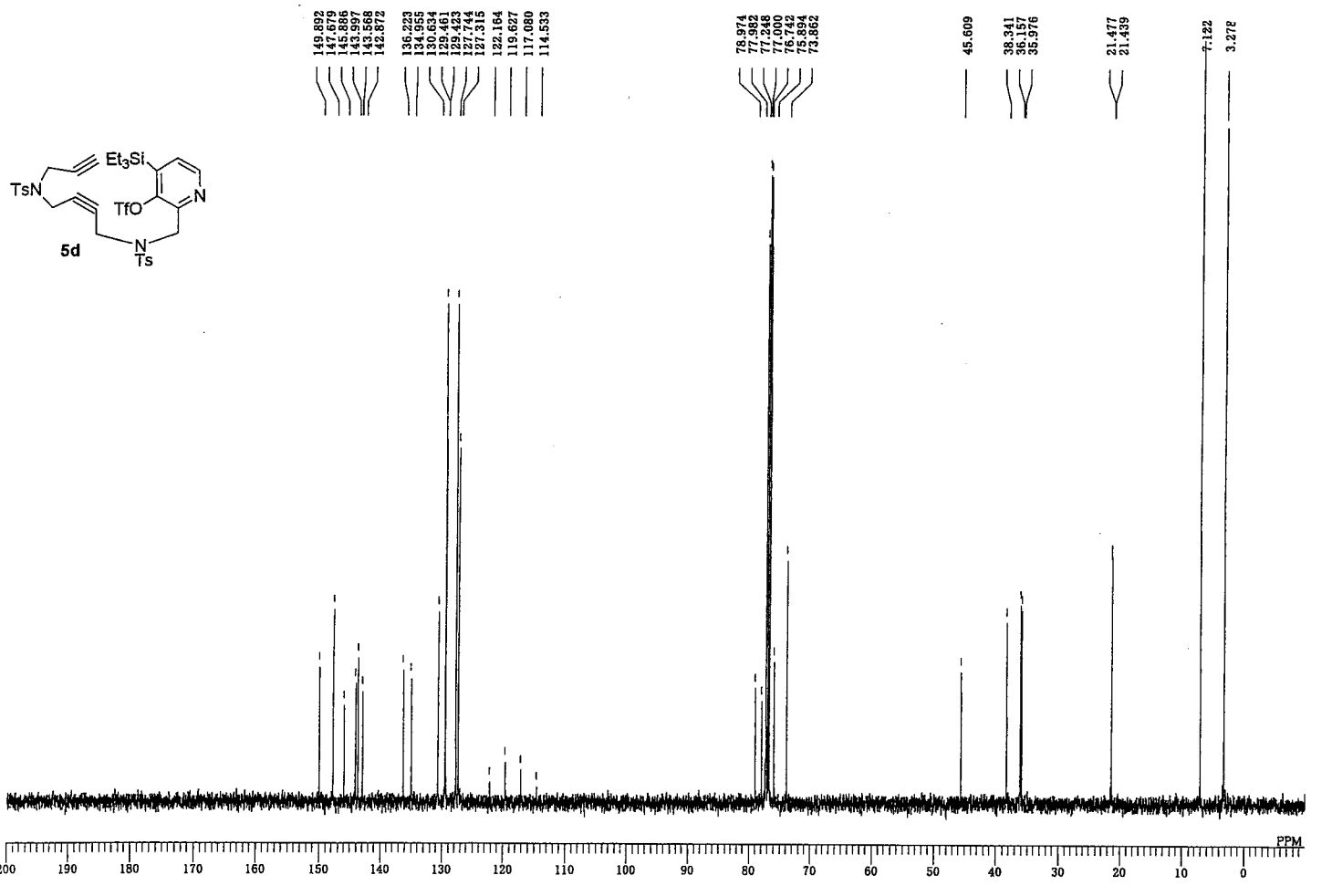

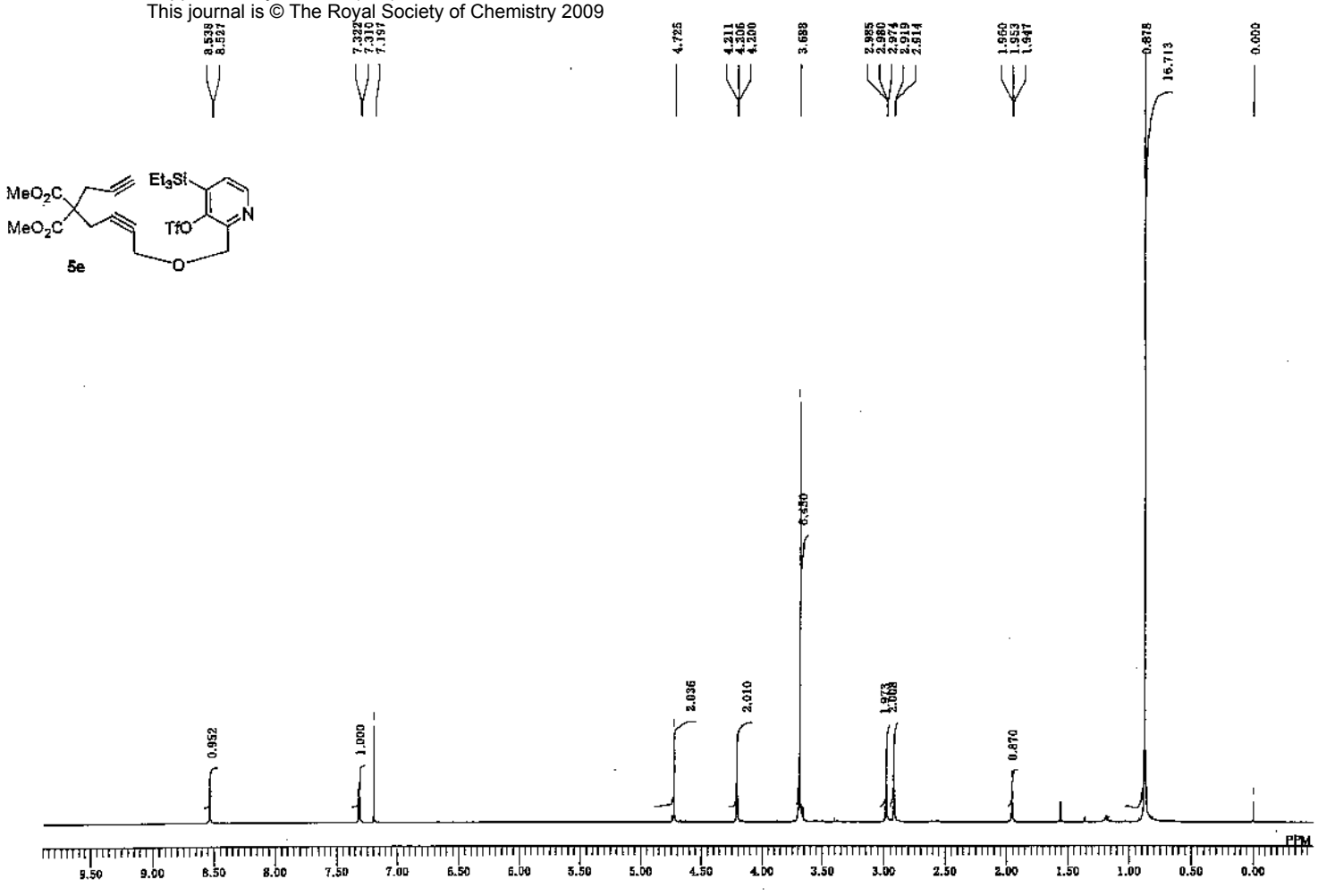
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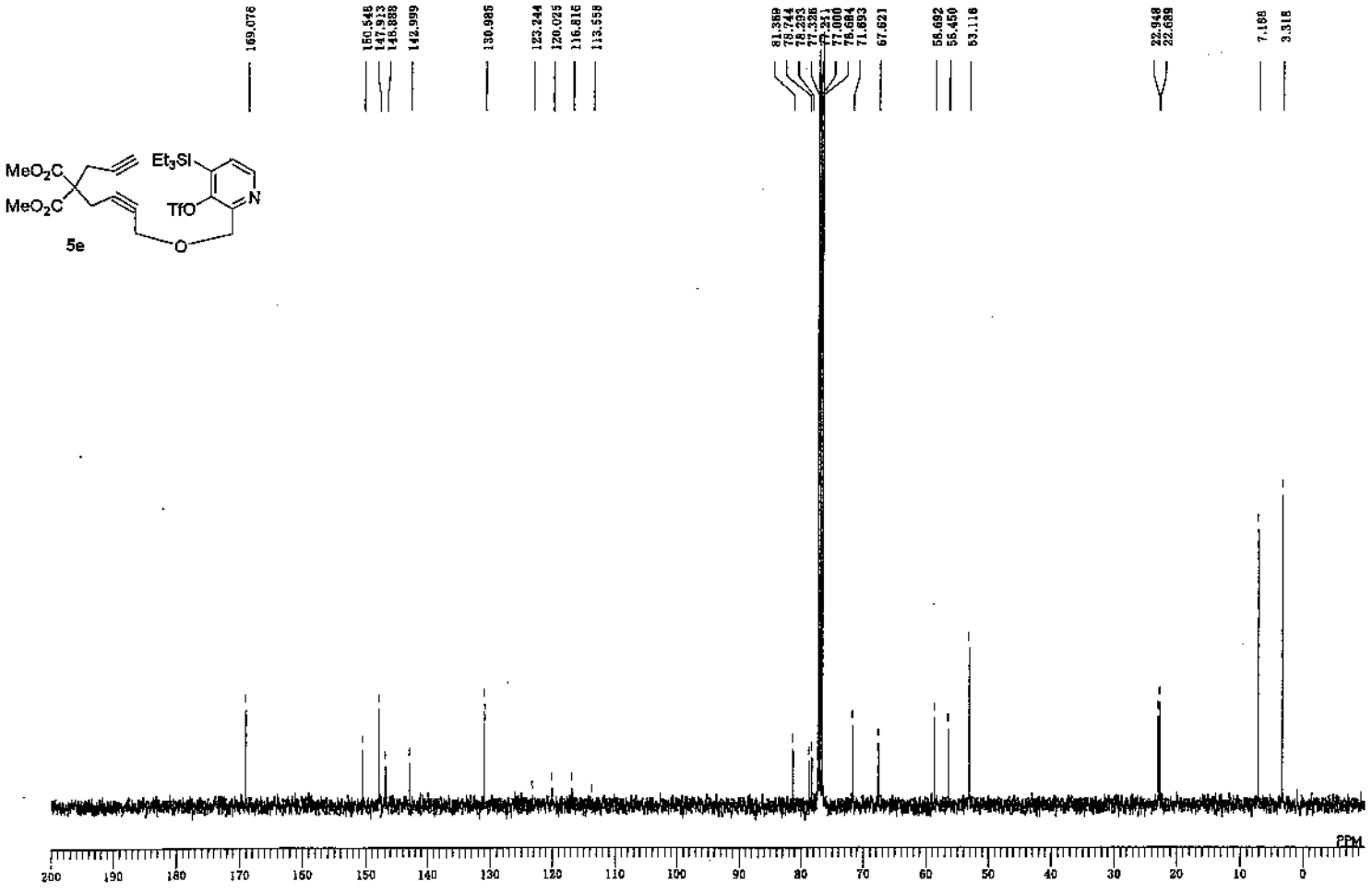
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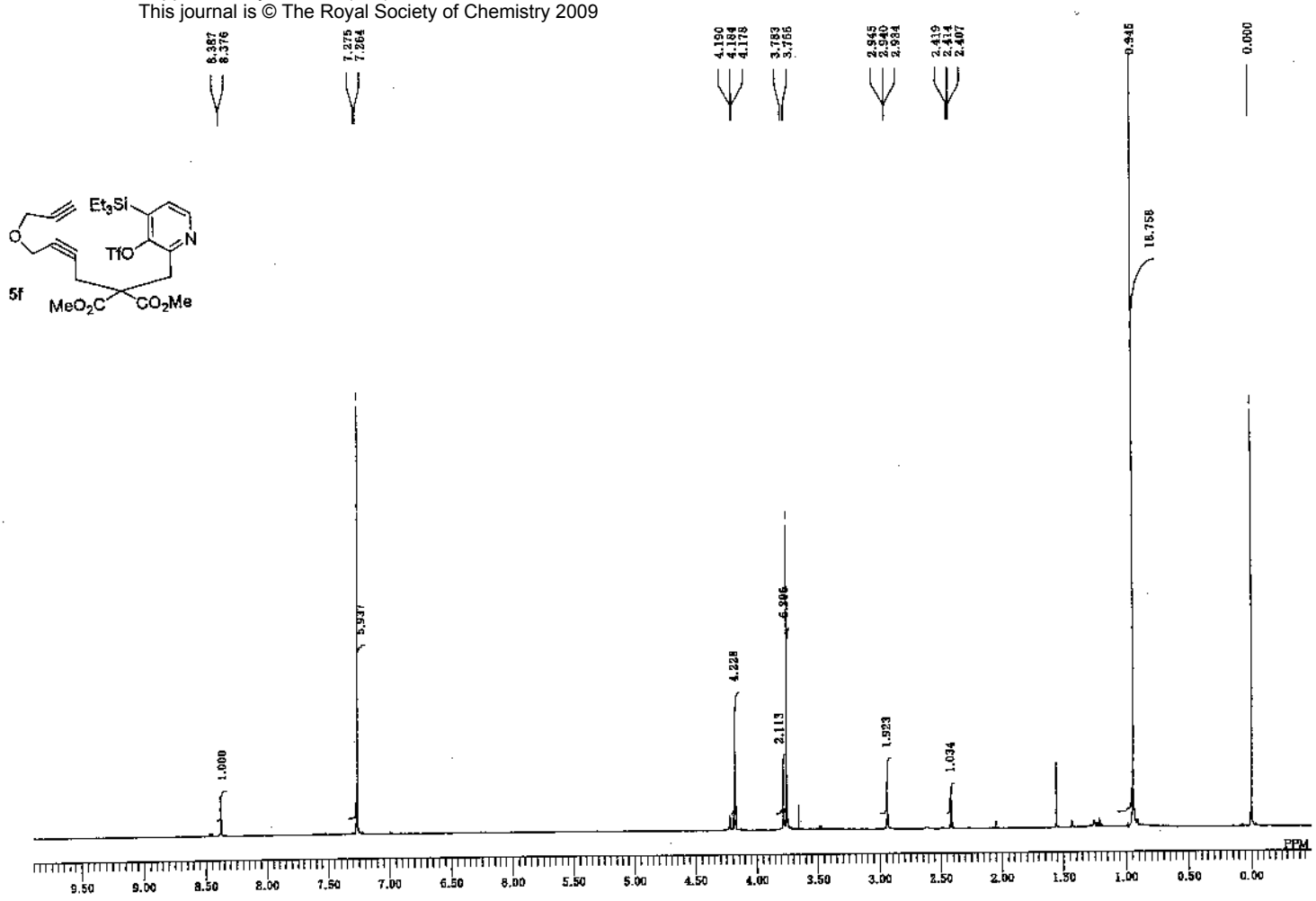
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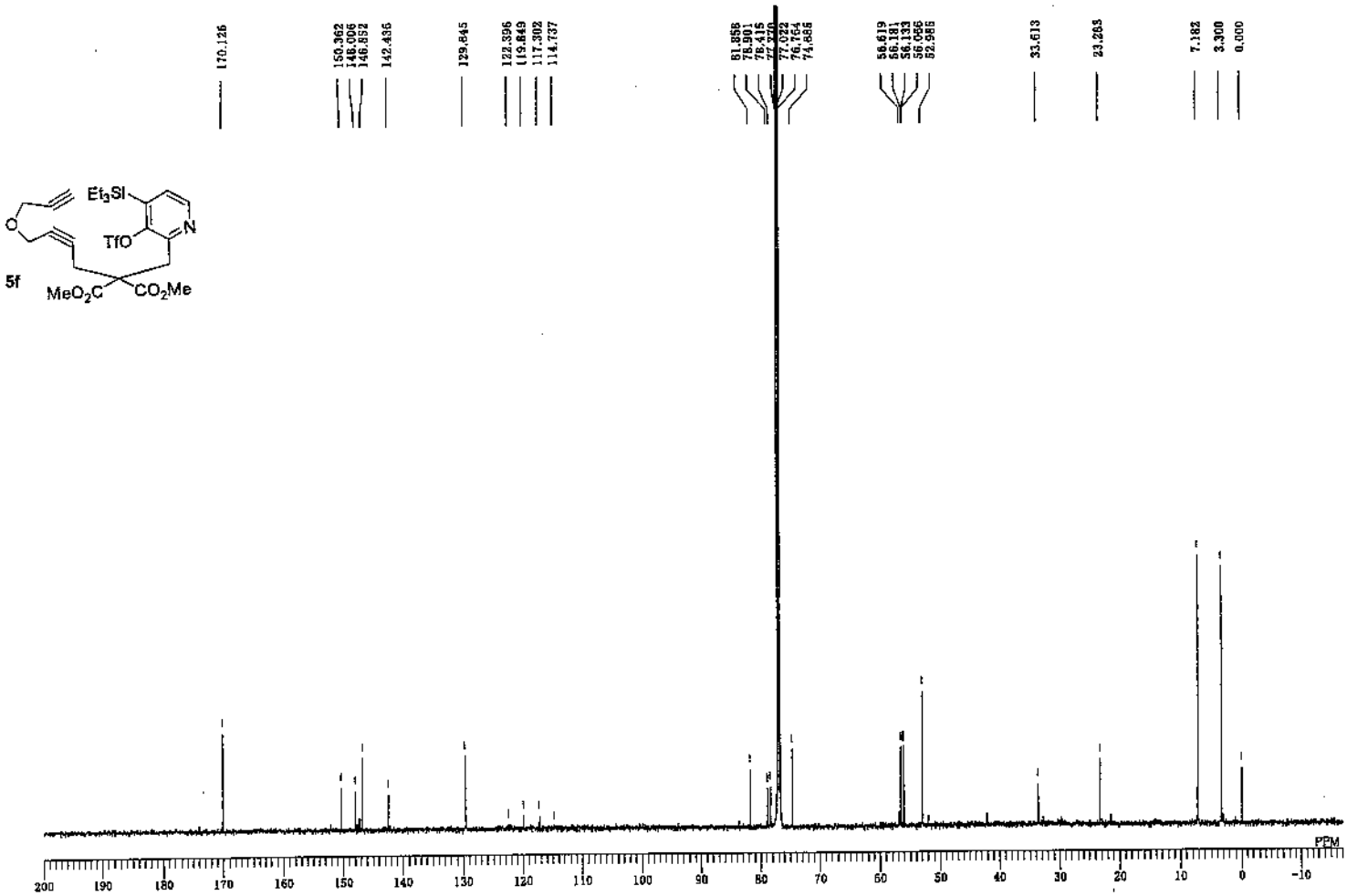
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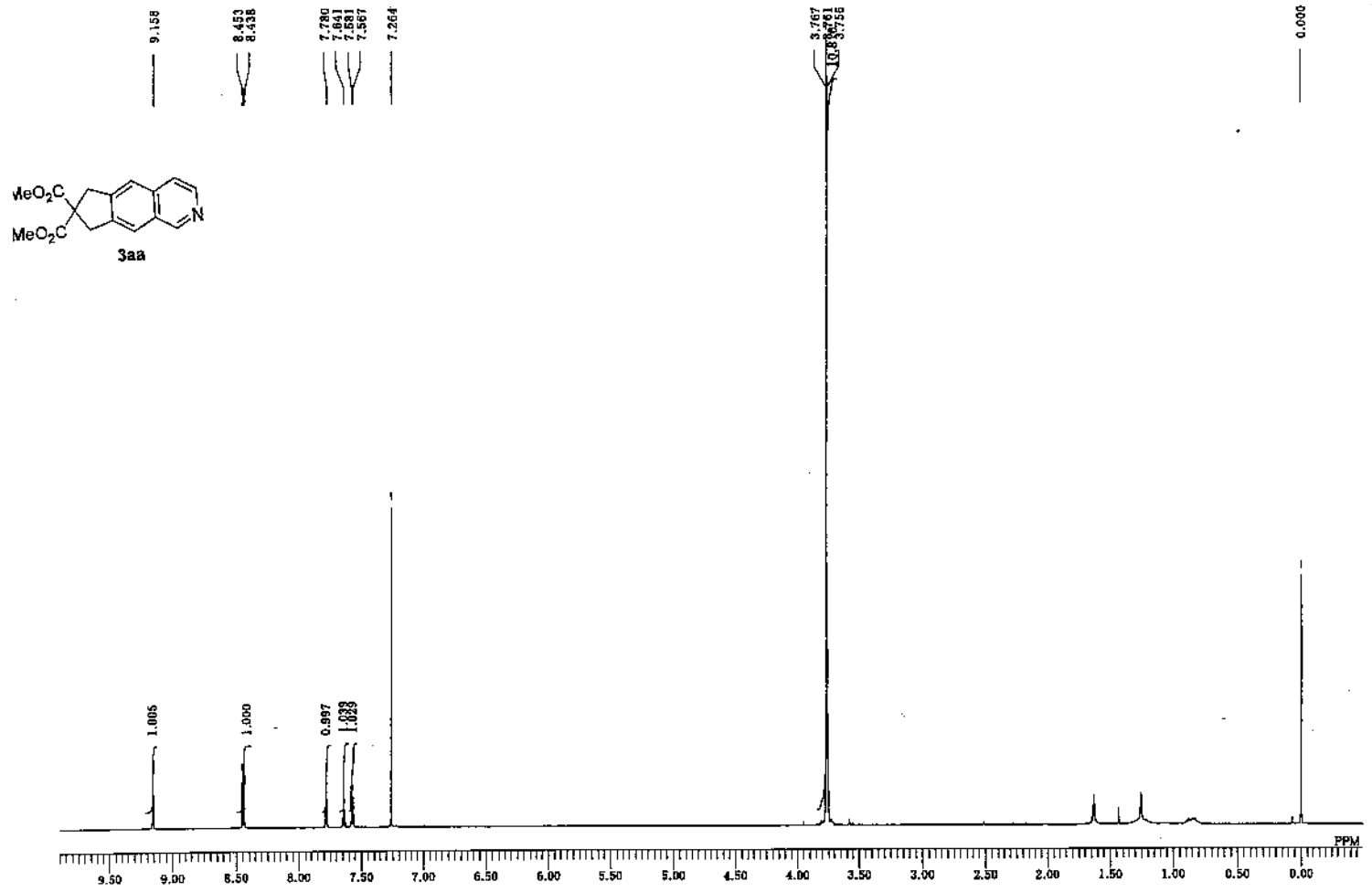
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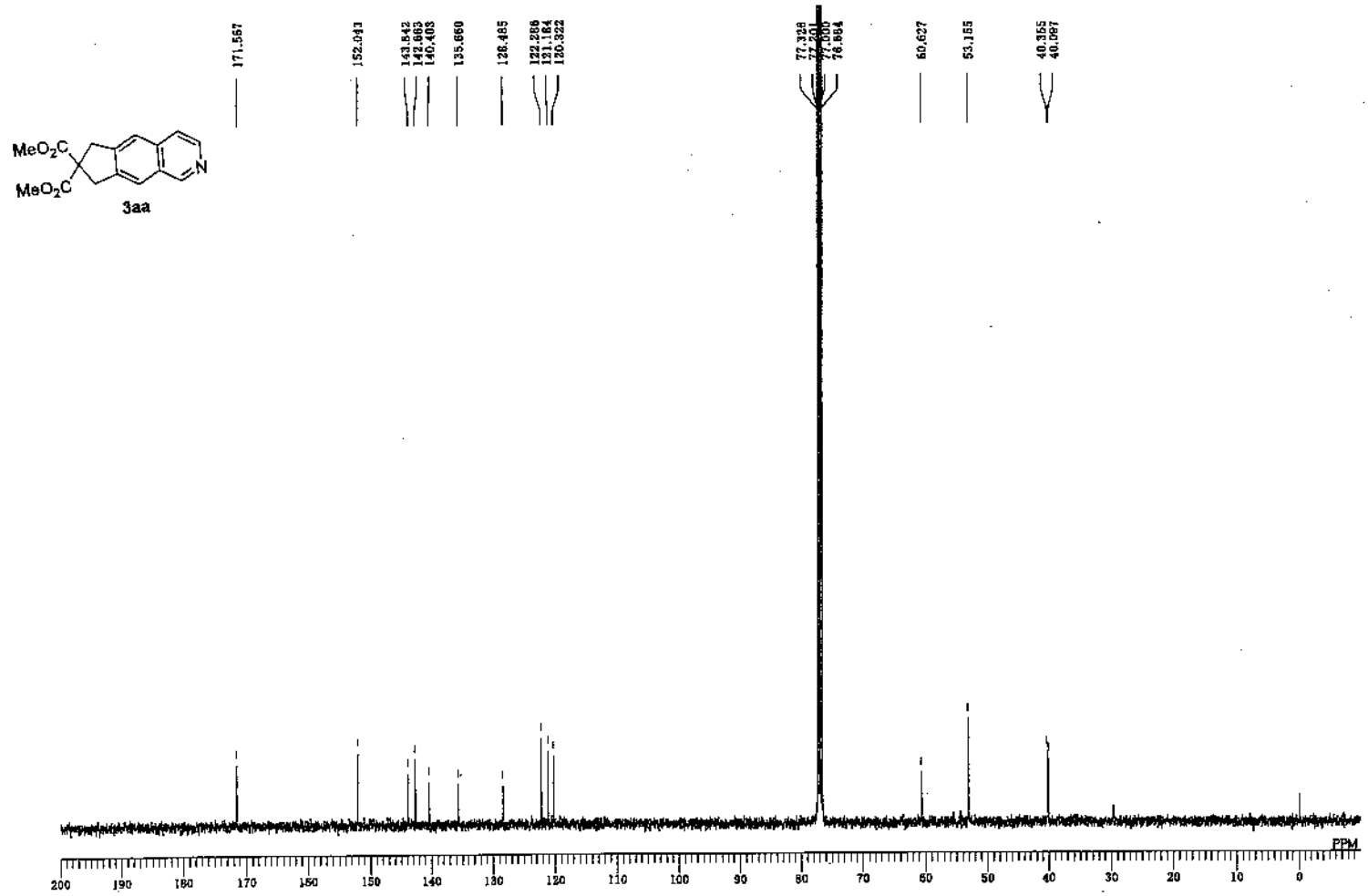
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MeO2C
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3aa
PPM
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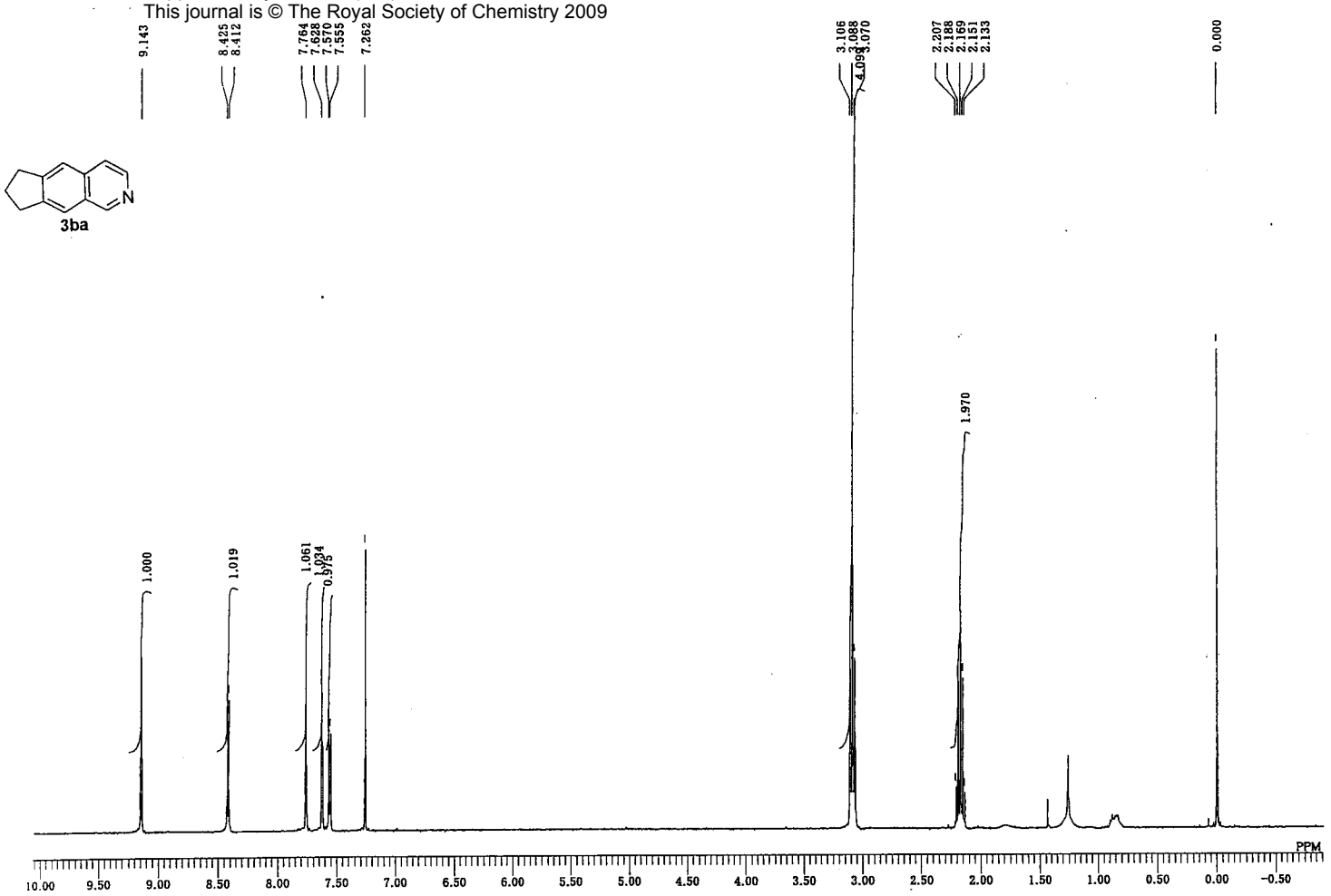
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1.000
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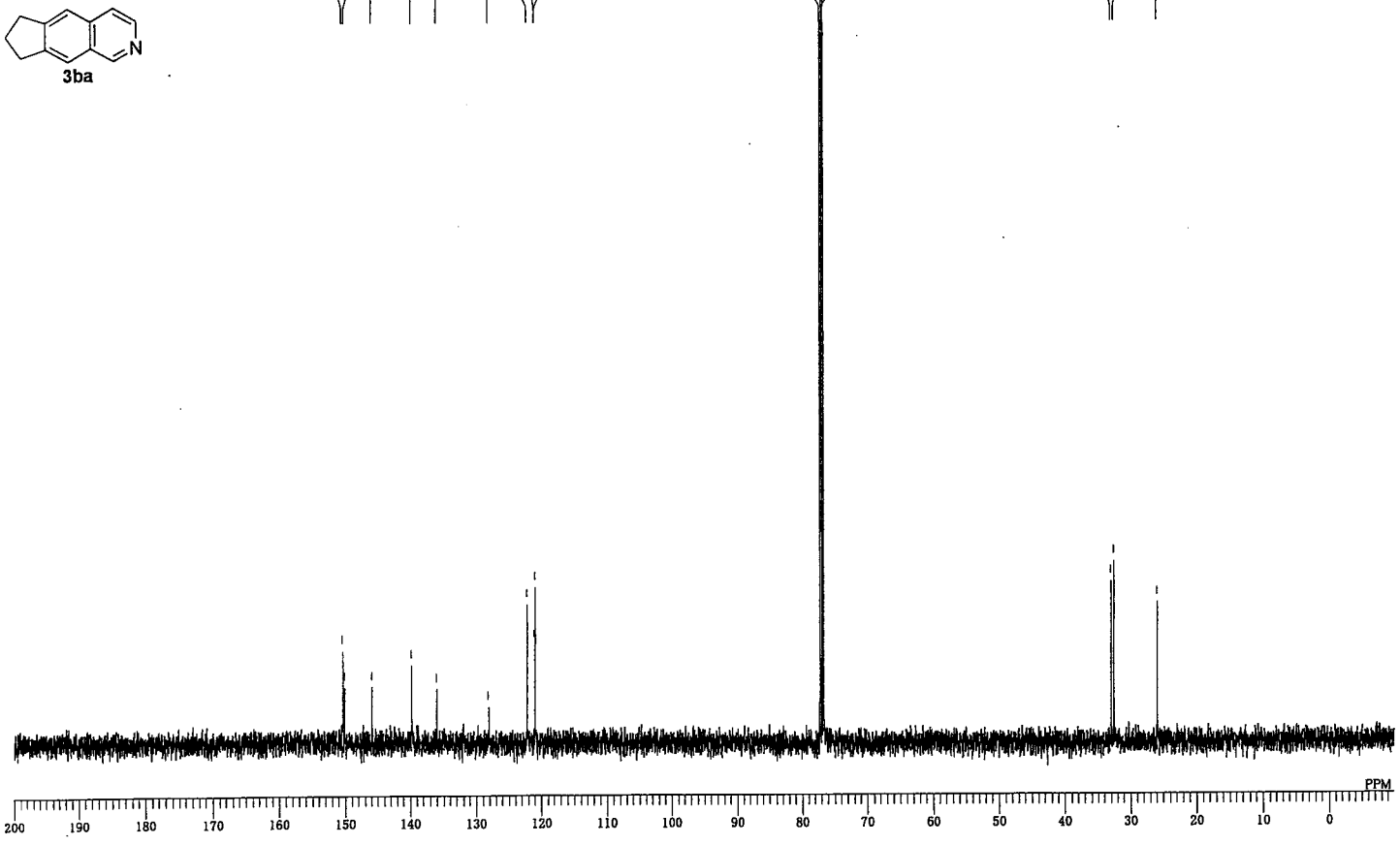
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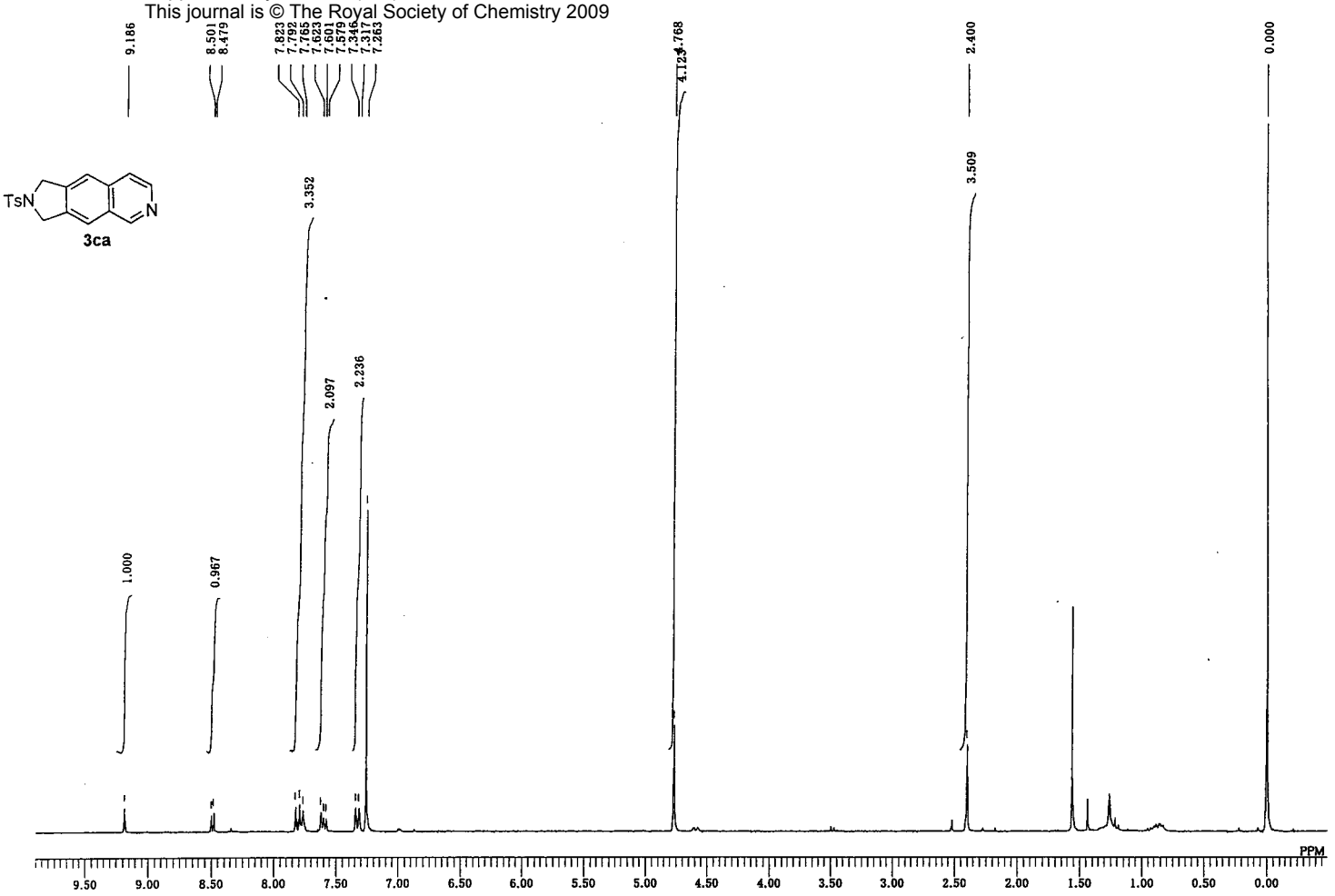
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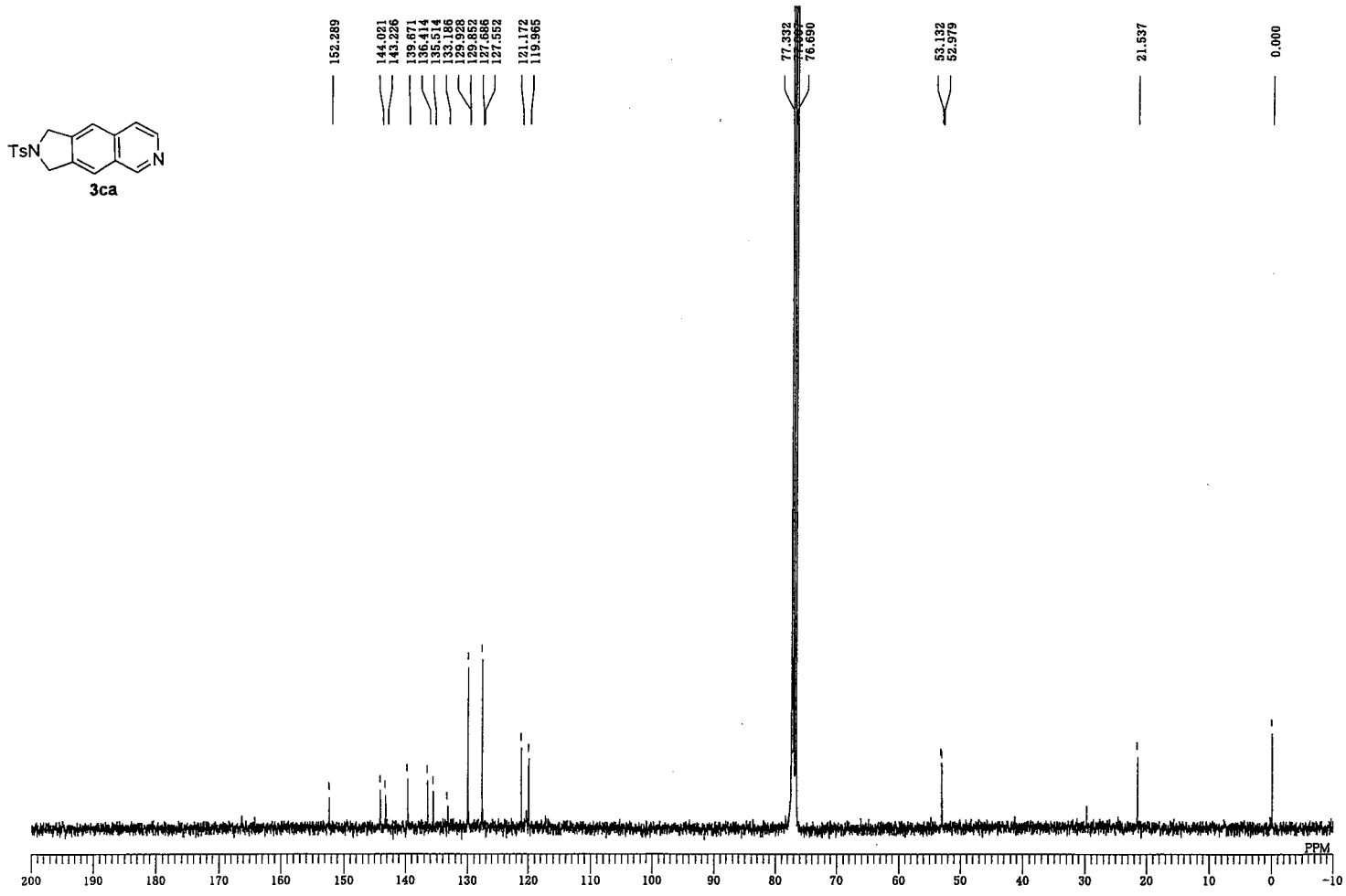
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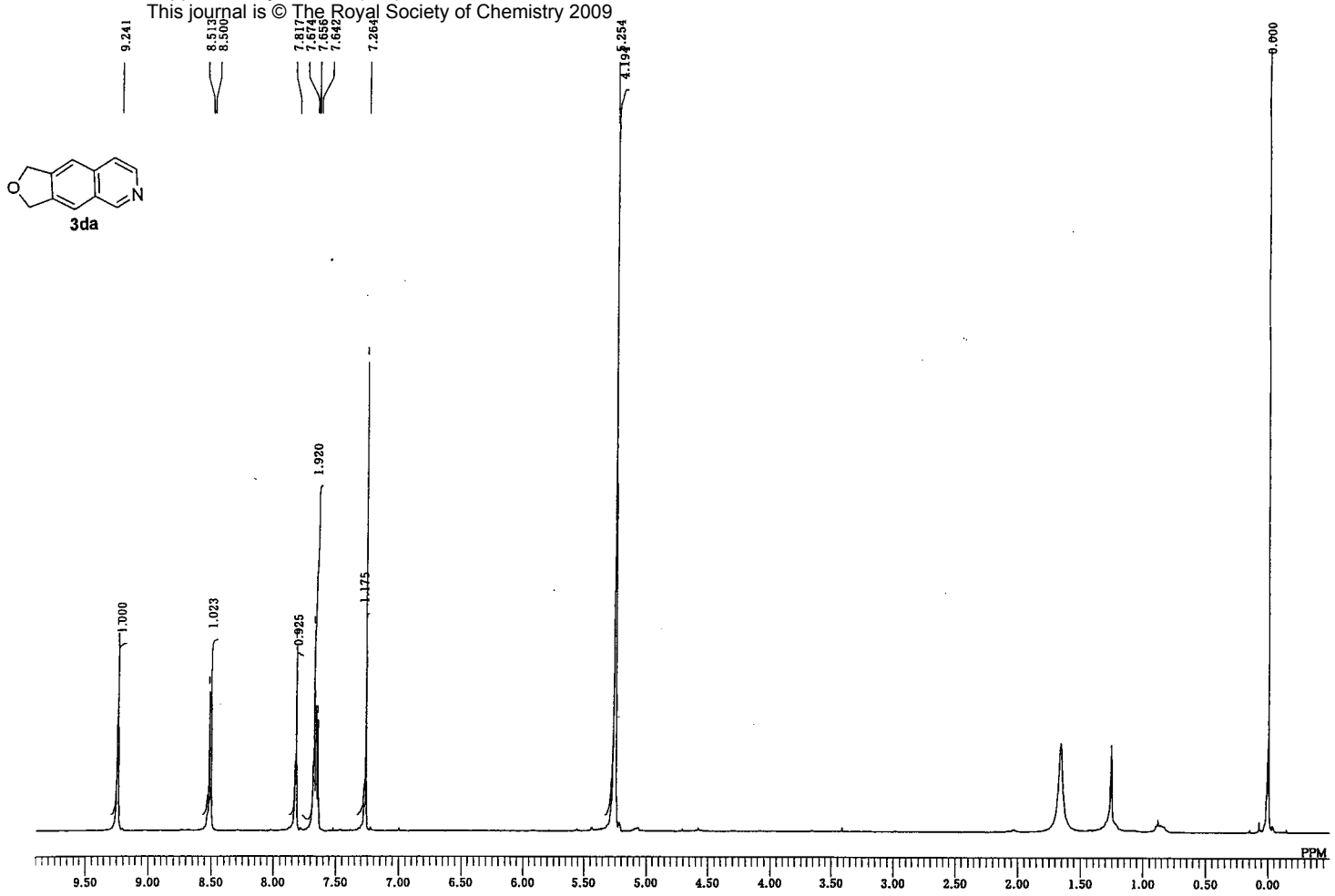
PPM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4.194
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1.175
1.000
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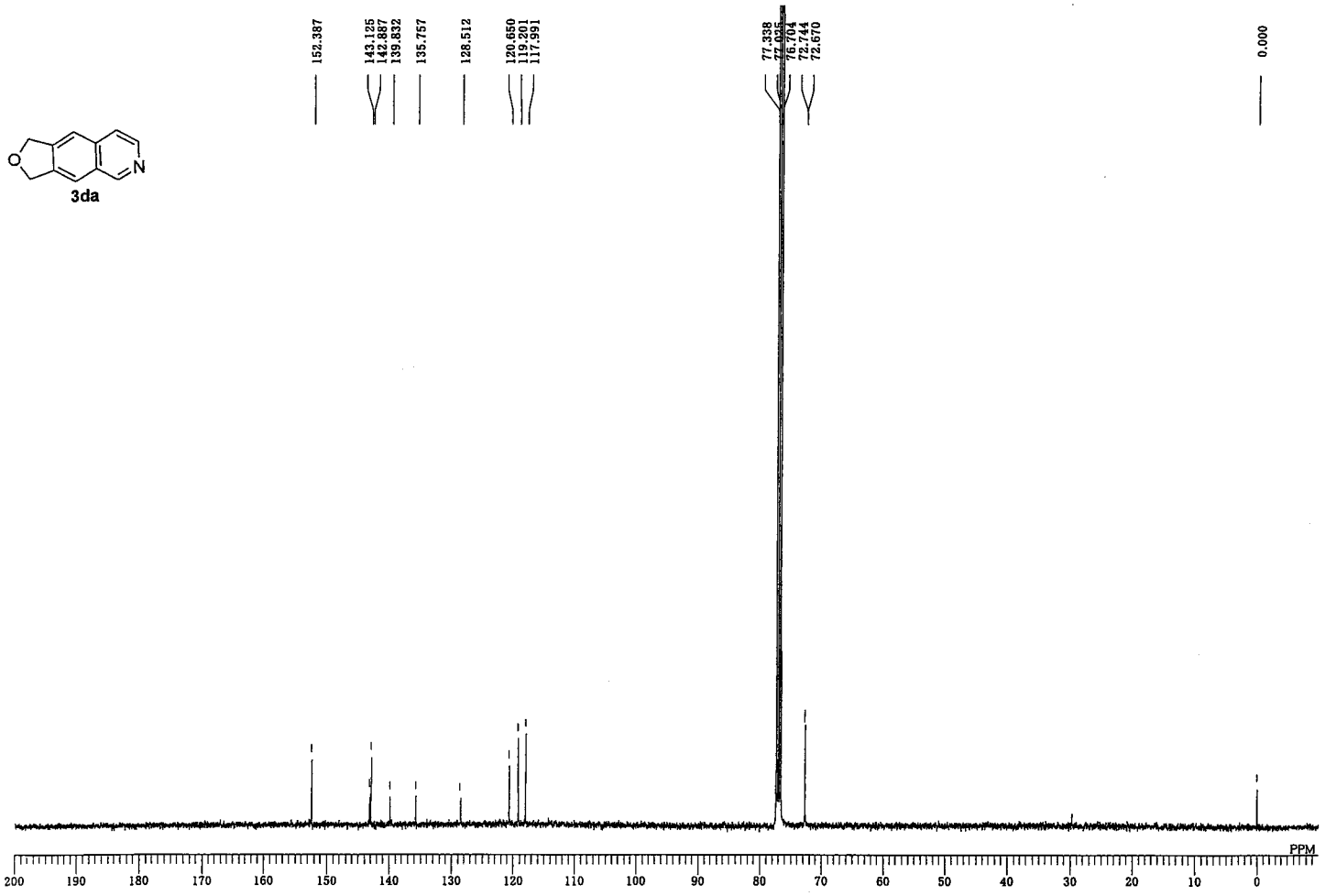
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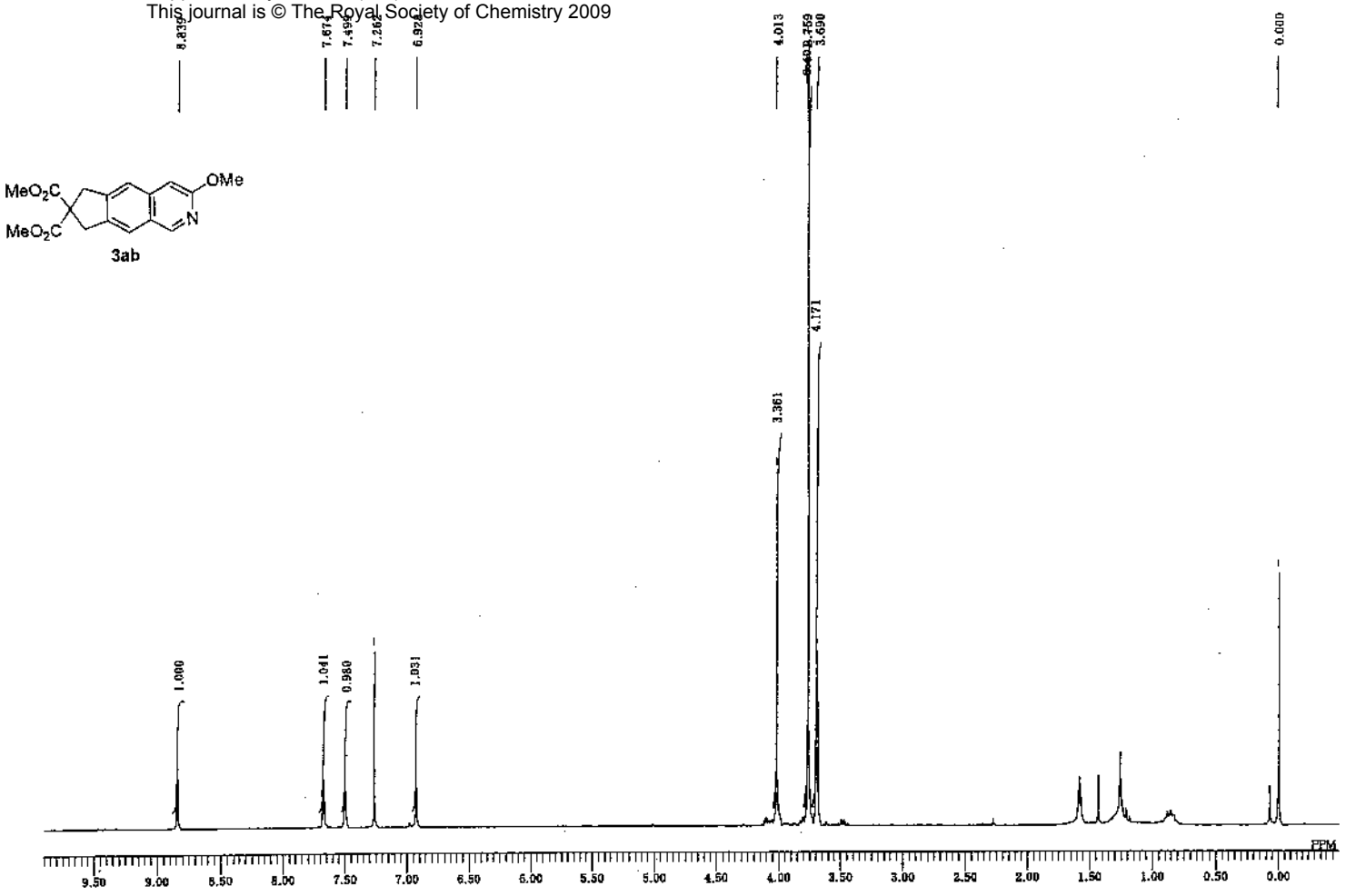

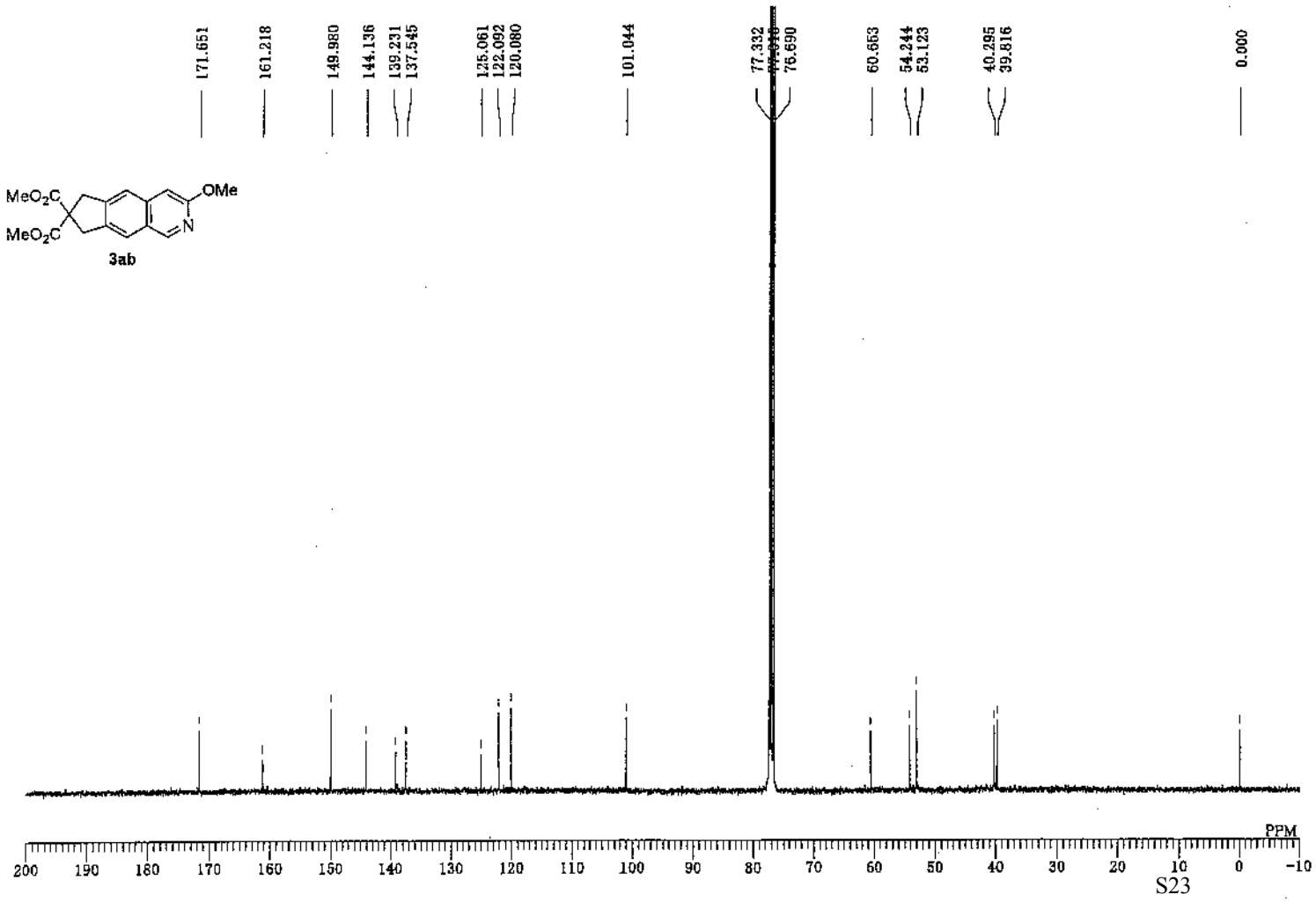

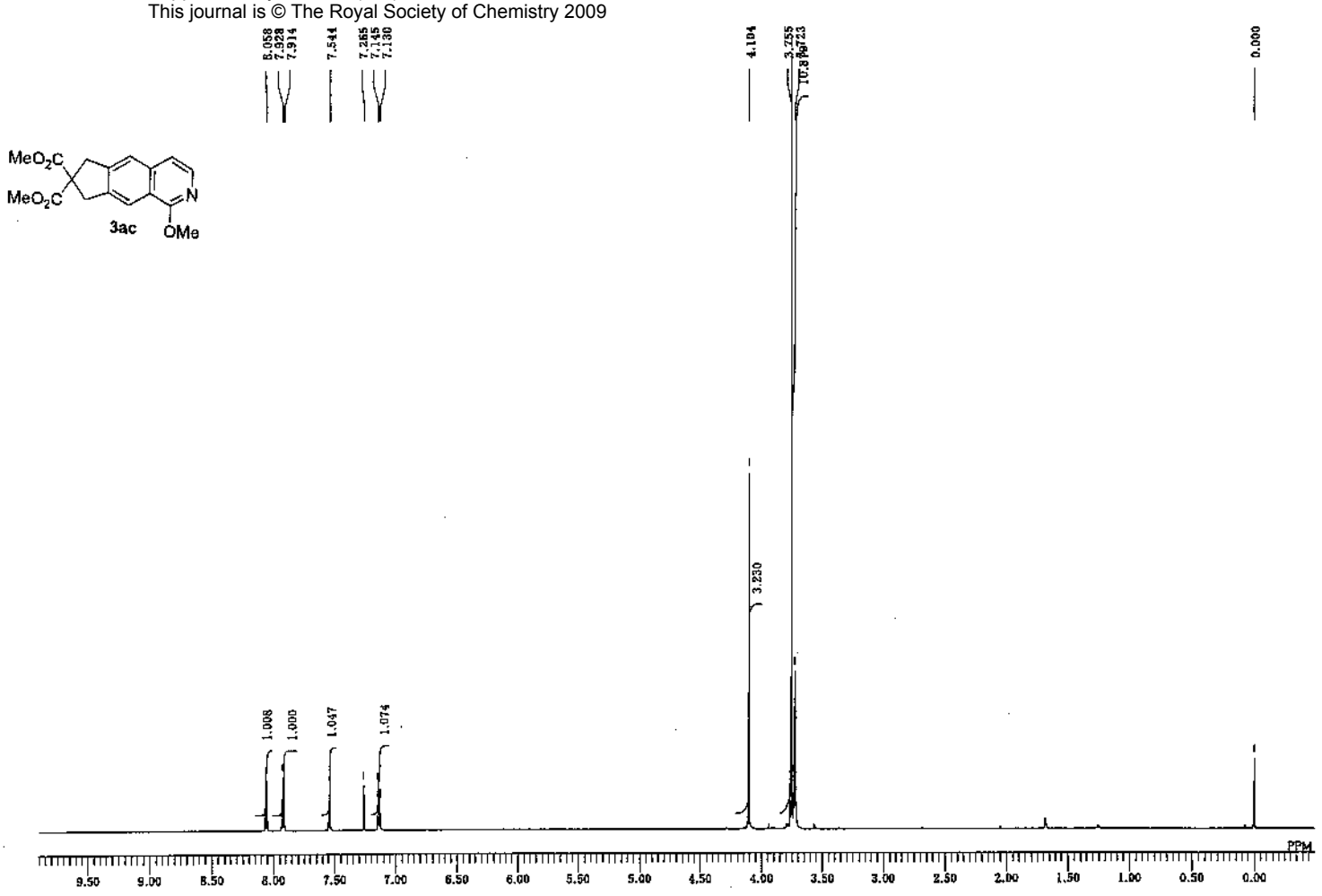
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1.000
1.047
1.074
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MeO2C
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3ac
OMe
PPM
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6.50
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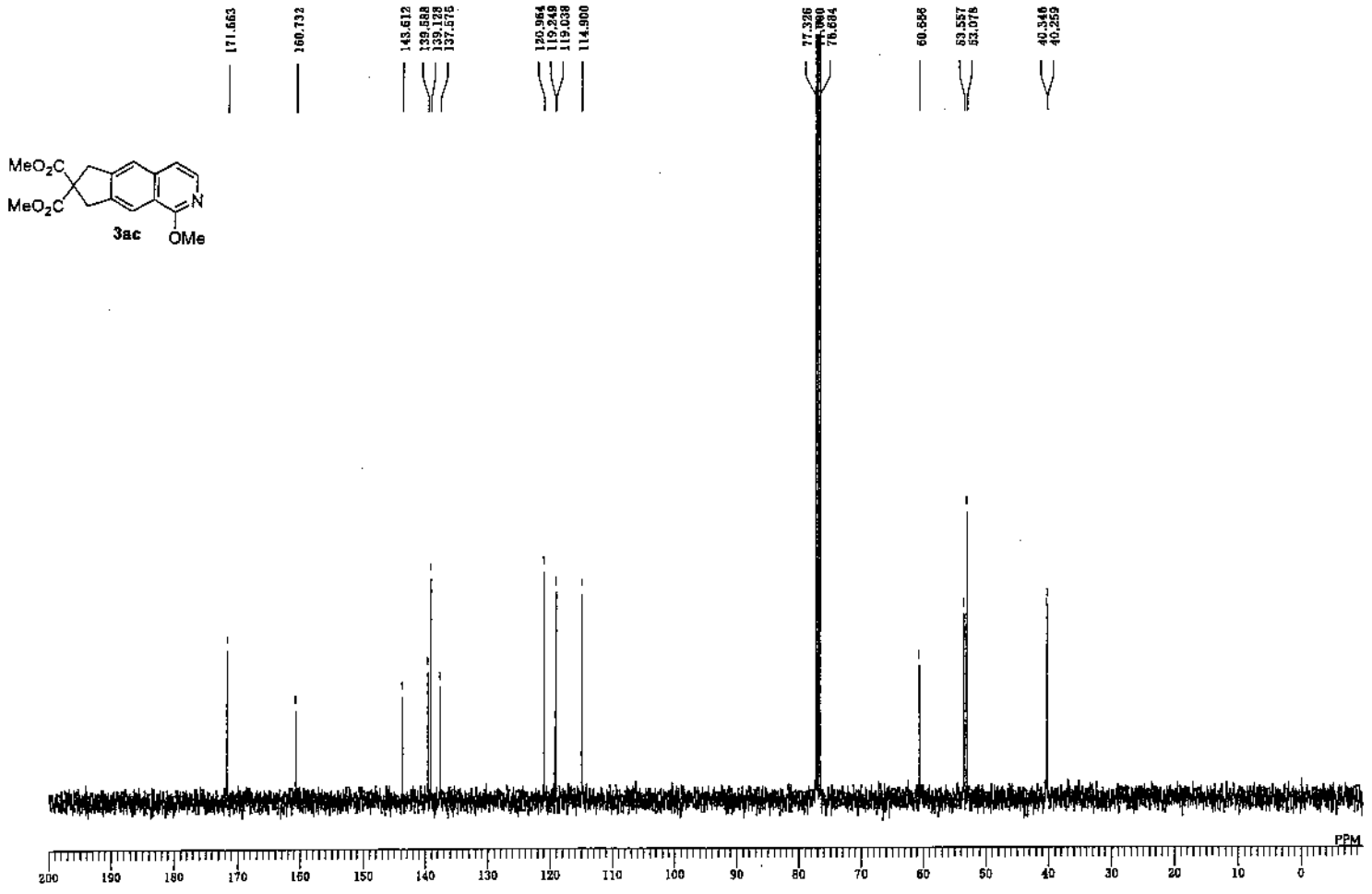
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N
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OMe
PPM
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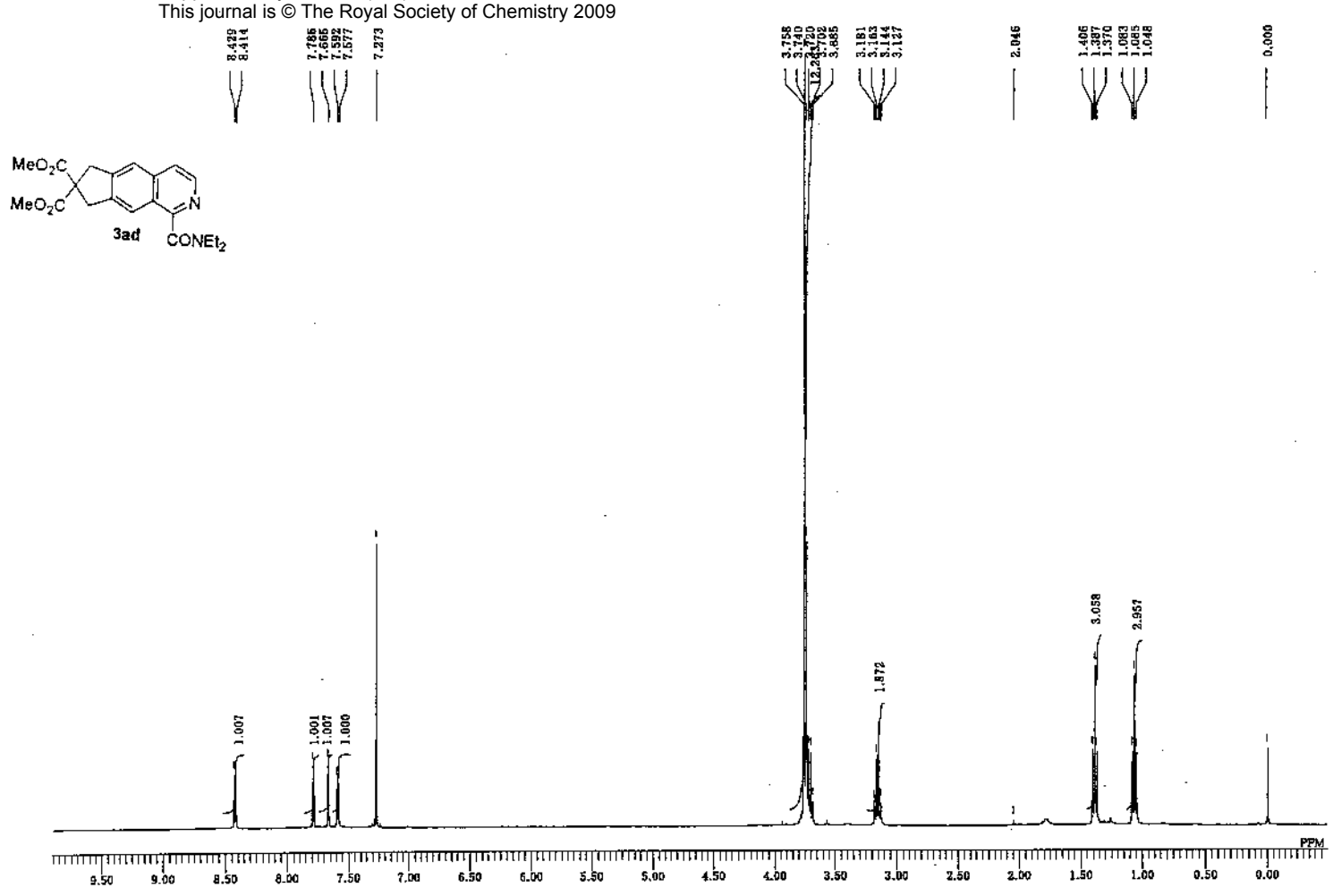
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1.063
1.065
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0.000
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MeO2C
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3ad
CONEt2
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1.007
1.000
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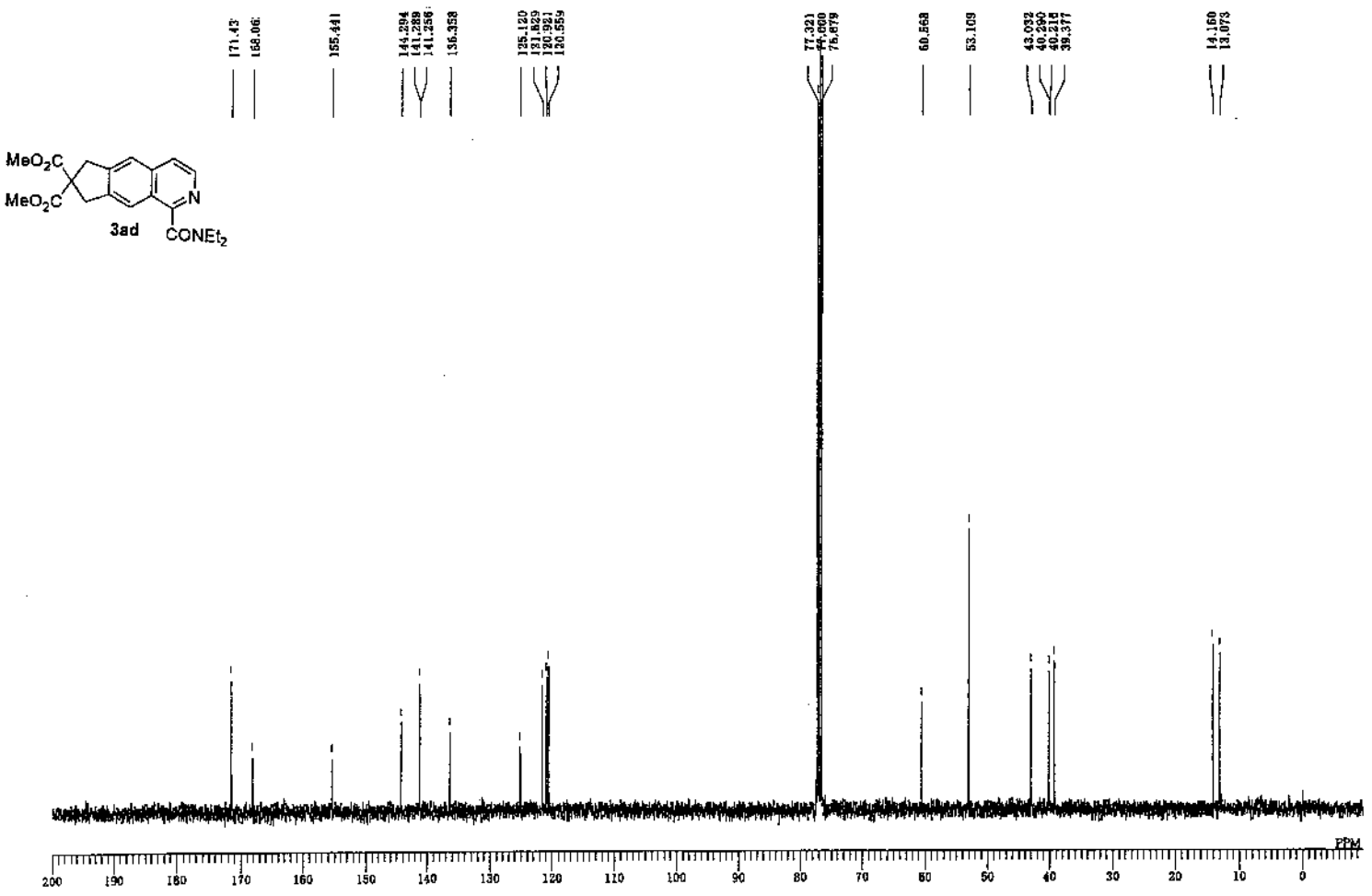
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MeO2C
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PPM
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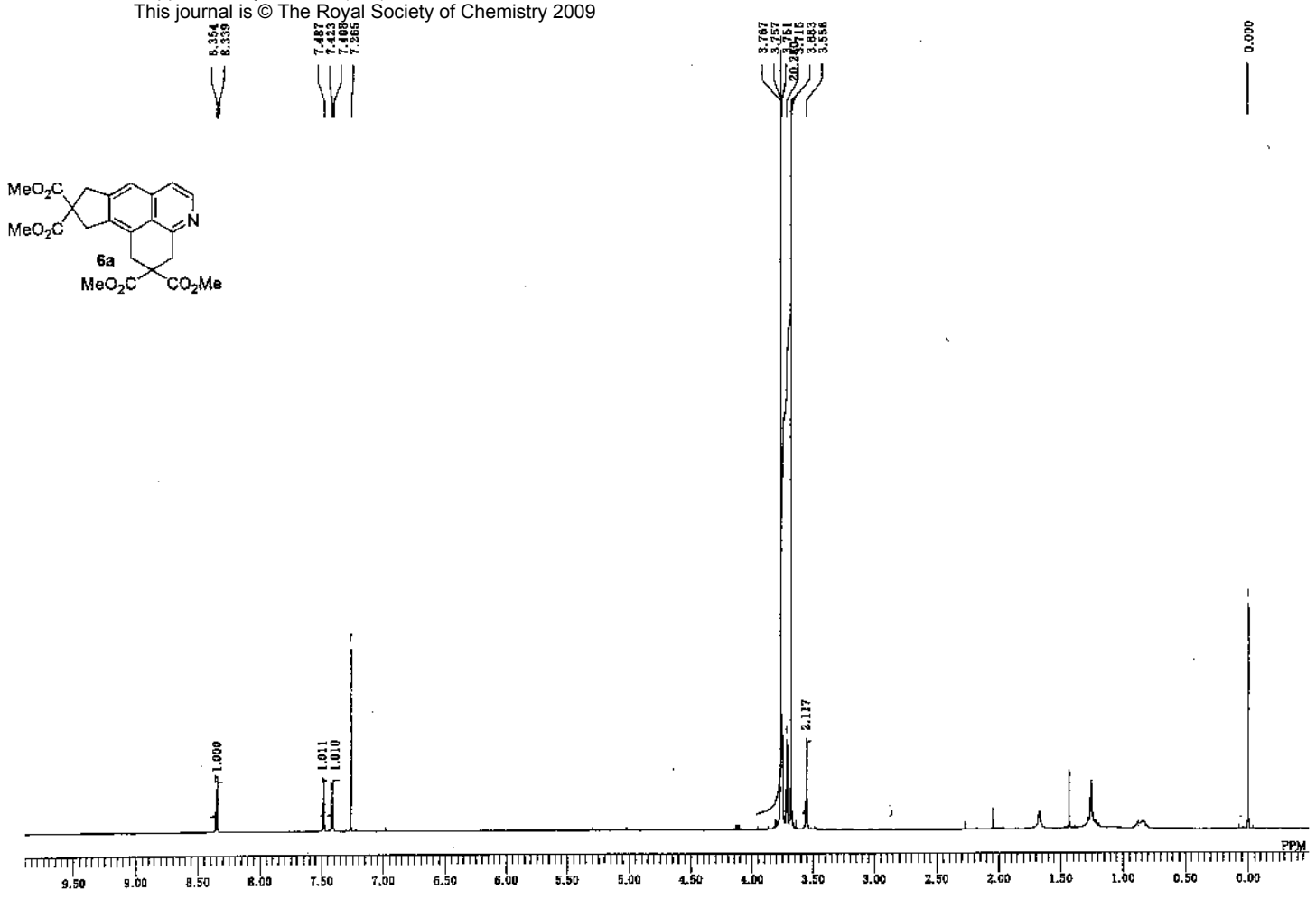
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2.117
1.000
1.011
1.010
PPM
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8.50
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7.00
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6.00
5.50
5.00
4.50
4.00
3.50
3.00
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1.00
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0.00

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MeO2C
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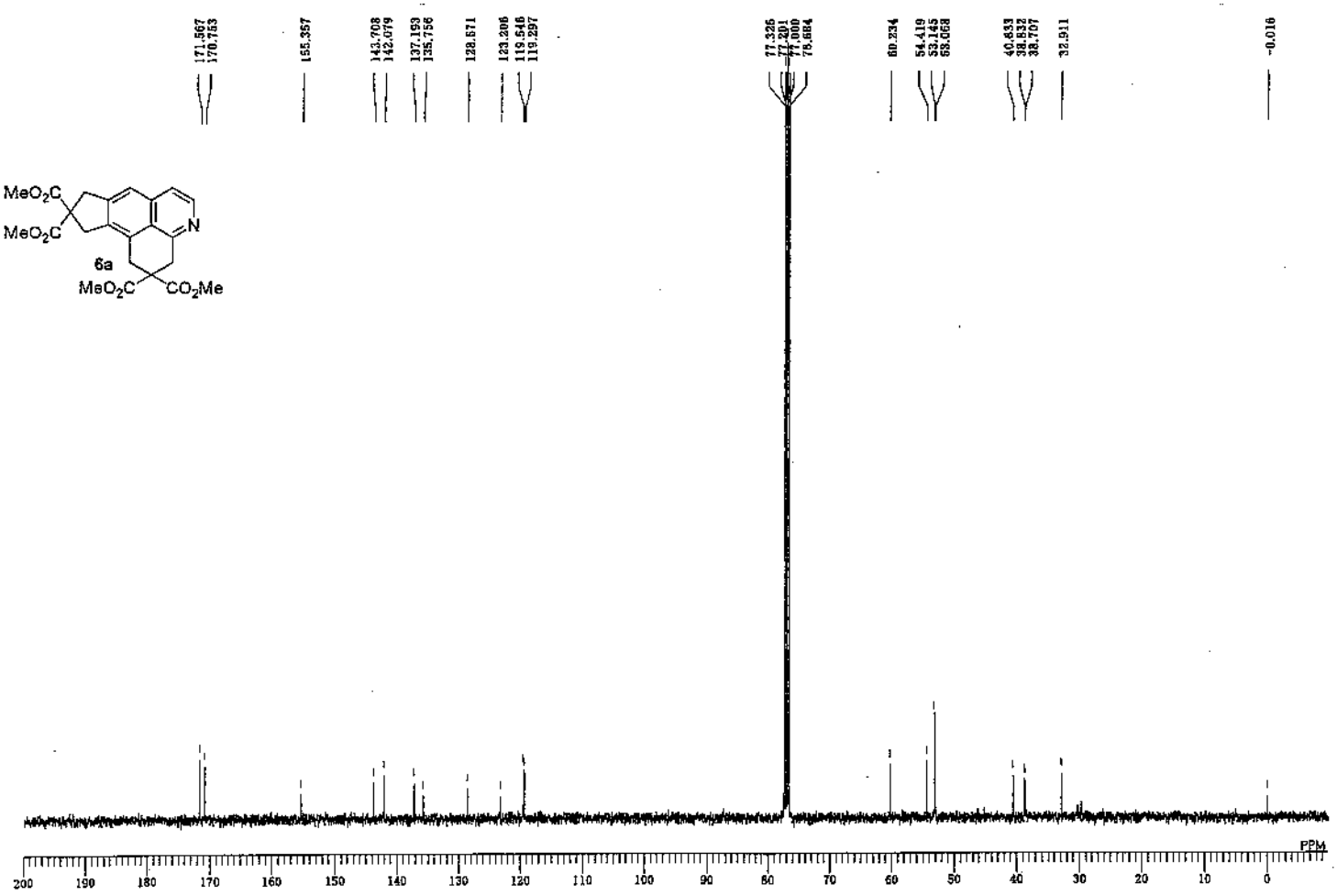
6a
MeO2C
CO2Me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MeO2C
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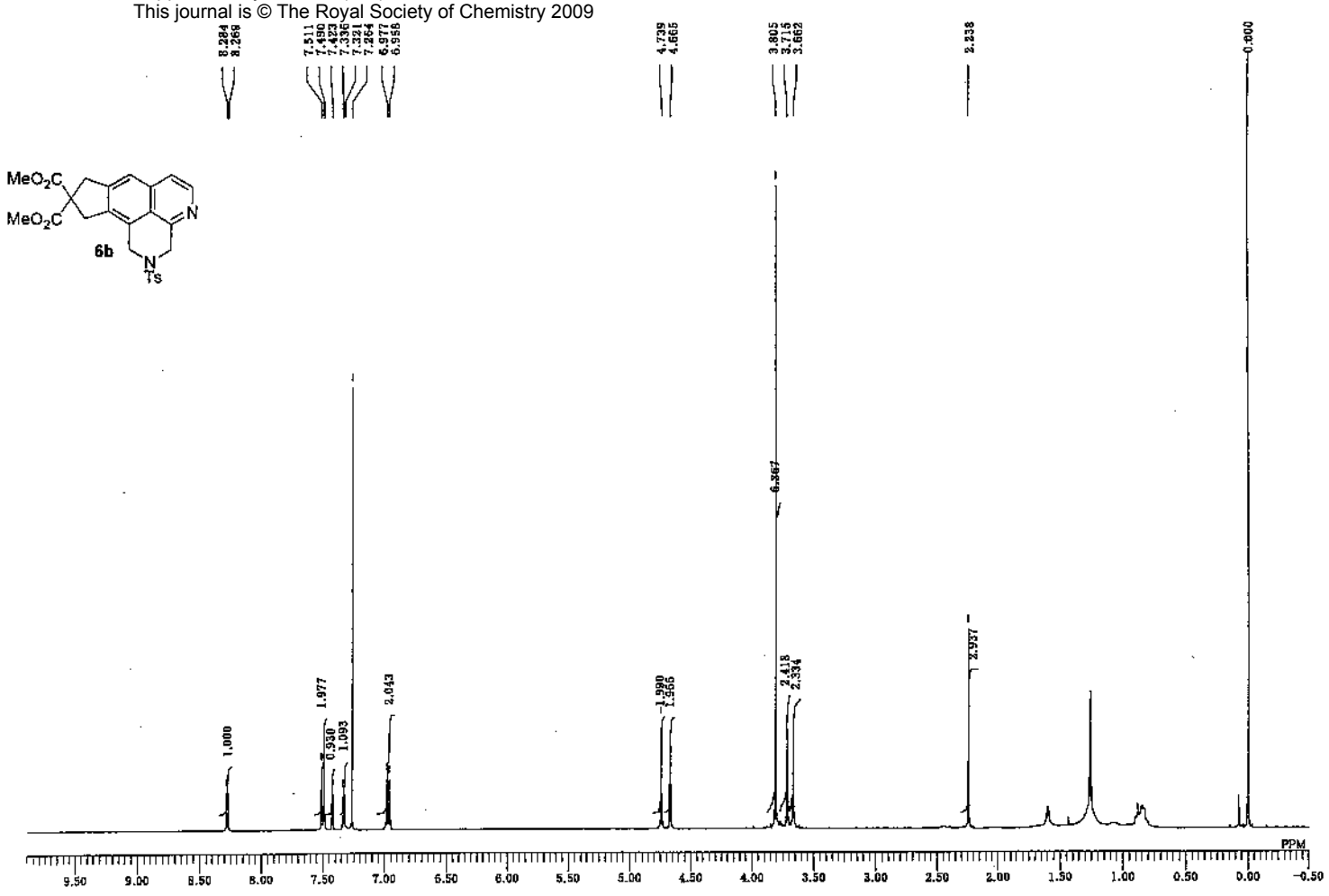
MeO2C
CO2Me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3.715
3.662
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MeO2C
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6b
N
Ts
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1.000
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7.50
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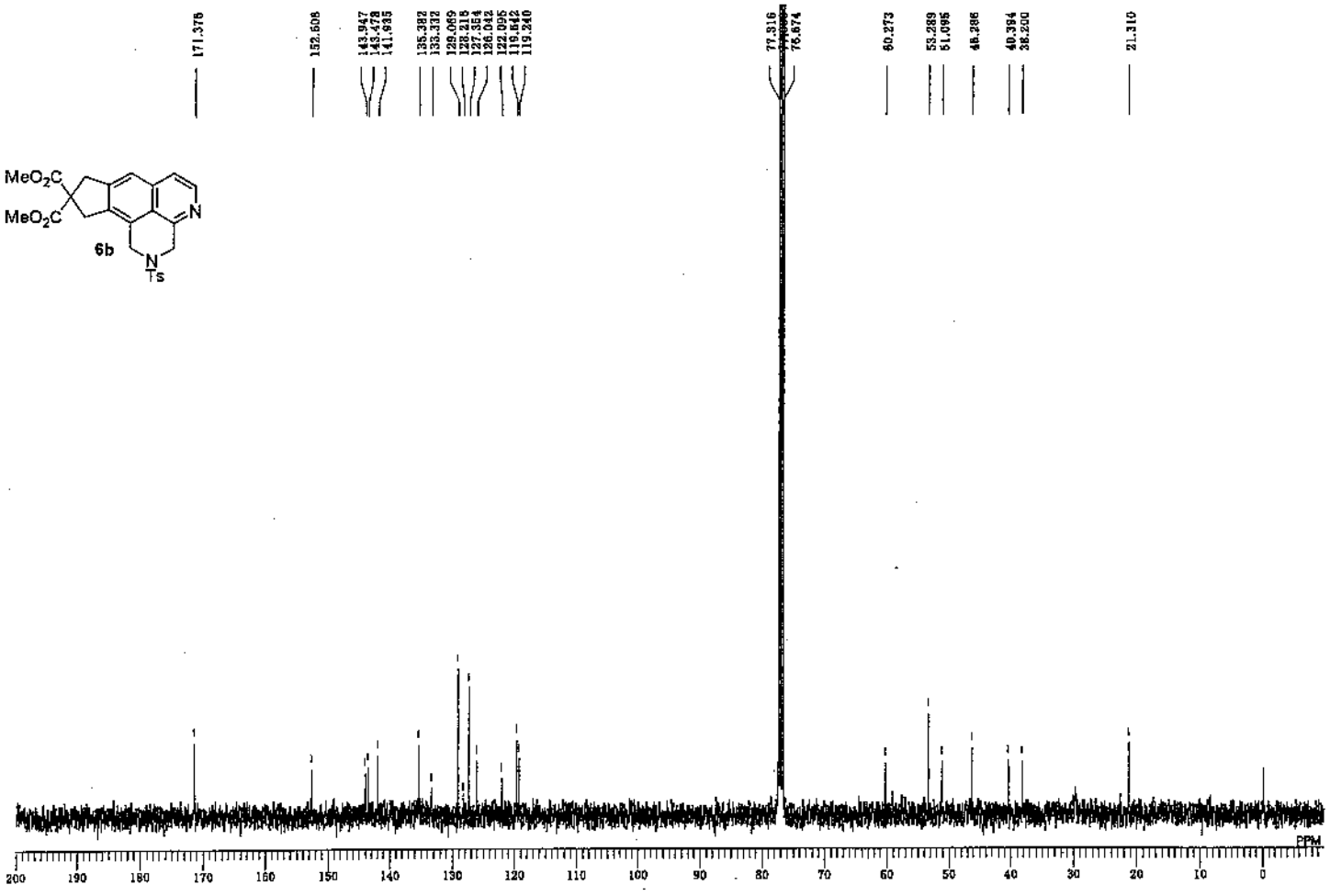
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MeO2C
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6b
N
Ts
PPM
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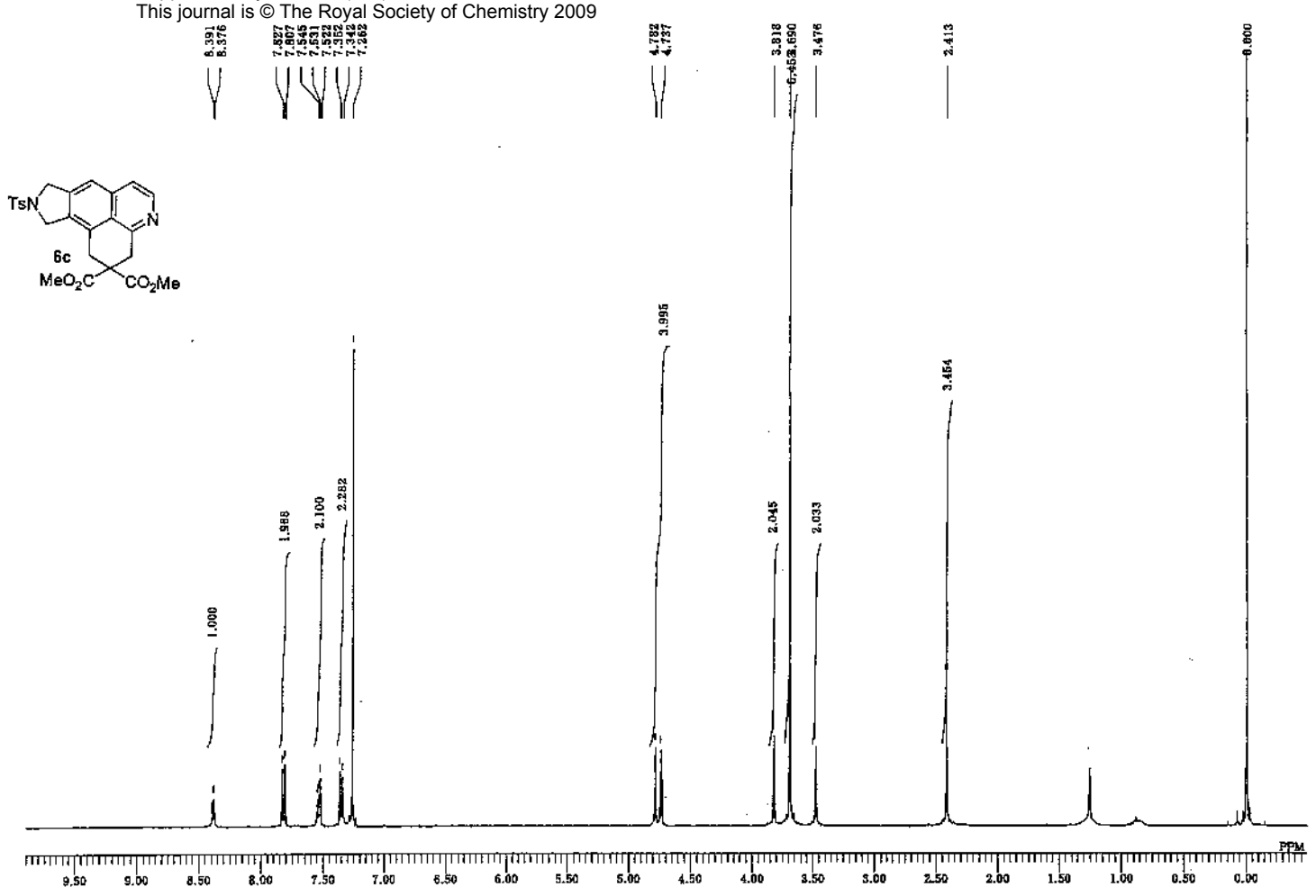
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CO2Me
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1.000
PPM
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9.00
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8.00
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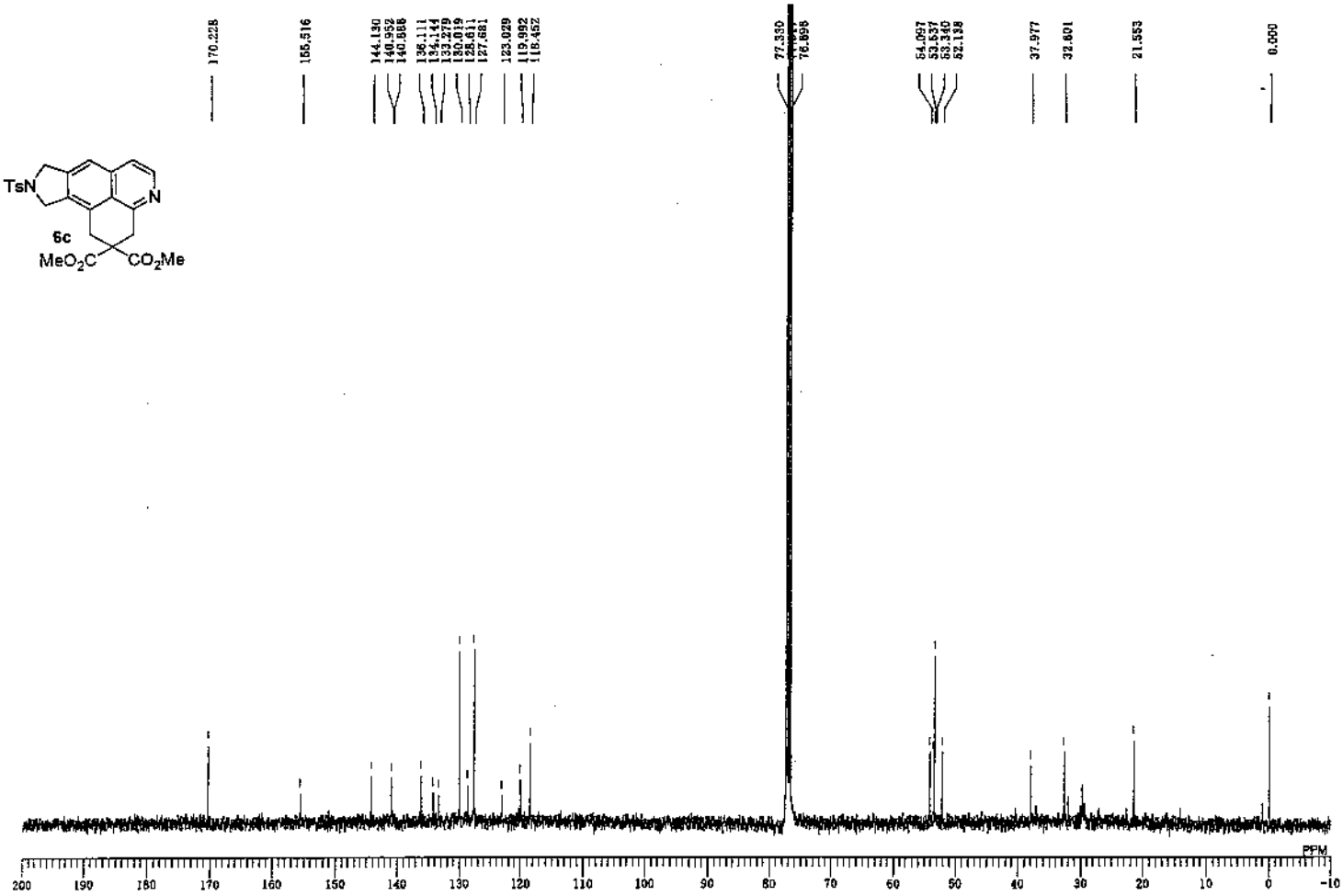
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21.553
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6c
MeO2C
CO2Me
PPM
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190
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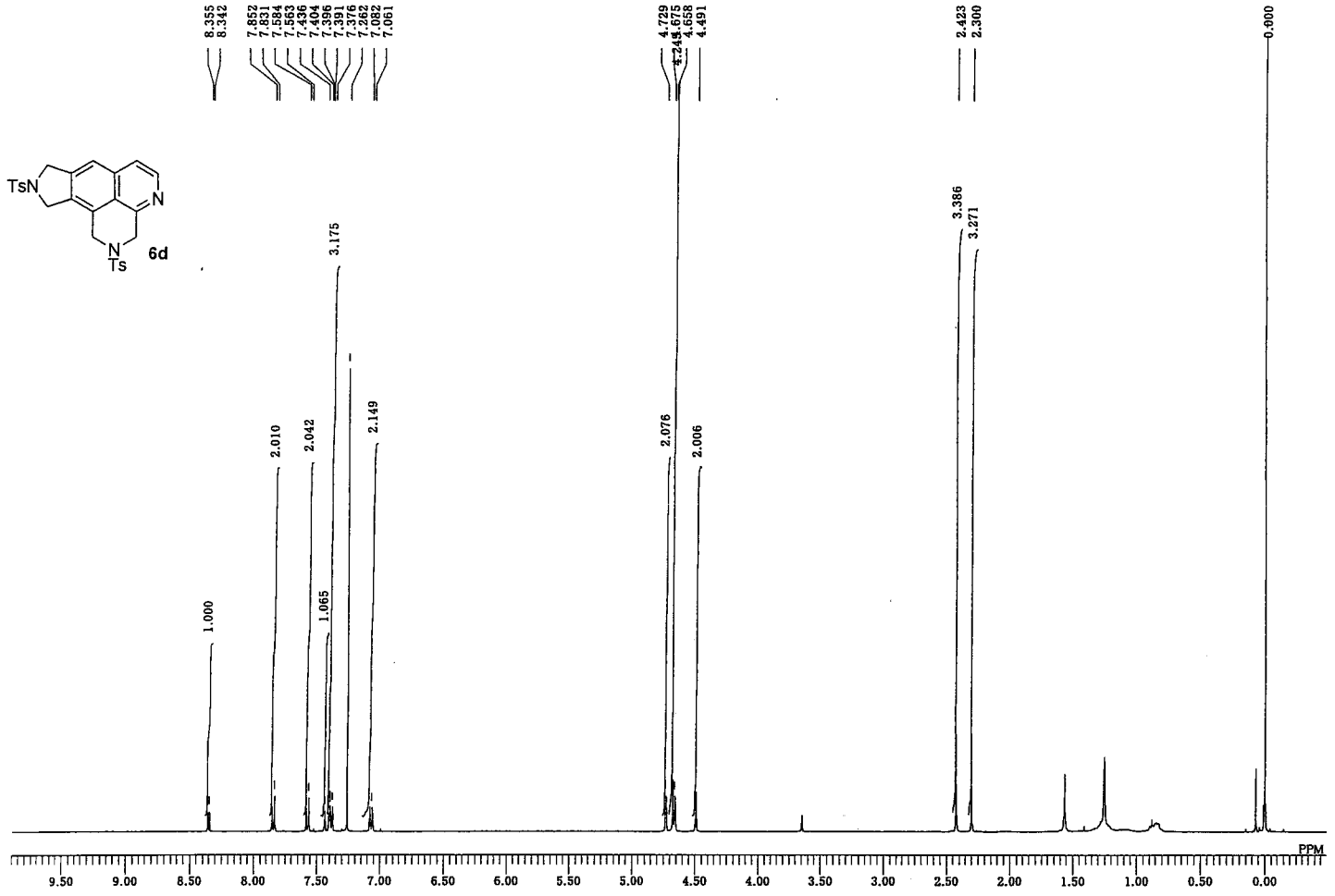
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3.271
3.175
2.149
2.076
2.042
2.010
2.006
1.065
1.000
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N
N
Ts
6d
PPM
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8.00
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6.50
6.00
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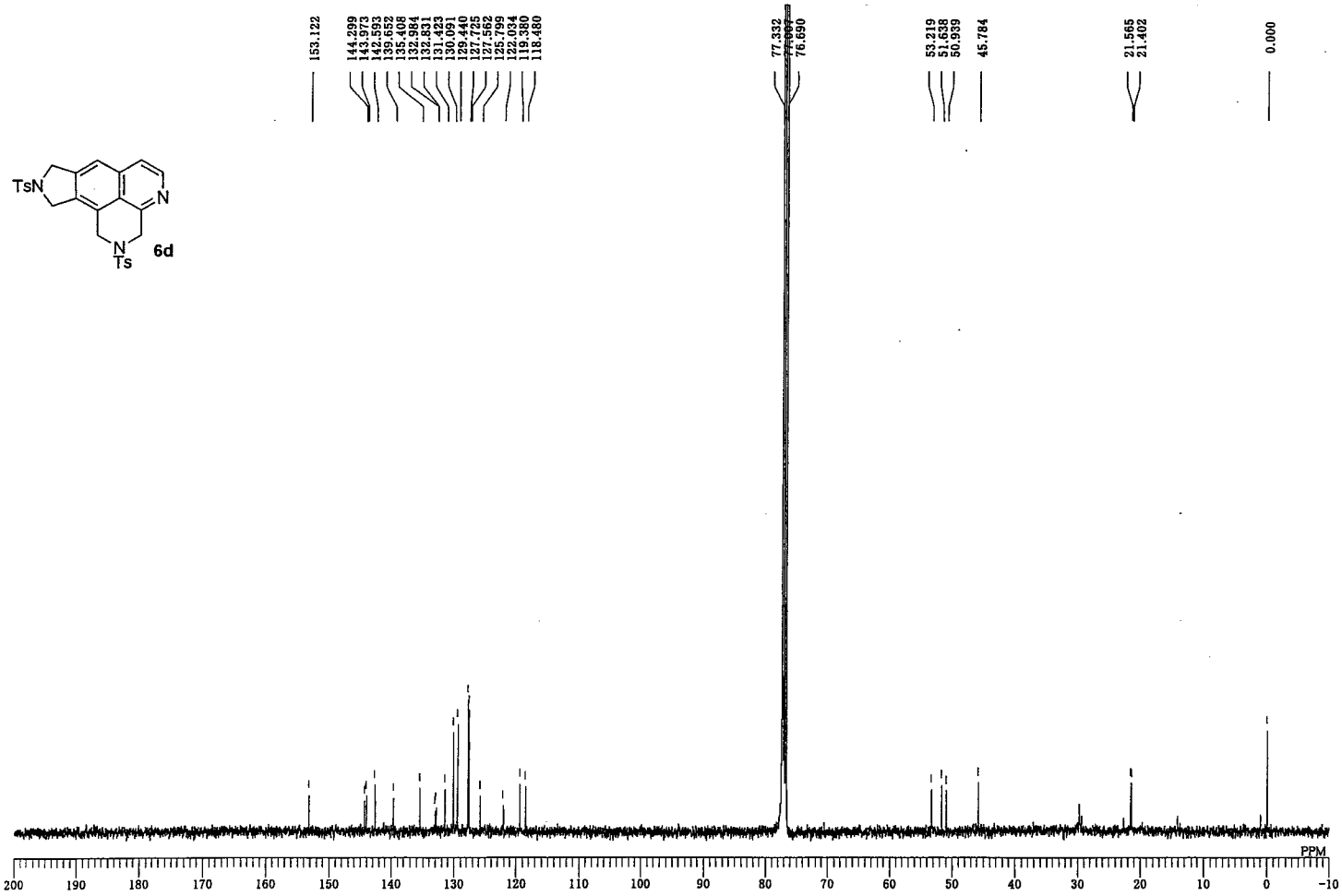
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Ts
6d
PPM
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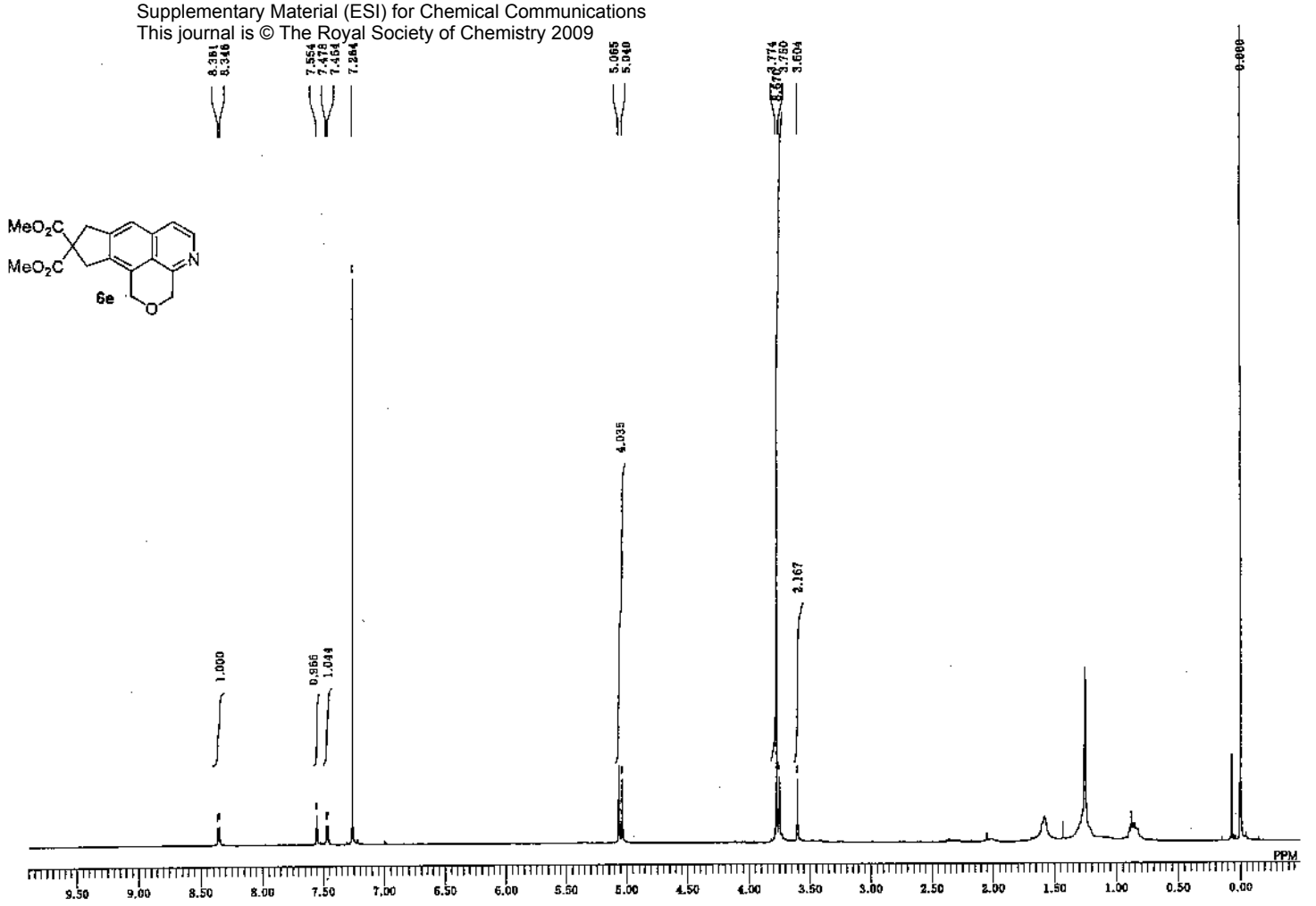
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MeO2C
N
O
6e
PPM
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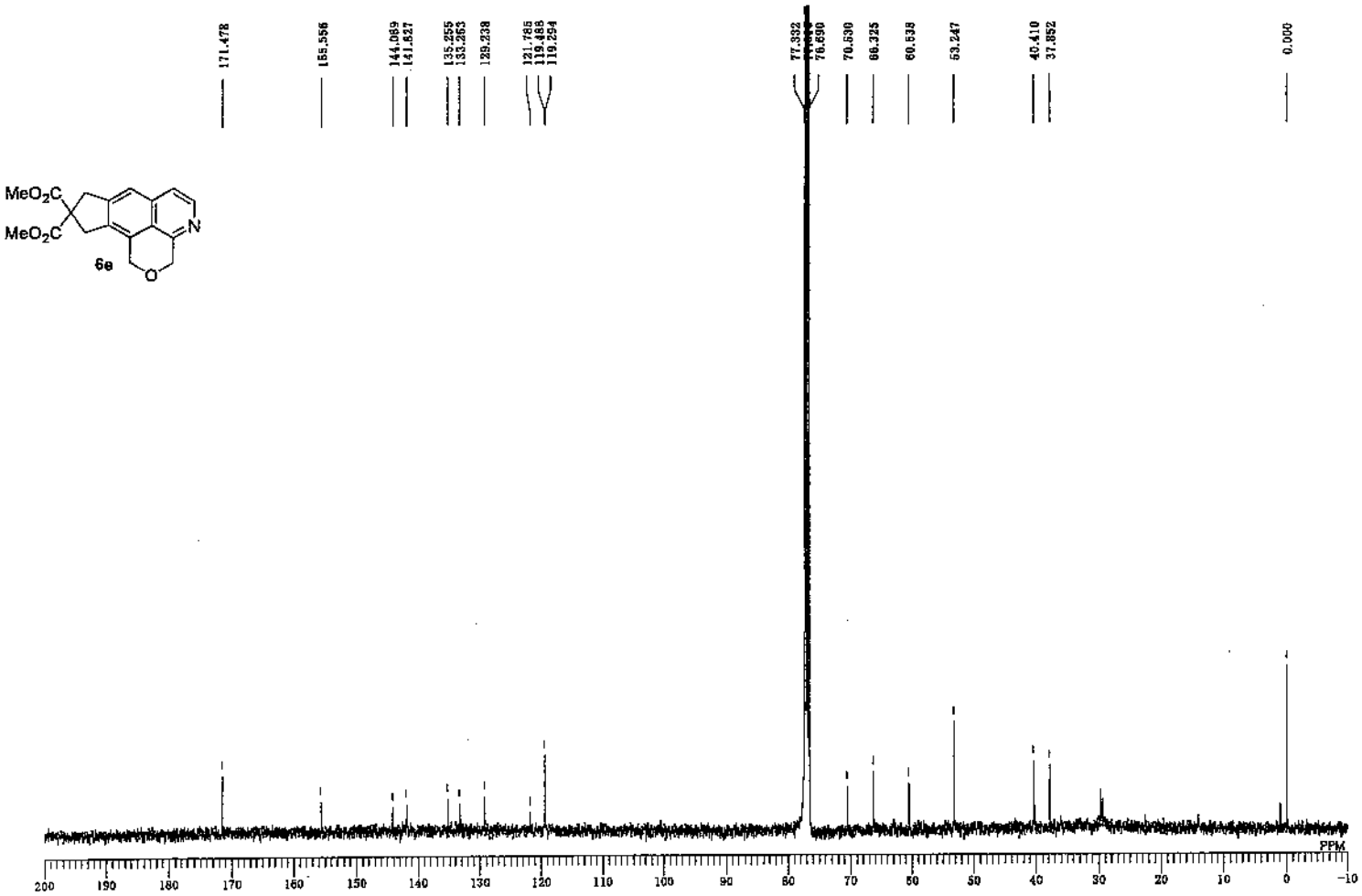
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MeO2C
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6e
PPM
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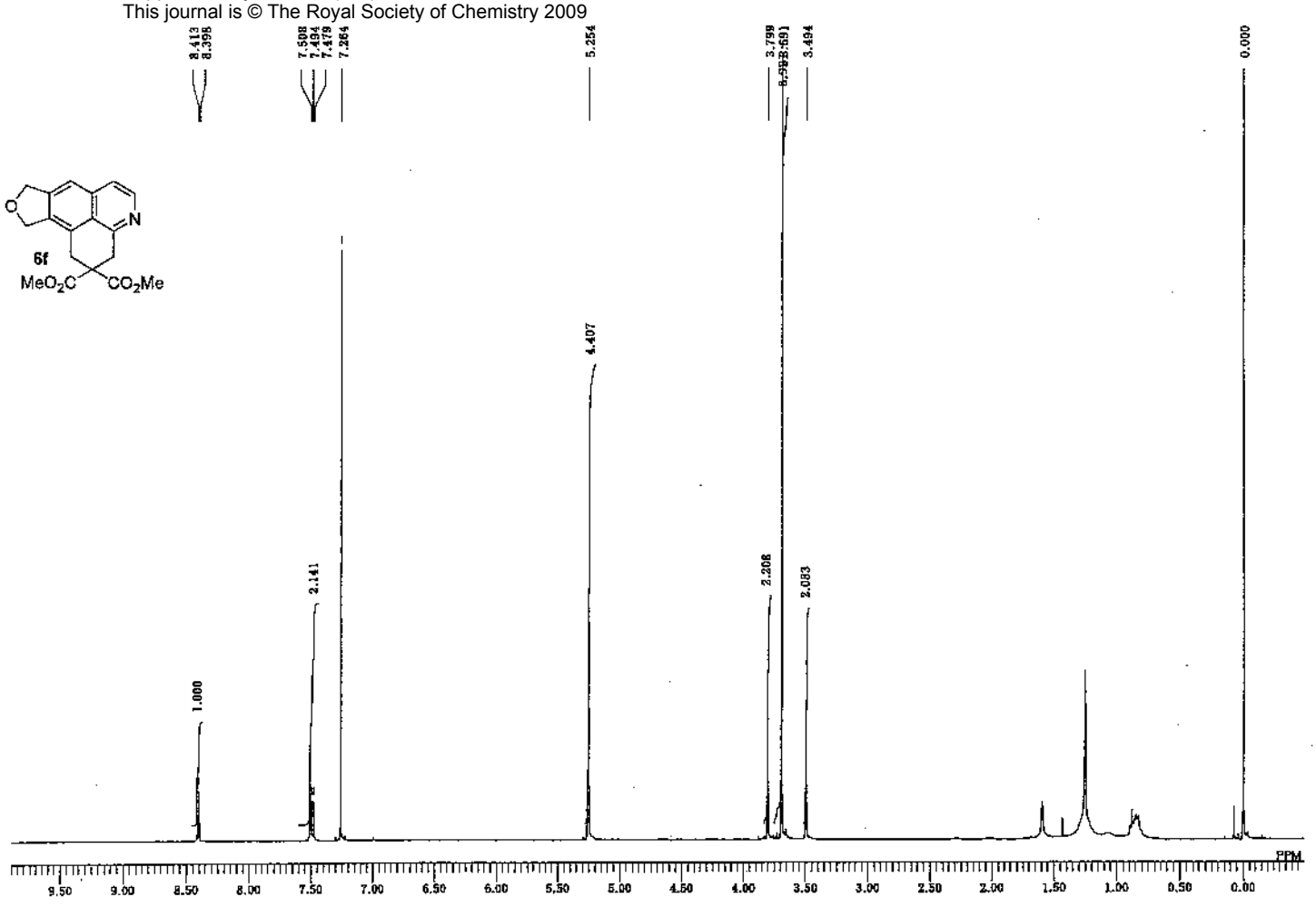
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CO2Me
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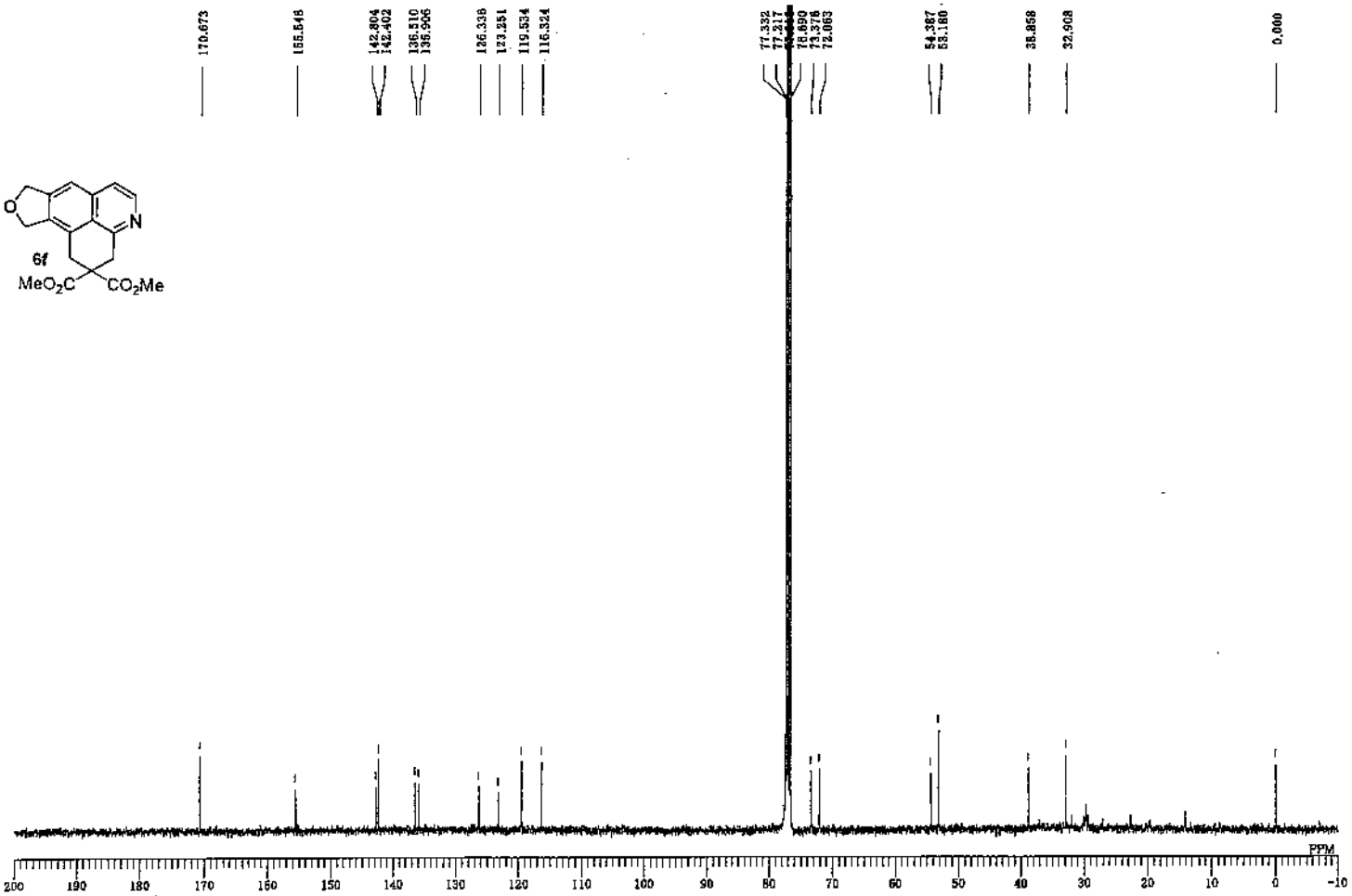
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6f
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CO2Me
PPM