Electronic Supplementary Information

Synthesis of Phenanthridinones via Palladium-Catalyzed C(sp^2)−H Aminocarbonylation of Unprotected *o*-Arylanilines

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I. General Information

Substrates **1** were synthesized according to literature method.[1] Dioxane was distilled before use. Reactions were monitored using thin-layer chromatography (TLC) on commercial silica gel plates (GF 254). Visualization of the developed plates was performed under UV lights (254 nm). Flash column chromatography was performed on silica gel (200-300 mesh). ^{1}H and ^{13}C NMR spectra were recorded on a 400 or 500 MHz spectrometer. Chemical shifts (δ) were reported in ppm referenced to an internal tetramethylsilane standard or the DMSO-d_6 residual peak (δ 2.50) for ^{1}H NMR. Chemical shifts of ^{13}C NMR were reported relative to $CDCl_3$ (δ 77.0) or DMSO-d_6 (δ 39.5). The following abbreviations were used to describe peak splitting patterns when appropriate: br s = broad singlet, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. Coupling constant, *J*, was reported in Hertz unit (Hz). Infrared (IR) spectra were recorded using a potassium bromide pellet. Frequencies were given in reciprocal centimeters (cm^{-1}) and only selected absorbance was reported. High resolution mass spectra (HRMS) were obtained on an ESI-LC-MS/MS spectrometer.

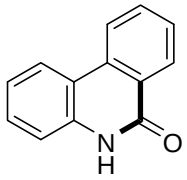
II. General Procedure and Product Characterization

General procedure

A mixture of *o*-arylanilines **1** (0.2 mmol), $Pd(MeCN)_2Cl_2$ (2.6 mg, 5.0 mol %), $Cu(TFA)_2$ (58 mg, 1.0 equiv), TFA (15 μL, 1.0 equiv) in 1,4-dioxane (1.0 mL) was stirred at 110 ^{o}C for 12-65 h under balloon pressure of CO. The reaction was cooled down to room temperature after complete consumption of **1** as monitored by TLC analysis. EtOAc (20 mL) and H_2O (20 mL) were added to the reaction mixture successively. The organic phase was separated, and the aqueous phase was further extracted with EtOAc (2 × 10 mL). The combined organic phases were washed with brine and dried over Na_2SO_4. The concentrated residue was purified by column chromatography over silica gel using petroleum ether/ethyl acetate as eluent to give the desired product **2.**

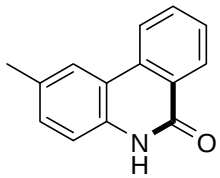
Product Characterization

phenanthridin-6(5*H*)-one (2a)[2]



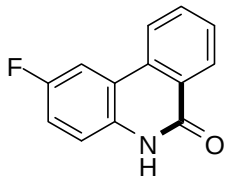
Pale yellow solid, 35 mg, 90% yield. ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.68 (br s, 1H), 8.50 (d, *J* = 8.0 Hz, 1H), 8.38 (d, *J* = 8.0 Hz, 1H), 8.32 (d, *J* = 8.0 Hz, 1H), 7.87-7.83 (m, 1H), 7.64 (t, *J* = 7.2 Hz, 1H), 7.49 (t, *J* = 7.6 Hz, 1H), 7.37 (d, *J* = 8.0 Hz, 1H), 7.26 (d, *J* = 7.2 Hz, 1H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 160.7, 136.4, 134.1, 132.6, 129.3, 127.7, 127.3, 125.6, 123.0, 122.4, 122.1, 117.4, 116.0; IR (KBr) ν 3457, 1665, 1609, 1512, 1470, 1424, 1370, 1154, 785, 751, 728, 670, 624 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{13}H_{10}NO$ $[M+H]^+$ 196.0757, found 196.0758.

2-methylphenanthridin-6(5*H*)-one (2b)[3]



Gray white powder, 35 mg, 91% yield. ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.60 (br s, 1H), 8.49 (d, *J* = 8.0 Hz, 1H), 8.31 (dd, *J* = 8.0, 0.8 Hz, 1H), 8.20 (s, 1H), 7.86-7.82 (m, 1H), 7.62 (t, *J* = 7.6 Hz, 1H), 7.32-7.24 (m, 2H), 2.41 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 160.6, 134.4, 134.2, 132.6, 131.2, 130.5, 127.7, 127.4, 125.7, 123.0, 122.5, 117.4, 116.0, 20.7; IR (KBr) ν 3449, 2878, 1690, 1507, 1269, 1184, 1152, 815, 769, 655 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{14}H_{12}NO$ $[M+H]^+$ 210.0913, found 210.0912.

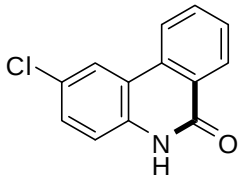
2-fluorophenanthridin-6(5*H*)-one (2c)[3]



Pale yellow solid, 31 mg, 73% yield. ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.74 (br s, 1H), 8.52 (d, *J* = 8.0 Hz, 1H), 8.32 (dd, *J* = 8.0, 1.2 Hz, 1H), 8.27 (d, *J* = 9.2 Hz, 1H), 7.88-7.84 (m, 1H), 7.68 (t, *J* = 7.6 Hz, 1H), 7.38 (d, *J* = 1.2 Hz, 1H), 7.37 (s, 1H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 160.5, 158.7, 156.8, 133.5, 133.1, 132.8, 127.4, 125.8,

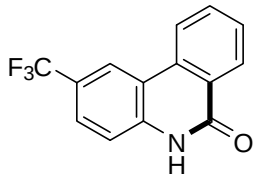
123.2, 118.84, 118.78, 117.8, 117.7, 117.2, 117.0, 109.2, 109.0; IR (KBr) ν 3415, 2878, 1690, 1507, 1369, 1269, 1151, 815, 654 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{13}H_9FNO$ $[M+H]^+$ 214.0663, found 214.0663.

2-chlorophenanthridin-6(5*H*)-one (2d)[3]



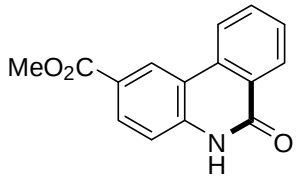
White solid, 37 mg, 80% yield. ^1H NMR (400 MHz, DMSO-d_6): δ 11.80 (br s, 1H), 8.55 (d, *J* = 8.0 Hz, 1H), 8.46 (d, *J* = 2.4 Hz, 1H), 8.32 (d, *J* = 7.2 Hz, 1H), 7.88-7.84 (m, 1H), 7.68 (t, *J* = 7.6 Hz, 1H), 7.53 (dd, *J* = 8.8, 2.0 Hz, 1H), 7.37 (d, *J* = 8.8 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 160.6, 135.3, 133.2, 133.0, 129.4, 128.7, 127.5, 126.6, 125.8, 123.1, 122.8, 119.2, 117.9.; IR (KBr) ν 3419, 1690, 1497, 1364, 819, 773 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{13}H_9ClNO$ $[M+H]^+$ 230.0367, found 230.0368.

2-(trifluoromethyl)phenanthridin-6(5*H*)-one (2e)[4]



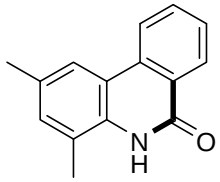
Gray white solid, 30 mg, 57% yield. ^1H NMR (400 MHz, DMSO-d_6): δ 12.01 (br s, 1H), 8.72 (s, 1H), 8.67 (d, *J* = 8.0 Hz, 1H), 8.34 (d, *J* = 7.6 Hz, 1H), 7.91-7.87 (m, 1H), 7.81 (dd, *J* = 8.1, 1.2 Hz, 1H), 7.71 (t, *J* = 7.6 Hz, 1H), 7.52 (d, *J* = 8.4 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 160.9, 139.3, 133.3, 133.1, 128.8, 127.5, 125.93, 125.90, 125.8, 125.6, 123.4, 123.2, 122.9, 122.6, 120.89, 120.85, 117.7, 116.9; IR (KBr) ν 3416, 1691, 1360, 1329, 1273, 1123, 777, 641 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{14}H_9F_3NO$ $[M+H]^+$ 264.0631, found 264.0629.

methyl 6-oxo-5,6-dihydrophenanthridine-2-carboxylate (2f)[5]



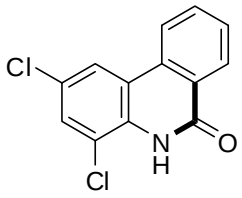
Pale yellow solid, 37 mg, 61% yield. ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.86 (br s, 1H), 8.55 (d, *J* = 8.0 Hz, 1H), 8.51 (d, *J* = 8.4 Hz, 1H), 8.34 (d, *J* = 8.0 Hz, 1H), 7.98 (d, *J* = 1.6 Hz, 1H), 7.98-7.88 (m, 1H), 7.77 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.12 (t, *J* = 7.6 Hz, 1H), 3.89 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 165.5, 160.5, 136.4, 133.1, 132.8, 129.9, 128.8, 127.4, 126.2, 123.5, 123.1, 122.0, 121.2, 116.9, 52.1; IR (KBr) ν 3417, 2956, 1727, 1679, 1608, 1393, 1357, 1283, 1222, 1103, 894, 749 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{15}H_{12}NO_3$ $[M+H]^+$ 254.0812, found 254.0810.

2,4-dimethylphenanthridin-6(5*H*)-one (2g)[6]



Pale yellow solid, 31 mg, 70% yield. ^{1}H NMR (400 MHz, DMSO-d_6): δ 10.62 (br s, 1H), 8.47 (d, *J* = 8.0 Hz, 1H), 8.32 (d, *J* = 7.6 Hz, 1H), 8.06 (s, 1H), 7.83 (t, *J* = 7.6 Hz, 1H), 7.62 (t, *J* = 7.2 Hz, 1H), 7.17 (s, 1H), 2.43 (s, 3H), 2.37 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 161.1, 134.5, 132.8, 132.6, 132.2, 130.9, 127.7, 127.5, 125.4, 124.0, 122.7, 120.9, 117.5, 20.6, 17.5; IR (KBr) ν 3416, 1655, 1340, 1183, 772 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{15}H_{14}NO$ $[M+H]^+$ 224.1070, found 224.1068.

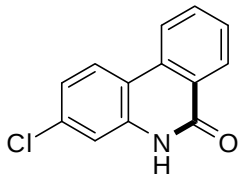
2,4-dichlorophenanthridin-6(5*H*)-one (2h)



White solid, 34 mg, 64% yield. ^{1}H NMR (400 MHz, DMSO-d_6): δ 10.94 (br s, 1H), 8.60 (d, *J* = 7.6 Hz, 1H), 8.52 (d, *J* = 2.0 Hz, 1H), 8.35 (dd, *J* = 8.0, 0.8 Hz, 1H), 7.92-7.88 (m, 1H), 7.79 (d, *J* = 2.0 Hz, 1H), 7.34 (t, *J* = 7.6 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 160.7, 133.5, 132.7, 132.0, 129.5, 129.1, 127.6, 126.6, 125.8,

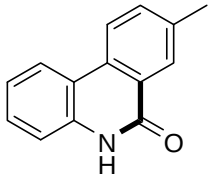
123.6, 122.3, 120.7, 120.3; IR (KBr) ν 3422, 1639, 1025, 667 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{13}H_8Cl_2NO$ $[M+H]^+$ 263.9977, found 263.9977.

3-chlorophenanthridin-6(5*H*)-one (2i)[7]



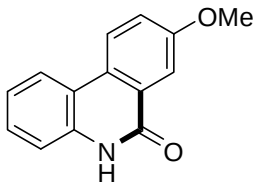
Pale yellow solid, 29 mg, 63% yield. ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.78 (br s, 1H), 8.49 (d, *J* = 8.4 Hz, 1H), 8.41 (d, *J* = 8.8 Hz, 1H), 8.31 (d, *J* = 8.0 Hz, 1H), 7.89-7.85 (m, 1H), 7.66 (t, *J* = 7.2 Hz, 1H), 7.39 (d, *J* = 2.0 Hz, 1H), 7.29 (dd, *J* = 8.8, 2.0 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 160.8, 137.6, 133.7, 133.5, 133.0, 128.3, 127.5, 125.5, 125.3, 122.8, 122.1, 116.5, 115.3; IR (KBr) ν 3418, 1666, 1610, 1357, 762 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{13}H_9ClNO$ $[M+H]^+$ 230.0367, found 230.0369.

8-methylphenanthridin-6(5*H*)-one (2j)[8]



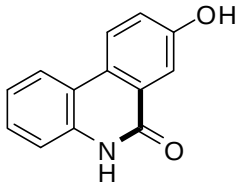
Pray white solid, 38 mg, 91% yield. ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.62 (br s, 1H), 8.39 (d, *J* = 8.0 Hz, 1H), 8.33 (d, *J* = 8.0 Hz, 1H), 8.12 (s, 1H), 7.67 (d, *J* = 8.0 Hz, 1H), 7.45 (t, *J* = 7.2 Hz, 1H), 7.34 (d, *J* = 7.6 Hz, 1H), 7.24 (t, *J* = 7.2 Hz, 1H), 2.48 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 160.8, 137.5, 136.2, 133.9, 131.8, 129.0, 127.2, 125.6, 122.9, 122.6, 122.1, 117.6, 116.0, 20.9; IR (KBr) ν 3419, 1676, 1358, 747 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{14}H_{12}NO$ $[M+H]^+$ 210.0913, found 210.0912.

8-methoxyphenanthridin-6(5*H*)-one (2k)[3]



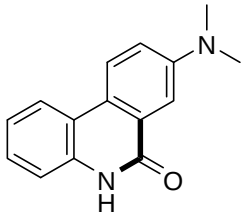
Pale yellow solid, 41 mg, 91% yield. ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.7 (br s, 1H), 8.43 (d, *J* = 9.2 Hz, 1H), 8.29 (d, *J* = 8.0 Hz, 1H), 7.75 (d, *J* = 2.8 Hz, 1H), 7.45-7.40 (m, 2H), 7.34 (d, *J* = 8.0 Hz, 1H), 7.25-7.21 (m, 1H), 3.91 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 160.3, 158.9, 135.4, 128.2, 127.5, 127.0, 124.3, 122.4, 122.0, 121.3, 117.6, 115.8, 108.8, 55.3; IR (KBr) ν3415, 1667, 1616, 1486, 1275, 833, 750, 628 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{14}H_{12}NO_2[M+H]^+$ 226.0863, found 226.0862.

8-hydroxyphenanthridin-6(5*H*)-one (2l)[9]



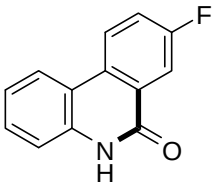
Pale yellow solid, 31 mg, 74% yield. ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.57 (br s, 1H), 10.14 (br s, 1H), 8.33 (d, *J* = 8.8 Hz, 1H), 8.23 (d, *J* = 8.0 Hz, 1H), 7.65 (d, *J* = 2.8 Hz, 1H), 7.38 (t, *J* = 7.2 Hz, 1H), 7.33-7.26 (m, 2H), 7.20 (t, *J* = 7.2 Hz, 1H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 160.4, 157.2, 135.1, 127.7, 127.1, 126.0, 124.2, 122.0, 121.9, 121.5, 117.9, 115.7, 111.5; IR (KBr) ν 3417, 1661, 1479, 1358, 738 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{13}H_{10}NO_2$ $[M+H]^+$ 212.0706, found 212.0702.

8-(dimethylamino)phenanthridin-6(5*H*)-one (2m)



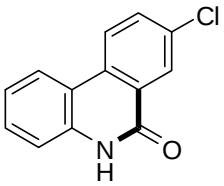
Pale yellow solid, 19 mg, 40% yield. ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.53 (br s, 1H), 8.29 (d, *J* = 8.8 Hz, 1H), 8.20 (d, *J* = 7.6 Hz, 1H), 7.47 (d, *J* = 2.8 Hz, 1H), 7.35-7.28 (m, 3H), 7.21-7.17 (m, 1H), 3.04 (s, 6H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 160.8, 149.7, 134.6, 126.9, 126.6, 123.5, 122.9, 121.8, 121.5, 118.2, 118.0, 115.6, 107.5; IR (KBr) ν 3443, 1653, 1521, 1429, 1373, 1234, 1091, 1002, 818, 750, 630 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{15}H_{15}N_2O$ $[M+H]^+$ 239.1179, found 239.1178.

8-fluorophenanthridin-6(5*H*)-one (2n)[10]



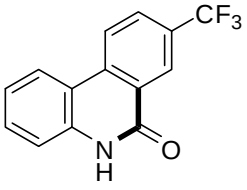
Pale yellow solid, 19 mg, 45% yield. ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.83 (br s, 1H), 8.60 (dd, *J* = 8.8, 4.8 Hz, 1H), 8.37 (d, *J* = 8.0 Hz, 1H), 7.97 (dd, *J* = 9.2, 2.8 Hz, 1H), 7.76-7.71 (m, 1H), 7.49 (t, *J* = 7.6 Hz, 1H), 7.37 (d, *J* = 8.0 Hz, 1H), 7.28 (t, *J* = 7.6 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 162.6, 160.6, 160.0, 136.1, 131.14, 131.12, 129.6, 127.7, 127.6, 126.0, 125.9, 123.3, 122.6, 121.2, 121.0, 117.1, 116.3, 112.6, 112.5; IR (KBr) ν 3413, 1644, 1605, 1183, 744 cm^{-1} HRMS (ESI): Exact mass calcd for $C_{13}H_9FNO$ $[M+H]^+$ 214.0663, found 214.0663.

8-chlorophenanthridin-6(5*H*)-one (2o)[11]



Pale yellow solid, 24 mg, 53% yield. ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.85 (br s, 1H), 8.54 (d, *J* = 8.8 Hz, 1H), 8.37 (d, *J* = 8.0 Hz, 1H), 8.24 (d, *J* = 2.4 Hz, 1H), 7.89 (dd, *J* = 8.8, 2.4 Hz, 1H), 7.53-7.49 (m, 1H), 7.37 (d, *J* = 7.6 Hz, 1H), 7.29-7.25 (m, 1H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 159.5,145.9 136.3, 132.9, 132.5, 129.8, 127.1, 126.4, 124.9, 123.2, 122.3, 116.7, 116.1; IR (KBr) ν 3417, 1675, 1606, 1469, 1360, 1262, 818, 740, 628 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{13}H_9ClNO$ $[M+H]^+$ 230.0367, found 230.0366.

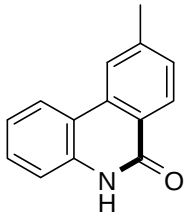
8-(trifluoromethyl)phenanthridin-6(5*H*)-one (2p)[12]



Brown solid, 20 mg, 38% yield. ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.96 (br s, 1H), 8.71 (d, *J* = 8.8 Hz, 1H), 8.53 (s, 1H), 8.43 (d, *J* = 8.0 Hz, 1H), 8.14 (d, *J* = 8.0 Hz,

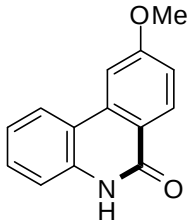
1H), 7.56 (t, *J* = 7.6 Hz, 1H), 7.40 (d, *J* = 8.0 Hz, 1H), 7.30 (t, *J* = 8.0 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 160.0, 137.6, 137.3, 131.0, 128.8, 128.7, 128.1, 127.9, 125.9, 125.1, 124.5, 124.45, 124.41, 122.9, 122.8, 116.6, 116.5; IR (KBr) ν 3423, 1734, 1717, 1699, 1635, 1540, 1507, 1397, 1126, 668 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{14}H_9F_3NO$ $[M+H]^+$ 264.0631, found 264.0632.

9-methylphenanthridin-6(5*H*)-one (2q)[2]



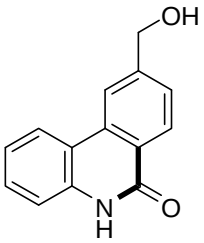
Gray white powder, 35 mg, 84% yield. ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.56 (br s, 1H), 8.35 (d, *J* = 8.0 Hz, 1H), 8.31 (s, 1H), 8.20 (d, *J* = 8.4 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 7.35 (d, *J* = 7.6 Hz, 1H), 7.24 (t, *J* = 7.2 Hz, 1H), 2.53 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 160.6,142.7, 136.6, 134.1, 129.1, 128.9, 127.3, 123.3, 122.9, 122.2, 121.8, 117.3, 115.9, 21.3; IR (KBr) ν 3450, 1673, 1617, 1429, 1366, 873, 827, 754, 674, 448 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{14}H_{12}NO$ $[M+H]^+$ 210.0913, found 210.0911.

9-methoxyphenanthridin-6(5*H*)-one (2r)[13]



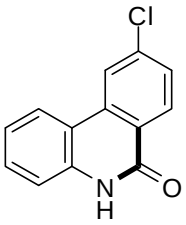
Pale yellow solid, 34 mg, 76% yield. ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.50 (br s, 1H), 8.41 (d, *J* = 8.4 Hz, 1H), 8.23 (d, *J* = 8.8 Hz, 1H), 7.89 (d, *J* = 2.0 Hz, 1H), 7.48 (t, *J* = 7.6 Hz, 1H), 7.34 (d, *J* = 8.0 Hz, 1H), 7.26-7.20 (m, 2H), 3.98 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 162.7, 160.4, 136.9, 136.2, 129.4, 123.3, 121.7, 119.1, 117.3, 115.92, 115.89, 105.0, 55.6; IR (KBr) ν 3002, 1658, 1609, 1509, 1458, 1428, 1362, 1298, 1236, 1223, 1031, 907, 676 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{14}H_{12}NO_2$ $[M+H]^+$ 226.0863, found 226.0862.

9-(hydroxymethyl)phenanthridin-6(5*H*)-one (2s)



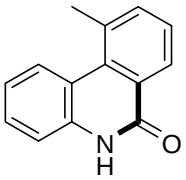
White powder, 27 mg, 60% yield. ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.63 (br s, 1H), 8.40 (s, 1H), 8.34 (d, *J* = 8.0 Hz, 1H), 8.28 (d, *J* = 8.0 Hz, 1H), 7.60 (d, *J* = 8.0 Hz, 1H), 7.48 (t, *J* = 7.6 Hz, 1H), 7.36 (d, *J* = 8.0 Hz, 1H), 7.26 (t, *J* = 7.6 Hz, 1H), 5.49 (t, *J* = 6.0 Hz, 1H), 4.73 (d, *J* = 6.0 Hz, 2H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 160.8, 147.7, 136.7, 134.1, 129.4, 127.4, 126.1, 124.3, 123.0, 122.2, 119.6, 117.6, 116.1, 62.7; IR (KBr) ν 3416, 1656, 1620, 1421, 745 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{14}H_{12}NO_2$ $[M+H]^+$ 226.0863, found 226.0862.

9-chlorophenanthridin-6(5*H*)-one (2t)[13]



Brown solid, 25 mg, 54% yield. ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.77 (br s, 1H), 8.60 (d, *J* = 1.6 Hz, 1H), 8.43 (d, *J* = 8.0 Hz, 1H), 8.30 (d, *J* = 7.6 Hz, 1H), 7.66 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.52 (t, *J* = 7.6 Hz, 1H), 7.36 (d, *J* = 8.0 Hz, 1H), 7.26 (t, *J* = 7.6 Hz, 1H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 160.0, 138.1, 136.9, 135.9, 130.1, 129.5, 127.8, 124.3, 123.5, 122.2, 122.1, 116.4, 116.0; IR (KBr) ν 3449, 1676, 1606, 749 cm^{-1} HRMS (ESI): Exact mass calcd for $C_{13}H_9ClNO$ $[M+H]^+$ 230.0367, found 230.0367.

10-methylphenanthridin-6(5*H*)-one (2u)[3]



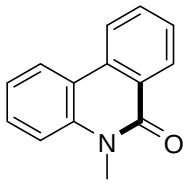
Brown solid, 18 mg, 43% yield. ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.70 (br s, 1H),

8.44 (d, *J* = 8.4 Hz, 1H), 8.31 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.71 (d, *J* = 6.8 Hz, 1H), 7.54 (t, *J* = 7.6 Hz, 1H), 7.51-7.46 (m, 1H), 7.41 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.28-7.23 (m, 1H), 2.92 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 160.9, 137.0, 136.96, 135.2, 133.3, 128.8, 127.4, 127.3, 127.2, 126.0, 121.7, 118.8, 116.2, 25.6; IR (KBr) ν 3415, 2925, 1657, 1524, 1449, 1208, 1043, 755 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{14}H_{12}NO$ $[M+H]^+$ 210.0913, found 210.0912.

10-fluorophenanthridin-6(5*H*)-one (2v)

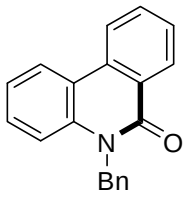
Pale yellow solid, 23 mg, 55% yield. ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.87 (br s, 1H), 8.49 (d, *J* = 8.0 Hz, 1H), 8.23 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.78-7.72 (m, 1H), 7.70-7.64 (m, 1H), 7.54 (t, *J* = 7.2 Hz, 1H), 7.41 (d, *J* = 7.2 Hz, 1H), 7.29 (t, *J* = 7.6 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 160.6, 159.62, 159.60, 158.6, 136.6, 129.8, 128.8, 128.7, 128.22, 128.19, 127.0, 126.8, 123.94, 123.91, 122.7, 122.6, 122.5, 120.3, 120.1, 116.2, 114.8, 114.78; IR (KBr) ν 3449, 1671, 1561, 1460, 1374, 1226, 825, 754, 692 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{13}H_9FNO$ $[M+H]^+$ 214.0663, found 214.0675.

5-methylphenanthridin-6(5*H*)-one (3a)[3]



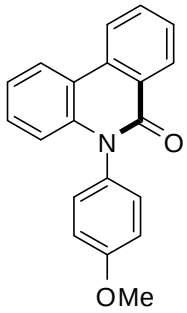
White solid, 36 mg, 86% yield. ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.50 (d, *J* = 8.0 Hz, 1H), 8.47 (d, *J* = 7.6 Hz, 1H), 8.35 (dd, *J* = 8.0, 0.8 Hz, 1H), 7.86-7.81 (m, 1H), 7.65-7.55 (m, 3H), 7.37-7.33 (m, 1H), 3.71 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 160.3, 137.6, 133.2, 132.7, 129.9, 128.1, 128.0, 124.9, 123.5, 122.5, 122.4, 118.4, 115.5, 29.7; IR (KBr) ν 3416, 1347, 1613, 1441, 1350, 744, 722, 685, 615 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{14}H_{12}NO$ $[M+H]^+$ 210.0913, found 210.0912.

5-benzylphenanthridin-6(5*H*)-one (3b)[14]



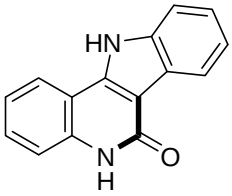
Gray white powder, 37 mg, 65% yield. ^{1}H NMR (400 MHz, DMSO-d$_6$): δ 8.57 (d, *J* = 7.6 Hz, 1H), 8.51 (d, *J* = 8.0 Hz, 1H), 8.44 (d, *J* = 8.0 Hz, 1H), 7.90 (t, *J* = 7.2 Hz, 1H), 7.70 (t, *J* = 7.6 Hz, 1H), 7.48 (t, *J* = 7.2 Hz, 1H), 7.41 (d, *J* = 7.6 Hz, 1H), 7.33-7.28 (m, 3H), 7.25-7.21 (m, 3H), 5.65 (s, 2H); ^{13}C NMR (125 MHz, DMSO-d$_6$): δ 160.8, 136.8, 136.7, 133.4, 133.1, 129.8, 128.6, 128.3, 127.0, 126.4, 124.7, 123.8, 122.7, 122.5, 118.7, 116.2, 45.2; IR (KBr) ν 3547, 1639, 1607, 1492, 1435, 1374, 1333, 1173, 759, 725, 466 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{20}H_{16}NO$ $[M+H]^+$ 286.1226, found 286.1224.

5-(4-methoxyphenyl)phenanthridin-6(5*H*)-one (3c)[15]



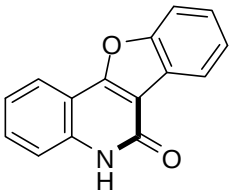
Brown solid, 46 mg, 77% yield. ^{1}H NMR (400 MHz, DMSO-d$_6$): δ 8.60 (d, *J* = 8.0 Hz, 1H), 8.51 (d, *J* = 7.6 Hz, 1H), 8.34 (d, *J* = 7.6 Hz, 1H), 7,92-7.89 (m, 1H), 7.68 (t, *J* = 7.6 Hz, 1H), 7.39 (t, *J* = 7.2 Hz, 1H), 7.33-7.27 (m, 3H), 7.16 (d, *J* = 8.8 Hz, 2H), 6.60 (d, *J* = 8.0 Hz, 1H), 3.86 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d$_6$): δ 160.7, 159.1, 139.2, 133.7, 133.2, 130.6, 130.3, 129.5, 128.3, 128.1, 125.3, 123.6, 122.64, 122.61, 118.3, 116.5, 115.3, 55.5; IR (KBr) ν 3421, 1657, 1514, 1318, 1251, 1029, 824, 751, 725 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{20}H_{16}NO_2$ $[M+H]^+$ 302.1176, found 302.1178

5*H*-indolo[3,2-*c*]quinolin-6(11*H*)-one (3d)[16]



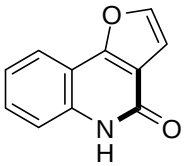
Gray white solid, 36 mg, 77% yield. ^{1}H NMR (400 MHz, DMSO-d_6): δ 12.56 (br s, 1H), 11.42 (br s, 1H), 8.20 (d, *J* = 8.0 Hz, 2H), 7.61 (d, *J* = 8.0 Hz, 1H), 7.52-7.46 (m, 2H), 7.37 (t, *J* = 7.6 Hz, 1H), 7.31-7.24 (m, 2H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 159.8, 140.7, 137.9, 137.7, 129.1, 124.4, 124.0, 122.1, 121.5, 121.0, 120.7, 116.0, 111.9, 111.7, 106.4; IR (KBr) ν 3449, 3219, 1639, 1555, 1398, 747 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{15}H_{11}N_2O$ $[M+H]^+$ 235.0866, found 235.0865.

benzofuro[3,2-*c*]quinolin-6(5*H*)-one (3e)[17]



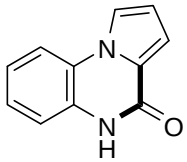
Pale yellow solid, 28 mg, 60% yield. ^{1}H NMR (400 MHz, DMSO-d_6): δ 12.03 (br s, 1H), 8.11 (d, *J* = 7.2 Hz, 1H), 8.07 (d, *J* = 8.0 Hz, 1H), 7.86 (d, *J* = 8.4 Hz, 1H), 7.62 (t, *J* = 8.0 Hz, 1H), 7.54-7.46 (m, 3H), 7.35 (t, *J* = 7.6 Hz, 1H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 158.8, 157.7, 154.7, 138.3, 130.6, 126.1, 124.4, 123.7, 122.2, 121.1, 120.9, 116.0, 111.6, 110.6, 109.9; IR (KBr) ν 3417, 1685, 1450, 1107, 736, 542 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{15}H_{10}NO_2$ $[M+H]^+$ 236.0706, found 236.0707.

furo[3,2-*c*]quinolin-4(5*H*)-one (3f)[18]



Pale yellow solid, 17 mg, 46% yield. ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.76 (br s, 1H), 8.09 (d, *J* = 2.0 Hz, 1H), 7.90 (d, *J* = 7.6 Hz, 1H), 7.53-7.45 (m, 2H), 7.30-7.36 (m, 1H), 7.06 (d, *J* = 2.0 Hz, 1H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 158.6, 155.2, 145.1, 136.9, 129.2, 122.0, 119.9, 115.8, 115.4, 111.1, 107.4; IR (KBr) ν 3416, 2852, 1670, 1564, 1364, 1265, 1197, 887, 760, 731, 656, 453 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{11}H_8NO_2$ $[M+H]^+$ 186.0550 found 186.0548.

pyrrolo[1,2-*a*]quinoxalin-4(5*H*)-one (3g)[19]



Pale yellow solid, 28 mg, 76% yield. ^{1}H NMR (400 MHz, DMSO-d_6): δ 11.25 (br s, 1H), 8.18 (t, *J* = 1.2 Hz, 1H), 8.04 (d, *J* = 8.4 Hz, 1H), 7.31-7.26 (m, 2H), 7.23-7.18 (m, 1H), 7.03-7.02(m, 1H), 6.69-6.68 (m, 1H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 154.8, 128.4, 125.4, 123.2, 122.4, 122.3, 117.7, 116.3, 114.7, 112.4, 111.1; IR (KBr) ν 3451, 1655, 1514, 1433, 1382, 738, 651 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{11}H_9N_2O$ $[M+H]^+$ 185.0709, found 185.0711.

III. References

(1) J. Xi, Q.-L. Dong, G.-S. Liu, S. Wang, L. Chen and Z.-J. Yao, *Synlett.,* 2010, **11**, 1674.

(2) G.-W. Wang, T.-T. Yuan and D.-D. Li, *Angew. Chem., Int. Ed*., 2011, **50**, 1380.

(3) B. S. Bhakuni, A. Kumar, S. J. Balkrishna, J. A. Sheikh, S. Konar and S. Kumar, *Org. Lett.,* 2012, **14**, 2838.

(4) C. C. Kofink, B. Blank, S. Pagano, N. Götz and P. Knochel, *Chem. Commun*., 2007, **19**, 1954.

(5) R. F. Cookson, J. W. James, R. E. Rodway and R. G. Simmonds, *J. Heterocyclic Chem*., 1972, **9**, 475.

(6) S. Patil, S. Kamath, T. Sanchez, N. Neamati, R. F. Schinazi and J. F. Buolamwini, *Bioorg. Med. Chem*., 2007, **15**. 1212.

(7) E. Dubost, R. Magnelli, C. Cailly, R. Legay, F. Fabis and S. Rault, *Tetrahedron*, 2010, *66,* 5008.

(8) B. E. C. Taylojrr and E. J. Strojny, *J. Am. Chem. Soc*., 1956, **78**, 5104.

(9) E. M. Beccalli, F. Clerici and M. L. Gelmi, *Tetrahedron*, 2003, **59**, 4615.

(10) B. Roberta, C. Sandro, F. Giancarlo and S. Alessio, *Synthesis,* 2008, **5**, 729.

(11) H.-L. Pan and T. L. Fletcher, *J. Heterocyclic Chem*., 1970. **7**, 313.

(12) C. C. Woodroofe, B. Zhong, X. Lu and R. B. Silverman, *J. Chem. Soc., Perkin Trans*., 2000, **2**, 55.

(13) N. A. Al-Awadi, B. J. George,H. H. Dib, M. R. Ibrahim, Y. A. Ibrahim and O. M.

E. El-Dusouqui, *Tetrahedron*, 2005, **61**, 8257.

(14) L. Donati, S. Michel, F. Tillequin and F.-H. Porée, *Org. Lett*., 2010, **12** ,156.

(15) L.-C. Campeau, M. Parisien, A. Jean and K. Fagnou, *J. Am. Chem. Soc*., 2006, **128**, 581.

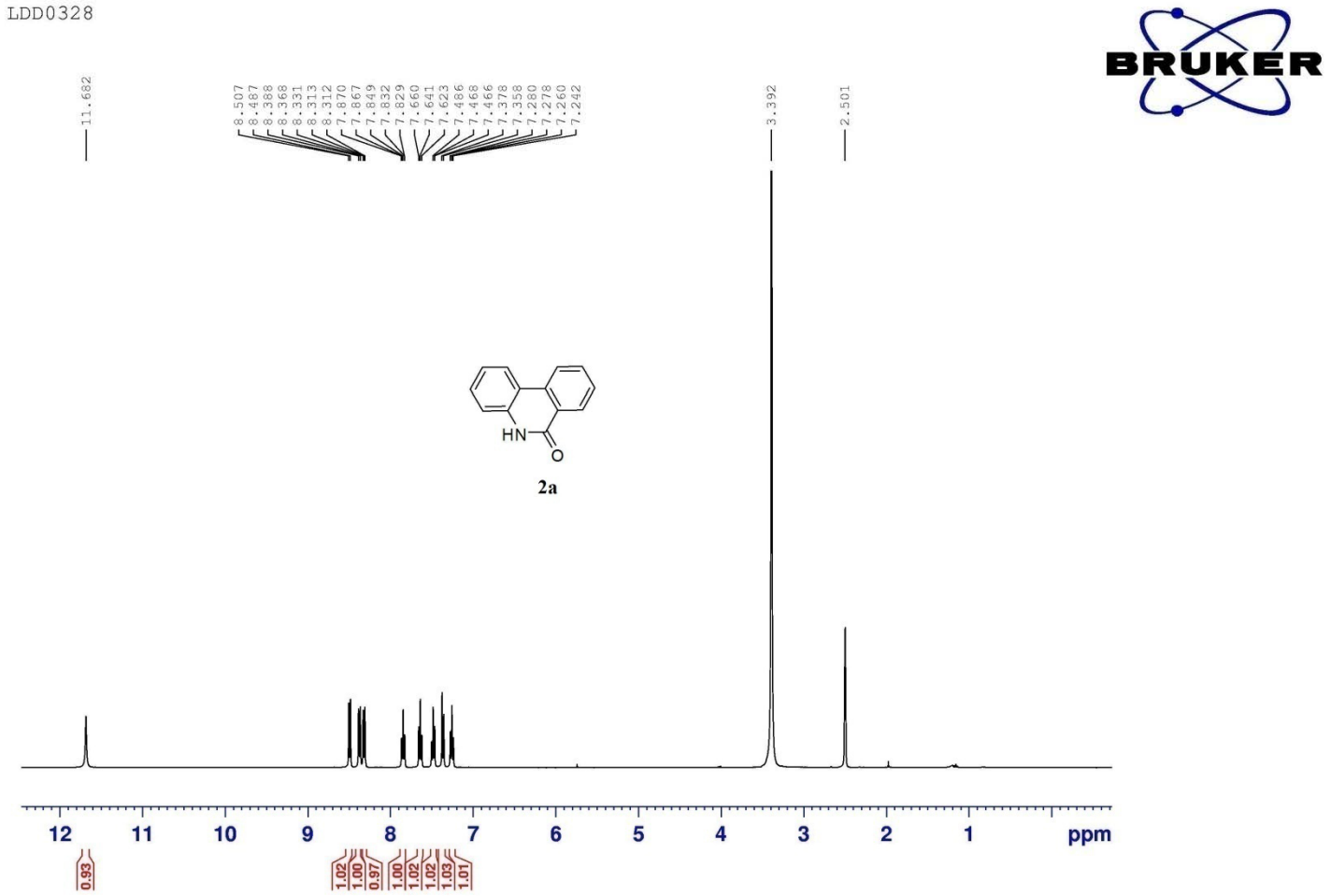
(16) K. Hayashi, T. Choshi, K. Chikaraishi, A. Oda, R. Yoshinaga, N. Hatae, M. Ishikura and S. Hibino, *Tetrahedron*, 2012, **68**, 4274.

(17) H. Li, H. Yang, J. L. Petersen, k. K. Wang, *J. Org. Chem.,* 2004, **69**, 4500.

(18) B. Glover, K. A. Harvey, B. Liu, M. J. Sharp and M. F. Tymoschenko, *Org. Lett.,* 2003, **5**, 301.

(19) Q. Yuan and D. Ma, *J. Org. Chem*., 2008, **73**, 5159.

IV. Copies of NMR Spectra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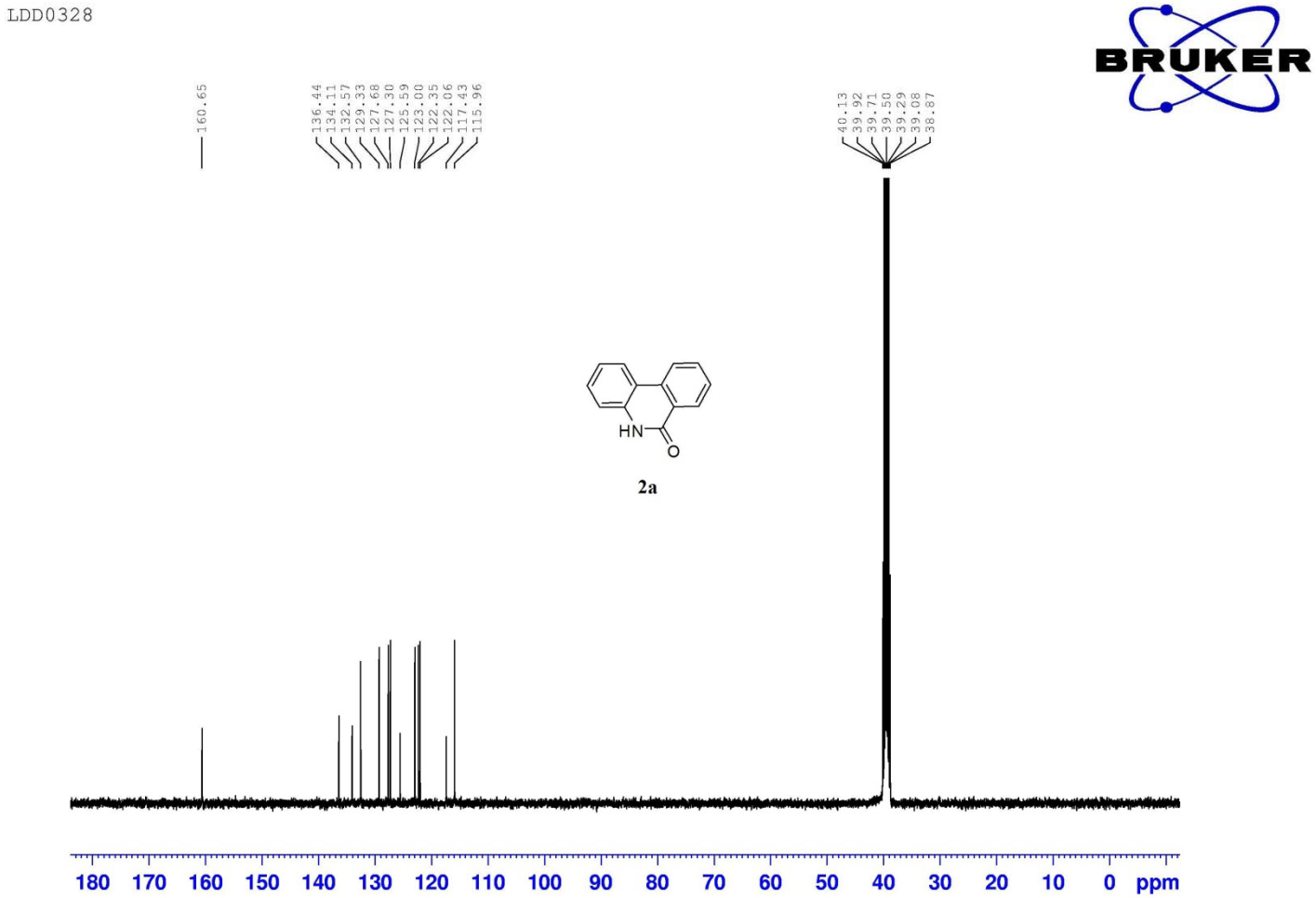

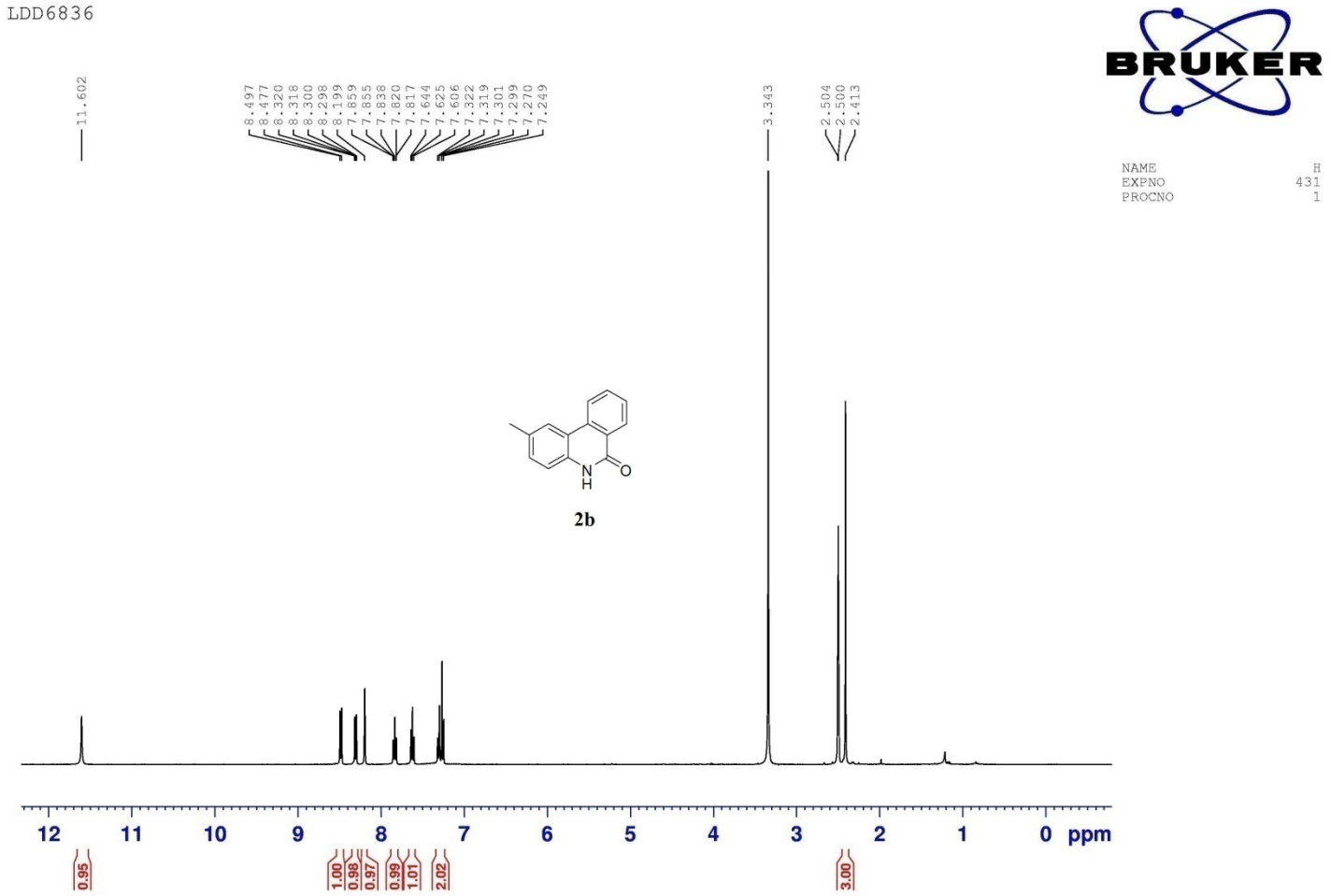
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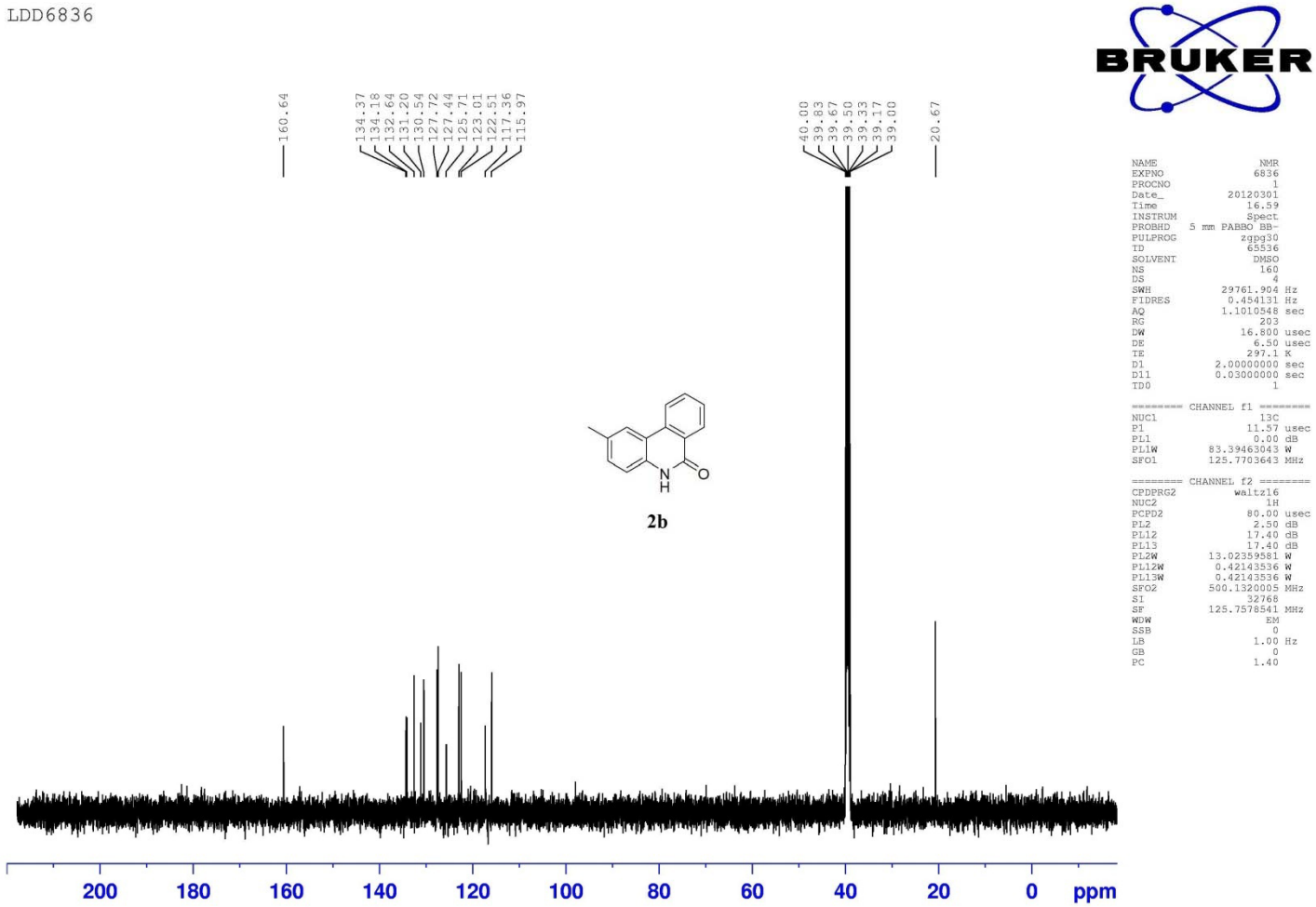
3.00



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PL1	0.00 dB
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PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
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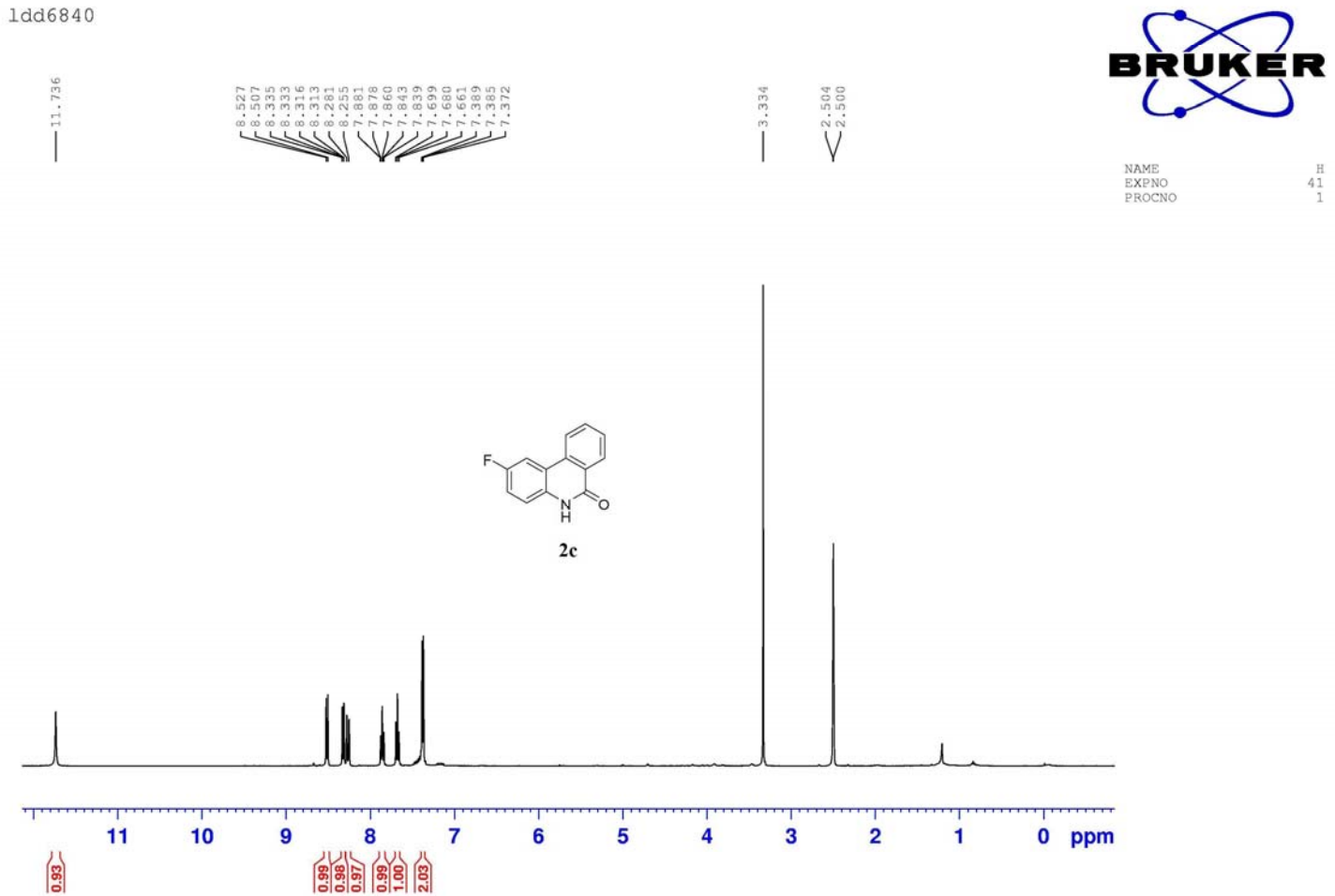
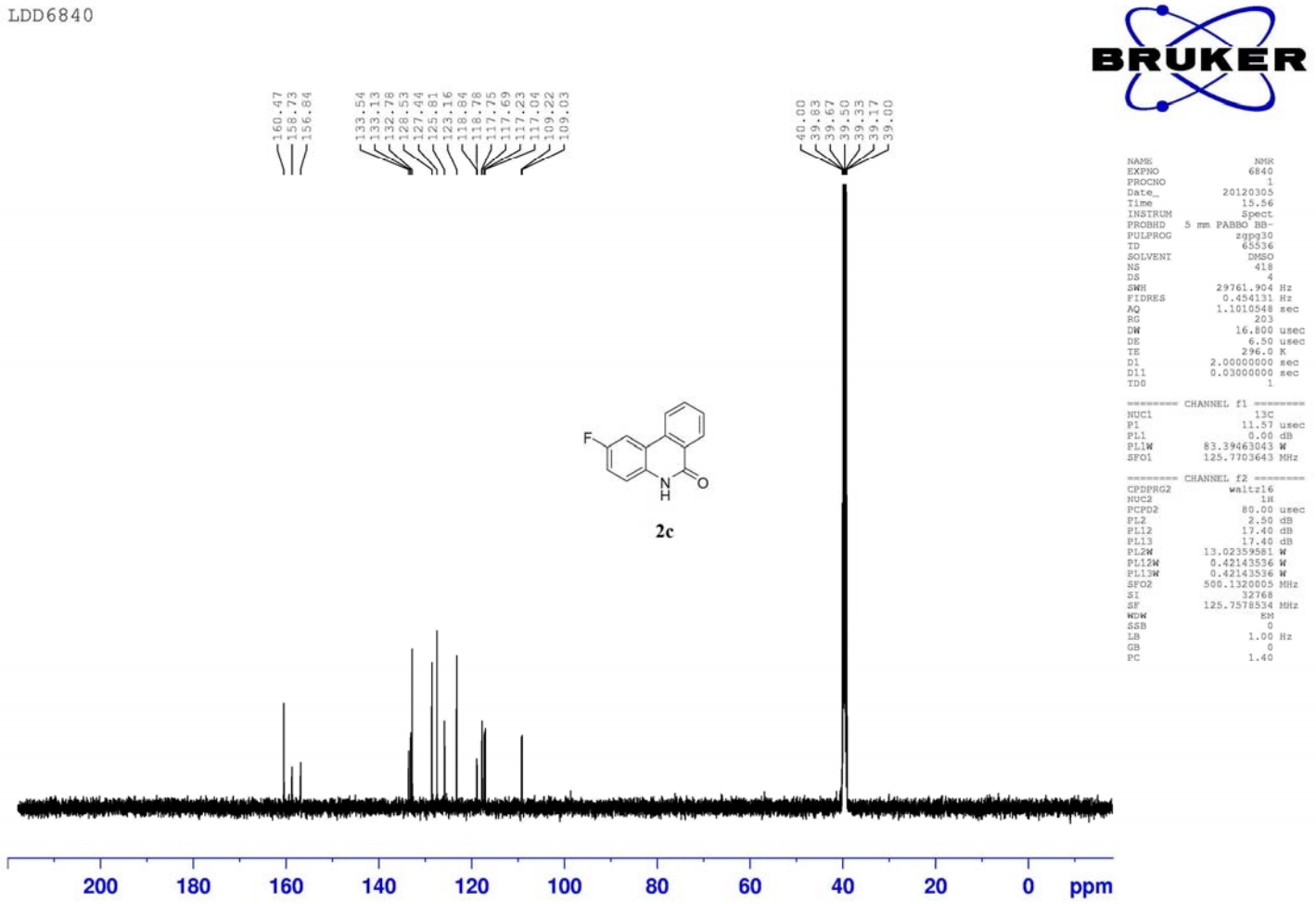
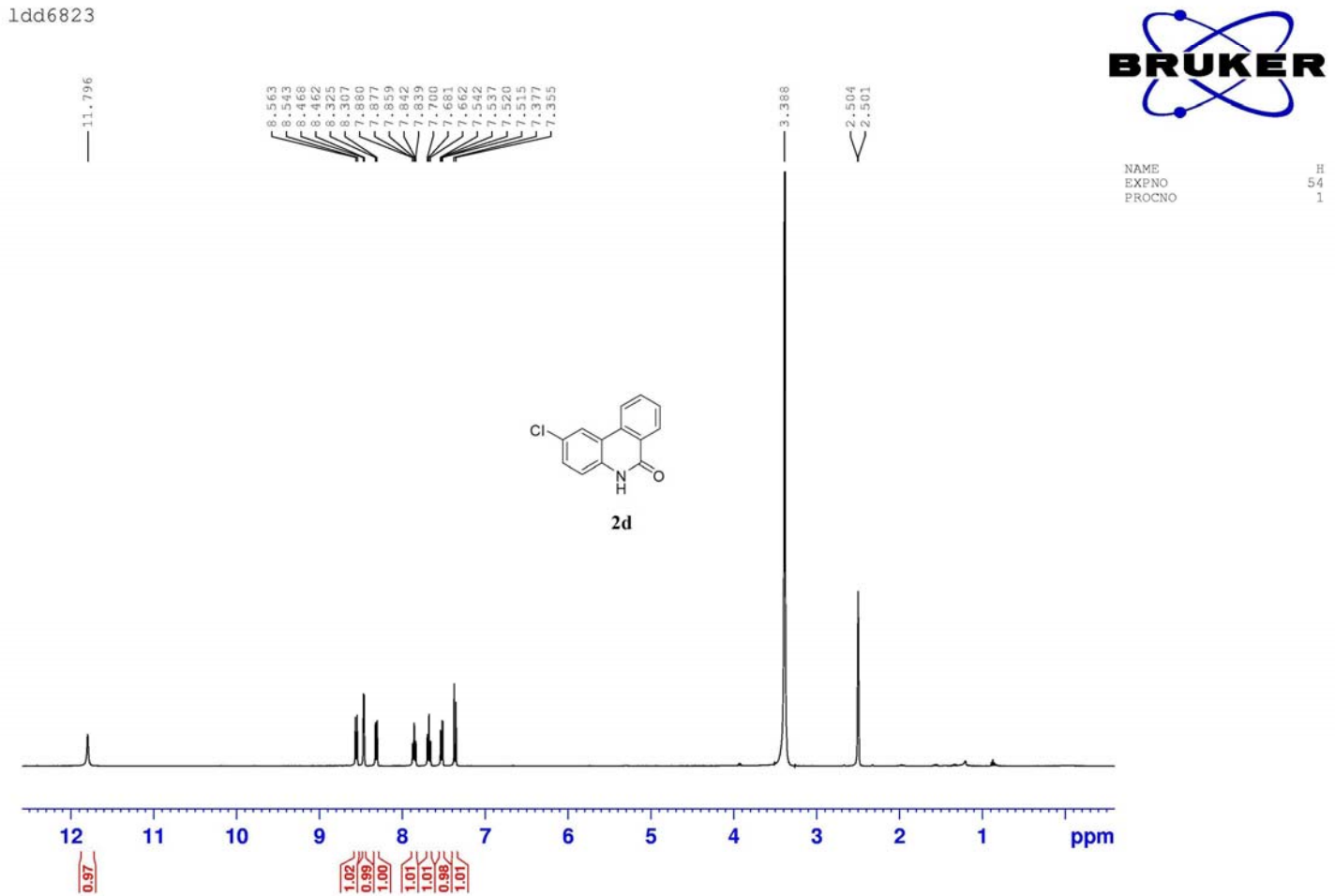
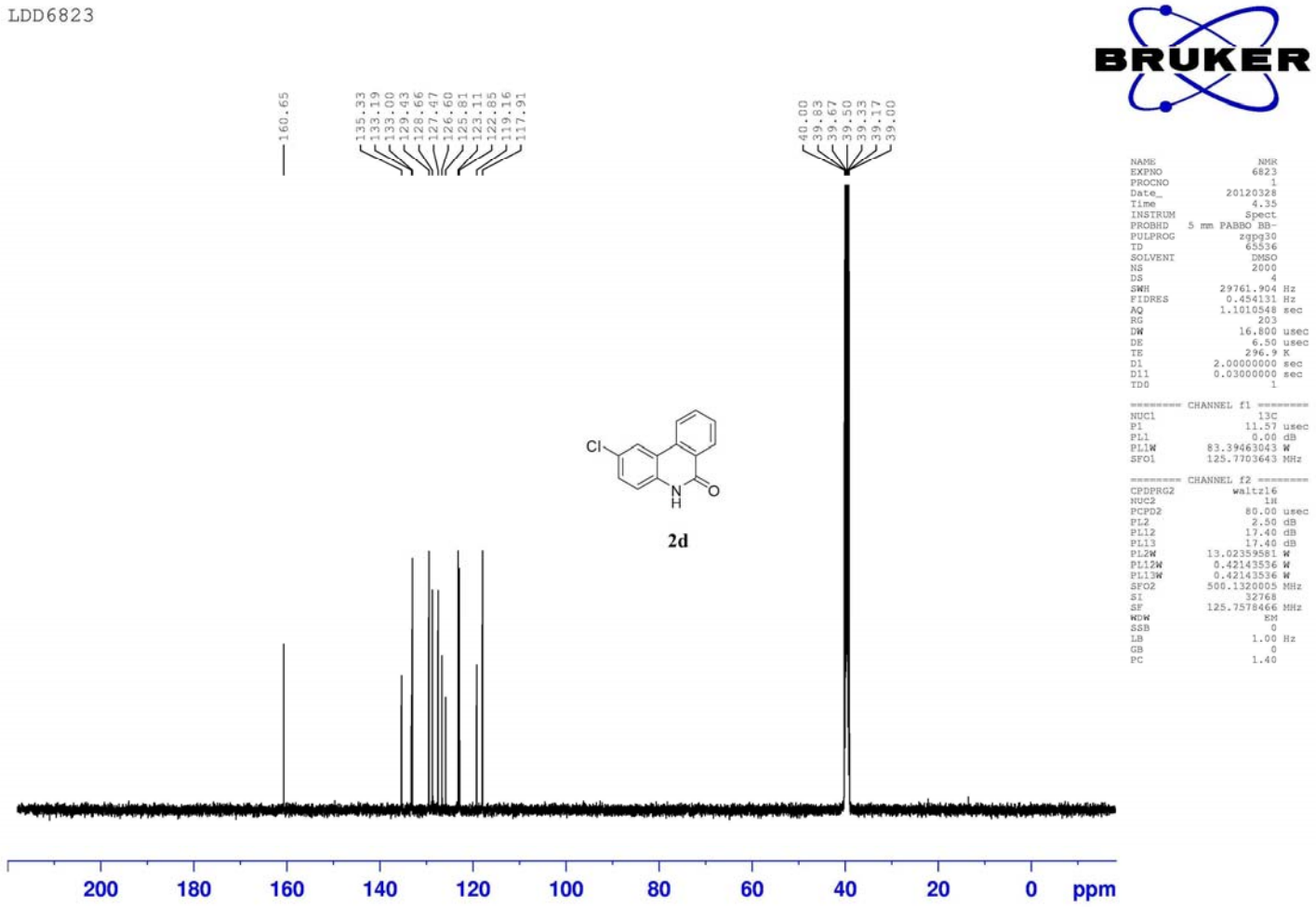
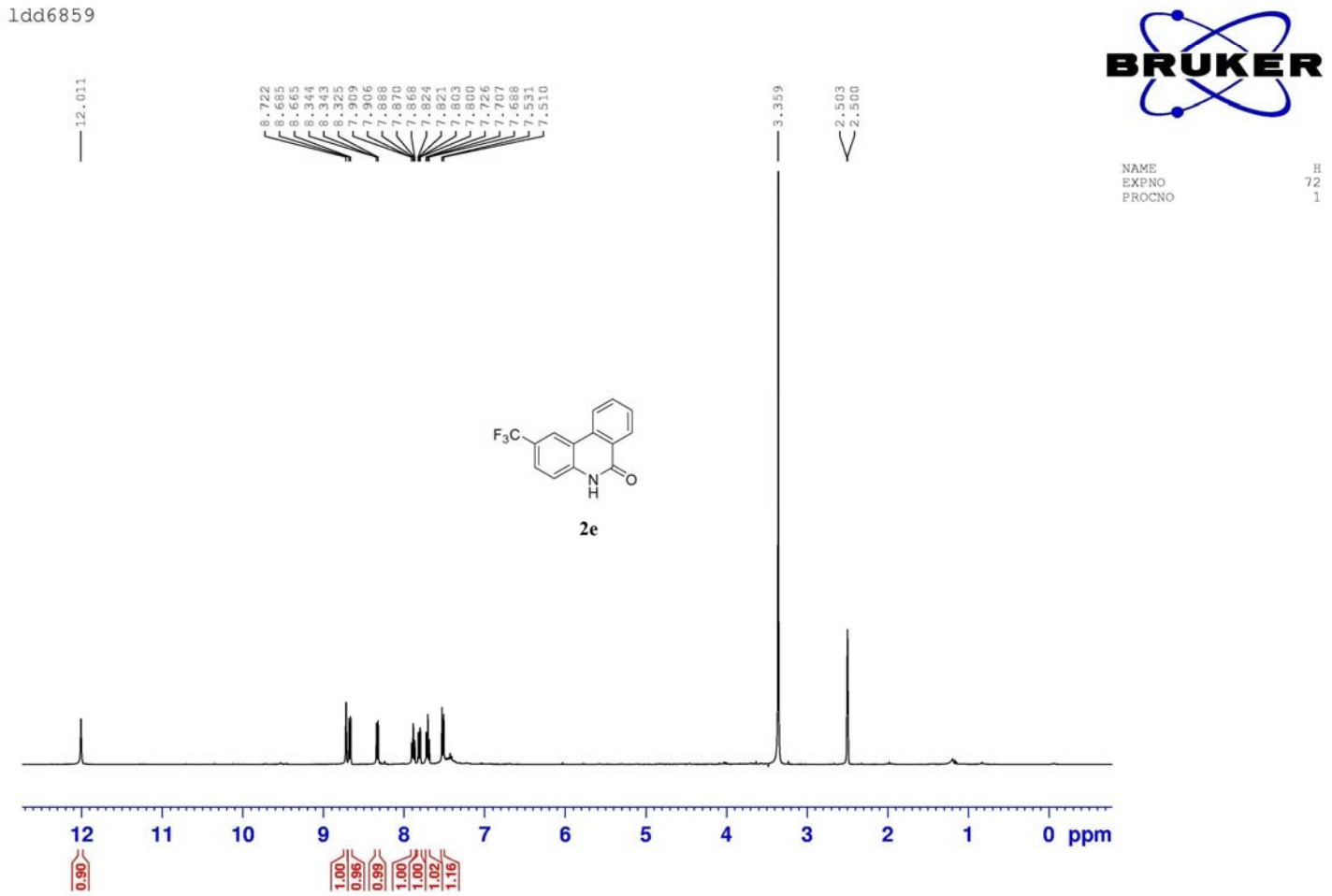
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7578541 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

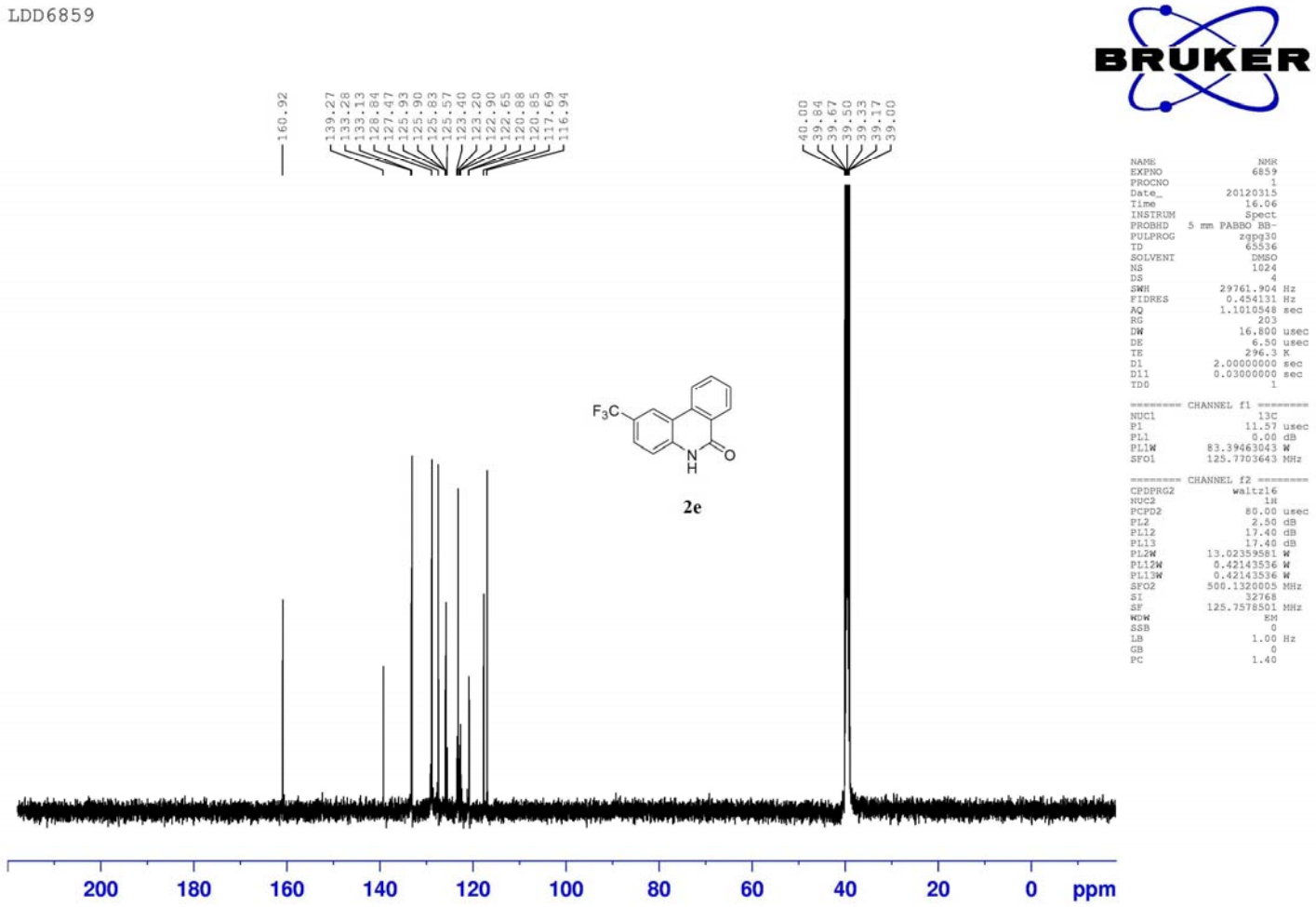

ldd6840
11.736
8.527
8.507
8.335
8.333
8.316
8.313
8.281
8.255
7.881
7.878
7.860
7.843
7.839
7.699
7.680
7.661
7.389
7.385
7.372
3.334
2.504
2.500
BRUKER
NAME H
EXPNO 41
PROCNO 1
F
N
H
O
2c
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.93
0.99
0.98
0.97
0.99
1.00
2.03

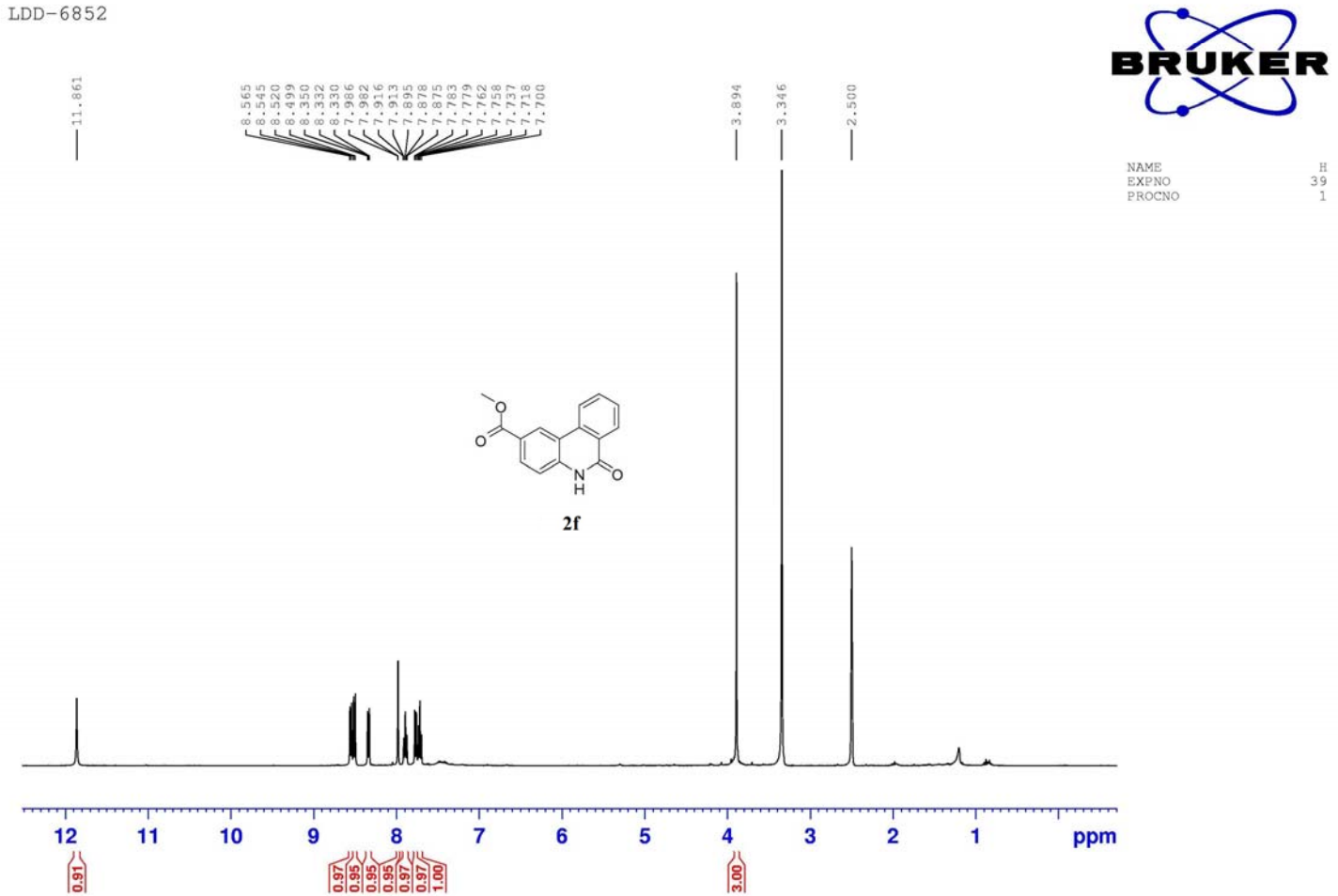

LDD6840
160.47
158.73
156.84
133.54
133.13
132.78
128.53
127.44
125.81
123.16
118.84
118.78
117.75
117.69
117.23
117.04
109.22
109.03
40.00
39.83
39.67
39.50
39.33
39.17
39.00
F
N
H
O
2c
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER
NAME NMR
EXPNO 6840
PROCNO 1
Date_ 20120305
Time 15.56
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 418
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 296.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.57 usec
PL1 0.00 dB
PL1W 83.3946304 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.0235958 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7578534 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

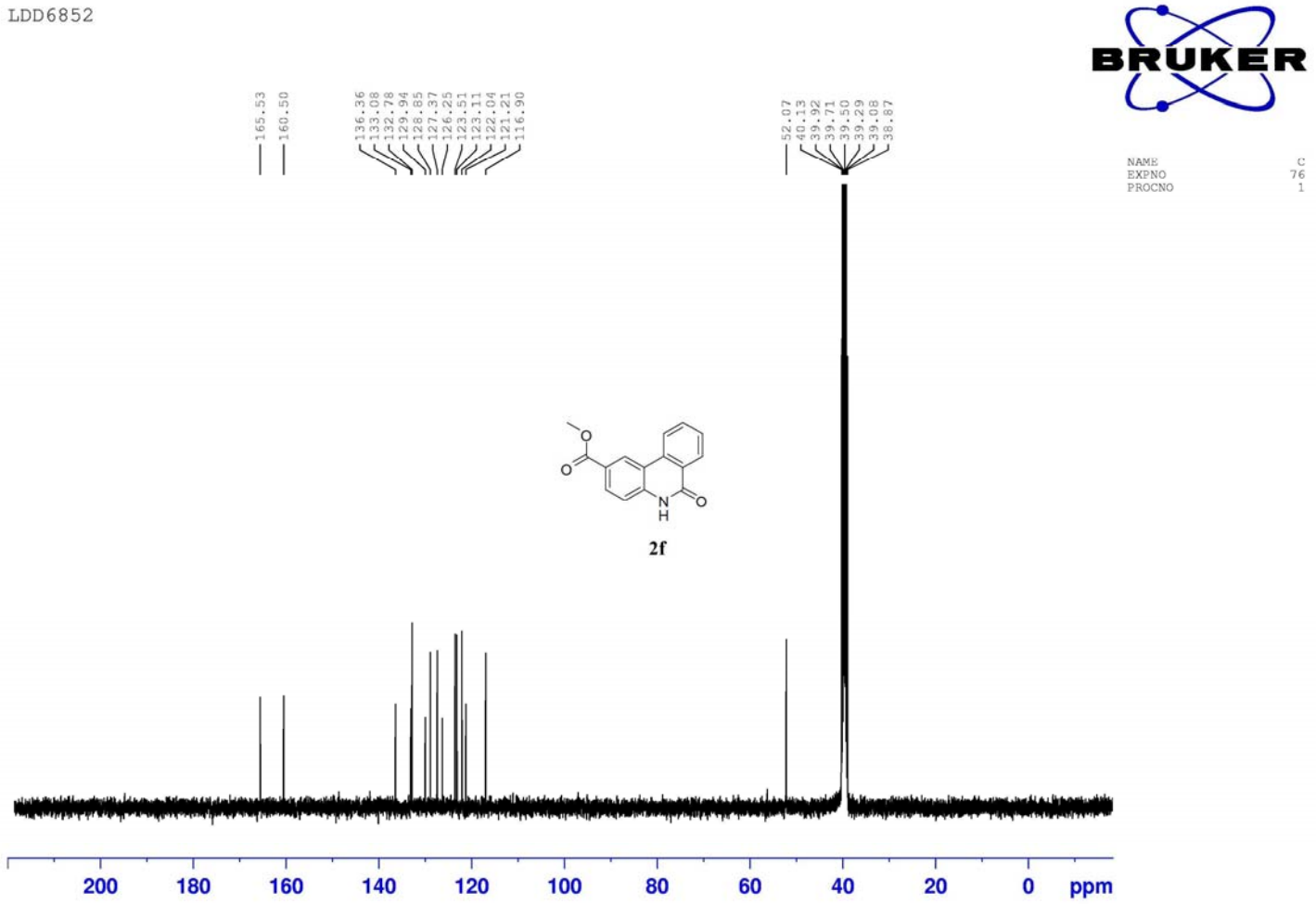

ldd6823
11.796
8.563
8.543
8.468
8.462
8.325
8.307
7.880
7.877
7.859
7.842
7.839
7.700
7.681
7.662
7.542
7.537
7.520
7.515
7.377
7.355
3.388
2.504
2.501
BRUKER
NAME H
EXPNO 54
PROCNO 1
Cl
N
H
O
2d
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.97
1.02
0.99
1.00
1.01
1.01
0.98
1.01

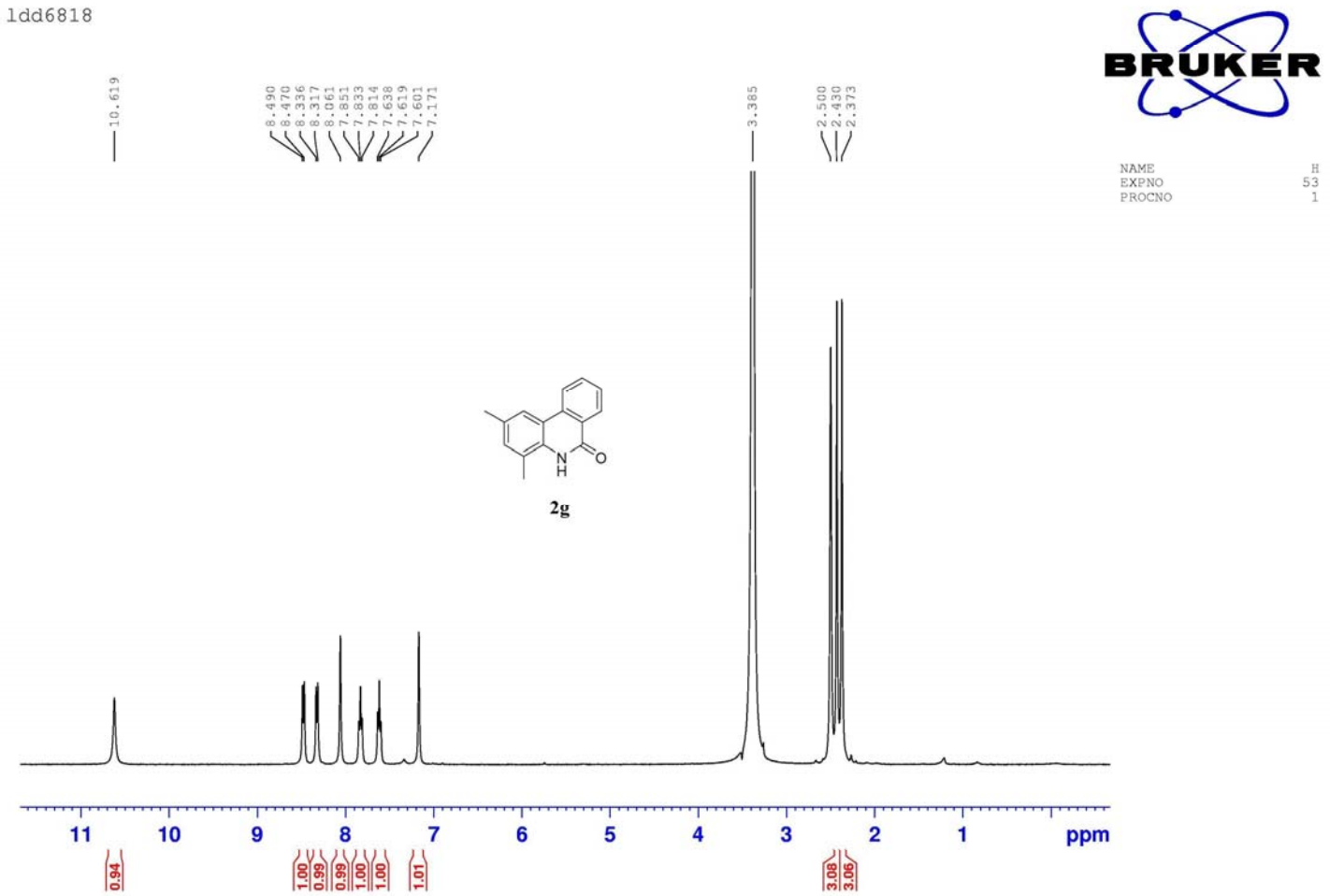

LDD6823
160.65
135.33
133.19
133.00
129.43
128.66
127.47
126.60
125.81
123.11
122.85
119.16
117.91
40.00
39.83
39.67
39.50
39.33
39.17
39.00
Cl
N
H
O
2d
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER
NAME NMR
EXPNO 6823
PROCNO 1
Date_ 20120328
Time 4.35
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2000
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 296.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.57 usec
PL1 0.00 dB
PL1W 83.3946304 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.0235958 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7578466 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

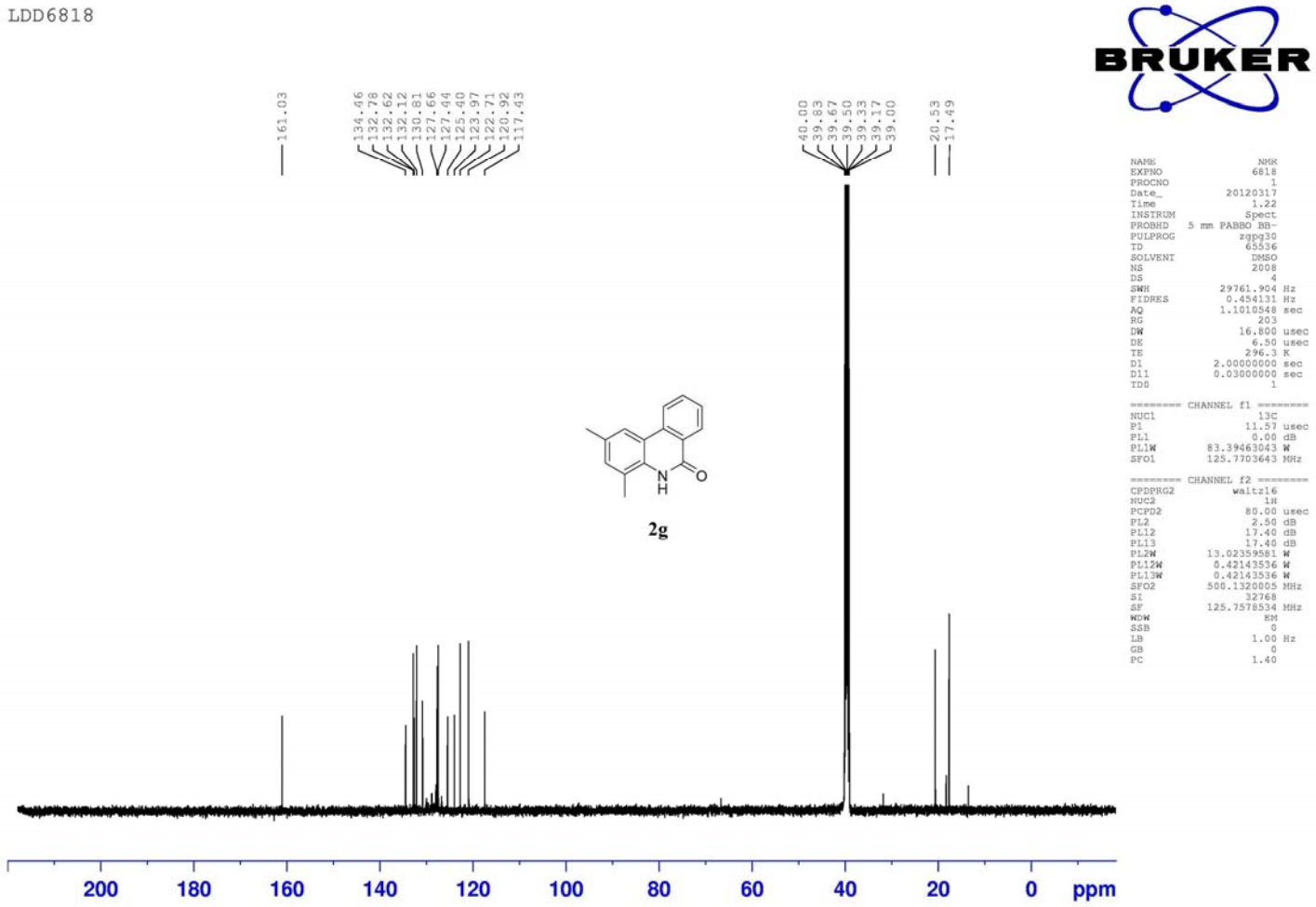


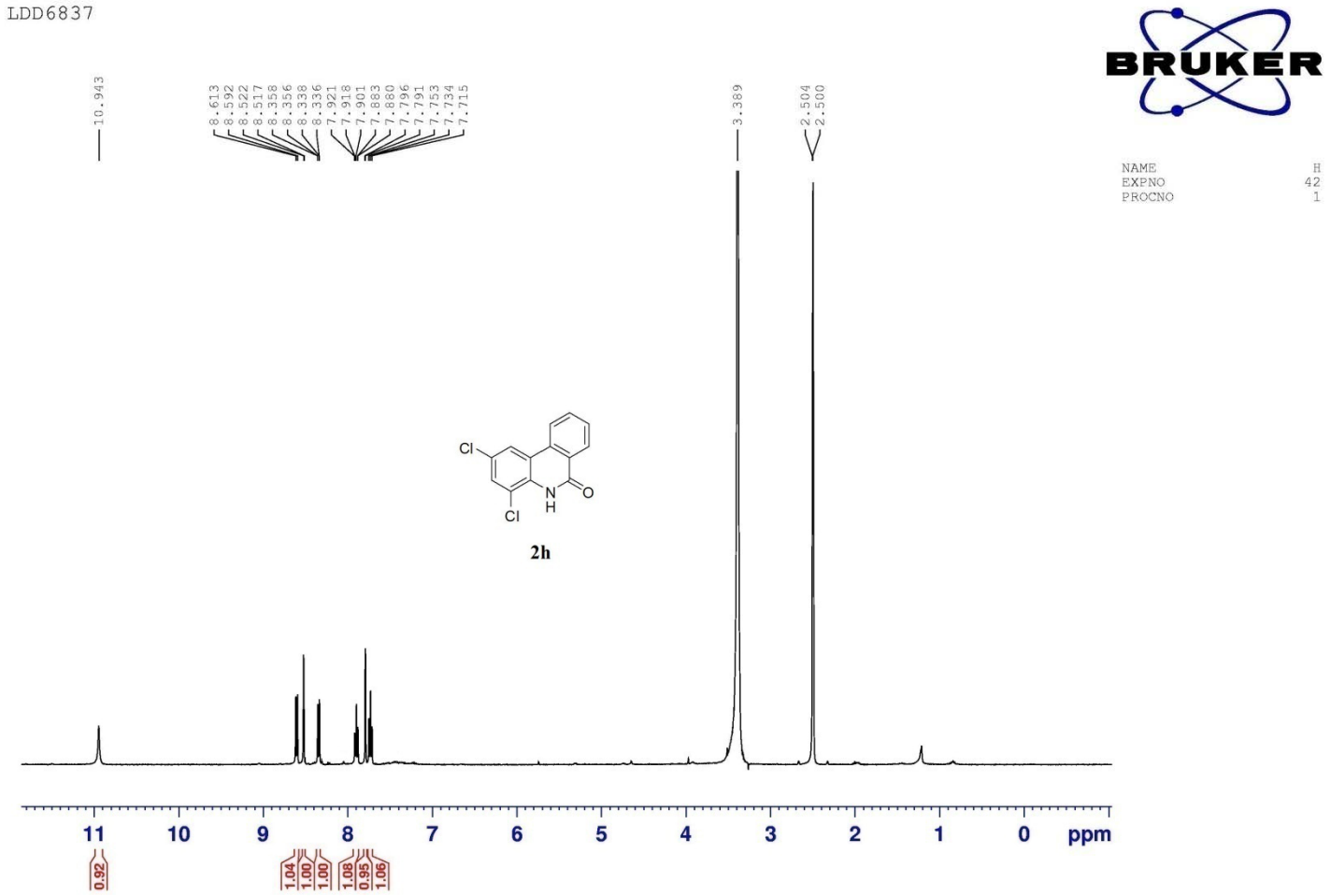

LDD6859
160.92
139.27
133.28
133.13
128.84
127.47
125.93
125.90
125.83
125.57
123.40
123.20
122.90
122.65
120.88
120.85
117.69
116.94
40.00
39.84
39.67
39.50
39.33
39.17
39.00
F3C
N
H
O
2e
BRUKER
NAME NMR
EXPNO 6859
PROCNO 1
Date_ 20120315
Time 16.06
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 296.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.57 usec
PL1 0.00 dB
PL1W 83.3946304 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.0235958 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7578501 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

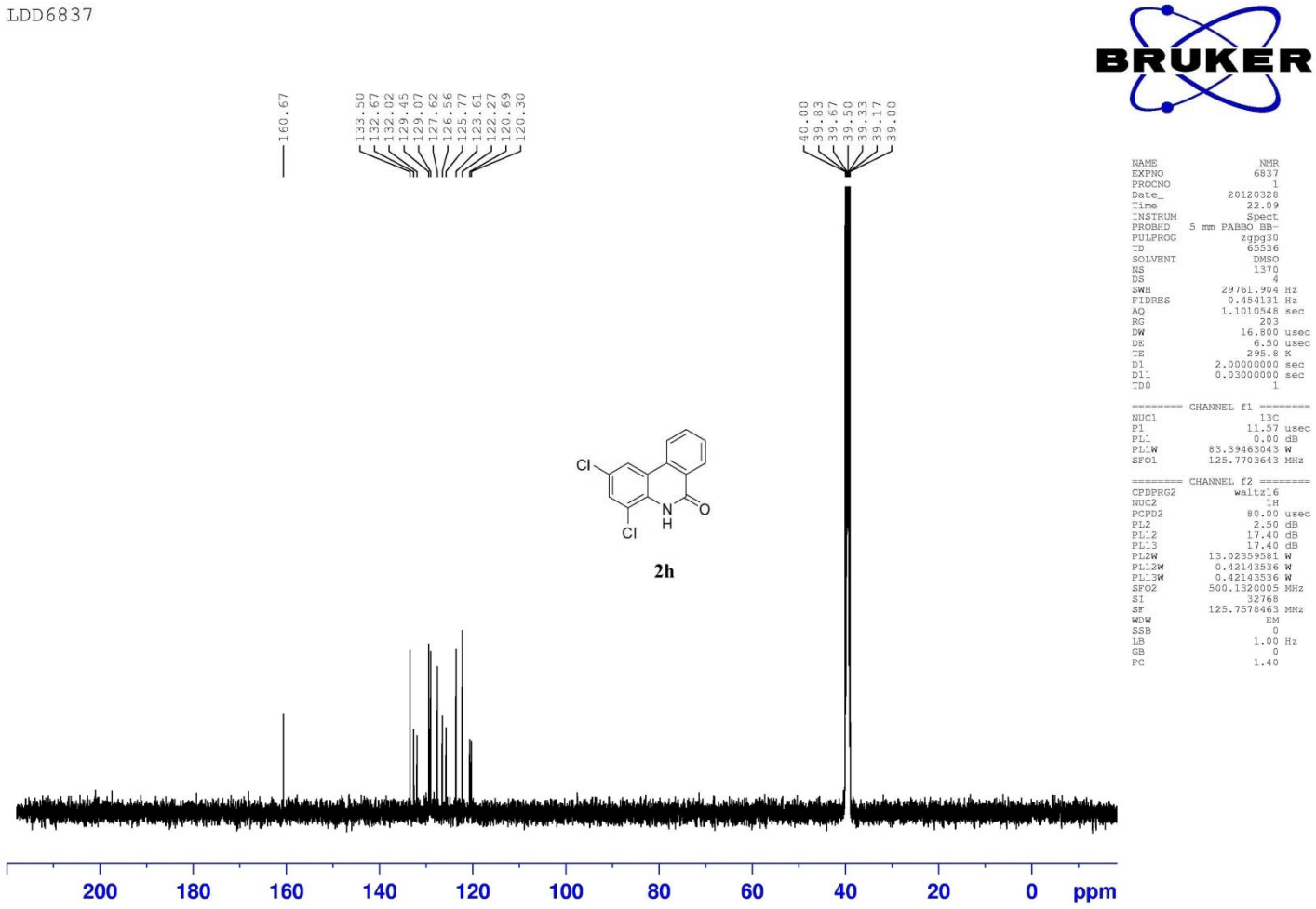

LDD-6852
11.861
8.565
8.545
8.520
8.499
8.350
8.332
8.330
7.986
7.982
7.916
7.913
7.895
7.878
7.875
7.783
7.779
7.762
7.758
7.737
7.718
7.700
3.894
3.346
2.500
NAME H
EXPNO 39
PROCNO 1
O
O
N
H
O
2f
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.91
0.97
0.95
0.95
0.95
0.97
0.97
1.00
3.00


LDD6852
165.53
160.50
136.36
133.08
132.78
129.94
128.85
127.37
126.25
123.51
123.11
122.04
121.21
116.90
52.07
40.13
39.92
39.71
39.50
39.29
39.08
38.87
NAME C
EXPNO 76
PROCNO 1
2f
200
180
160
140
120
100
80
60
40
20
0
ppm


ldd6818
10.619
8.490
8.470
8.336
8.317
8.061
7.851
7.833
7.814
7.638
7.619
7.601
7.171
3.385
2.500
2.430
2.373
NAME H
EXPNO 53
PROCNO 1
2g
11
10
9
8
7
6
5
4
3
2
1
ppm
0.94
1.00
0.99
0.99
1.00
1.00
1.01
3.08
3.06

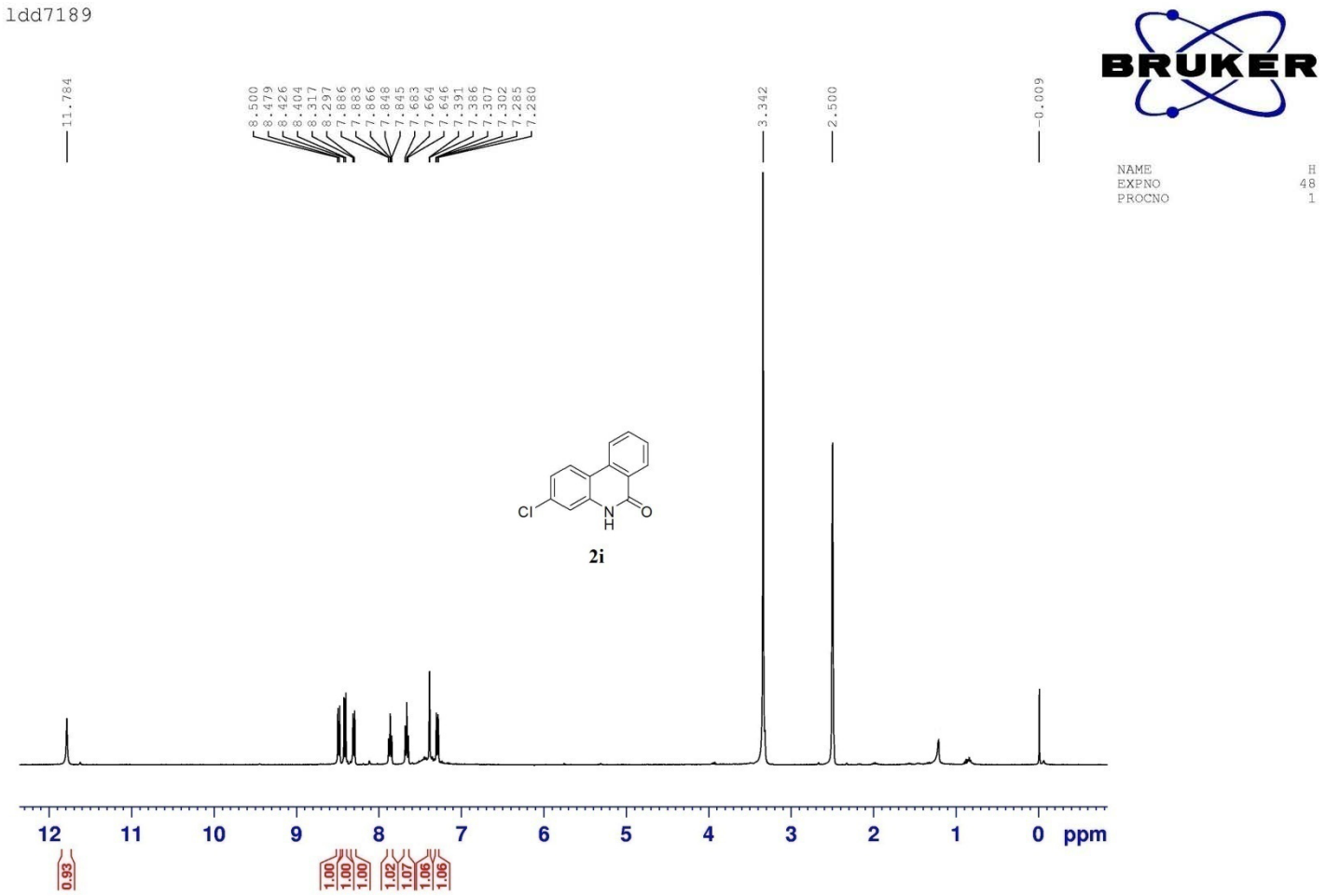

LDD6818
161.03
134.46
132.78
132.62
132.12
130.81
127.66
127.44
125.40
123.97
122.71
120.92
117.43
40.00
39.83
39.67
39.50
39.33
39.17
39.00
20.53
17.49
2g
200
180
160
140
120
100
80
60
40
20
0
ppm
NAME NMR
EXPNO 6818
PROCNO 1
Date_ 20120317
Time 1.22
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2008
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 296.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 11.57 usec
PL1 0.00 dB
PL1W 83.3946304 W
SFO1 125.7703643 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.0235958 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7578534 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

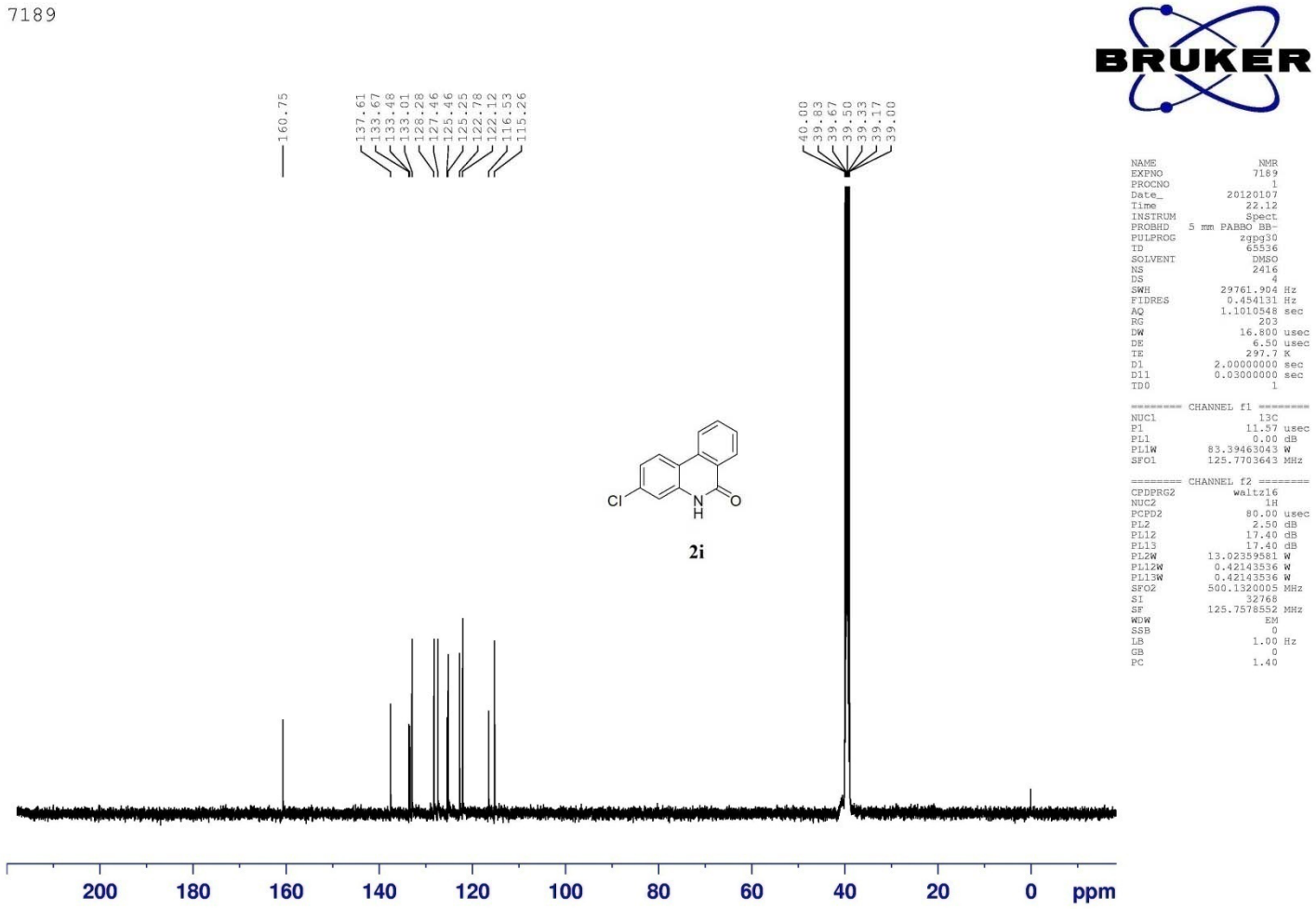

LDD6837
10.943
8.613
8.592
8.522
8.517
8.358
8.356
8.338
8.336
7.921
7.918
7.901
7.883
7.880
7.796
7.791
7.753
7.734
7.715
3.389
2.504
2.500
BRUKER
NAME H
EXPNO 42
PROCNO 1
Cl
N
H
O
Cl
2h
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.92
1.04
1.00
1.00
1.08
0.95
1.06

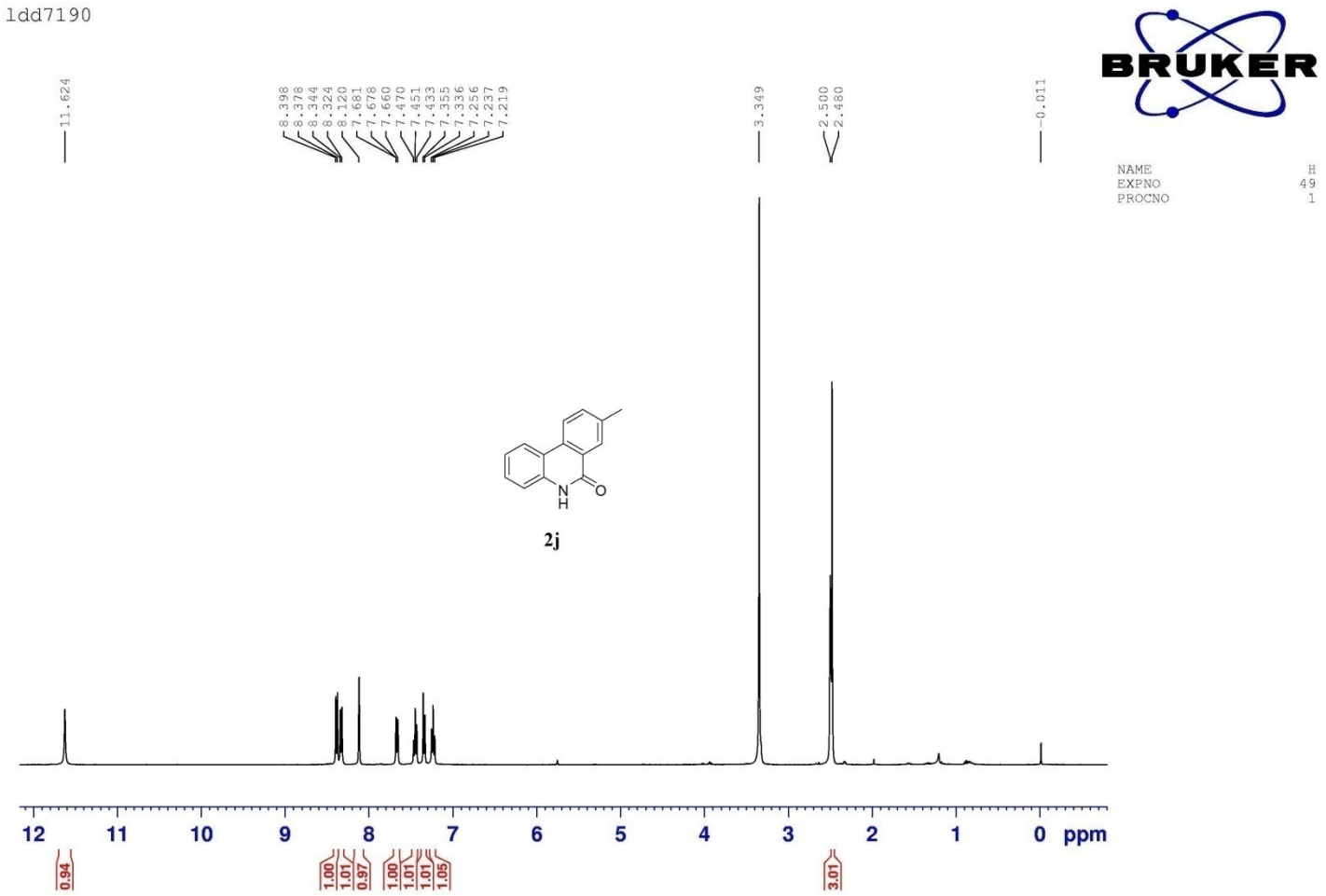


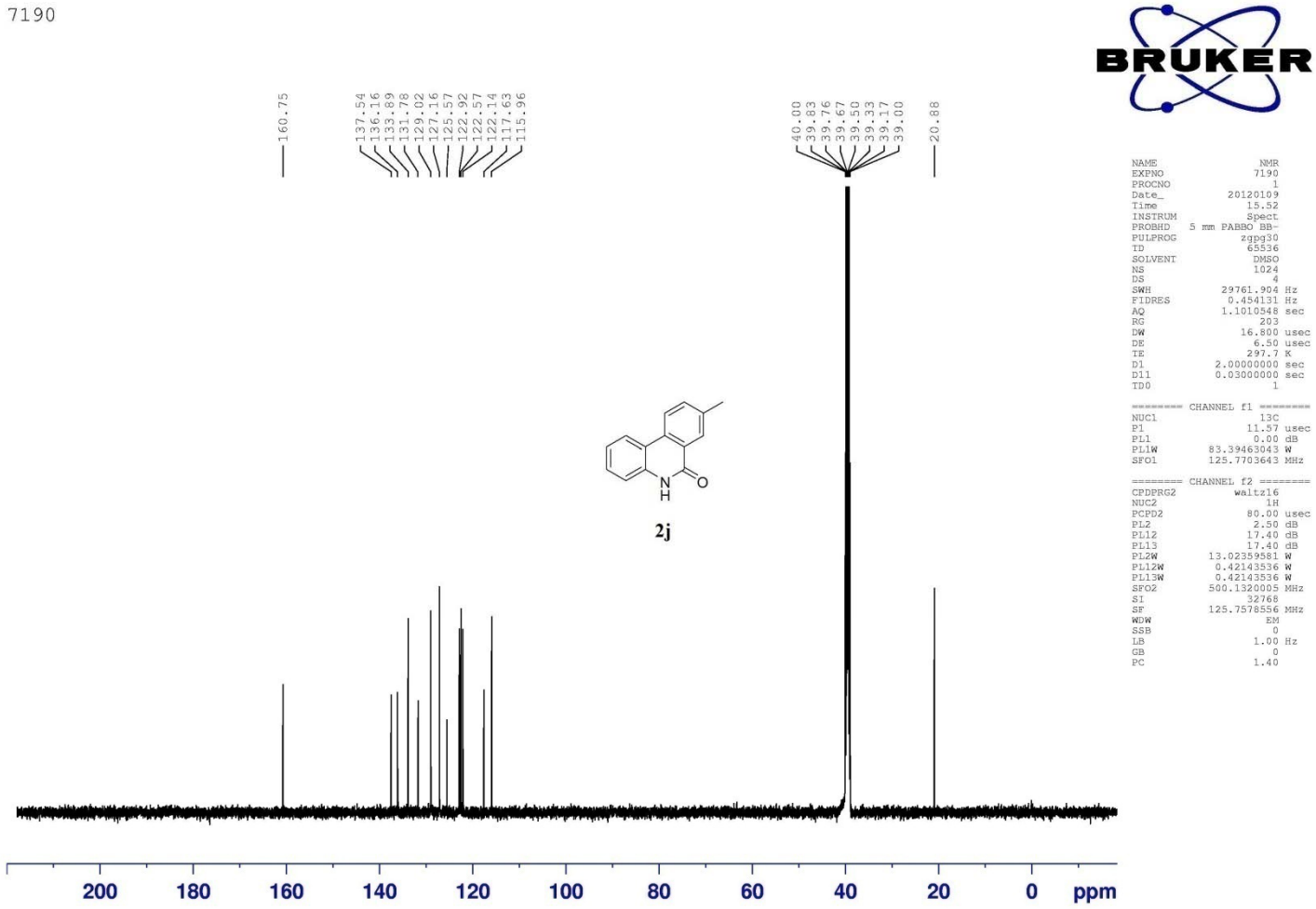
BRUKER

NAME	NMR
EXPNO	6837
PROCNO	1
Date_	20120328
Time	22.09
INSTRUM	Spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	DMSO
NS	1370
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	295.8 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.57 usec
PL1	0.00 dB
PL1W	83.39463043 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7578463 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

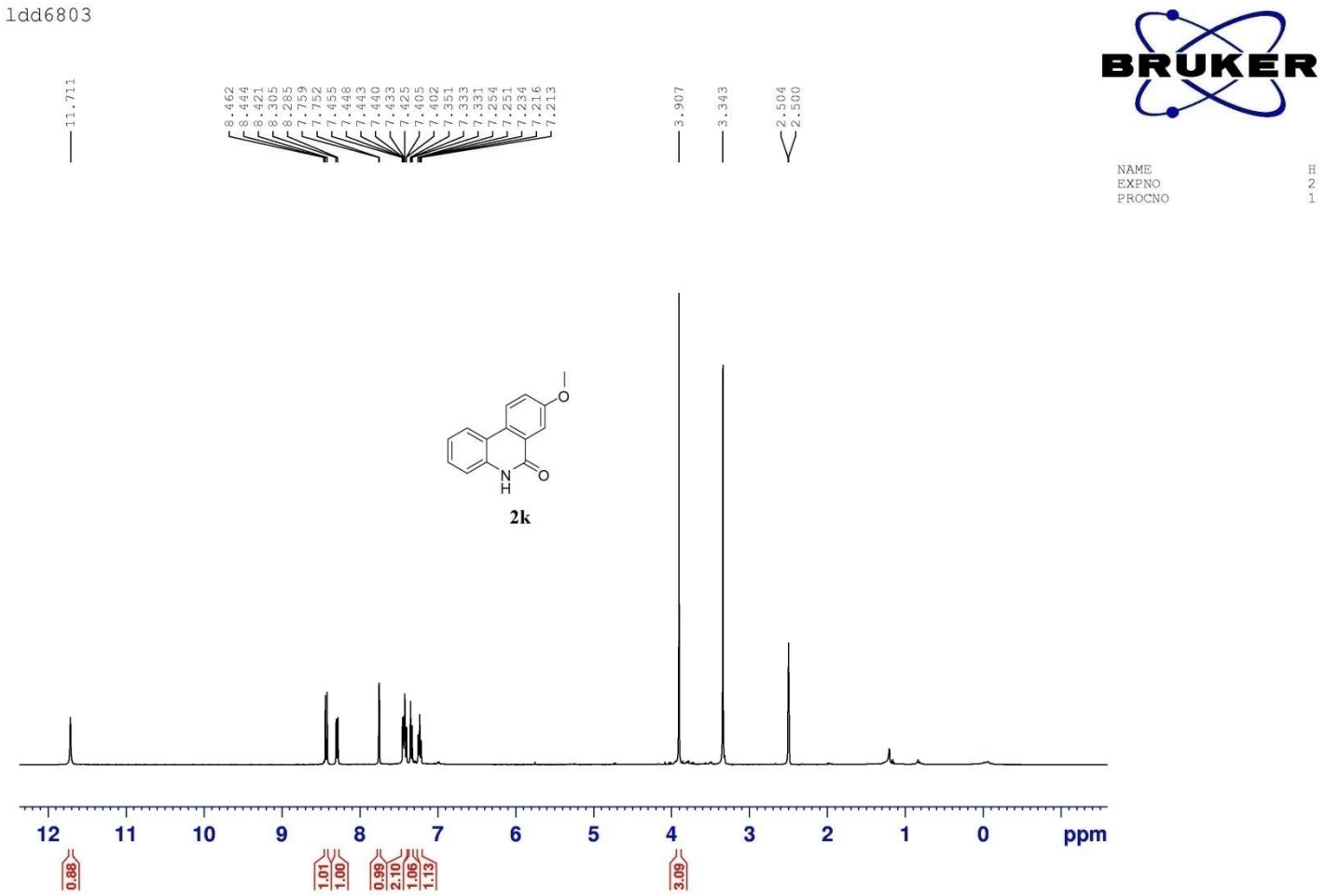

ldd7189
11.784
8.500
8.479
8.426
8.404
8.317
8.297
7.886
7.883
7.866
7.848
7.845
7.683
7.664
7.646
7.391
7.386
7.307
7.302
7.285
7.280
3.342
2.500
0.009
BRUKER
NAME H
EXPNO 48
PROCNO 1
Cl
N
H
O
2i
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.93
1.00
1.00
1.00
1.02
1.07
1.06
1.06

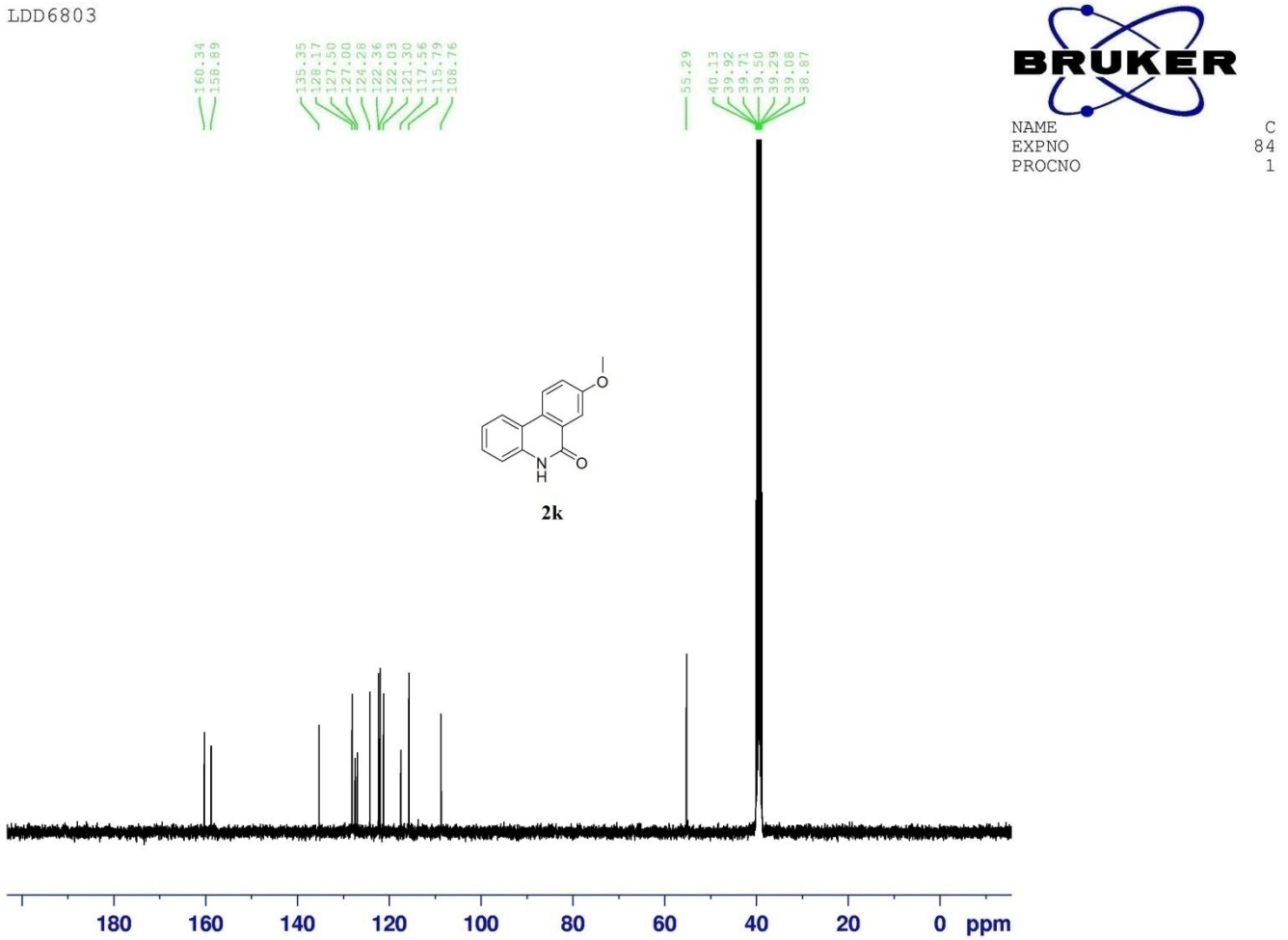

7189
160.75
137.61
133.67
133.48
133.01
128.28
127.46
125.46
125.25
122.78
122.12
116.53
115.26
40.00
39.83
39.67
39.50
39.33
39.17
39.00
Cl
N
H
O
2i
200
180
160
140
120
100
80
60
40
20
0
ppm
NAME NMR
EXPNO 7189
PROCNO 1
Date_ 20120107
Time 22.12
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2416
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 297.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.57 usec
PL1 0.00 dB
PL1W 83.39463043 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7578552 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

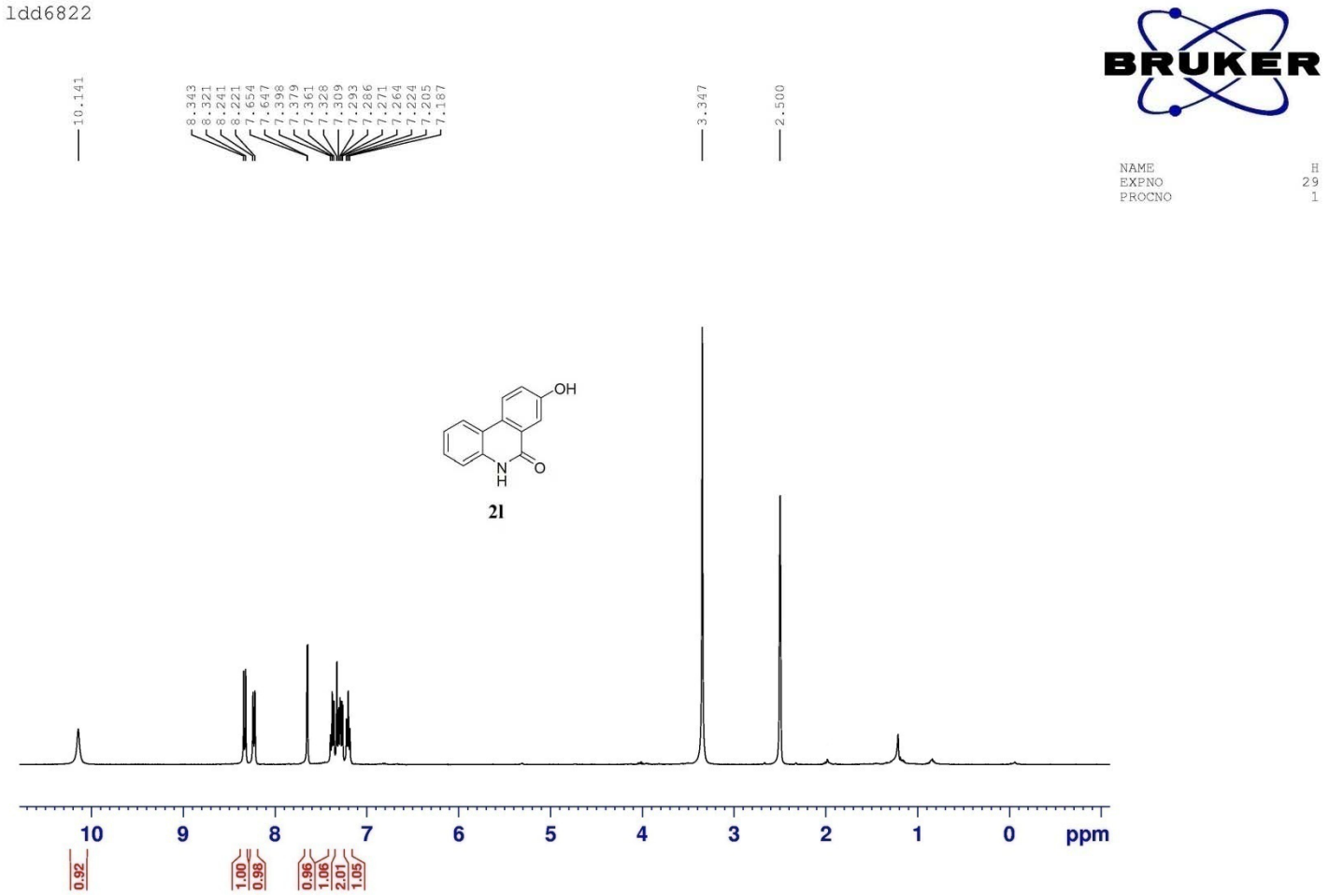

ldd7190
11.624
8.398
8.378
8.344
8.324
8.120
7.681
7.678
7.660
7.470
7.451
7.433
7.355
7.336
7.256
7.237
7.219
3.349
2.500
2.480
-0.011
BRUKER
NAME H
EXPNO 49
PROCNO 1
N
H
O
2j
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.94
1.00
1.01
0.97
1.00
1.01
1.01
1.05
3.01

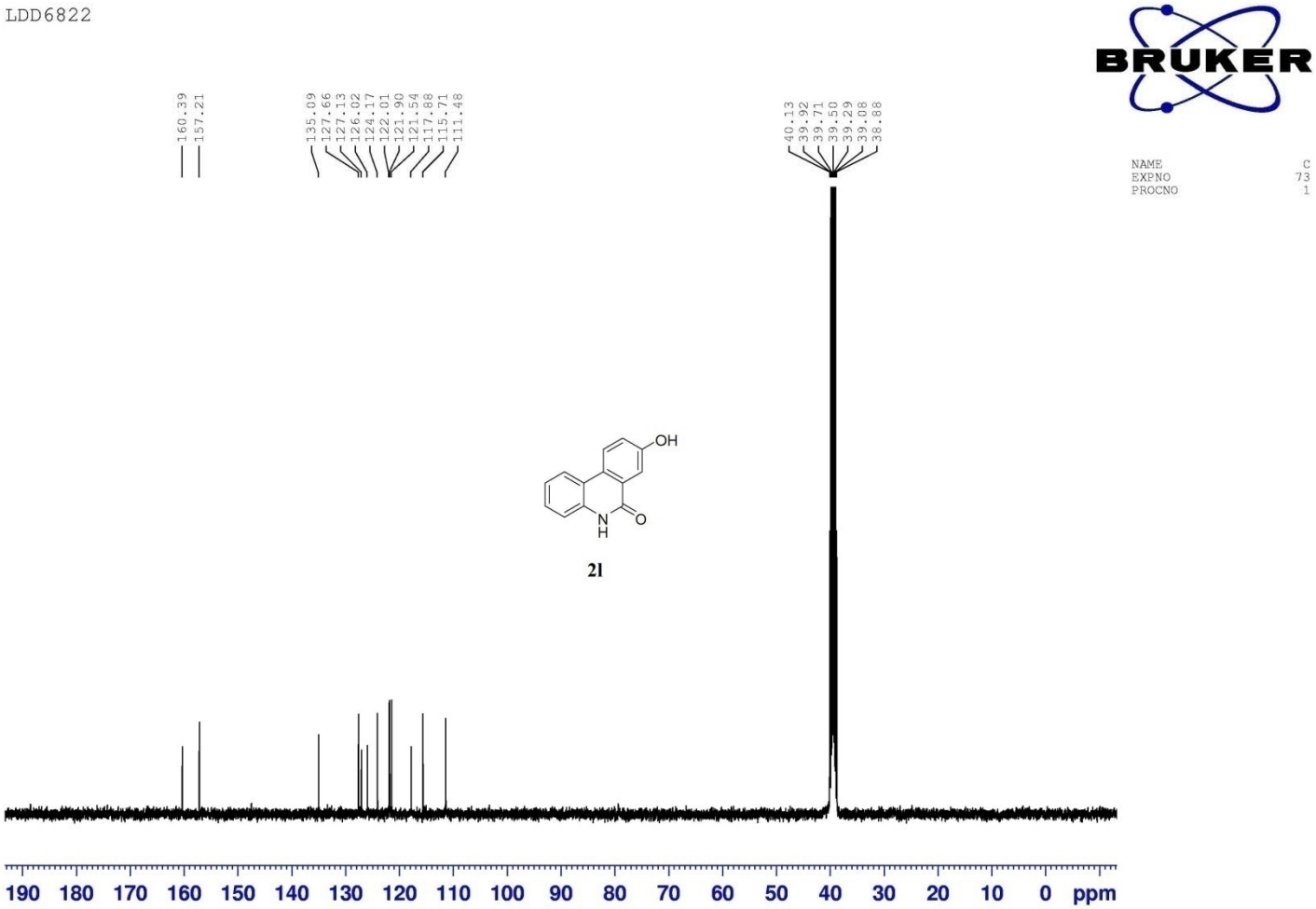


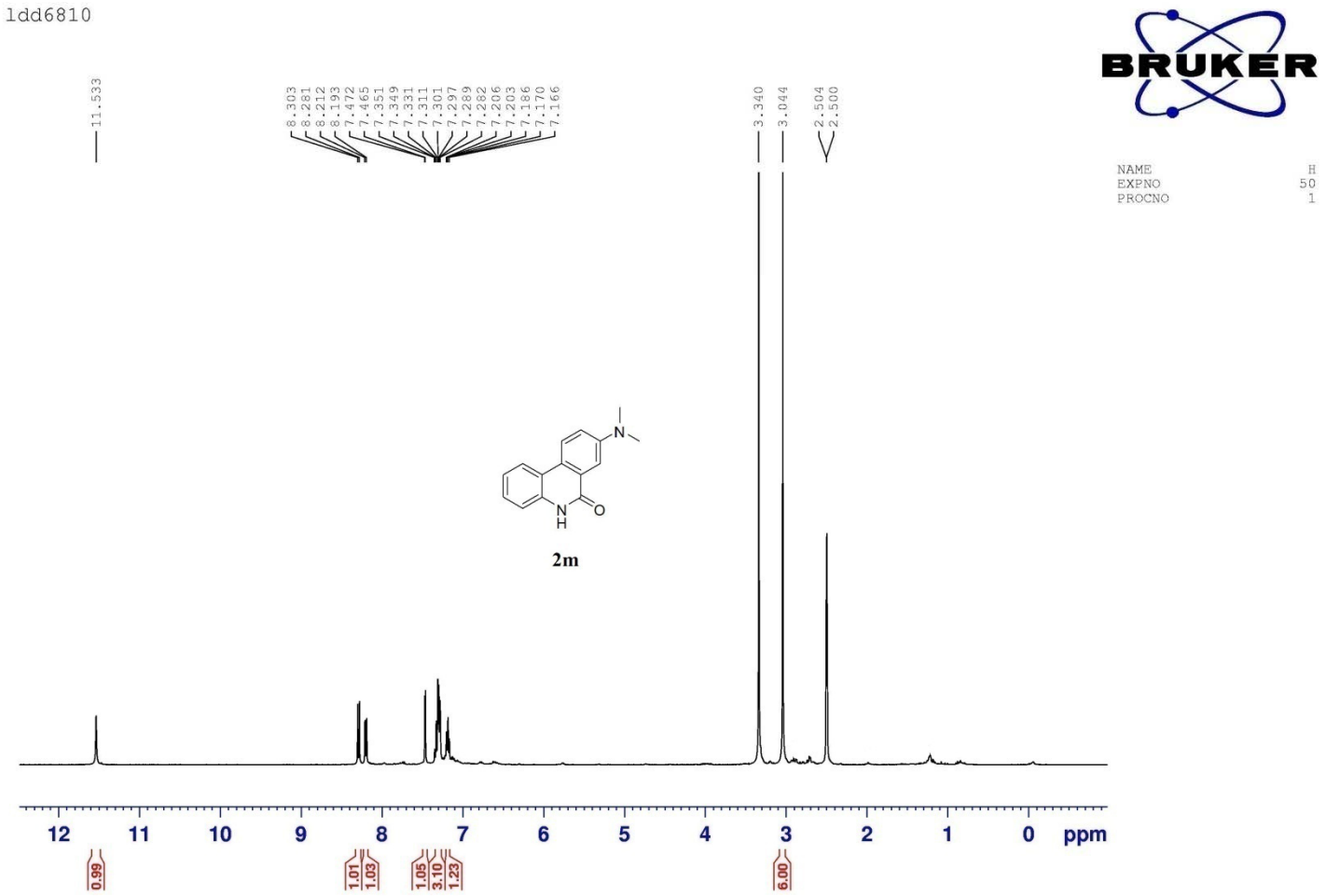
NAME	NMR
EXPNO	7190
PROCNO	1
Date_	20120109
Time	15.52
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	DMSO
NS	1024
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	297.7 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.57 usec
PL1	0.00 dB
PL1W	83.39463043 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7578556 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

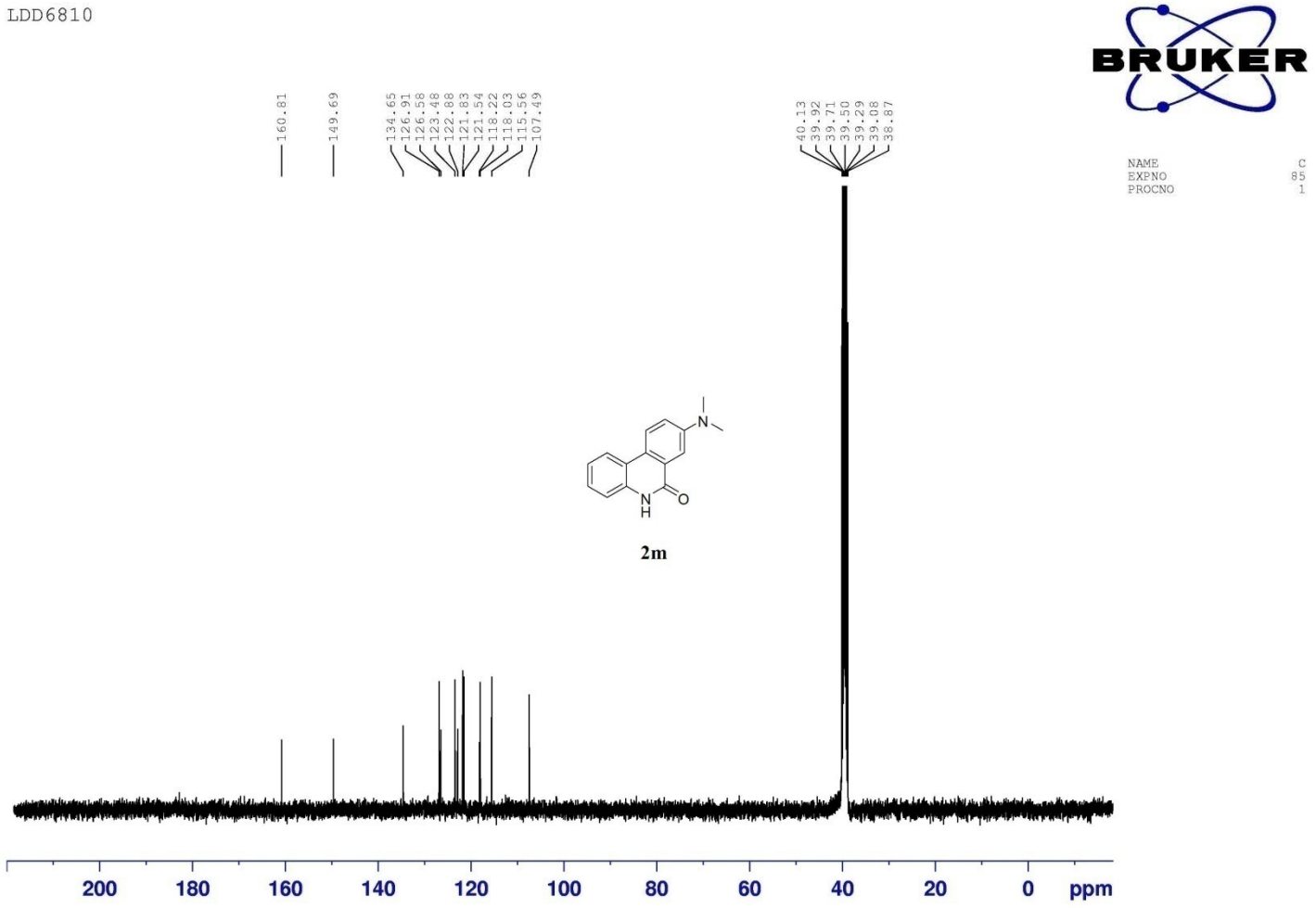

ldd6803
11.711
8.462
8.444
8.421
8.305
8.285
7.759
7.752
7.455
7.448
7.443
7.440
7.433
7.425
7.405
7.402
7.351
7.333
7.331
7.254
7.251
7.234
7.216
7.213
3.907
3.343
2.504
2.500
NAME H
EXPNO 2
PROCNO 1
2k
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.88
1.01
1.00
0.99
2.10
1.06
1.13
3.09

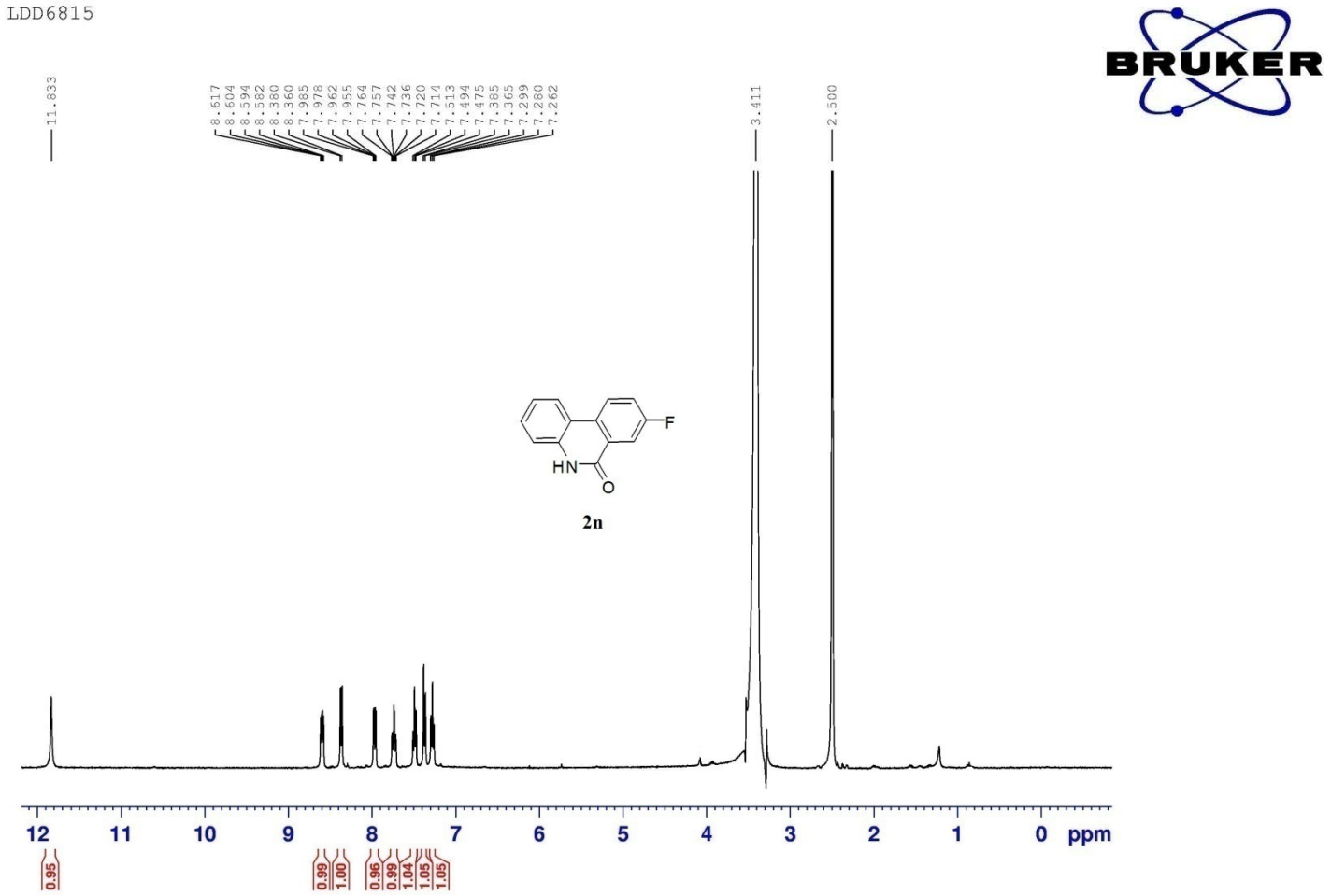

LDD6803
160.34
158.89
135.35
128.17
127.50
127.00
124.28
122.36
122.03
121.30
117.56
115.79
108.76
55.29
40.13
39.92
39.71
39.50
39.29
39.08
38.87
BRUKER
NAME C
EXPNO 84
PROCNO 1
O
N
H
O
2k
180
160
140
120
100
80
60
40
20
0
ppm

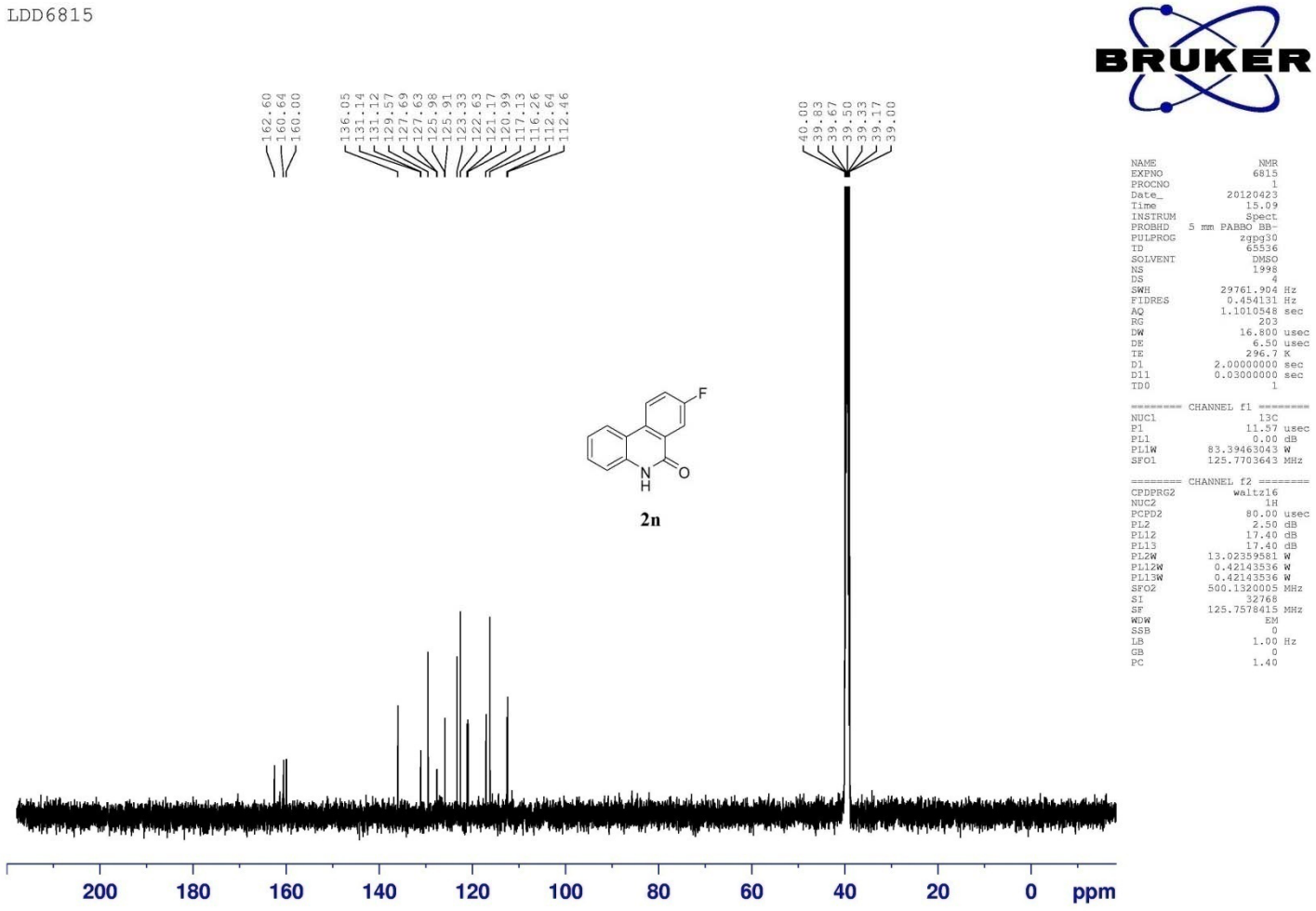

ldd6822
10.141
8.343
8.321
8.241
8.221
7.654
7.647
7.398
7.379
7.361
7.328
7.309
7.293
7.286
7.271
7.264
7.224
7.205
7.187
3.347
2.500
BRUKER
NAME H
EXPNO 29
PROCNO 1
OH
N
H
O
21
10
9
8
7
6
5
4
3
2
1
0
ppm
0.92
1.00
0.98
0.96
1.06
2.01
1.05


LDD6822
160.39
157.21
135.09
127.66
127.13
126.02
124.17
122.01
121.90
121.54
117.88
115.71
111.48
40.13
39.92
39.71
39.50
39.29
39.08
38.88
BRUKER
NAME C
EXPNO 73
PROCNO 1
OH
N
H
O
2l
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


ldd6810
11.533
8.303
8.281
8.212
8.193
7.472
7.465
7.351
7.349
7.331
7.311
7.301
7.297
7.289
7.282
7.206
7.203
7.186
7.170
7.166
3.340
3.044
2.504
2.500
BRUKER
NAME H
EXPNO 50
PROCNO 1
2m
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.99
1.01
1.03
1.05
3.10
1.23
6.00

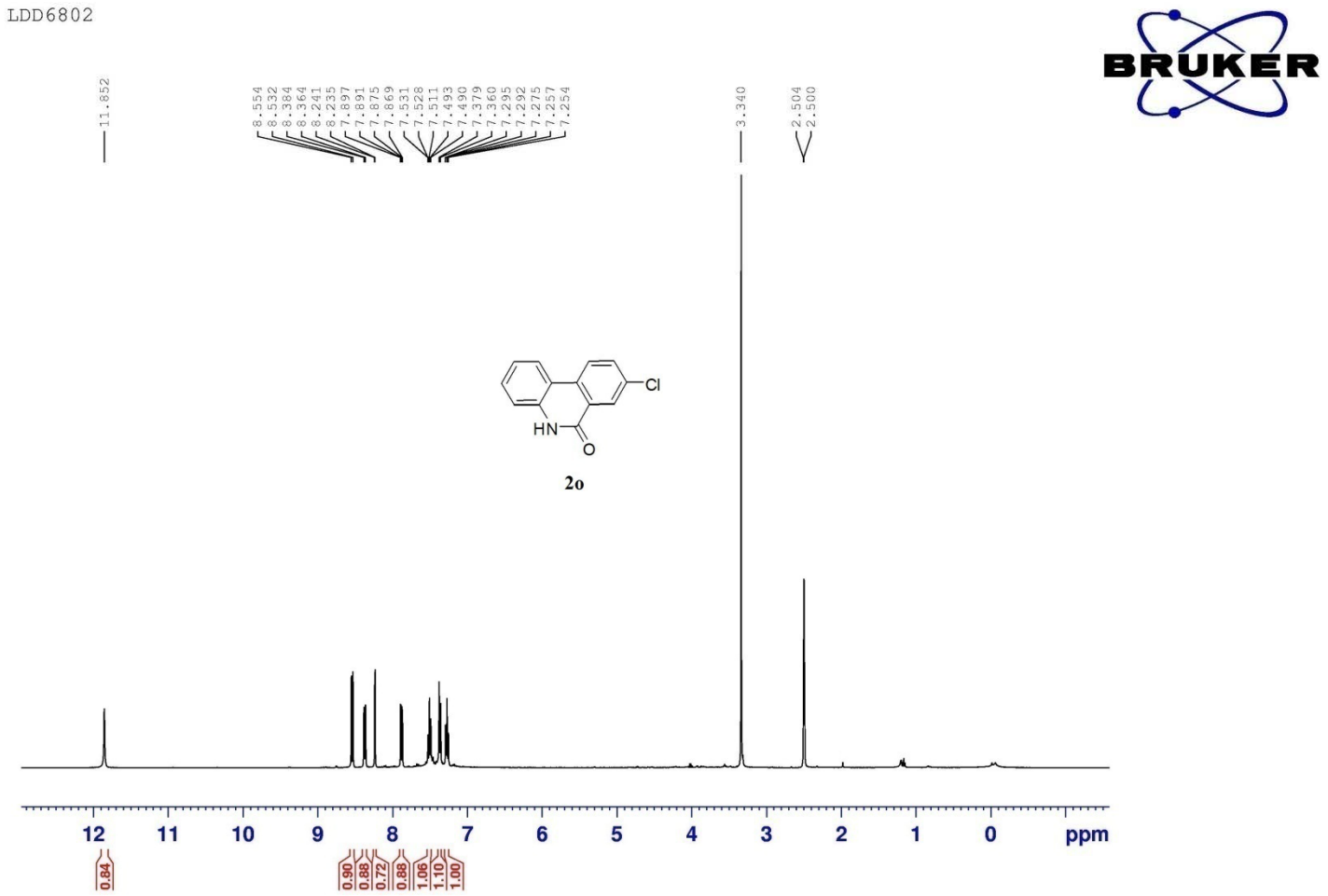


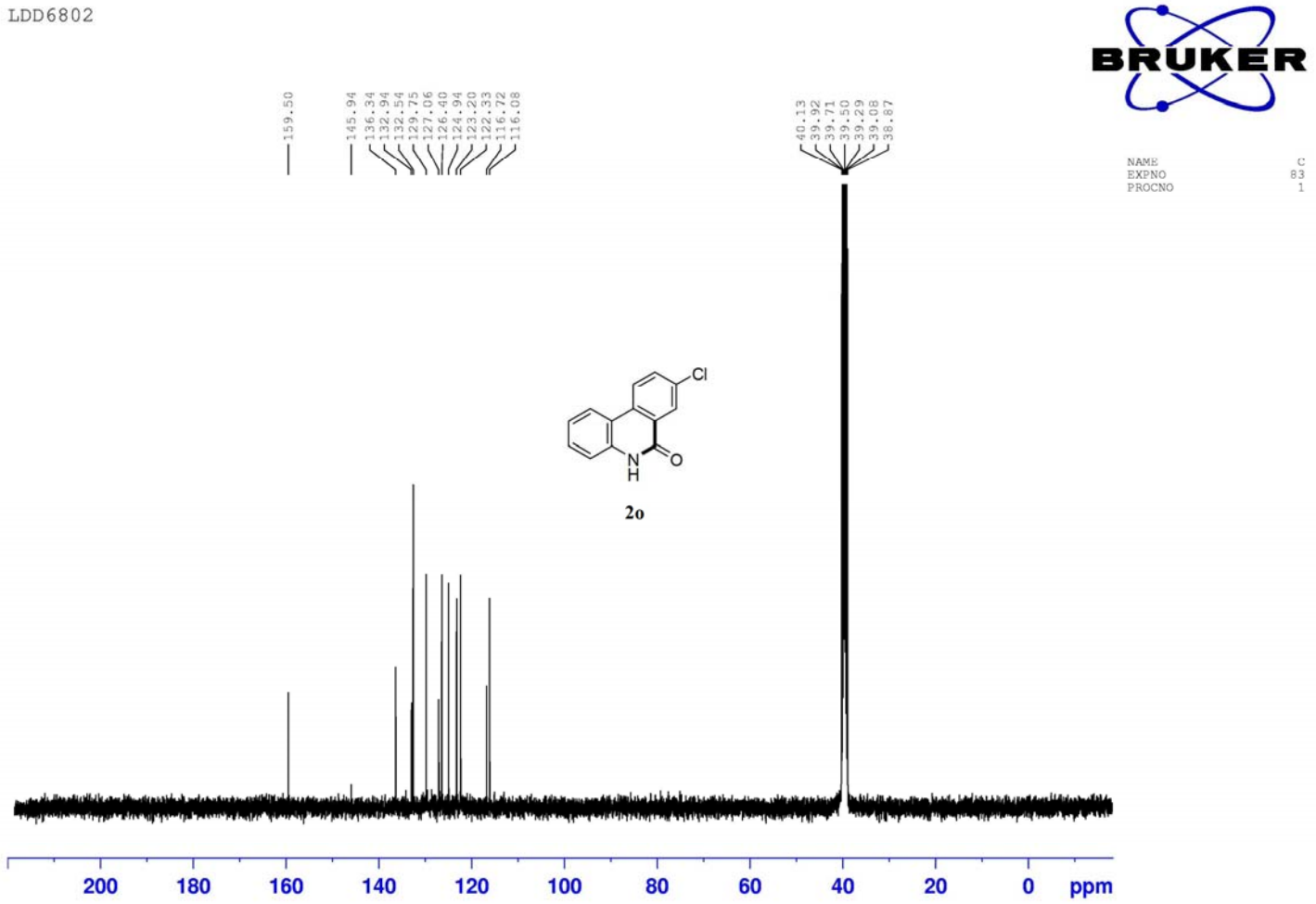


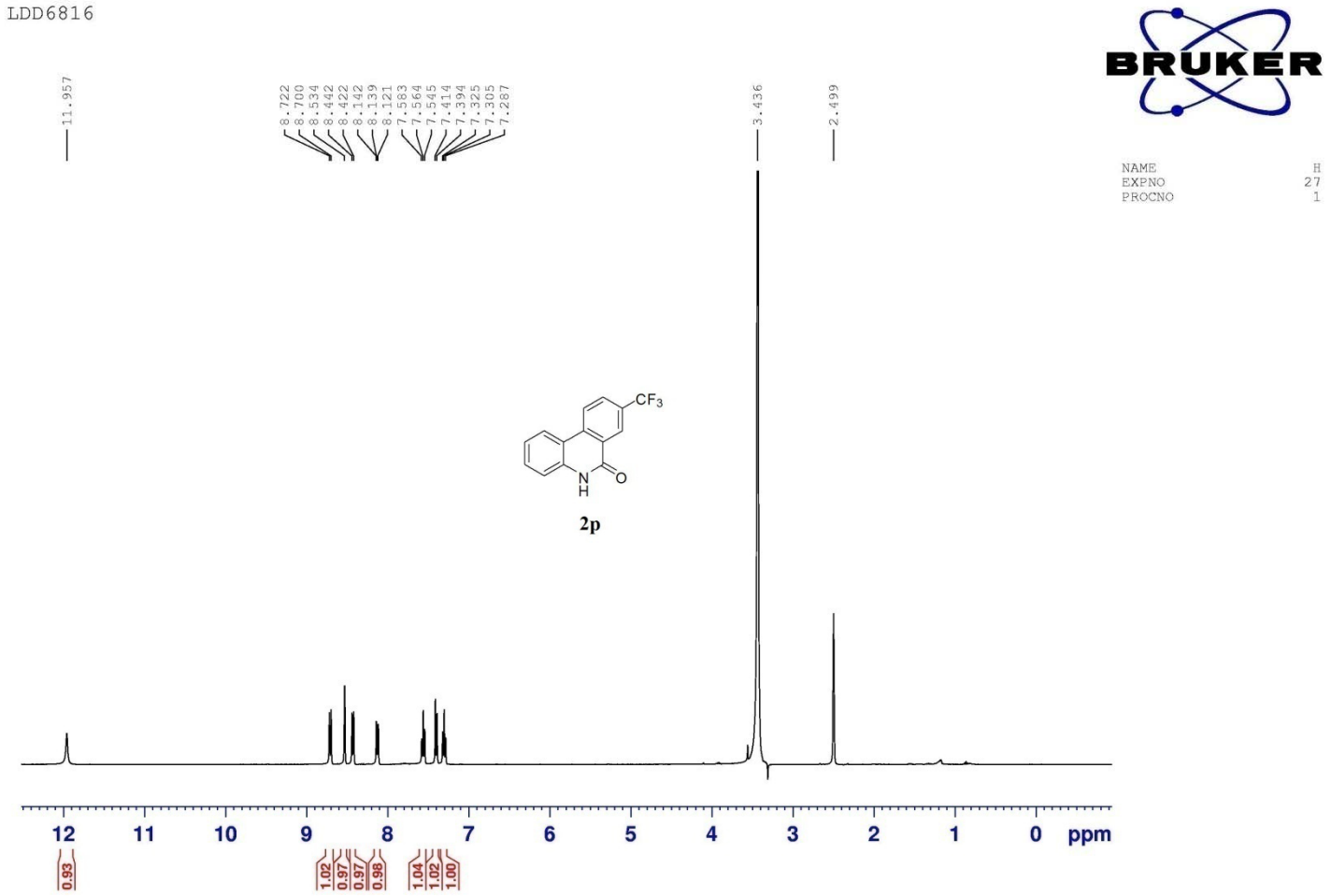


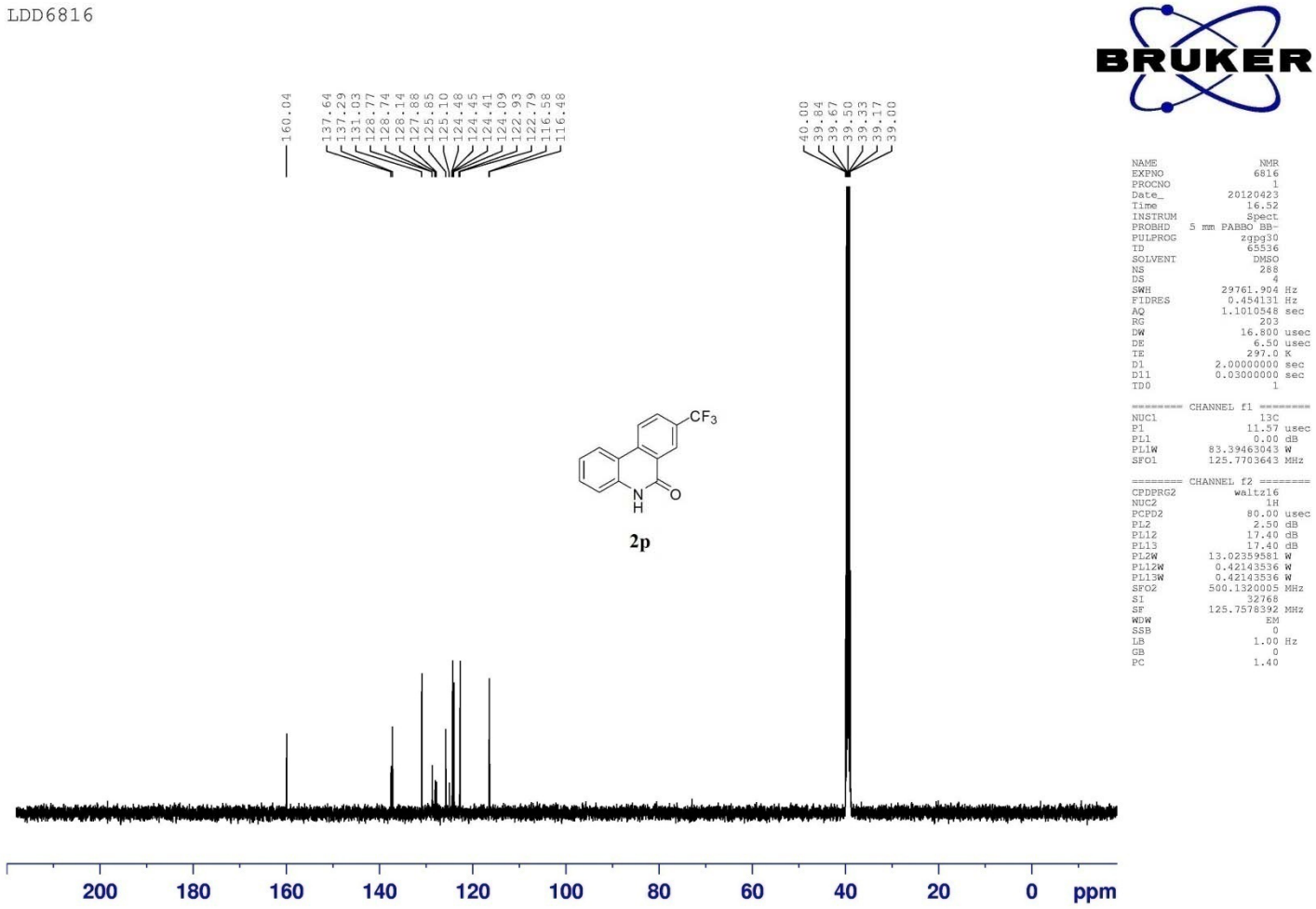
BRUKER

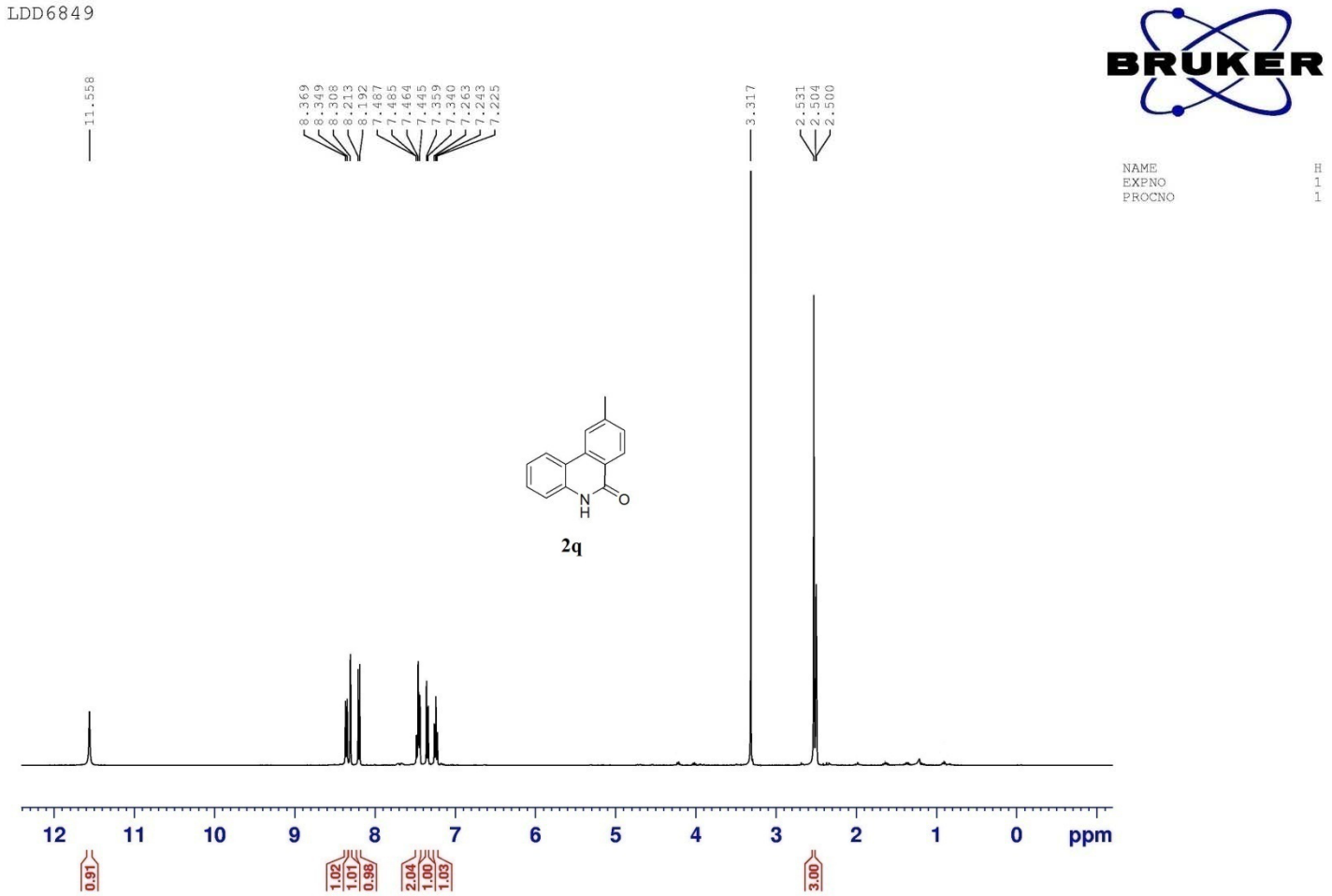
NAME	NMR
EXPNO	6815
PROCNO	1
Date_	20120423
Time	15.09
INSTRUM	Spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	DMSO
NS	1998
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	296.7 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.57 usec
PL1	0.00 dB
PL1W	83.39463043 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7578415 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

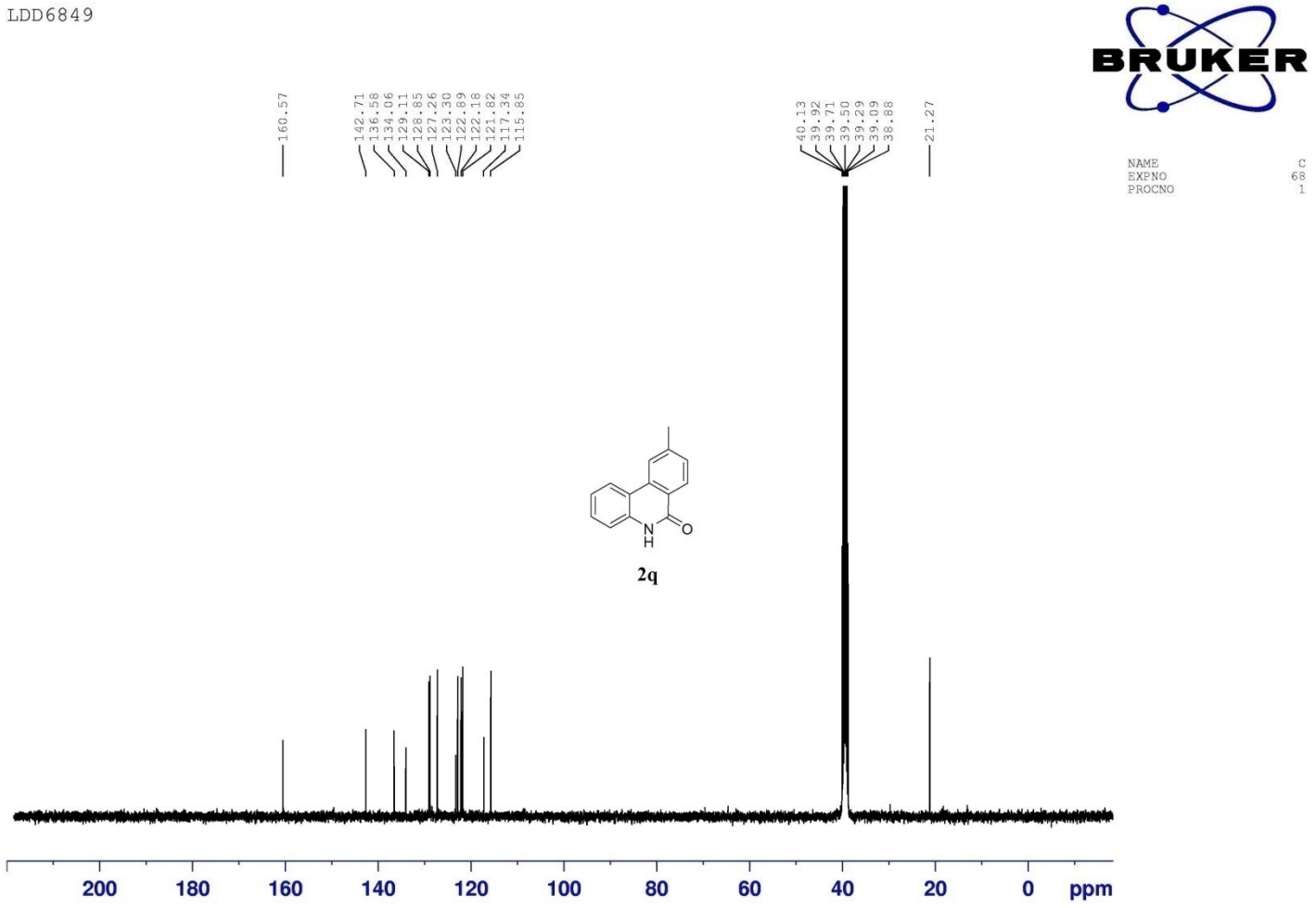

LDD6802
11.852
8.554
8.532
8.384
8.364
8.241
8.235
7.897
7.891
7.875
7.869
7.531
7.528
7.511
7.493
7.490
7.379
7.360
7.295
7.292
7.275
7.257
7.254
3.340
2.504
2.500
Cl
HN
O
2o
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.84
0.90
0.88
0.72
0.88
1.06
1.10
1.00

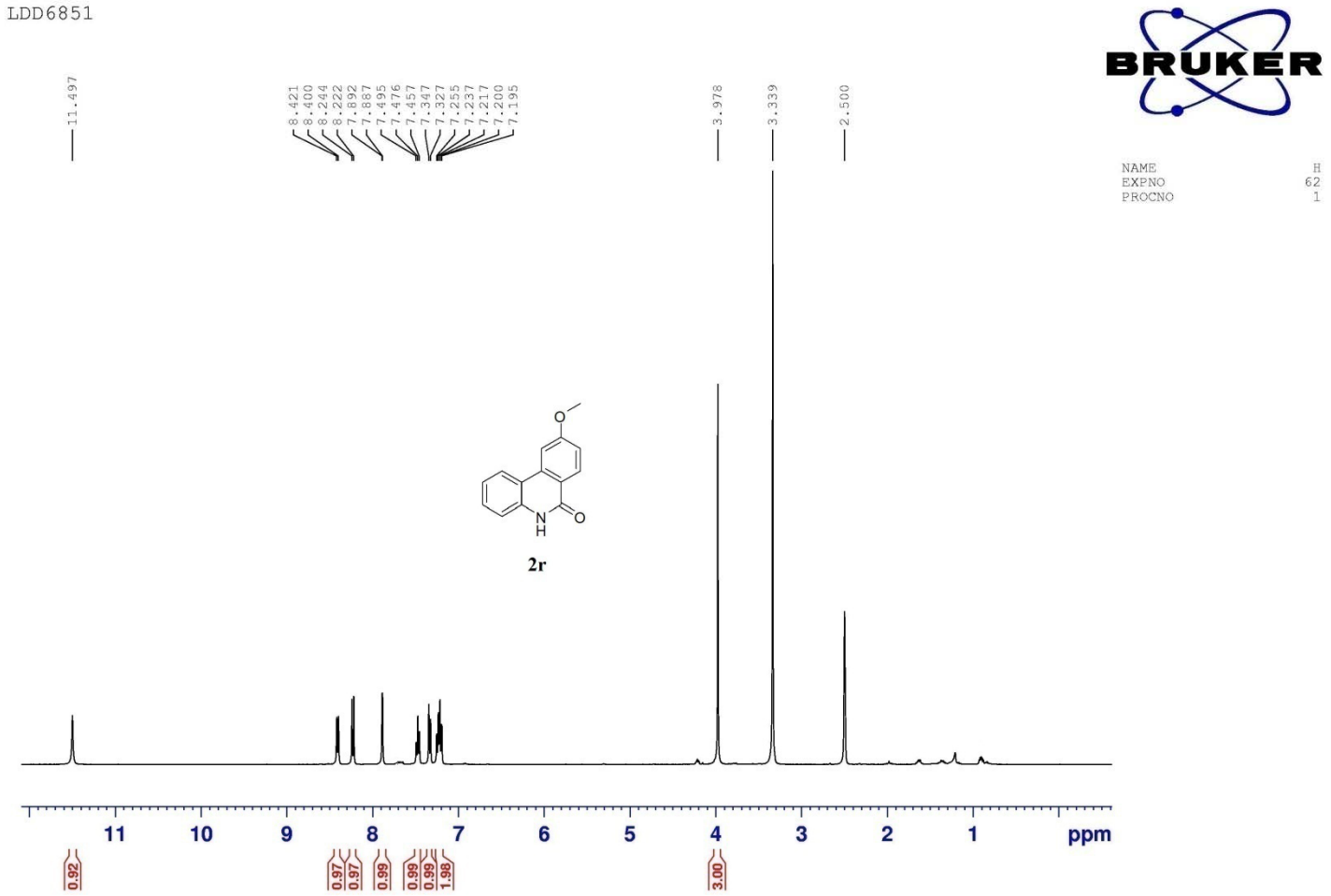

LDD6802
159.50
145.94
136.34
132.94
132.54
129.75
127.06
126.40
124.94
123.20
122.33
116.72
116.08
40.13
39.92
39.71
39.50
39.29
39.08
38.87
NAME C
EXPNO 83
PROCNO 1
Cl
N
H
O
2o
200
180
160
140
120
100
80
60
40
20
0
ppm

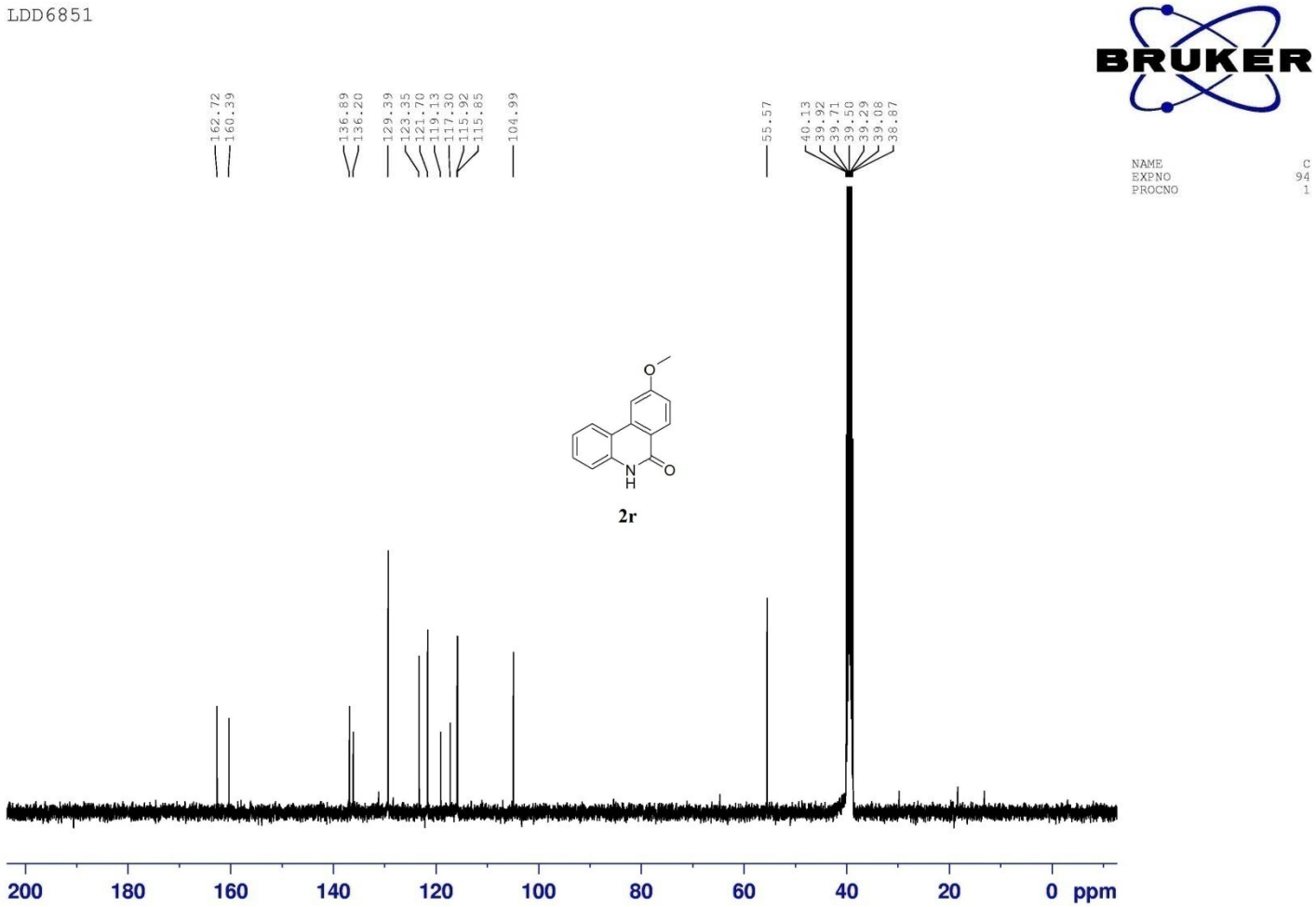

LDD6816
11.957
8.722
8.700
8.534
8.442
8.422
8.142
8.139
8.121
7.583
7.564
7.545
7.414
7.394
7.325
7.305
7.287
3.436
2.499
NAME H
EXPNO 27
PROCNO 1
CF3
N
H
O
2p
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.93
1.02
0.97
0.97
0.98
1.04
1.02
1.00

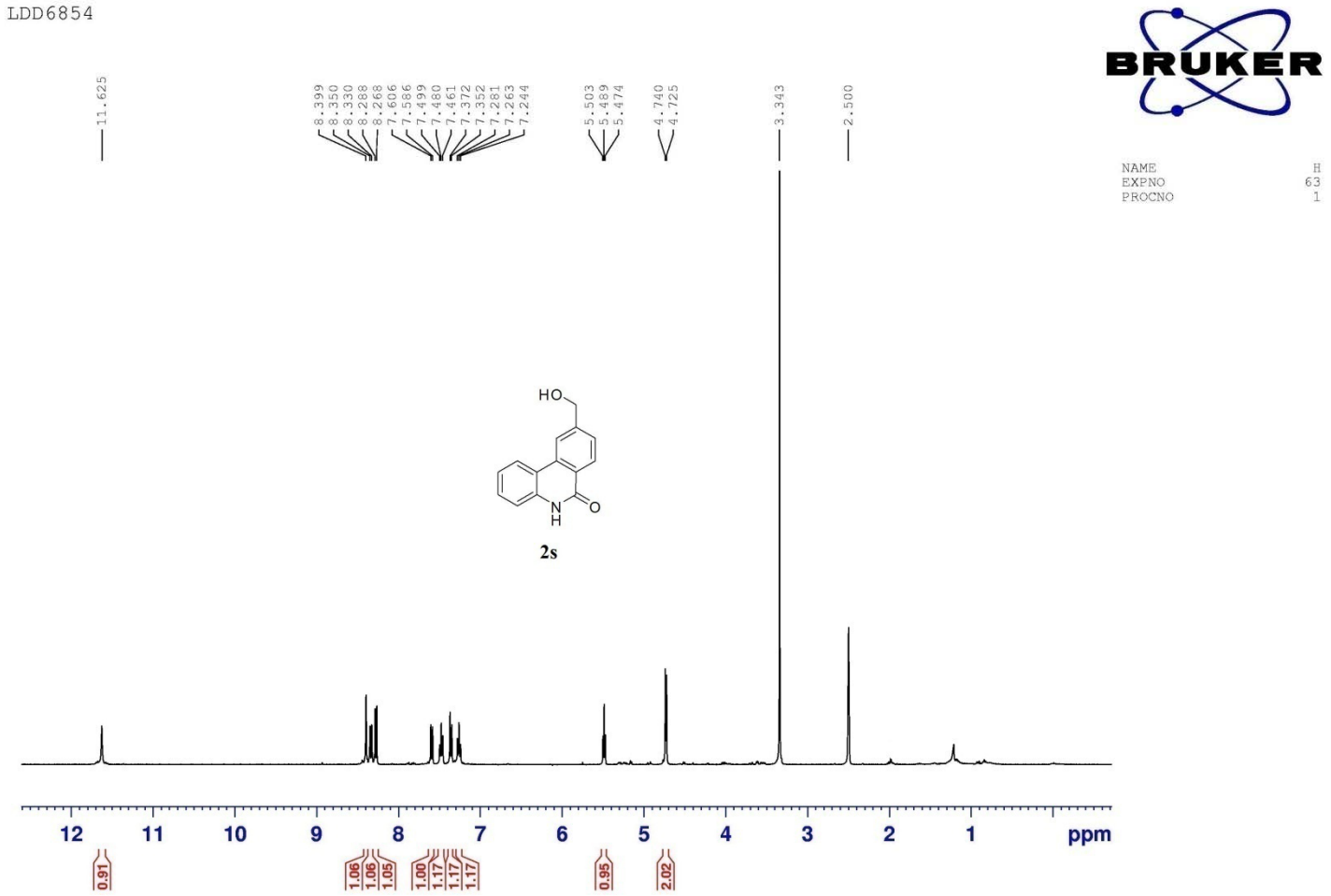

LDD6816
160.04
137.64
137.29
131.03
128.77
128.74
128.14
127.88
125.85
125.10
124.48
124.45
124.41
124.09
122.93
122.79
116.58
116.48
40.00
39.84
39.67
39.50
39.33
39.17
39.00
CF3
N
H
O
2p
200
180
160
140
120
100
80
60
40
20
0
ppm
NAME NMR
EXPNO 6816
PROCNO 1
Date_ 20120423
Time 16.52
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 288
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 297.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.57 usec
PL1 0.00 dB
PL1W 83.39463043 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7578392 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

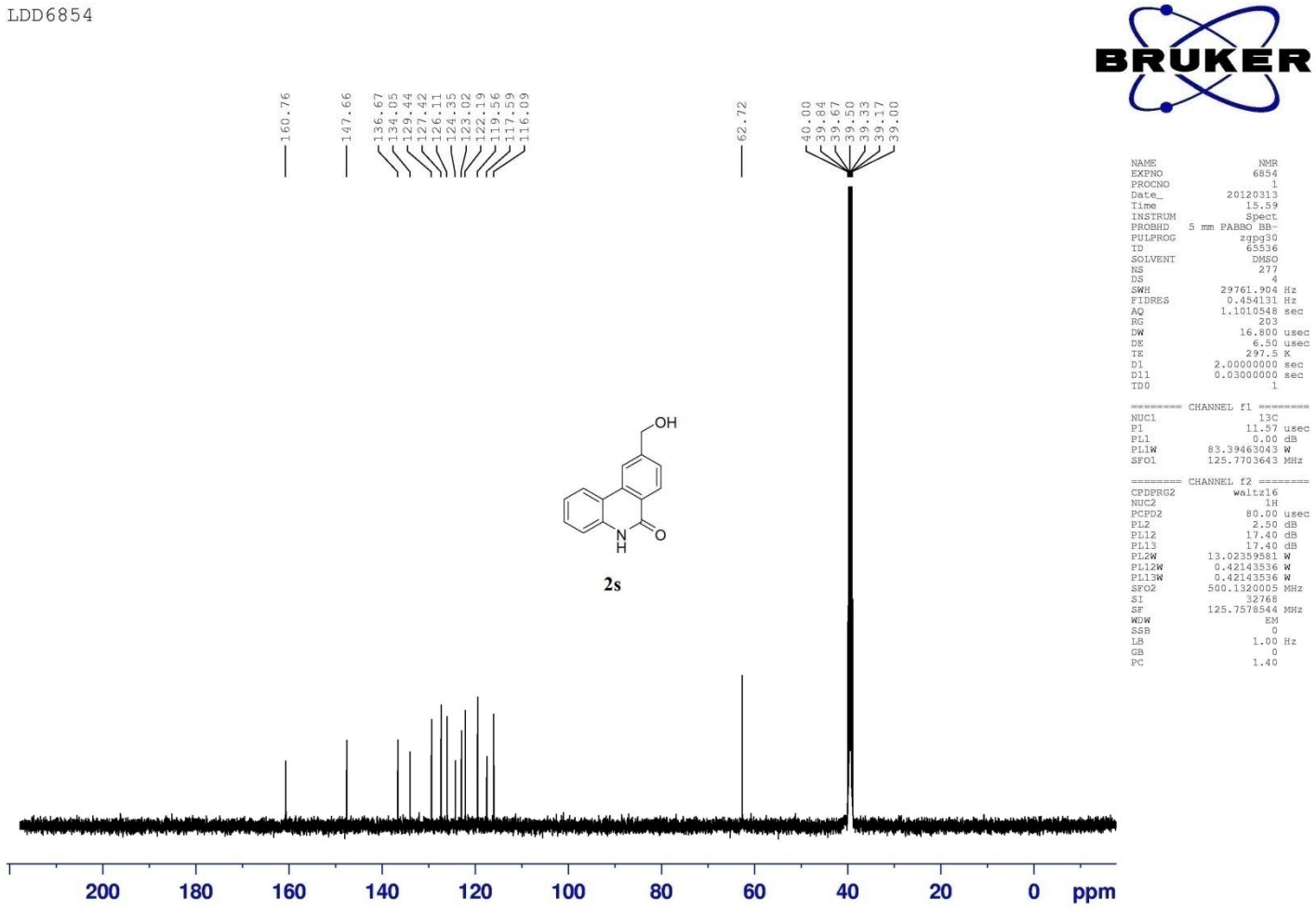

LDD6849
11.558
8.369
8.349
8.308
8.213
8.192
7.487
7.485
7.464
7.445
7.359
7.340
7.263
7.243
7.225
3.317
2.531
2.504
2.500
NAME H
EXPNO 1
PROCNO 1
2q
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.91
1.02
1.01
0.98
2.04
1.00
1.03
3.00

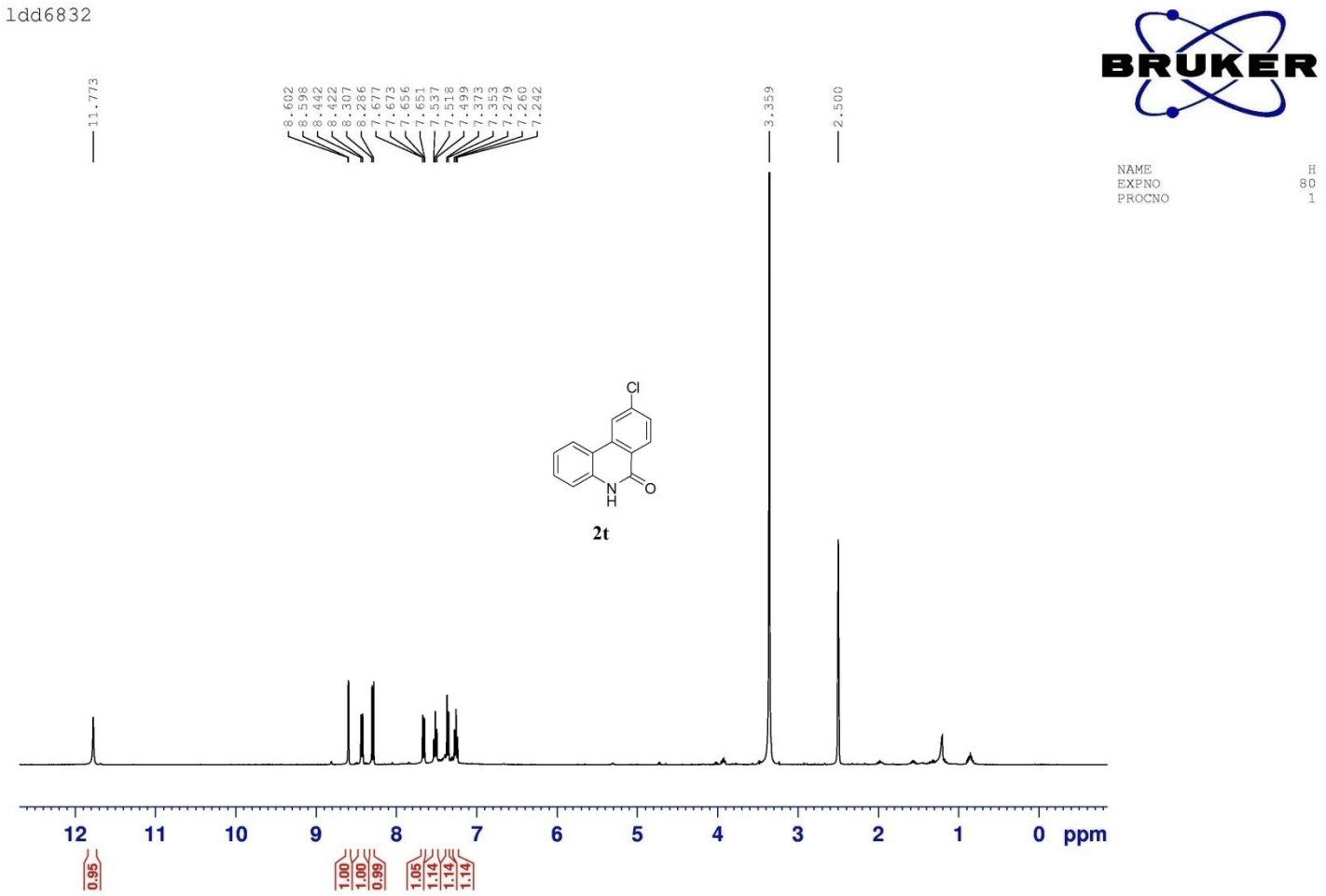


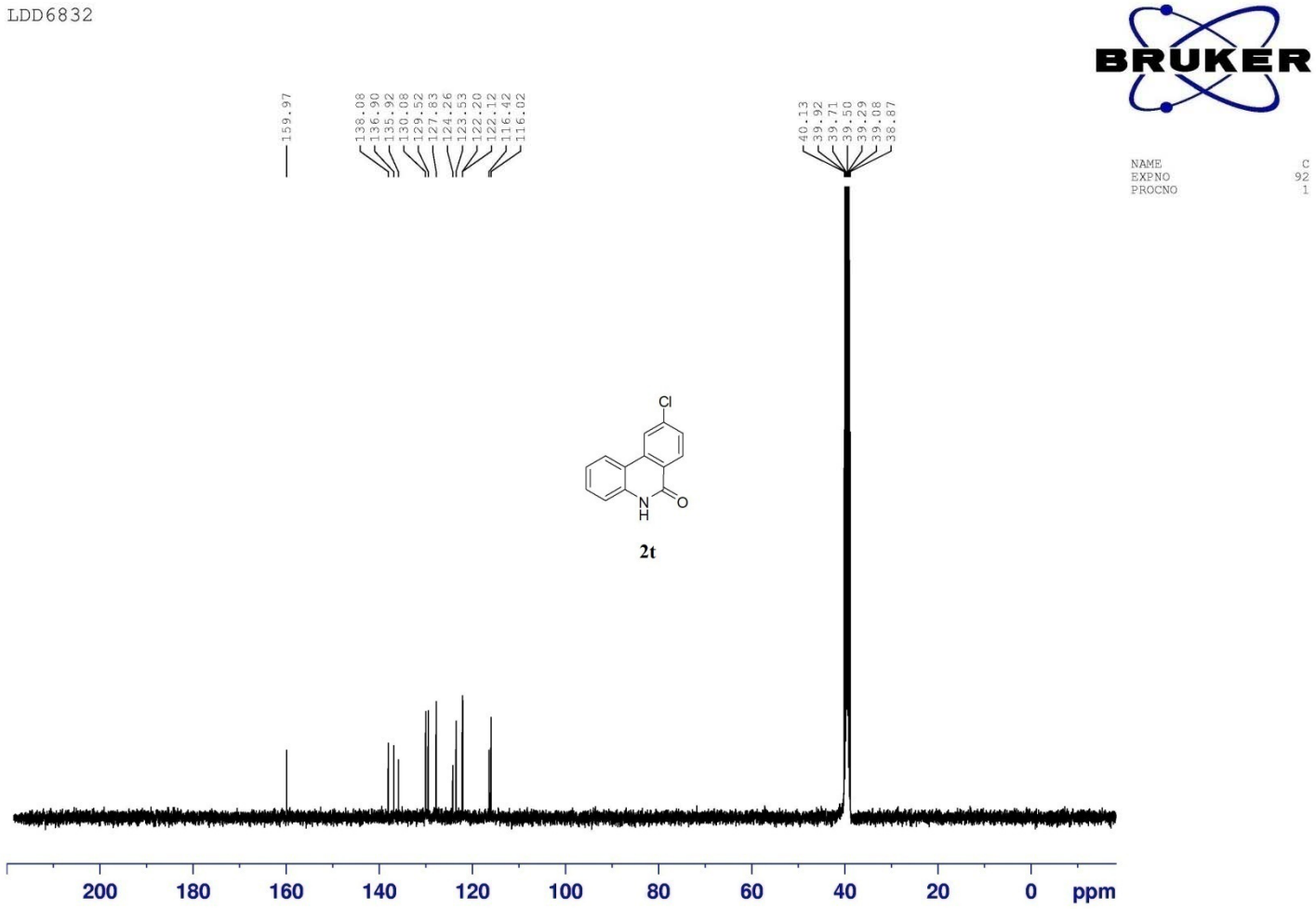

LDD6851
11.497
8.421
8.400
8.244
8.222
7.892
7.887
7.495
7.476
7.457
7.347
7.327
7.255
7.237
7.217
7.200
7.195
3.978
3.339
2.500
NAME H
EXPNO 62
PROCNO 1
O
N
H
O
2r
11
10
9
8
7
6
5
4
3
2
1
ppm
0.92
0.97
0.97
0.99
0.98
0.99
1.98
3.00

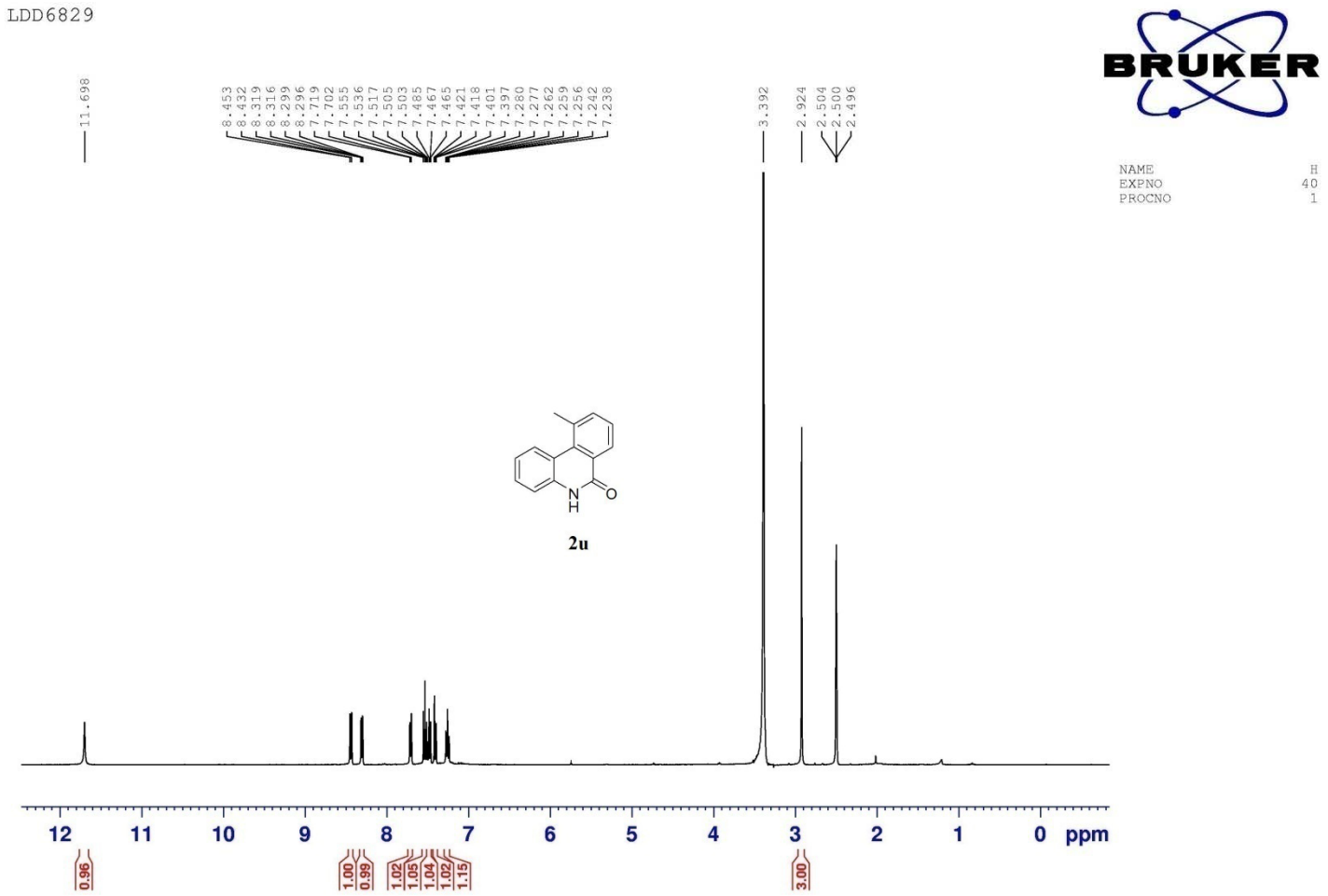

LDD6851
BRUKER
NAME C
EXPNO 94
PROCNO 1
162.72
160.39
136.89
136.20
129.39
123.35
121.70
119.13
117.30
115.92
115.85
104.99
55.57
40.13
39.92
39.71
39.50
39.29
39.08
38.87
O
N
H
O
2r
200
180
160
140
120
100
80
60
40
20
0 ppm

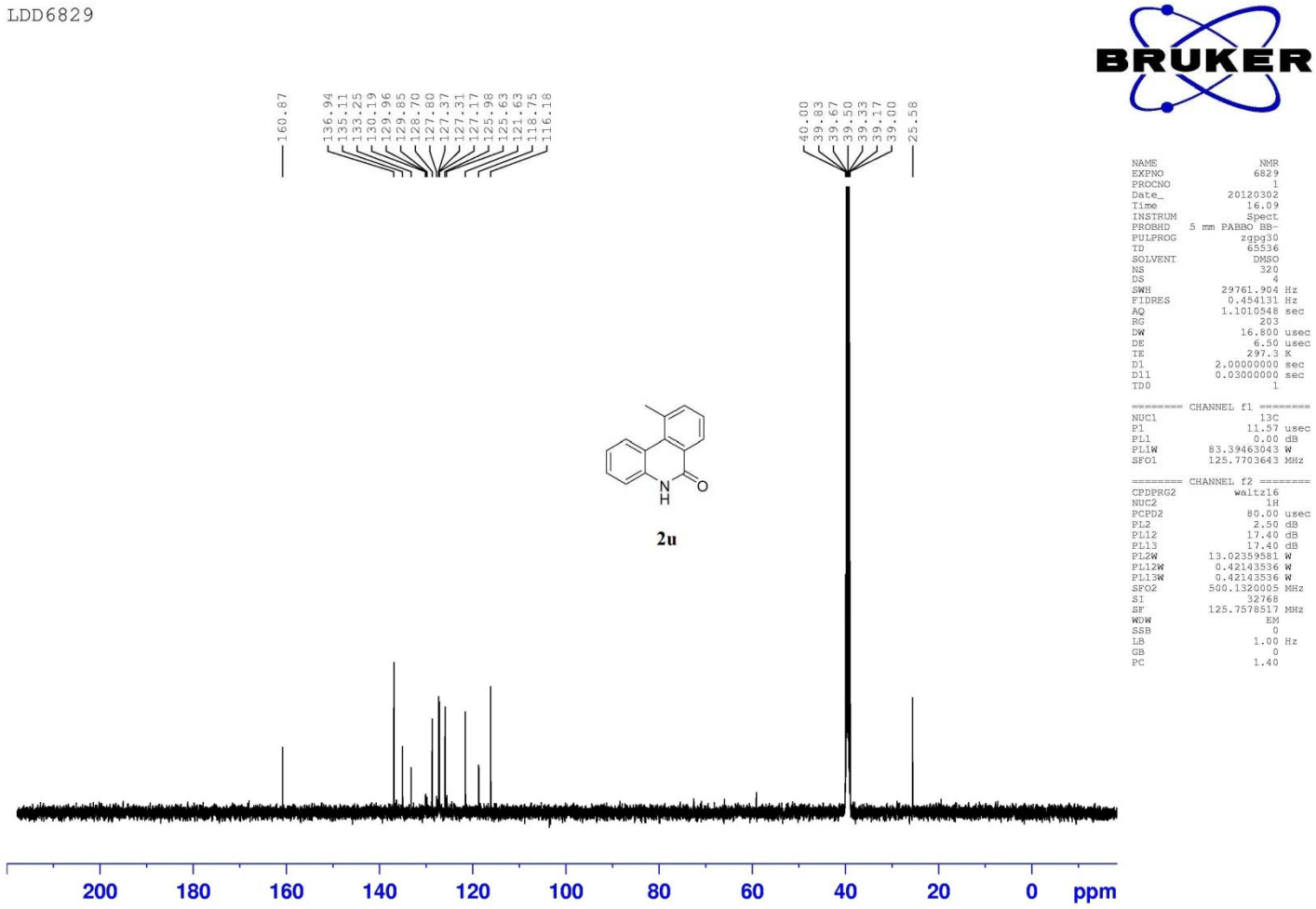

LDD6854
11.625
8.399
8.350
8.330
8.288
8.268
7.606
7.586
7.499
7.480
7.461
7.372
7.332
7.281
7.263
7.244
5.503
5.489
5.474
4.740
4.725
3.343
2.500
NAME H
EXPNO 63
PROCNO 1
HO
N
H
O
2s
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.91
1.06
1.06
1.05
1.00
1.17
1.17
1.17
0.95
2.02


LDD6854
160.76
147.66
136.67
134.05
129.44
127.42
126.11
124.35
123.02
122.19
119.56
117.59
116.09
62.72
40.00
39.84
39.67
39.50
39.33
39.17
39.00
OH
N
H
O
2s
200
180
160
140
120
100
80
60
40
20
0
ppm
NAME NMR
EXPNO 6854
PROCNO 1
Date_ 20120313
Time 15.59
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 277
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 297.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.57 usec
PL1 0.00 dB
PL1W 83.39463043 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7578544 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


ldd6832
11.773
8.602
8.598
8.442
8.422
8.307
8.286
7.677
7.673
7.656
7.651
7.537
7.518
7.499
7.373
7.353
7.279
7.260
7.242
3.359
2.500
NAME H
EXPNO 80
PROCNO 1
Cl
N
H
O
2t
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.95
1.00
1.00
0.99
1.05
1.14
1.14
1.14

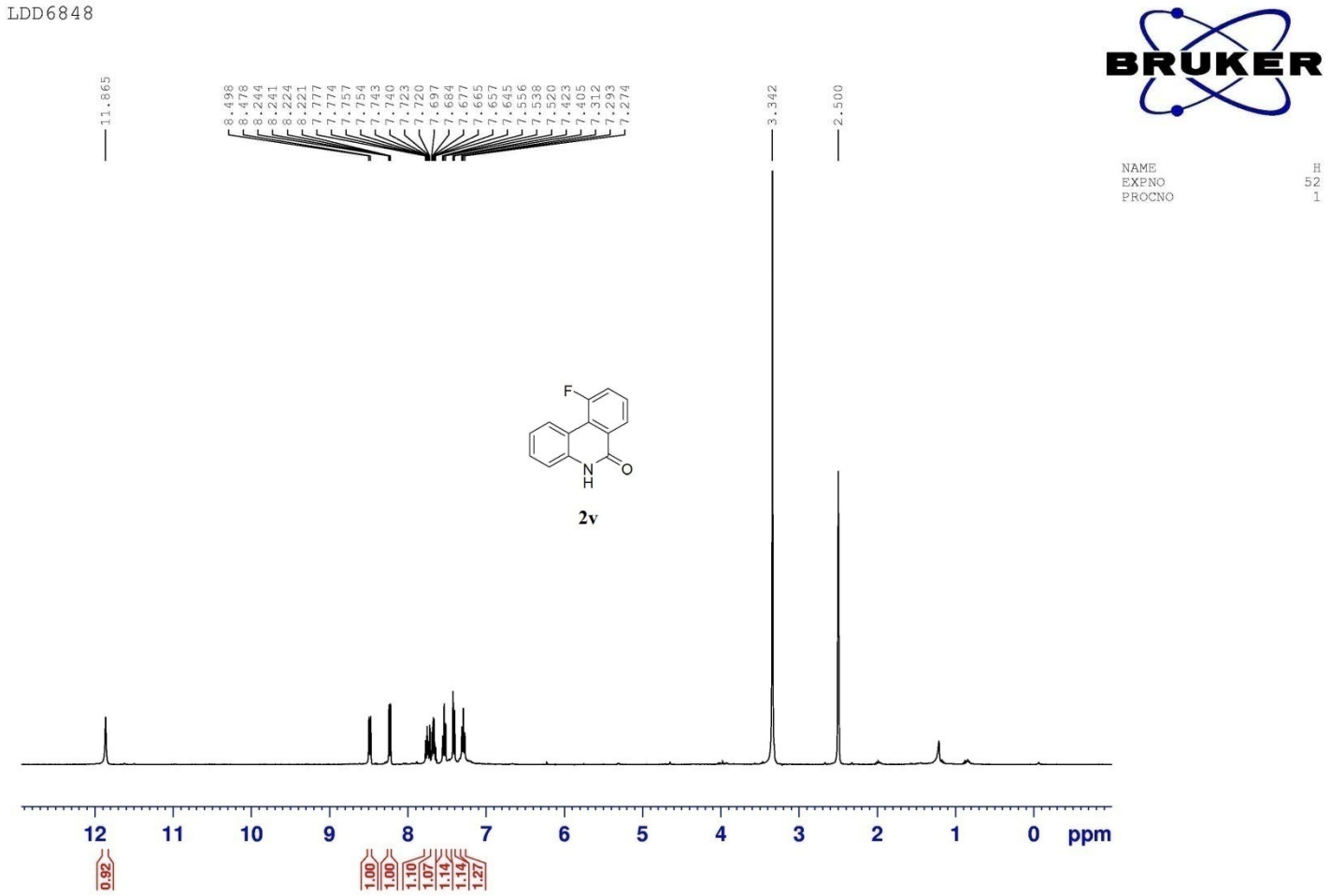


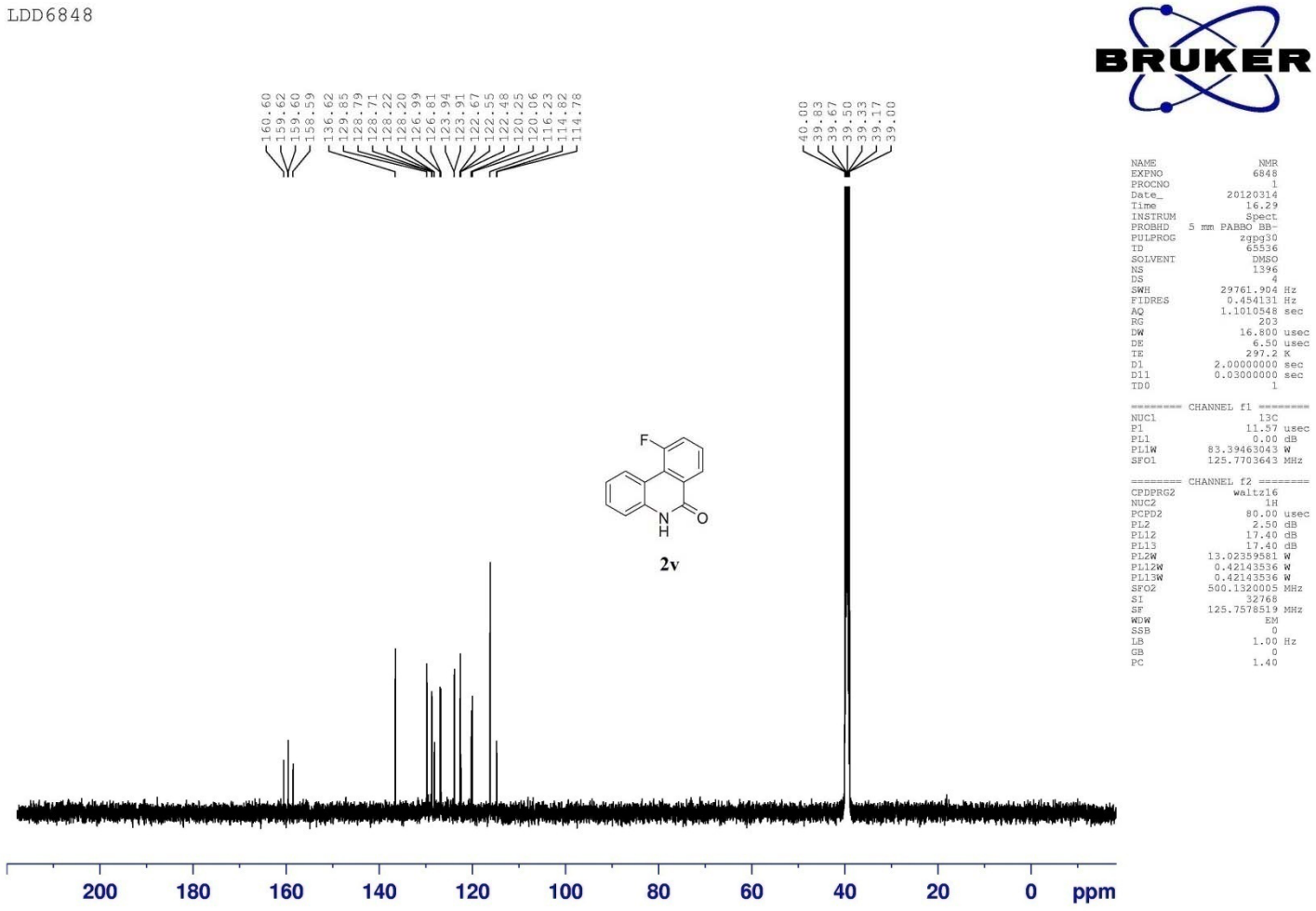

LDD6829
11.698
8.453
8.432
8.319
8.316
8.299
8.296
7.719
7.702
7.555
7.536
7.517
7.505
7.503
7.485
7.467
7.465
7.421
7.418
7.401
7.397
7.280
7.277
7.262
7.259
7.256
7.242
7.238
3.392
2.924
2.504
2.500
2.496
NAME H
EXPNO 40
PROCNO 1
2u
12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.96
1.00
0.99
1.02
1.05
1.04
1.02
1.15
3.00

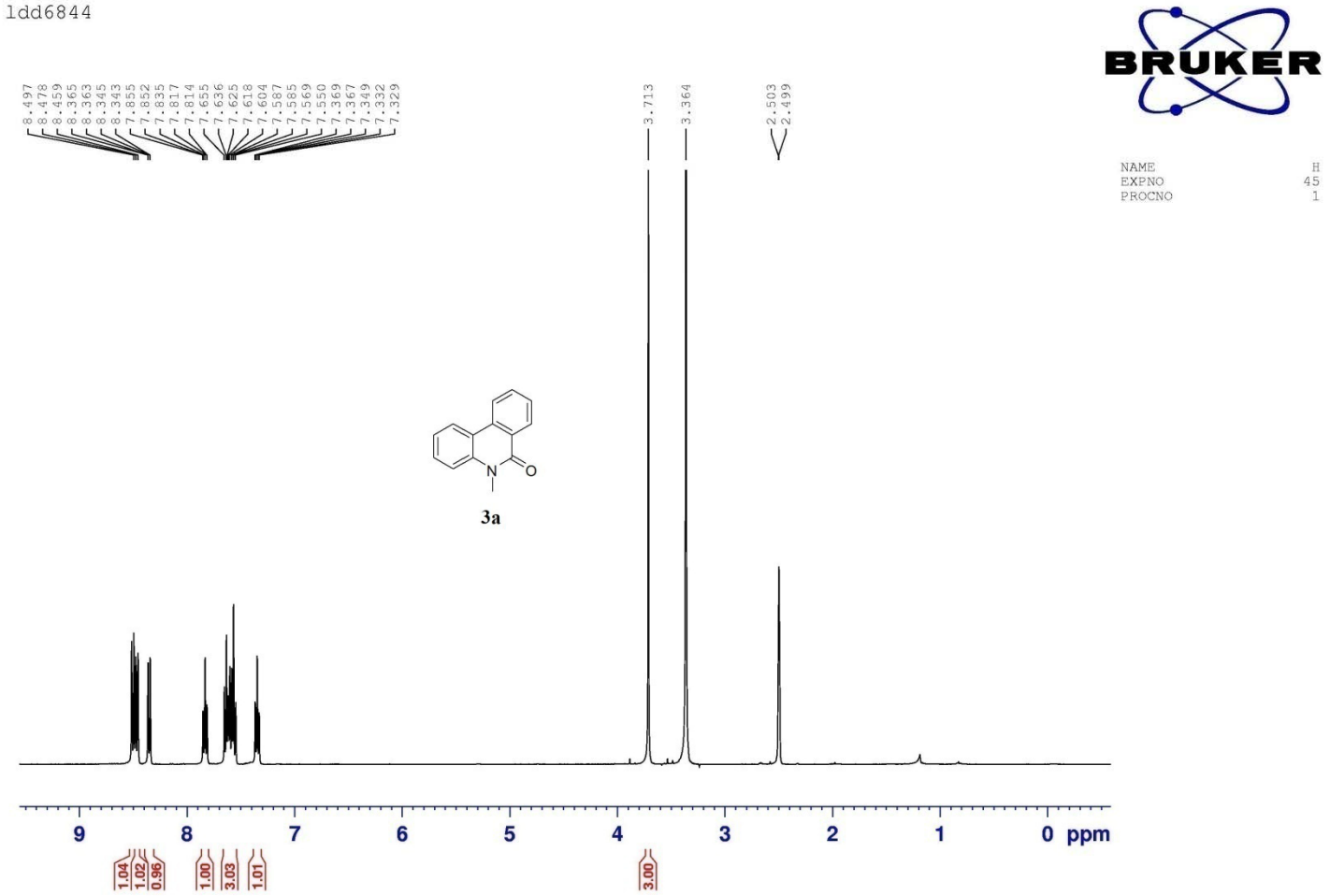


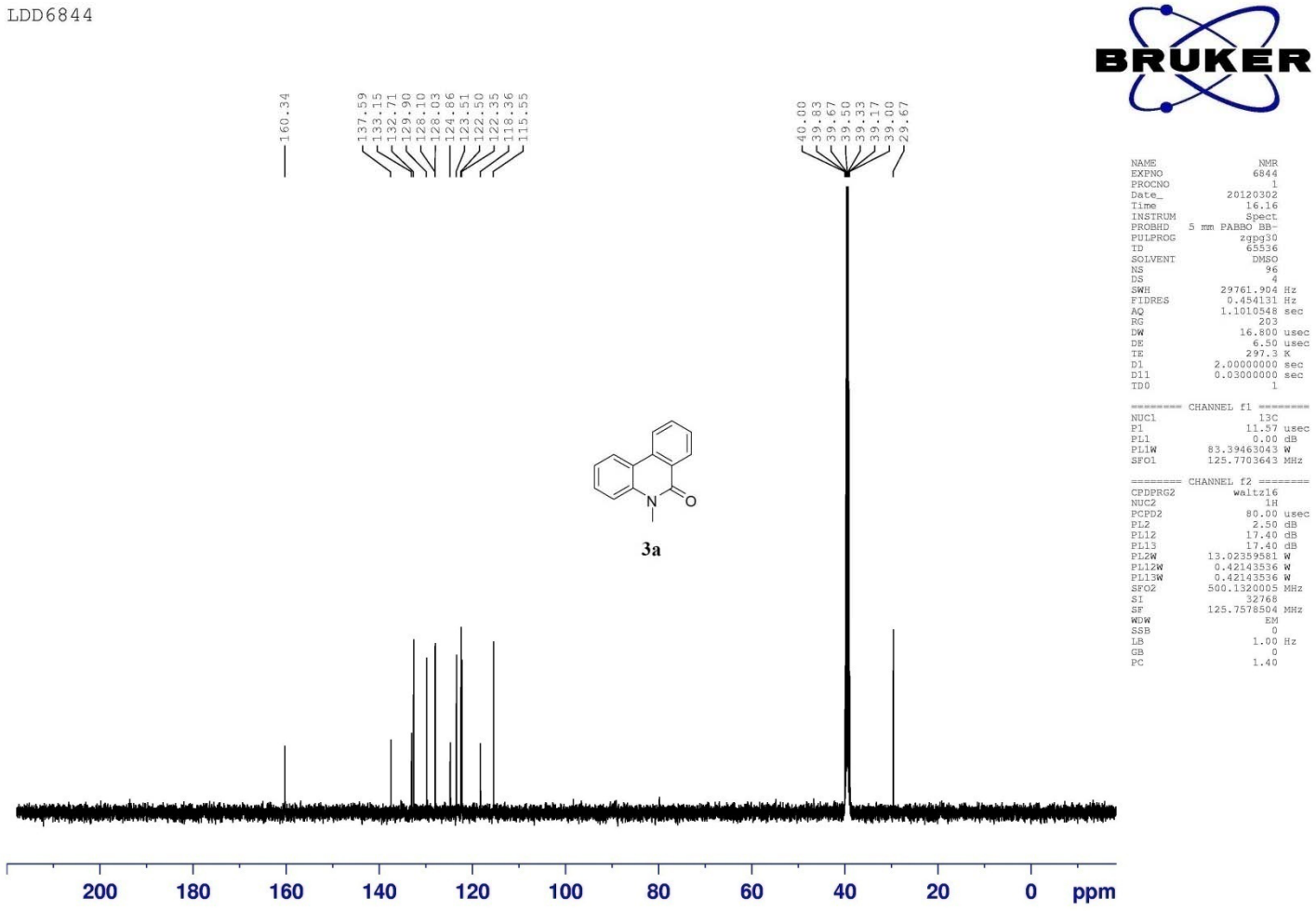
BRUKER

NAME	NMR
EXPNO	6829
PROCNO	1
Date_	20120302
Time	16.09
INSTRUM	Spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	DMSO
NS	320
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	297.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.57 usec
PL1	0.00 dB
PL1W	83.39463043 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7578517 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

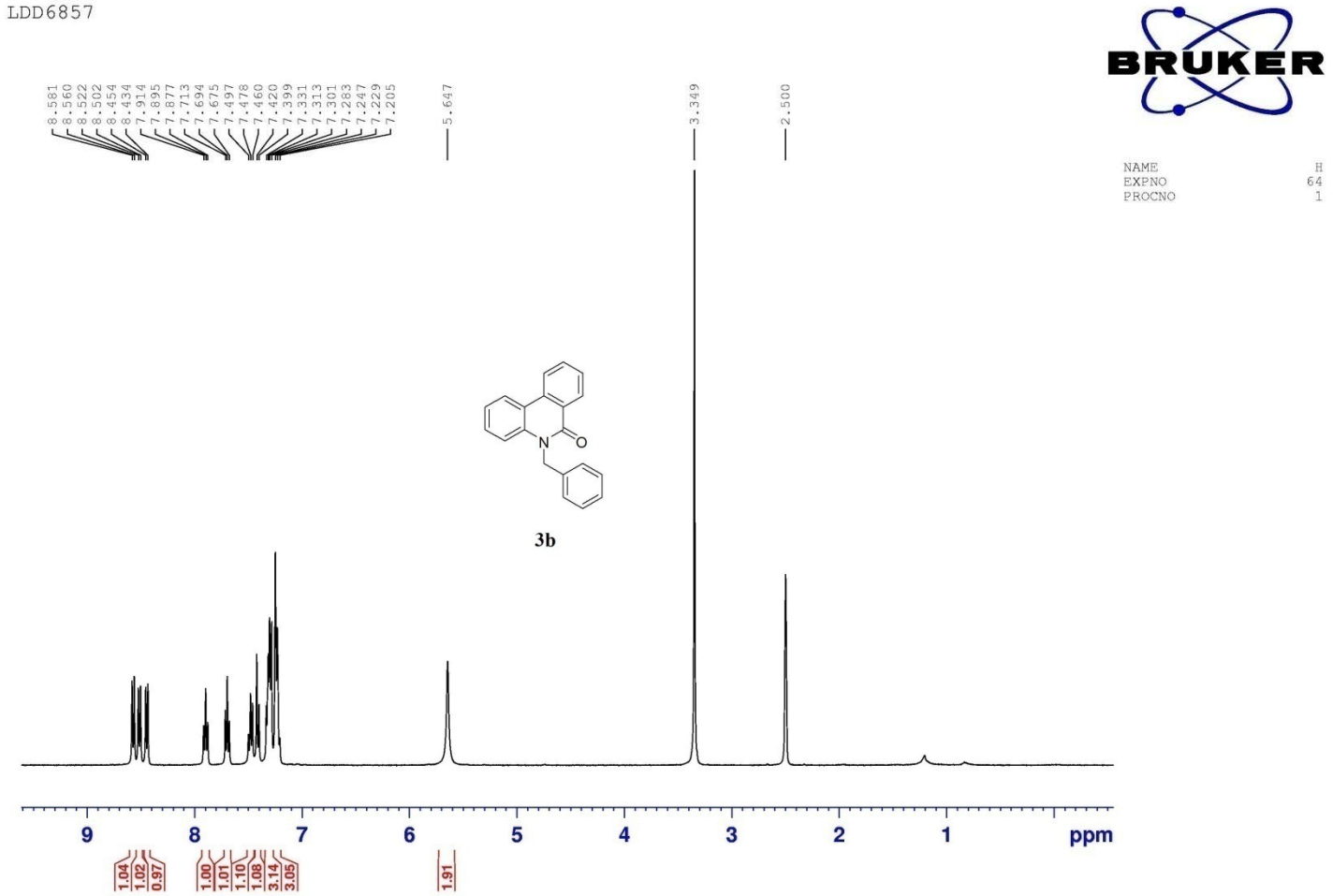

LDD6848
11.865
8.498
8.478
8.244
8.241
8.224
8.221
7.777
7.774
7.757
7.754
7.743
7.740
7.723
7.720
7.697
7.684
7.677
7.665
7.657
7.645
7.556
7.538
7.520
7.423
7.405
7.312
7.293
7.274
3.342
2.500
BRUKER
NAME H
EXPNO 52
PROCNO 1
F
N
H
O
2v
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.92
1.00
1.00
1.10
1.07
1.14
1.27

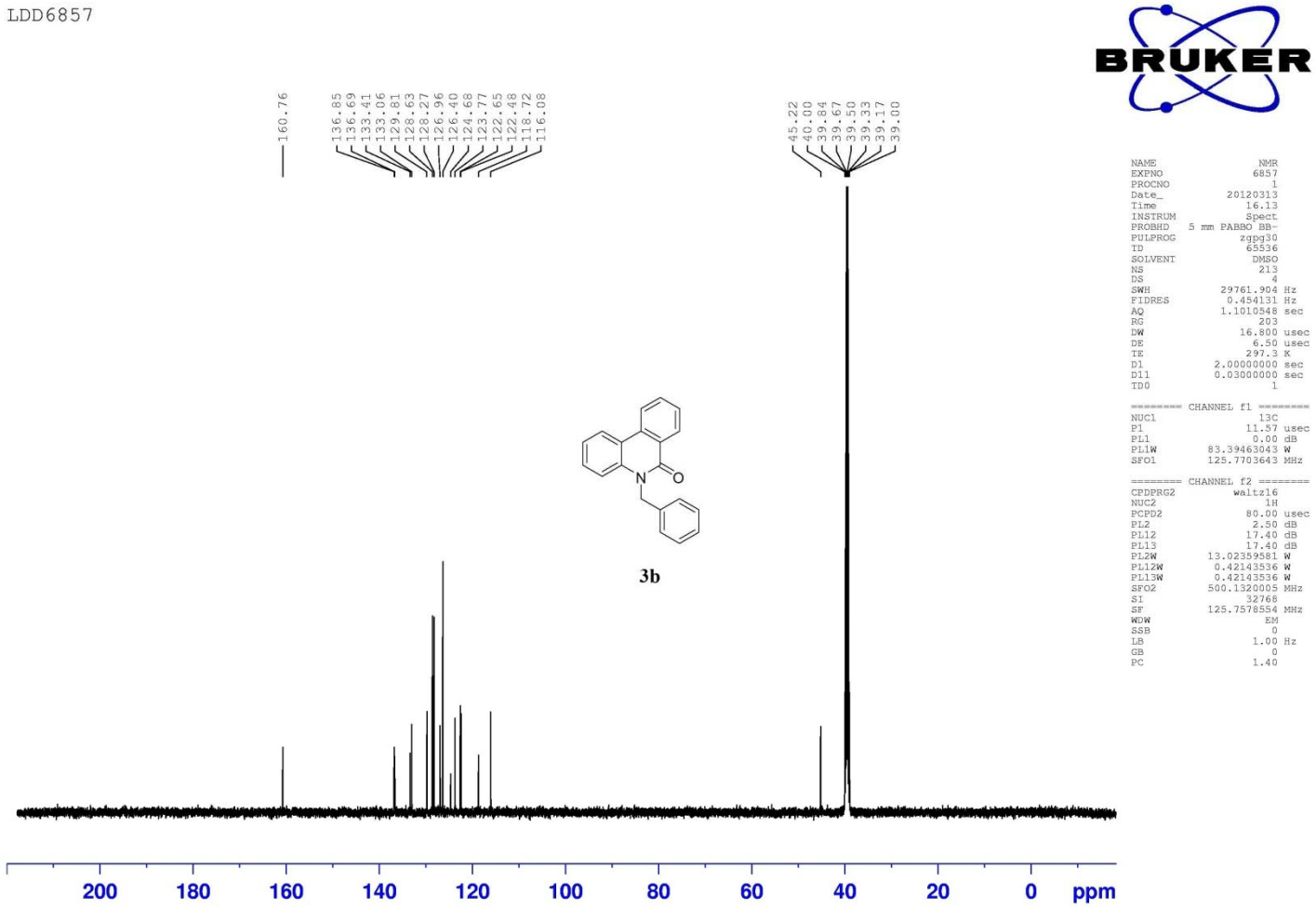

LDD6848
160.60
159.62
159.60
158.59
136.62
129.85
128.79
128.71
128.22
128.20
126.99
126.81
123.94
123.91
122.67
122.55
122.48
120.25
120.06
116.23
114.82
114.78
40.00
39.83
39.67
39.50
39.33
39.17
39.00
F
N
H
O
2v
200
180
160
140
120
100
80
60
40
20
0
ppm
NAME NMR
EXPNO 6848
PROCNO 1
Date_ 20120314
Time 16.29
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1396
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 297.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.57 usec
PL1 0.00 dB
PL1W 83.39463043 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7578519 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


ldd6844
8.497
8.478
8.459
8.365
8.363
8.345
8.343
7.855
7.852
7.835
7.817
7.814
7.655
7.636
7.625
7.618
7.604
7.587
7.585
7.569
7.550
7.369
7.367
7.349
7.332
7.329
3.713
3.364
2.503
2.499
BRUKER
NAME H
EXPNO 45
PROCNO 1
3a
9
8
7
6
5
4
3
2
1
0 ppm
1.04
1.02
0.96
1.00
3.03
1.01
3.00



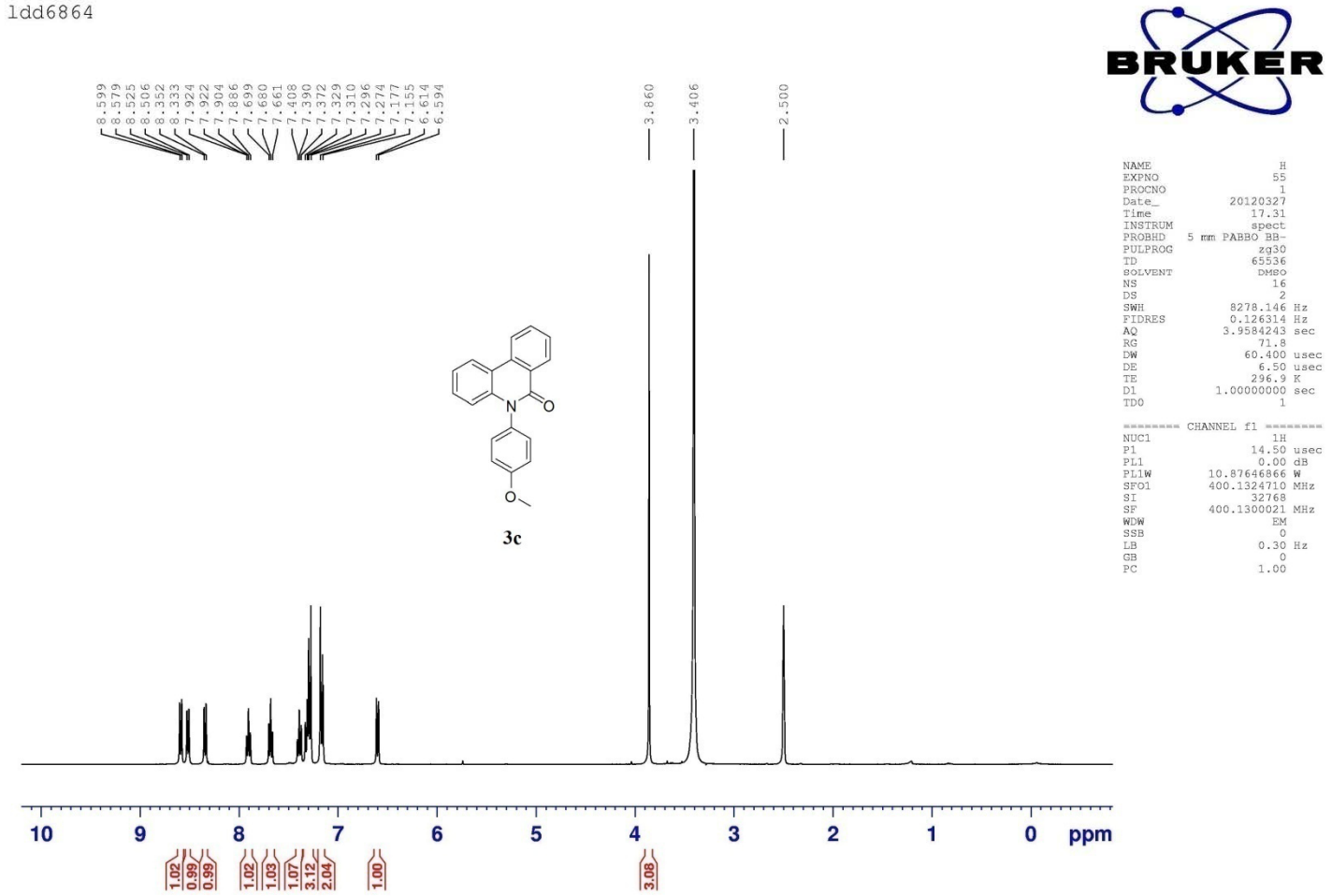
NAME	NMR
EXPNO	6844
PROCNO	1
Date_	20120302
Time	16.16
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	DMSO
NS	96
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	297.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.57 usec
PL1	0.00 dB
PL1W	83.39463043 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7578504 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

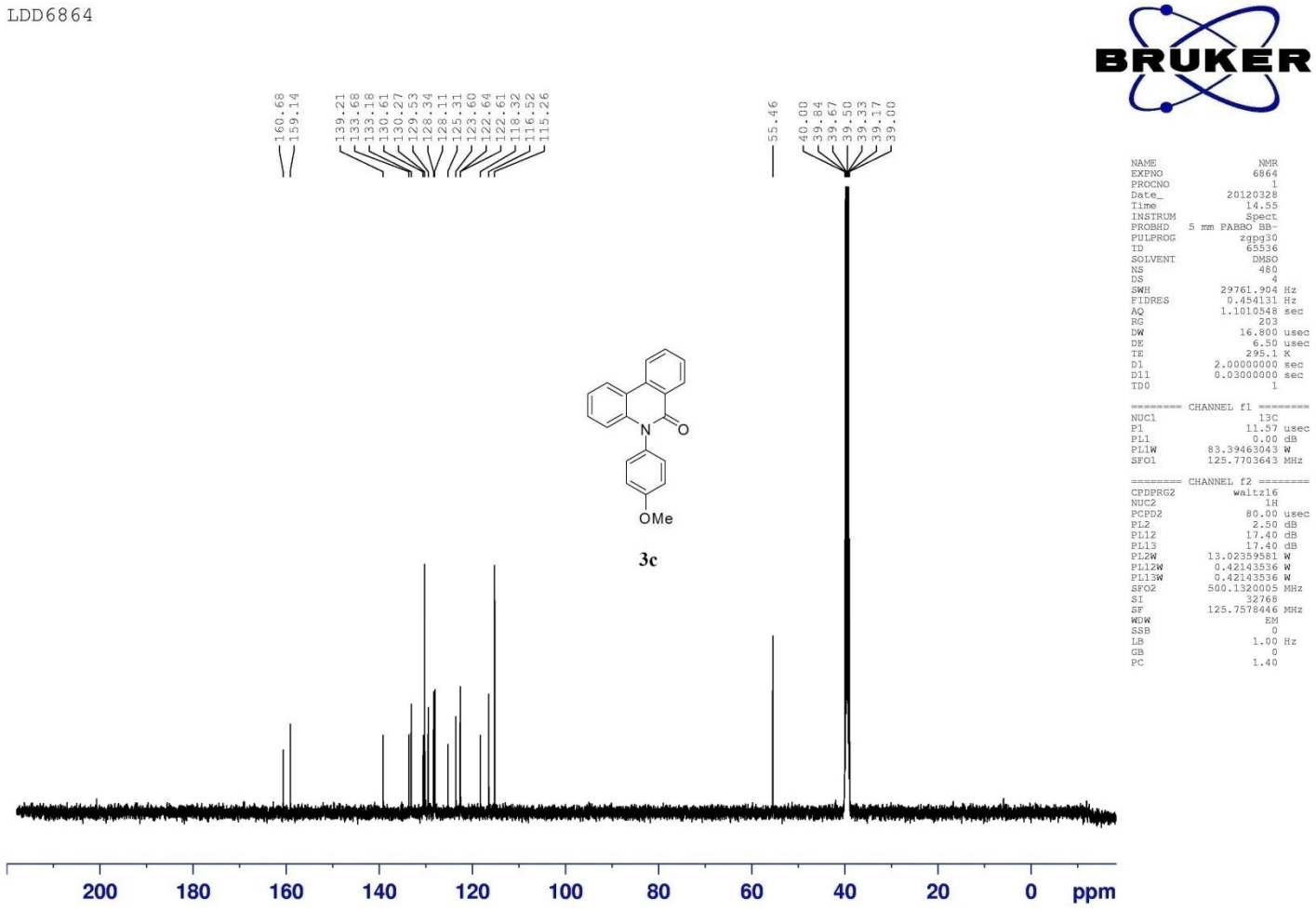

LDD6857
8.581
8.560
8.522
8.502
8.454
8.434
7.914
7.895
7.877
7.713
7.694
7.675
7.497
7.478
7.460
7.420
7.399
7.331
7.313
7.301
7.283
7.247
7.229
7.205
5.647
3.349
2.500
NAME H
EXPNO 64
PROCNO 1
3b
9
8
7
6
5
4
3
2
1
ppm
1.04
1.02
0.97
1.00
1.01
1.10
1.08
3.14
3.05
1.91



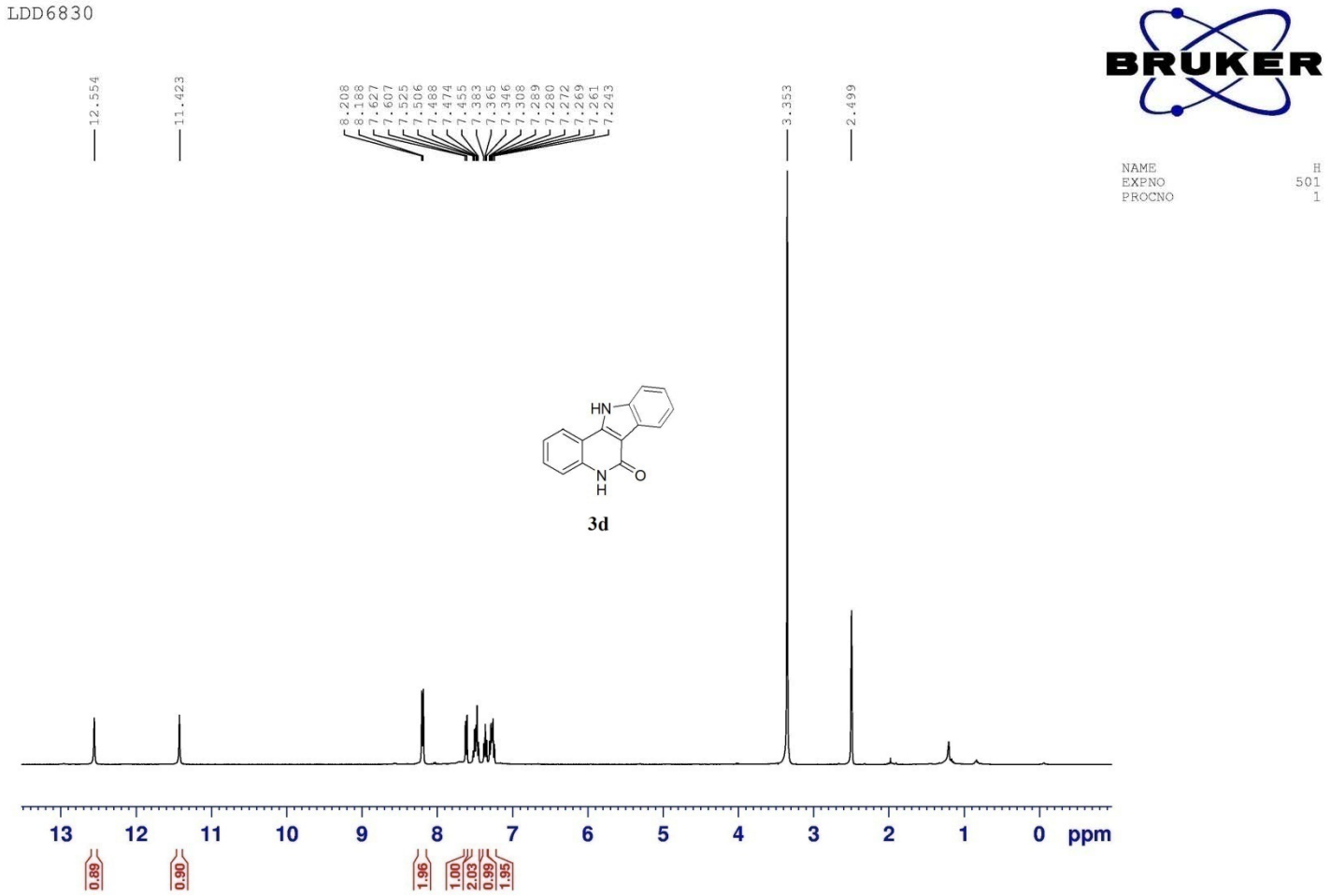
BRUKER

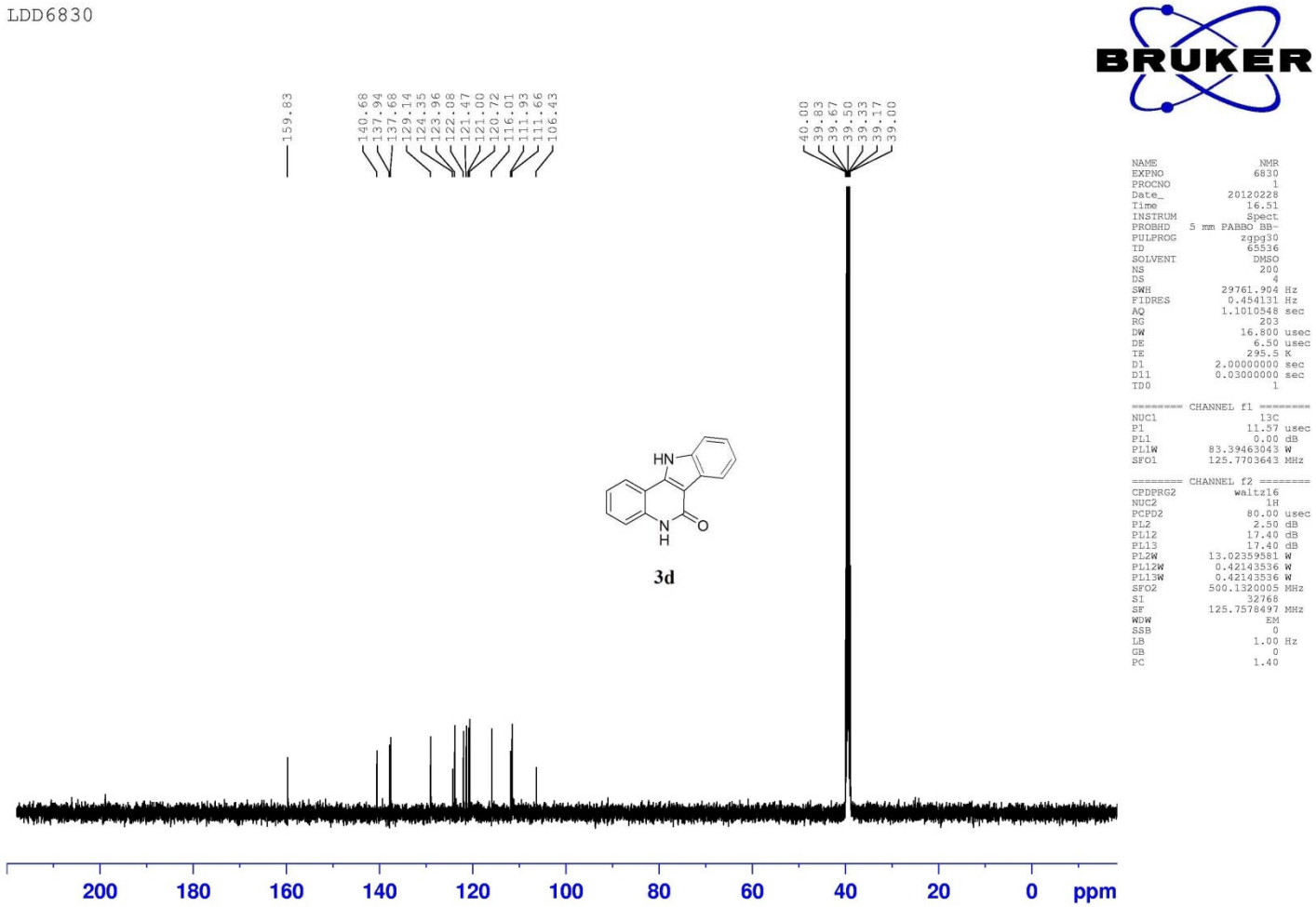
NAME	NMR
EXPNO	6857
PROCNO	1
Date_	20120313
Time	16.13
INSTRUM	Spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	DMSO
NS	213
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	297.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.57 usec
PL1	0.00 dB
PL1W	83.39463043 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	2.50 dB
PL12	17.40 dB
PL13	17.40 dB
PL2W	13.02359581 W
PL12W	0.42143536 W
PL13W	0.42143536 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7578554 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

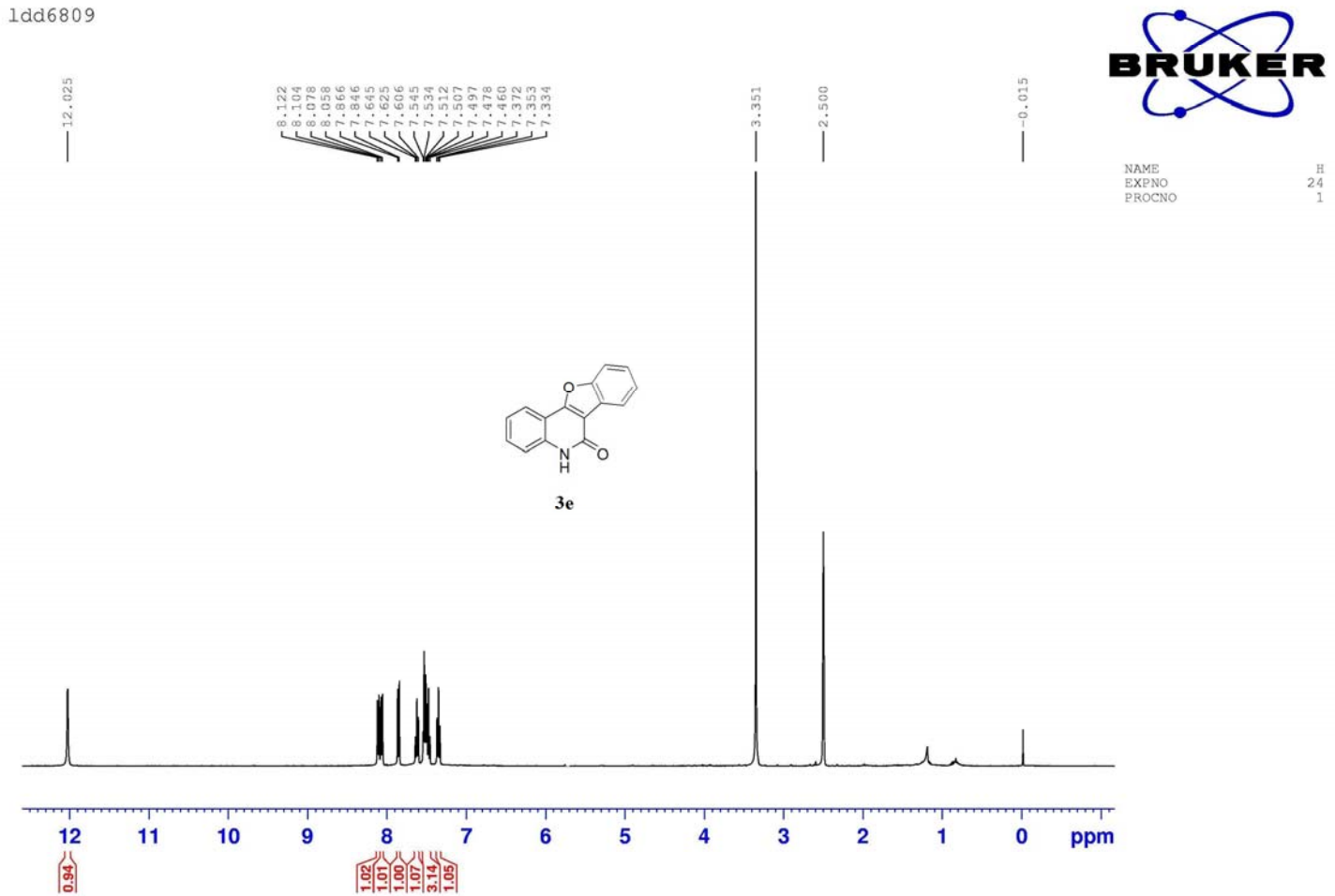

ldd6864
8.599
8.579
8.525
8.506
8.352
8.333
7.924
7.922
7.904
7.886
7.699
7.680
7.661
7.408
7.390
7.372
7.329
7.310
7.296
7.274
7.177
7.155
6.614
6.594
3.860
3.406
2.500
3c
10
9
8
7
6
5
4
3
2
1
0
ppm
1.02
0.99
0.99
1.02
1.03
1.07
3.12
2.04
1.00
3.08
NAME H
EXPNO 55
PROCNO 1
Date_ 20120327
Time 17.31
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 71.8
DW 60.400 usec
DE 6.50 usec
TE 296.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.50 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300021 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

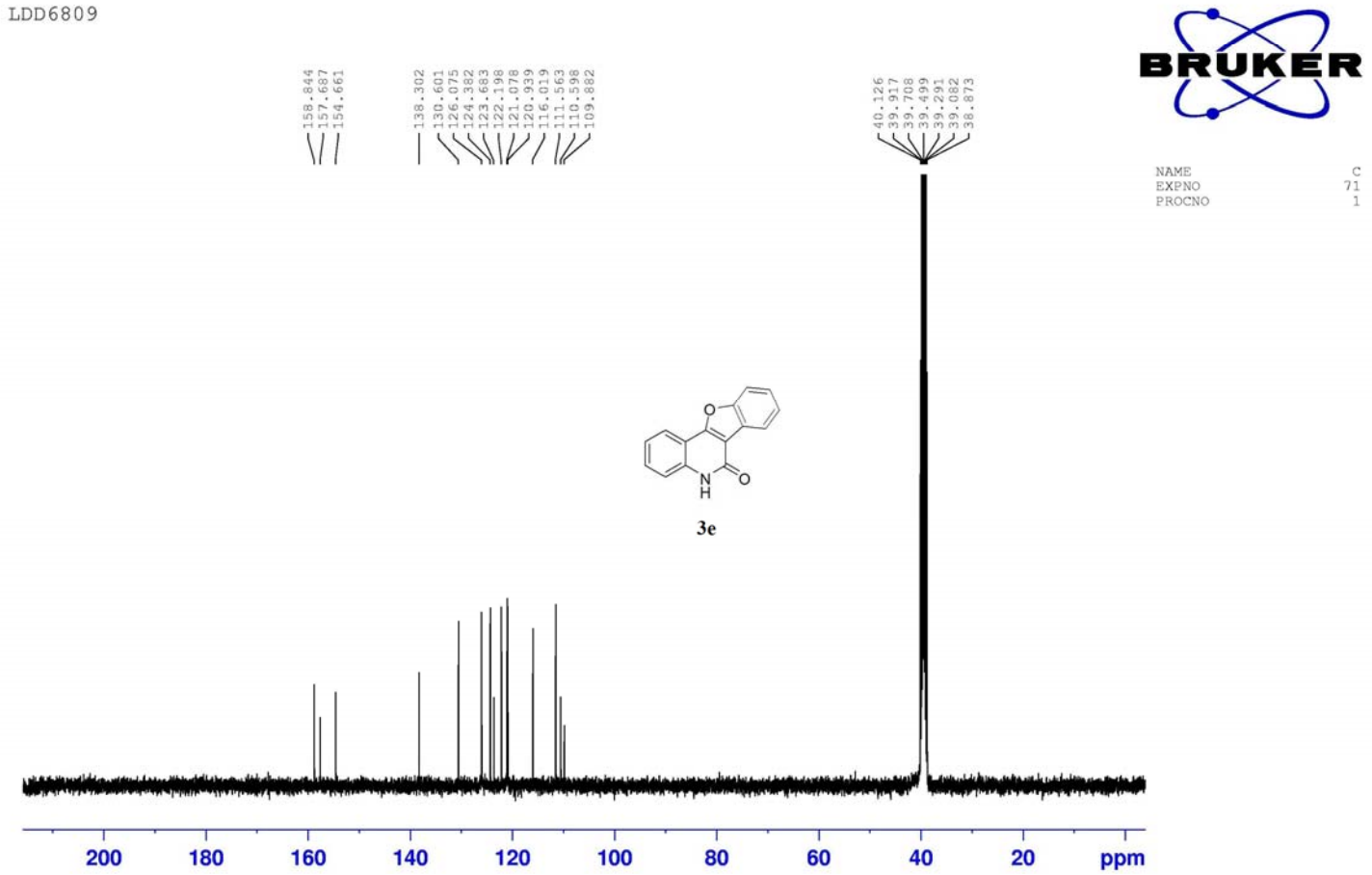


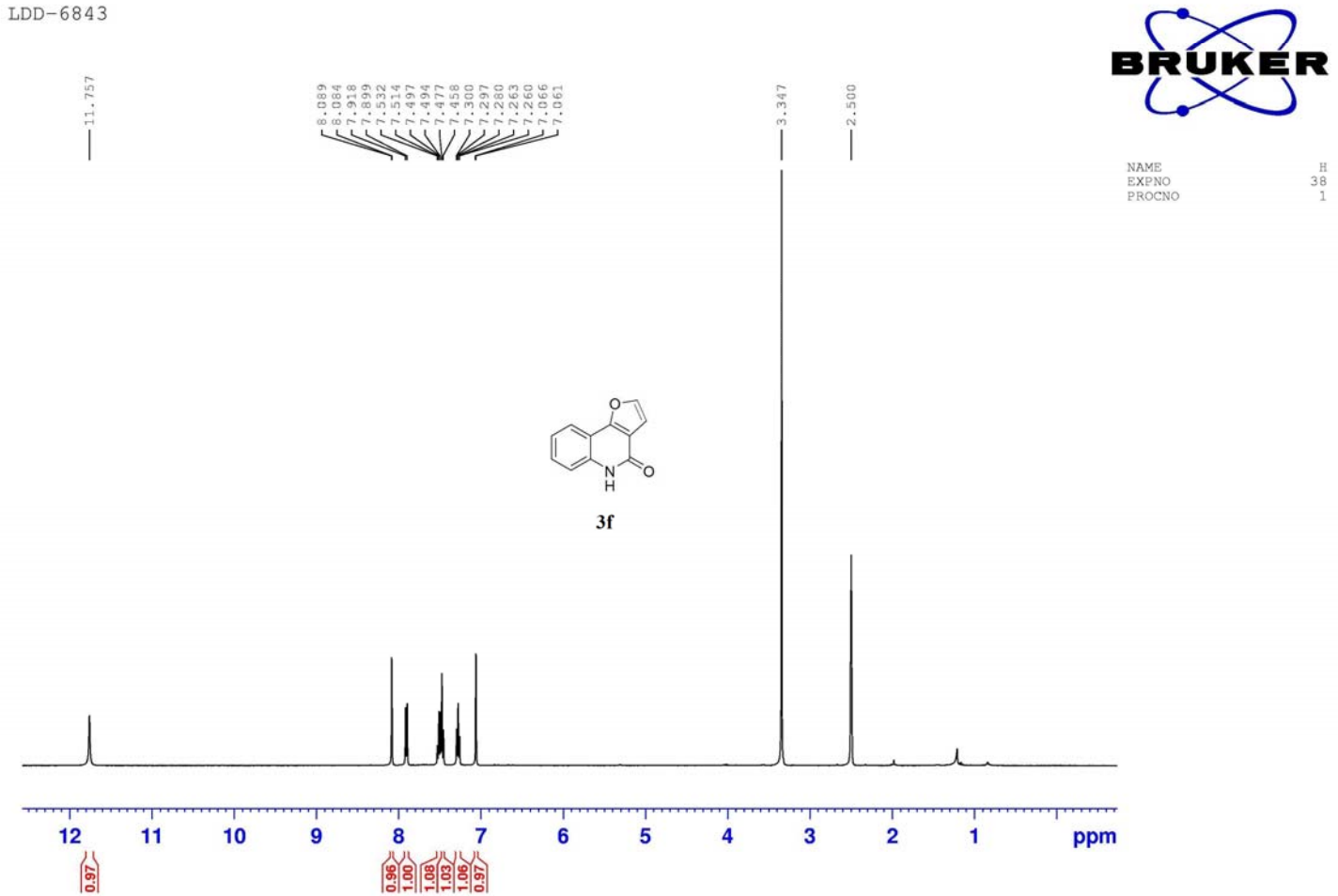

```
NAME                  NMR
EXPNO                6864
PROCNO                  1
Date_             20120328
Time                14.55
INSTRUM             Spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT              DMSO
NS                    480
DS                      4
SWH             29761.904 Hz
FIDRES          0.454131 Hz
AQ              1.1010548 sec
RG                    203
DW                16.800 usec
DE                 6.50 usec
TE                295.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                 11.57 usec
PL1                 0.00 dB
PL1W          83.39463043 W
SFO1         125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 2.50 dB
PL12               17.40 dB
PL13               17.40 dB
PL2W          13.02359581 W
PL12W          0.42143536 W
PL13W          0.42143536 W
SFO2         500.1320005 MHz
SI                  32768
SF           125.7578446 MHz
WDW                    EM
SSB                     0
LB                 1.00 Hz
GB                      0
PC                 1.40
```

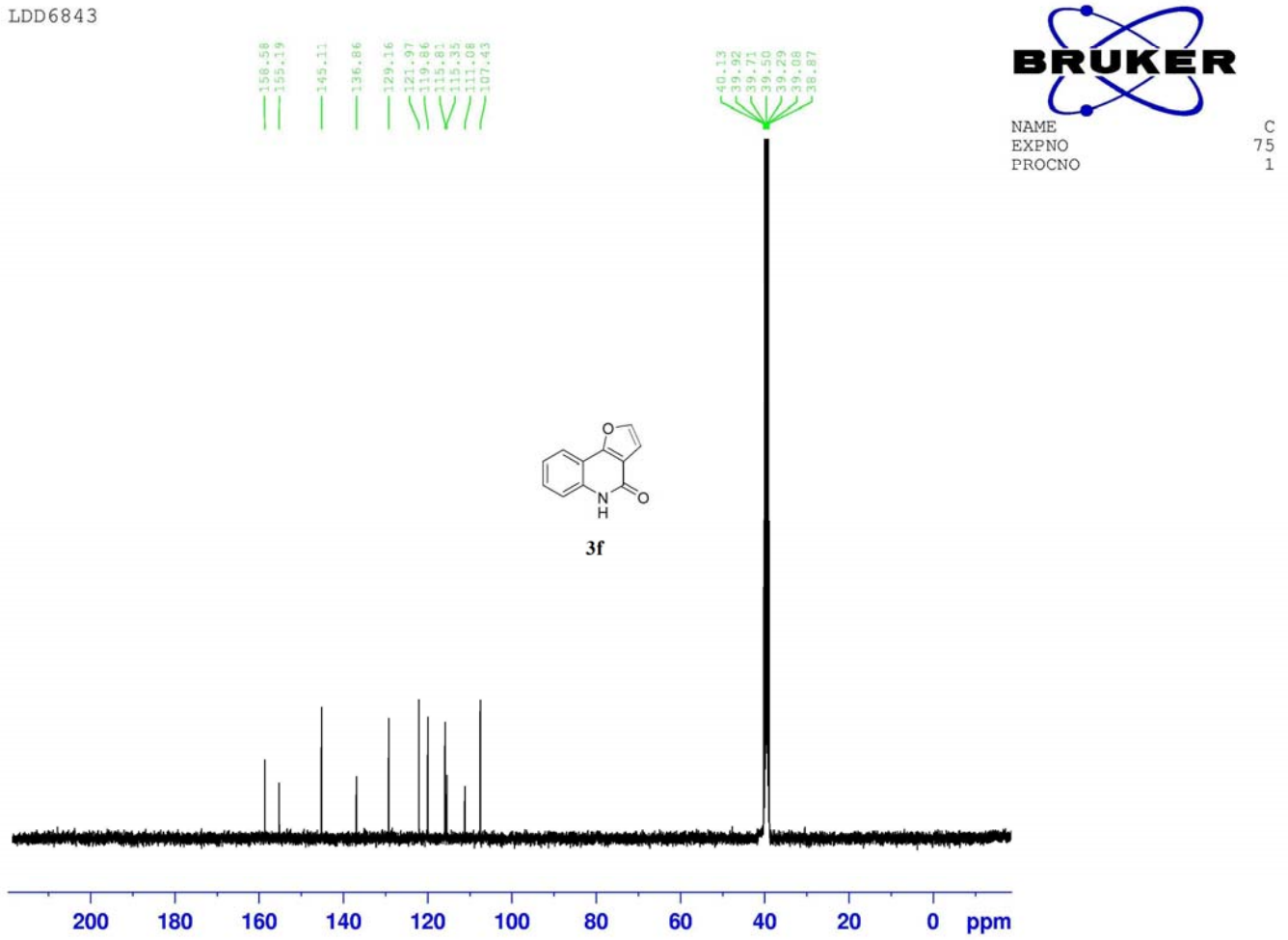

LDD6830
12.554
11.423
8.208
8.188
7.627
7.607
7.525
7.506
7.488
7.474
7.455
7.383
7.365
7.346
7.308
7.289
7.280
7.272
7.269
7.261
7.243
3.353
2.499
NAME H
EXPNO 501
PROCNO 1
HN
N
H
O
3d
13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.89
0.90
1.96
1.00
2.03
0.99
1.95

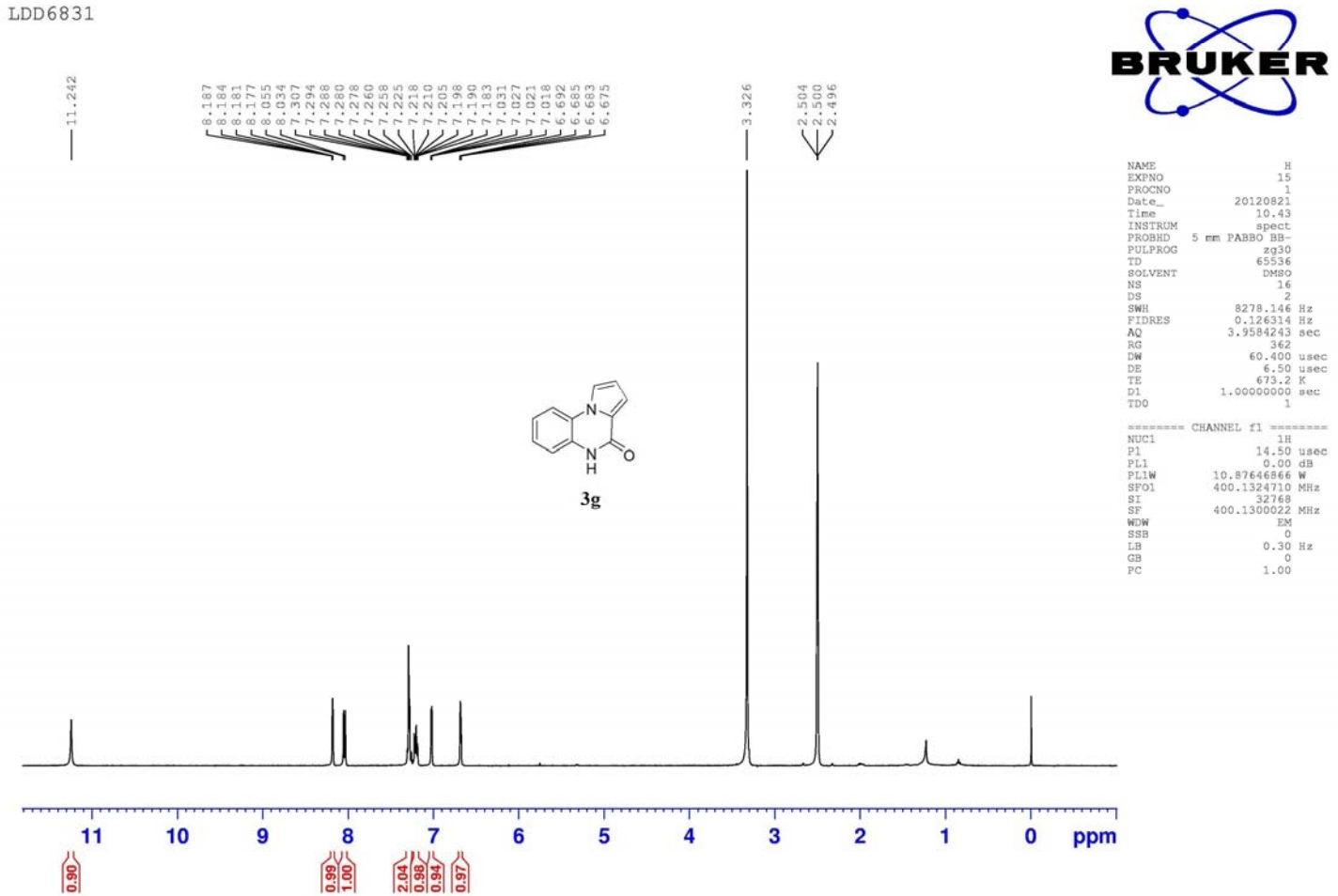

LDD6830
159.83
140.68
137.94
137.68
129.14
124.35
123.96
122.08
121.47
121.00
120.72
116.01
111.93
111.66
106.43
40.00
39.83
39.67
39.50
39.33
39.17
39.00
HN
N
H
O
3d
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER
NAME NMR
EXPNO 6830
PROCNO 1
Date_ 20120228
Time 16.51
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 200
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 295.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.57 usec
PL1 0.00 dB
PL1W 83.39463043 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7578497 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


ldd6809
12.025
8.122
8.104
8.078
8.058
7.866
7.846
7.645
7.625
7.606
7.545
7.534
7.512
7.507
7.497
7.478
7.460
7.372
7.353
7.334
3.351
2.500
-0.015
BRUKER
NAME H
EXPNO 24
PROCNO 1
O
N
H
O
3e
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.94
1.02
1.01
1.00
1.07
3.14
1.05


LDD6809
158.844
157.687
154.661
138.302
130.601
126.075
124.382
123.683
122.198
121.078
120.939
116.019
111.563
110.598
109.882
40.126
39.917
39.708
39.499
39.291
39.082
38.873
BRUKER
NAME C
EXPNO 71
PROCNO 1
O
N
H
O
3e
200
180
160
140
120
100
80
60
40
20
ppm


LDD-6843
11.757
8.089
8.084
7.918
7.899
7.532
7.514
7.497
7.494
7.477
7.458
7.300
7.297
7.280
7.263
7.260
7.066
7.061
3.347
2.500
NAME H
EXPNO 38
PROCNO 1
O
N
H
O
3f
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.97
0.96
1.00
1.08
1.03
1.06
0.97


LDD6843
158.58
155.19
145.11
136.86
129.16
121.97
119.86
115.81
115.35
111.08
107.43
40.13
39.92
39.71
39.50
39.29
39.08
38.87
BRUKER
NAME C
EXPNO 75
PROCNO 1
O
N
H
O
3f
200
180
160
140
120
100
80
60
40
20
0
ppm


LDD6831
11.242
8.187
8.184
8.181
8.177
8.055
8.034
7.307
7.294
7.288
7.280
7.278
7.260
7.258
7.225
7.218
7.210
7.205
7.198
7.190
7.183
7.031
7.027
7.021
7.018
6.692
6.685
6.683
6.675
3.326
2.504
2.500
2.496
3g
NAME H
EXPNO 15
PROCNO 1
Date_ 20120821
Time 10.43
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.50 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300022 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.90
0.99
1.00
2.04
0.98
0.94
0.97