Supporting Information

Palladium-Catalyzed C(sp^2)-H Pyridocarbonylation of *N*-Aryl-2-aminopyridines: Dual Function of the Pyridyl Moiety

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1. General Information

Substrates **1** were synthesized according to the literature method.[1] 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). IR spectra were recorded on a Bruker Tensor 27 spectrometer using a diamond comb. High resolution mass spectra (HRMS) were obtained on an ESI-LC-MS/MS spectrometer.

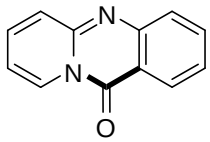
2. General Procedure and Product Characterization

2.1 General procedure

In an oven-dried Schlenk tube equipped with a stir bar, $Pd(OAc)_2$ (2.3 mg, 5.0 mol %), $K_2S_2O_8$ (162 mg, 3.0 equiv) and *N*-aryl-2-aminopyridines (0.2 mmol) were combined. A balloon filled with CO was connected to the Schlenk tube by the side tube and purged three times. Then, TFA (1.0 mL) was added to the tube through a syringe. The Schlenk tube was heated at 70 ^{o}C for 1.5-8 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 saturated aqueous $NaHCO_3$ (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.**

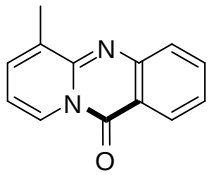
2.2. Product Characterization

11*H*-pyrido[2,1-*b*]quinazolin-11-one (2a)[2,3]



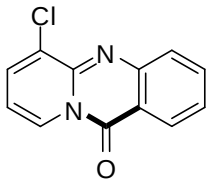
Yellow solid, 34 mg, 87% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.87 (d, J = 7.2 Hz, 1H), 8.45 (dd, *J* = 8.4 Hz, *J* = 1.2 Hz, 1H), 7.84-7.82 (m, 1H), 7.78 (d, *J* = 8.0 Hz, 1H), 7.51-7.45 (m, 3H), 6.88-6.84 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 159.0, 148.5, 147.7, 135.0, 134.1, 127.3, 126.9, 126.7, 126.3, 125.2, 116.3, 112.4; IR (KBr): 3412, 3111, 2924, 1974, 1700, 1641, 1569, 1545, 1455, 1384, 1302, 1150 764, 685 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{12}H_9N_2O$ $[M+H]^+$ 196.0709, found 196.0710.

6-methyl-11*H*-pyrido[2,1-*b*]quinazolin-11-one (2b)[4]



Yellow solid, 36 mg, 86% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.81 (d, *J* = 7.2 Hz, 1H), 8.44 (d, *J* = 8.0 Hz, 1H), 7.84-7.82 (m, 2H), 7.48-7.45 (m, 1H), 7.37 (dd, *J* = 5.2 Hz, *J* = 1.2 Hz, 1H), 6.78 (t, *J* = 7.2 Hz, 1H), 2.60 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 159.5, 148.3, 147.6, 134.7, 134.5, 132.2, 127.5, 127.2, 125.1, 124.8, 116.1, 112.1, 18.6; IR (KBr): 3551, 3478, 3413, 2978, 2917, 1702, 1640, 1608, 1556, 1529, 1457, 1164, 1137, 1072, 765, 736 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{13}H_{11}N_2O$ $[M+H]^+$ 211.0866, found 211.0865.

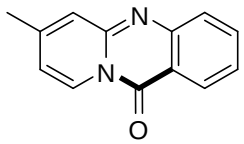
6-chloro-11*H*-pyrido[2,1-*b*]quinazolin-11-one (2c)



Pale yellow solid, 31 mg, 67% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.84 (d, *J* = 7.2 Hz, 1H), 8.44 (d, *J* = 8.0 Hz, 1H), 7.94 (d, *J* = 8.4 Hz, 1H), 7.90-7.86 (m, 1H), 7.71-7.69 (m, 1H), 7.53 (t, *J* = 7.2 Hz, 1H), 6.79 (t, *J* = 7.2 Hz, 1H); ^{13}C NMR (125

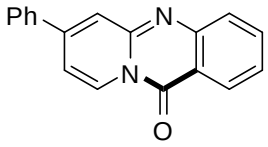
MHz, $CDCl_3$): δ 159.0, 147.8, 144.4, 135.3, 133.0, 130.4, 127.8, 127.3, 126.1, 126.0, 116.2, 111.0; IR (KBr): 3474, 3453, 3347, 3098, 2925, 1834, 1701, 1633, 1605, 1544, 1523, 1470, 1455, 1248, 1152, 897, 771, 693 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{12}H_8ClN_2O$ $[M+H]^+$ 231.0320, found 231.0321.

7-methyl-11*H*-pyrido[2,1-*b*]quinazolin-11-one (2d)[2]



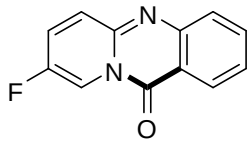
Pale yellow solid, 32 mg, 76% yield. ^1H NMR (400 MHz, $CDCl_3$): δ 8.78 (d, J = 7.2 Hz, 1H), 8.42 (dd, J = 8.4 Hz, J = 1.2 Hz, 1H), 7.83-7.79 (m, 1H), 7.74 (d, J = 8.4 Hz, 1H), 7.45-7.41 (m, 1H), 7.27 (s, 1H), 6.70 (dd, J = 7.2 Hz, J = 1.2 Hz, 1H), 2.41 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 167.9, 150.7, 142.5, 135.9, 134.6, 134.1, 130.4, 129.6, 128.6, 127.6, 110.5, 107.7, 37.1; IR (KBr): 3530, 3369, 3277, 3065, 2919, 1727, 1695, 1649, 1606, 1528, 1457, 1238, 776, 693 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{13}H_{11}N_2O$ $[M+H]^+$ 211.0866, found 211.0868.

7-phenyl-11*H*-pyrido[2,1-*b*]quinazolin-11-one (2e)



Yellow solid, 25 mg, 46% yield. ^1H NMR (400 MHz, $CDCl_3$): δ 8.92 (d, J = 7.6 Hz, 1H), 8.44 (d, J = 8.0 Hz, 1H), 7.84 (t, J = 8.0 Hz, 1H), 7.78 (d, J = 8.4 Hz, 1H), 7.73 (d, J = 8.0 Hz, 3H), 7.54-7.44 (m, 4H), 7.17 (d, J = 8.0, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 158.9, 149.0, 148.0, 146.0, 136.4, 135.1, 129.9, 129.3, 127.3, 126.9, 126.82, 126.80, 125.0, 121.9, 116.2, 112.3; IR (KBr): 3551, 3414, 3050, 2923, 1690, 1648, 1530, 1453, 1261, 1160, 1147, 941, 755, 690 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{18}H_{13}N_2O$ $[M+H]^+$ 273.1022, found 273.1022.

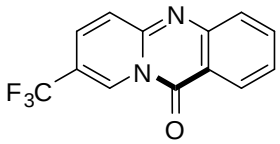
8-fluoro-11*H*-pyrido[2,1-*b*]quinazolin-11-one (2f)



Pale yellow solid, 34 mg, 79% yield. ^1H NMR (400 MHz, $CDCl_3$): δ 8.76 (dd, J = 5.2

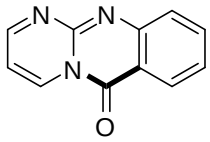
Hz, *J* = 2.4 Hz, 1H), 8.41 (dd, *J* = 8.0 Hz, *J* = 1.2 Hz, 1H), 7.86-7.82 (m, 1H), 7.77 (d, *J* = 8.4 Hz, 1H), 7.53-7.43 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 158.4 (d, *J* = 1.8 Hz), 152.3 (d, *J* = 241.9 Hz), 148.1, 145.7, 135.0, 128.3 (d, *J* = 3.9 Hz), 127.7 (d, *J* = 27.1 Hz), 127.10, 127.08, 125.9, 115.6, 112.1 (d, *J* = 41.5 Hz); IR (KBr): 3412, 3069, 3052, 2923, 1972, 1690, 1608, 1568, 1550, 1531, 1311, 1472, 1457, 1345, 769, 694 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{12}H_8FN_2O$ $[M+H]^+$ 215.0615, found 215.0617.

8-(trifluoromethyl)-11*H*-pyrido[2,1-*b*]quinazolin-11-one (2g)



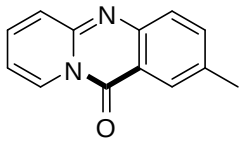
Yellow solid, 40 mg, 76% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.18 (d, *J* = 1.6 Hz, 1H), 8.43 (dd, *J* = 8.0 Hz, *J* = 0.8 Hz, 1H), 7.90-7.86 (m, 1H), 7.78 (d, *J* = 8.0 Hz, 1H), 7.55-7.51 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.6, 148.2, 146.6, 135.8, 128.9 (q, *J* = 1.9 Hz), 127.7, 127.5, 127.3, 126.6 (q, *J* = 6.1 Hz), 126.3, 122.9 (q, *J* = 269.8 Hz), 116.5, 116.3; IR (KBr): 3551, 3468, 3413, 3103, 3047, 1809, 1712, 1662, 1610, 1580, 1556, 1462, 1335, 1262, 1116, 1058, 769, 675 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{13}H_8F_3N_2O$ $[M+H]^+$ 265.0583, found 265.0584.

6*H*-pyrimido[2,1-*b*]quinazolin-6-one (2h)[5]



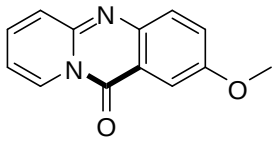
Yellow solid, 16 mg, 40% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.12 (dd, *J* = 7.2 Hz, *J* = 2.0 Hz, 1H), 8.91 (dd, *J* = 7.6 Hz, *J* = 2.4 Hz, 1H), 8.42 (d, *J* = 8.0 Hz, 1H), 7.94-7.87 (m, 2H), 7.55-7.52 (m, 1H), 6.89 (dd, *J* = 7.2 Hz, *J* = 2.0 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 161.5, 159.3, 149.0, 147.3, 135.8, 135.7, 128.0, 127.3, 126.2, 116.2, 108.9; IR (KBr): 3413, 2962, 2922, 2851, 1696, 1638, 1570, 1547, 1458, 1402, 1228, 1145, 775, 690 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{11}H_8N_3O$ $[M+H]^+$ 198.0662, found 198.0661.

2-methyl-11*H*-pyrido[2,1-*b*]quinazolin-11-one (2i)[3]



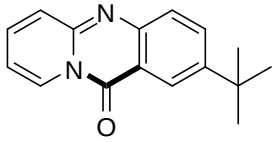
Yellow solid, 32 mg, 76% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.86 (dd, J = 7.2 Hz, 1H), 8.23 (s, 1H), 7.71-7.65 (m, 2H), 7.49-7.46 (m, 2H), 6.85-6.81 (m, 1H), 2.51 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 158.9, 147.2, 146.7, 136.9, 135.4, 133.5, 126.7, 126.6, 126.3, 126.2, 116.1, 112.3, 21.4; IR (KBr): 3413, 3052, 2920, 1697, 1642, 1545, 1526, 1481, 1257, 1140, 826, 765, 696, 569 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{13}H_{11}N_2O$ $[M+H]^+$ 211.0866, found 211.0867.

2-methoxy-11*H*-pyrido[2,1-*b*]quinazolin-11-one (2j)[3,5]



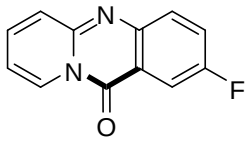
Pray white solid, 28 mg, 62% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.87 (d, J = 7.2 Hz, 1H), 7.76-7.73 (m, 2H), 7.51-7.43 (m, 3H), 6.88-6.84 (m, 1H), 3.96 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 158.7, 157.4, 146.1, 143.7, 132.8, 128.7, 126.8, 126.41, 126.39, 116.9, 112.6, 105.1, 55.8; IR (KBr): 3478, 3037, 2923, 1731, 1692, 1599, 1546, 1432, 1375, 1142, 1022, 763 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{13}H_{11}N_2O_2$ $[M+H]^+$ 227.0815, found 227.0817.

2-tert-butyl-11*H*-pyrido[2,1-*b*]quinazolin-11-one (2k)



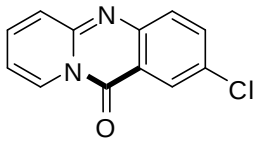
Pale yellow solid, 39 mg, 77% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.87 (dd, J = 7.6 Hz, J = 1.2 Hz, 1H), 8.40 (d, J = 2.0 Hz, 1H), 7.93 (dd, J = 8.8 Hz, J = 2.4 Hz, 1H), 7.74 (d, J = 8.8 Hz, 1H), 7.49-7.47 (m, 2H), 6.86-6.82 (m, 1H), 1.42 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 159.1, 148.7, 147.3, 146.6, 133.6, 126.7, 126.6, 126.3, 122.5, 115.7, 112.4, 35.0, 31.2; IR (KBr): 3033, 3961, 2867, 2636, 1684, 1644, 1547, 1525, 1483, 1265, 1150, 1225, 834, 766, 601 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{16}H_{17}N_2O[M+H]^+$ 253.1335, found 253.1338.

2-fluoro-11*H*-pyrido[2,1-*b*]quinazolin-11-one (2l)



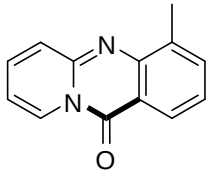
White solid, 30 mg, 70% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.85 (d, J = 7.2 Hz, 1H), 8.06 (dd, J = 8.8 Hz, J = 2.8 Hz, 1H), 7.80 (dd, J = 8.8 Hz, J = 4.8 Hz, 1H), 7.62-7.57 (m, 1H), 7.52-7.51 (m, 2H), 6.91-6.87 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 159.7 (d, J = 246.1 Hz), 158.4 (d, J = 4.1 Hz), 147.1, 145.4, 133.9, 129.5, (d, J = 8.1 Hz), 126.5, 126.4, 124.5(d, J = 25 Hz), 117.0 (d, J = 8.8 Hz), 112.9, 111.2 (d, J = 23.3 Hz); IR (KBr): 3550, 3476, 3413, 3234, 1694, 1649, 1546, 1549, 1482, 1440, 1364, 1133, 839, 796, 760 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{12}H_8FN_2O$ $[M+H]^+$ 215.0615, found 215.0614.

2-chloro-11*H*-pyrido[2,1-*b*]quinazolin-11-one (2m)



Yellow solid, 35 mg, 77% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.85 (dd, J = 7.2 Hz, J = 1.2 Hz, 1H), 8.39 (d, J = 2.0 Hz, 1H), 7.77-7.71 (m, 2H), 7.56-7.49 (m, 2H), 6.92-6.88 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 158.1, 147.8, 147.1, 135.6, 134.4, 130.7, 128.7, 126.7, 126.4, 126.3, 117.0, 113.0; IR (KBr): 3114, 3038, 1690, 1643, 1547, 1524, 1466, 1153, 1014, 886, 762, 732, 692 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{12}H_8ClN_2O$ $[M+H]^+$ 231.0320, found 231.0321.

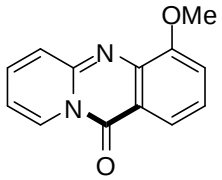
4-methyl-11*H*-pyrido[2,1-*b*]quinazolin-11-one (2n)



Pale yellow solid, 34 mg, 81% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.86 (d, J = 7.2 Hz, 1H), 8.31 (d, J = 8.0, 1H), 7.69 (d, J = 6.8 Hz, 1H), 7.55 (d, J = 9.2Hz, 1H), 7.50-7.46 (m, 1H), 7.37 (t, J = 7.6 Hz, 1H), 6.87-6.83 (m, 1H), 2.71 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 159.4, 147.5, 146.7, 135.4, 135.1, 133.4, 126.9, 126.6, 124.9, 124.8, 116.2, 112.4, 17.7; IR (KBr): 3474, 3453, 3423, 2946, 1692, 1640, 1605, 1578, 1544, 1476, 1302, 1242, 1136, 761, 699 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{13}H_{11}N_2O$

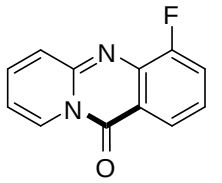
$[M+H]^+$ 211.0866, found 211.0866.

4-methoxy-11*H*-pyrido[2,1-*b*]quinazolin-11-one (2o)



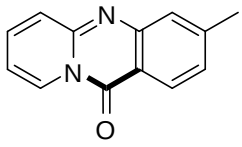
Pale yellow solid, 30 mg, 66% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.88 (d, J = 7.2 Hz, 1H), 8.03 (dd, J = 8.0 Hz, J = 1.2 Hz, 1H), 7.69 (d, J = 9.2 Hz, 1H), 7.53-7.49 (m, 1H), 7.41 (t, J = 8.0 Hz, 1H), 7.25 (d, J = 7.6 Hz, 1H), 6.91-6.87 (m, 1H), 4.08 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 158.8, 154.1, 147.2, 140.0, 133.7, 127.0, 126.6, 125.2, 118.5, 117.2, 113.4, 112.9, 56.3; IR (KBr): 3474, 3453, 3449, 3240, 2922, 2839, 1709, 1639, 1576, 1528, 1486, 1384, 1266, 1253, 1144, 1078, 757, 705 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{13}H_{11}N_2O_2$ $[M+H]^+$ 227.0815, found 227.0816.

4-fluoro-11*H*-pyrido[2,1-*b*]quinazolin-11-one (2p)



Brown solid, 33 mg, 77% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.88 (d, J = 7.2 Hz 1H), 8.23 (d, J = 8.4 Hz, 1H), 7.65 (d, J = 9.2Hz, 1H), 7.59-7.54 (m, 2H), 7.42-7.37 (m, 1H), 6.95-6.91 (m, 1H), 7.30; ^{13}C NMR (125 MHz, $CDCl_3$): δ 158.2 (d, J = 3.4 Hz), 156.7 (d, J = 253.6 Hz), 148.1, 138.8 (d, J = 12.1 Hz), 134.7, 126.8, 126.7, 124.7 (d, J = 7.5 Hz), 122.9 (d, J = 4.5 Hz), 119.5 (d, J = 18.4 Hz), 118.1, 113.1; IR (KBr): 3551, 3474, 3414, 3043, 2924, 1689, 1644, 1617, 1545, 1531, 1251, 1099, 755, 675 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{12}H_8FN_2O$ $[M+H]^+$ 215.0615, found 215.0616.

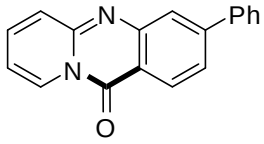
3-methyl-11*H*-pyrido[2,1-*b*]quinazolin-11-one (2q)



Gray white powder, 25 mg, 59% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.86 (d, J = 8.0 Hz, 1H), 8.33 (d, J = 8.4 Hz, 1H), 7.57 (s, 1H), 7.49-7.48 (m, 2H), 7.30 (dd, J = 8.4

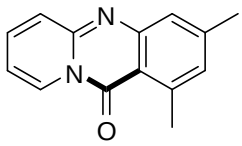
Hz, *J* = 1.2 Hz, 1H), 6.86-6.82 (m, 1H), 2.54 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 158.8, 148.7, 147.9, 146.2, 134.0, 127.2, 127.1, 126.7, 126.3, 114.0, 112.3, 22.2; IR (KBr): 3411, 3076, 2917, 1709, 1680, 1644, 1616, 1545, 1459, 1348, 1210, 1225, 761, 654 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{13}H_{11}N_2O$ $[M+H]^+$ 211.0866, found 211.0866.

3-phenyl-11*H*-pyrido[2,1-*b*]quinazolin-11-one (2r)



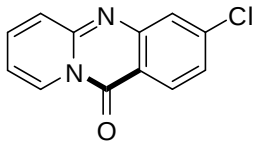
Pale yellow solid, 41 mg, 75% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.88 (d, *J* = 7.2 Hz, 1H), 8.49 (d, *J* = 8.8 Hz, 1H), 8.00 (d, *J* = 1.6 Hz, 1H), 7.76-7.72 (m, 3H), 7.52-7.48 (m, 4H), 7.45-7.41 (m, 1H), 6.88-6.85 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 158.8, 148.9, 148.1, 147.7, 139.7, 134.2, 129.0, 128.5, 127.8, 127.5, 126.8, 126.3, 124.63, 124.57, 115.0, 112.4; IR (KBr): 3551, 3413, 3114, 3030, 1692, 1642, 1614, 1546, 1530, 1464, 1210, 1160, 878, 768, 757, 688 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{18}H_{13}N_2O$ $[M+H]^+$ 273.1022, found 273.1023.

1,3-dimethyl-11*H*-pyrido[2,1-*b*]quinazolin-11-one (2s)



Yellow solid, 32 mg, 72% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.83 (d, *J* = 7.6 Hz, 1H), 7.48-7.40 (m, 3H), 7.05 (s, 1H), 6.81-6.77 (m, 1H), 2.93 (s, 3H), 2.47 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 158.9, 150.3, 147.8, 145.2, 141.2, 134.0, 129.5, 126.6, 126.0, 124.6, 112.9, 112.0, 23.5, 21.9; IR (KBr): 3551, 3479, 3413, 3109, 1732, 1689, 1641, 1616, 1548, 1529, 1293, 857, 621 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{14}H_{13}N_2O$ $[M+H]^+$ 225.1022, found 225.1021.

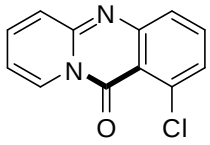
3-chloro-11*H*-pyrido[2,1-*b*]quinazolin-11-one (2t)[3]



Gray white powder, 20 mg, 43% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.85 (d, *J* = 7.2

Hz, 1H), 8.35 (d, *J* = 8.8 Hz, 1H), 7.75 (d, *J* = 2.0 Hz, 1H), 7.57-7.53 (m, 1H), 7.49 (d, *J* = 9.2 Hz, 1H), 7.39 (dd, *J* = 8.8 Hz, *J* = 2.0 Hz, 1H), 6.92-6.88 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.8, 149.9, 149.0, 141.8, 135.0, 129.2, 127.2, 126.8, 126.6, 126.3, 115.1, 113.1; IR (KBr): 3412, 3064, 3033, 2924, 1990, 1690, 1642, 1598, 1544, 1526, 1453, 1147, 768, 687 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{12}H_8ClN_2O$ $[M+H]^+$ 231.0320, found 231.0321.

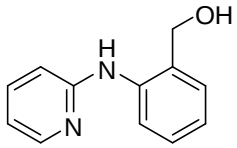
1-chloro-11*H*-pyrido[2,1-*b*]quinazolin-11-one (2t')



Pale yellow solid, 12 mg, 27% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.89 (d, *J* = 7.2 Hz, 1H), 7.68-7.66 (m, 2H), 7.58-7.54 (m, 1H), 7.48-7.46 (m, 2H), 6.91-6.88 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 156.9, 150.9, 148.1, 135.0, 134.4, 134.1, 127.6, 126.8, 126.2, 126.0, 113.5, 112.8; IR (KBr): 3779, 3574, 3216, 2957, 2924, 2854, 1698, 1643, 1597, 1560, 1517, 1462, 1378, 1279, 813, 687 cm^{-1}; HRMS (ESI): Exact mass calcd for $C_{12}H_8ClN_2O$ $[M+H]^+$ 231.0320, found 231.0318.

3. Diversification of 2a

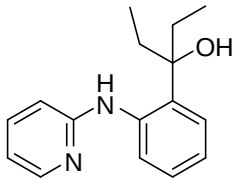
(2-(pyridin-2-ylamino)phenyl)methanol (3)



A solution of **2a** (196 mg, 1 mmol) in dry THF (2 mL) was added slowly to an ice-cold suspension of $LiAlH_4$ (152 mg, 4 mmol) in dry THF (5 mL). Then the solution was refluxed for 4 h, during which the conversion was complete. The reaction was then carefully quenched by ice. The citric acid (1g) in H_2O (10 mL) and 1 M of HCl (10 mL) was added, and then extracted by EtOAc. 10% of NaOH was added to adjust the PH to 8. The aqueous phase was extracted by EtOAc again. 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 **3** (98 mg)

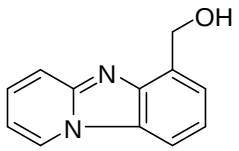
in 49% yield as a yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.11 (d, J = 4.8 Hz, 1H), 7.82 (br s, 1H), 7.59 (d, J = 8.4 Hz, 1H), 7.50 (t, J = 8.0 Hz, 1H), 7.29 (t, J = 1.2 Hz, 1H), 7.03 (t, J = 7.6 Hz, 1H), 6.88 (d, J = 8.4 Hz, 1H), 6.73 (t, J = 6.0 Hz, 1H), 4.73 (s, 2H), 3.97 (br s 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 156.0, 147.7, 139.8, 137.9, 131.5, 129.5, 128.7, 122.9, 120.6, 114.9, 109.0, 63.6.

3-(2-(pyridin-2-ylamino)phenyl)pentan-3-ol (4)



A solution of **2a** (392 mg, 2 mmol) in dry THF (10 mL) was cooled to 0 ^{o}C, after a dropwise addition of EtMgBr (1.0 M in THF, 4 mL) to this stirring solution, the resulting reaction mixture was stirred for 12 h at 25 ^{o}C. The reaction was quenched by H_2O (20 mL), EtOAc (20 mL) was added. The aqueous phase was extracted by EtOAc (20 mL) again. 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 **4** (344 mg) in 67% yield as yellow liquid**.** ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.03 (s, 1H), 8.08 (d, J = 4.8 Hz, 1H), 7.64 (d, J = 7.6 Hz, 1H), 7.42 (t, J = 8.0 Hz, 1H), 7.20 (t, J = 8.0 Hz, 2H), 6.97 (t, J = 7.6 Hz, 1H), 6.86 (d, J = 8.4 Hz, 1H), 6.65 (t, J = 6.4 Hz, 1H), 4.13 (br s, 1H), 1.96 (q, J = 7.6 Hz, 4H), 0.85 (t, J = 7.6 Hz, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 156.5, 147.8, 140.4, 137.6, 133.7, 127.8, 127.3, 122.0, 121.7, 114.4, 108.6,79.4, 32.2, 8.3.

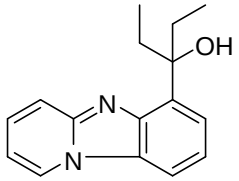
benzo[4,5]imidazo[1,2-*a*]pyridin-6-ylmethanol (5)



To a solution of **3** (0.20 mmol, 1 equiv) in HFIP (1.5 mL) was added $PhI(OAc)_2$ (0.22 mmol, 1.1 equiv) at 25 ^{o}C under air. The resulting mixture was stirred at 25 ^{o}C for 1.5 h. The reaction was monitored by TLC until the starting material was completely consumed. The reaction mixture was diluted with EtOAc (10 mL) and washed with

brine (10 mL). The organic layer was dried over Na_2SO_4, and concentrated under vacuum. The residue was purified by column chromatography to give **5** in 69% yield . ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.04 (d, J = 6.8 Hz, 1H), 8.15 (d, J = 8.0 Hz, 1H), 7.68 (d, J = 9.2 Hz, 1H), 7.57-7.51 (m, 2H), 7.35 (t, J = 7.6 Hz, 1H), 6.98 (t , J = 6.8 Hz, 1H), 5.29 (br s, 1H), 5.05 (s, 2H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 147.8, 141.8, 133.5, 130.3, 128.5, 127.4, 122.5, 120.8, 117.4, 110.7, 110.4, 59.5; IR (KBr): 3408, 2925, 1636, 1503, 1428, 1356, 1018, 757; HRMS (ESI): Exact mass calcd for $C_{12}H_{11}N_2O$ $[M+H]^+$ 199.0871, found 199.0864.

3-(benzo[4,5]imidazo[1,2-*a*]pyridin-6-yl)pentan-3-ol (6)



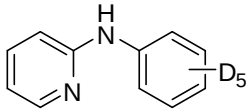
To a solution of **3** (0.20 mmol, 1 equiv) in HFIP (1.5 mL) was added $PhI(OAc)_2$ (0.22 mmol, 1.1 equiv) at 25 ^{o}C under air. The resulting mixture was stirred at 25 ^{o}C for 1.5 h. The reaction was monitored by TLC until the starting material was completely consumed. The reaction mixture was diluted with EtOAc (10 mL) and washed with brine (10 mL). The organic layer was dried over Na_2SO_4, and concentrated under vacuum. The residue was purified by column chromatography to give the **6** in 88% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.43 (d, J = 6.8 Hz, 1H), 7.76 (d, J = 8.0 Hz, 1H), 7.66 (d, J = 9.2 Hz, 1H), 7.43 (t, J = 8.8 Hz, 1H), 7.35 (t, J = 7.6 Hz, 1H), 7.28 (t, J = 7.6 Hz, 1H), 7.10 (br s, 1H), 6.85 (t, J = 6.8 Hz, 1H), 2.13-1.96 (m, 4H), 0.85 (t, 7.6 Hz, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 146.7, 143.0, 136.6, 129.4, 128.7, 125.1, 122.4, 120.9, 117.9, 110.4, 108.4, 79.5, 34.7, 8.3; IR (KBr): 3301, 2961, 1639, 1509, 1415, 1359, 1288, 965, 760; HRMS (ESI): Exact mass calcd for $C_{16}H_{20}N_2O$ $[M+H]^+$ 255.1497, found 255.1494.

4. Mechanistic studies

4.1 Isotop Labeling Experiments

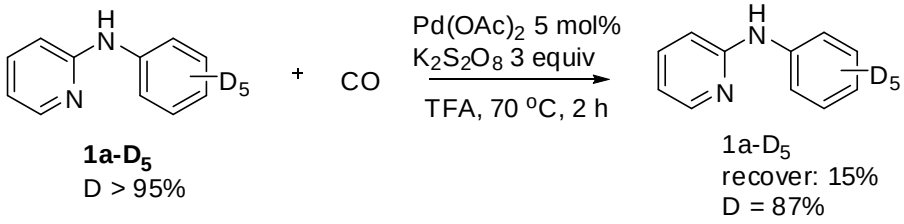
Synthesis and characterization of deuterated substrate 1a-D_5

N-pencadeuteriumphenylpyridin-2-amine (1a-D_5)



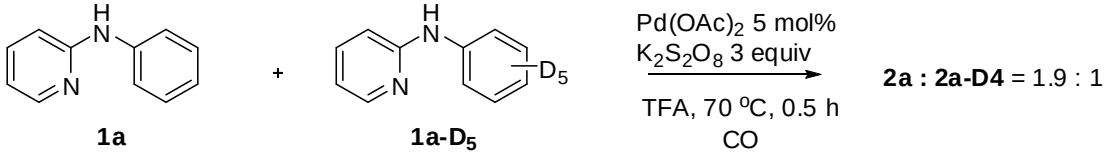
Following the literature method for the synthesis of substrate **1**, 2-bromopyridine and pentadeuteriumaniline were used. **1a-D_5** was obtained in 85% yield as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$): 8.21 (dd, *J* = 4.8 Hz, *J* = 1.2 Hz, 1H), 7.51-7.47 (m, 1H), 6.87 (d, *J* = 8.4 Hz, 1H), 6.75-6.71 (m, 1H), 6.60 (br s, 1H).

H/D Exchange experiment



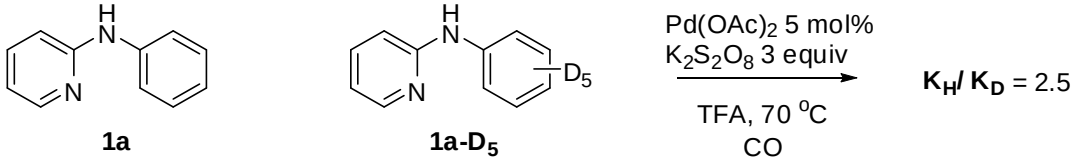
A mixture of **1a-D_5** (35 mg, 0.2 mmol, the deuterium rate is over 95%), $Pd(OAc)_2$ (2.3 mg, 5 mol %), $K_2S_2O_8$ (162 mg, 3 equiv) in TFA was stirred at 70 °C under balloon pressure of CO for 2 h. The mixture was diluted with EtOAc (20 mL), washed saturated aqueous $NaHCO_3$ (20 mL) and brine (20 mL), and dried over Na_2SO_4. The concentrated residue was purified by column chromatography to recover **1a-D_5** (D% was about 87%) in 15% yield.

Intermolecular competition reaction of a mixture of 1a and 1a-D_5



A mixture of **1a** (17 mg, 0.1 mmol, 0.5 equiv), **1a-D_5** (19.5 mg, 0.1 mmol, 0.5 equiv), $Pd(OAc)_2$ (2.3 mg, 5 mol %), $K_2S_2O_8$ (162 mg, 3 equiv) in TFA (1 mL) was stirred at 70 °C under balloon pressure of CO for 0.5 h. The mixture was diluted with EtOAc (20 mL), washed Saturated aqueous $NaHCO_3$ (20 mL) and brine (20 mL), and dried over Na_2SO_4. The concentrated residue was purified by column chromatography to give a mixture of **2a** and **2a-D_4** in a total yield of 38% in a ratio of 1.9:1 as determined by ^{1}H NMR.

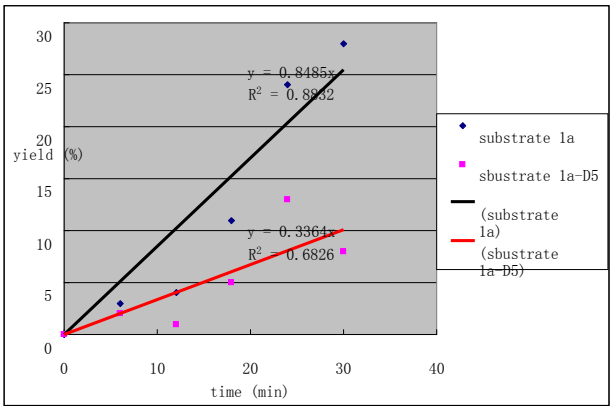
Parallel competition reactions of 1a and 1a-D_5 in separate tubes[6]



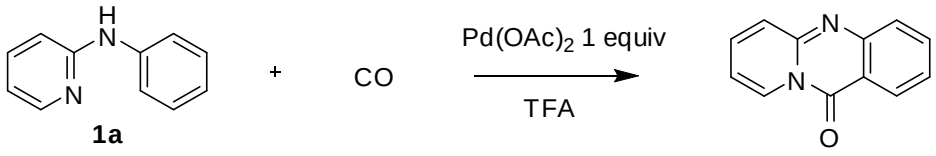
1a **1a-D_5**

parallel experiment

Five identical reactions were set side-by-side. Each reaction tube was charged with **1a** (17 mg, 0.1 mmol), $Pd(OAc)_2$ (1.2 mg, 5 mol%), $K_2S_2O_8$ (81 mg, 3 equiv) in TFA (0.5 mL) was stirred at 70 °C under balloon pressure of CO. The reactions were stopped in 6, 12, 18, 24 and 30 min, respectively. In a parallel experiment, the same five reactions were performed using **1a-D_5** (19.5 mg, 0.1 mmol) as a substrate under otherwise identical conditions. Each of the reaction was worked up following procedures mentioned above. The crude reaction mixture was analyzed by ^{1}H NMR using 4-iodoanisole as an internal standard. The yields of **2a** and **2a-D_4** of the 10 reactions were plotted against reaction time. The ratio of product formation was determined to be 2.5 by comparing the slopes.



4.2 Evidence of oxidation state of the palladium intermediate



1a **2a**, 48%

In an oven-dried Schlenk tube equipped with a stirring bar, $Pd(OAc)_2$ (45 mg, 1 equiv) and *N*-aryl-2-aminopyridines **1a** (35 mg, 0.2 mmol) were combined. A balloon filled with CO was connected to the Schlenk tube by the side tube and purged three times. Then, TFA (1.0 mL) was added to the tube through a syringe. The Schlenk tube was

heated at 70 ^{o}C for 4 h under balloon pressure of CO. The reaction was cooled down to room temperature. EtOAc (20 mL) and saturated aqueous $NaHCO_3$ (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 **2a** in 48% yield. 45% of **1a** was recovered. The result indicates that Pd(II) intermediate maybe involve in the course of reaction, instead of Pd(IV) ($K_2S_2O_8$ often used as an oxidant to oxidize the Pd(II) intermediate to Pd(IV).

5. References

(1) (a) Grasa, G. A.; Viciu, M. S.; Huang, J.; Nolan, S. P. *J. Org. Chem.* **2001,** *66*, 7729. (b)Wang, H.; Wang, Y.; Peng, C.; Zhang, J.; Zhu, Q. *J. Am. Chem. Soc*. **2010**, *132*, 13217.

(2) Maity, A.; Mondal, S.; Paira, R.; Hazra, A.; Naskar, S.; Sahu, K. B.; Saha, P.; Banerjee, S.; Mondal, N. B. *Tetrahedron Lett.* **2011**, *52*, 3033.

(3) Docampo, M. L.; Pellón, R. F. *Synth. Commun.* **2003**, *33*, 1777.

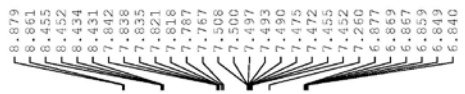
(4) Pellón, R. F.; Carrasco, R.; Rodés, L. *Synth. Commun*. **1996**, *26*, 3869

(5) Deetz, M. J.; Malerich, J. P.; Beatty, A. M.; Smith, B. D. *Tetrahedron Lett.* **2001**, *42*, 1851.

(6) Simmons, E. M.; Hartwig, J. F. *Angew. Chem., Int. Ed.* **2012**, *51*, 3066.

6. Copies of NMR Spectra

LDD-1314



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PULPROG	zg30
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SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	322.5
DW	60.400 usec
DE	6.50 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
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P1	12.58 usec
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PL1W	10.87646866 W
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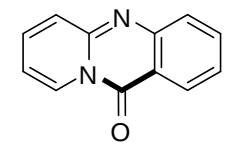
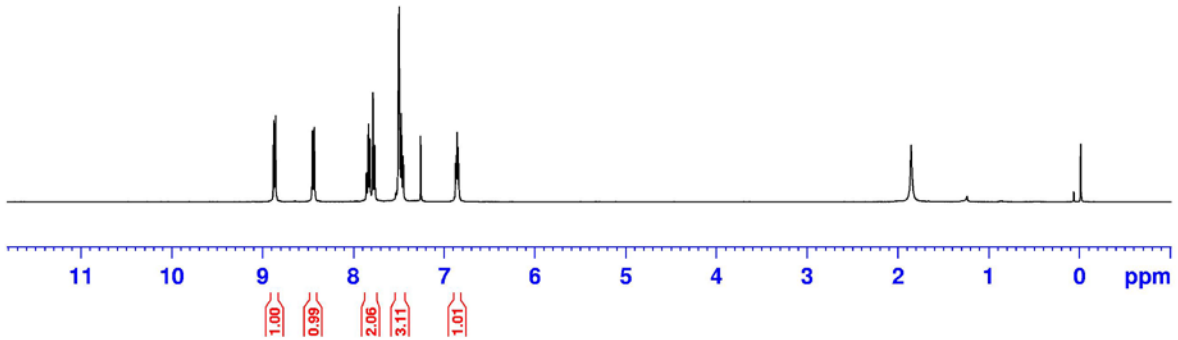
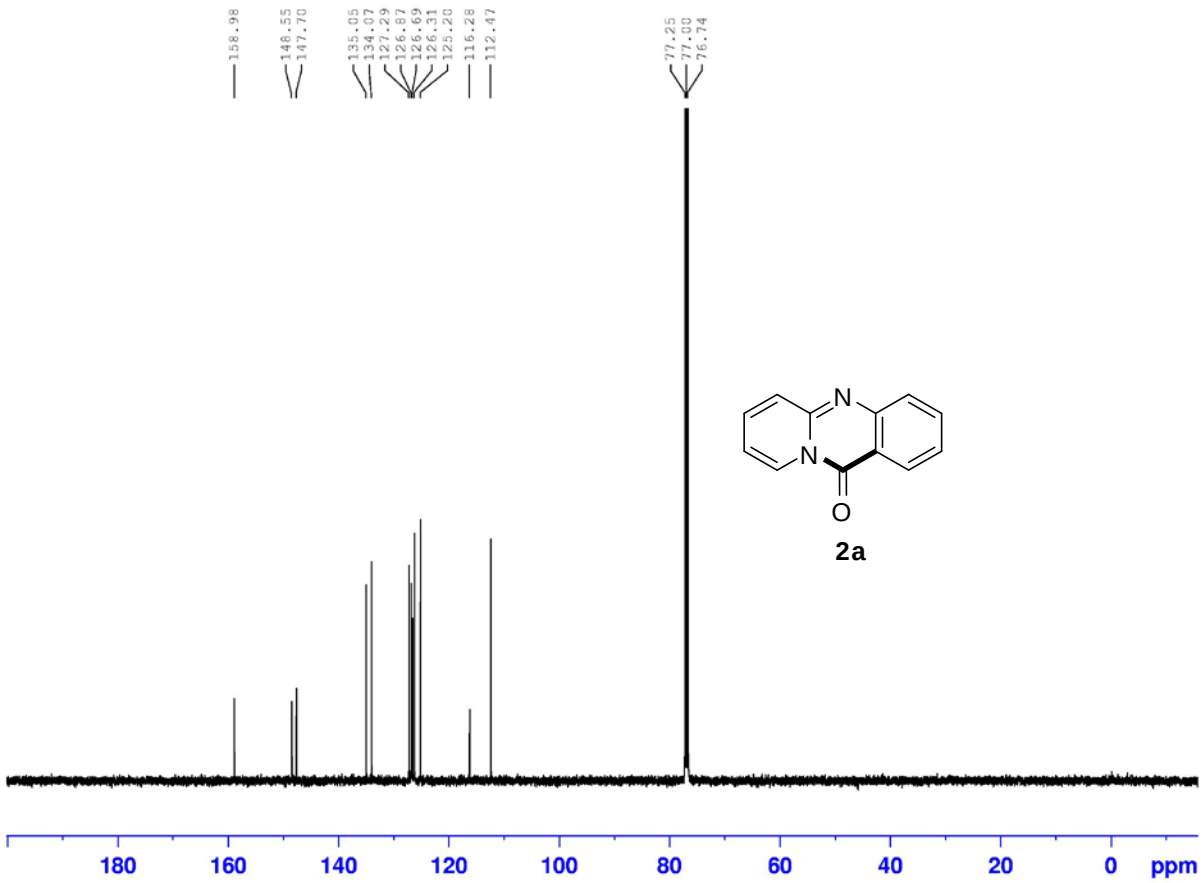


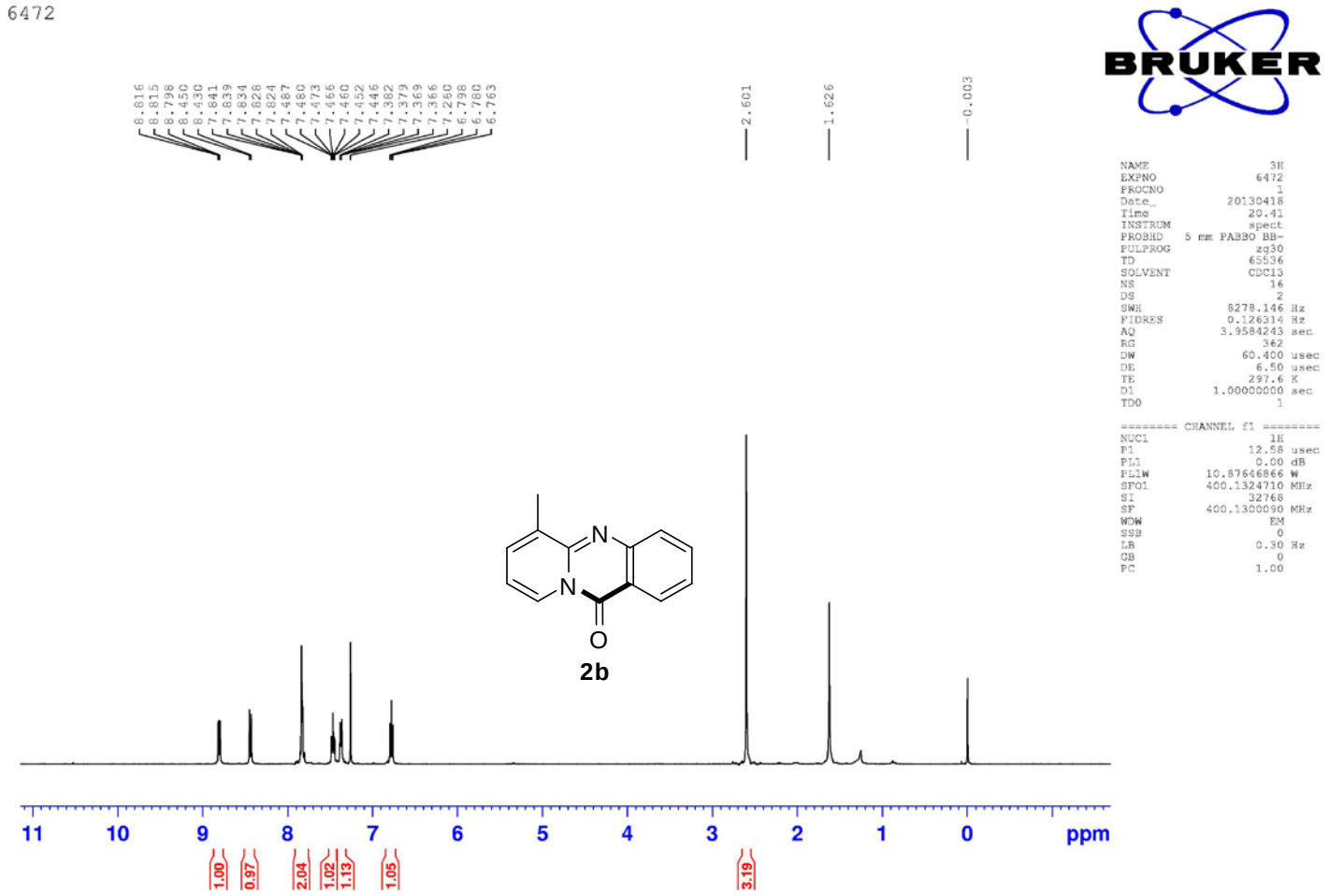
2a



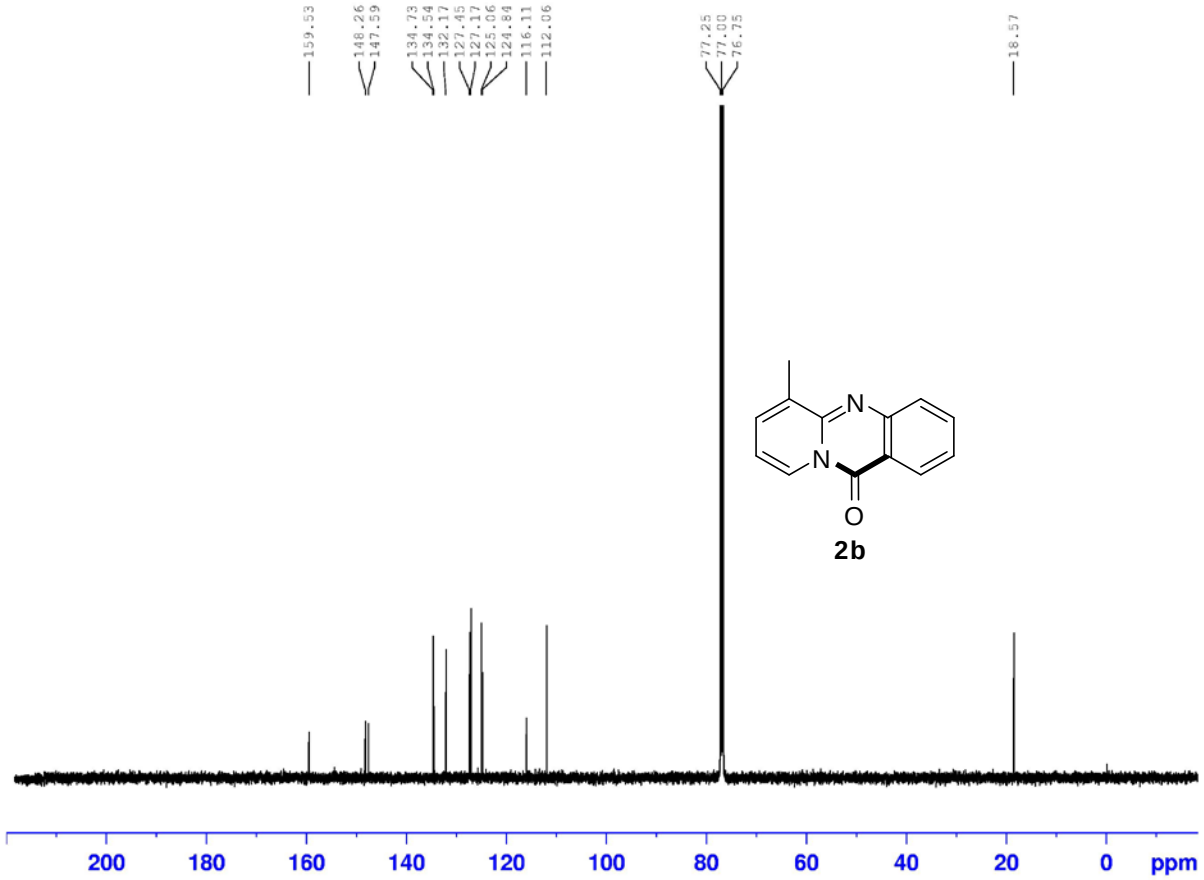
LDD1314



NAME	3C
EXPNO	1314
PROCNO	1
Date_	20130419
Time	17.21
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	655
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	295.7 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.66 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	125.7577959 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

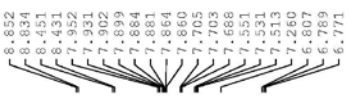

6472
8.816
8.815
8.798
8.450
8.430
7.841
7.839
7.834
7.828
7.824
7.487
7.480
7.473
7.466
7.460
7.452
7.446
7.382
7.379
7.369
7.366
7.260
6.798
6.780
6.763
2.601
1.626
0.003
BRUKER
NAME 3H
EXPNO 6472
PROCNO 1
Date_ 20130418
Time 20.41
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
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 297.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300090 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
N
N
O
2b
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
0.97
2.04
1.02
1.13
1.05
3.19

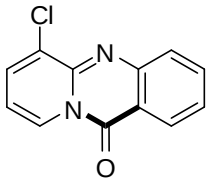
LDD6472



NAME	2C
EXPNO	6472
PROCNO	1
Date_	20130419
Time	19.52
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
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	295.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.66 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	125.7577933 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

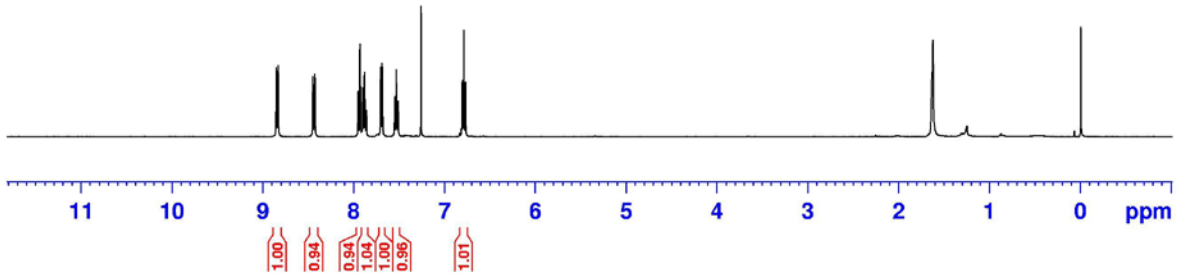
LDD-6506

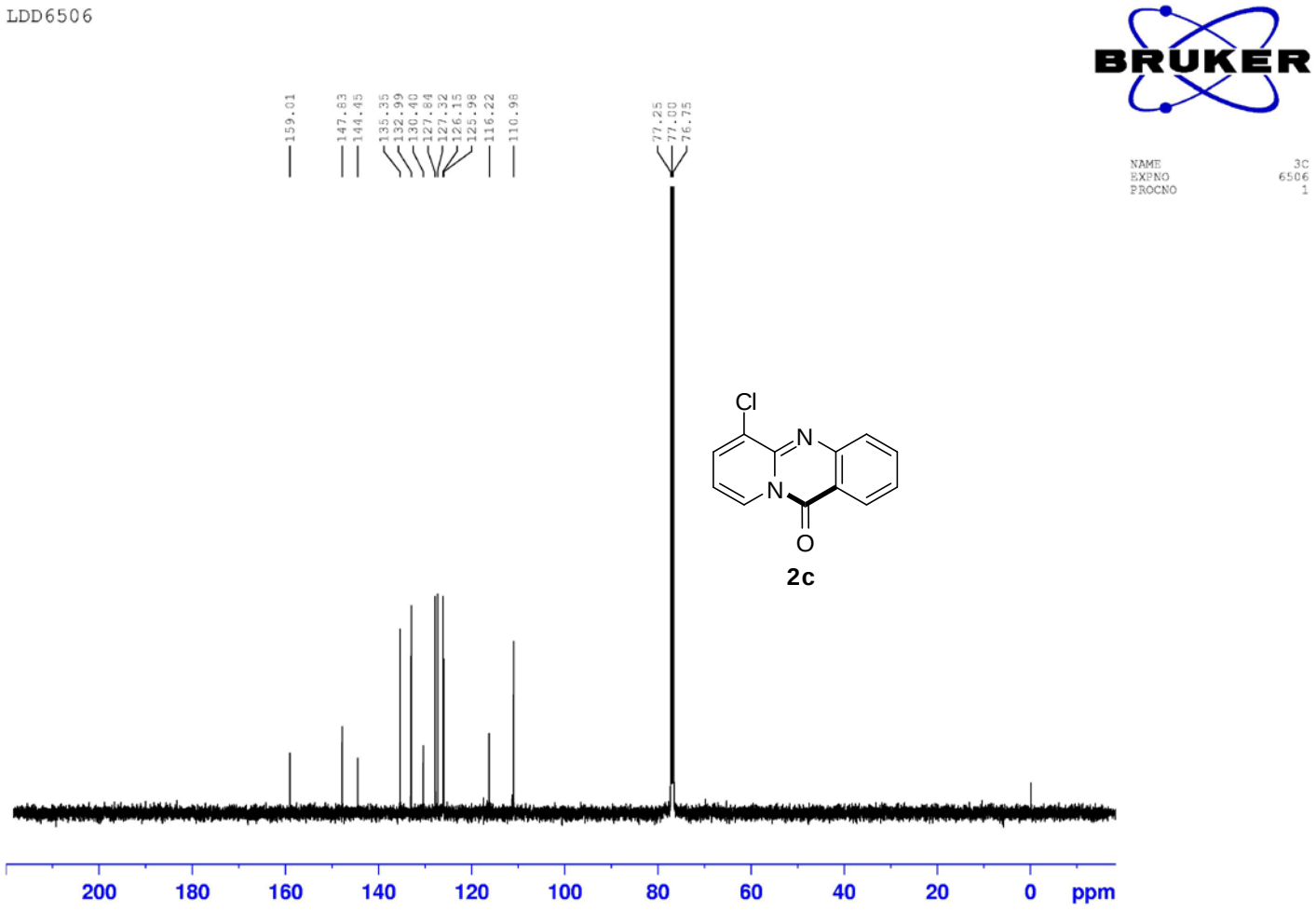


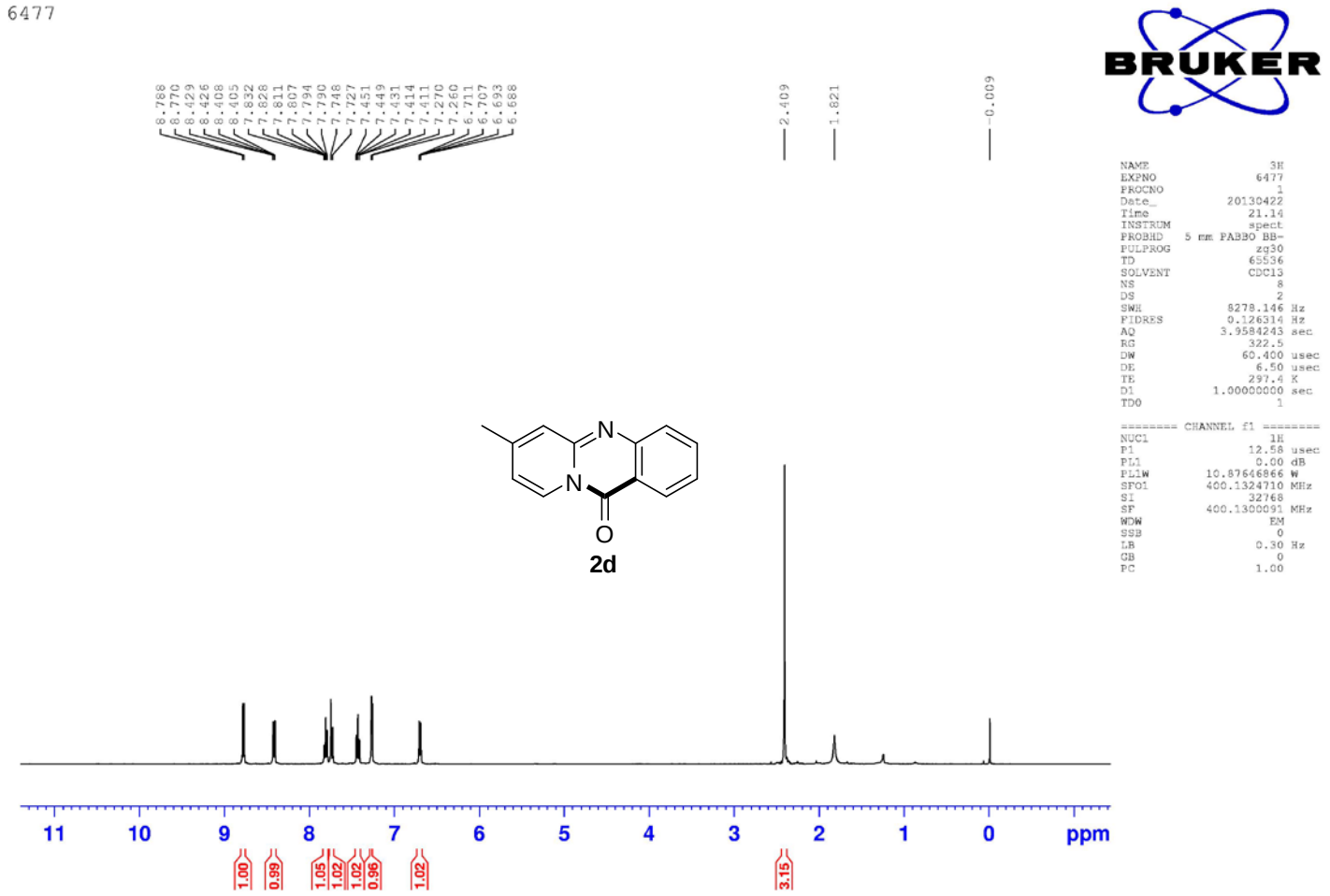


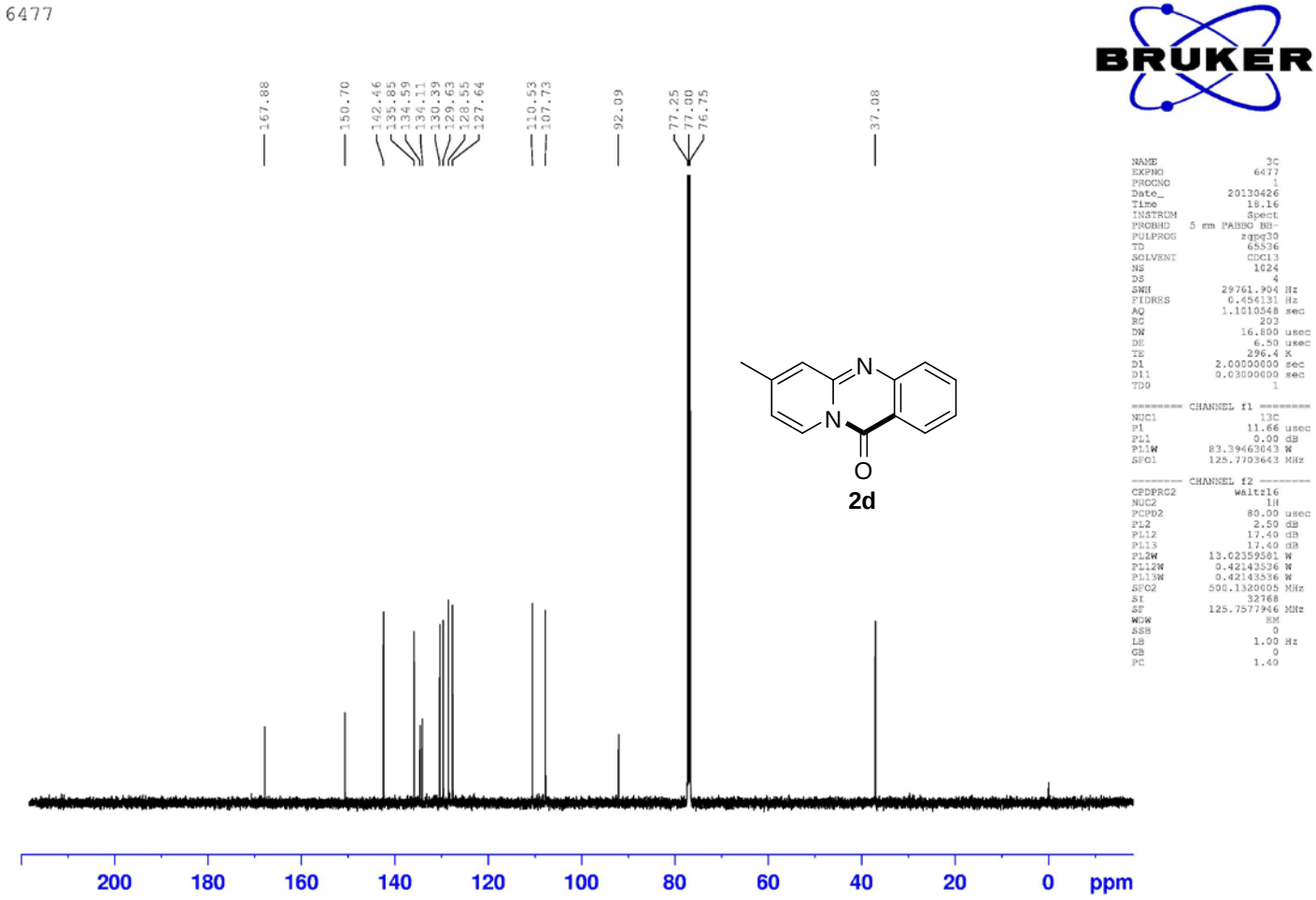
2c

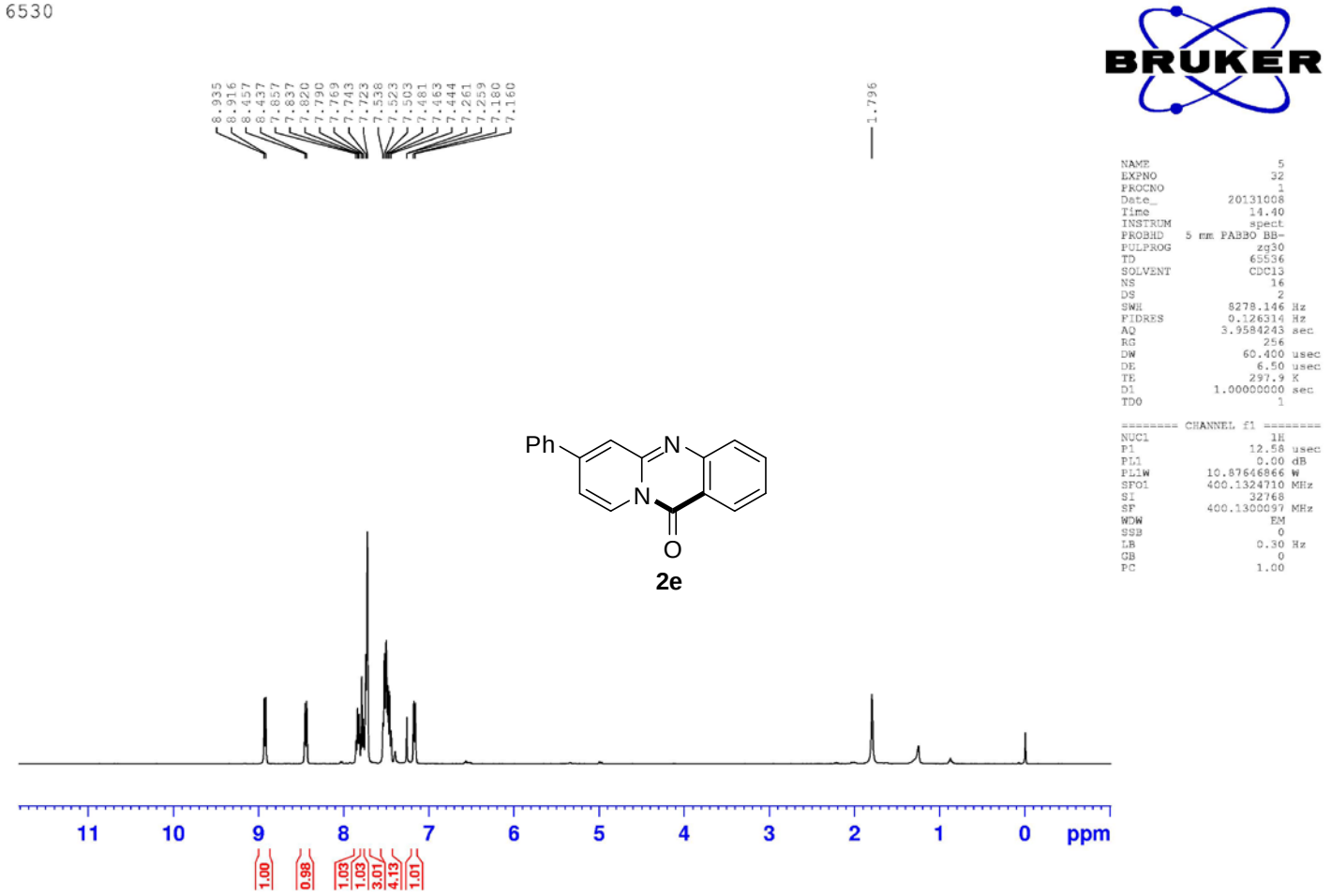
NAME	3H
EXPNO	6506
PROCNO	6506
Date_	20130508
Time	19.29
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	322.5
DW	60.400 usec
DE	6.50 usec
TE	297.8 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300090 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

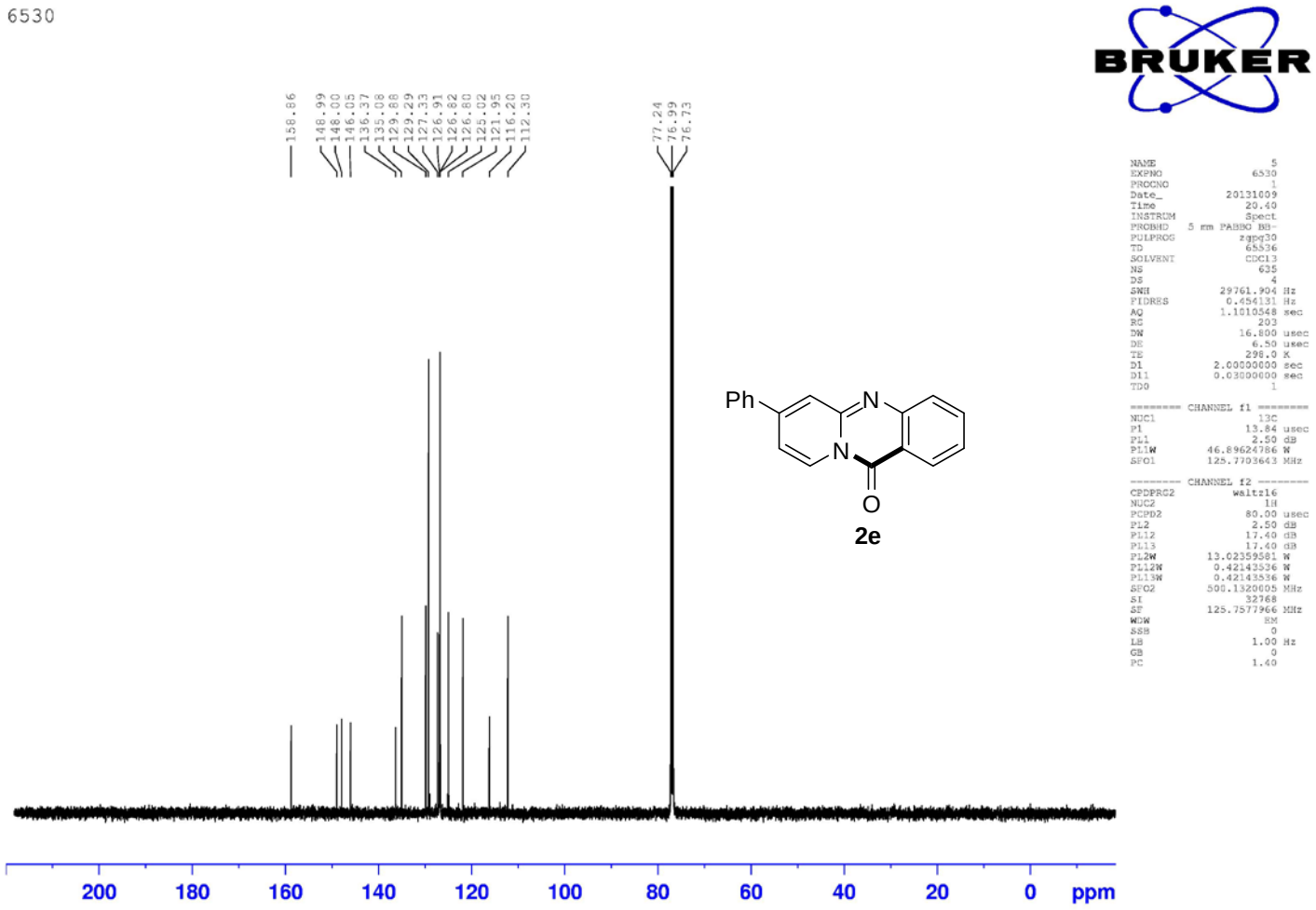


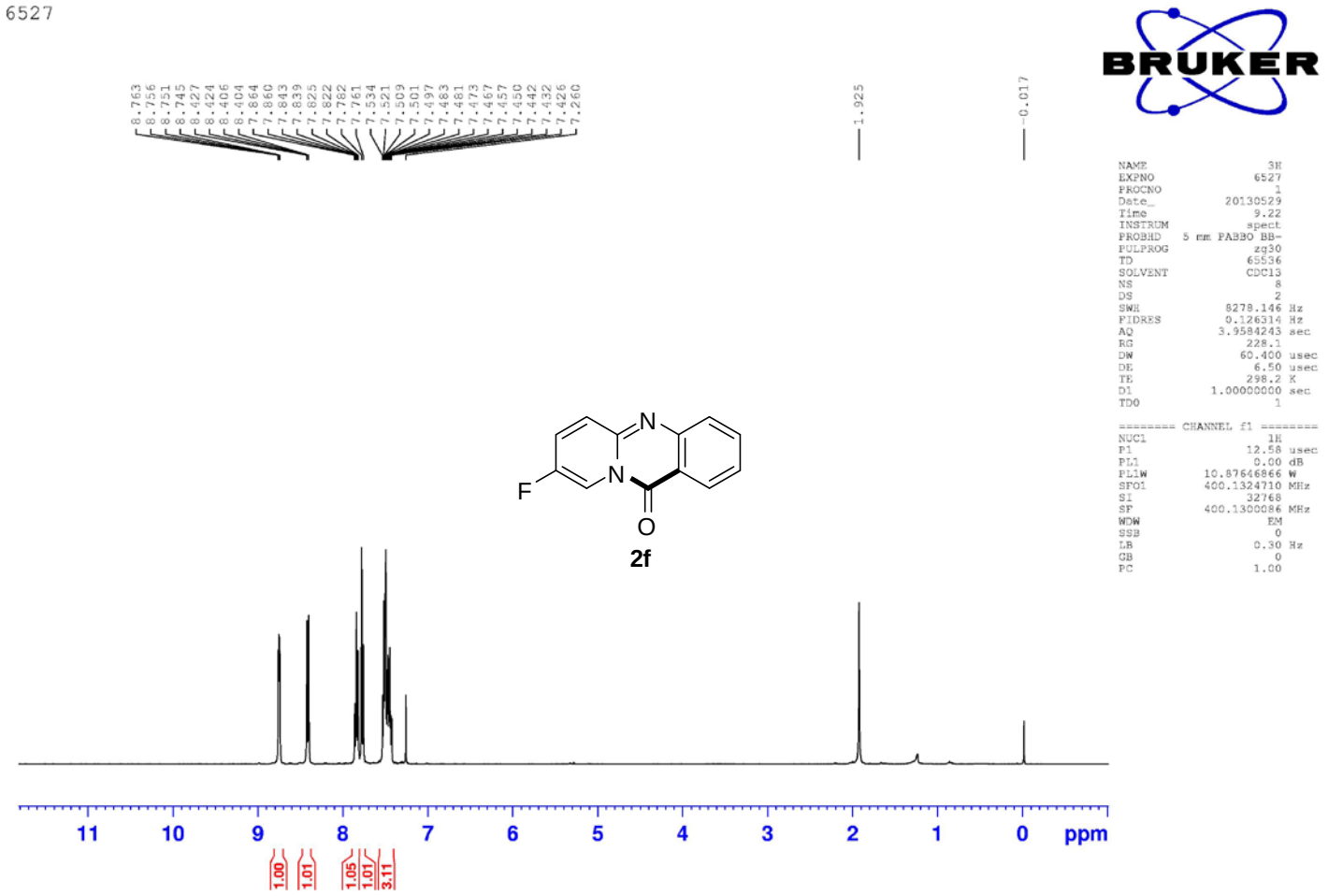

LDD6506
BRUKER
159.01
147.83
144.45
135.35
132.99
130.40
127.84
127.32
126.15
125.98
116.22
110.98
77.25
77.00
76.75
NAME 3C
EXPNO 6506
PROCNO 1
Cl
N
N
O
2c
200
180
160
140
120
100
80
60
40
20
0
ppm

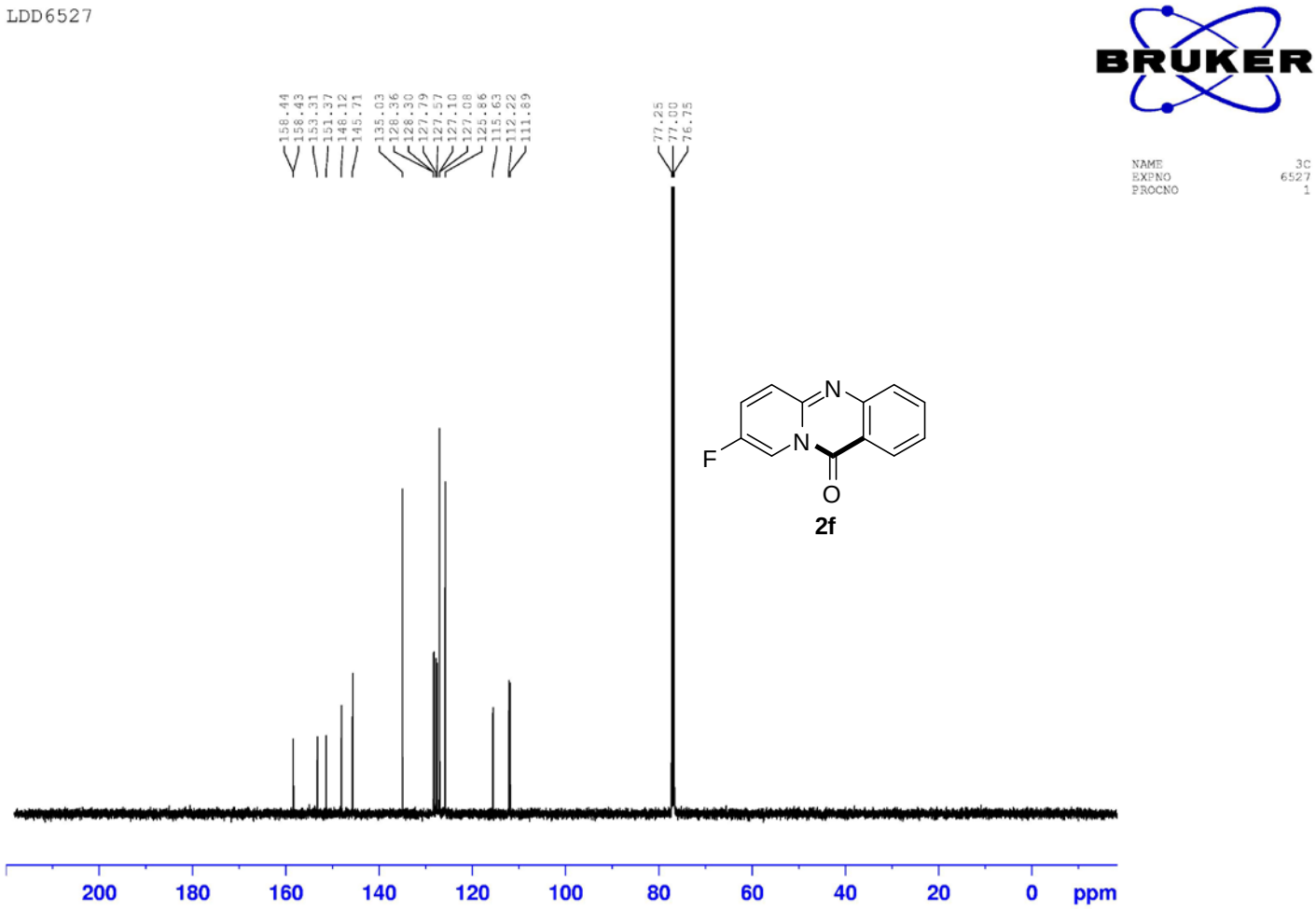

6477
8.788
8.770
8.429
8.426
8.408
8.405
7.832
7.828
7.811
7.807
7.794
7.790
7.748
7.727
7.451
7.449
7.431
7.414
7.411
7.270
7.260
6.711
6.707
6.693
6.688
2.409
1.821
-0.009
BRUKER
NAME 3H
EXPNO 6477
PROCNO 1
Date_ 20130422
Time 21.14
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.50 usec
TE 297.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300091 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
N
N
O
2d
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
0.99
1.05
1.02
1.02
0.96
1.02
3.15


6477
167.88
150.70
142.46
135.85
134.59
134.11
130.39
129.63
128.55
127.64
110.53
107.73
92.09
77.25
77.00
76.75
37.08
N
N
O
2d
BRUKER
NAME 3C
EXPNO 6477
PROCNO 1
Date_ 20130426
Time 18.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
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 296.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.66 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 125.7577946 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

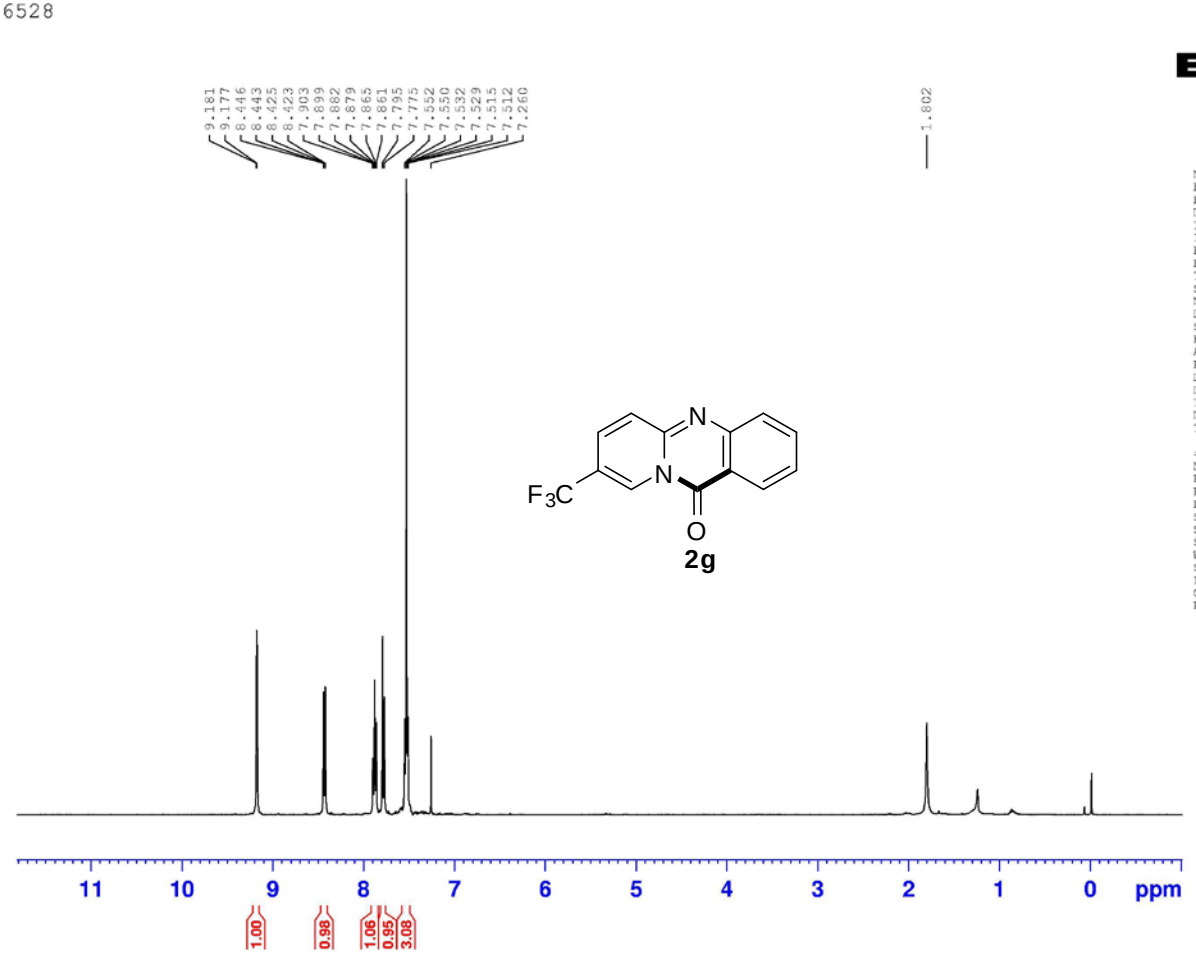

6530
8.935
8.916
8.457
8.437
7.857
7.837
7.820
7.790
7.769
7.743
7.723
7.538
7.523
7.503
7.481
7.463
7.444
7.261
7.259
7.180
7.160
1.796
BRUKER
NAME 5
EXPNO 32
PROCNO 1
Date_ 20131008
Time 14.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DW 60.400 usec
DE 6.50 usec
TE 297.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Ph
N
N
O
2e
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
0.98
1.03
1.03
3.01
4.13
1.01


6530
158.86
148.99
148.00
146.05
136.37
135.08
129.88
129.29
127.33
126.91
126.82
126.80
125.02
121.95
116.20
112.30
77.24
76.99
76.73
Ph
N
N
O
2e
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER
NAME 5
EXPNO 6530
PROCNO 1
Date_ 20131009
Time 20.40
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 635
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 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 13.84 usec
PL1 2.50 dB
PL1W 46.89624786 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 125.7577966 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

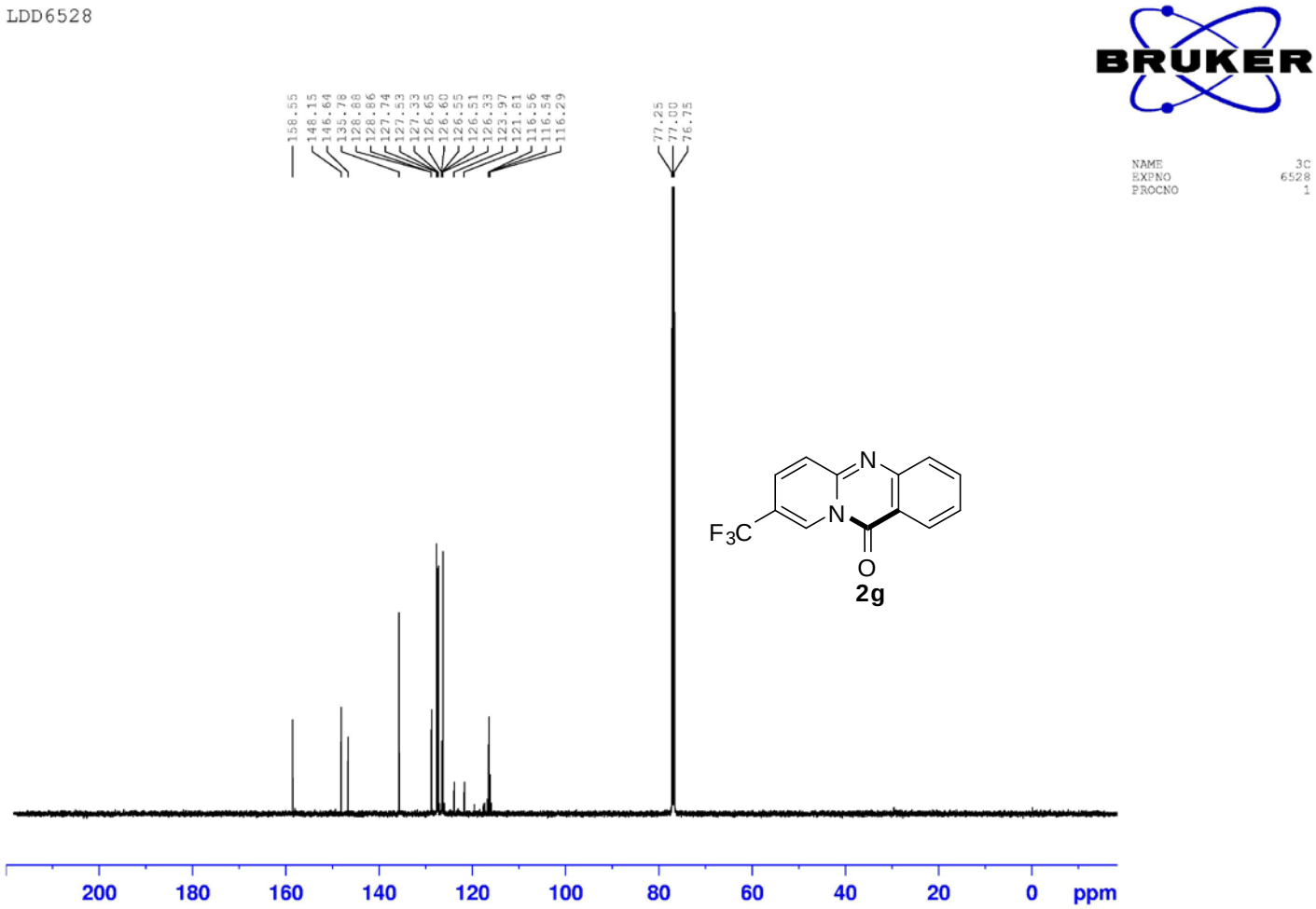

6527
8.763
8.756
8.751
8.745
8.427
8.424
8.406
8.404
7.864
7.860
7.843
7.839
7.825
7.822
7.782
7.761
7.534
7.521
7.509
7.501
7.497
7.483
7.481
7.473
7.467
7.457
7.450
7.442
7.432
7.426
7.260
1.925
0.017
BRUKER
NAME 3H
EXPNO 6527
PROCNO 1
Date_ 20130529
Time 9.22
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 228.1
DW 60.400 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300086 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
F
N
N
O
2f
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.01
1.05
1.01
3.11


LDD6527
158.44
158.43
153.31
151.37
146.12
145.71
135.03
128.36
128.30
127.79
127.57
127.10
127.08
125.86
115.63
112.22
111.89
77.25
77.00
76.75
NAME 3C
EXPNO 6527
PROCNO 1
N
N
F
O
2f
200
180
160
140
120
100
80
60
40
20
0
ppm

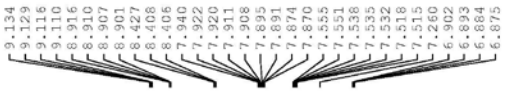
6528



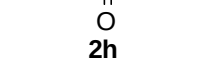
NAME	3H
EXPNO	6528
PROCNO	1
Date_	20130529
Time	9.28
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	228.1
DW	60.400 usec
DE	6.50 usec
TE	298.1 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300087 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00


LDD6528
BRUKER
NAME 3C
EXPNO 6528
PROCNO 1
158.55
148.15
146.64
135.78
128.88
128.86
127.74
127.53
127.33
126.65
126.60
126.55
126.51
126.33
123.97
121.81
116.56
116.54
116.29
77.25
77.00
76.75
N
N
F3C
O
2g
200
180
160
140
120
100
80
60
40
20
0
ppm

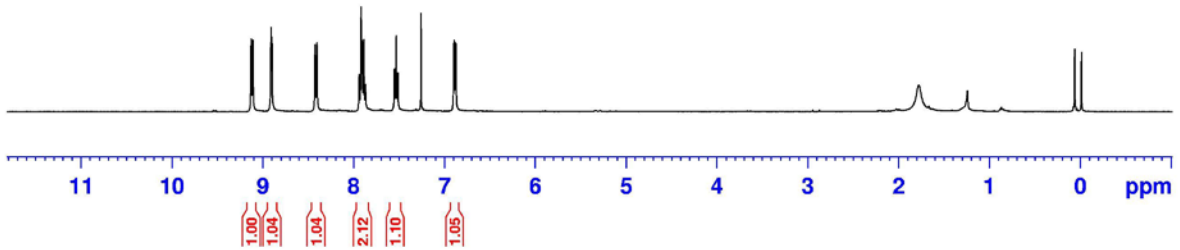
LDD-6487

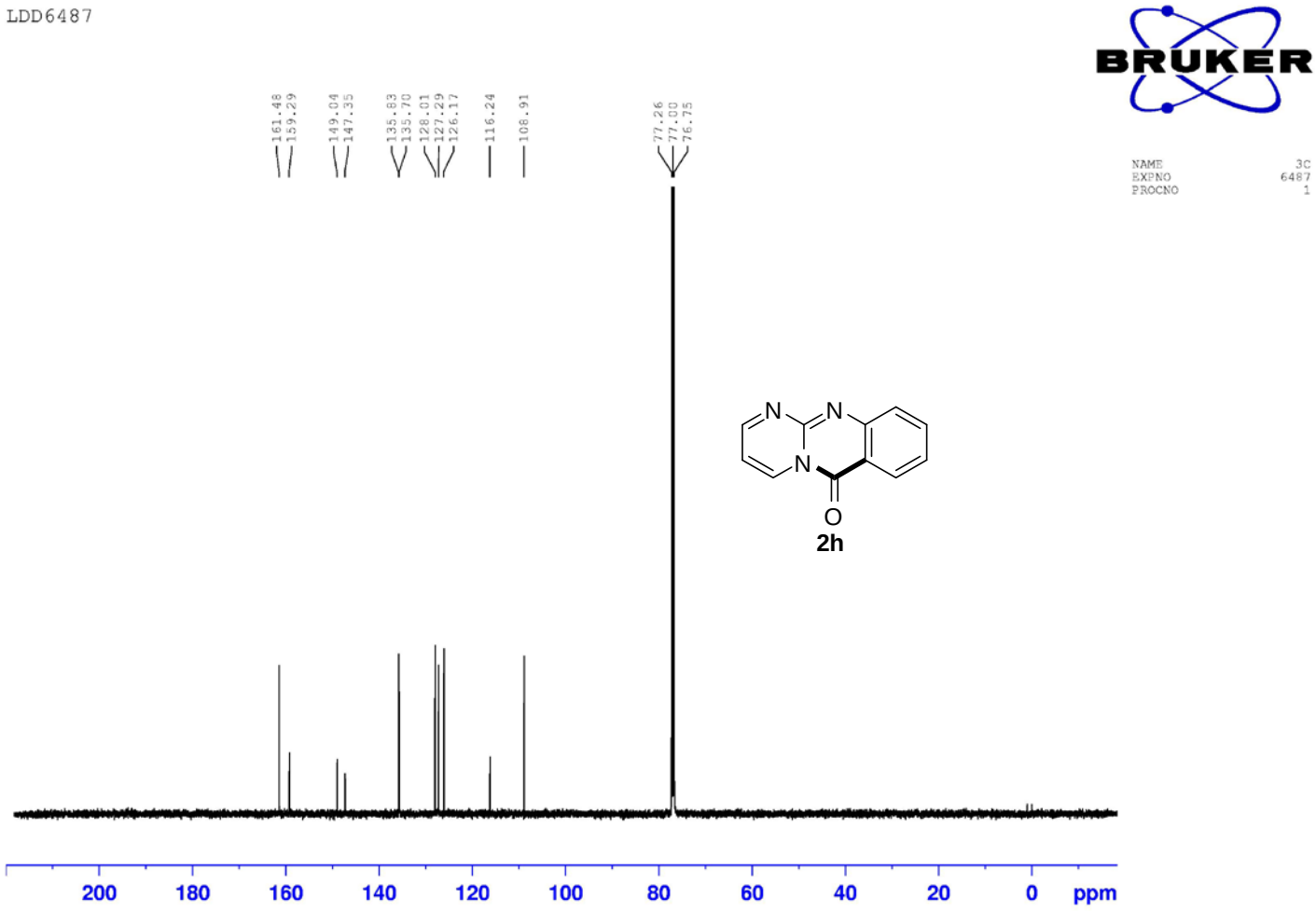


NAME	3H
EXPNO	6487
PROCNO	1
Date_	20130424
Time	23.39
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	322.5
DW	60.400 usec
DE	6.50 usec
TE	298.8 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300090 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

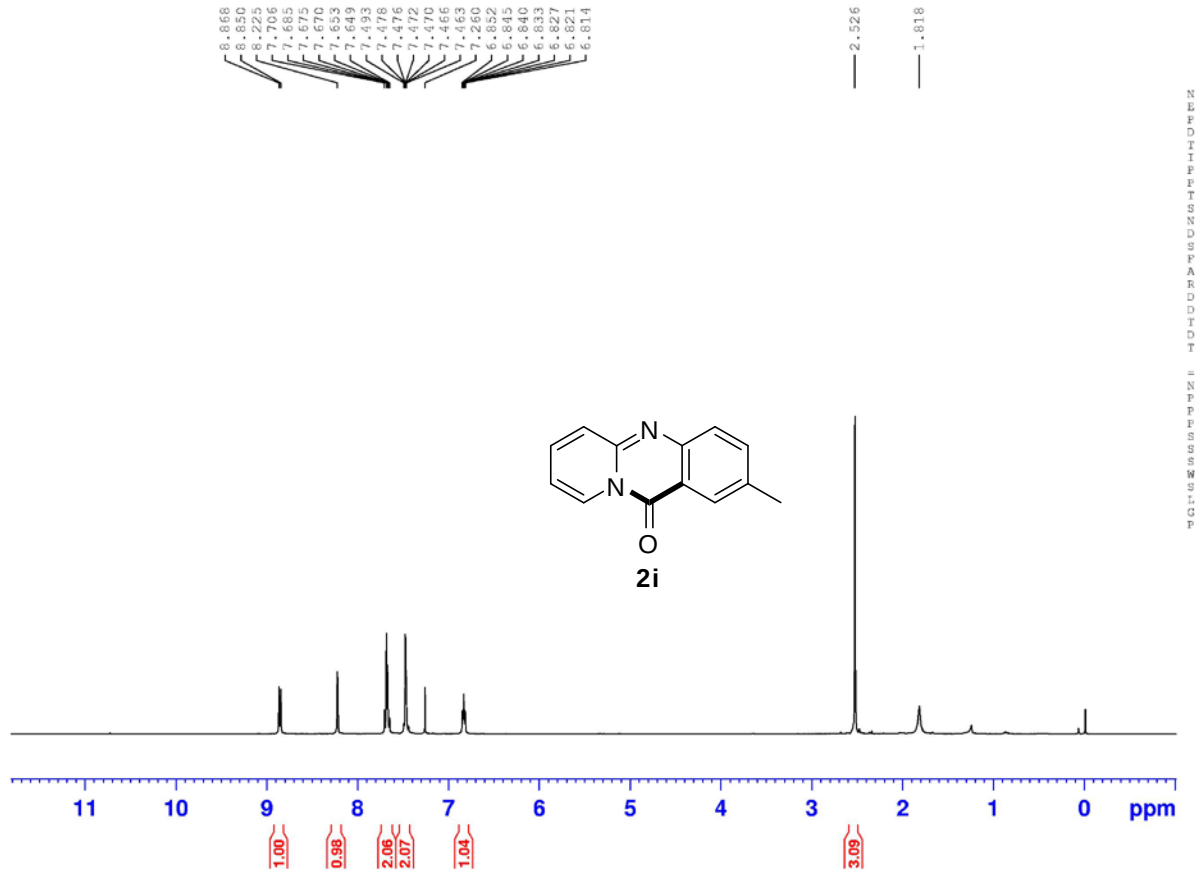


2h




LDD6487
BRUKER
NAME 3C
EXPNO 6487
PROCNO 1
161.48
159.29
149.04
147.35
135.83
135.70
128.01
127.29
126.17
116.24
108.91
77.26
77.00
76.75
N
N
N
O
2h
200
180
160
140
120
100
80
60
40
20
0
ppm

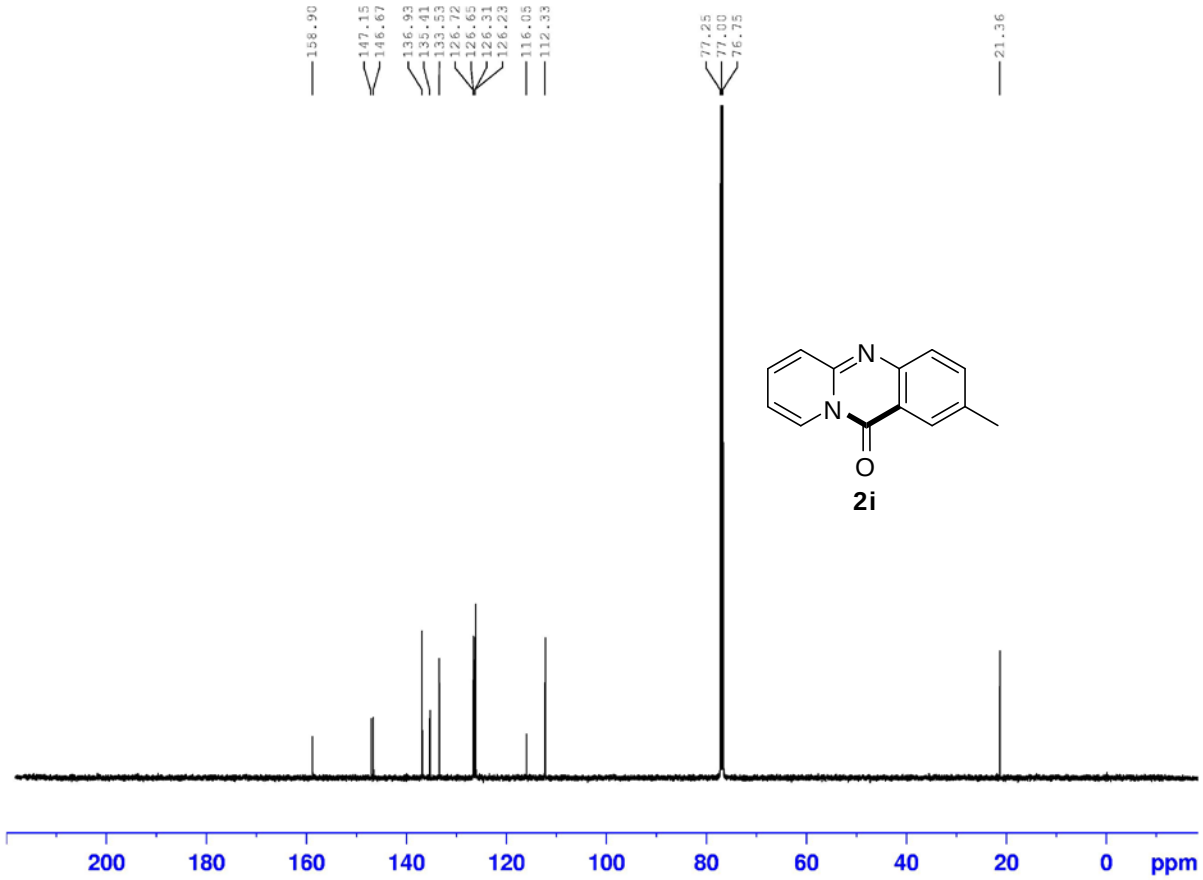
LDD-6467



BRUKER

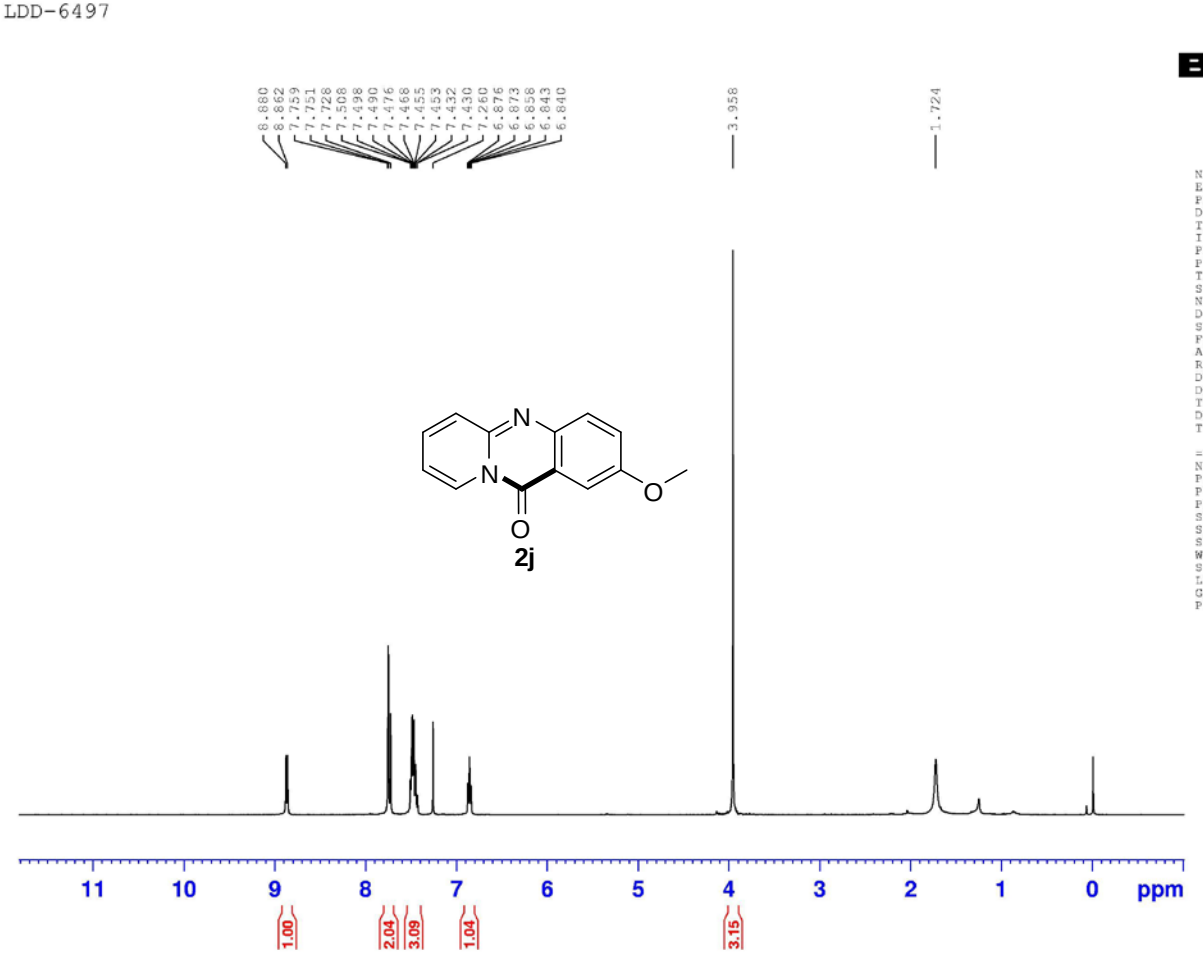
NAME	3H
EXPNO	62
PROCNO	1
Date_	20130417
Time	22.17
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	322.5
DW	60.400 usec
DE	6.50 usec
TE	297.9 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300090 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

LDD6467



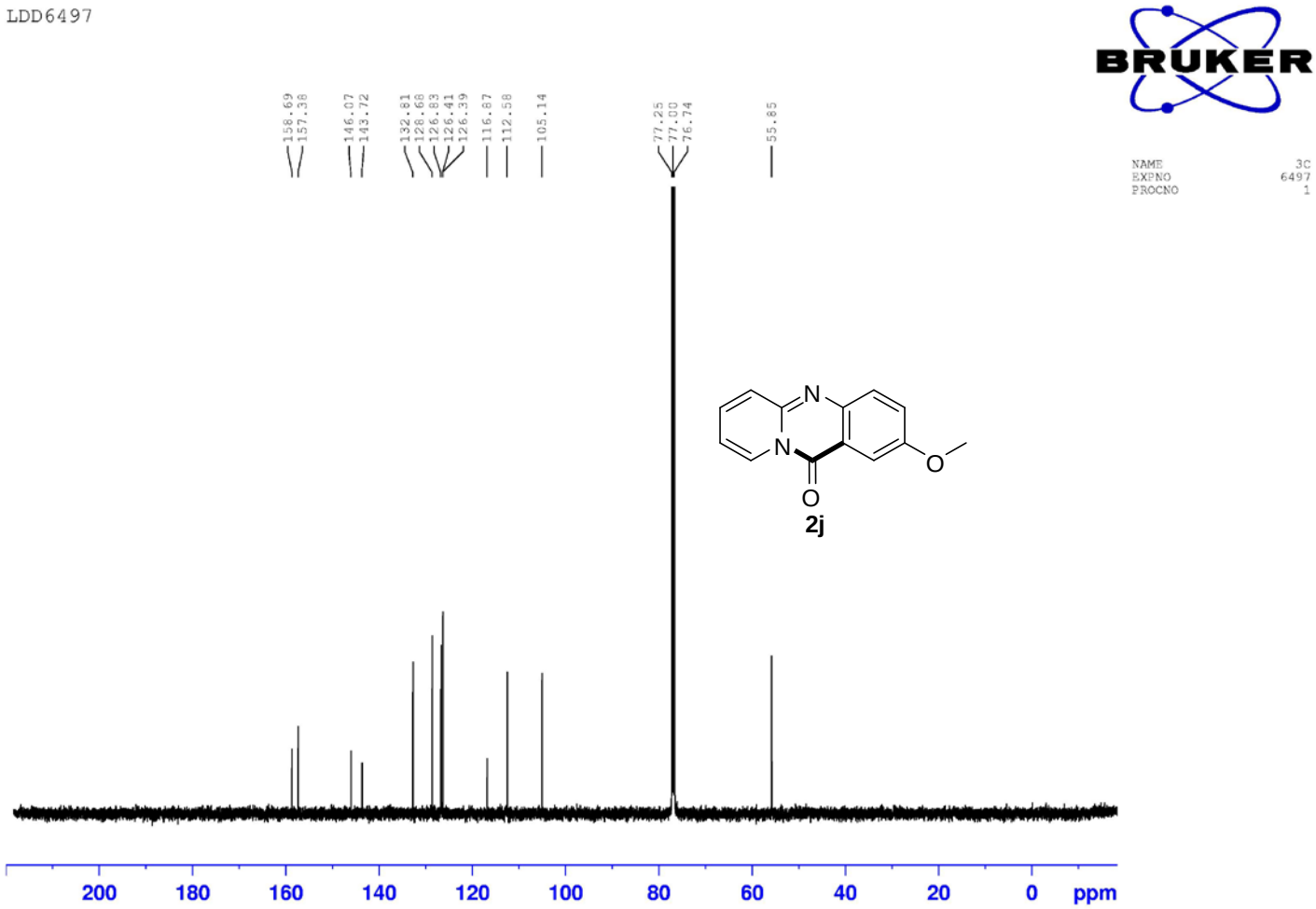

```
NAME                  3C
EXPNO                 6467
PROCNO                   1
Date_             20130419
Time                 16.03
INSTRUM              spect
PROBHD     5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   883
DS                     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                11.66 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        125.7577950 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

LDD-6497

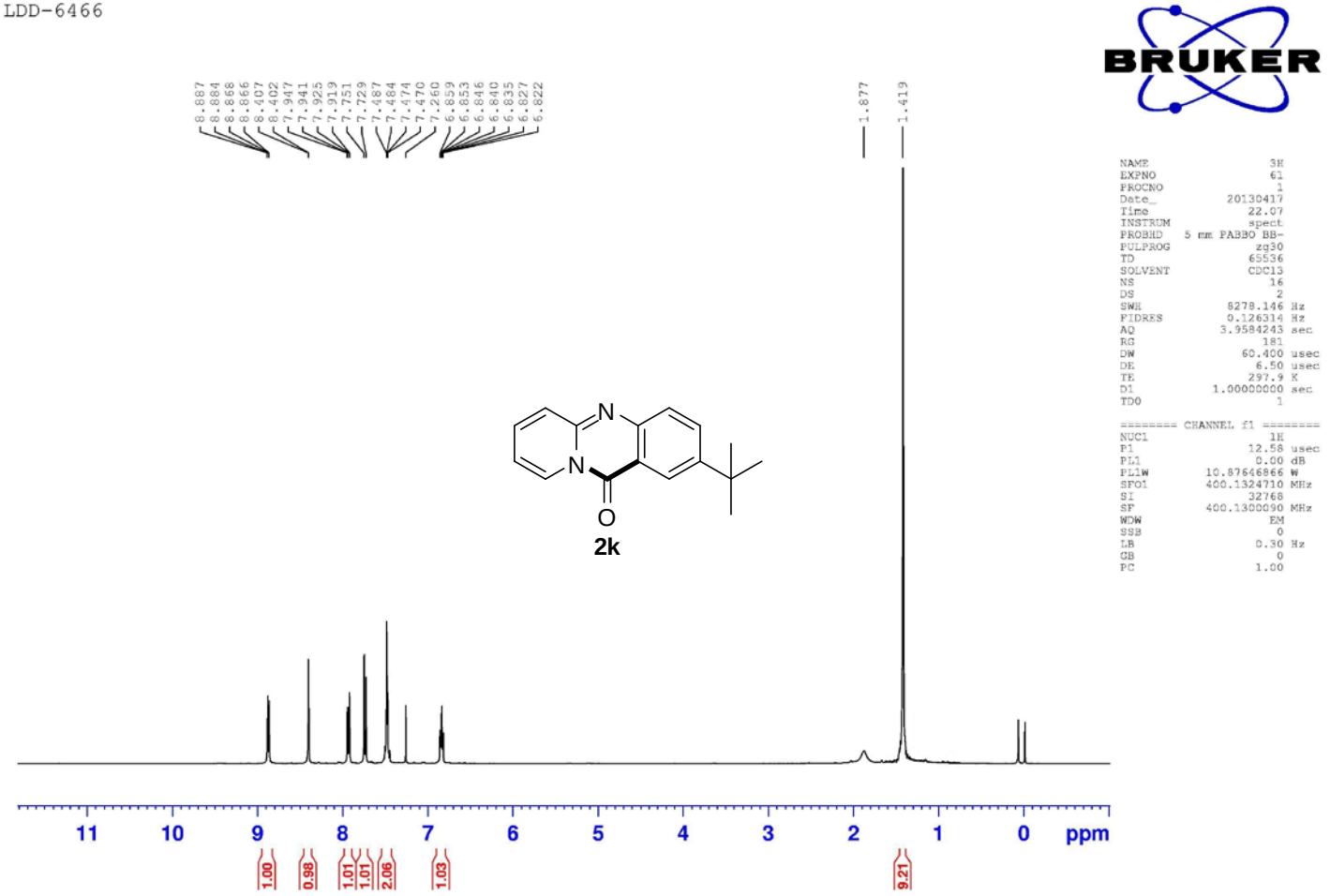


BRUKER

NAME	3H
EXPNO	6497
PROCNO	1
Date_	20130504
Time	19.07
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	322.5
DW	60.400 usec
DE	6.50 usec
TE	297.5 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300087 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

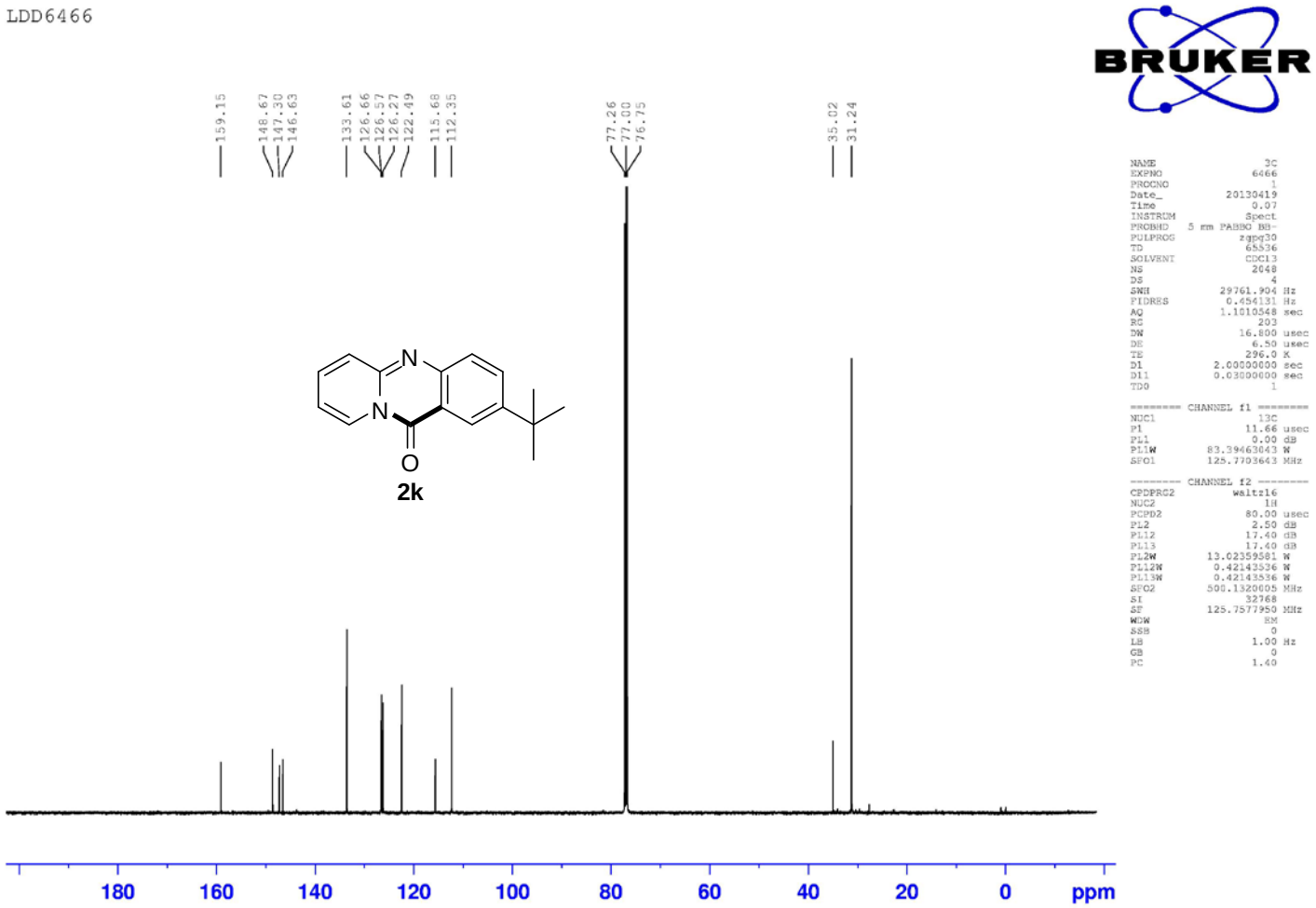

LDD6497
BRUKER
158.69
157.38
146.07
143.72
132.81
126.68
126.83
126.41
126.39
116.87
112.58
105.14
77.25
77.00
76.74
55.85
NAME 3C
EXPNO 6497
PROCNO 1
N
N
O
O
2j
200
180
160
140
120
100
80
60
40
20
0
ppm

LDD-6466



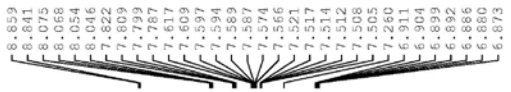
BRUKER

NAME	3H
EXPNO	61
PROCNO	1
Date_	20130417
Time	22.07
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	181
DW	60.400 usec
DE	6.50 usec
TE	297.9 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300090 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

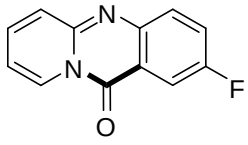


NAME	3C
EXPNO	6466
PROCNO	1
Date_	20130419
Time	0.07
INSTRUM	Spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	2048
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	296.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.66 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	125.7577950 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

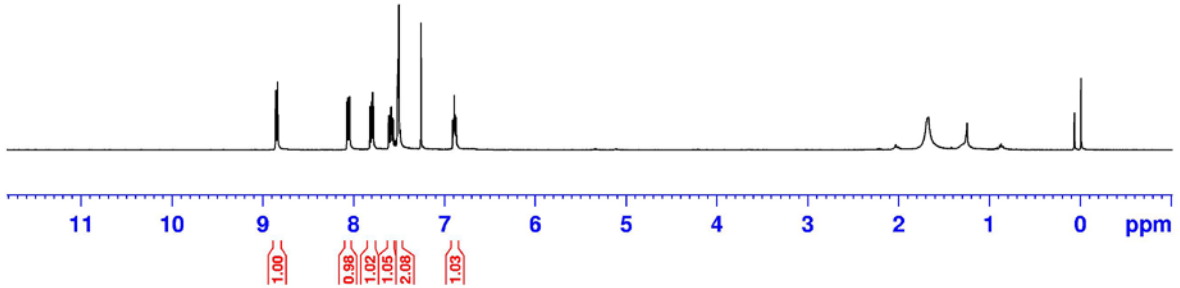
LDD-6476



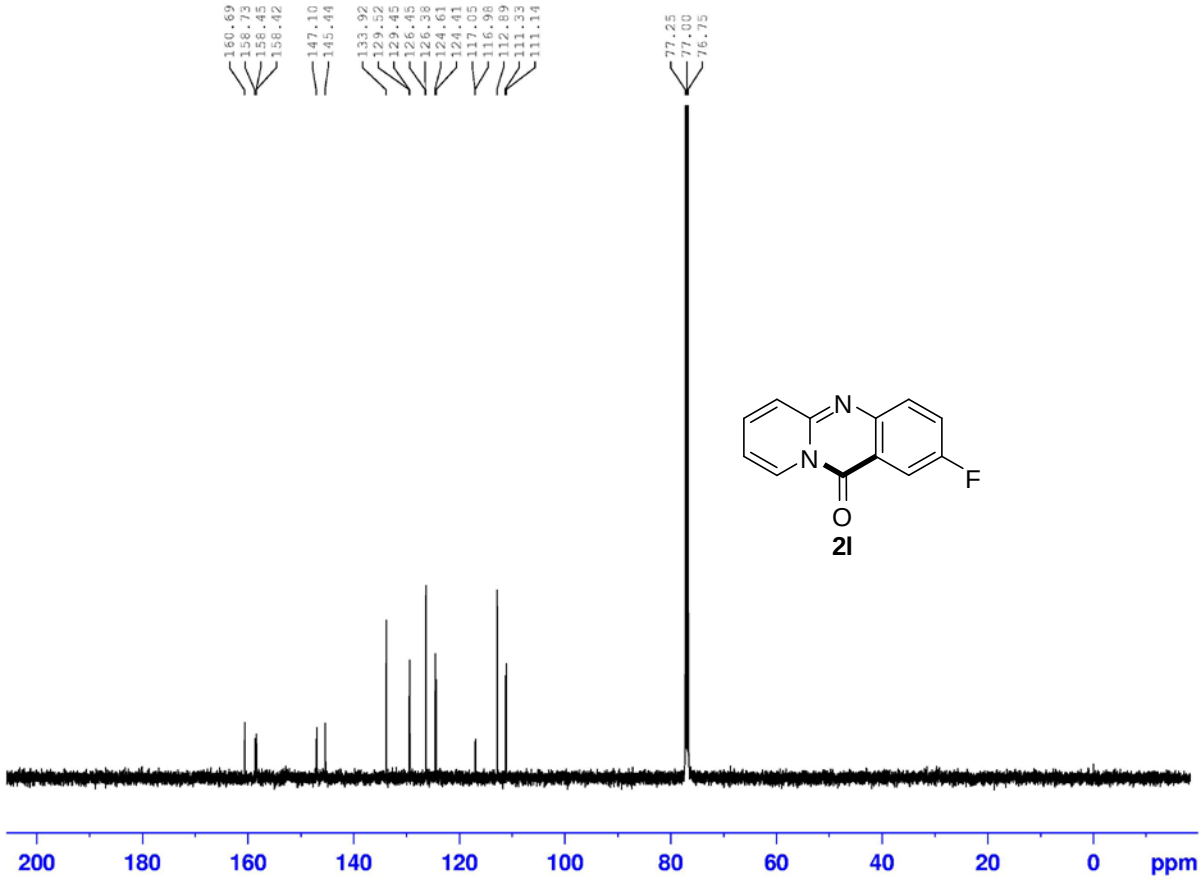
NAME	3H
EXPNO	6476
PROCNO	1
Date_	20130424
Time	23.26
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	322.5
DW	60.400 usec
DE	6.50 usec
TE	298.8 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300090 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



2l



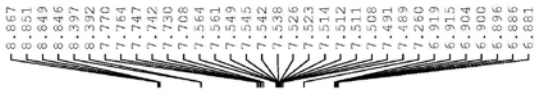
LDD6476



2l

```
NAME                  2C
EXPNO                 6476
PROCNO                   1
Date_              20130427
Time                 20.48
INSTRUM              spect
PROBHD     5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
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               296.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 11.66 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         125.7577937 MHz
WDW                EM
SSB                0
LB              1.00 Hz
GB                 0
PC                1.40
```

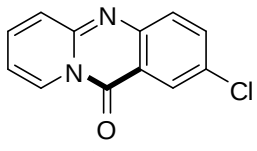
LDD-6482



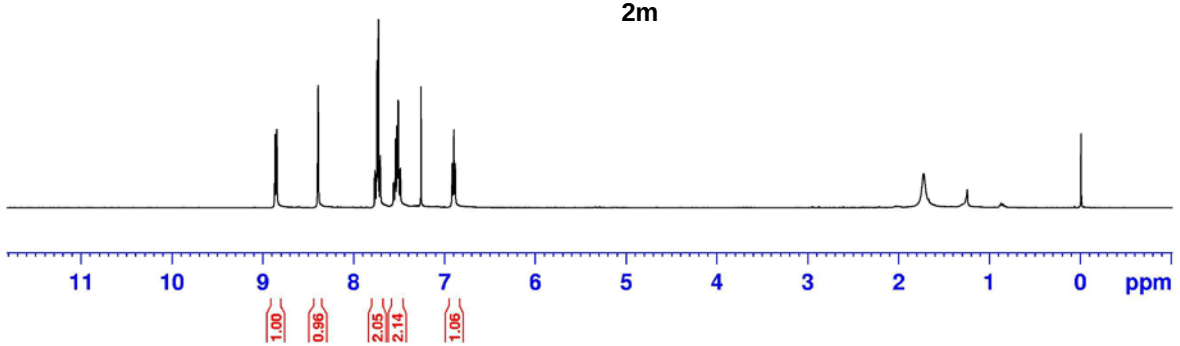
NAME	3H
EXPNO	6482
PROCNO	1
Date_	20130424
Time	23.33
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	322.5
DW	60.400 usec
DE	6.50 usec
TE	298.9 K
D1	1.00000000 sec
TD0	1

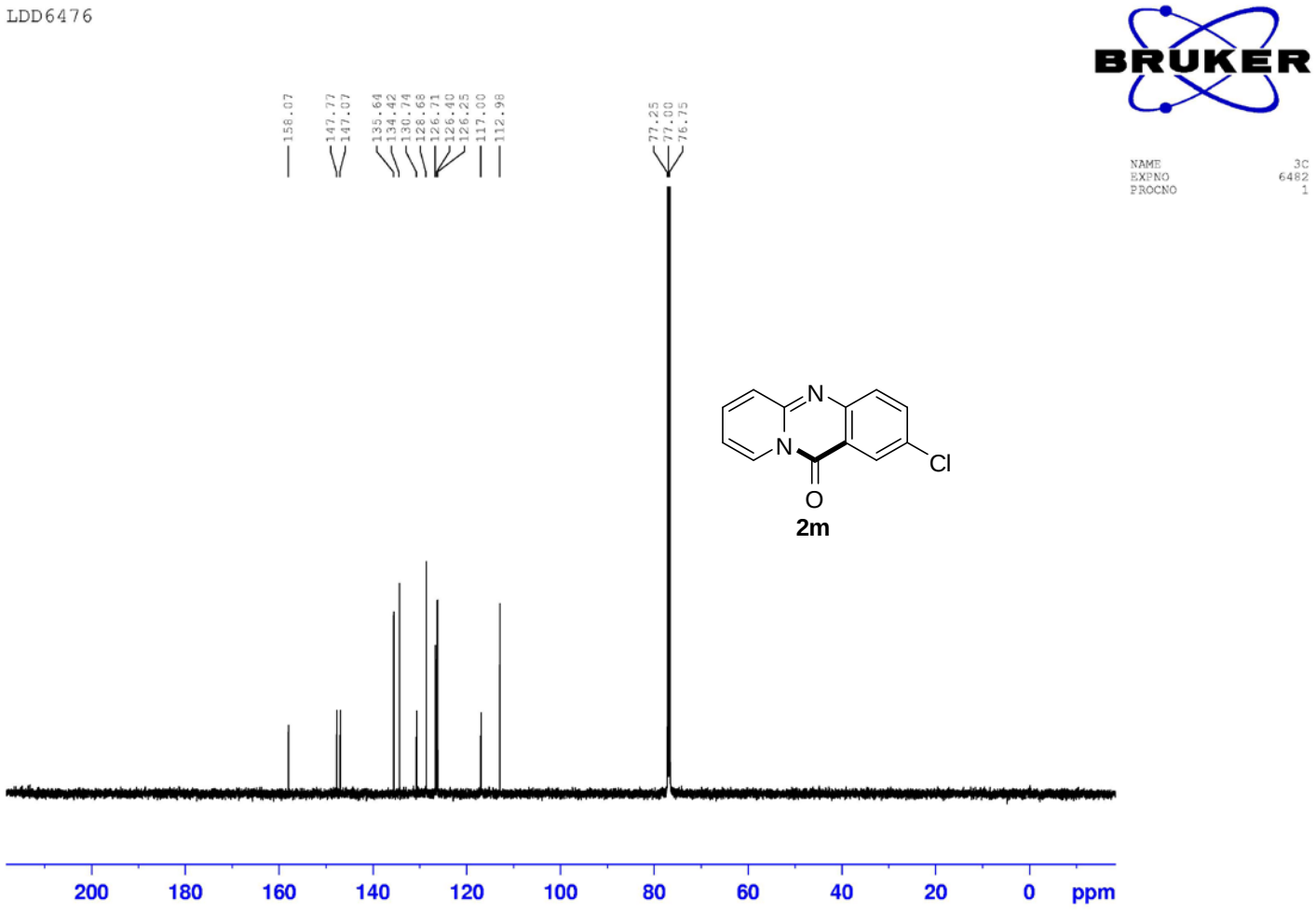
======== CHANNEL f1 ========

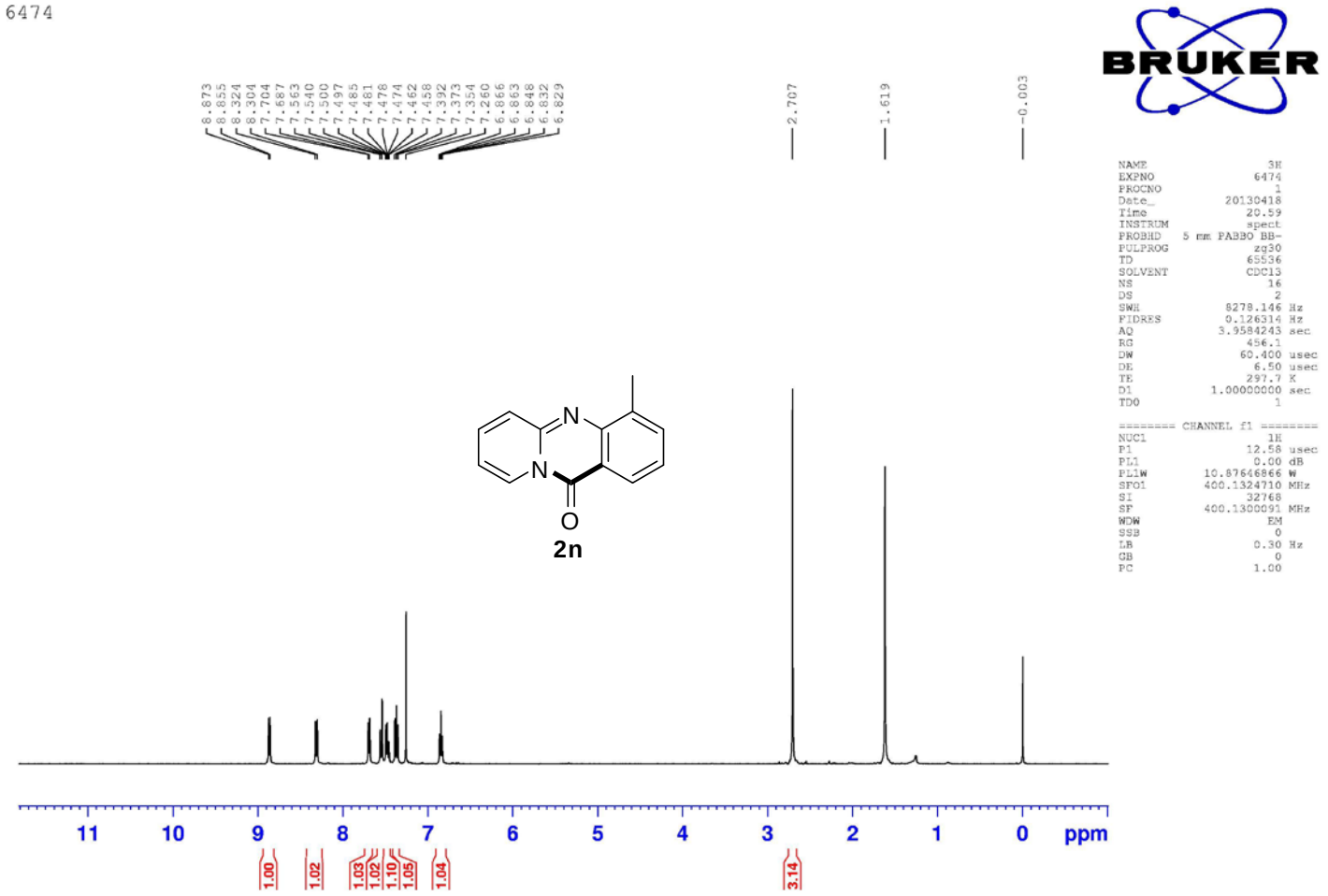
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300090 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



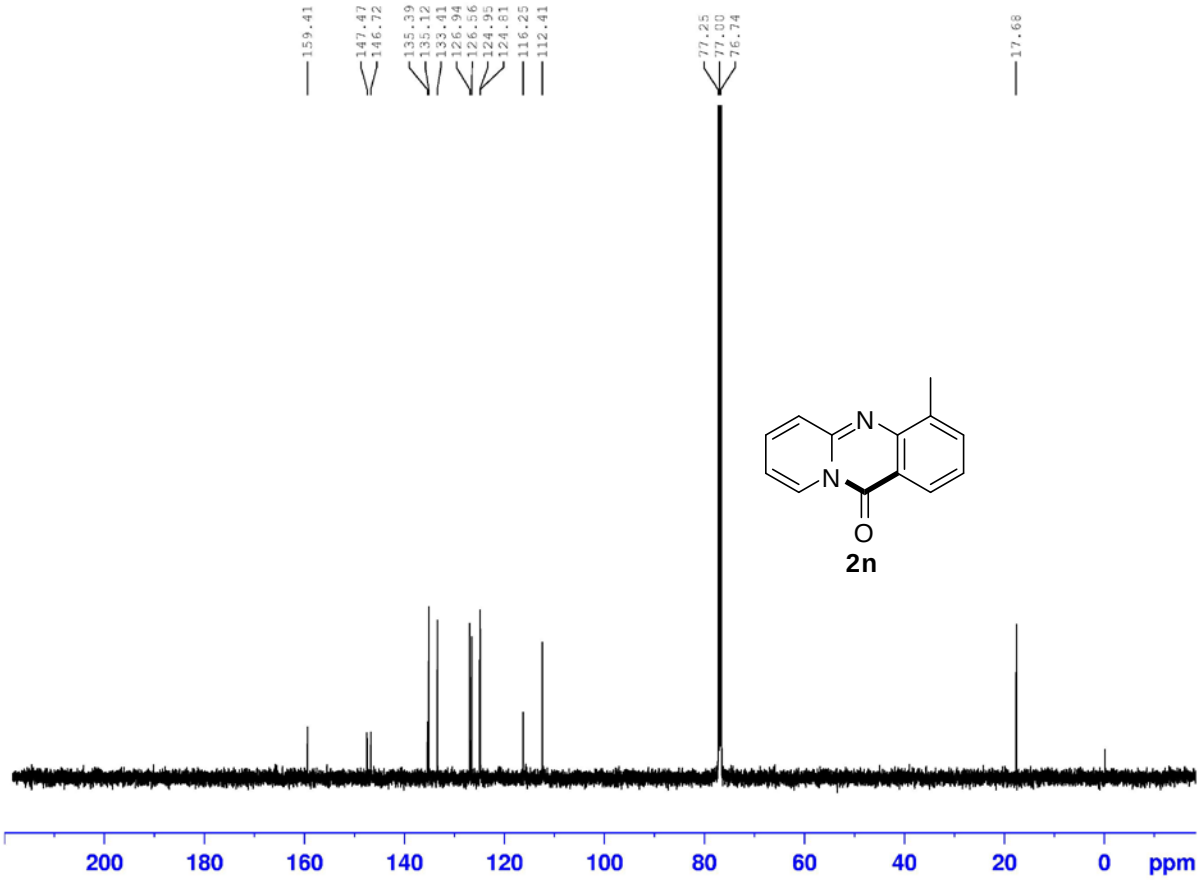
2m




LDD6476
BRUKER
NAME 3C
EXPNO 6482
PROCNO 1
156.07
147.77
147.07
135.64
134.42
130.74
128.68
126.71
126.40
126.25
117.00
112.98
77.25
77.00
76.75
N
N
Cl
O
2m
200
180
160
140
120
100
80
60
40
20
0
ppm


6474
8.873
8.855
8.324
8.304
7.704
7.687
7.563
7.540
7.500
7.497
7.485
7.481
7.478
7.474
7.462
7.458
7.392
7.373
7.354
7.260
6.866
6.863
6.848
6.832
6.829
2.707
1.619
-0.003
BRUKER
NAME 3H
EXPNO 6474
PROCNO 1
Date_ 20130418
Time 20.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 456.1
DW 60.400 usec
DE 6.50 usec
TE 297.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300091 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
N
N
O
2n
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.02
1.03
1.02
1.10
1.05
1.04
3.14

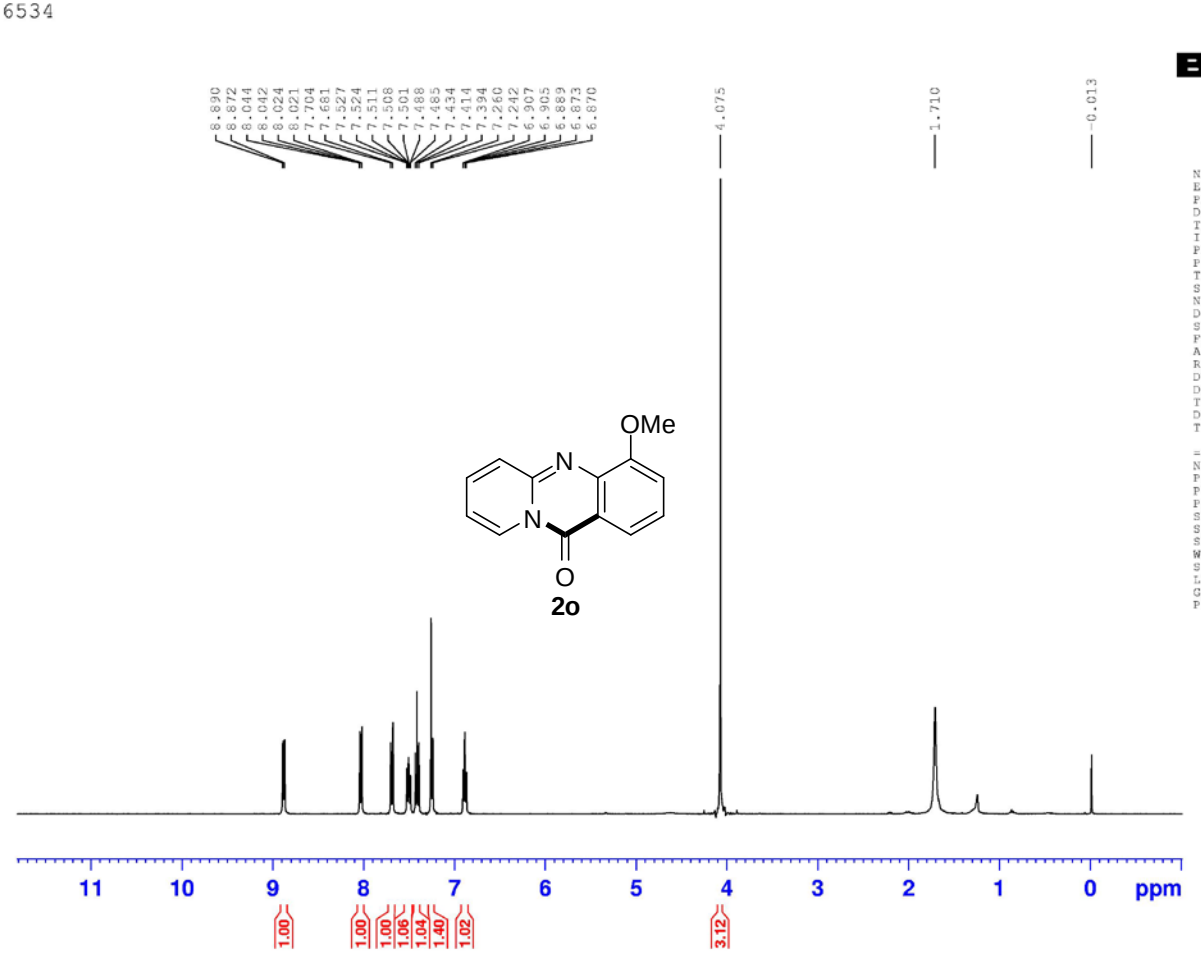
LDD6474



BRUKER

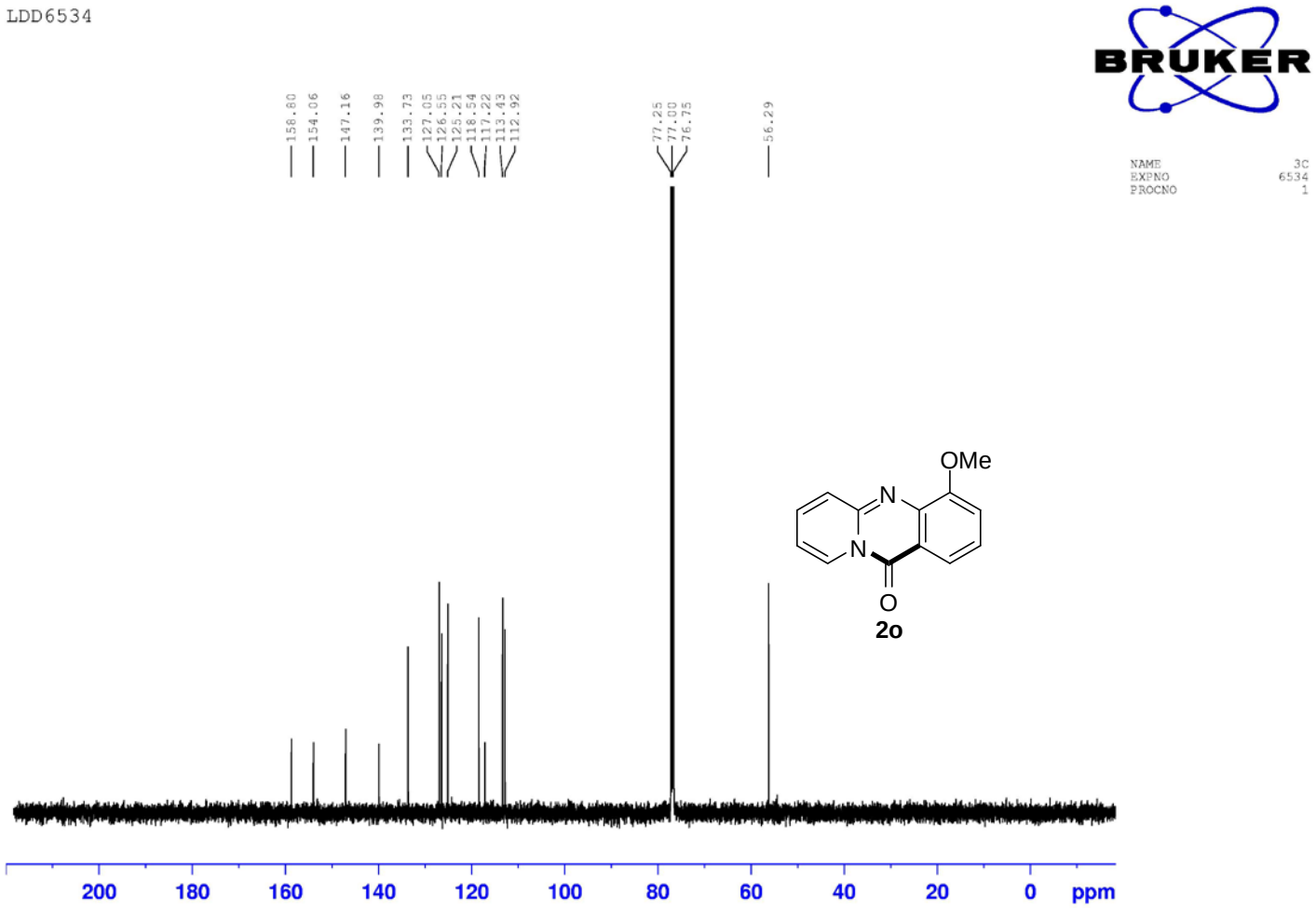
NAME	2C
EXPNO	6474
PROCNO	1
Date_	20130419
Time	22.57
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	296.7 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.66 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	125.7577931 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

6534

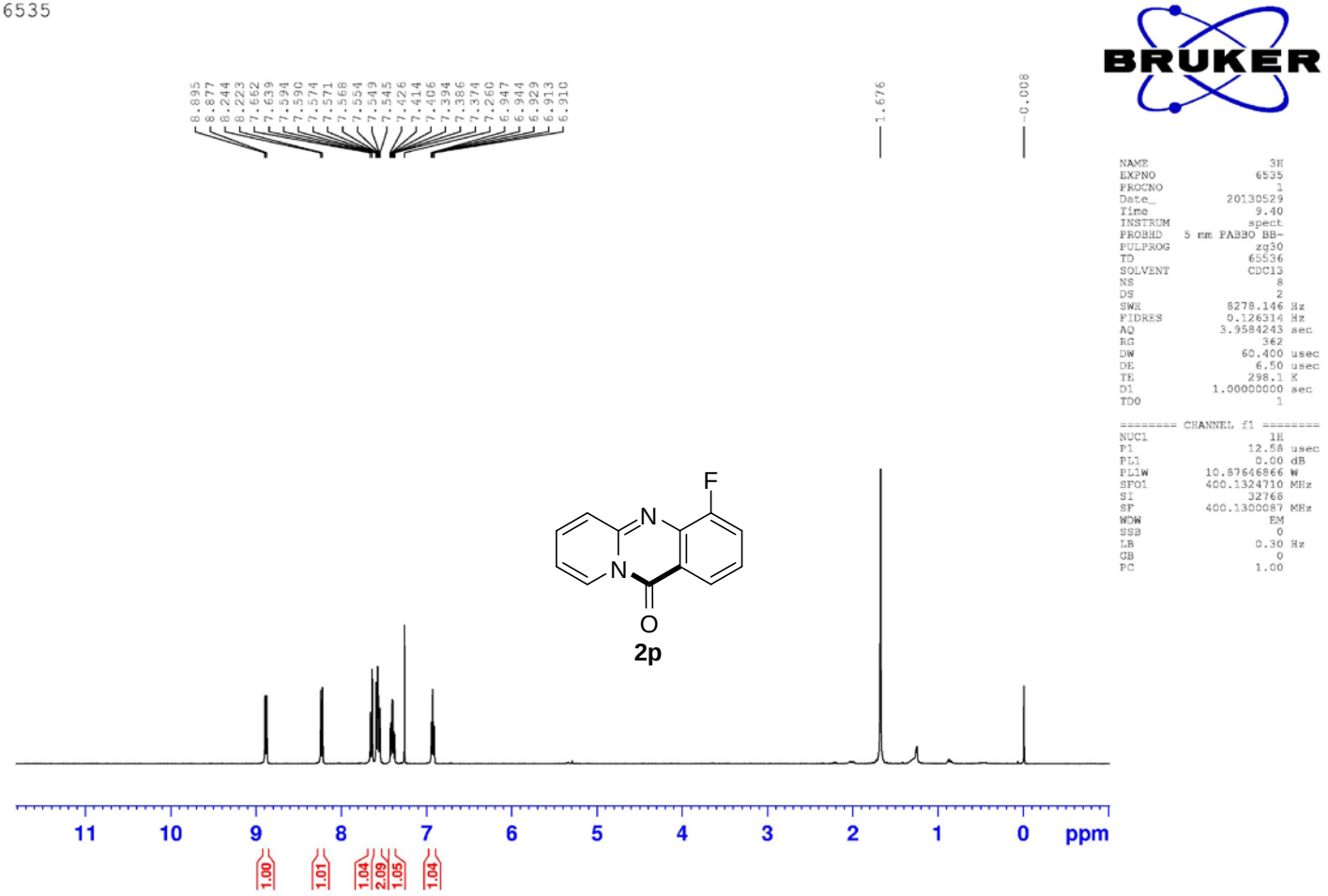


BRUKER

NAME	3H
EXPNO	6534
PROCNO	1
Date_	20130529
Time	9.34
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	322.5
DW	60.400 usec
DE	6.50 usec
TE	298.1 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300087 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

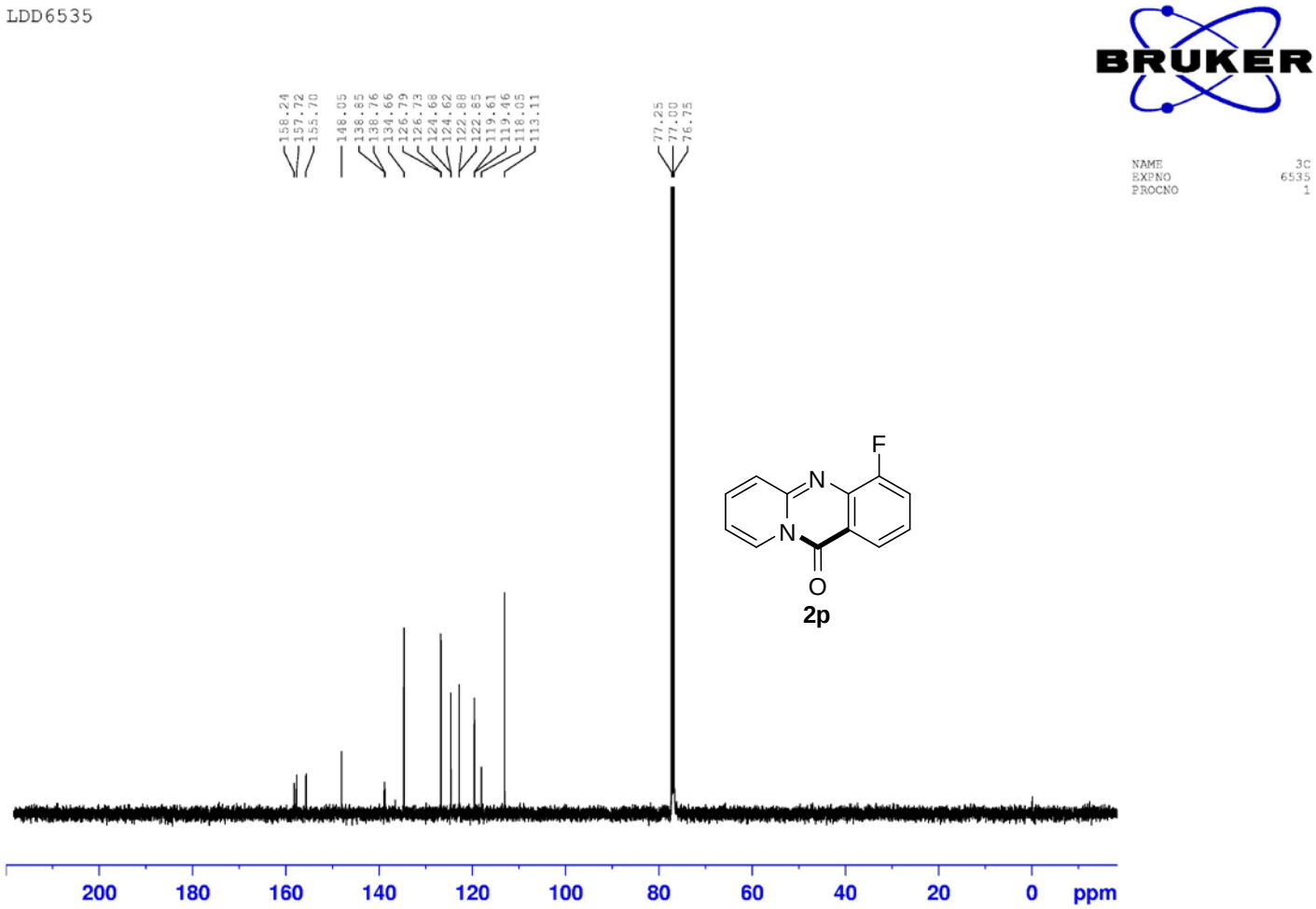

LDD6534
BRUKER
158.80
154.06
147.16
139.98
133.73
127.05
126.55
125.21
118.54
117.22
113.43
112.92
77.25
77.00
76.75
56.29
NAME 3C
EXPNO 6534
PROCNO 1
OMe
N
N
O
2o
200
180
160
140
120
100
80
60
40
20
0
ppm

6535

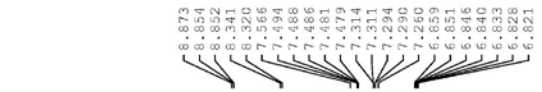


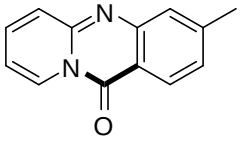
BRUKER

NAME	3H
EXPNO	6535
PROCNO	1
Date_	20130529
Time	9.40
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
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	298.1 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300087 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

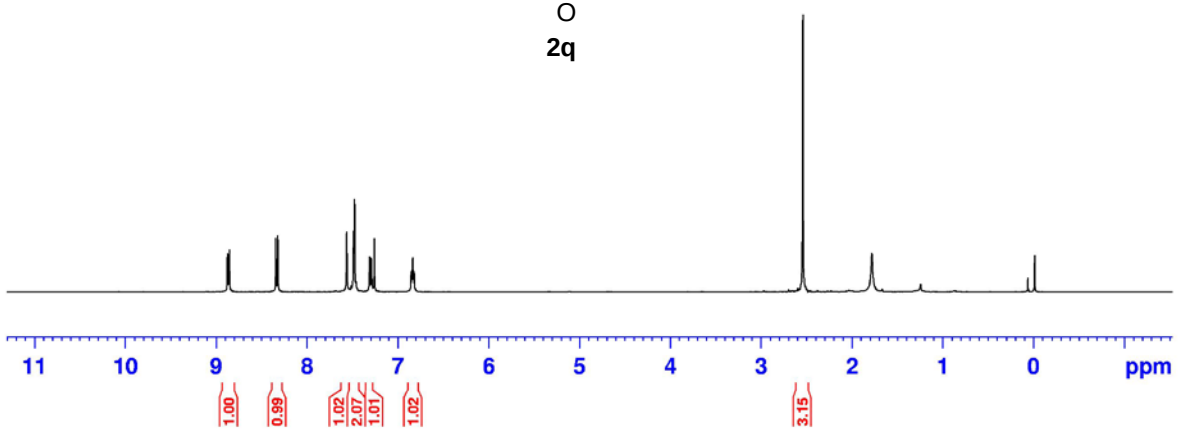

LDD6535
158.24
157.72
155.70
148.05
138.85
138.76
134.66
126.79
126.73
124.68
124.62
122.88
122.85
119.61
119.46
118.05
113.11
77.25
77.00
76.75
NAME 3C
EXPNO 6535
PROCNO 1
F
N
N
O
2p
200
180
160
140
120
100
80
60
40
20
0
ppm

6473



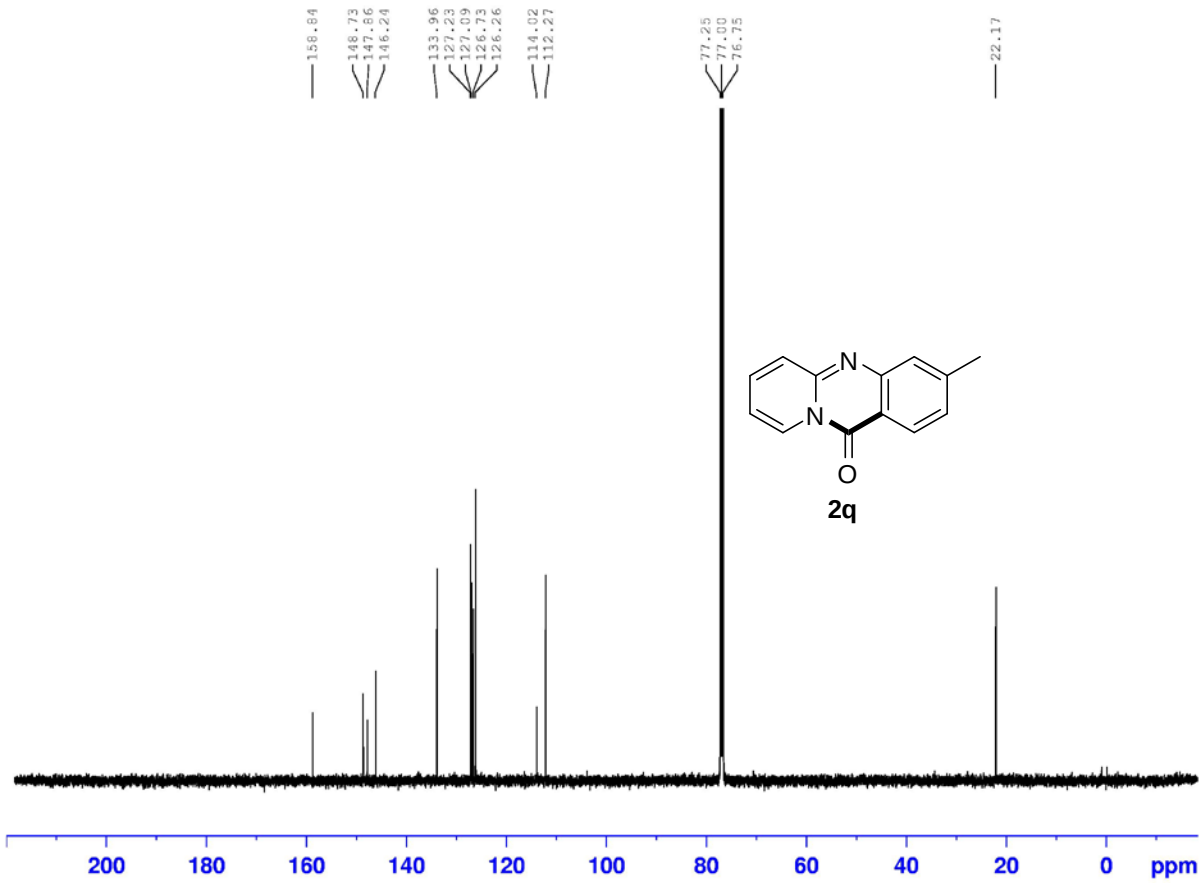


2q



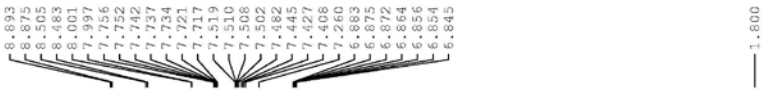
NAME	3H
EXPNO	6473
PROCNO	1
Date_	20130418
Time	20.50
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	322.5
DW	60.400 usec
DE	6.50 usec
TE	297.7 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300091 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

LDD6473



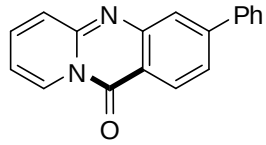
NAME	3C
EXPNO	6473
PROCNO	1
Date_	20130419
Time	22.44
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	296.9 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	11.66 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	125.7577939 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

6514

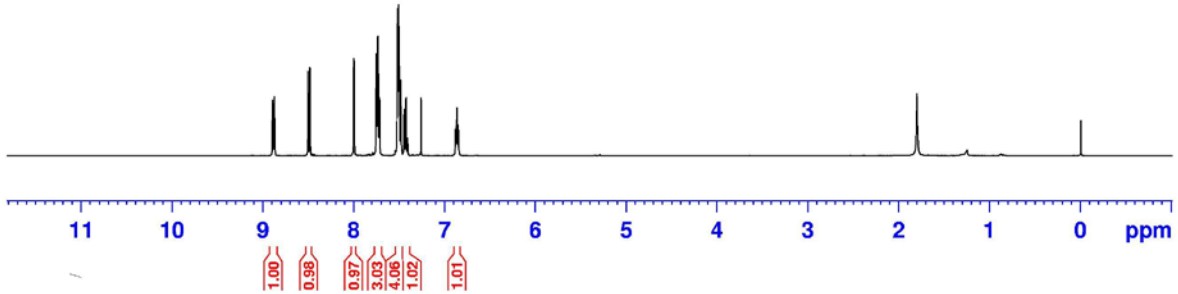


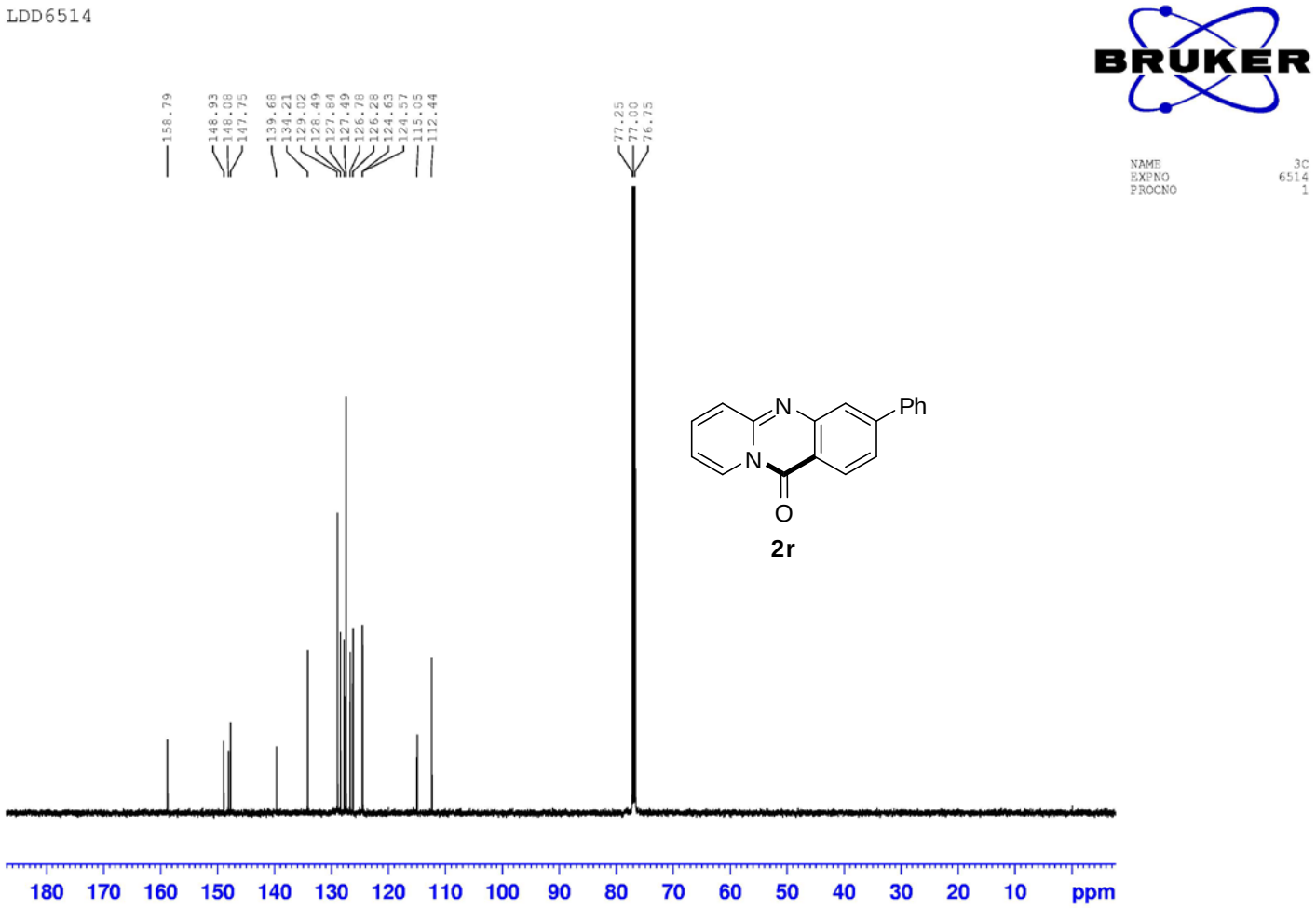
BRUKER

NAME	3H
EXPNO	6514
PROCNO	1
Date_	20130515
Time	10.15
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	256
DW	60.400 usec
DE	6.50 usec
TE	297.9 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300090 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

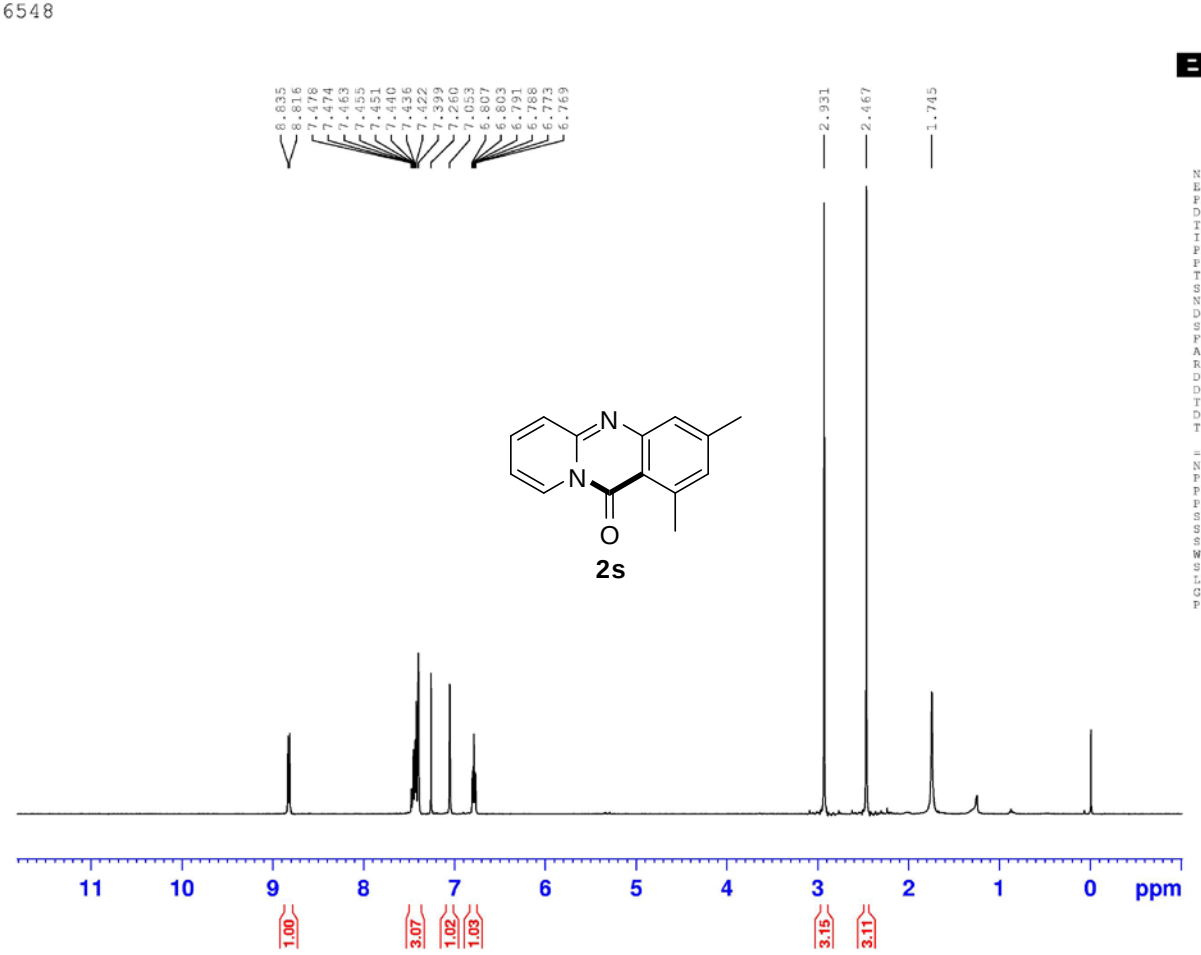


2r

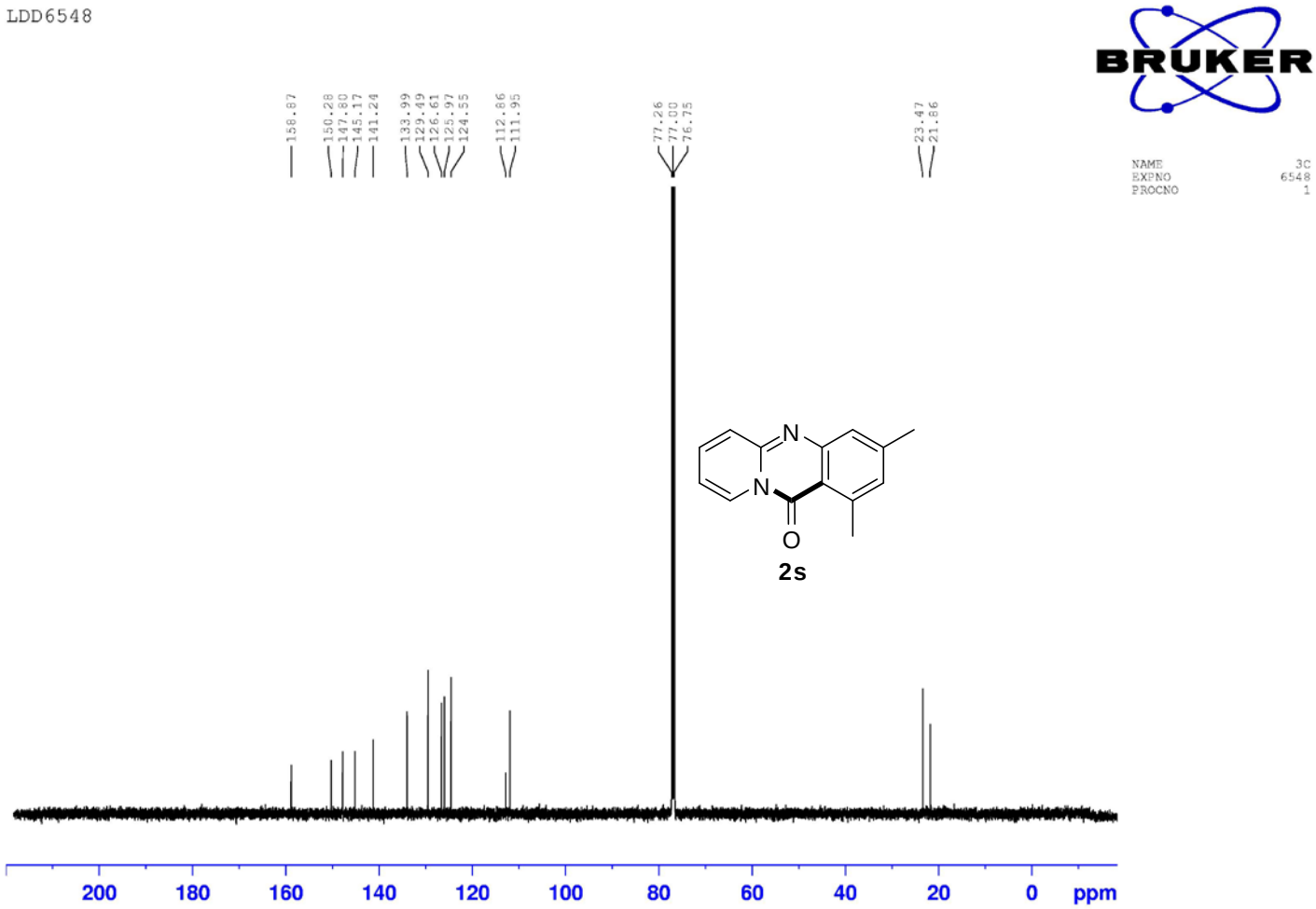



LDD6514
158.79
148.93
146.08
147.75
139.68
134.21
129.02
126.49
127.84
127.49
126.78
126.28
124.63
124.57
115.05
112.44
77.25
77.00
76.75
NAME 3C
EXPNO 6514
PROCNO 1
N
N
O
Ph
2r
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

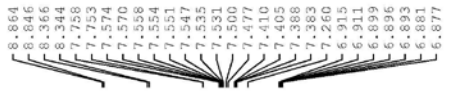
6548

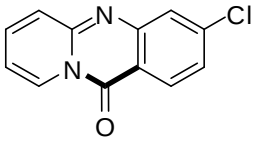


NAME	3H
EXPNO	6548
PROCNO	1
Date_	20130605
Time	20.47
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	362
DW	60.400 usec
DE	6.50 usec
TE	298.4 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300088 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00


LDD6548
BRUKER
158.87
150.28
147.80
145.17
141.24
133.99
129.49
126.61
125.97
124.55
112.86
111.95
77.26
77.00
76.75
23.47
21.86
NAME 3C
EXPNO 6548
PROCNO 1
N
N
O
2s
200
180
160
140
120
100
80
60
40
20
0
ppm

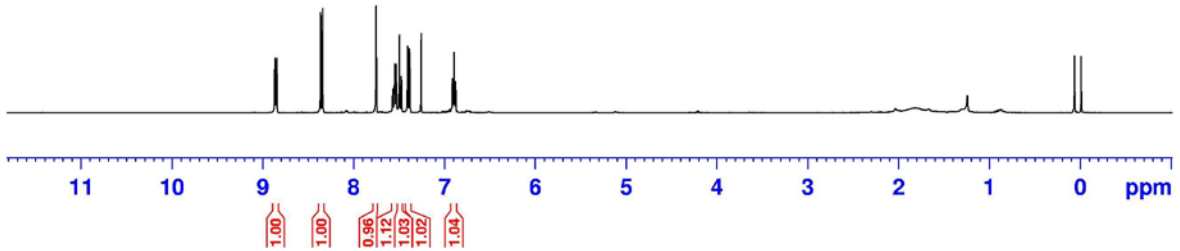
6531-1

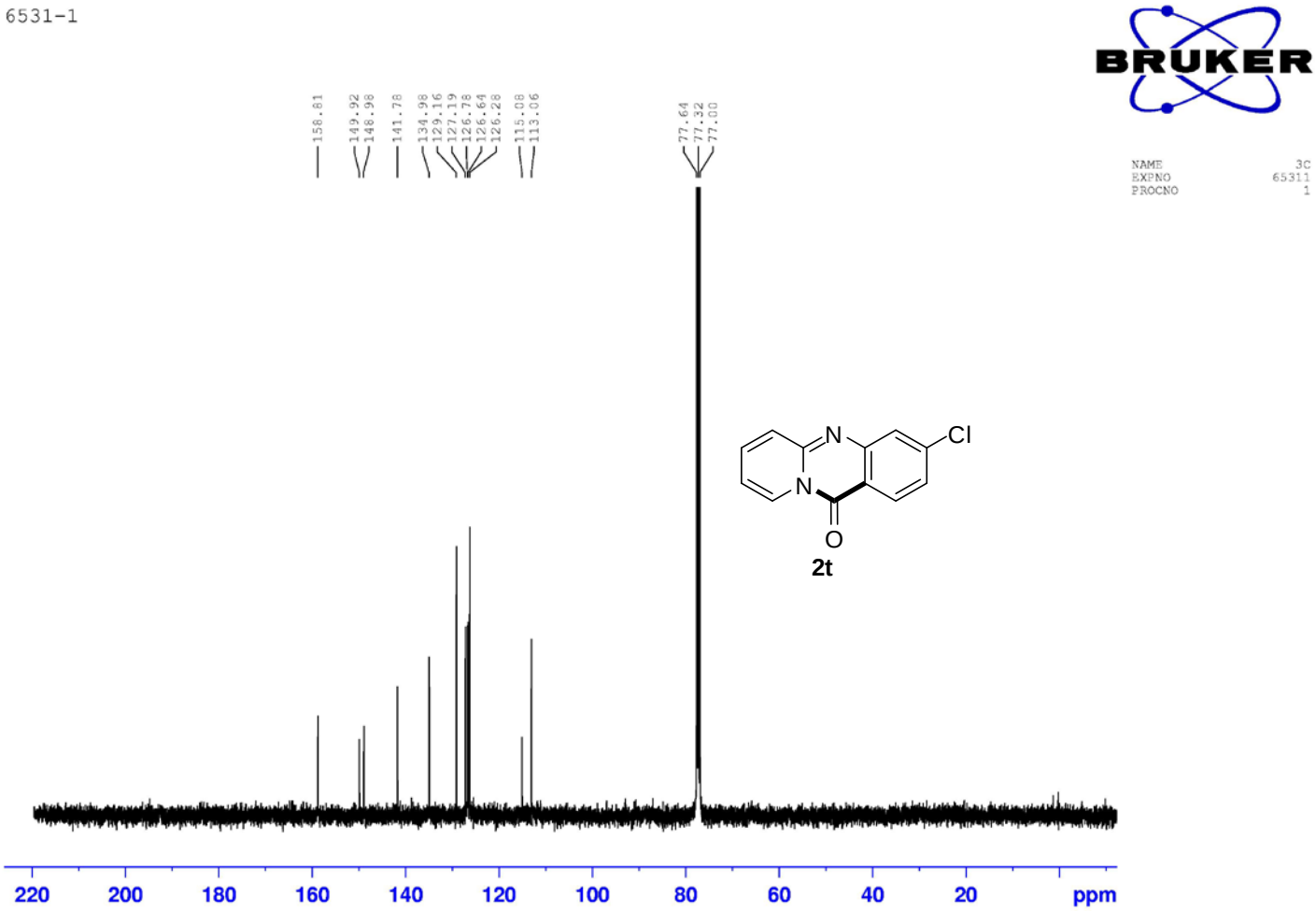




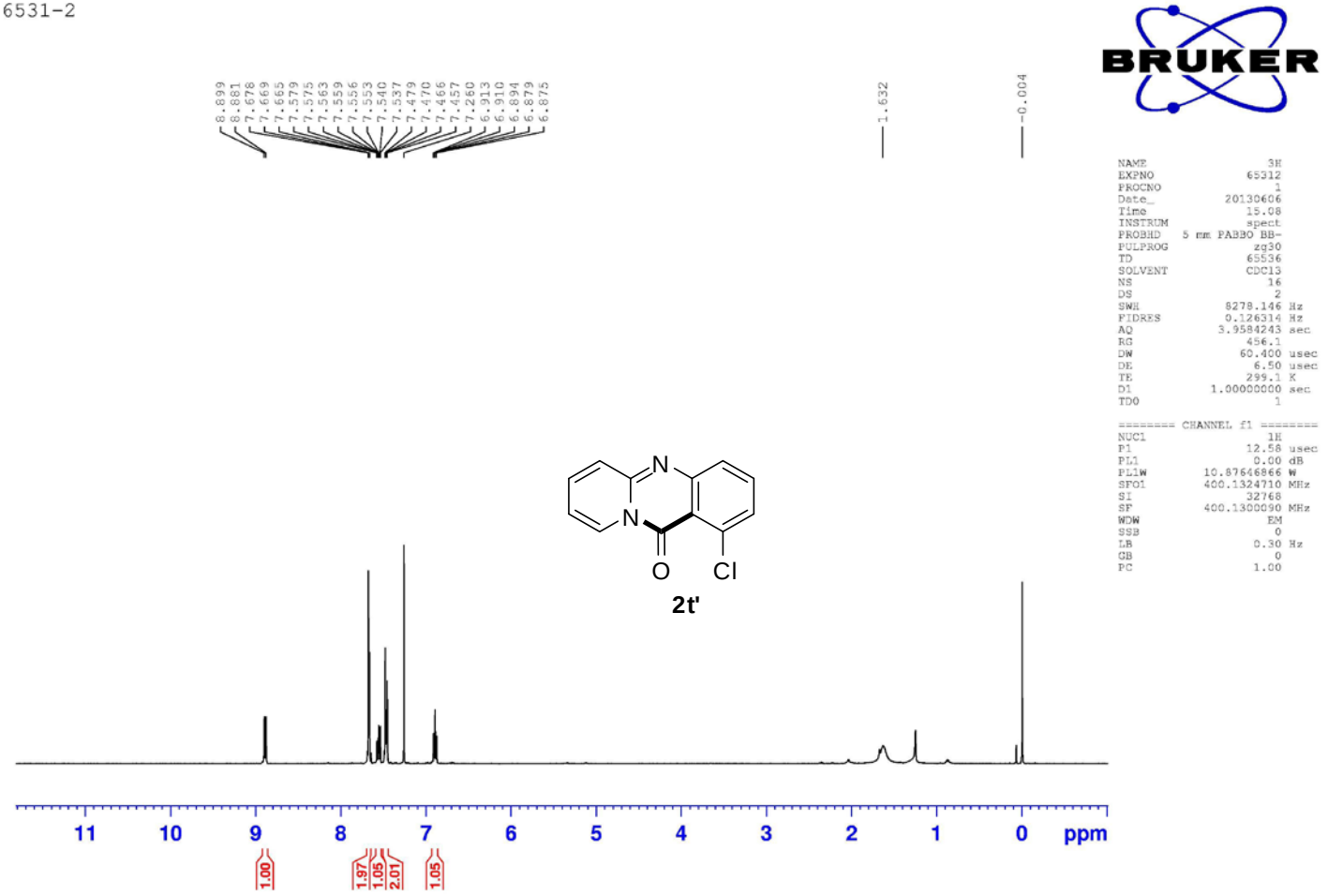
2t

NAME	3H
EXPNO	65311
PROCNO	1
Date_	20130609
Time	1.16
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	322.5
DW	60.400 usec
DE	6.50 usec
TE	299.7 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300091 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



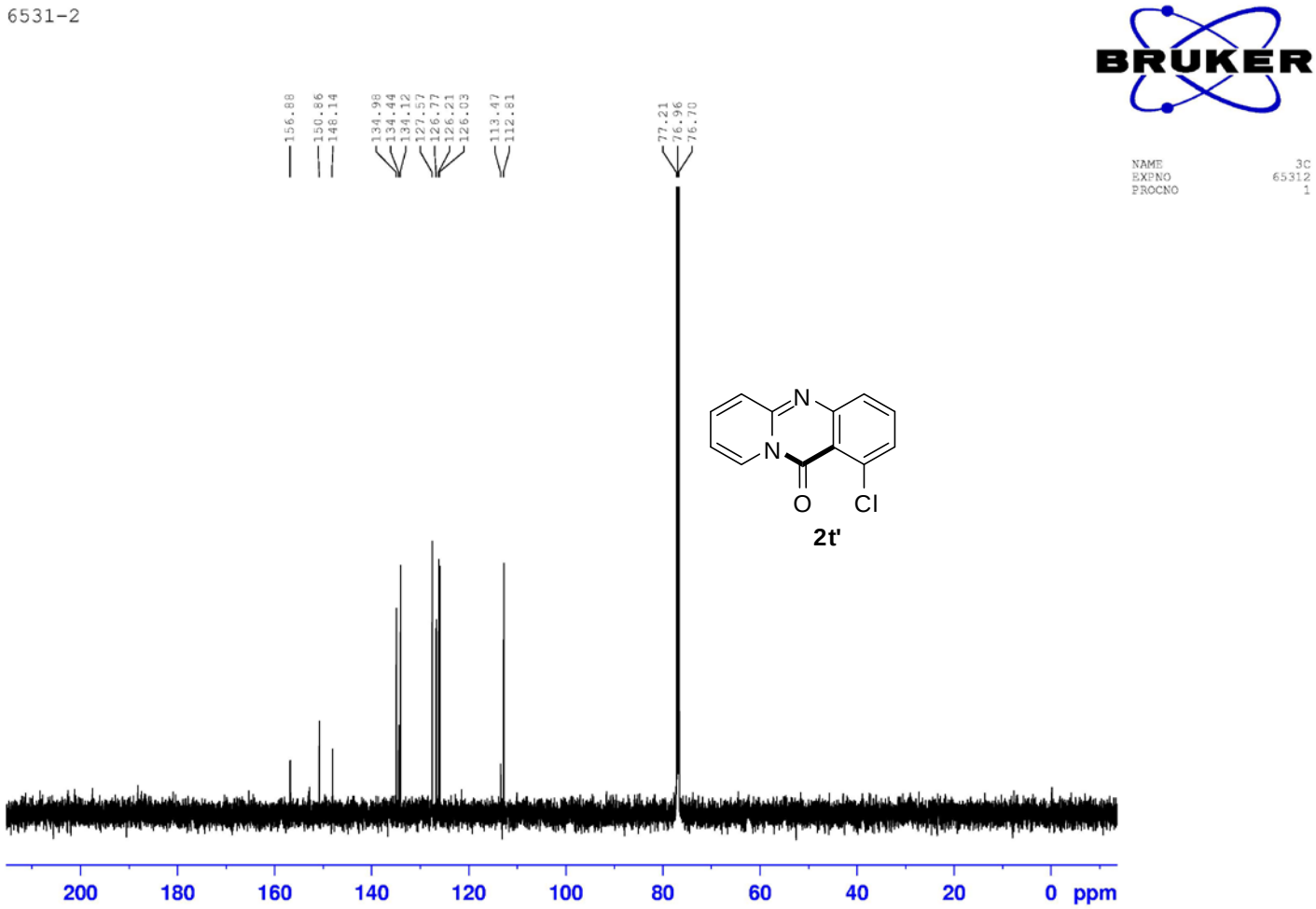

6531-1
156.81
149.92
148.98
141.78
134.98
129.16
127.19
126.78
126.64
126.28
115.08
113.06
77.64
77.32
77.00
BRUKER
NAME 3C
EXPNO 6531
PROCNO 1
N
N
Cl
O
2t
220
200
180
160
140
120
100
80
60
40
20
ppm

6531-2

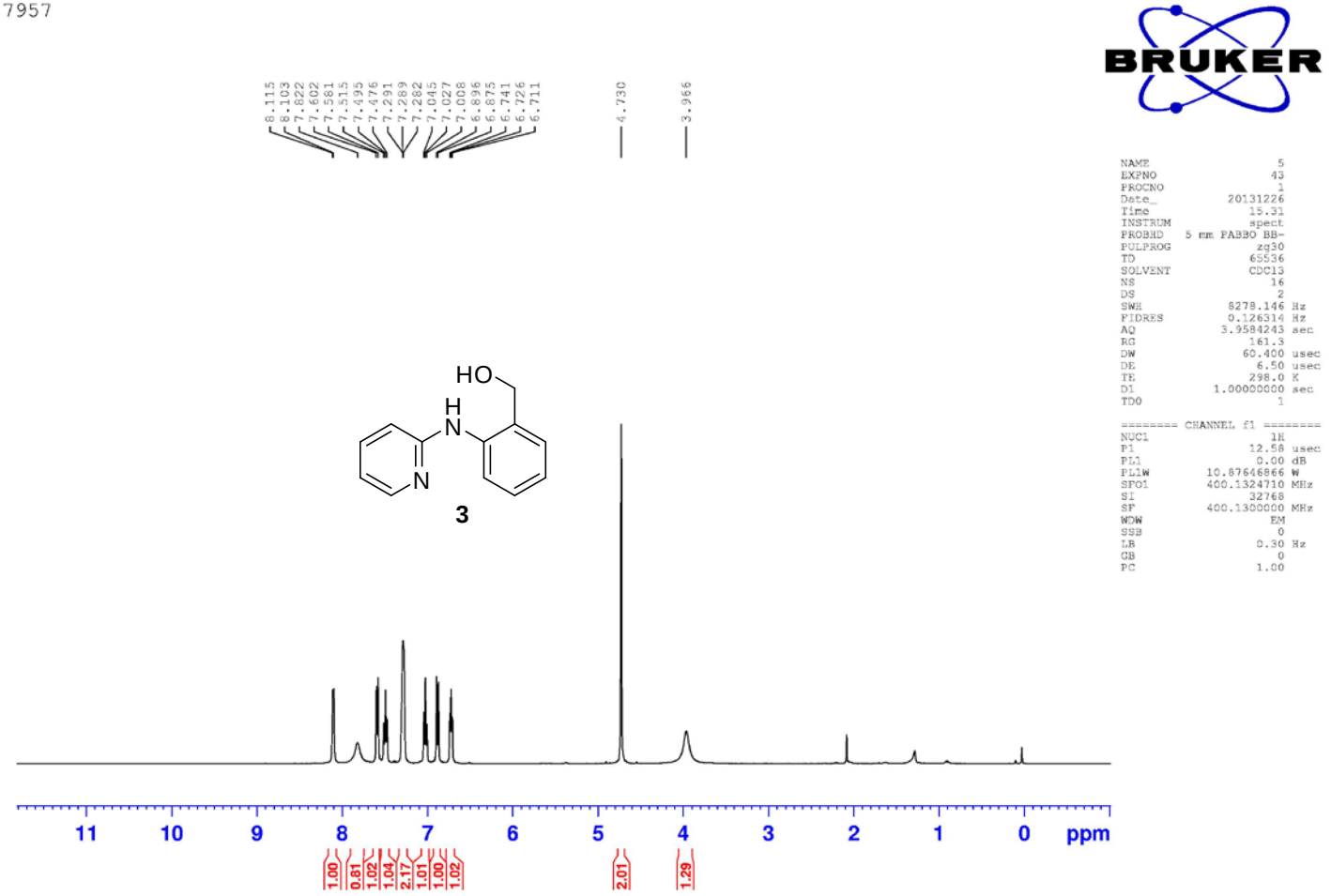


BRUKER

NAME	3H
EXPNO	65312
PROCNO	1
Date_	20130606
Time	15.08
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	456.1
DW	60.400 usec
DE	6.50 usec
TE	299.1 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300090 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

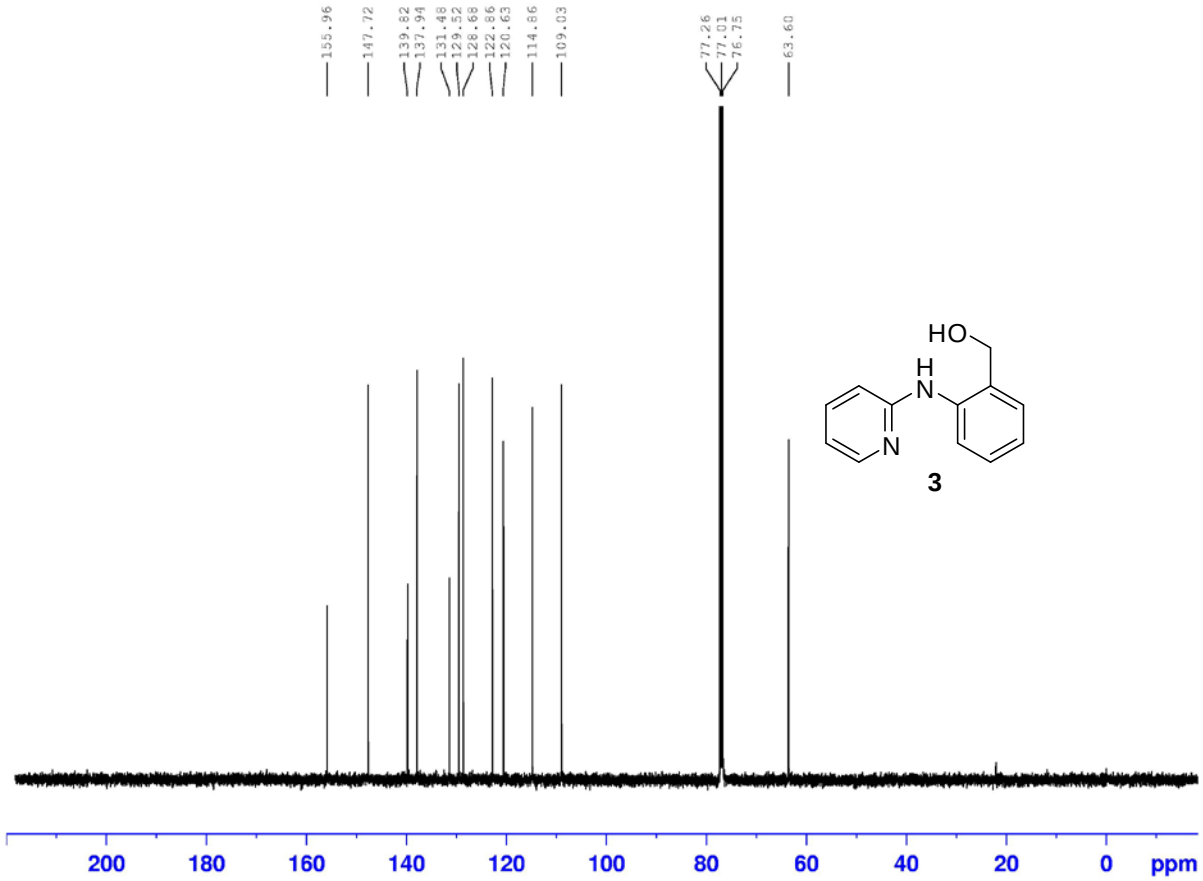

6531-2
156.88
150.86
148.14
134.98
134.44
134.12
127.57
126.77
126.21
126.03
113.47
112.81
77.21
76.96
76.70
NAME 3C
EXPNO 65312
PROCNO 1
N
N
O
Cl
2t'
200
180
160
140
120
100
80
60
40
20
0 ppm

7957



NAME	5
EXPNO	43
PROCNO	1
Date_	20131226
Time	15.31
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	161.3
DW	60.400 usec
DE	6.50 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

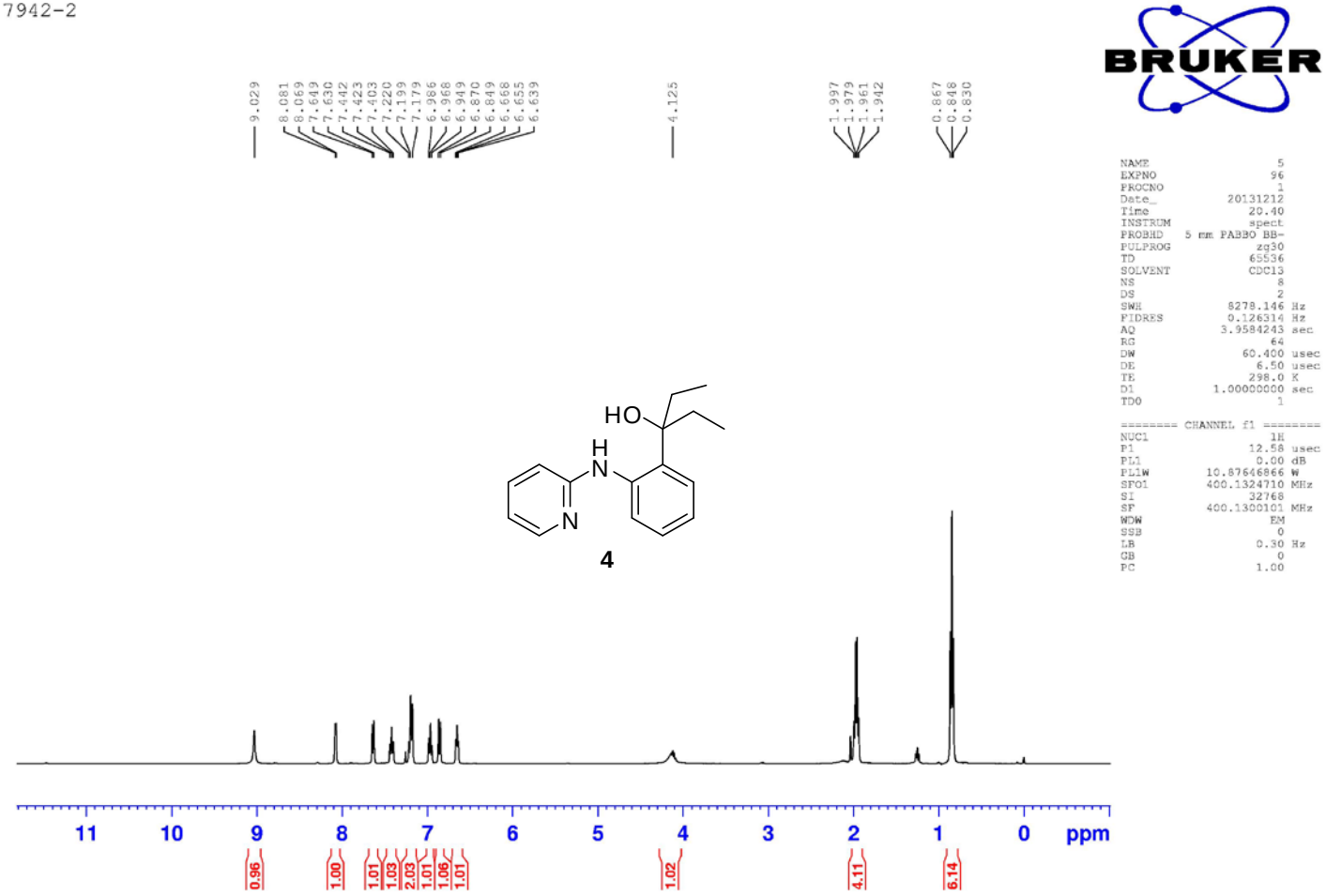
7957



BRUKER

NAME	5
EXPNO	7957
PROCNO	1
Date_	20131230
Time	20.40
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	589
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	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	13.84 usec
PL1	2.50 dB
PL1W	46.89624786 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	125.7577966 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

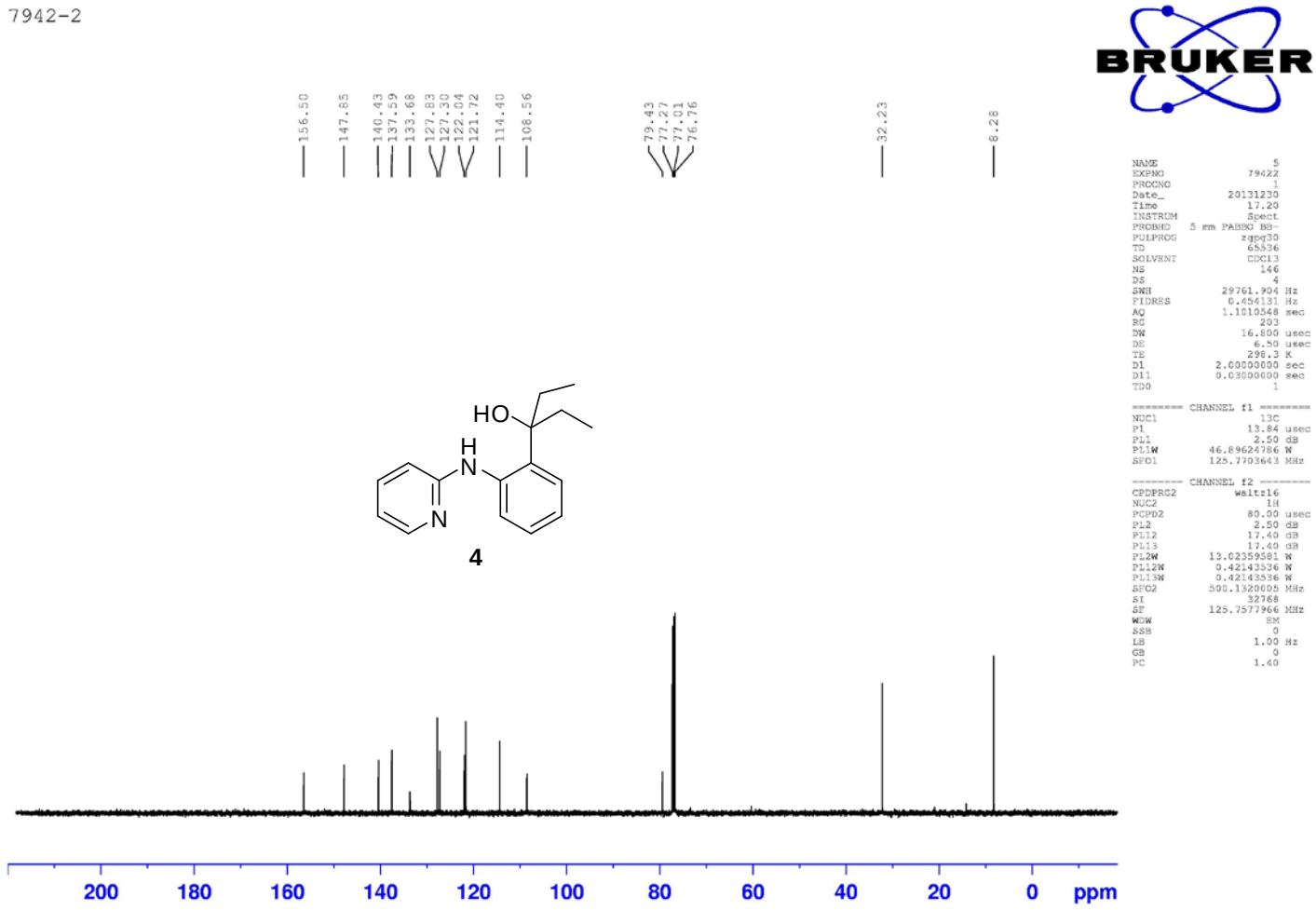
7942-2



BRUKER

NAME	5
EXPNO	96
PROCNO	1
Date_	20131212
Time	20.40
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	64
DW	60.400 usec
DE	6.50 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300101 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

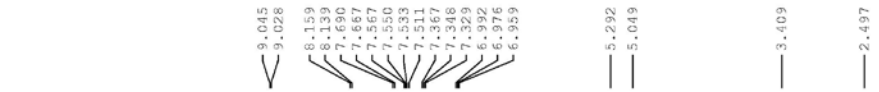
7942-2

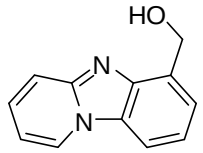


BRUKER

NAME	5
EXPNO	79422
PROCNO	1
Date_	20131230
Time	17.20
INSTRUM	Spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	146
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	298.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	13.84 usec
PL1	2.50 dB
PL1W	46.89624786 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	125.7577966 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

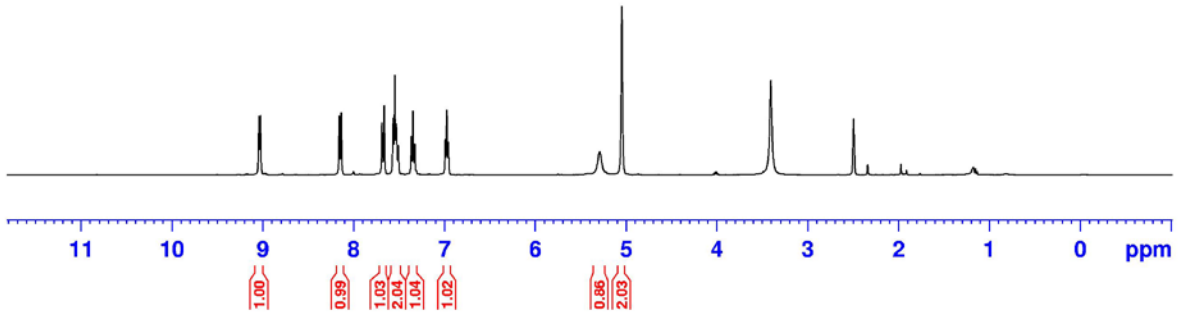
7956



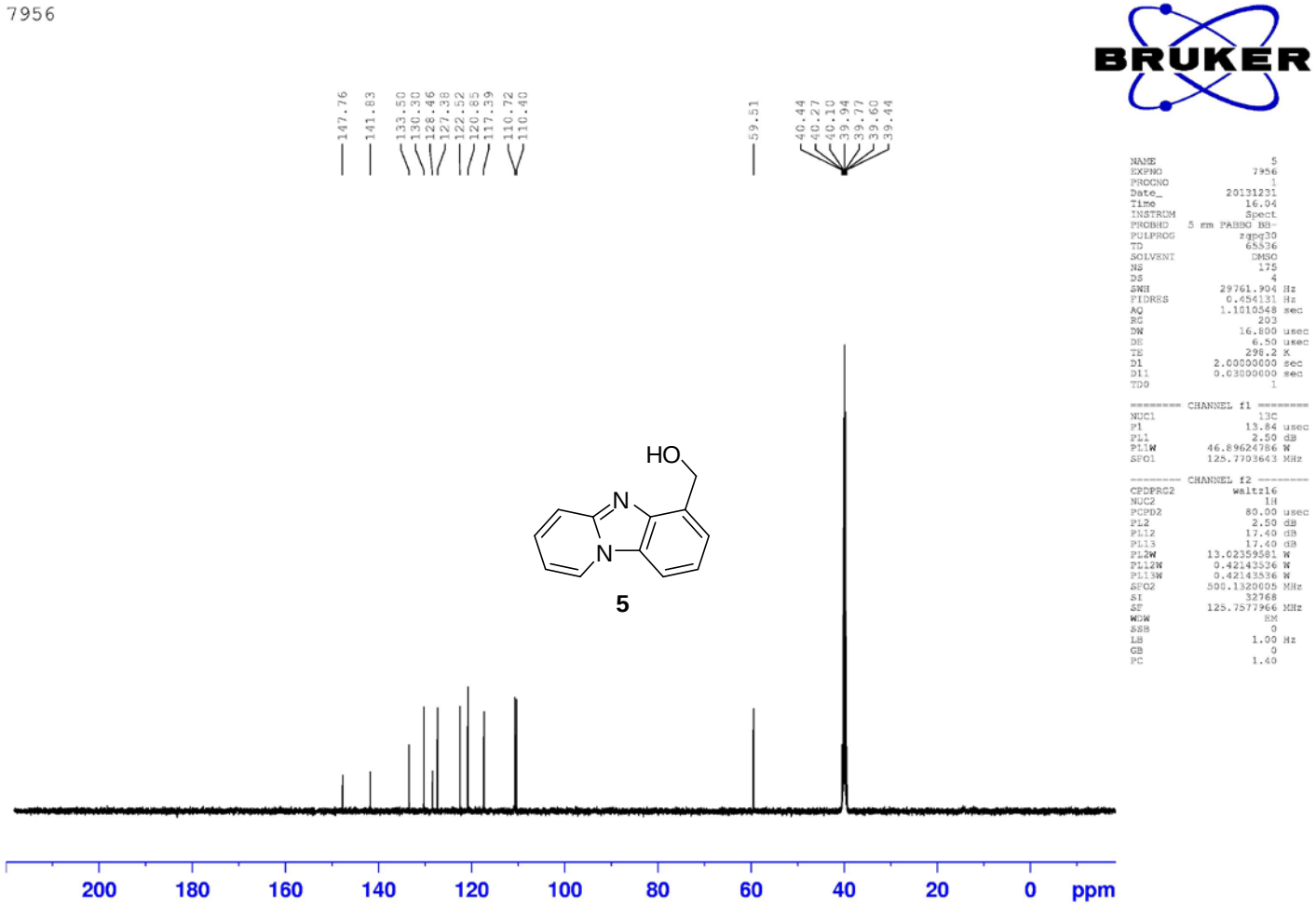


5

NAME	5	
EXPNO	42	
PROCNO	1	
Date_	20131226	
Time	15.23	
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	90.5	
DW	60.400	usec
DE	6.50	usec
TE	298.0	K
D1	1.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	1H	
P1	12.58	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	



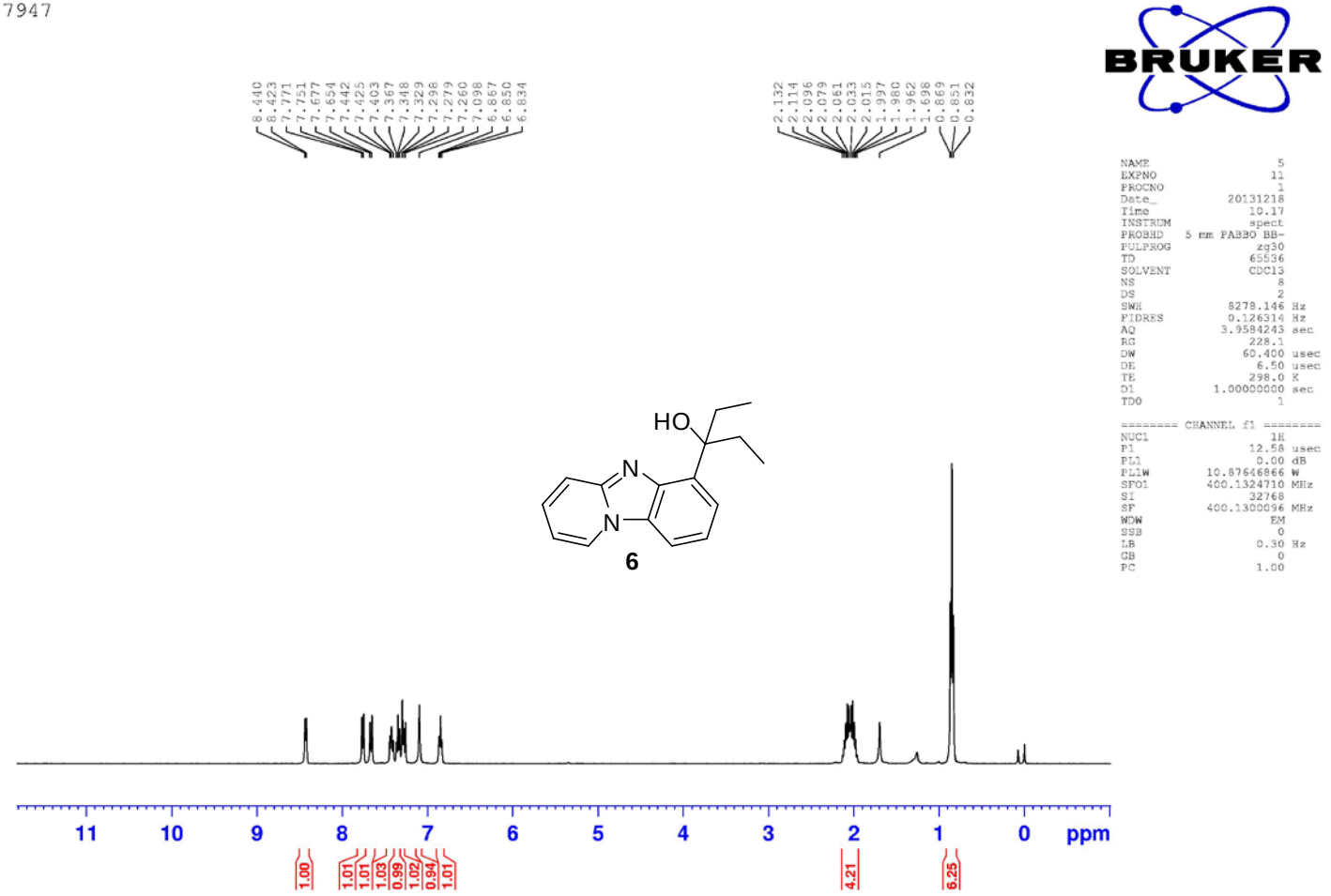
7956

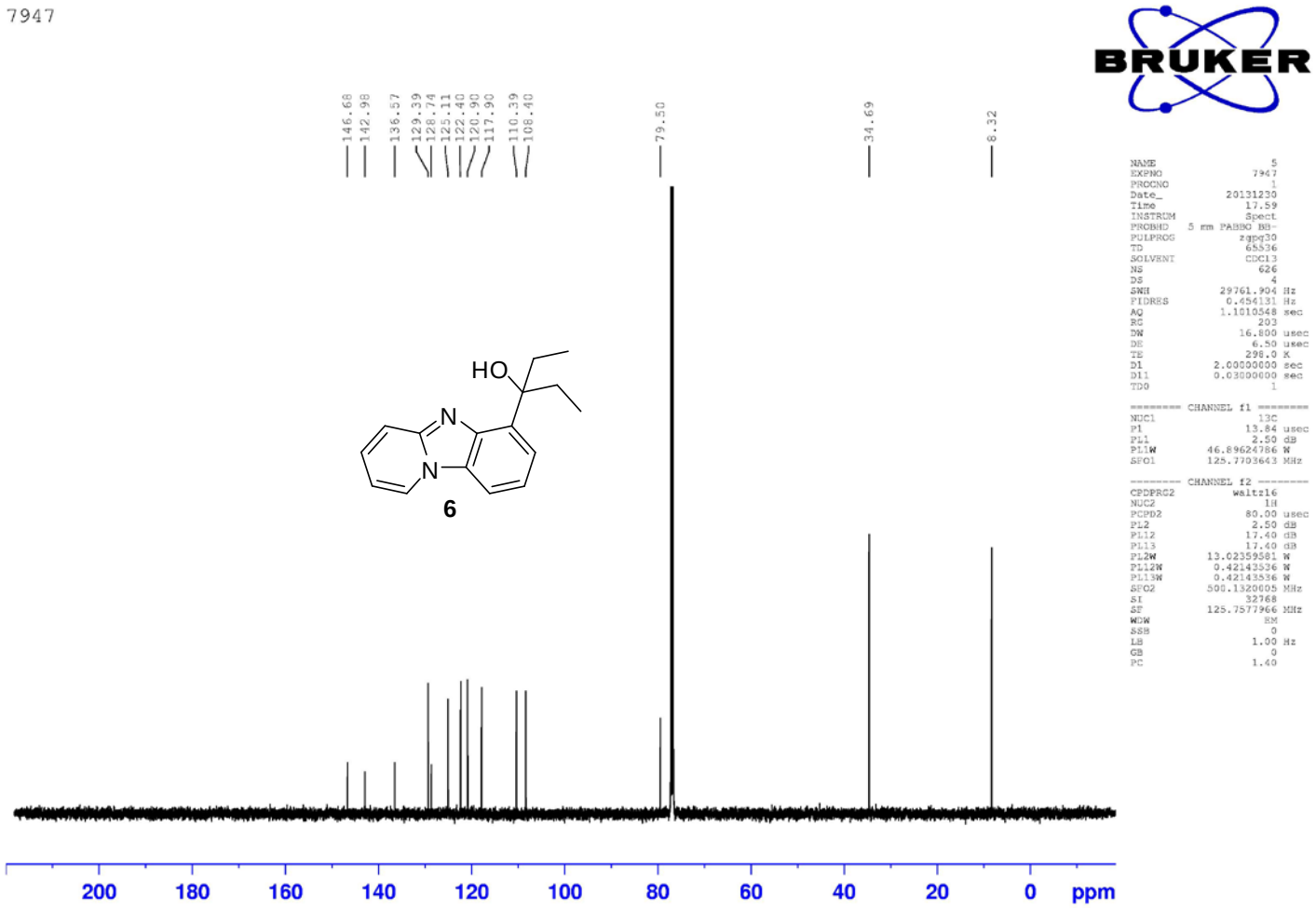


BRUKER

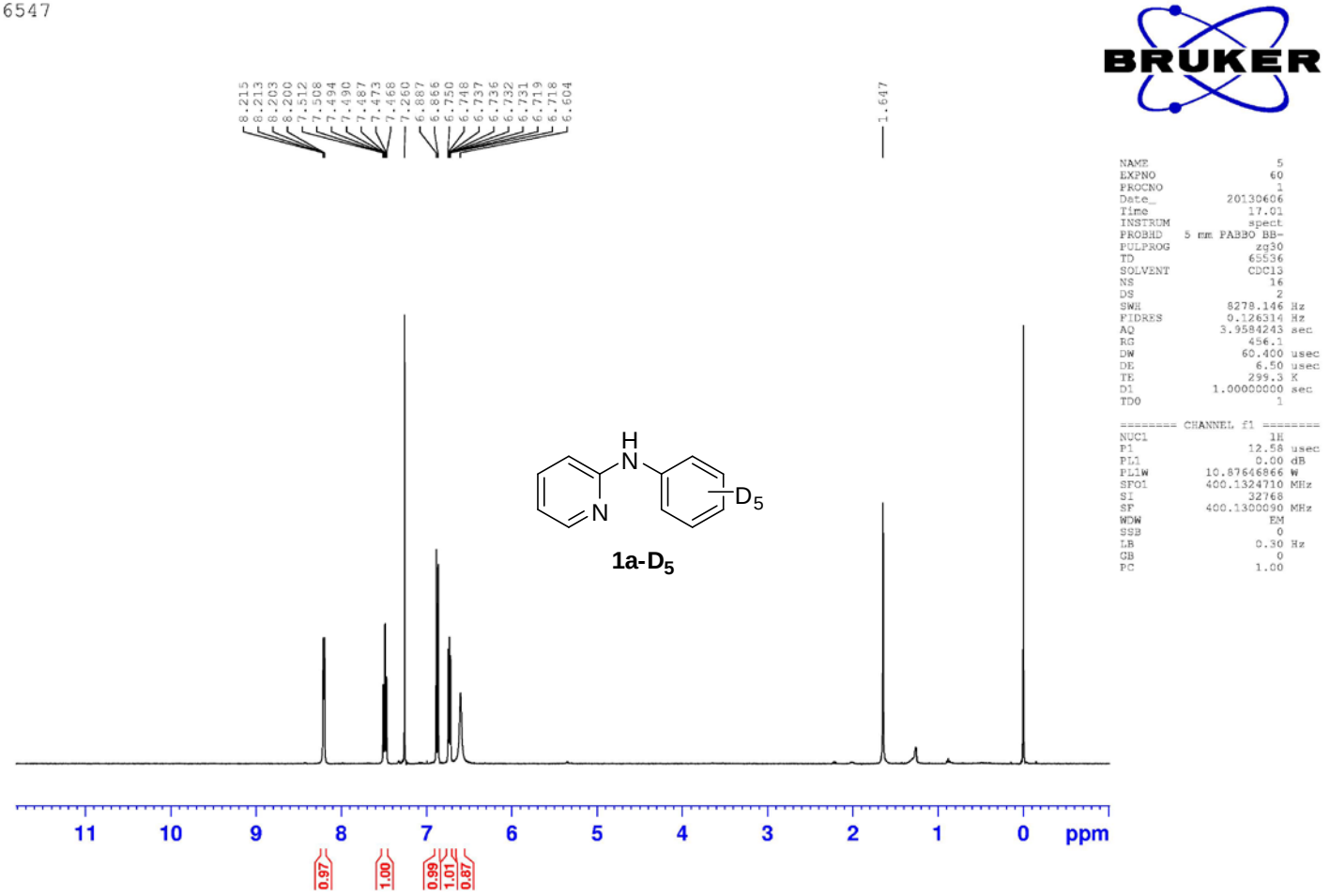
NAME	5
EXPNO	7956
PROCNO	1
Date_	20131231
Time	16.04
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	DMSO
NS	175
DS	4
SWH	29761.904 Hz
FIDRES	0.454131 Hz
AQ	1.1010548 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	298.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	13.84 usec
PL1	2.50 dB
PL1W	46.89624786 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	125.7577966 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

7947


8.440
8.423
7.771
7.751
7.677
7.654
7.442
7.425
7.403
7.367
7.348
7.329
7.298
7.279
7.260
7.098
6.867
6.850
6.834
2.132
2.114
2.096
2.079
2.061
2.033
2.015
1.997
1.980
1.962
1.698
0.869
0.851
0.832
HO
N
N
6
NAME 5
EXPNO 11
PROCNO 1
Date_ 20131218
Time 10.17
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 228.1
DW 60.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300096 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
1.00
1.01
1.01
1.03
0.99
1.02
0.94
1.01
4.21
6.25


7947
146.66
142.98
136.57
129.39
128.74
125.11
122.40
120.90
117.90
110.39
108.40
79.50
34.69
8.32
HO
N
N
6
BRUKER
NAME 5
EXPNO 7947
PROCNO 1
Date_ 20131230
Time 17.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 626
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 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 13.84 usec
PL1 2.50 dB
PL1W 46.89624786 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 125.7577966 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

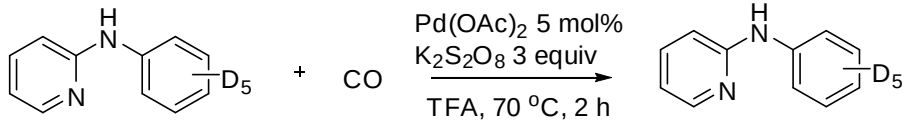
6547



BRUKER

NAME	5
EXPNO	60
PROCNO	1
Date_	20130606
Time	17.01
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	456.1
DW	60.400 usec
DE	6.50 usec
TE	299.3 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300090 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

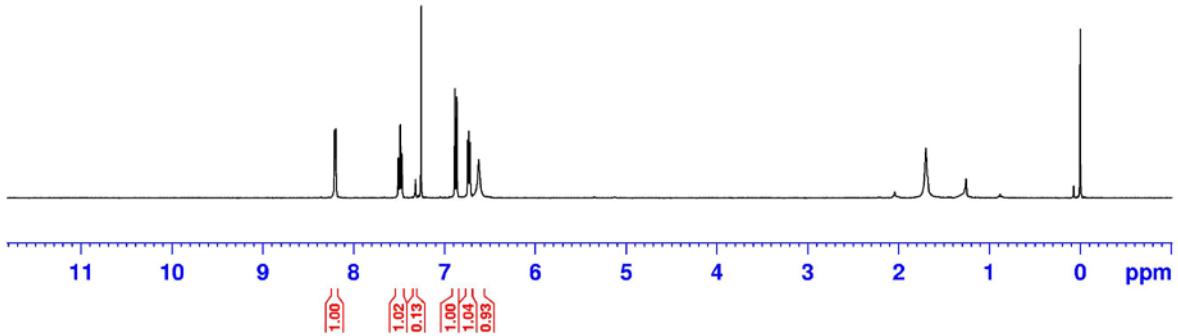
12

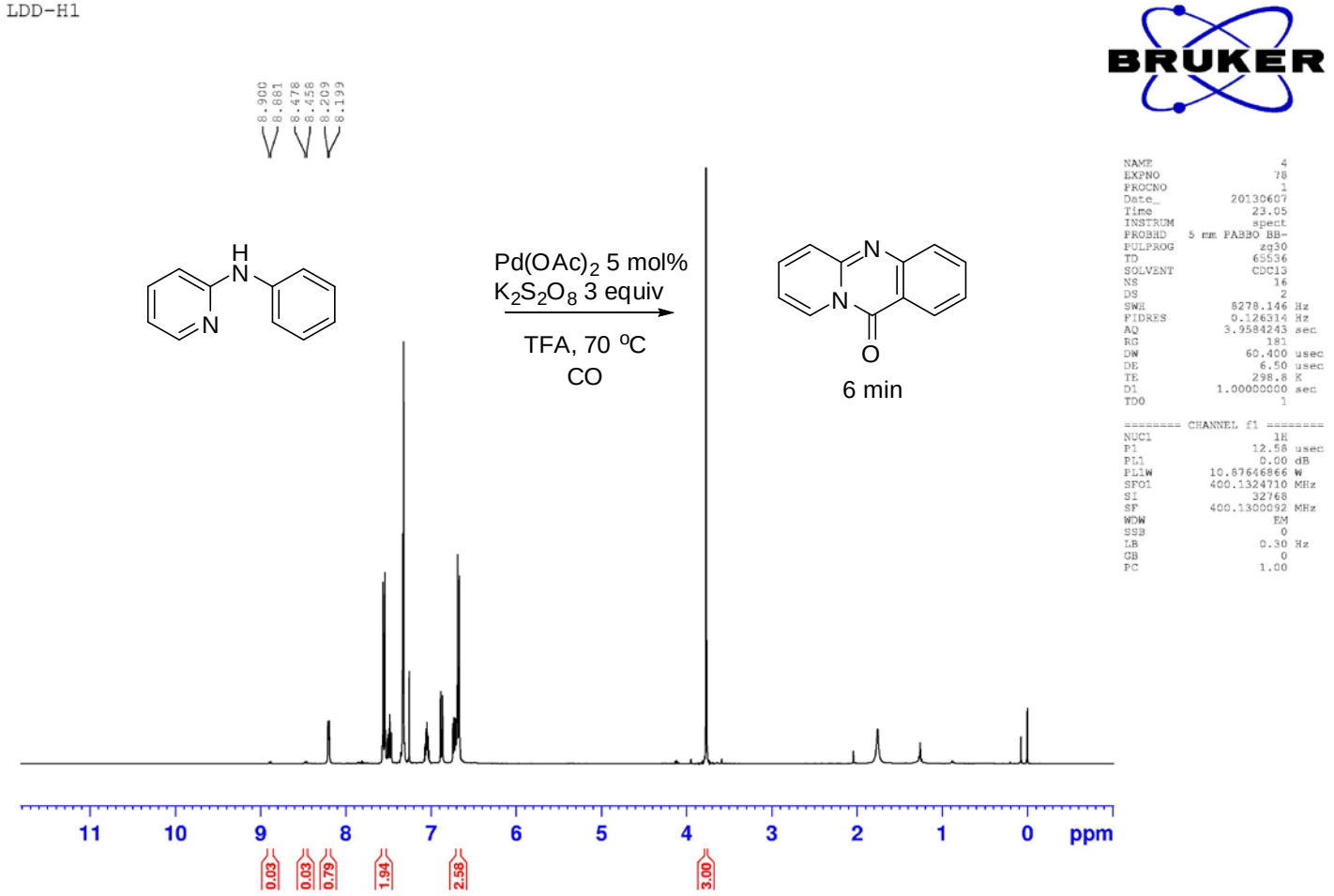


1a-D_5
D > 95%

1a-D_5
recover: 15%
D = 87%

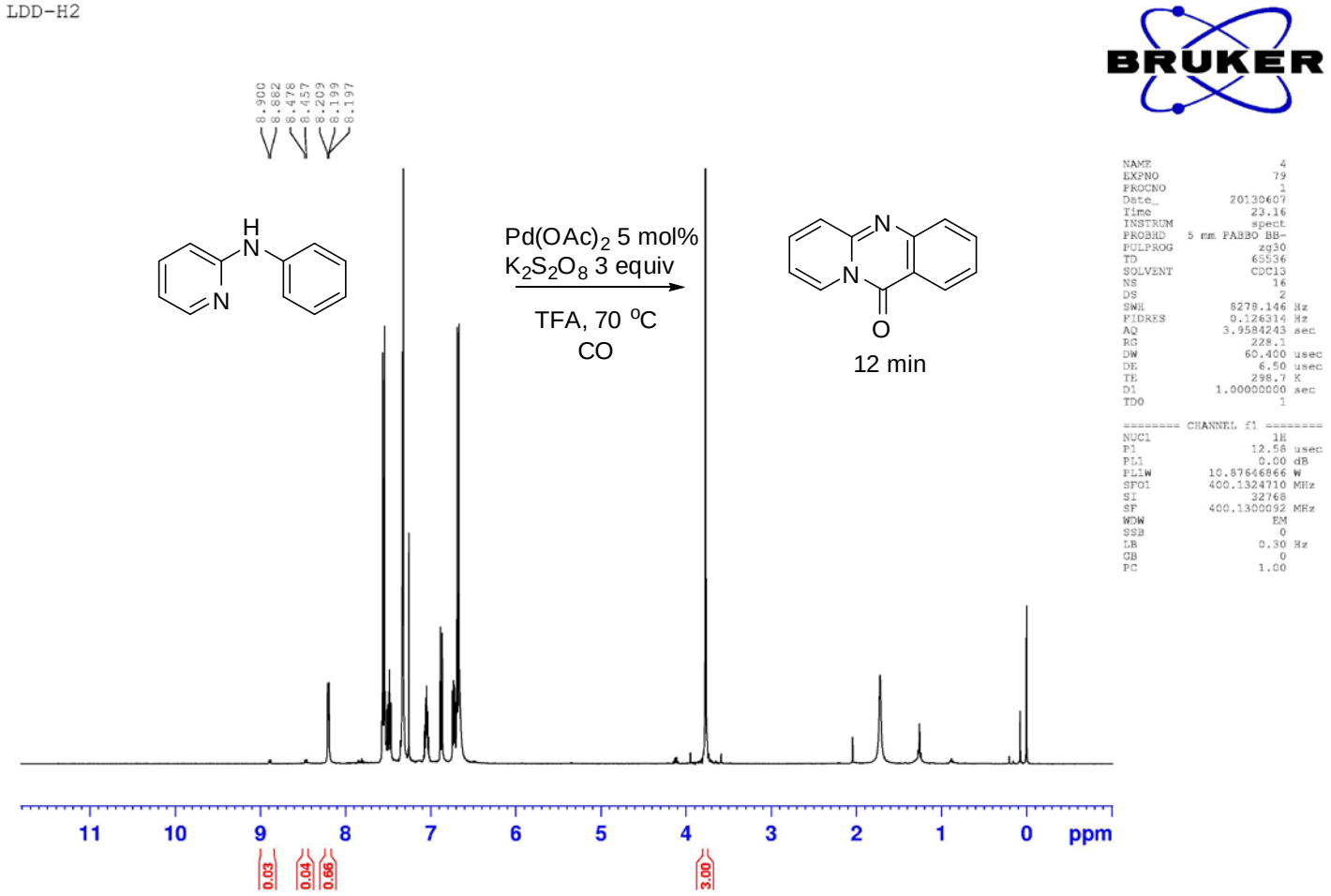
```
NAME                  5
EXPNO                53
PROCNO                1
Date_          20130608
Time              16.59
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            8278.146 Hz
FIDRES         0.126314 Hz
AQ             3.9584243 sec
RG                456.1
DW                60.400 usec
DE                 6.50 usec
TE                300.2 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                12.58 usec
PL1                0.00 dB
PL1W        10.87646866 W
SFO1        400.1324710 MHz
SI                32768
SF          400.1300088 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

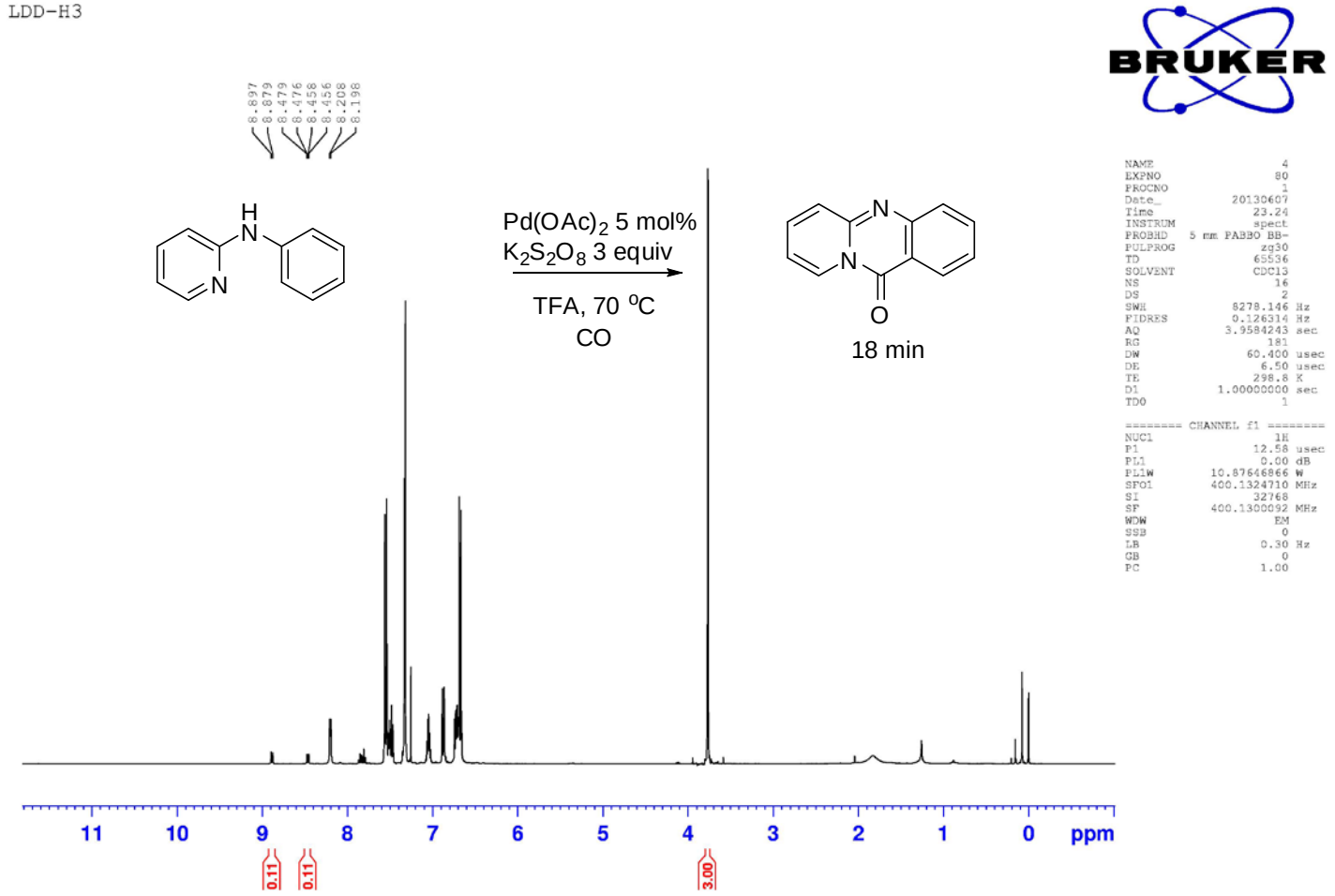




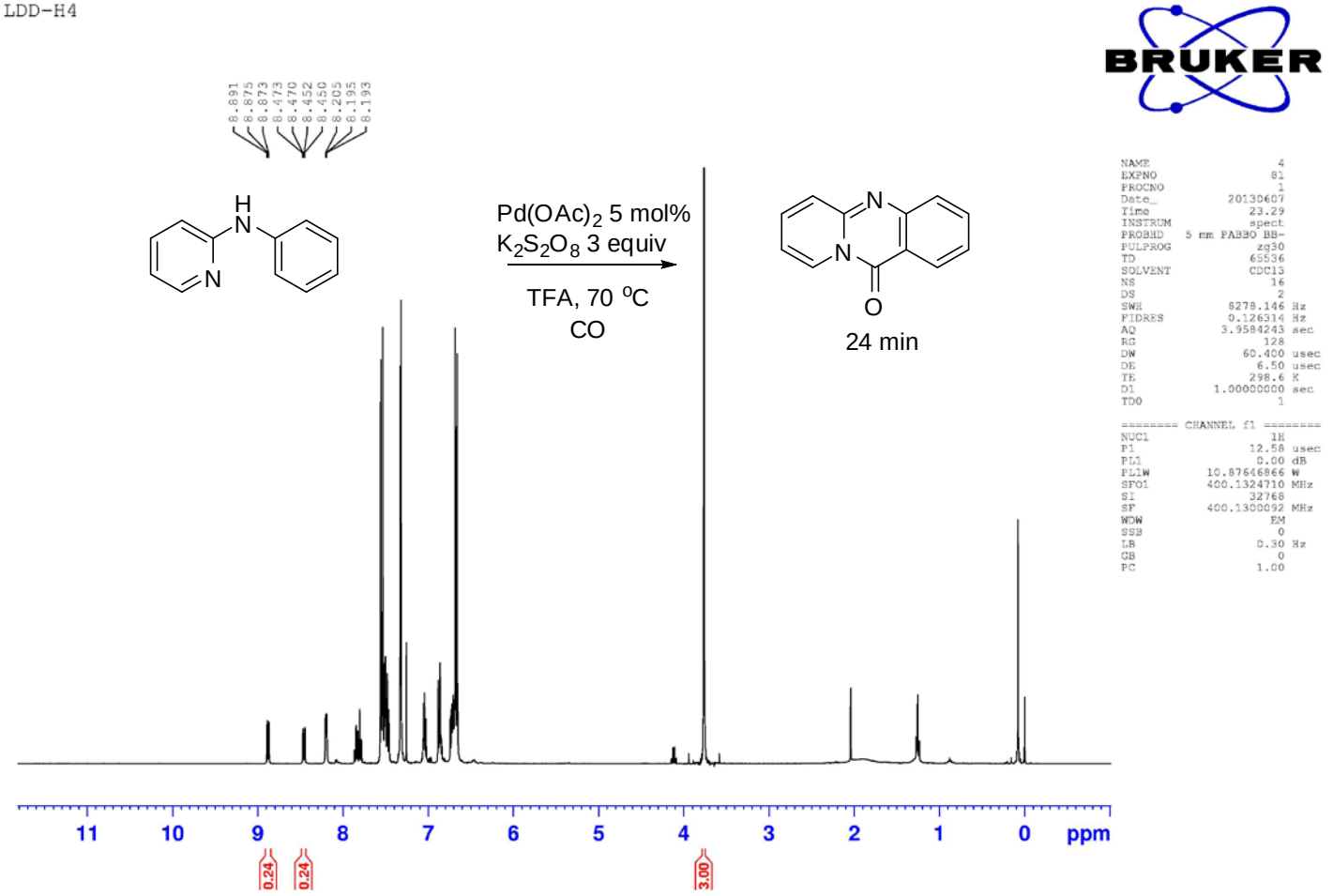
BRUKER

NAME	4
EXPNO	78
PROCNO	1
Date_	20130607
Time	23.05
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	181
DW	60.400 usec
DE	6.50 usec
TE	298.8 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300092 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

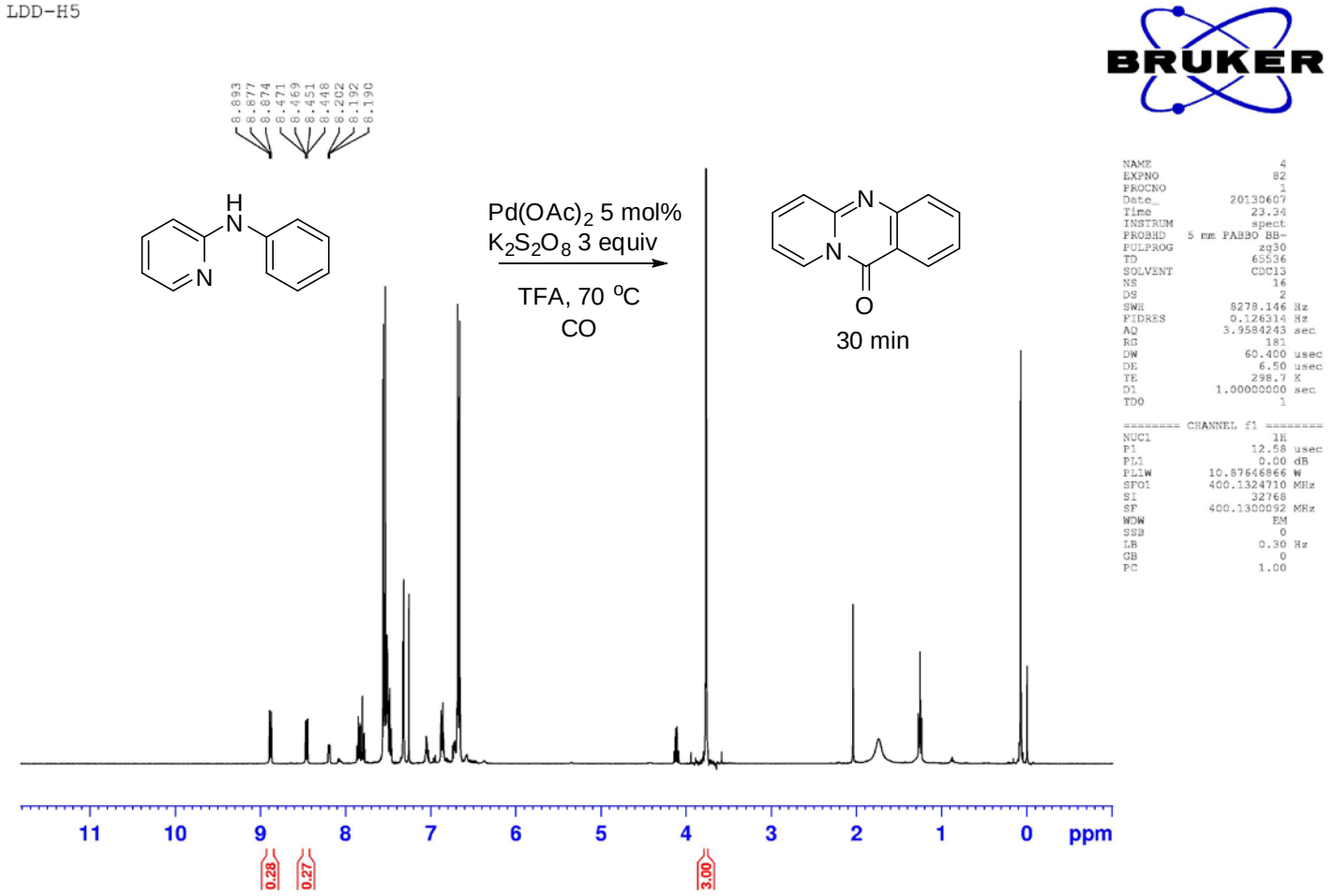

LDD-H2
8.900
8.882
8.476
8.457
8.209
8.199
8.197
H
N
N
Pd(OAc)2 5 mol%
K2S2O8 3 equiv
TFA, 70 °C
CO
12 min
N
N
O
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.03
0.04
0.66
3.00
NAME 4
EXPNO 79
PROCNO 1
Date_ 20130607
Time 23.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 228.1
DW 60.400 usec
DE 6.50 usec
TE 298.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


LDD-H3
8.897
8.879
8.479
8.476
8.458
8.456
8.208
8.198
H
N
N
Pd(OAc)2 5 mol%
K2S2O8 3 equiv
TFA, 70 °C
CO
N
N
O
18 min
NAME 4
EXPNO 80
PROCNO 1
Date_ 20130607
Time 23.24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 181
DW 60.400 usec
DE 6.50 usec
TE 298.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300092 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
0.11
0.11
3.00

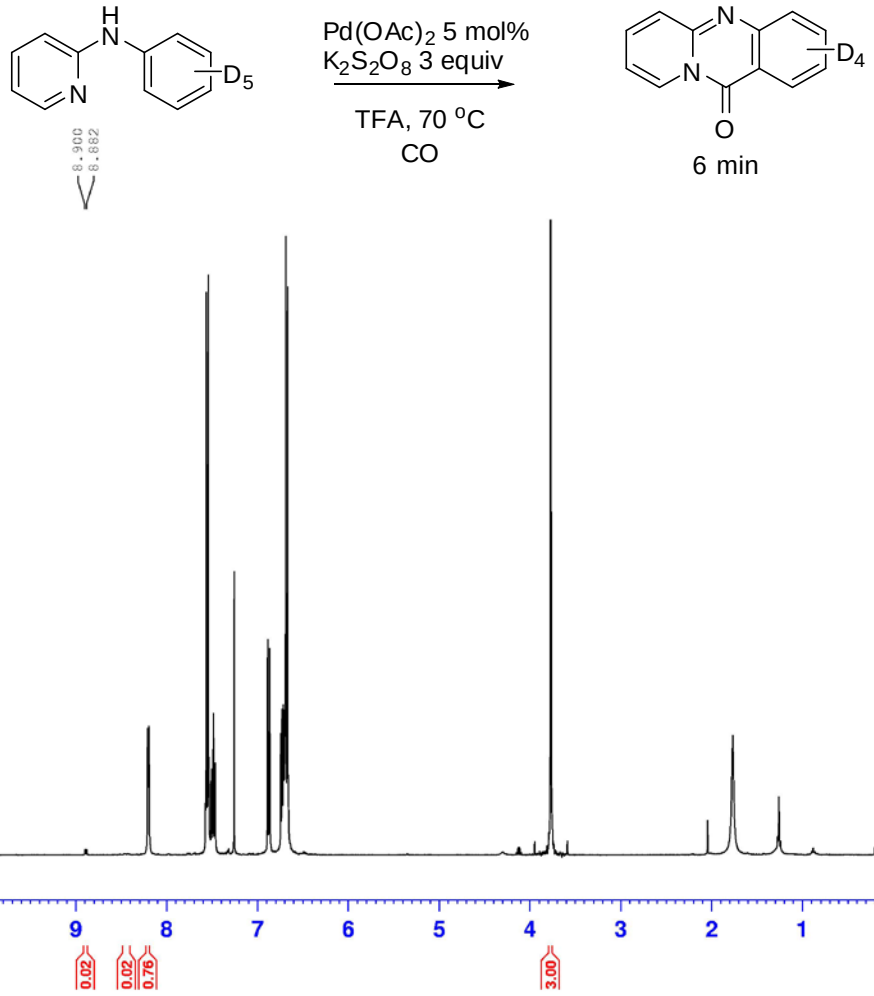
LDD-H4




```
NAME                  4
EXPNO                81
PROCNO               1
Date_         20130607
Time             23.29
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG          zg30
TD              65536
SOLVENT         CDCl3
NS                 16
DS                  2
SWH          8278.146 Hz
FIDRES       0.126314 Hz
AQ           3.9584243 sec
RG                128
DW             60.400 usec
DE              6.50 usec
TE             298.6 K
D1          1.00000000 sec
TD0                 1

======== CHANNEL f1 ========
NUC1                1H
P1              12.58 usec
PL1              0.00 dB
PL1W       10.87646866 W
SFO1       400.1324710 MHz
SI              32768
SF         400.1300092 MHz
WDW                EM
SSB                 0
LB              0.30 Hz
GB                  0
PC              1.00
```

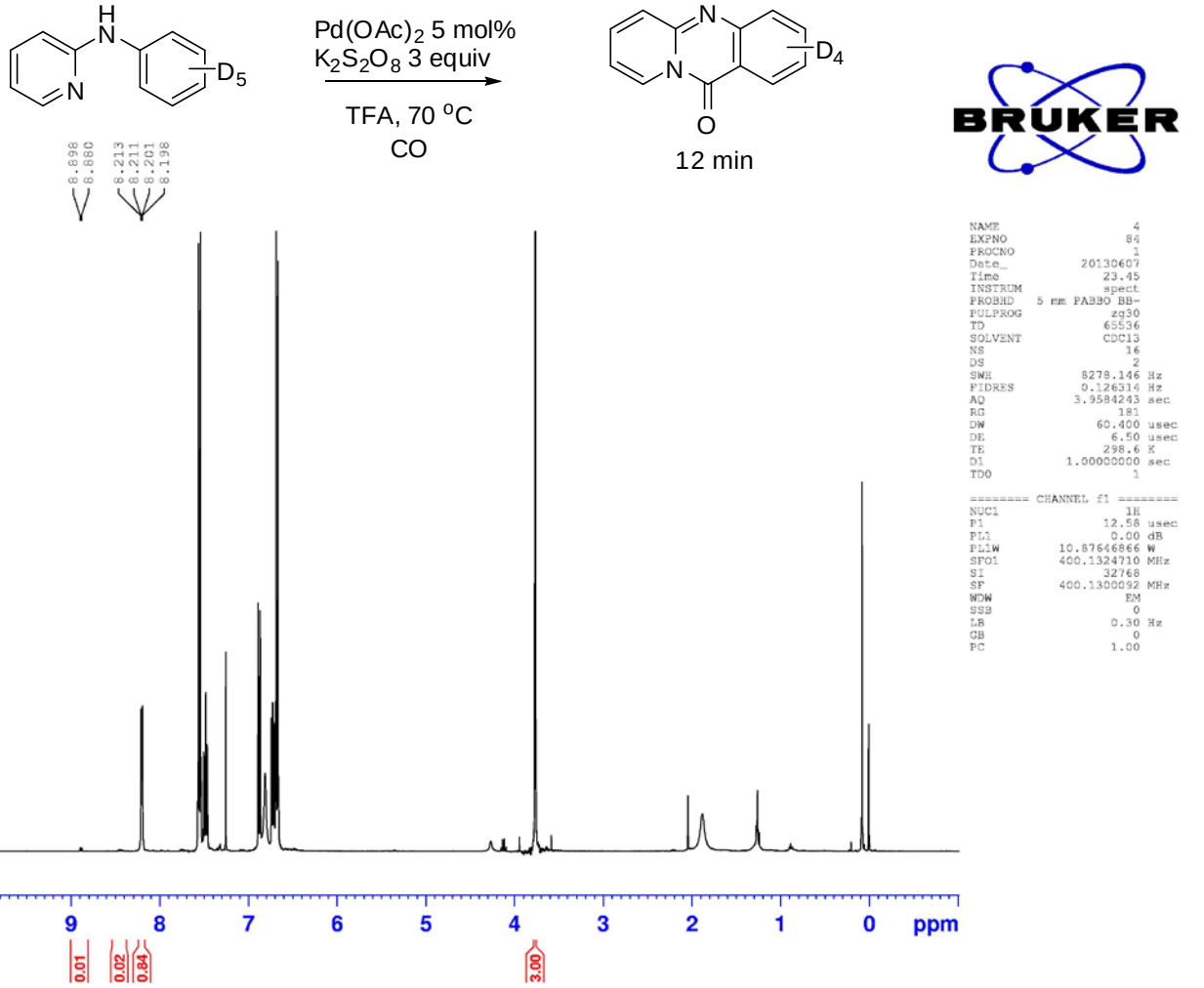

LDD-H5
6.893
6.877
6.874
6.471
6.469
6.451
6.448
6.202
6.192
6.190
H
N
N
Pd(OAc)2 5 mol%
K2S2O8 3 equiv
TFA, 70 °C
CO
30 min
N
N
O
BRUKER
NAME 4
EXPNO 82
PROCNO 1
Date_ 20130607
Time 23.34
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 181
DW 60.400 usec
DE 6.50 usec
TE 298.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300092 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
0.28
0.27
3.00

LDD-D1



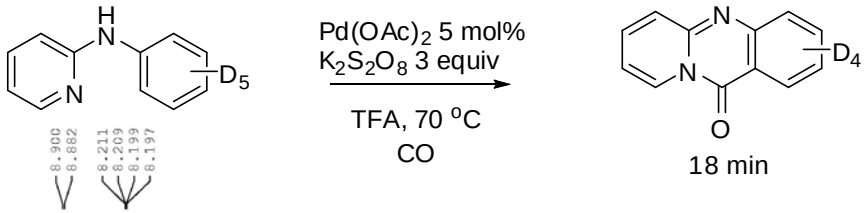

```
NAME                 4
EXPNO               83
PROCNO               1
Date_         20130607
Time              23.40
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                 256
DW                60.400 usec
DE                 6.50 usec
TE                298.6 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1                12.58 usec
PL1                0.00 dB
PL1W        10.87646866 W
SFO1        400.1324710 MHz
SI               32768
SF          400.1300092 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```

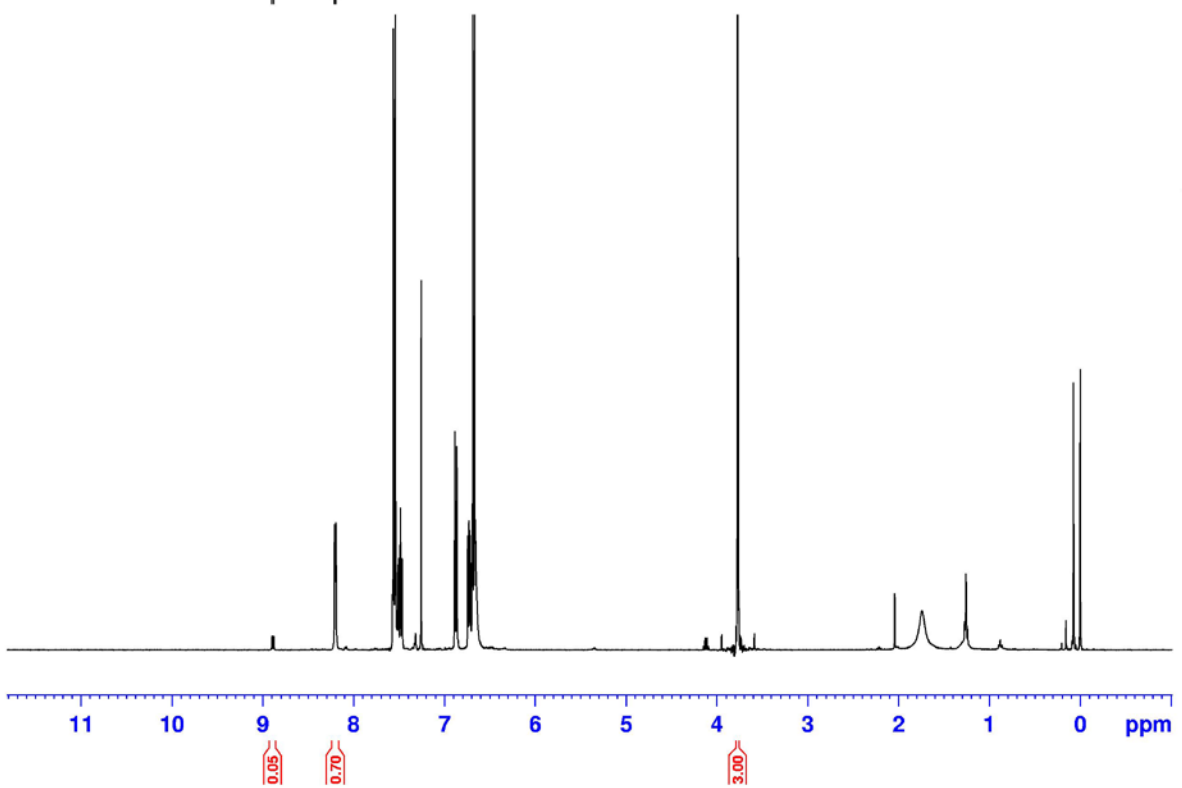
LDD-D2



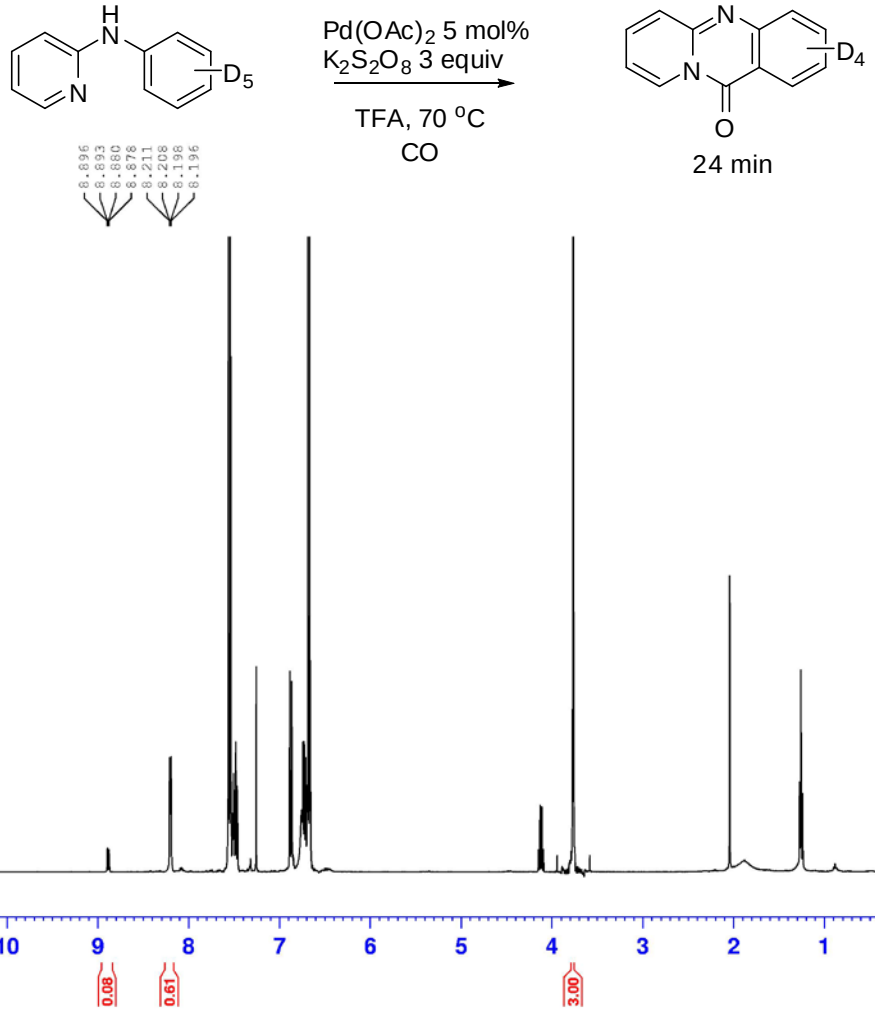
NAME	4
EXPNO	84
PROCNO	1
Date_	20130607
Time	23.45
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	181
DW	60.400 usec
DE	6.50 usec
TE	298.6 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300092 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

LDD-D3

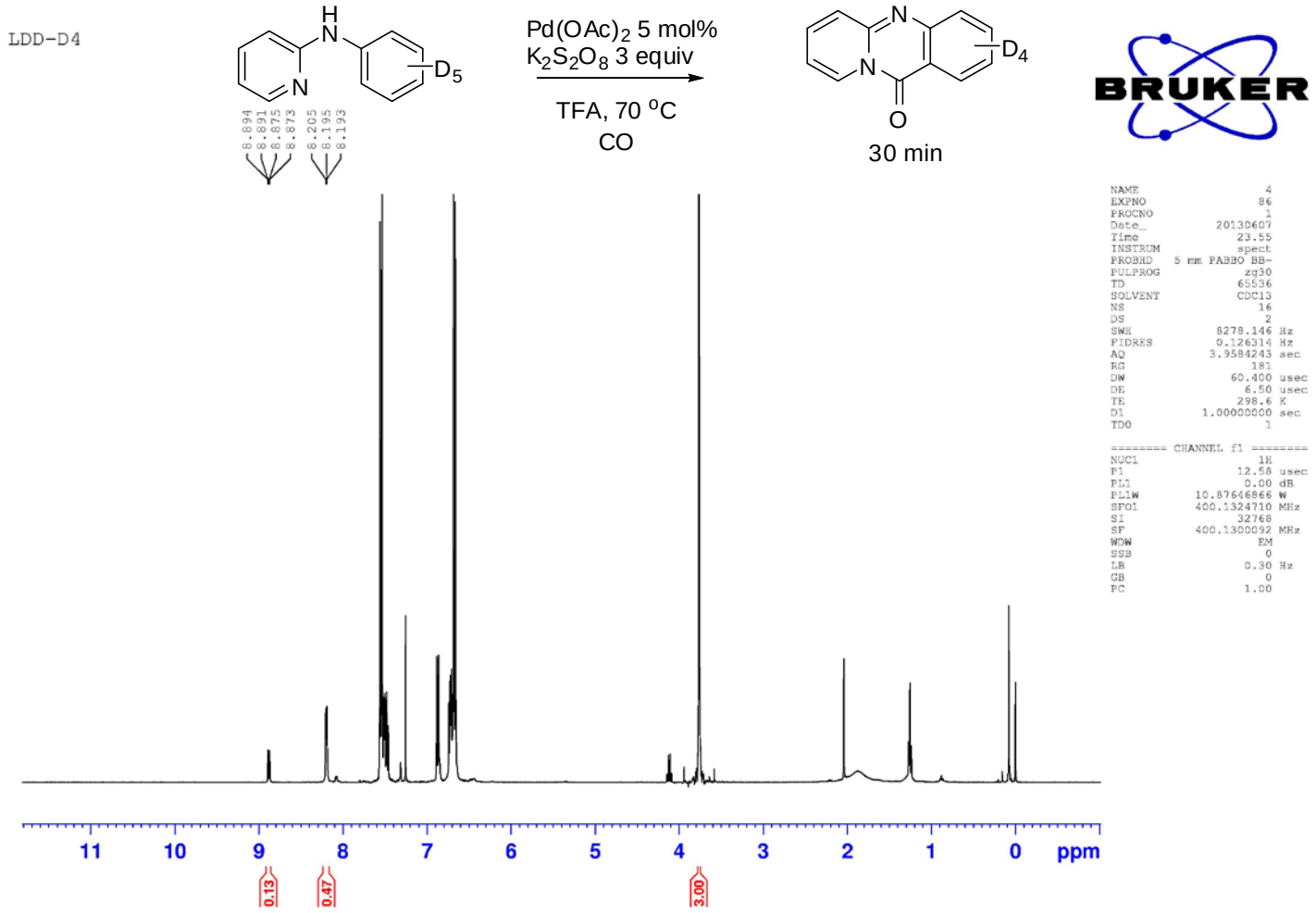





```
NAME                 4
EXPNO               85
PROCNO               1
Date_         20130607
Time             23.50
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG               322.5
DW              60.400 usec
DE               6.50 usec
TE              298.5 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                 1H
P1                12.58 usec
PL1                0.00 dB
PL1W        10.87646866 W
SFO1       400.1324710 MHz
SI               32768
SF         400.1300092 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```



NAME	4
EXPNO	87
PROCNO	1
Date_	20130608
Time	0.00
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	181
DW	60.400 usec
DE	6.50 usec
TE	298.6 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300092 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00


LDD-D4
Pd(OAc)2 5 mol%
K2S2O8 3 equiv
TFA, 70 °C
CO
30 min
D5
D4
8.894
8.891
8.875
8.873
8.205
8.195
8.193
NAME 4
EXPNO 86
PROCNO 1
Date_ 20130607
Time 23.55
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 181
DW 60.400 usec
DE 6.50 usec
TE 298.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300092 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
0.13
0.47
3.00