Electronic Supplementary Information

Brønsted acid assisted activation of imide carbonyl group: Regioselective synthesis of isoindoloisoquinolinone alkaloid (±)-nuevamine

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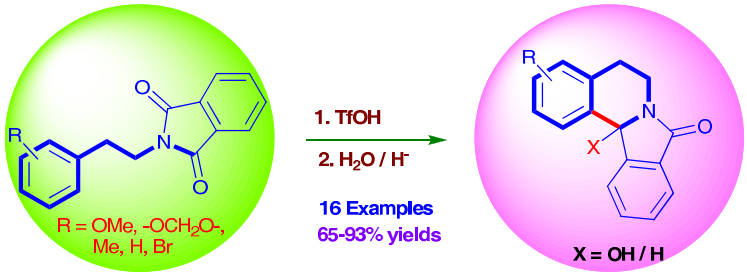
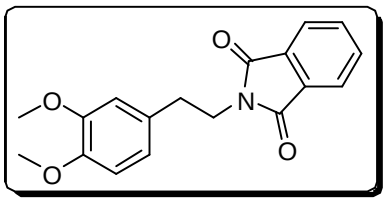


General information: Melting points reported in this paper are uncorrected and were determined using EZ Melt, Stanford Research Systems, USA. Infrared spectra were recorded on Thermo Nicolet 6700 FT-IR Spectrophotometer and are reported in frequency of absorption (cm^{-1}). Mass spectra were measured with micro mass Q-TOF (ESI-HRMS), MALDI-MS were recorden on ABI Voyager DE-STR, ^{1}H and ^{13}C NMR were recorded on Brucker AVANCE 400 spectrometer. NMR spectra for all the samples were measured in $CDCl_3$ or DMSO-d_6 using TMS as an internal standard. The chemical shifts are expressed in δ ppm down field from the signal of internal TMS. Triflic acid was purchased from Aldrich and used without further purification. Phenethylamines were prepared following the reported methods.[1] Solvents used for the reactions were dried using standard procedures.[2] Column chromatography was performed on Merck silica gel 100-200 mesh and TLC analysis was facilitated using phosphomolybdic acid stain in addition to UV light with Merck 60 F_{254} pre-coated silica plates.

Preparation of substituted phenethyl *N*-phthalimides: [3]

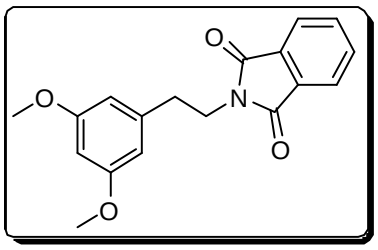
A suspension of phthalic anhydride (12 mmol) in toluene in an oven dried round bottom flask fitted with Dean-Stark apparatus was heated to reflux until complete dissolution of the anhydride and no additional water was removed. To this solution was added appropriate phenethylamines (10 mmol) and refluxing was continued until the water evolution was completed (2-3 h). Reaction mixture was concentrated under reduced pressure to give a residue which was purified through column chromatography to give **1a-k and 1m** in pure form.

***N*-[2-(3,4-Dimethoxyphenyl)ethyl]phthalimide (1a)**[4]



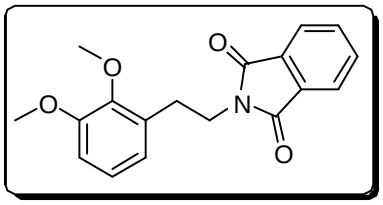
2.713g. white solid, 87% yield; m.p. 171 ºC (*Lit.*[4] 168-170 ºC); IR (KBr, cm^{-1}): 2941, 1759, 1713, 1596; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.8-7.81 (m, 2H) 7.71-7.69 (m, 2H), 6.78-6.78 (m, 2H), 6.74 (d, *J* = 1.2 Hz, 1H), 3.93-3.89 (m, 2H), 3.84 (s, 3H), 3.81 (s, 3H), 2.96-2.92 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.1, 148.8, 147.7, 133.9, 132.0, 130.4, 123.1, 120.8, 111.9, 111.2, 55.8, 55.7, 39.3, 34.0.

_N_-[2-(3,5-Dimethoxyphenyl)ethyl]phthalimide (1b)



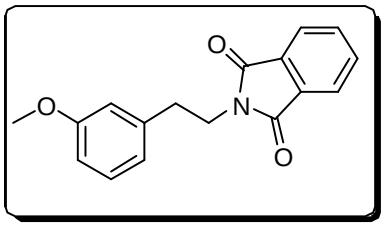
2.182 g. white solid, 70% yield; m.p.140-142 ºC; IR (KBr, cm^{-1}): 2940, 1765, 1707, 1591; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84-7.82 (m, 2H), 7.72-7.69 (m, 2H), 6.41 (d, *J* = 2.0 Hz, 2H), 6.32 (t, *J* = 2.0 Hz, 1H), 3.94-3.90 (m, 2H), 3.74 (s, 6H), 2.95-2.91 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.1, 160.8, 140.2, 133.9, 132.1, 123.2, 106.7, 98.9, 55.2, 39.0, 34.8.

_N_-[2-(2,3-Dimethoxyphenyl)ethyl]phthalimide (1c)



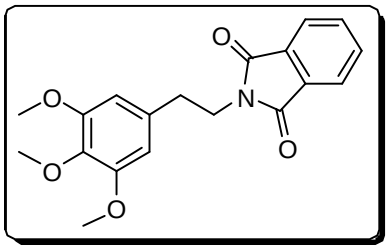
2.649 g. white solid, 85% yield; m.p. 119 ºC; IR (KBr, cm^{-1}): 2943, 1766, 1708, 1586; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83-7.81 (m, 2H), 7.70-7.68 (m, 2H), 6.96-6.92 (m, 1H), 6.81-6.76 (m, 2H), 3.95-3.91 (m, 2H), 3.89 (s, 3H), 3.86 (s, 3H), 3.03-2.99 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.2, 152.7, 147.6, 133.7, 132.1, 131.9, 123.8, 123.1, 122.3, 111.2, 60.7, 55.7, 38.5, 29.1.

_N_-[2-(3-Methoxyphenyl)ethyl]phthalimide (1d)



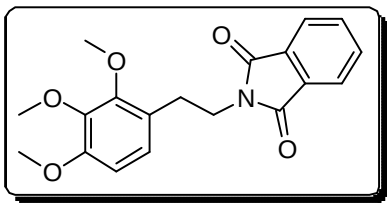
2.06 g. white solid, 73% yield; m.p. 91 ºC; IR (KBr, cm^{-1}): 2937, 1767, 1719, 1602; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83-7.81 (m, 2H), 7.70-7.68 (m, 2H), 7.19 (t, *J* = 7.6 Hz, 1H), 6.84 (d, *J* = 7.6 Hz, 1H), 6.79-6.78 (m, 1H), 6.75 (dd, *J* = 8.0, 2.4 Hz, 1H), 3.93-3.89 (m, 2H), 3.75 (s, 3H), 2.97-2.94 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.1, 159.7, 139.5, 133.9, 132.0, 129.5, 123.2, 121.1, 114.2, 112.2, 55.1, 39.1, 34.6.

_N_-[2-(3,4,5-Trimethoxyphenyl)ethyl]phthalimide (1e)



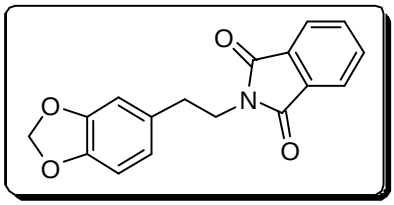
2.683 g. white solid, 78% yield; m.p. 168 ºC; IR (KBr, cm^{-1}): 2940, 1767, 1710, 1589; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84-7.82 (m, 2H), 7.72-7.70 (m, 2H), 6.46 (s, 2H), 3.94-3.90 (m, 2H), 3.81 (s, 6H), 3.80 (s, 3H), 2.96-2.92 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.1, 153.1, 136.5, 133.9, 133.5, 131.9, 105.5, 123.1, 60.7, 55.9, 39.0, 34.7.

_N_-[2-(2,3,4-Trimethoxyphenyl)ethyl]phthalimide (1f)[5]



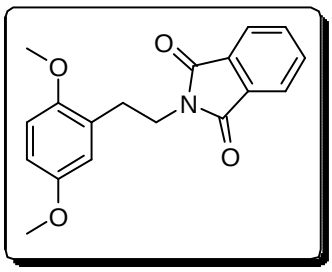
2.8 g. white solid, 82% yield; m.p. 105-107 °C (*Lit.*[5] 110 ºC); IR (KBr, cm^{-1}): 3011, 2942, 2832, 1765, 1712, 1603, 1497, 1397, 1280, 1110, 1000, 907, 796; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82-7.80 (m, 2H), 7.69-7.67 (m, 2H), 6.83 (d, *J* = 8.5 Hz, 1H), 6.55 (d, *J* = 8.5 Hz, 1H), 3.91-3.87 (m, 5H), 3.81 (s, 3H), 3.75 (s, 3H), 2.94-2.91 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.2, 152.6, 152.2, 142.1, 133.7, 132.2, 124.4, 124.0, 123.0, 107.0, 60.8, 60.5, 55.9, 38.6, 28.9.

_N_-[2-(3,4-Methylenedioxyphenyl)ethyl]phthalimide (1g)[6]



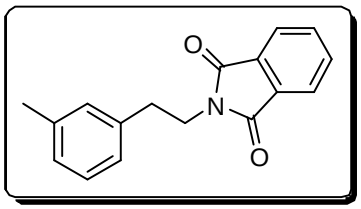
2.596 g. white solid, 88% yield; m.p. 139-141 ºC (*Lit.*[6] 139-140 ºC); IR (KBr, cm^{-1}): 2938, 2888, 1767, 1706, 1611, 1496, 1399, 1099, 929, 715; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.75 (d, *J* = 1.2 Hz, 1H), 6.70 (d, *J* = 8.0 Hz, 1H), 6.67 (dd, *J* = 8.0, 1.2 Hz, 1H), 5.91 (s, 2H), 3.89-3.85 (m, 2H), 2.92-2.88 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.1, 147.7, 146.2, 133.9, 132.0, 131.7, 123.2, 121.7, 109.2, 108.3, 100.8, 39.4, 34.3.

_N_-[2-(2,5-Dimethoxyphenyl)ethyl]phthalimide (1h)



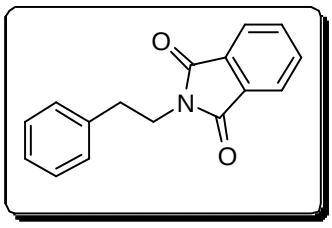
2.457 g. white solid, 79% yield; m.p. 95 °C; IR (KBr, cm^{-1}): 2938, 1705, 1575, 1501, 13821256; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82-7.77 (m, 2H), 7.70-7.66 (m, 2H), 6.73-6.68 (m, 3H), 3.93 (t, *J* = 7.2 Hz, 2H), 3.70 (s, 3H), 3.66 (s, 3H), 2.97 (t, *J* = 7.2 Hz, 2H).; ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.2, 153.3, 152.0, 133.7, 132.1, 127.6, 123.0, 116.6, 112.2, 111.1, 55.7, 55.6, 37.8, 29.7.

_N_-[2-(3-Methylphenyl)ethyl]phthalimide (1i)



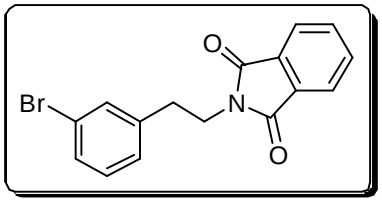
2.2 g. white solid, 83% yield; m.p. 88 °C; IR (KBr, cm^{-1}): 3009, 2934, 2862, 1773, 1731, 1610, 1457, 1434, 1391, 1351, 1184, 1081, 1001, 865, 782, 717, 529, 495, 439; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84-7.82 (m, 2H), 7.71-7.69 (m, 2H), 7.17 (t, *J* = 7.6, 1H), 7.07-7.02 (m, 3H), 3.92-3.88 (m, 2H), 2.96-2.92 (m, 2H), 2.30 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.1, 138.1, 137.9, 133.8, 132.1, 129.6, 128.4, 127.3, 125.8, 123.1, 39.3, 34.5, 21.3.

_N_-[2-Phenyl ethyl]phthalimide (1j)[7]



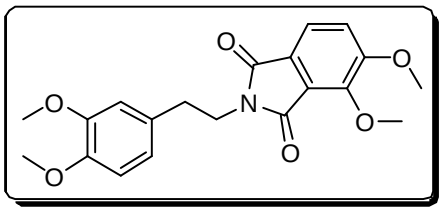
2.08 g. white solid, 83% yield; m.p.125 ºC (*Lit.*[7] 125 ºC); IR (KBr, cm^{-1}): 2932, 1763, 1711, 1602, 1400; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.97-3.01 (m, 2H), 3.90-3.94 (m, 2H), 7.19-7.30 (m, 5H), 7.69-7.29 (m, 2H), 7.81 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 34.6, 39.2, 123.2, 126.6, 128.5, 128.8, 132.0, 133.9, 138.0, 168.1.

_N_-[2-(3-Bromophenyl)ethyl]phthalimide (1k)



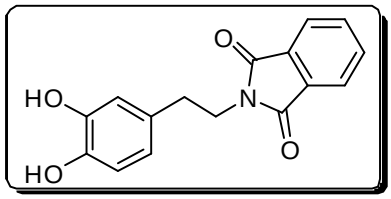
2.64 g, white solid, 80% yield; m.p. 97 ºC; IR (KBr, cm^{-1}): 3054, 2947, 1768, 1708, 1566, 1427, 1395, 1359, 1075, 717; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83-7.80 (m, 2H), 7.72-7.69 (m, 2H), 7.30-7.19 (m, 4H), 3.94-3.90 (m, 2H), 3.01-2.97 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.1, 138.0, 133.9, 132.0, 128.8, 128.5 126.6, 123.2, 39.2, 34.6.

2-(3,4-dimethoxyphenethyl)]-4,5-dimethoxyisoindoline-1,3-dione (1m)



3.31 g. white solid, 87% yield; m.p. 120 °C; IR (KBr, cm^{-1}): 3007, 2937, 2843, 1765, 1712, 1591, 1517, 1497, 1435, 1390, 1278, 1262, 1240, 1026.; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.51 (d, J = 8.0 Hz, 1H), 7.08 (d, J = 8.0 Hz, 1H), 6.784-6.781 (m, 2H), 6.75 (s, 1H), 4.11 (s, 3H), 3.94 (s, 3H), 3.87-3.84 (m, 2H), 3.83 (s, 3H), 3.82 (s, 3H), 2.93-2.89 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.5, 166.1, 157.6, 148.8, 147.6, 147.1, 130.6, 124.6, 121.8, 120.8, 119.2, 115.7, 112.0, 111.2, 62.5, 56.6, 55.86, 55.80, 39.3, 34.0.

_N_-[2-(3,4-Dihydroxyphenyl)ethyl]phthalimide (1l)[8]



An oven dried two neck round bottom flask bearing septum in side arm was cooled to room temperature under a steady stream of nitrogen gas flow. The flask was charged with stirring bar, substrate **1f** (1 mmol) and dry dichloromethane (15 ml) and cooled down to −15 °C (using ice and salt as a freezing mixture). To this solution was added BBr_3 (3 ml, 1M soln. in dichloromethane) with stirring. After 2 h, the reaction mixture was quenched with water (10 ml). The organic layer was separated and aqueous layer was extracted with

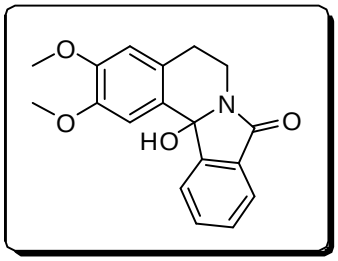
dichloromethane (2 x 15 ml). The combined organic extract was washed with brine solution and dried over anhydrous Na_2SO_4. Filtered and the solvent was removed under vacuum on rotary evaporator to dryness. The dried compound was purified through silica gel column chromatography using ethyl acetate and hexane (50:50) as eluent; 252 mg. white solid, 89% yield; m.p. 175 ºC; IR (KBr, cm^{-1}): 3272, 2959, 1763, 1681, 1600, 1393, 1262; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.82 (s, 1H), 8.71 (s, 1H), 7.90-7.85 (m, 4H), 6.64-6.62 (m, 2H), 6.45 (dd, *J* = 2.0, 8.0 Hz, 1H), 3.76 (t, *J* = 3.8 Hz, 2H), 2.77 (t, *J* = 3.6 Hz, 2H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 167.6, 145.0, 143.6, 134.3, 131.4, 128.8, 122.9, 119.1, 115.8, 115.4, 39.1, 33.0.

Typical procedure for TfOH mediated cyclization.

An oven dried two neck round bottom flask bearing septum in side arm was cooled to room temperature under a steady stream of nitrogen gas flow. The flask was charged with stirring bar, imide (0.5 mmol) and dry dichloromethane (15 ml) and cooled down to 0 °C (using ice). To this solution was added TfOH (0.2 ml, 4 equiv) with stirring. After 30 minutes, the reaction mixture was quenched with water (10 ml) followed by $NaHCO_3$ (1g). The organic layer was separated and aqueous layer was extracted with dichloromethane (2 x 15 ml). The combined organic extract was washed with brine solution and dried over anhydrous Na_2SO_4. Filtered and the solvent was removed under vacuum on rotary evaporator to dryness. The dried compound was purified through the short silica gel column chromatography using ethyl acetate and hexane as eluent.

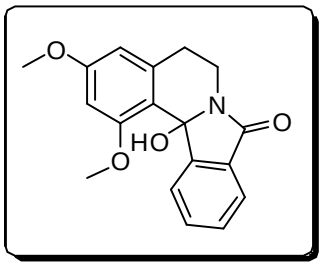
The isolation of compound **2d** was achieved using ethyl acetate as extracting solvent instead of dichloromethane.

12b-Hydroxy-2,3-dimethoxy-5,12b-dihydro-6*H*-isoindolo[1,2-a]isoquinolin-8-one (2a)[4]



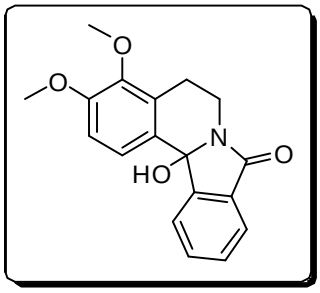
149 mg. white solid, 96% yield; m.p. 169 ºC (*Lit.*[4] 157-159 ºC); IR (KBr, cm^{-1}): 3331, 1672, 1614, 1518; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.99 (d, *J* = 7.6 Hz, 1H), 7.67 (d, *J* = 7.6 Hz, 1H), 7.63 (td, *J* = 7.6, 1.2 Hz, 1H), 7.47 (td, *J* = 7.6, 1.2 Hz, 1H), 7.41 (s, 1H), 6.56 (s, 1H), 4.16 (ddd, *J* = 13.2, 7.6, 1.6 Hz, 1H), 4.04 (s, 1H), 3.95 (s, 3H), 3.83 (s, 3H), 3.42-3.35 (m, 1H), 2.95-2.87 (m, 1H), 2.67 (ddd, *J* = 16.0, 8.0, 1.6 Hz, 1H), ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.4, 149.4, 148.2, 148.0, 132.7, 130.6, 129.5, 127.8, 127.6, 123.7, 123.0, 111.5, 110.4, 86.4, 56.2, 56.0, 34.9, 29.1.

12b-Hydroxy-1,3-dimethoxy-5,12b-dihydro-6*H*-isoindolo[1,2-a]isoquinolin-8-one (2b)



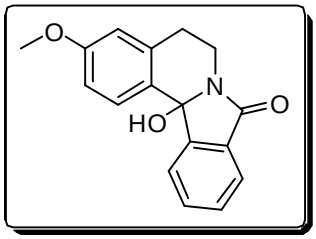
140 mg. white solid, 90% yield; m.p. 168 ºC; IR (KBr, cm^{-1}): 3307, 1675, 1594, 1443; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.14 (d, *J* = 7.6 Hz, 1H), 7.73 (d, *J* = 7.6 Hz, 1H), 7.51 (td, *J* = 7.6, 1.2 Hz, 1H), 7.42 (td, *J* = 7.6 ,1.2 Hz, 1H), 6.43 (d, *J* = 2.4 Hz, 1H), 6.27 (d, *J* = 2.0 Hz, 1H), 4.39 (ddd, *J* = 12.8, 5.6, 3.2 Hz, 1H), 4.31 (s, 1H), 4.04 (s, 3H), 3.76 (s, 3H), 3.33 (td, *J* = 12.8, 3.2 Hz, 1H), 3.00-2.91 (m, 1H), 2.62 (dd, *J* = 16.4, 2.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.7, 160.0, 158.6, 148.9, 138.5, 132.3, 130.6, 129.2, 124.1, 123.1, 117.2, 105.5, 97.8, 87.1, 55.34, 55.31, 35.1, 30.6.

12b-Hydroxy-3,4-dimethoxy-5,12b-dihydro-6*H*-isoindolo[1,2-a]isoquinolin-8-one (2c)



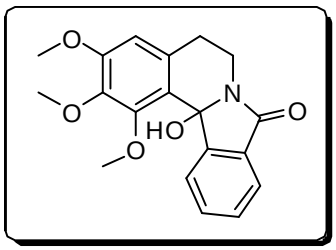
132 mg. white solid, 85% yield; m.p. 198 ºC; IR (KBr, cm^{-1}): 3293, 1678, 1602, 1458; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.99 (d, *J* = 8.0 Hz, 1H), 7.69-7.68 (m, 1H), 7.69 (s, 1H), 7.62 (td, *J* = 7.6, 1.2 Hz, 1H), 7.46 (td, *J* = 7.2, 0.8 Hz, 1H), 6.87 (d, *J* = 8.8 Hz, 1H), 4.20 (ddd, *J* = 13.2, 6.4, 2.4 Hz, 1H), 3.83 (s, 3H), 3.81 (s, 1H), 3.73 (s, 3H), 3.41-3.34 (m, 1H), 3.02 (ddd, *J* = 16.8, 4.4, 2.0 Hz, 1H), 2.81–2.72 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.1, 152.4, 147.9, 146.2, 132.5, 130.5, 129.6, 129.5, 128.9, 123.5, 123.4, 123.2, 110.8, 86.1, 60.1, 55.7, 34.2, 23.4.

12b-Hydroxy-3-methoxy-5,12b-dihydro-6*H*-isoindolo[1,2-a]isoquinolin-8-one (2d)



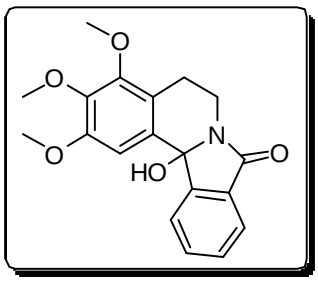
130 mg. white solid, 93% yield; m.p. 169 ºC; IR (KBr, cm^{-1}): 3265, 1681, 1613, 1580, 1407; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.12 (d, *J* = 8.0 Hz, 1H), 7.89 (d, *J* = 4.8 Hz, 1H), 7.63-7.68 (m, 2H), 7.51 (td, *J* = 7.6, 0.8 Hz, 1H), 6.89 (s, 1H), 6.84 (dd, *J* = 8.4, 2.4 Hz, 1H), 6.72 (d, *J* = 2.4 Hz, 1H), 4.23-4.17 (m, 1H), 3.71 (s, 3H), 3.45-3.38 (m, 1H), 2.79 (dd, *J* = 7.6, 4.0 Hz, 2H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 165.8, 158.5, 148.7, 135.9, 132.2, 130.2, 129.6, 129.2, 129.1, 123.7, 122.3, 113.0, 112.9, 85.4, 55.0, 29.0, 34.1.

12b-Hydroxy-1,2,3-trimethoxy-5,12b-dihydro-6*H*-isoindolo[1,2-a]isoquinolin-8-one (2e)



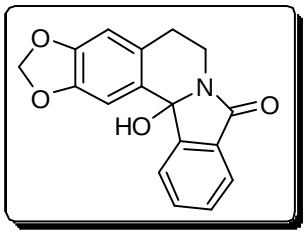
152 mg. white solid, 89% yield; m.p. 179 ºC; IR (KBr, cm^{-1}): 3282, 1675, 1600, 1456; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.49 (d, *J* = 7.6 Hz, 1H), 7.60 (dd, *J* = 7.6, 0.8 Hz, 1H), 7.55 (td, *J* = 7.6, 1.2 Hz, 1H), 7.41 (td, *J* = 7.6, 0.8 Hz, 1H), 6.38 (s, 1H), 4.30 (s, 1H), 4.13 (s, 3H), 4.05-4.01 (m, 1H), 3.85 (s, 3H), 3.80 (s, 3H), 3.25 (td, *J* = 12.8, 3.6 Hz, 1H), 2.81-2.89 (m, 1H), 2.56 (dd, *J* = 16.0, 2.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.5, 153.3, 153.2, 148.7, 141.3, 132.5, 131.4, 130.4, 129.2, 125.9, 122.8, 122.3, 107.7, 87.7, 61.5, 60.8, 55.8, 34.6, 30.3.

12b-Hydroxy-2,3,4-trimethoxy-5,12b-dihydro-6*H*-isoindolo[1,2-a]isoquinolin-8-one (2f)



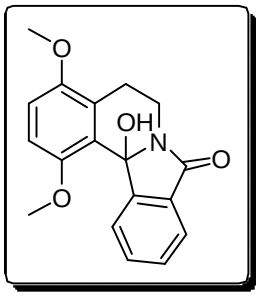
155 mg. white solid, 91% yield; m.p. 157-158 ºC; IR (KBr, cm^{-1}): 3289, 2937, 2835, 1684, 1605, 1457, 1323, 1417, 1112, 1030, 753; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.99 (d, *J* = 7.6 Hz, 1H), 7.69 (d, *J* = 7.6 Hz, 1H), 7.64 (td, *J* = 1.2, 7.6 Hz, 1H), 7.48 (td, *J* = 7.6, 0.8 Hz, 1H), 7.27 (s, 1H), 4.21 (dd, *J* = 6.2, 13.0 Hz, 1H), 3.94 (s, 3H), 3.83 (s, 3H), 3.79 (s, 4H), 3.34 (td, *J* = 4.4, 13.0, Hz, 1H), 2.88 (ddd, *J* = 1.7, 4.4, 16.7 Hz, 1H), 2.68 (ddd, *J* = 6., 12.0, 16.7 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.3, 152.1, 150.9, 148.0, 142.3, 132.5, 131.1, 130.4, 129.4, 123.4, 123.1, 121.8, 106.6, 86.2, 60.7, 60.5, 56.1, 34.3, 23.2; HRMS-ESI (*m/z*): Calculated for $C_{19}H_{19}NO_5$ (M+Na): 364.1161, Found (M+Na): 364.1168.

11b-Hydroxy-5,11b-dihydro-6H-1,3-dioxa-6a-azaindeno[5,6-c]fluoren-7-one (2g)[9]



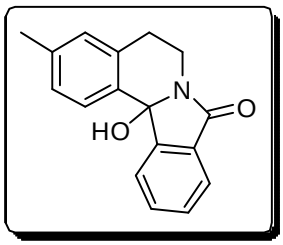
96 mg. white solid, 65% yield; m.p. 176-178 ºC (*Lit.*[9] 175-176 ºC); IR (KBr, cm^{-1}): 3251, 2995, 2910, 2767, 1679, 1617, 1482, 1431, 1234, 1034, 701; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.96 (d, *J* = 7.5 Hz, 1H), 7.69 (d, *J* = 7.5 Hz, 1H), 7.63 (td, *J* = 1.1, 7.5 Hz, 1H), 7.48 (td, *J* = 0.8, 7.5 Hz, 1H), 7.39 (s, 1H), 6.56 (s, 1H), 5.95 (d, *J* = 1.2 Hz, 1H), 5.90 (d, *J* = 1.2 Hz, 1H), 4.18 (ddd, *J* = 2.1, 6.0, 13.2 Hz, 1H), 3.63 (s, 1H), 3.39 (ddd, *J* = 4.4, 11.6, 13.2 Hz, 1H), 2.93-2.85 (m, 1H), 2.69 (ddd, *J* = 2.1, 4.4, 16.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.3, 147.8, 147.7, 146.6, 132.6, 130.4, 129.5, 128.8, 123.5, 123.1, 108.7, 107.3, 101.2, 86.4, 34.8, 29.4.

12b-Hydroxy-1,4-dimethoxy-5,12b-dihydro-6*H*-isoindolo[1,2-a]isoquinolin-8-one (2h)



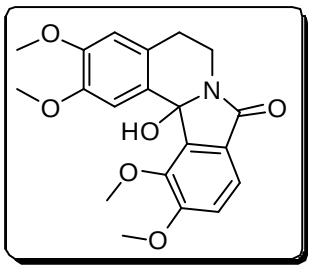
135 mg. white solid, 87% yield; m.p. 165-166 ºC; IR (KBr, cm^{-1}): 3398, 3054, 2925, 2854, 1717, 1675, 1586, 1458, , 1290, 1063, 1020, 887, 739; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.16-8.14 (m, 1H), 7.79-7.77 (m, 1H), 7.51 (td, *J* = 1.3, 7.5 Hz, 1H), 7.45 (td, *J* = 1.0, 7.5 Hz, 1H), 6.84 (d, *J* = 8.9 Hz, 1H), 6.76 (d, *J* = 8.9 Hz, 1H), 4.53 (ddd, *J* = 1.0, 5.8, 13.0 Hz, 1H), 4.45 (s, 1H), 4.07 (s, 3H), 3.75 (s, 3H), 3.30 (td, *J* = 3.5, 13.0 Hz, 1H), 2.91 (ddd, *J* = 1.0, 3.5 17.2 Hz, 1H), 2.68-2.59 (m, 1H).; ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.8, 151.7, 151.2, 148.8, 132.4, 130.8, 129.4, 126.7, 125.6, 124.1, 123.2, 110.1, 108.9, 87.1, 55.8, 55.5, 34.7, 24.1; HRMS-ESI (*m/z*): Calculated for $C_{18}H_{17}NO_4$ (M+Na): 334.1055, Found (M+Na): 334.1053

12b-Hydroxy-3-methyl-5,12b-dihydro-6*H*-isoindolo[1,2-a]isoquinolin-8-one (2i)



103 mg. white solid, 78% yield; m.p. 174-175 ºC; IR (KBr, cm^{-1}): 3269, 2947, 2893, 2840, 1695, 1614, 1576, 1417, 1294, 1107, 1027, 939, 824, 768, 701; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.01 (d, *J* = 8.0 Hz, 1H), 7.81 (d, *J* = 8.0 Hz, 1H), 7.62-7.58 (m, 2H), 7.42 (td, *J* = 0.8, 7.6 Hz, 1H), 7.09 (d, *J* = 12 Hz, 1H), 6.93 (s, 1H), 4.28 (s, 1H), 4.04 (ddd, *J* = 2.4, 6.0, 13.1 Hz, 1H), 3.36 (ddd, *J* = 4.4, 11.2, 13.1 Hz, 1H), 2.94-2.85 (m, 1H), 2.74 (ddd, *J* = 2.4, 4.4, 12.4 Hz, 1H), 2.28 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.3,147.9, 138.4, 134.5, 133.0, 132.5, 130.4, 129.7, 129.3, 127.6, 127.3, 123.4, 123.3, 86.4, 34.7, 29.2, 21.0; HRMS-ESI (*m/z*): Calculated for $C_{17}H_{15}NO_2$ (M+Na): 288.1000, Found (M+Na): 288.1010.

12b-Hydroxy-2,3,11,12-tetramethoxy-5,12b-dihydro-6*H*-isoindolo[1,2-a]isoquinolin-8-one (2m)

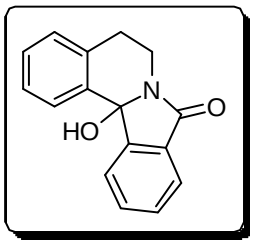


158 mg. white solid, 85% yield; m.p. 158-160 ºC; IR (KBr, cm^{-1}): 3241, 1996, 2936, 2836, 1680, 1614, 1494, 1334, 1135, 967; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.23 (s, 1H), 7.48 (d, *J* = 8.2, 1H), 7.03 (d, *J* = 8.2, 1H), 6.5 (s, 1H), 4.22 (ddd, *J* = 2.0, 6.1, 13.0 Hz, 1H), 4.12 (s, 3H), 3.96 (s, 3H), 3.91 (s, 3H), 3.83 (s, 3H), 3.66 (s, 1H), 3.44 (ddd, *J* = 4.5, 11.8, 13.0 Hz, 1H), 3.00-2.92 (m, 1H), 2.70 (ddd, *J* = 1.8, 4.3, 16.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.5, 157.2, 148.9, 147.7, 144.6, 140.3, 128.7, 127.0, 124.3, 119.9, 113.4, 112.0, 111.0, 87.9, 61.9, 56.4, 55.9, 55.8, 34.7, 28.7; HRMS-ESI (*m/z*): Calculated for $C_{20}H_{21}NO_6$ (M+Na): 394.1267, Found (M+Na): 394.1266.

Cyclization of the imides 1j and 1k

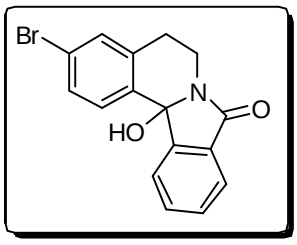
An oven dried two neck round bottom flask bearing septum in side arm was cooled to room temperature under a steady stream of nitrogen gas flow. The flask was charged with stirring bar, substrate (0.5 mmol) and TfOH (0.4 ml, 8 equiv) with stirring and heated for 48 h at 70 ˚C. This mixture was quenched with water (10 ml) followed by $NaHCO_3$ (1g). The precipitate was digested in dichloromethane and organic layer was separated and aqueous layer was extracted with dichloromethane (2 x 15 ml). The combined organic extract was washed with brine solution and dried over anhydrous Na_2SO_4. Filtered and the solvent was removed under vacuum on rotary evaporator to dryness. The dried compound was purified through short silica gel column chromatography using ethyl acetate and hexane as eluent.

12b-Hydroxy-5,12b-dihydro-6*H*-isoindolo[1,2-a]isoquinolin-8-one (2j)



98 mg. white solid, 78% yield; m.p. 144 ºC; IR (KBr, cm^{-1}): 3284, 3065, 2929, 1679, 1606, 1419, 1295, 1-39, 945, 769, 698; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.04 (d, *J* = 7.7 Hz, 1H), 7.95 (dd, *J* = 1.3, 7.5 Hz, 1H), 7.73 (dt, *J* = 1.3, 7.5 Hz, 1H), 7.64 (td, *J* = 1.2, 7.5 Hz, 1H), 7.49 (td, *J* = 0.9, 7.5 Hz, 1H), 7.32-7.28 (m, 1H), 7.27-7.23 (m, 1H), 7.16-7.14 (m, 1H), 4.29 (ddd, *J* = 2.4, 6.1, 13.1 Hz, 1H), 3.49 (ddd, *J* = 4.5, 11.5, 13.1 Hz, 1H), 3.40 (s, 1H), 3.06-2.97 (m, 1H), 2.85 (ddd, *J* = 2.3, 4.3, 16.2 Hz, 1H).; ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.3, 147.7, 136.9, 134.9, 132.7, 132.0, 130.3, 130.0, 129.7, 129.1, 123.5, 123.2, 122.5, 86.2, 34.4, 29.0; HRMS-ESI (*m/z*): Calculated for $C_{16}H_{13}NO_2$ (M+Na): 274.0844, Found (M+Na): 274.0844.

12b-Hydroxy-3-bromo-5,12b-dihydro-6*H*-isoindolo[1,2-a]isoquinolin-8-one (2k)



109 mg. white solid, 66% yield; m.p. 163 ºC; IR (KBr, cm^{-1}): 3246, 2951, 2894, 2838, 1683, 1593, 1472, 1414, 1181, 1114, 1040, 939,, 883, 765, 697; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.98 (d, *J* = 7.5 Hz, 1H), 7.80 (d, *J* = 8.4 Hz, 1H), 7.70-7.68 (m, 1H), 7.65 (td, *J* = 1.2, 7.5 Hz, 1H), 7.49 (td, *J* = 0.8, 7.5 Hz, 1H), 7.42 (dd, *J* = 1.8, 8.3 Hz, 1H), 7.316-7.311 (m, 1H), 4.18 (ddd, *J* = 2.4, 6.1, 13.1 Hz, 1H), 3.71 (s, 1H), 3.42 (ddd, *J* = 4.5, 11.4, 13.1 Hz, 1H), 2.99-2.91 (m, 1H), 2.80 (ddd, *J* = 2.3, 4.2, 16.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.3, 147.4, 136.9, 134.9, 132.7,

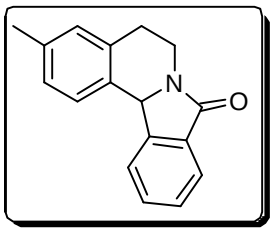
132.0, 130.3, 130.0, 129.7, 129.1, 123.5, 123.2, 122.5, 86.2, 34.4, 29.0; HRMS-ESI (*m/z*): Calculated for $C_{16}H_{12}BrNO_2$ (M+Na): 351.9949, Found (M+Na): 351.9948.

Typical procedure for cyclization followed by $NaBH_4$/TFA reduction.[10]

An oven dried two neck round bottom flask bearing septum in side arm was cooled to room temperature under a steady stream of nitrogen gas flow. The flask was charged with stirring bar, imide (0.5 mmol) and dry dichloromethane (15 ml) and cooled down to 0 °C. To this solution was added TfOH (0.2 ml, 2 mmol) with stirring. After the stipulated time the contents were brought to room temperature and $NaBH_4$ (2 mmol) was added followed by TFA (1 ml) and the solution was stirred until color disappears (additional $NaBH_4$ and TFA was used if color persists for long time). To this mixture acetone was added and evaporated under reduced pressure to dryness. The solid residue was dissolved in dichloromethane (20 ml) and the insoluble material was filtered off, the organic layer was dried over anhydrous Na_2SO_4 and filtered. The solvent was evaporated under vacuum and the crude product was purified through silica gel column chromatography.

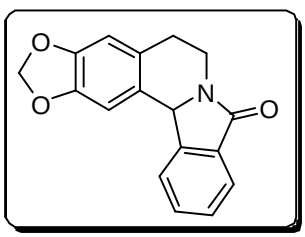
The isolation of compound **3l** was achieved using ethyl acetate as extracting solvent instead of dichloromethane.

3-methyl-5,12*b*-dihydroisoindolo[1,2-*a*]isoquinolin-8(6*H*)-one (3h)



114 mg. white semi solid, 92% yield; IR (KBr, cm^{-1}): 3018, 2929, 1696, 1614, 1468, 1394, 1298, 735.; ^1H NMR (400 MHz, $CDCl_3$): δ 7.88-7.84 (m, 2H), 7.60 (td, *J* = 1.2, 7.5 Hz, 1H), 7.52-7.46 (m, 2H), 7.08 (d, *J* = 7.9 Hz, 1H), 7.01 (s, 1H), 5.65 (s, 1H), 4.44 (ddd, *J* = 4.3, 5.7, 12.8 Hz, 1H), 3.47 (ddd, *J* = 4.5, 9.6, 12.8 Hz, 1H), 3.07-2.99 (m, 1H), 2.84 (dt, *J* = 4.5, 15.9 Hz, 1H), 2.31 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.9, 144.4, 137.1, 134.5, 132.7, 131.4, 131.3, 129.8, 128.3, 128.0, 127.4, 125.1, 123.7, 123.4, 59.0, 38.2, 29.3, 21.1, 20.9; MALDI-MS (*m/z*): Calculated for $C_{17}H_{15}NO$ (M+H): 250.1232, Found (M+H): 250.1281.

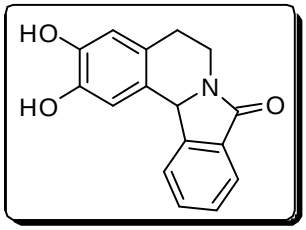
5,11b-Dihydro-6H-1,3-dioxa-6a-aza-indeno[5,6-c]-fluoren-7-one (3f)[9]



119 mg. white solid, 85% yield; m.p. 180-181 ºC (*Lit.*[9] 179-180 ºC); IR (KBr, cm^{-1}): 3031, 2948, 2907, 1691, 1499, 1238, 1029, 899, 837, 735, 688; ^1H NMR (400 MHz, $CDCl_3$): δ 7.88 (d, *J* = 7.5 Hz, 1H), 7.80 (dd, *J* = 0.7,

7.5 Hz, 1H), 7.61 (td, *J* = 1.2, 7.5 Hz, 1H), 7.50 (t, *J* = 7.5 Hz, 1H), 7.08 (s, 1H), 6.65 (s, 1H), 5.96 (d, *J* = 1.2 Hz, 1H), 5.89 (d, *J* = 1.2 Hz, 1H), 5.57 (s, 1H), 4.37 (ddd, *J* = 4.8, 5.5, 13.0 Hz, 1H), 3.45 (ddd, *J* = 4.7, 9.2, 13.0 Hz, 1H), 3.02-2.94 (m, 1H), 2.78 (dt, *J* = 4.7, 15.7 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.9, 146.8, 146.5, 144.2, 132.7, 131.5, 128.5, 128.2, 127.2, 123.9, 123.3, 109.1, 105.5, 101.1, 59.1, 38.2, 29.4.

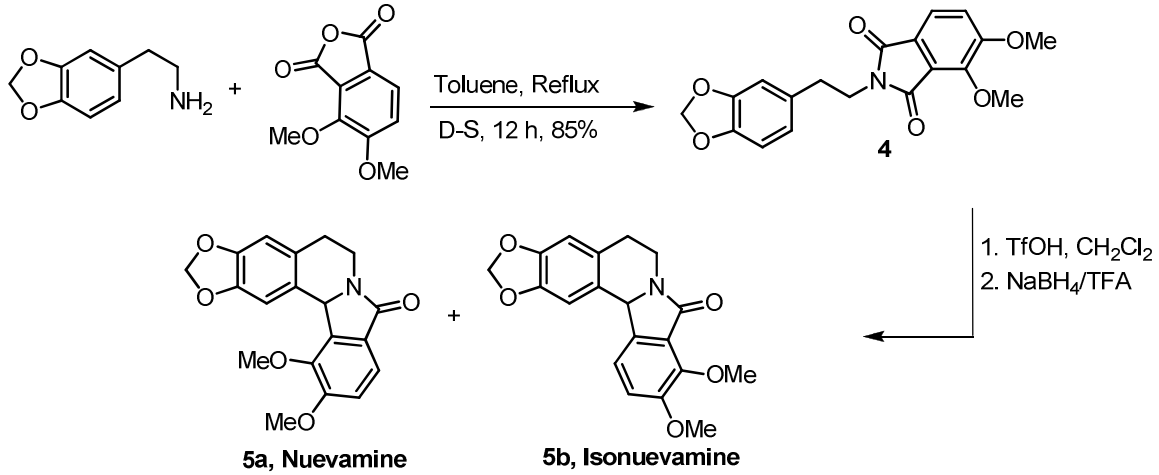
2,3-dihydroxy-5,6-dihydroisoindolo[1,2-a]isoquinolin-8(12bH)-one (3l)



110 mg. white solid, 77% yield; m.p. 225-227 ºC; IR (KBr, cm^{-1}): 3493, 1651, 1626, 1455, 1294, 1270, 234, 731; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.95 (s, 1H), 8.87 (s, 1H), 7.94 (d, *J* = 7.6 Hz, 1H), 7.71-7.66 (m, 2H), 7.52 (t, *J* = 7.6 Hz, 1H), 7.08 (s, 1H), 6.55 (s, 1H), 5.67 (s, 1H), 4.19-4.13 (m, 1H), 3.34-3.31 (m, 1H), 2.71-2.66 (m, 2H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 166.6, 145.1, 144.4, 143.9, 131.9, 131.5, 128.2, 124.6, 123.9, 122.8, 115.6, 112.6, 58.1, 37.8, 27.9; MALDI-MS (*m/z*): Calculated for $C_{16}H_{13}NO_3$ (M+H): 268.0974, Found (M+H): 268.0954.

Synthesis of Nuevamine:

Synthesis of Imide 4: Suspension of phthalic anhydride (1 mmol) in toluene in an oven dried round bottom flask fitted with Dean-Stark apparatus was heated to reflux until complete dissolution of the 3,4-dimethoxy phthalic anhydride and no additional water was removed. To this solution was added homopiperonyl amine (1.2 mmol) and refluxing was continued until the water evolution was completed (12 h). Reaction mixture was concentrated under reduced pressure to give a residue which was purified through column chromatography to give 2-(3,4-methylenedioxyphenyl)ethyl)-4,5-dimethoxyisoindoline-1,3-dione **4** in pure form.



Imide 4: 302 mg. white solid, 85% yield; m.p. 149 °C (*Lit.*[11] 155-157 ºC); IR (KBr, cm^{-1}): 2994, 2937, 2844, 1767, 1708, 1606, 1496, 1442, 1391, 1345, 1267, 1043, 926; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.52 (d, *J* = 8.0 Hz, 1H), 7.1 (d, *J* = 8.0 Hz, 1H), 6.74 (d, *J* = 1.3 Hz, 1H), 6.72-6.66 (m, 2H), 5.91 (s, 2H), 4.13 (s, 3H), 3.95 (s, 3H), 3.84-3.80 (m, 2H), 2.89-2.85 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.46, 166.1, 157.6, 147.6, 147.1, 146.2, 131.9, 124.6, 121.8, 121.7, 119.3, 115.7, 109.2, 108.2, 100.8, 62.5, 56.5, 39.4, 34.2.

Synthesis of Nuevamine 5a: An oven dried two neck round bottom flask bearing septum in side arm was cooled to room temperature under a steady stream of nitrogen gas flow. The flask was charged with stirring bar, imide **4** (71 mg, 0.2 mmol) and dry dichloromethane (10 ml) and cooled down to given temperature. To this solution was added TfOH (0.1 ml, 1 mmol) with stirring. After reported time the contents were brought to room temperature and $NaBH_4$ (1 mmol) was added followed by TFA (0.5 ml) and the solution was stirred until color disappears (additional $NaBH_4$ and TFA was used if color persists for long time). This mixture was evaporated to dryness under reduced pressure. The solid residue was dissolved in dichloromethane (20 ml) and the insoluble material was filtered off, the organic layer was dried over anhydrous Na_2SO_4 and filtered. The solvent was evaporated under vacuum and the crude product was purified through silica gel column chromatography using ethyl acetate/hexane (50:50) as eluent to give nuevamine **5a** and its regioisomer Isonuevamine **5b** as mixture.

S. No.	Temperature (˚C)	Time (h)	% of Nuevamine **5a**[a]	Yield of (**5a**+**5b**)[b]
1	0	0.5	52	86
2	-20	1	71	--[c]
3	-40	12	80	--[c]
4	-60	20	85	--[c]
5	-78	36	88	92

[a] % of regioisomeric composition was ascertained by ^{1}H-NMR data

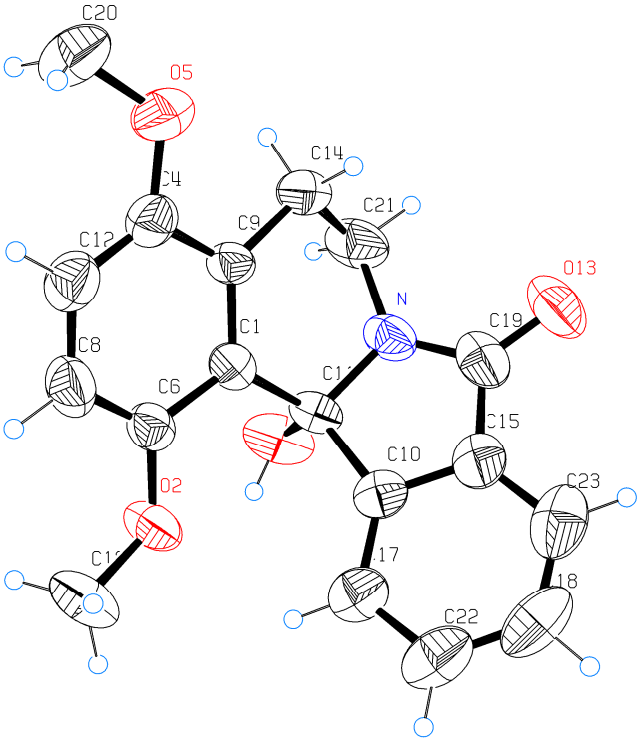
[b] Isolated Yield; [c] Yield was not calculated.

Mixture of 10,11-Dimethoxy-5,11b-dihydro-6H-1,3-dioxa-6a-aza-indeno[5,6-c]fluoren-7-one (5a) and 8,9-Dimethoxy-5,11b-dihydro-6H-1,3-dioxa-6a-aza-indeno[5,6-c]fluoren-7-one (5b)

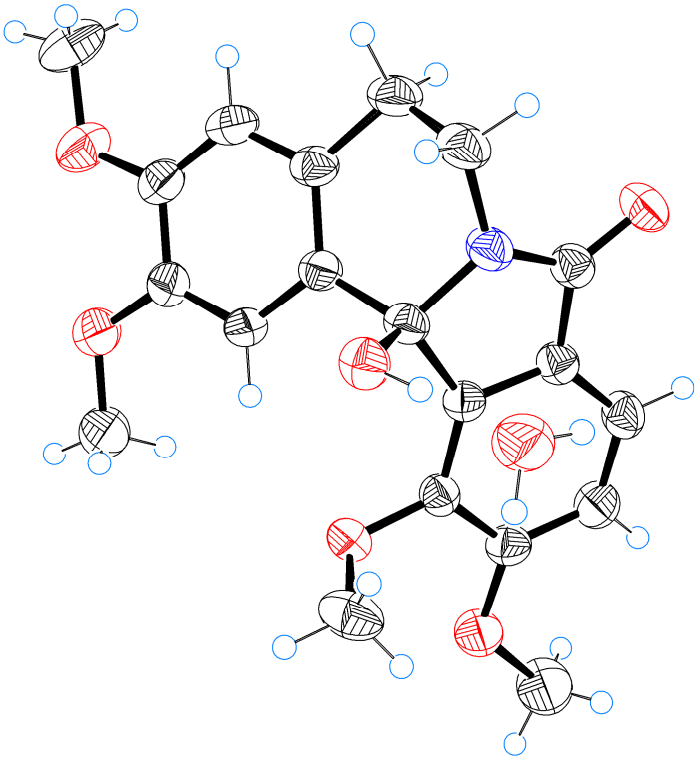
59 mg (86% yield) and 63 mg (92% yield), IR (KBr, cm^{-1}): 2972, 2940, 2841, 1681, 1620, 1446, 1408, 1273, 1220, 1035; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.58 (d, *J* = 8.0 Hz, 1H), 7.43 (dd, *J* = 0.8, 8.4 Hz, 1H), 7.32 (s, 1H), 7.14 (d, *J* = 8.0 Hz, 1H), 7.07 (d, J = 8.4, 1H), 7.03 (s, 1H), 6.66 (s, 1H), 6.65 (s, 1H), 5.95 (d, *J* = 1.2 Hz, 1H), 5.92 (d, *J* = 1.2 Hz, 1H), 5.89 (d, *J* = 1.2 Hz, 1H), 5.86 (d, *J* = 1.2 Hz, 1H), 5.63 (s, 1H), 5.45 (s, 1H), 4.34-4.28 (m, 1H), 4.07 (s, 3H), 4.0-4.09 (m, 1H), 3.99 (s, 3H), 3.97 (s, 3H), 3.90 (s, 3H), 3.59-3.53 (m, 1H), 3.42-3.36 (m, 1H), 3.05-2.94 (m, 2H), 2.90-2.82 (m, 1H), 2.79-2.75 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.6, 166.2, 155.5, 152.8, 146.7, 146.5, 146.4, 144.3, 137.6, 136.1, 128.8, 128.4, 128.3, 127.8, 126.6, 125.1, 119.7, 118.4, 116.2, 113.2, 109.0, 108.4, 107.5, 105.5, 101.1, 100.9, 62.5, 60.5, 58.4, 58.0, 56.7, 56.3, 38.8, 38.3, 29.3, 28.9.

Crystal Structure of the compounds 2h, 2m, 2j and 2k[12]

2h, CCDC 828126

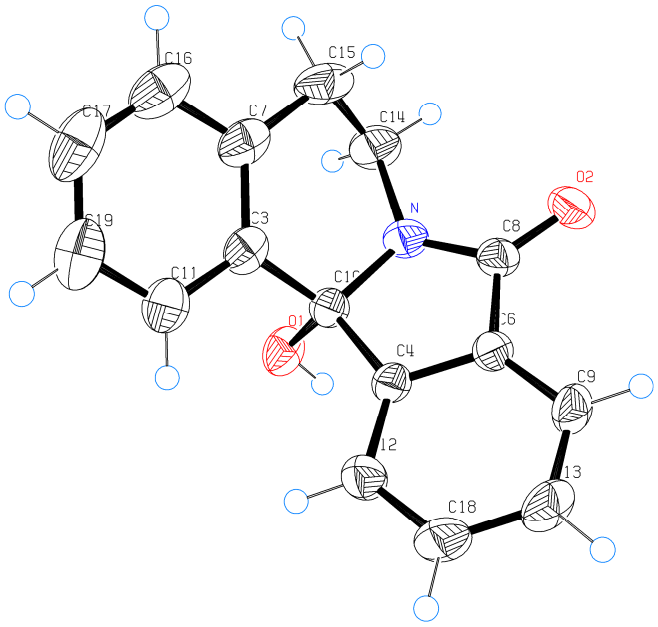


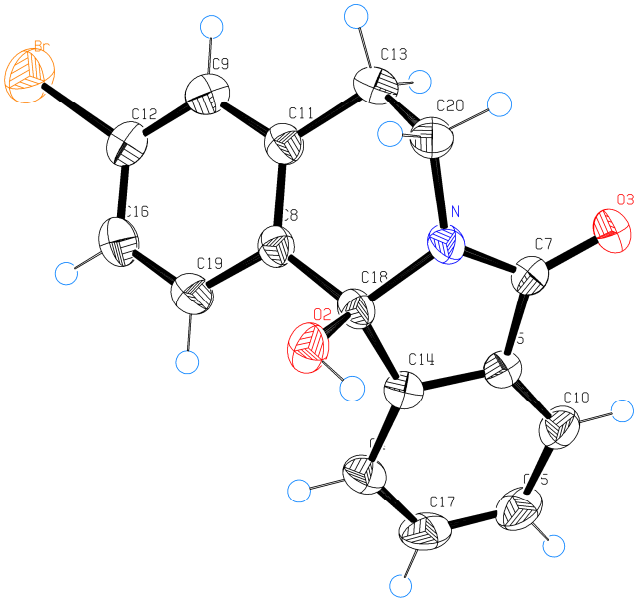
2m, CCDC 828128



2j, CCDC 828125

2k, CCDC 828124





References:

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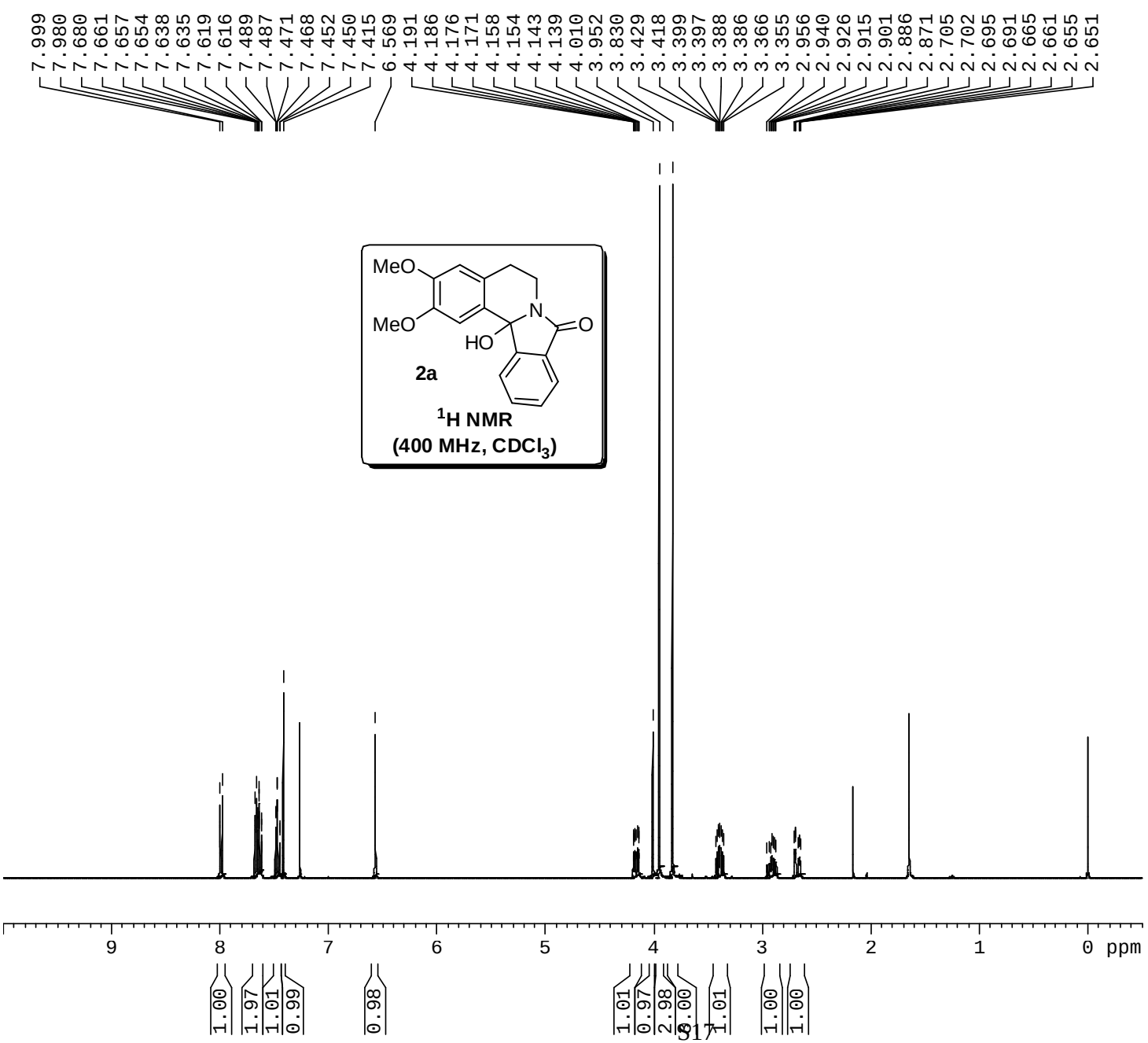
12. CCDC No 828124-828126, 828128 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data_request/cif.

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PROCNO                1

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TD                32768
SOLVENT           CDCl3
NS                   32
DS                    2
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FIDRES         0.250967 Hz
AQ            1.9923444 sec
RG                  362
DW               60.800 usec
DE                6.00 usec
TE               296.3 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
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PL1               -0.90 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
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WDW                  EM
SSB                   0
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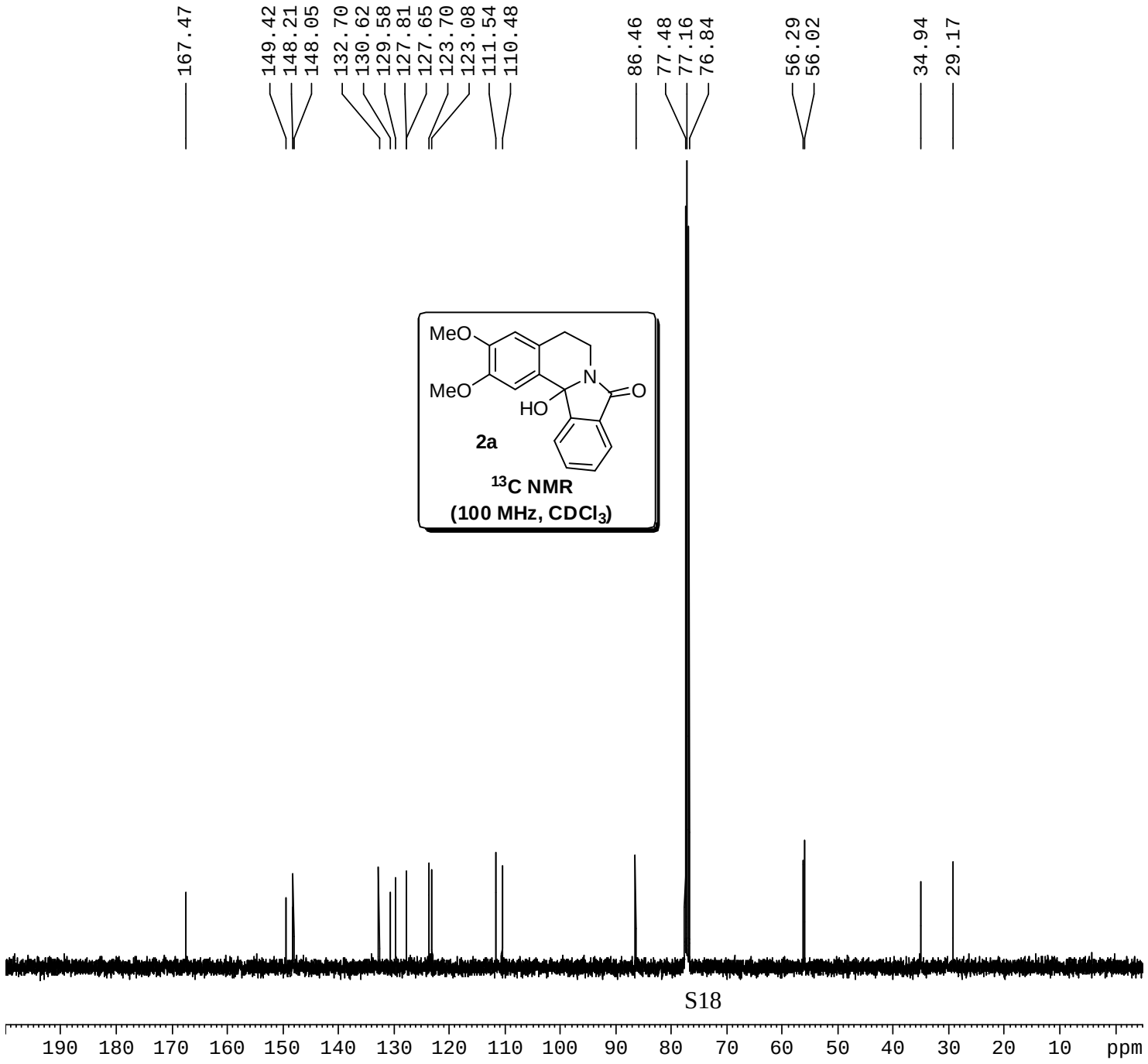


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PROCNO               1

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TD               65536
SOLVENT          CDCl3
NS                 256
DS                   4
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ           1.3631988 sec
RG                  57
DW               20.800 usec
DE                6.00 usec
TE                298.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                  1

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P1                9.50 usec
PL1               -0.60 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            90.00 usec
PL12              15.60 dB
PL13              15.60 dB
PL2               -0.90 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
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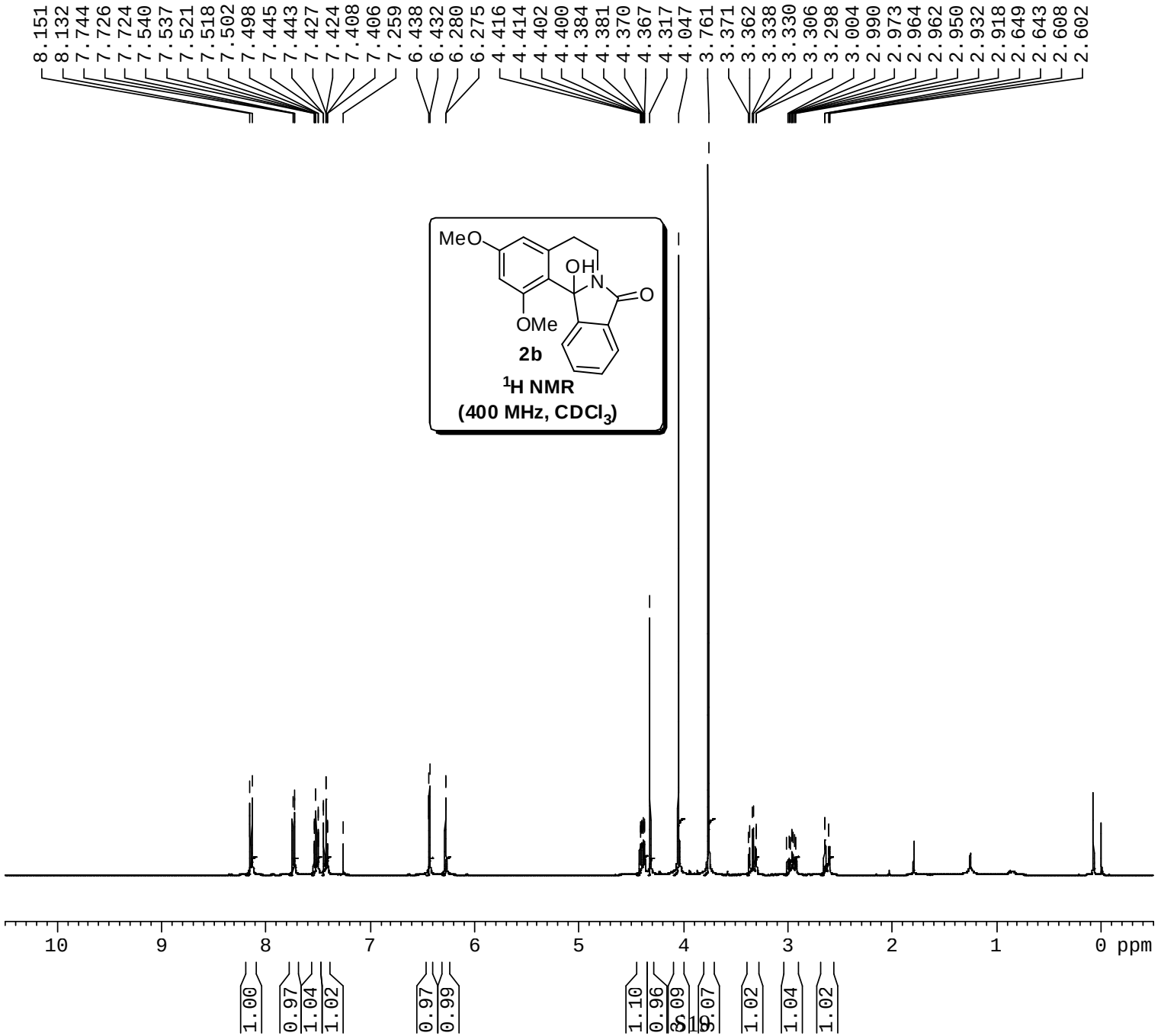


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TD                32768
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            8223.685 Hz
FIDRES         0.250967 Hz
AQ            1.9923444 sec
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TE               294.8 K
D1          2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.00 usec
PL1               -0.90 dB
SFO1        400.1324710 MHz

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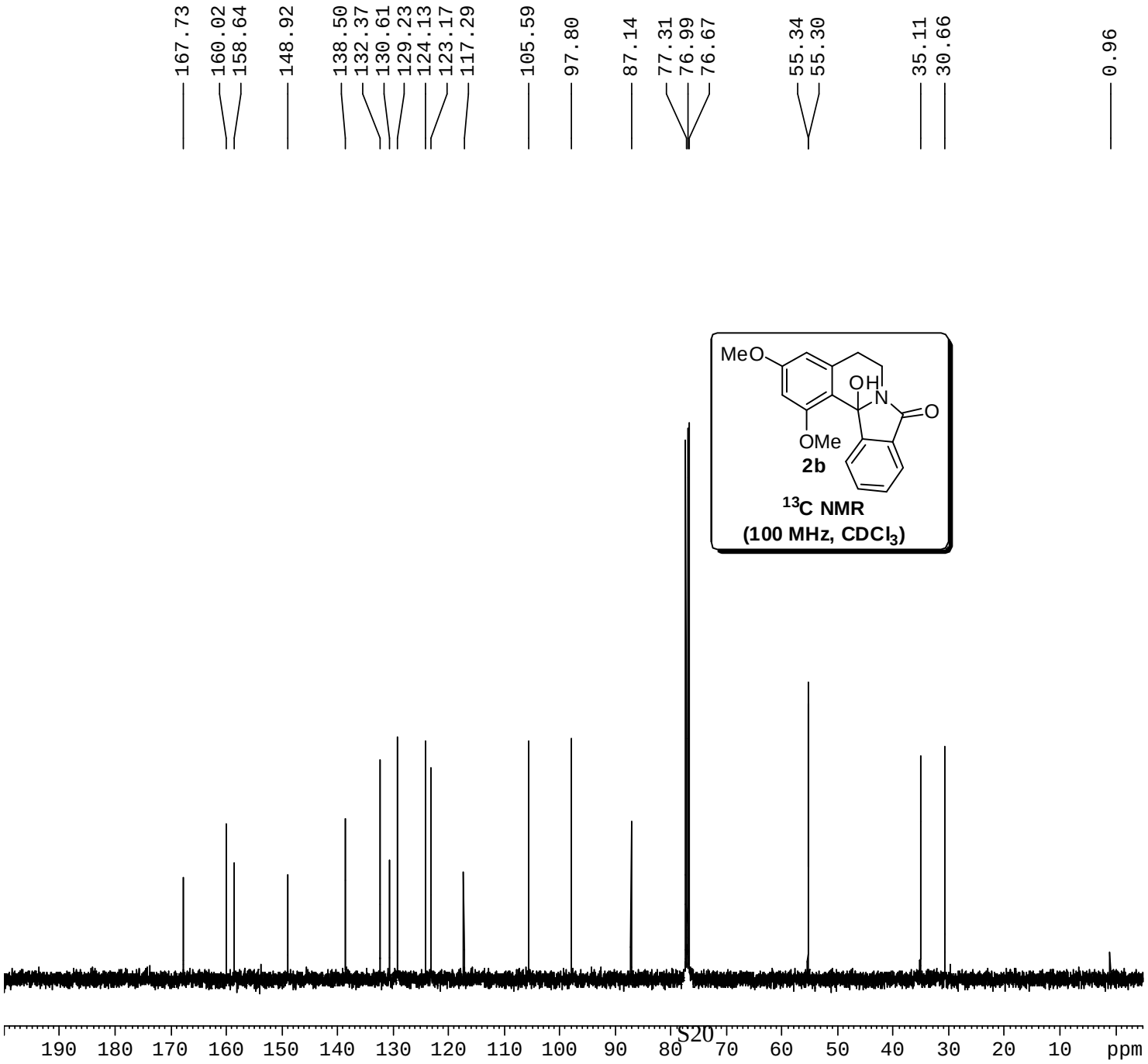


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PROCNO                1

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PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  256
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                  575
DW                20.800 usec
DE                 6.00 usec
TE                295.4 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.50 usec
PL1               -0.60 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PL12              15.60 dB
PL13              15.60 dB
PL2               -0.90 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
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SSB                   0
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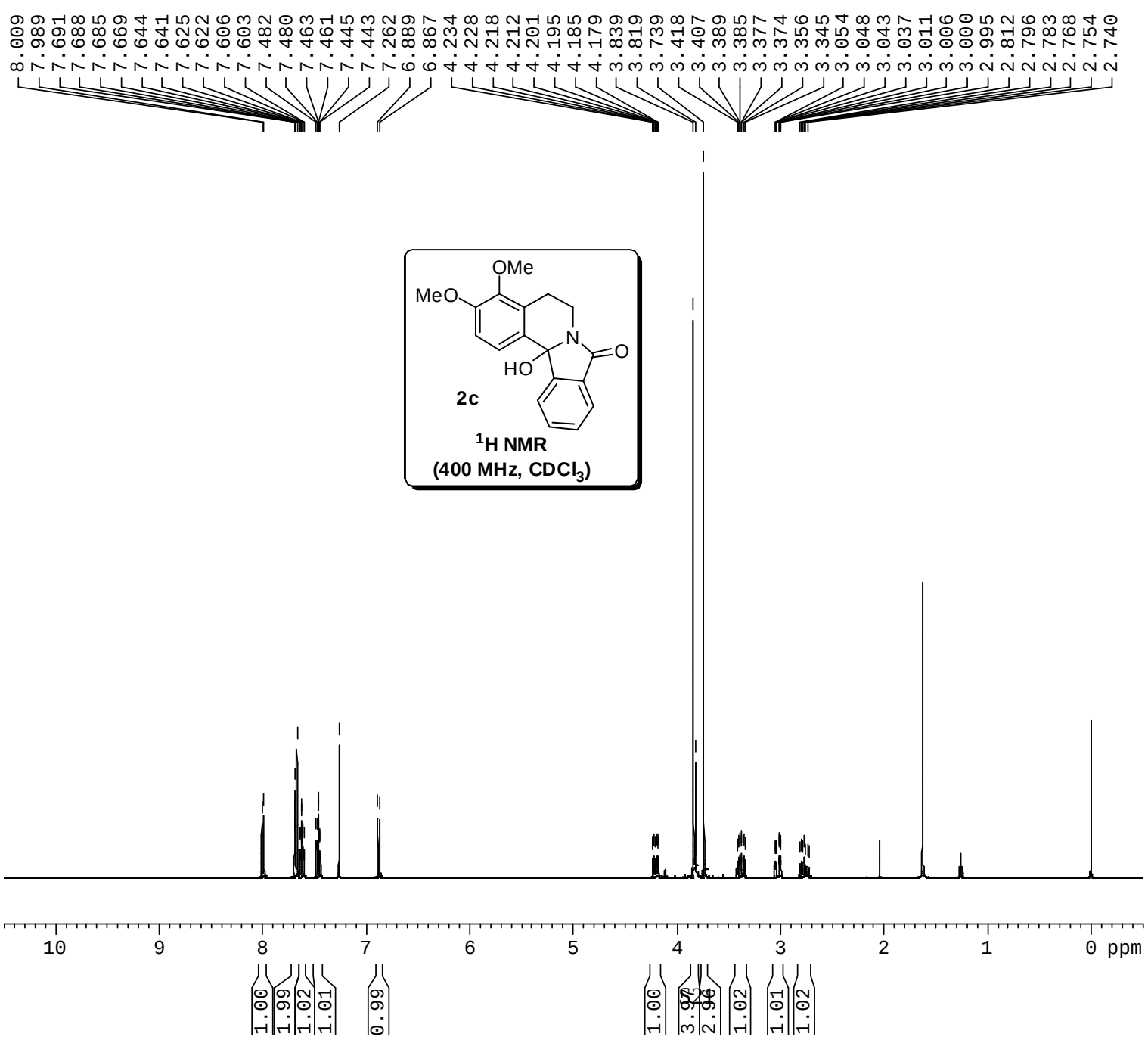


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PULPROG             zg30
TD                32768
SOLVENT           CDCl3
NS                    4
DS                    2
SWH            8223.685 Hz
FIDRES         0.250967 Hz
AQ            1.9923444 sec
RG                  456
DW               60.800 usec
DE                6.00 usec
TE               296.0 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.00 usec
PL1               -0.90 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
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SF          400.1300040 MHz
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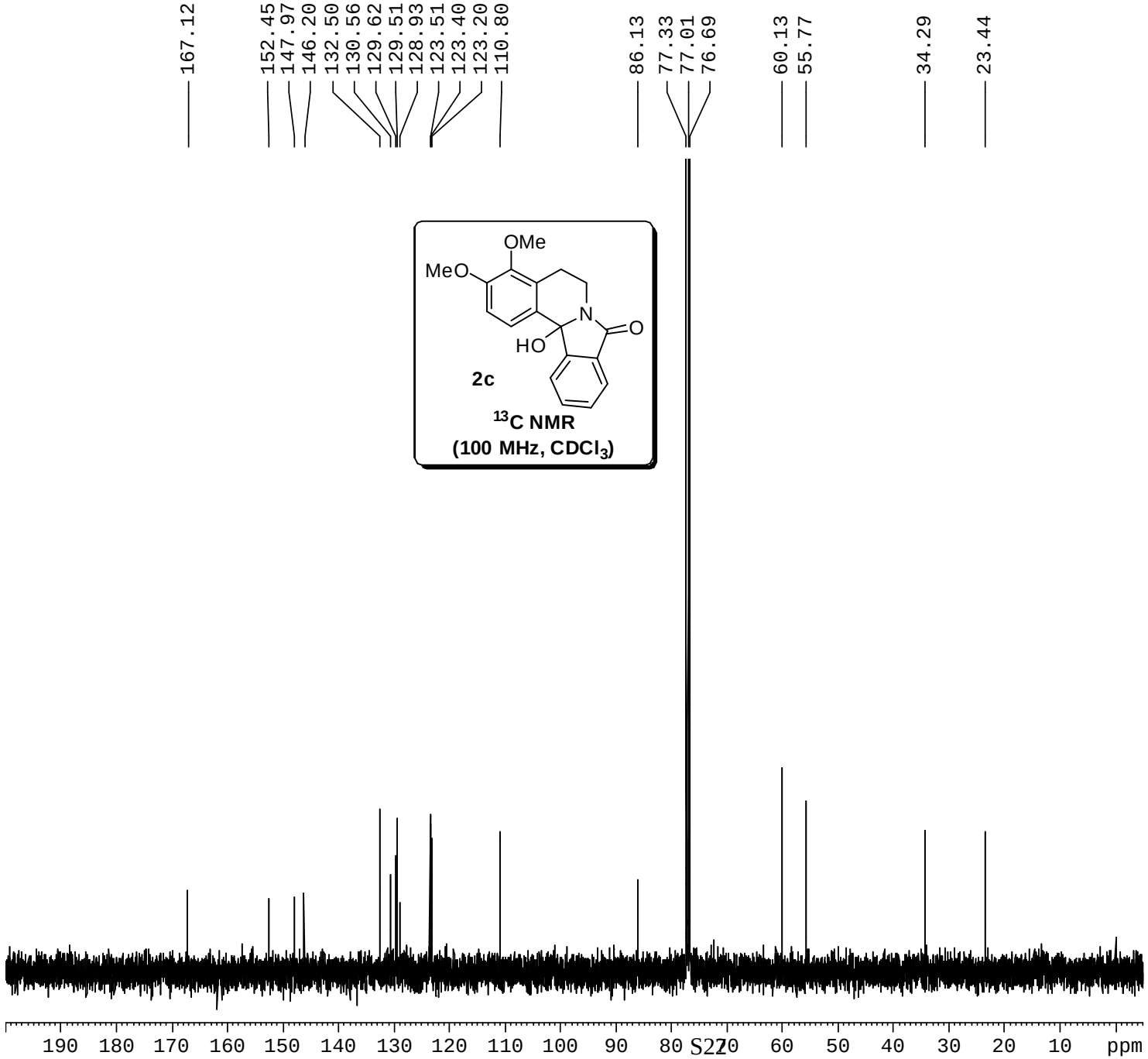


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PROCNO                 1

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INSTRUM            spect
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TD                65536
SOLVENT           CDCl3
NS                  256
DS                    2
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631988 sec
RG                  575
DW               20.800 usec
DE                6.00 usec
TE               296.7 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.50 usec
PL1               -0.60 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PL12              15.60 dB
PL13              15.60 dB
PL2               -0.90 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
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SF          100.6127690 MHz
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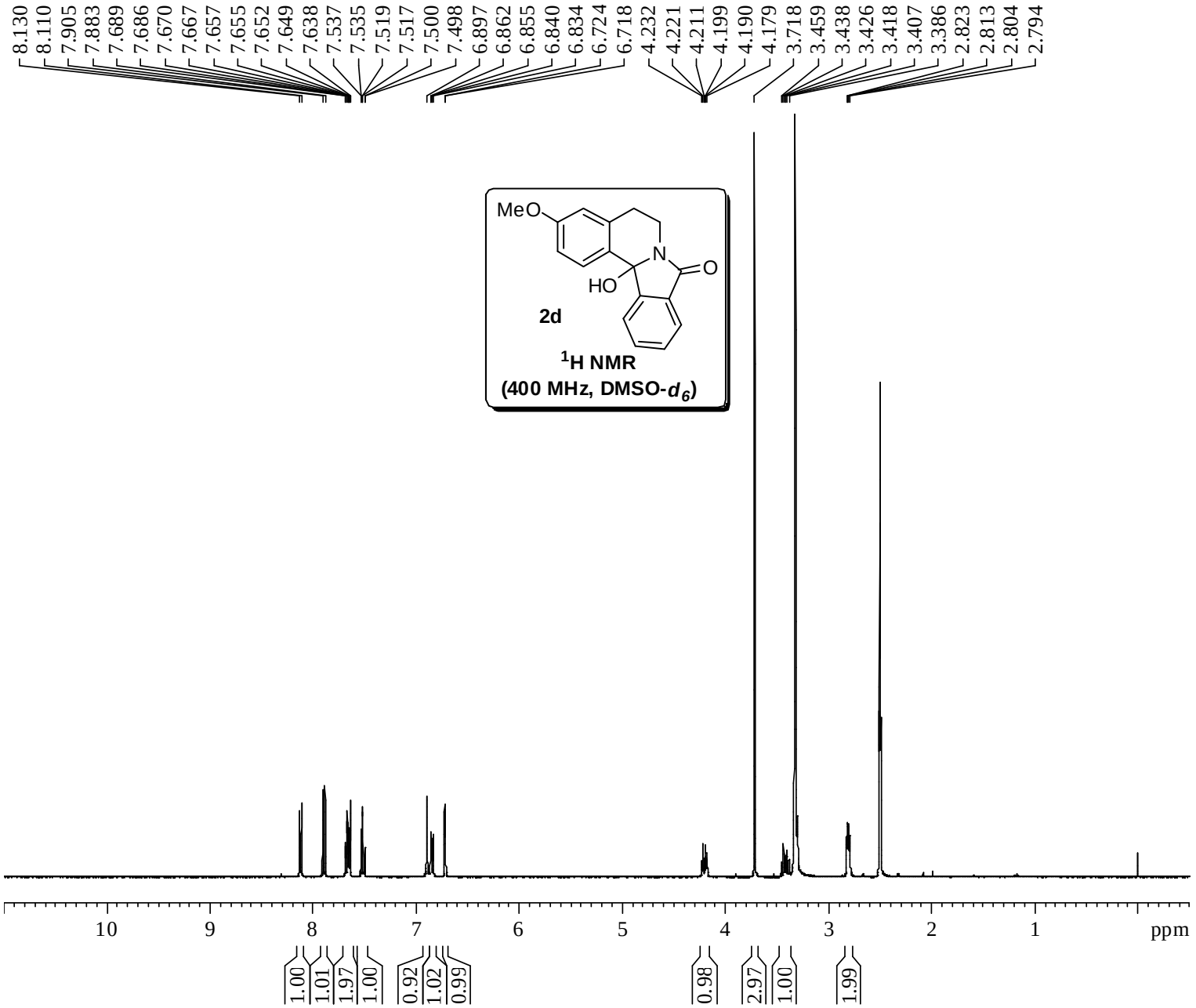


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PROCNO               1

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INSTRUM           spect
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PULPROG            zg30
TD                32768
SOLVENT            DMSO
NS                    16
DS                     2
SWH            8223.685 Hz
FIDRES         0.250967 Hz
AQ            1.9923444 sec
RG                   456
DW                60.800 usec
DE                 6.00 usec
TE                296.9 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.00 usec
PL1               -0.90 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                32768
SF          400.1299954 MHz
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LB                0.30 Hz
GB                    0
PC                 1.00
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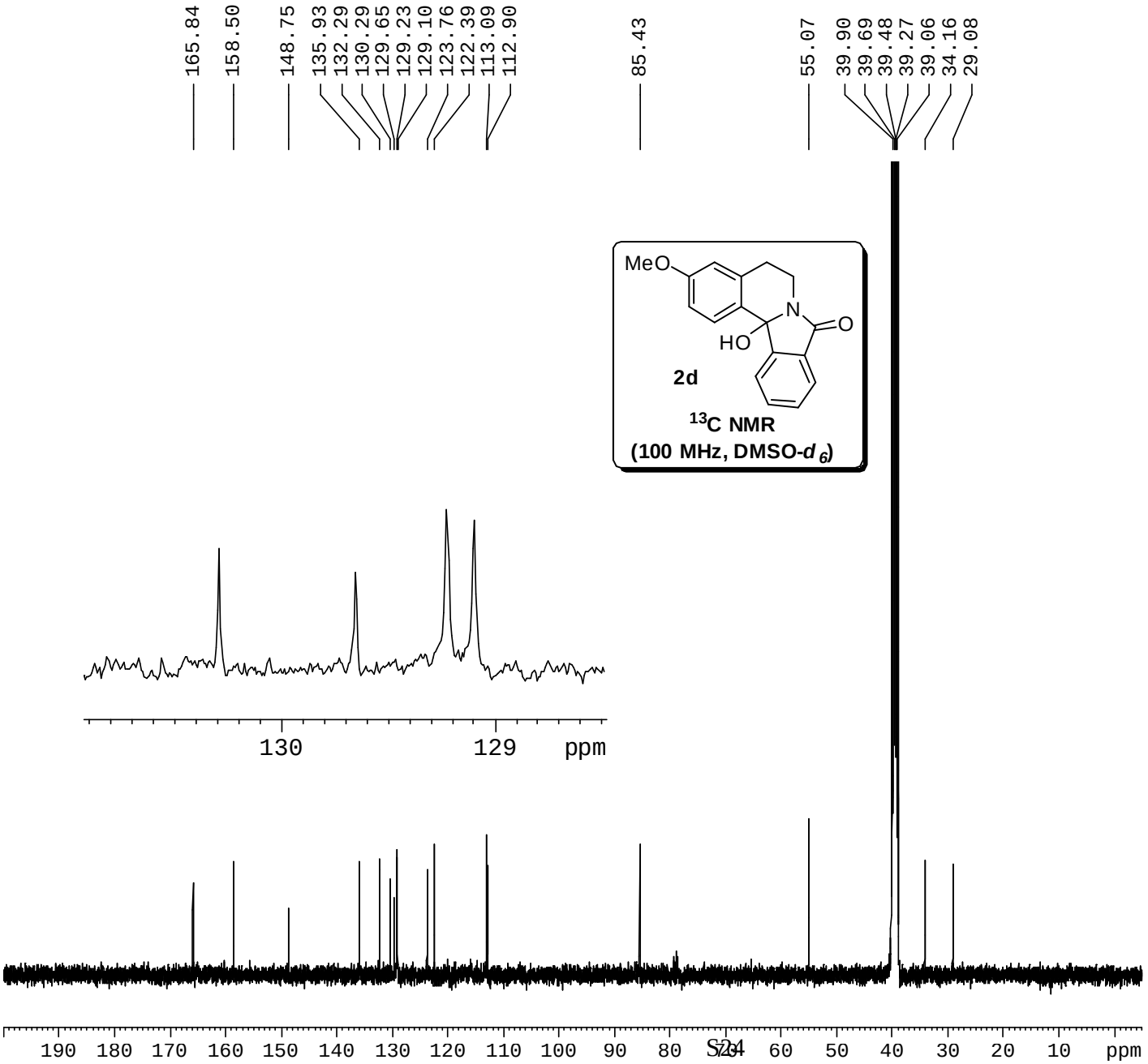


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Current Data Parameters
NAME       JS-3-PTH-CYCL
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
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INSTRUM          spect
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PULPROG         zgpg30
TD               65536
SOLVENT           DMSO
NS                7000
DS                   4
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                2050
DW              20.800 usec
DE               6.00 usec
TE               297.7 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                13C
P1                9.50 usec
PL1              -0.60 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            90.00 usec
PL12             15.60 dB
PL13             15.60 dB
PL2              -0.90 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
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LB               1.00 Hz
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PC               1.40
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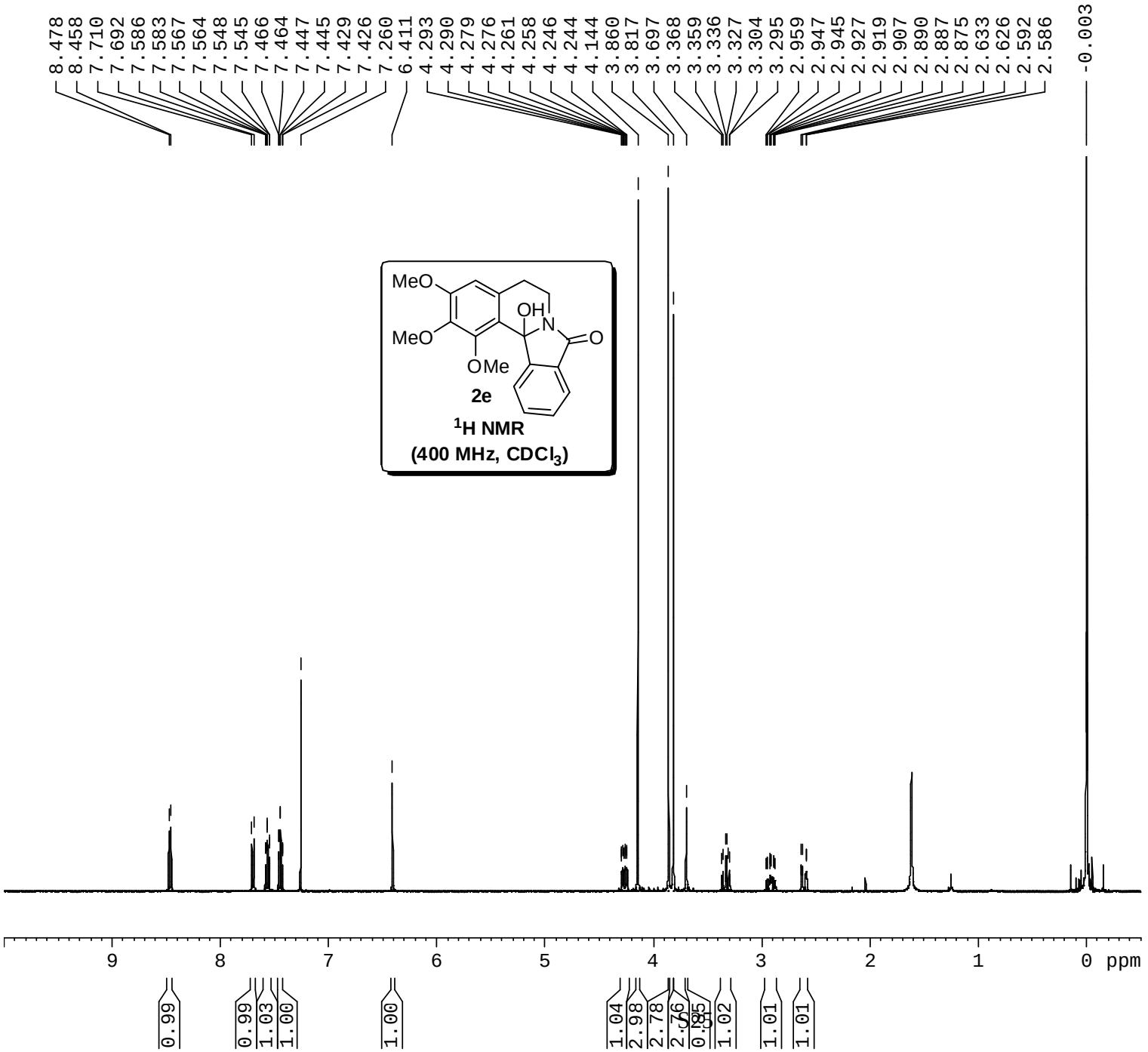

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PROCNO 1

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INSTRUM spect
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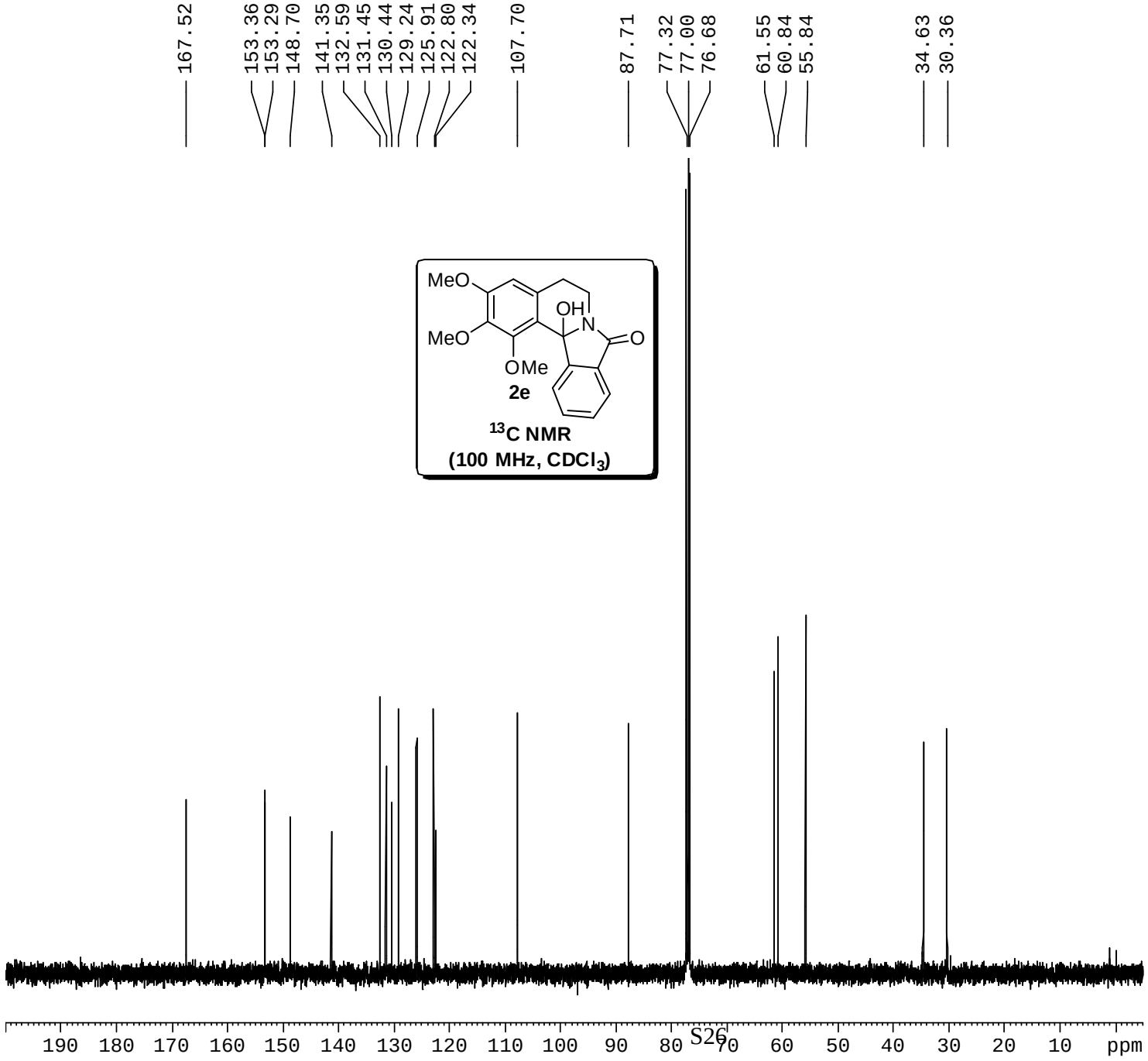
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 4
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 287
DW 60.800 usec
DE 6.00 usec
TE 293.6 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -0.90 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300048 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

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PROCNO                1

F2 - Acquisition Parameters
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TD                65536
SOLVENT           CDCl3
NS                  256
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631988 sec
RG                  645
DW               20.800 usec
DE                6.00 usec
TE               295.7 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.50 usec
PL1               -0.60 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            90.00 usec
PL12             15.60 dB
PL13             15.60 dB
PL2              -0.90 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
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SF          100.6127728 MHz
WDW                   EM
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LB                 1.00 Hz
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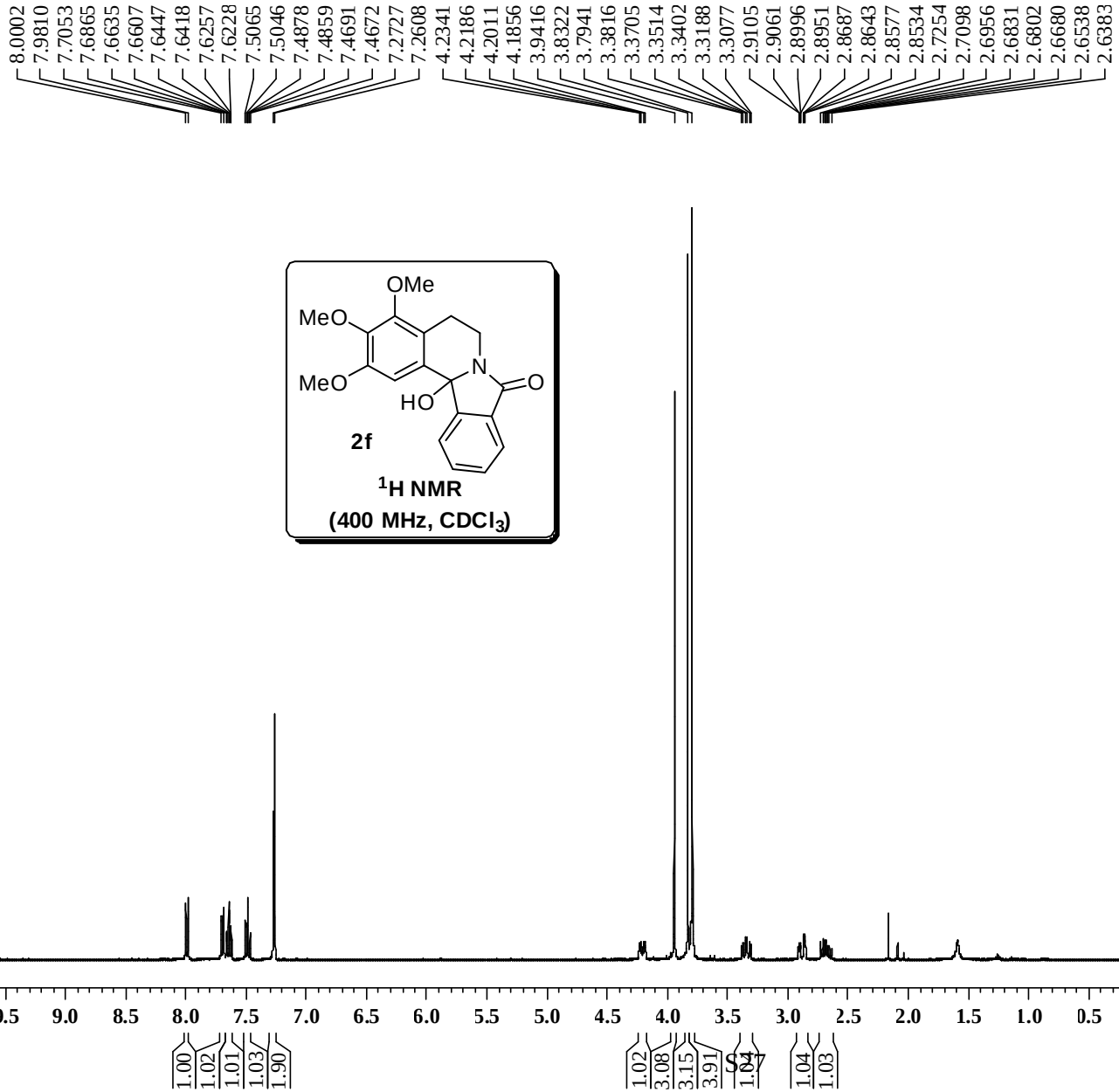

BRUKER

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Current Data Parameters
NAME       JS-III-233-1
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20100927
Time              10.16
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                  406
DW               60.800 usec
DE                6.00 usec
TE                297.7 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PL1               -0.90 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                32768
SF          400.1300035 MHz
WDW                  EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                1.00
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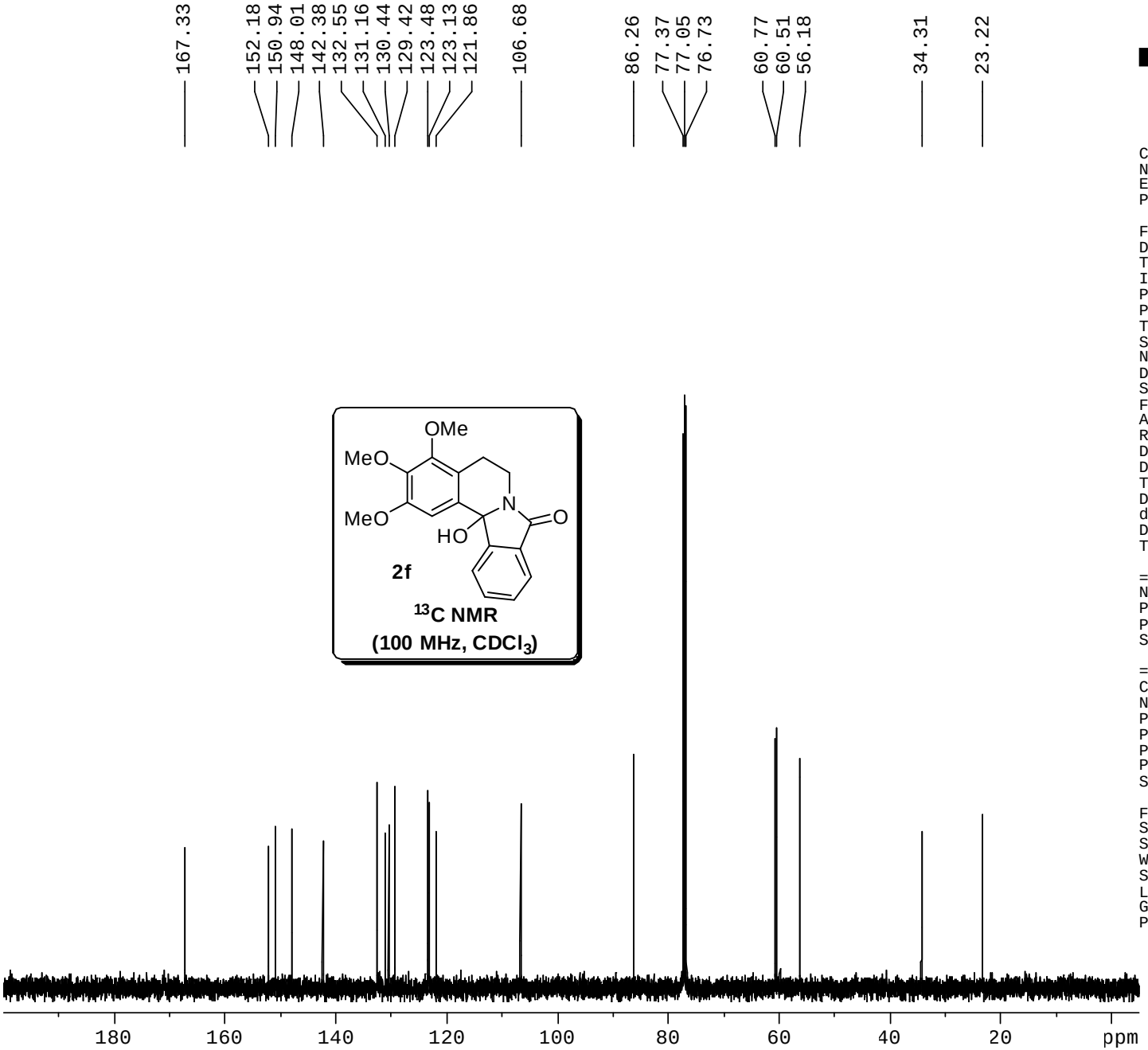


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Current Data Parameters
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PROCNO                1

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Time              13.11
INSTRUM           spect
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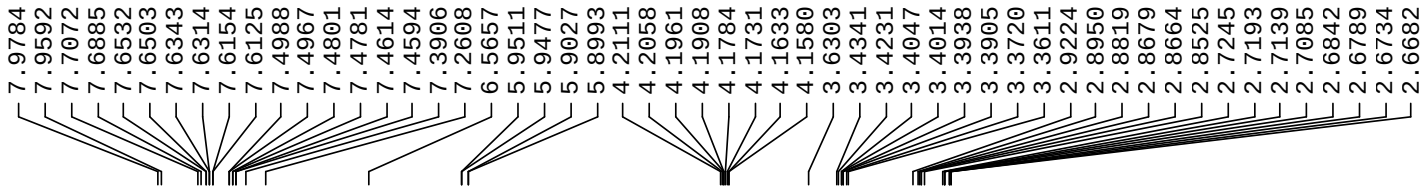
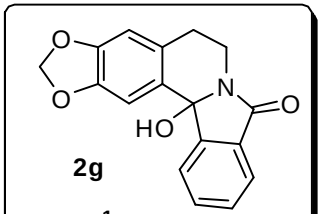
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                   77
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631988 sec
RG                   57
DW               20.800 usec
DE                6.00 usec
TE               299.5 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.50 usec
PL1                -0.60 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2              90.00 usec
PL12               15.60 dB
PL13               15.60 dB
PL2                -0.90 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

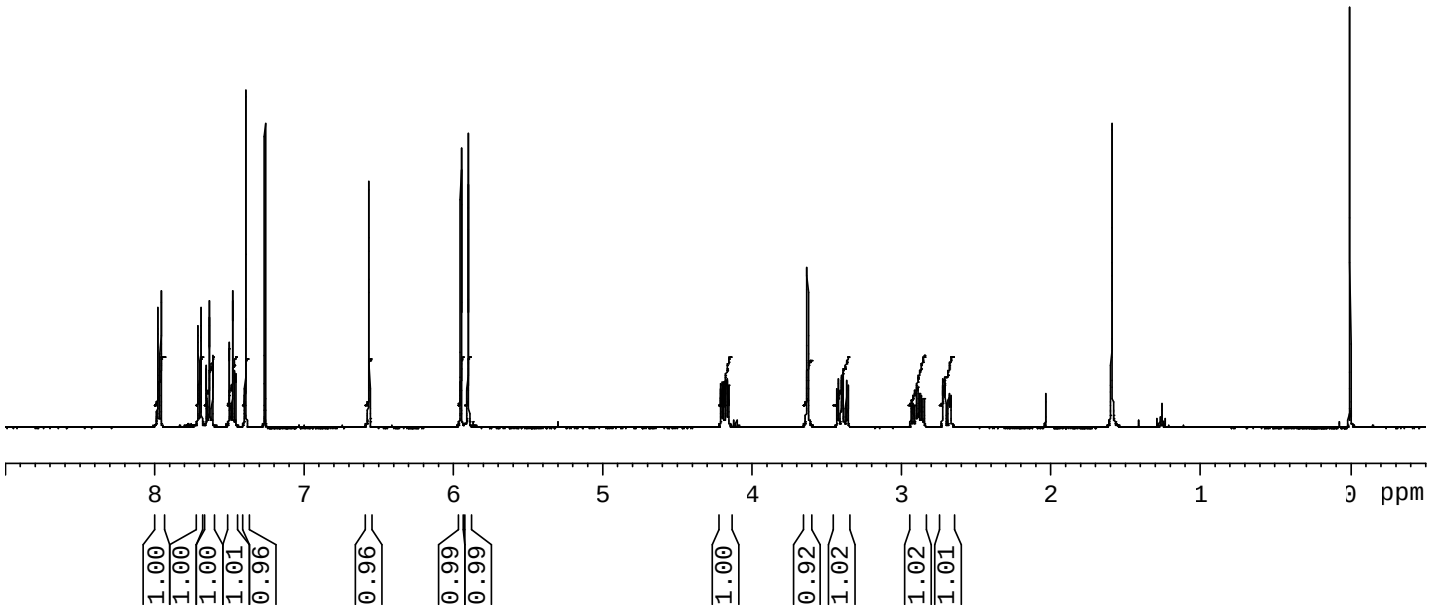




2g

^{1}H NMR

(400 MHz, $CDCl_3$)

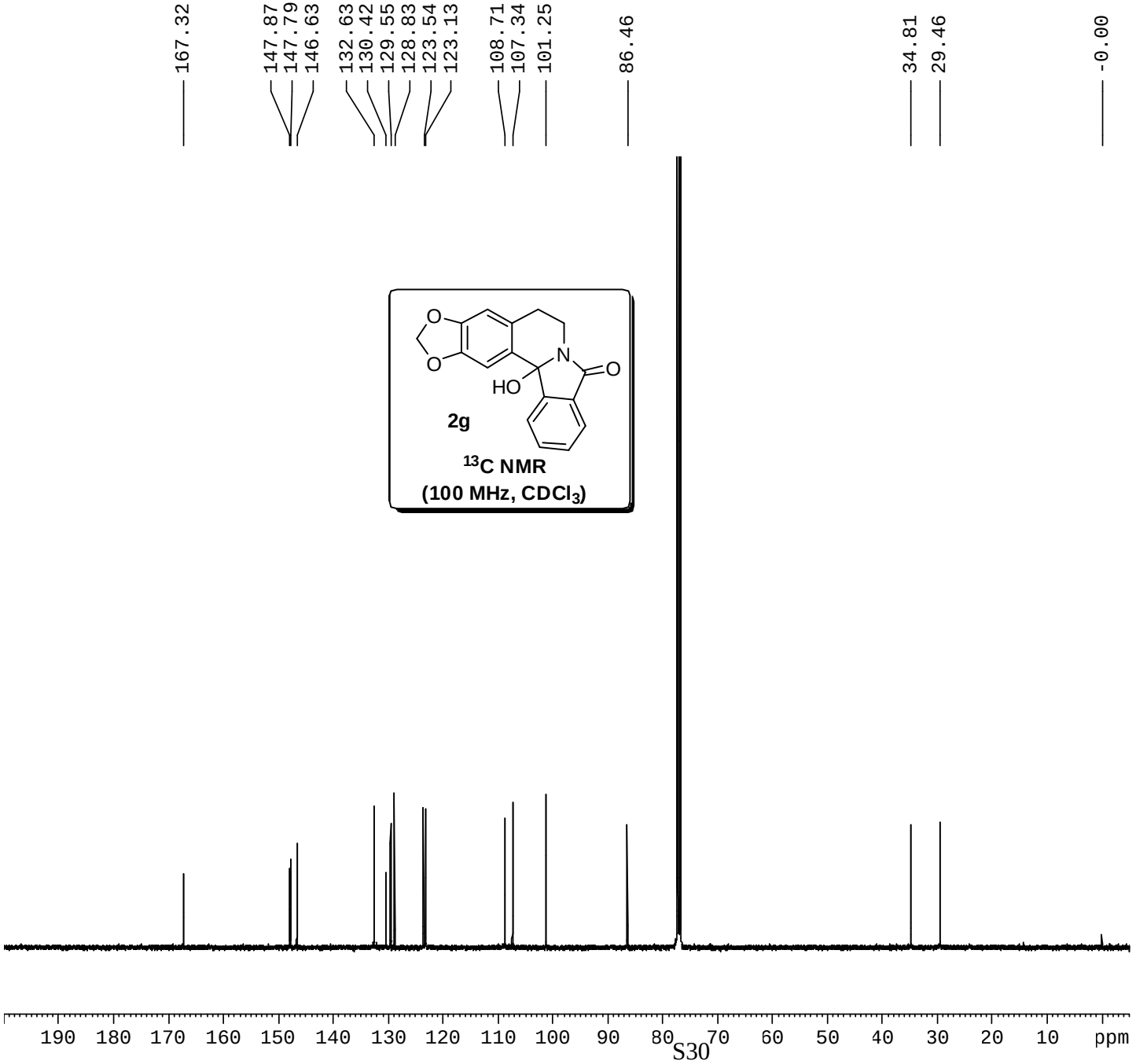



```
Current Data Parameters
NAME       JS-III-249-1
EXPNO                 6
PROCNO                1

F2 - Acquisition Parameters
Date_          20110205
Time              11.02
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ             3.9846387 sec
RG                  512
DW                60.800 usec
DE                 6.00 usec
TE                296.4 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PL1               -0.90 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                32768
SF          400.1300036 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



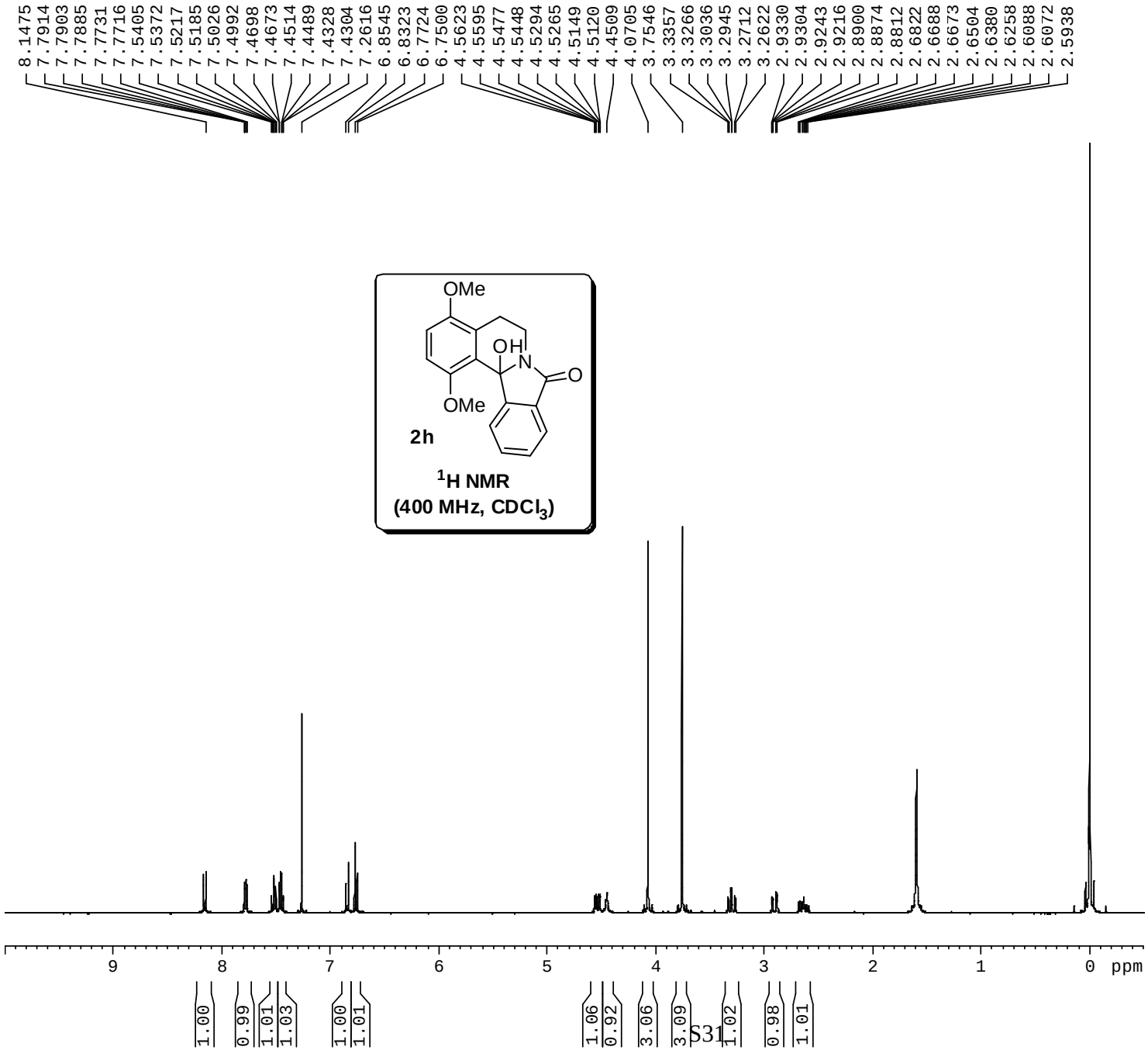

```
Current Data Parameters
NAME       JS-II-249-1b
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20110205
Time              21.11
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                 8000
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ           1.3631988 sec
RG                   57
DW                20.800 usec
DE                 6.00 usec
TE                297.2 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.50 usec
PL1                -0.60 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              90.00 usec
PL12               15.60 dB
PL13               15.60 dB
PL2                -0.90 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127675 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

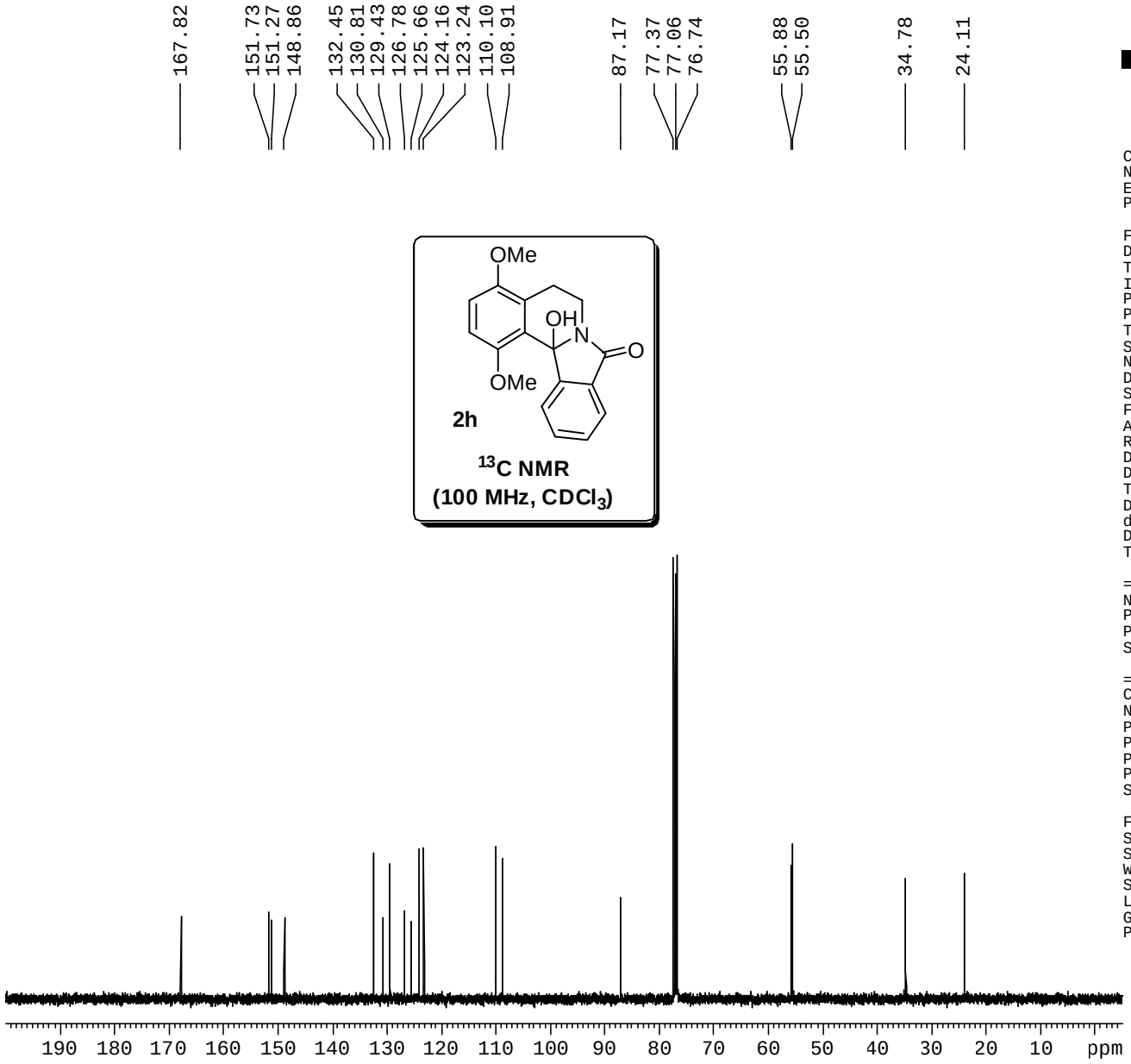



```
Current Data Parameters
NAME           JS-25C-1
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20110523
Time              15.50
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   27
DS                    2
SWH            8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                  322
DW               60.800 usec
DE                6.00 usec
TE                294.9 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.00 usec
PL1               -0.90 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                32768
SF          400.1300032 MHz
WDW                  EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                1.00
```



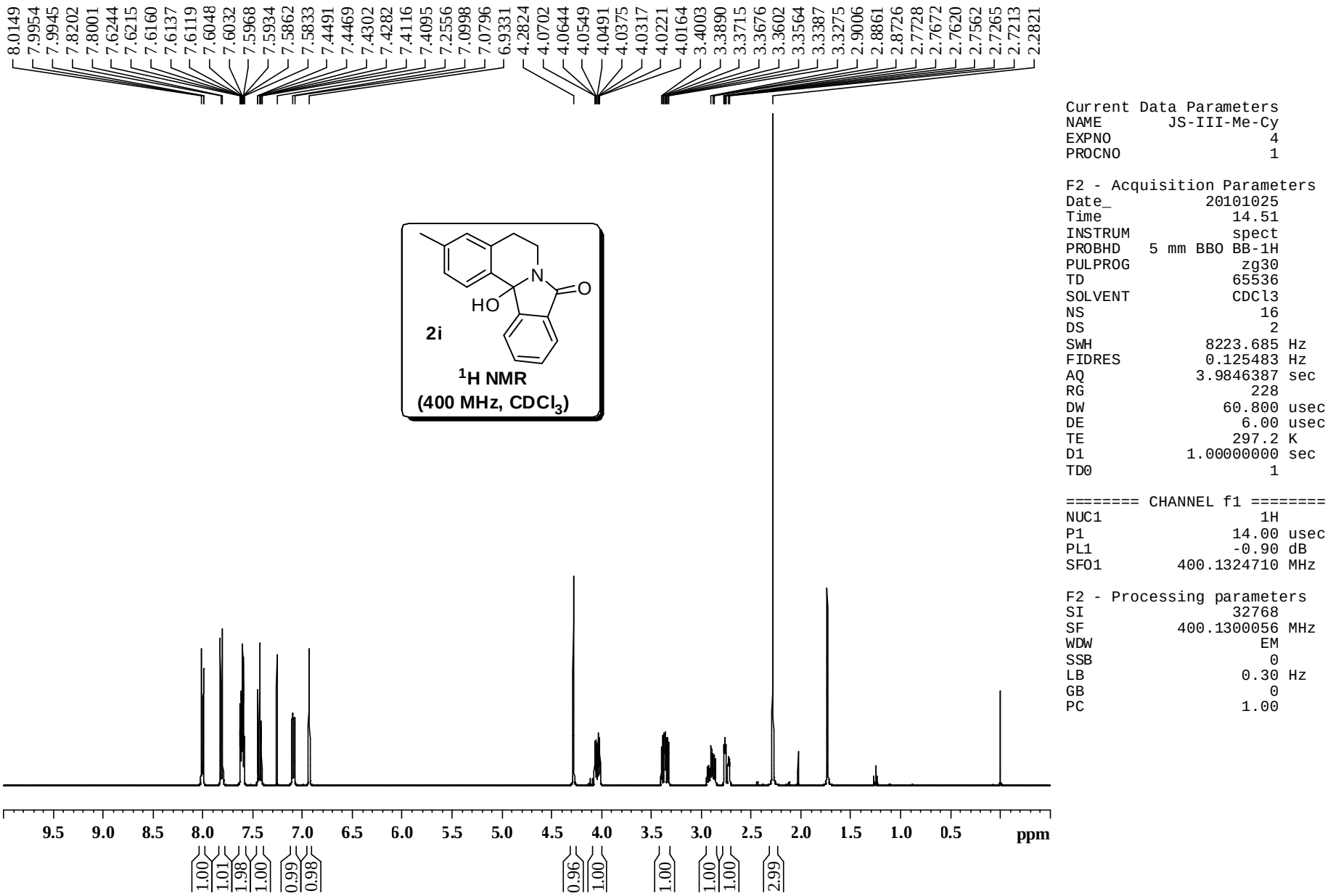

```
Current Data Parameters
NAME       JS-III-208-1
EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
Date_          20100825
Time              16.30
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  208
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631988 sec
RG                 50.8
DW                20.800 usec
DE                 6.00 usec
TE                297.3 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.50 usec
PL1                -0.60 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              90.00 usec
PL12               15.60 dB
PL13               15.60 dB
PL2                -0.90 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

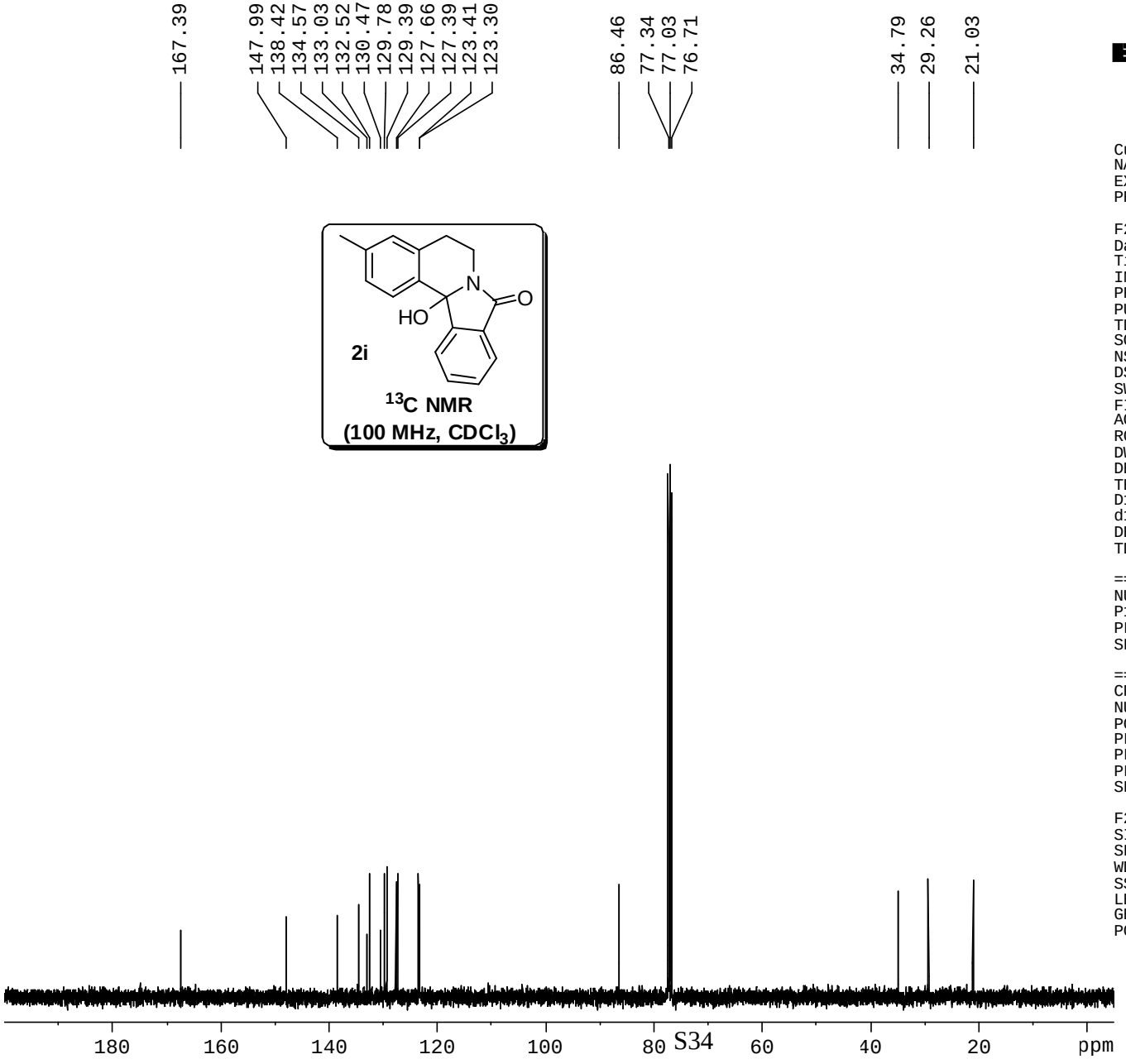



```
Current Data Parameters
NAME       JS-III-Me-Cy
EXPNO                 4
PROCNO                1

F2 - Acquisition Parameters
Date_          20101025
Time              14.51
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                  228
DW                60.800 usec
DE                 6.00 usec
TE                297.2 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.00 usec
PL1               -0.90 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                32768
SF          400.1300056 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



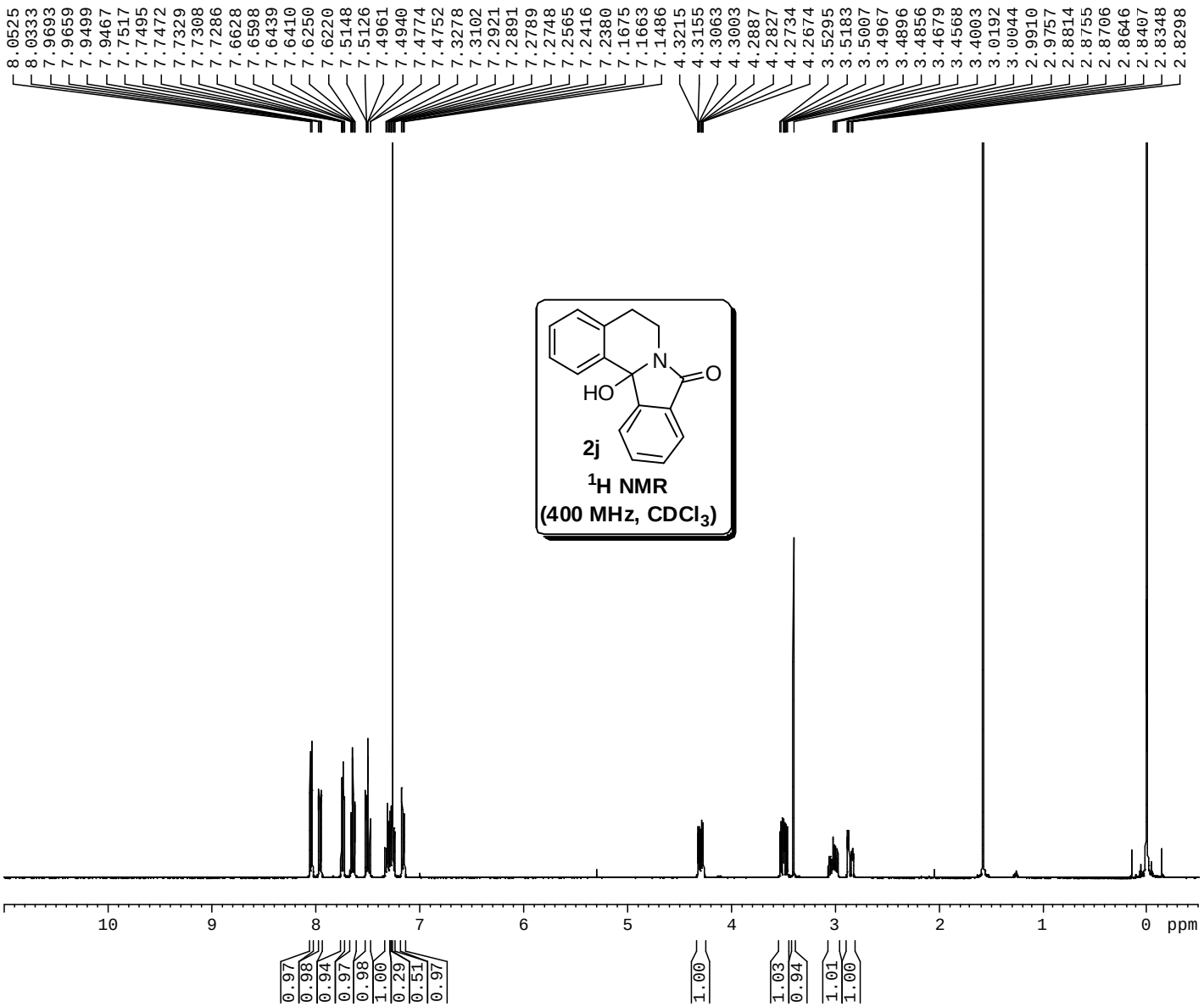

```
Current Data Parameters
NAME       JS-III-Me-Cy
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20101025
Time              14.46
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  126
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                 50.8
DW                20.800 usec
DE                 6.00 usec
TE                297.7 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.50 usec
PL1               -0.60 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PL12              15.60 dB
PL13              15.60 dB
PL2               -0.90 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

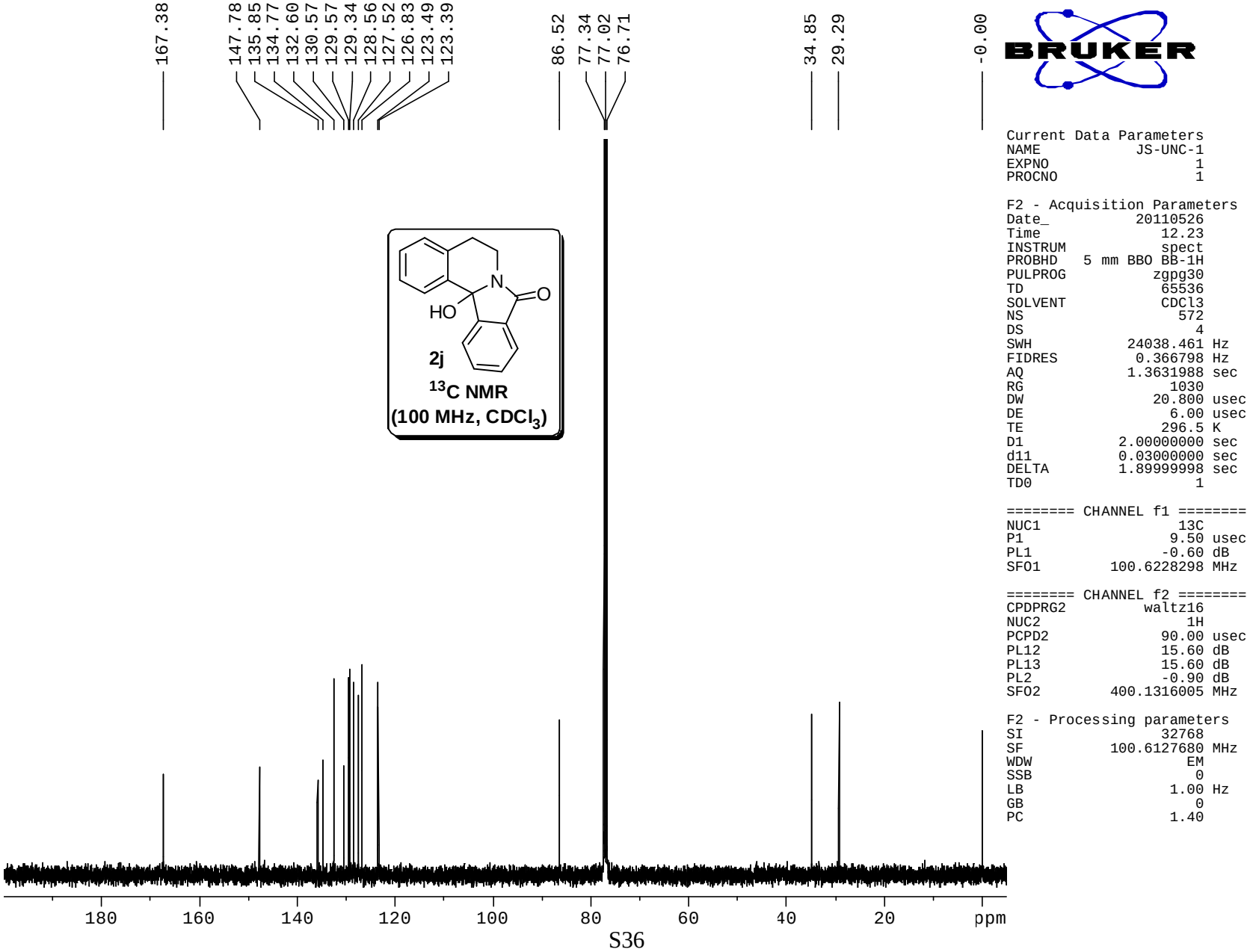



```
Current Data Parameters
NAME          JS-un-Cy-1
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20110516
Time               12.53
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9846387 sec
RG                   362
DW                60.800 usec
DE                 6.00 usec
TE                295.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.00 usec
PL1                -0.90 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF          400.1300038 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



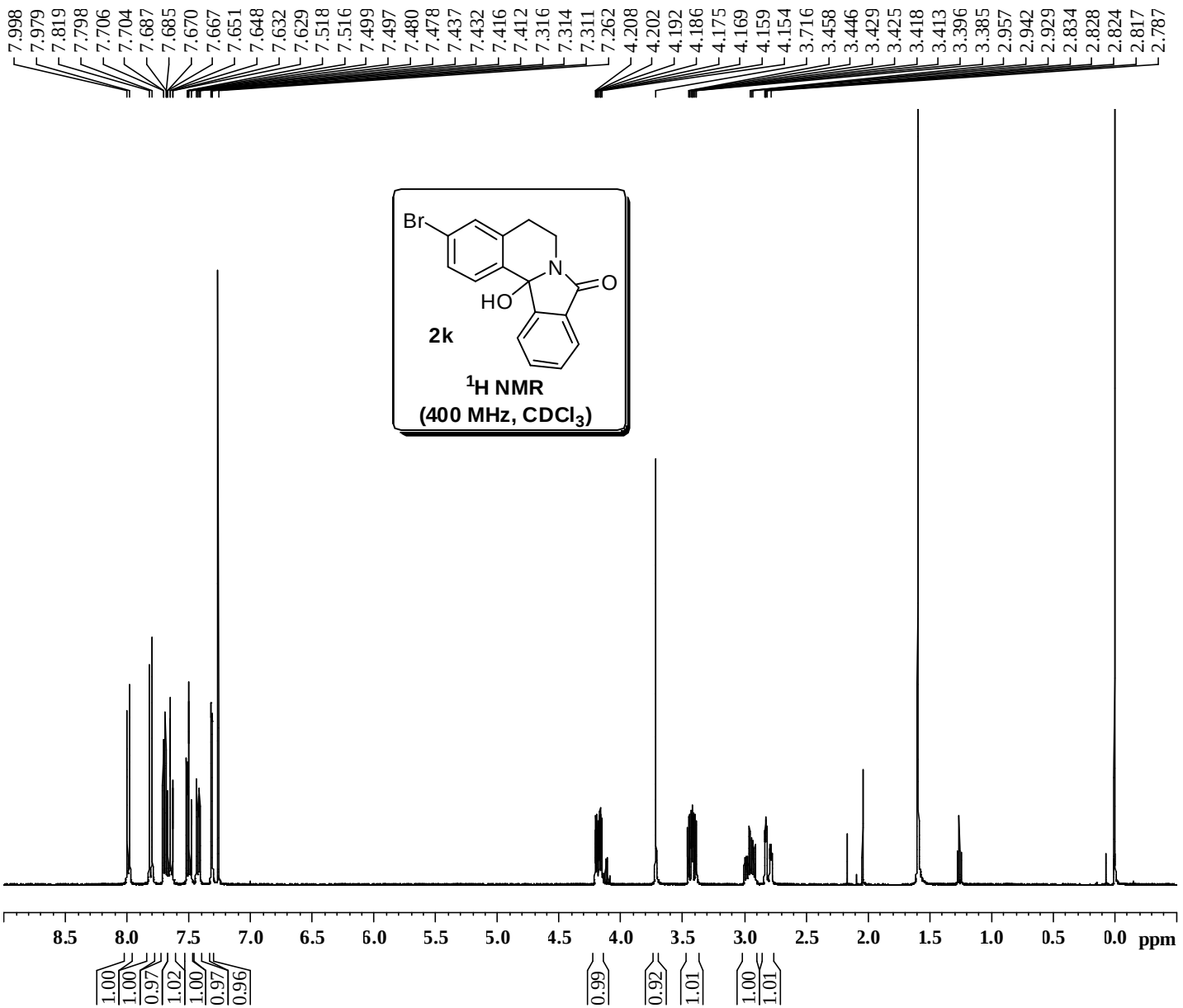

```
Current Data Parameters
NAME                  JS-UNC-1
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20110526
Time                     12.23
INSTRUM                  spect
PROBHD   5 mm BBO BB-1H
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         572
DS                           4
SWH                  24038.461 Hz
FIDRES                0.366798 Hz
AQ                    1.3631988 sec
RG                        1030
DW                      20.800 usec
DE                       6.00 usec
TE                      296.5 K
D1                   2.00000000 sec
d11                  0.03000000 sec
DELTA                1.89999998 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       13C
P1                        9.50 usec
PL1                      -0.60 dB
SFO1               100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                    90.00 usec
PL12                     15.60 dB
PL13                     15.60 dB
PL2                      -0.90 dB
SFO2               400.1316005 MHz

F2 - Processing parameters
SI                       32768
SF                 100.6127680 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```

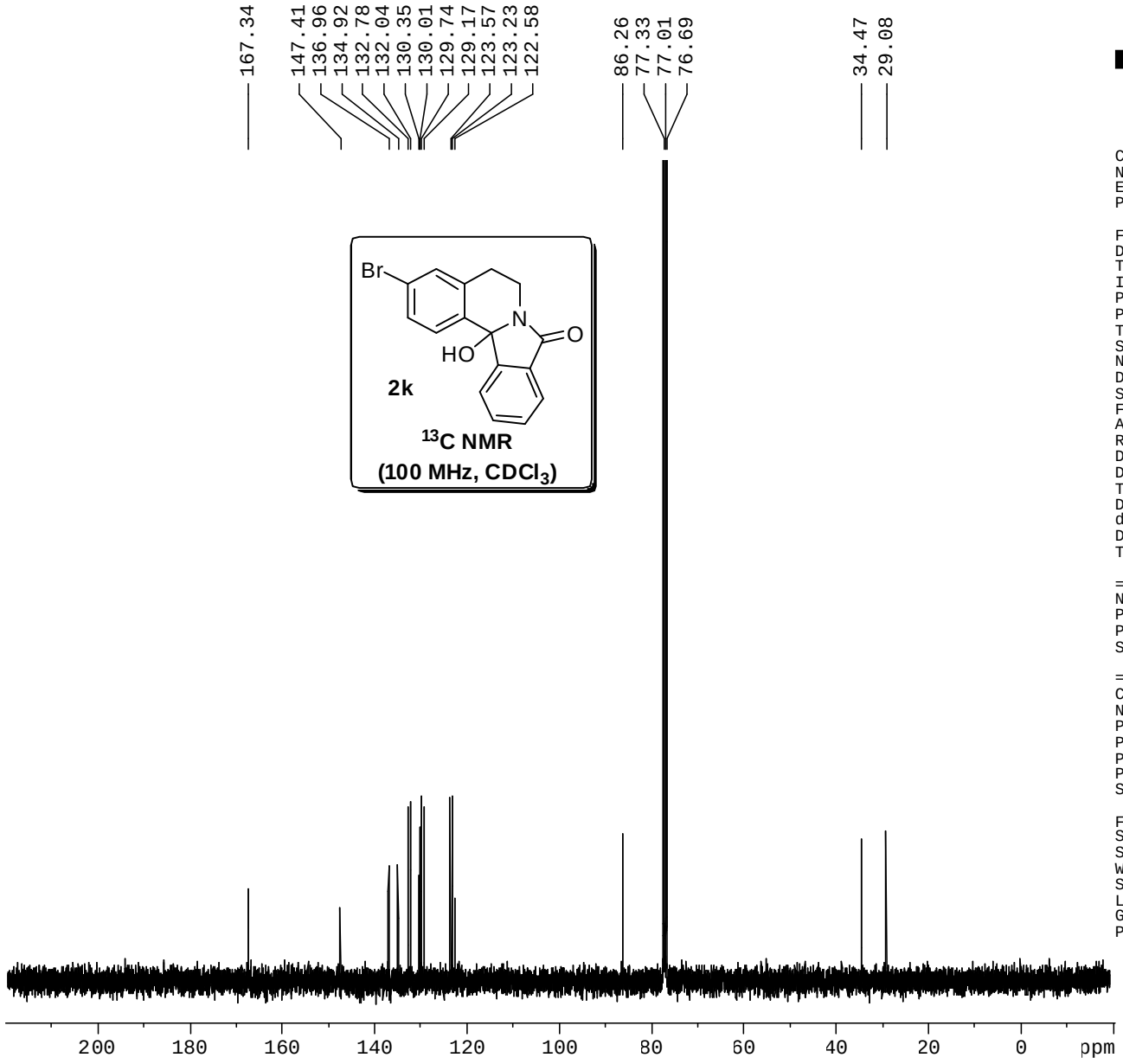



```
Current Data Parameters
NAME       JS-III-262-2
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20101214
Time              21.50
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                  575
DW               60.800 usec
DE                6.00 usec
TE                295.3 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PL1               -0.90 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                32768
SF          400.1300033 MHz
WDW                  EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                1.00
```




```
Current Data Parameters
NAME      JS-III-262-2a
EXPNO                1
PROCNO               1

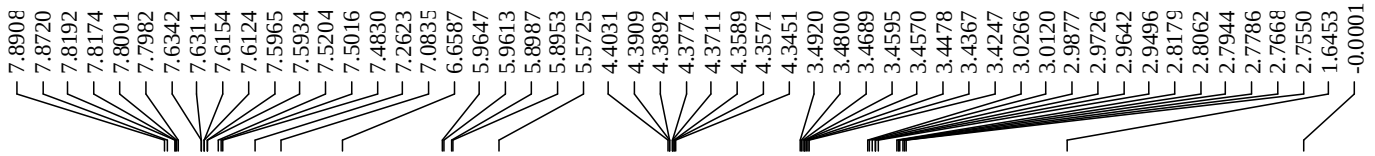
F2 - Acquisition Parameters
Date_          20101223
Time              16.40
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  150
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631988 sec
RG                  912
DW               20.800 usec
DE                6.00 usec
TE               297.6 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                  1

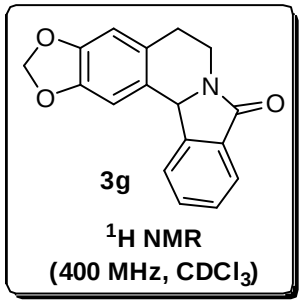
======== CHANNEL f1 ========
NUC1                13C
P1                9.50 usec
PL1              -0.60 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            90.00 usec
PL12             15.60 dB
PL13             15.60 dB
PL2              -0.90 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```

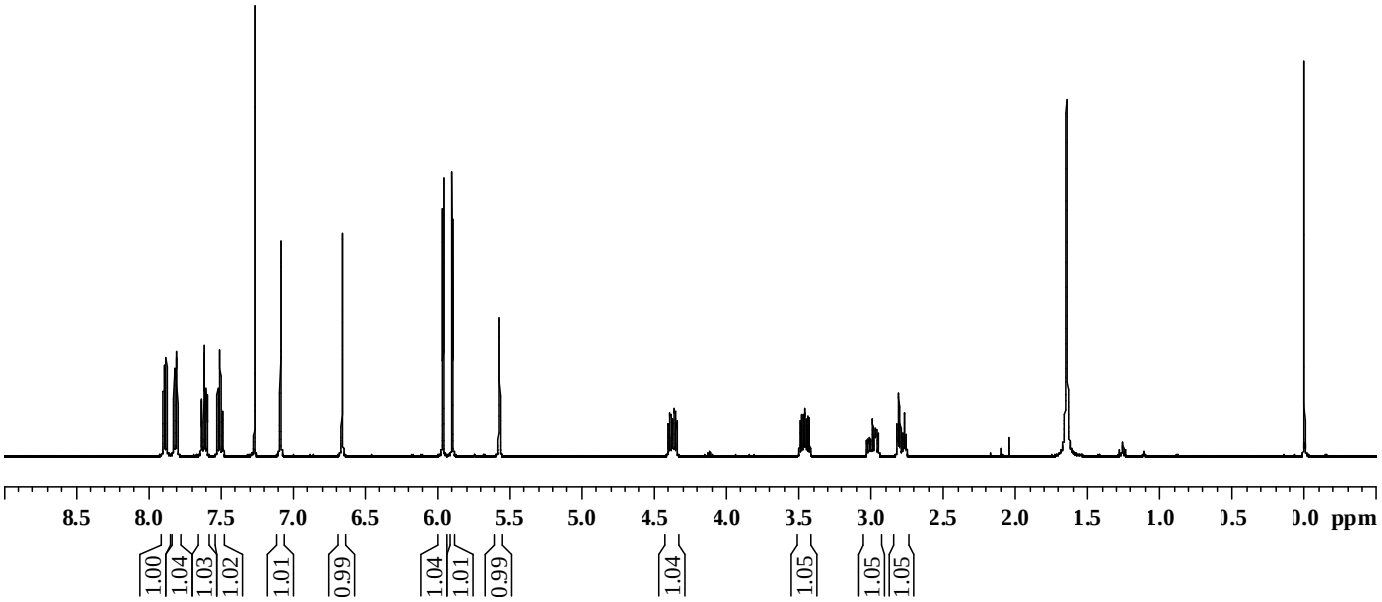
PROTON CDCl3 {D:\CRR} KOPAL 1





3g

^{1}H NMR
(400 MHz, $CDCl_3$)

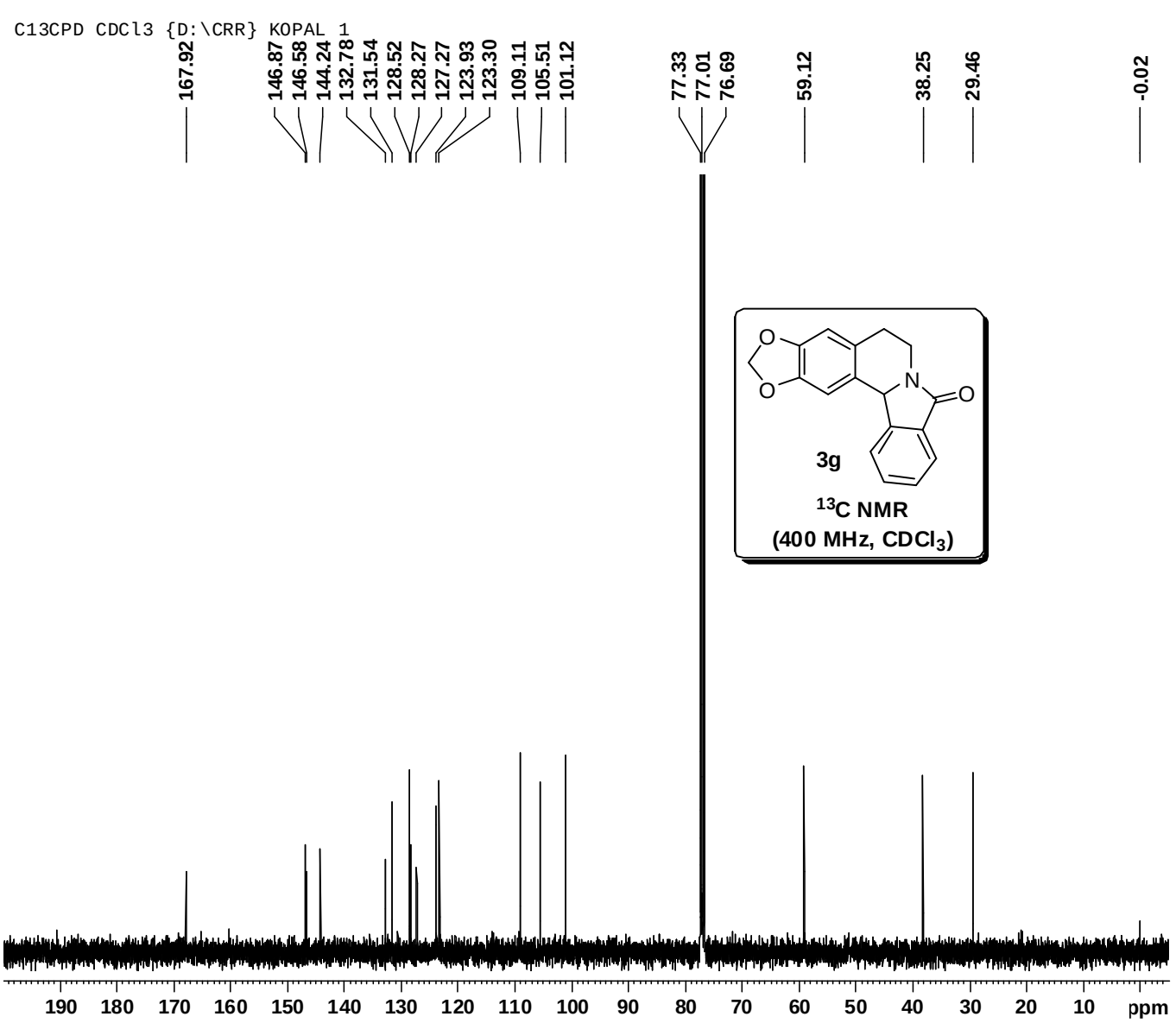



```
Current Data Parameters
NAME          JS-PiP-ted
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20101021
Time               15.26
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9846387 sec
RG                   406
DW                60.800 usec
DE                 6.00 usec
TE                296.5 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.00 usec
PL1                -0.90 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF           400.1300029 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```



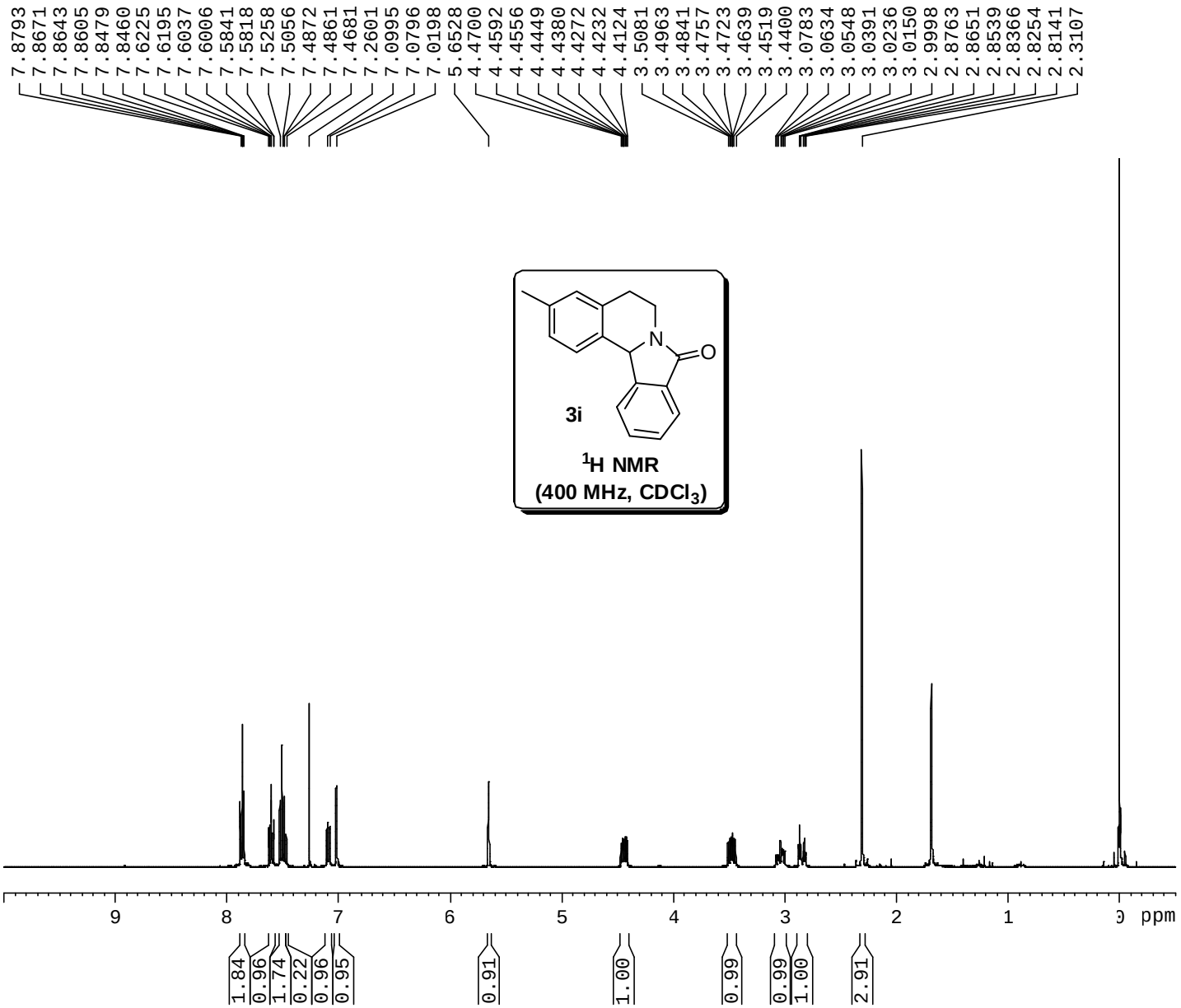

```
Current Data Parameters
NAME         JS-PIPC-RED
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20110208
Time               14.21
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631988 sec
RG                    57
DW                20.800 usec
DE                 6.00 usec
TE                297.3 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                  9.50 usec
PL1                -0.60 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL12               15.60 dB
PL13               15.60 dB
PL2                -0.90 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

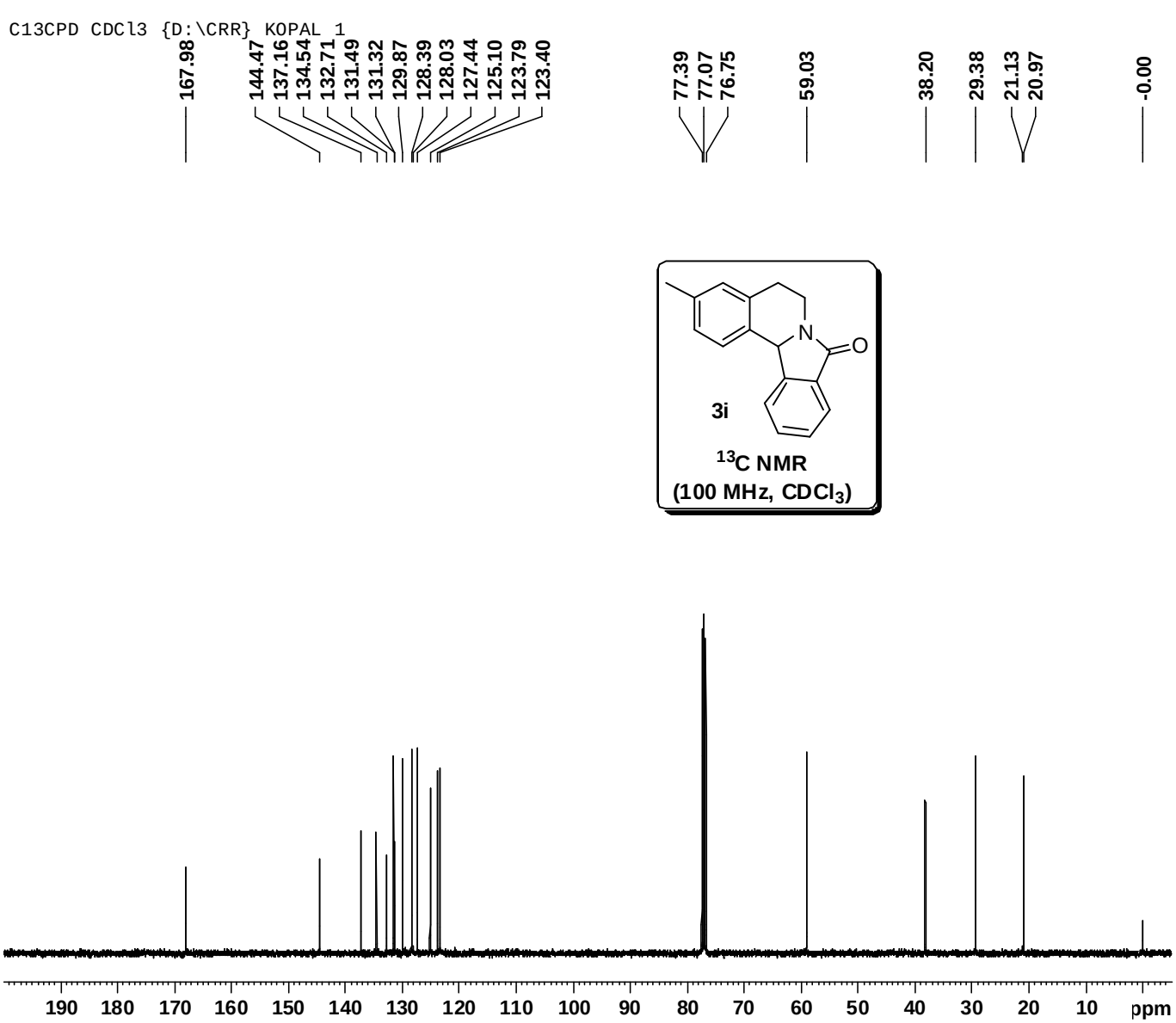



```
Current Data Parameters
NAME            JS-MERE-1
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20110523
Time                10.51
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                    256
DW                 60.800 usec
DE                  6.00 usec
TE                 295.6 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.00 usec
PL1                -0.90 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                  32768
SF           400.1300040 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                 1.00
```



BRUKER

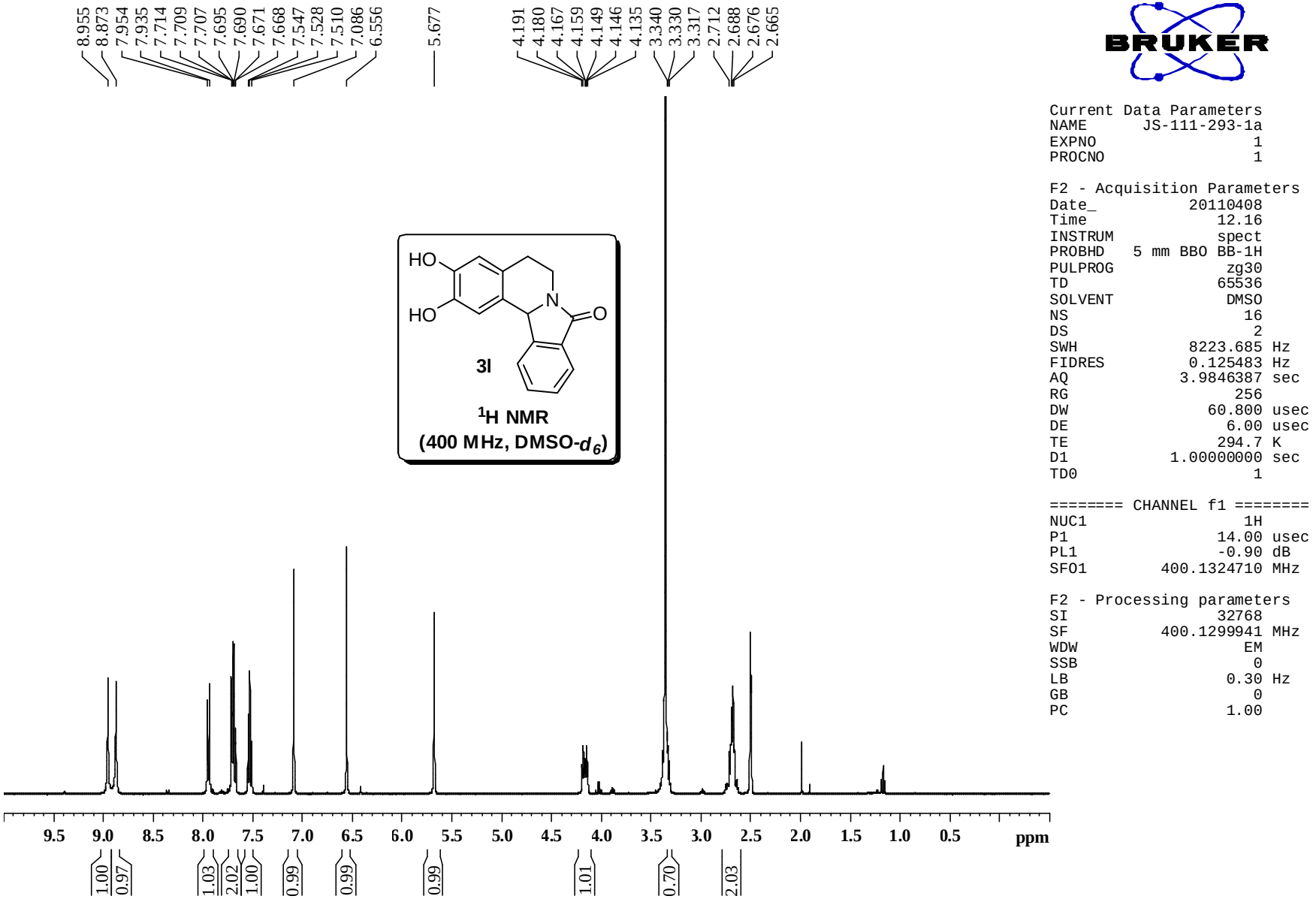
```
Current Data Parameters
NAME                  JS-MERE-1
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20110523
Time                      11.51
INSTRUM                   spect
PROBHD   5 mm BBO BB-1H
PULPROG                 zgpg30
TD                         65536
SOLVENT                   CDCl3
NS                          315
DS                            4
SWH                   24038.461 Hz
FIDRES                 0.366798 Hz
AQ                     1.3631988 sec
RG                         1150
DW                       20.800 usec
DE                        6.00 usec
TE                       295.7 K
D1                  2.00000000 sec
d11                 0.03000000 sec
DELTA               1.89999998 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                        13C
P1                        9.50 usec
PL1                      -0.60 dB
SFO1               100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                    90.00 usec
PL12                     15.60 dB
PL13                     15.60 dB
PL2                      -0.90 dB
SFO2               400.1316005 MHz

F2 - Processing parameters
SI                         32768
SF                 100.6127708 MHz
WDW                           EM
SSB                           0
LB                        1.00 Hz
GB                            0
PC                        1.40
```

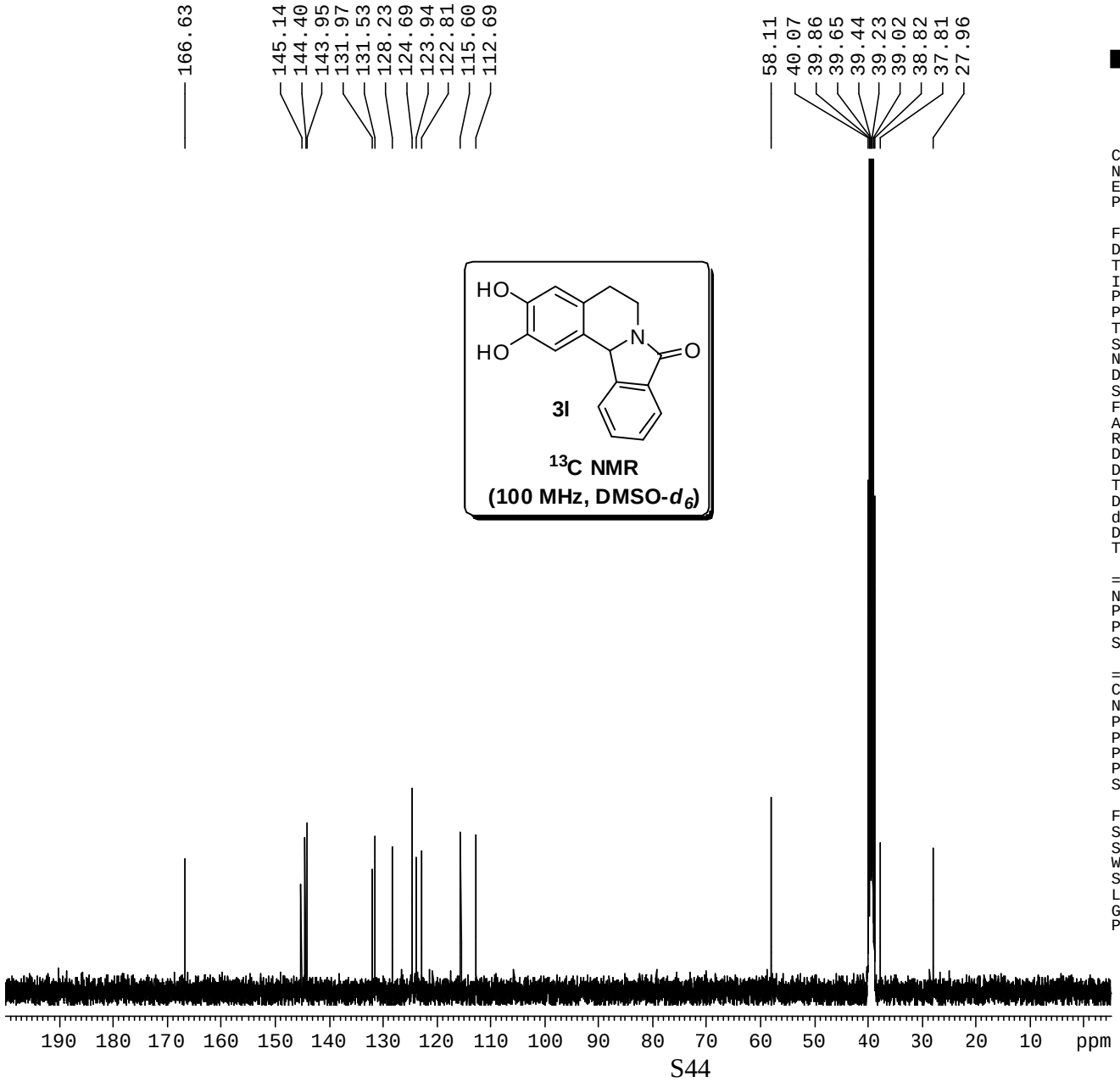



```
Current Data Parameters
NAME      JS-111-293-1a
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20110408
Time              12.16
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG            zg30
TD                65536
SOLVENT            DMSO
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                  256
DW                60.800 usec
DE                 6.00 usec
TE                294.7 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1                14.00 usec
PL1               -0.90 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                32768
SF          400.1299941 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



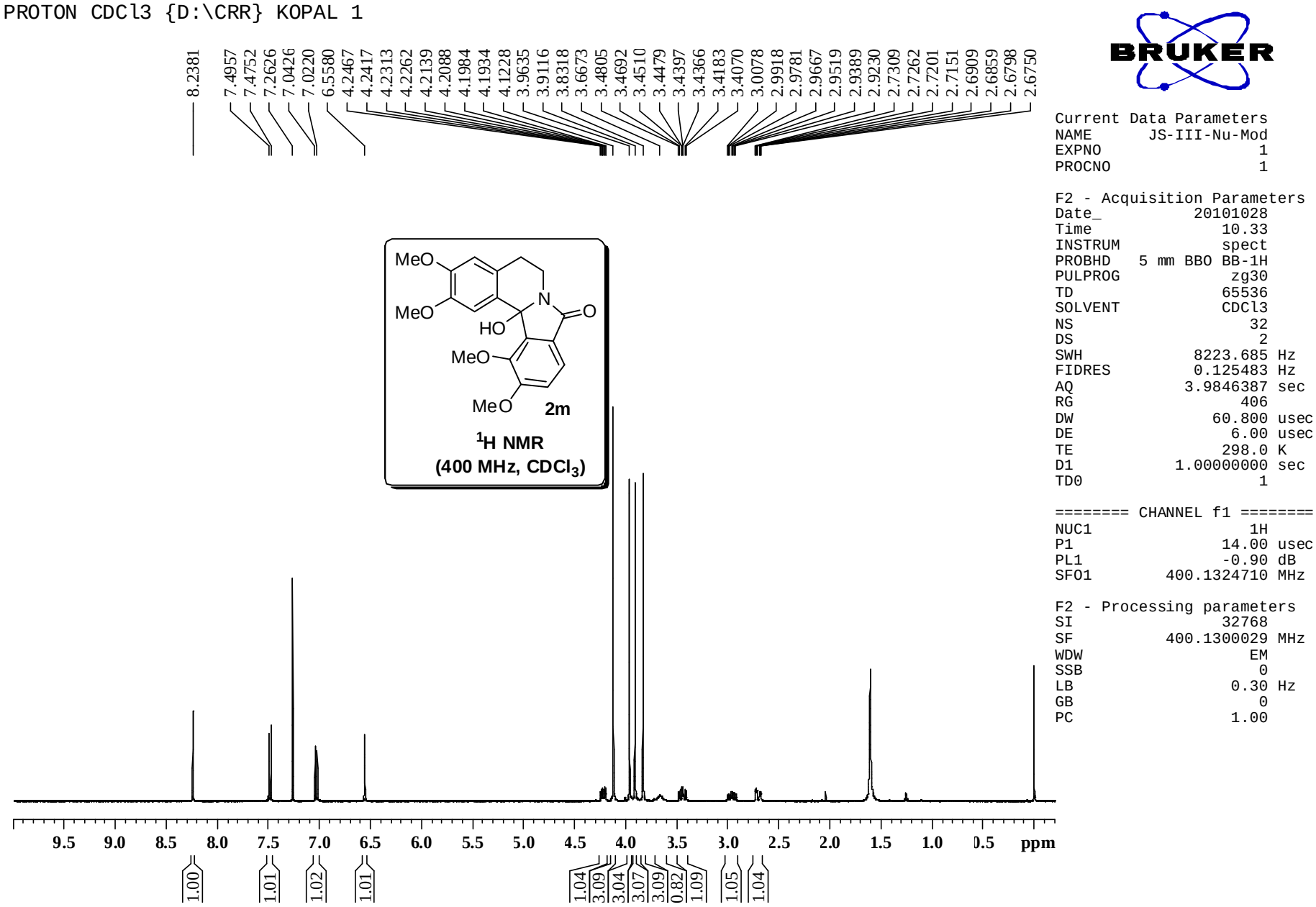

```
Current Data Parameters
NAME      JS-111-293-1a
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20110408
Time              12.46
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG         zgpg30
TD                65536
SOLVENT             DMSO
NS                  512
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631988 sec
RG                  812
DW               20.800 usec
DE                6.00 usec
TE               295.1 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.50 usec
PL1               -0.60 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PL12              15.60 dB
PL13              15.60 dB
PL2               -0.90 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6128193 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

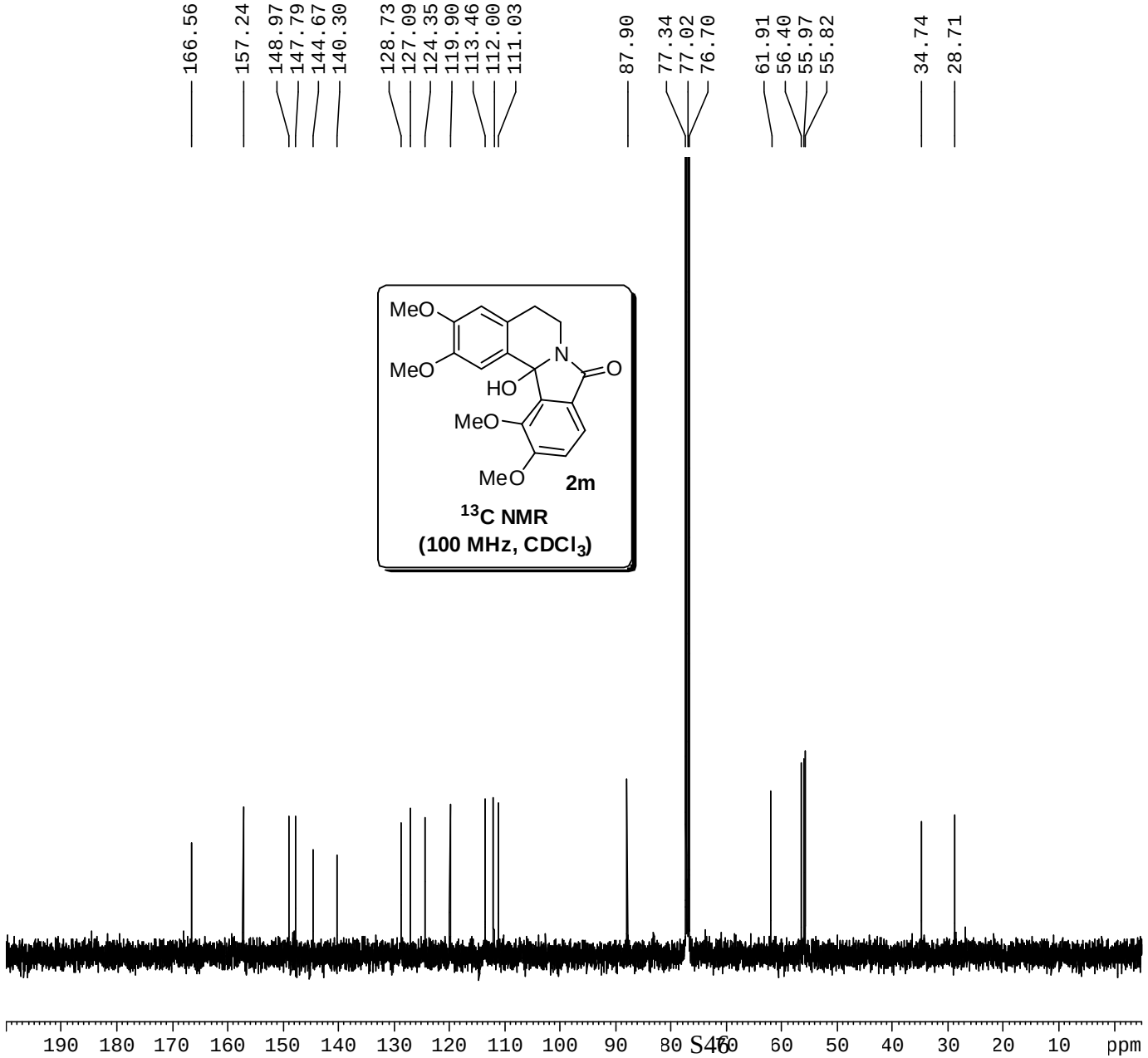



```
Current Data Parameters
NAME      JS-III-Nu-Mod
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20101028
Time              10.33
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   32
DS                    2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ            3.9846387 sec
RG                  406
DW                60.800 usec
DE                 6.00 usec
TE                298.0 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PL1               -0.90 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                32768
SF          400.1300029 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



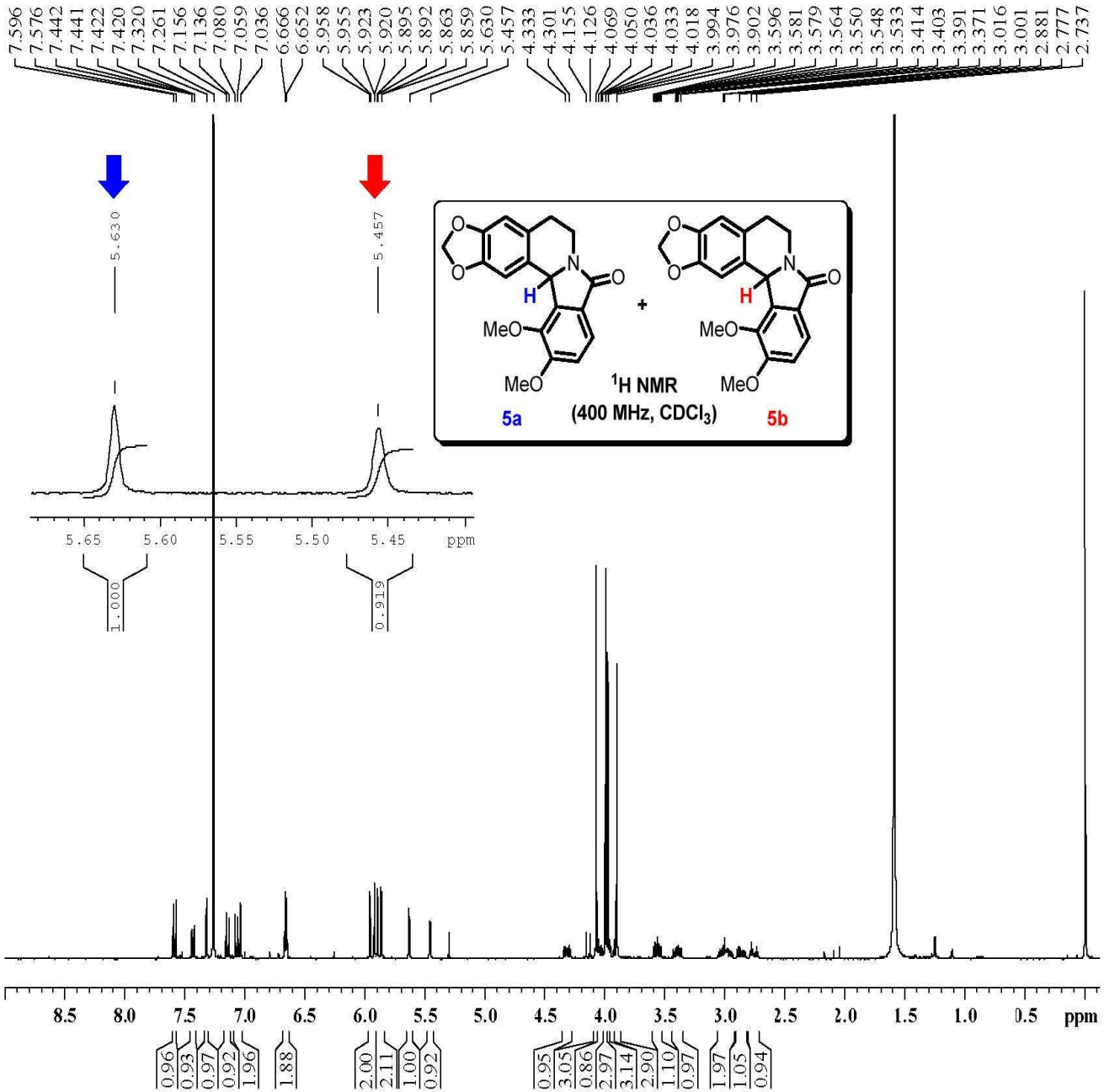

```
Current Data Parameters
NAME      JS-III-Nu-Mod
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20101101
Time              13.43
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631988 sec
RG                 50.8
DW                20.800 usec
DE                 6.00 usec
TE                297.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.50 usec
PL1                -0.60 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              90.00 usec
PL12               15.60 dB
PL13               15.60 dB
PL2                -0.90 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

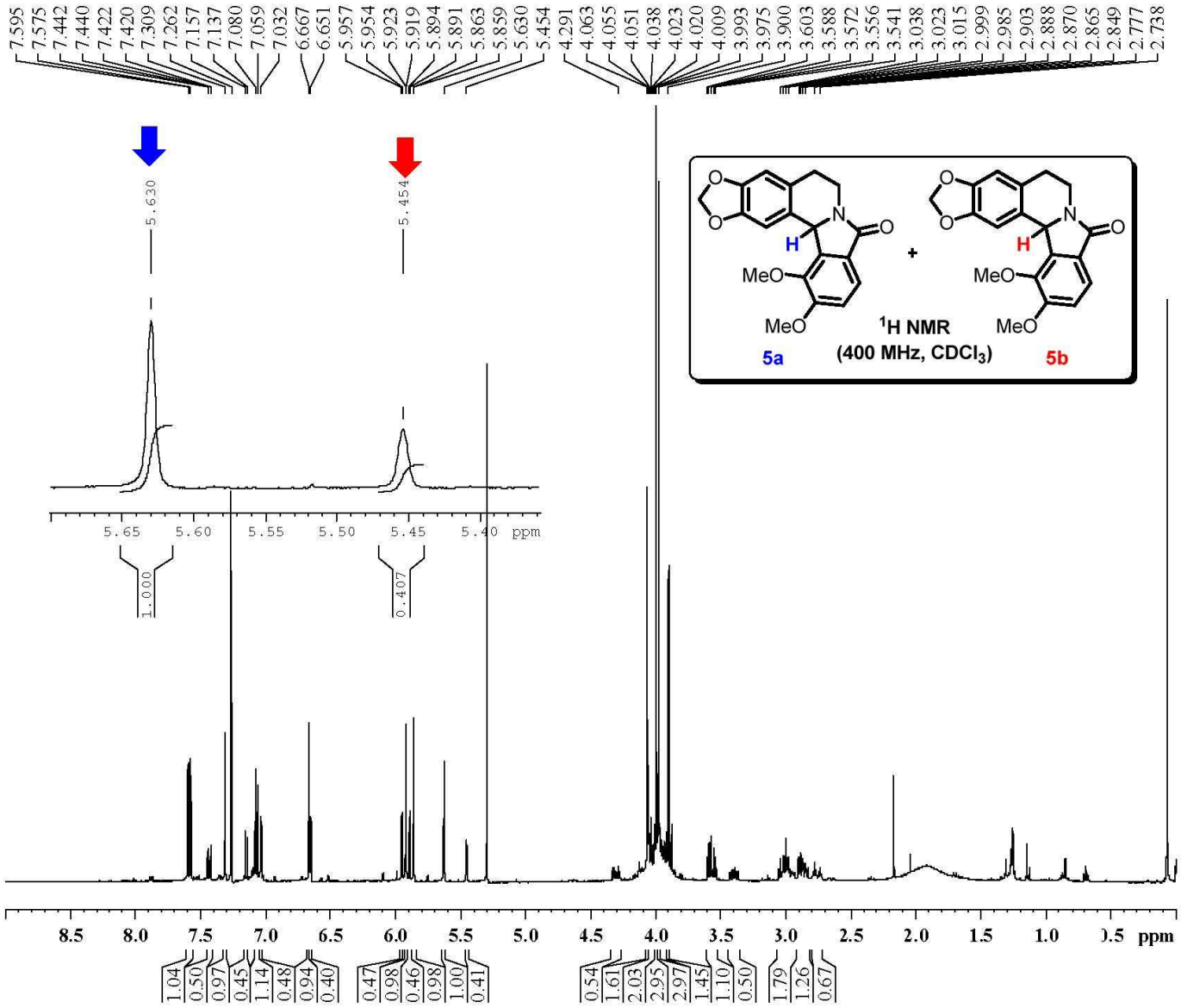



```
Current Data Parameters
NAME          JS-III-Nuv
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20101026
Time               11.49
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9846387 sec
RG                   812
DW                60.800 usec
DE                 6.00 usec
TE                296.7 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.00 usec
PL1                -0.90 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF          400.1300036 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

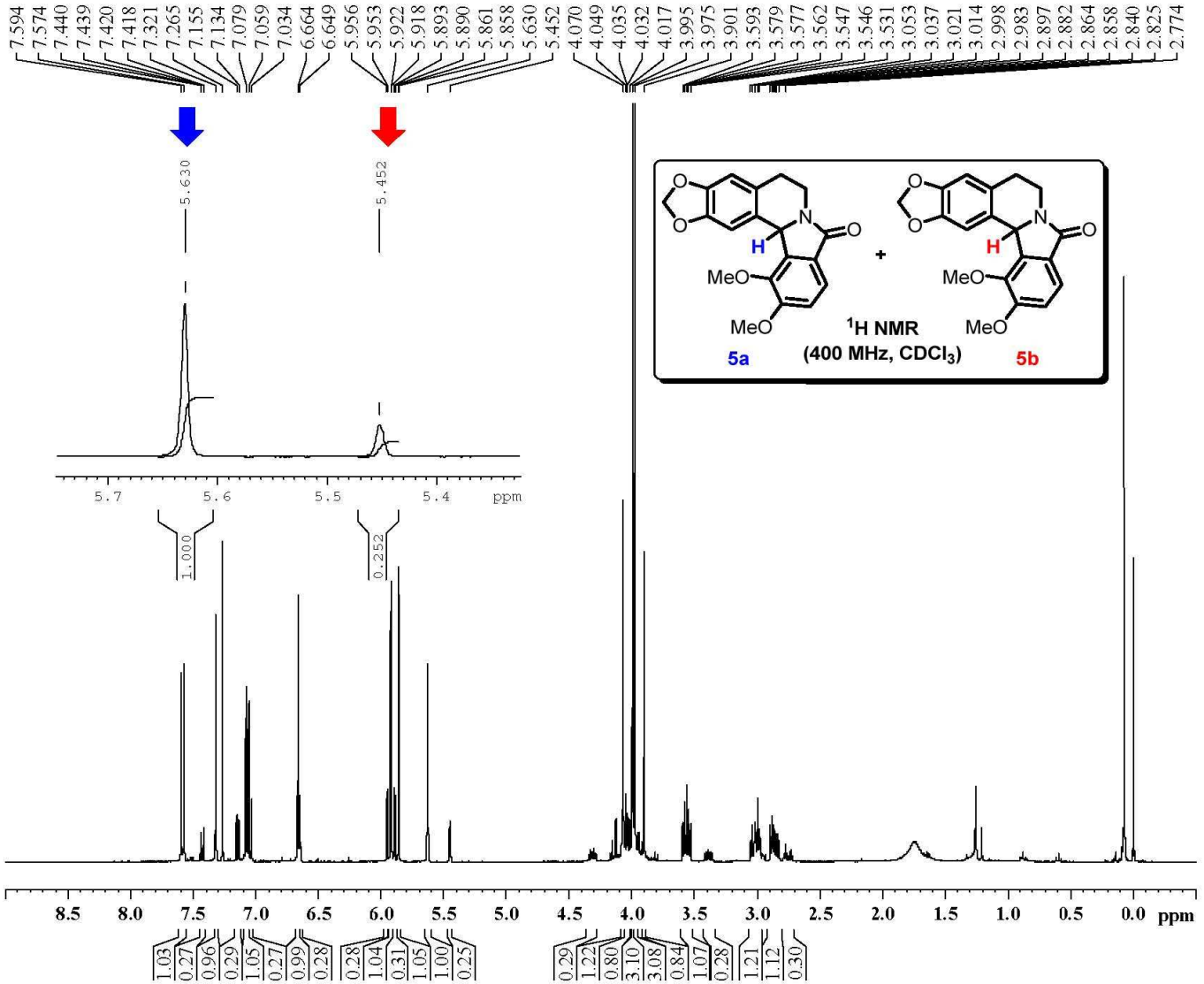



```
Current Data Parameters
NAME                  JS-III-255c
EXPNO                           1
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20101214
Time                        21.43
INSTRUM                     spect
PROBHD    5 mm BBO BB-1H
PULPROG                      zg30
TD                          65536
SOLVENT                     CDCl3
NS                             16
DS                              2
SWH                      8223.685 Hz
FIDRES                   0.125483 Hz
AQ                       3.9846387 sec
RG                            406
DW                         60.800 usec
DE                          6.00 usec
TE                         295.5 K
D1                    1.00000000 sec
TD0                             1

======== CHANNEL f1 ========
NUC1                           1H
P1                         14.00 usec
PL1                        -0.90 dB
SFO1                400.1324710 MHz

F2 - Processing parameters
SI                             32768
SF                  400.1300028 MHz
WDW                            EM
SSB                             0
LB                          0.30 Hz
GB                              0
PC                          1.00
```

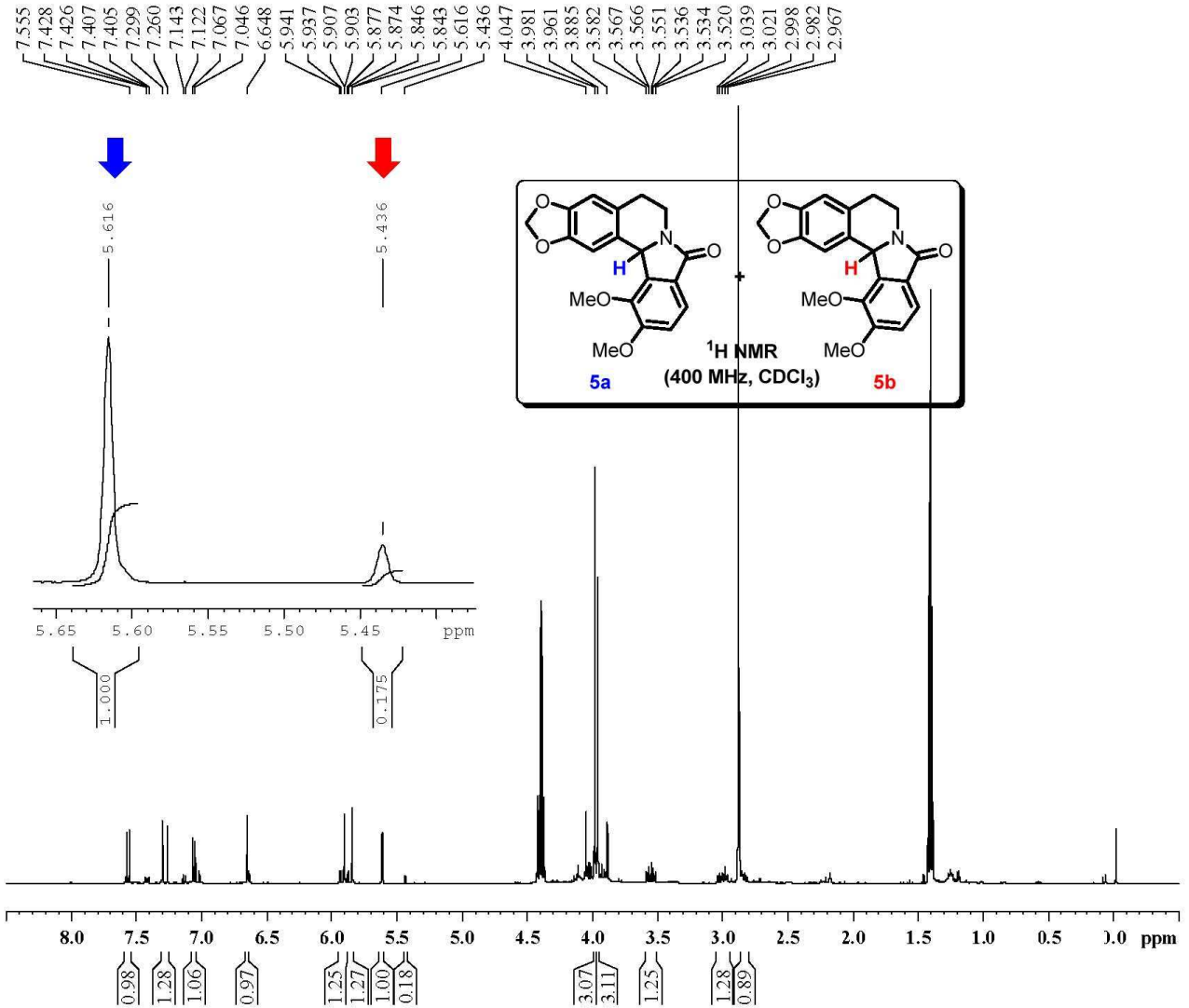



```
Current Data Parameters
NAME                  JS-NUV-40
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20101228
Time                      12.15
INSTRUM                   spect
PROBHD    5 mm BBO BB-1H
PULPROG                    zg30
TD                        65536
SOLVENT                   CDCl3
NS                           16
DS                            2
SWH                    8223.685 Hz
FIDRES                 0.125483 Hz
AQ                     3.9846387 sec
RG                          256
DW                       60.800 usec
DE                        6.00 usec
TE                       296.5 K
D1                     1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         1H
P1                       14.00 usec
PL1                      -0.90 dB
SFO1                400.1324710 MHz

F2 - Processing parameters
SI                        32768
SF                  400.1300021 MHz
WDW                          EM
SSB                           0
LB                       0.30 Hz
GB                            0
PC                        1.00
```

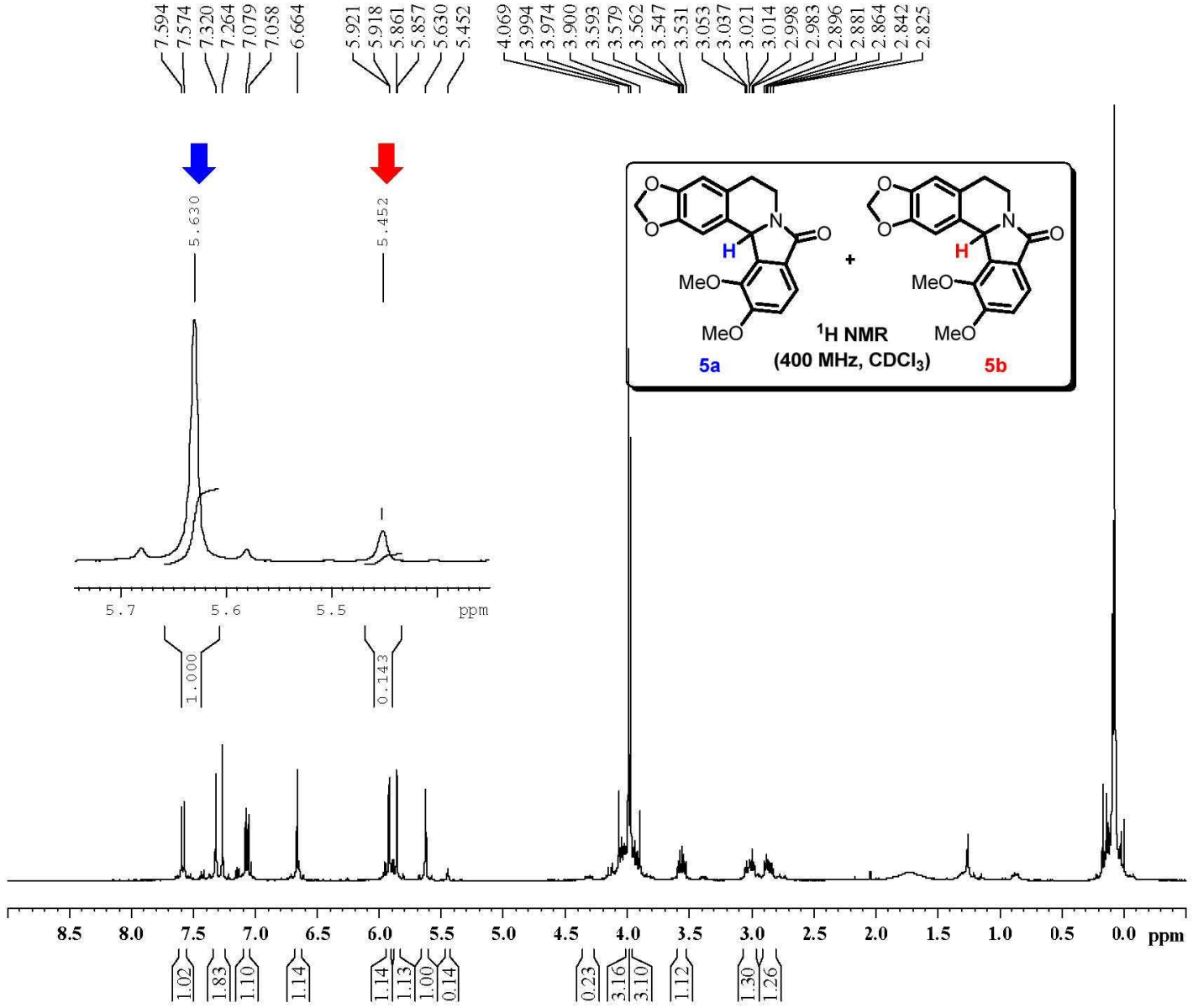



```
Current Data Parameters
NAME      JS-III-Nuv-60C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20110112
Time              12.02
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ             3.9846387 sec
RG                  161
DW               60.800 usec
DE                6.00 usec
TE                296.4 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PL1               -0.90 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                32768
SF          400.1300038 MHz
WDW                  EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                 1.00
```

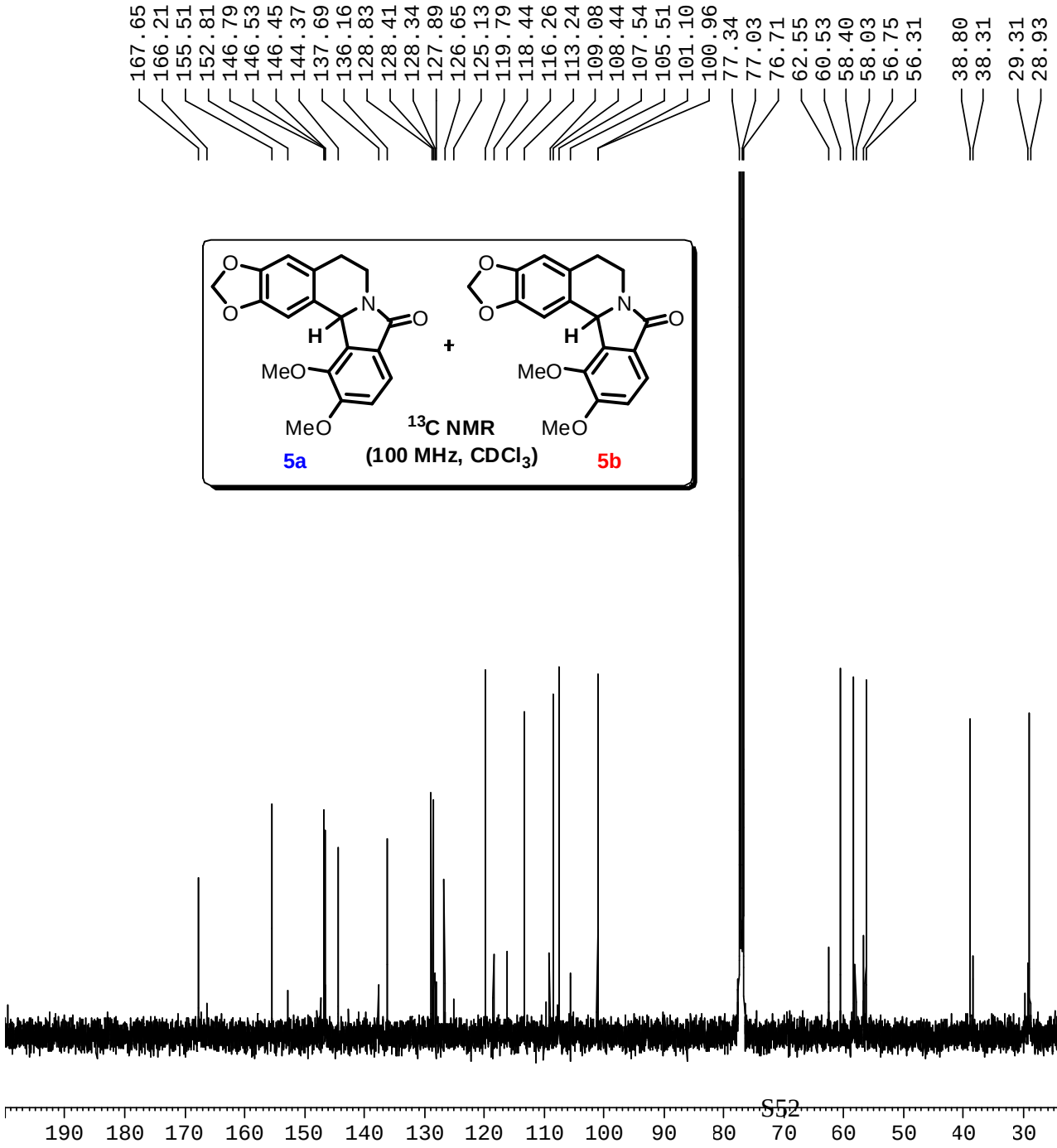



```
Current Data Parameters
NAME                  JS-NUV-78
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20101231
Time                      15.56
INSTRUM                   spect
PROBHD    5 mm BBO BB-1H
PULPROG                    zg30
TD                        65536
SOLVENT                   CDCl3
NS                           16
DS                            2
SWH                    8223.685 Hz
FIDRES                 0.125483 Hz
AQ                     3.9846387 sec
RG                          181
DW                       60.800 usec
DE                        6.00 usec
TE                       296.0 K
D1                  1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         1H
P1                       14.00 usec
PL1                      -0.90 dB
SFO1                400.1324710 MHz

F2 - Processing parameters
SI                        32768
SF                  400.1300016 MHz
WDW                          EM
SSB                           0
LB                       0.30 Hz
GB                            0
PC                        1.00
```




```
Current Data Parameters
NAME          JS-III-Nuv
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20110604
Time               17.33
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  5000
DS                     4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                  50.8
DW                20.800 usec
DE                 6.00 usec
TE                296.7 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.50 usec
PL1                -0.60 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              90.00 usec
PL12               15.60 dB
PL13               15.60 dB
PL2                -0.90 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127670 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```