Supporting information for

Synthesis of Spirocyclic β-keto-Lactams: Copper Catalyzed Process

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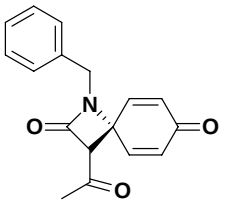
General Experimental: Proton nuclear magnetic resonance (^{1}H-NMR) spectra were recorded on a Bruker Avance 300 spectrometer at 300 MHz. Carbon-13 nuclear magnetic resonance (^{13}C-NMR) was recorded on a Bruker Avance 300 spectrometer at 75 MHz. Chemical shifts are reported as δ values in parts per million (ppm) relative to tetramethylsilane (TMS) for all recorded NMR spectra. Low-resolution Mass spectra were recorded on a VG Auto Spec-3000 magnetic sector MS spectrometer. High Resolution Mass spectra were taken on an AB QSTAR Pulsar mass spectrometer. Starting materials and reagents used in reactions were obtained commercially from Acros, Aldrich, Fluka and were used without purification, unless otherwise indicated.

General procedure for the synthesis of spirocyclic azetidinones, copper sulphate penthydrate catalyzed coupling:

To a solution of amide (1.0 mmol) in anhydrous ethanol (50 mL) was added copper (II) sulfate pentahydrate ($CuSO_4$•$5H_2O$, 13 mg, 0.05 mmol, 0.05 eq.) and DMAP (4-Dimethylaminopyridine, 7 mg, 0.06 mmol, 0.06 eq.). The resulting solution was stirred at 0 ºC for 10 minutes. To this mixture, IBD (iodobenzene diacetate, 387 mg, 1.20 mmol, 1.2 eq.) was added in one portion at 0ºC. After stirring at 0 ºC for 1 hour, the reaction mixture was concentrated and the residue was diluted with ethyl acetate (50 mL) and washed with water (10 mL) and brine (15 mL). The organic phase was then dried over anhydrous sodium sulfate. After filtration and removal of the solvent, the crude products were chromatographed on silica gel (Petroleum ether 60-90 ºC: ethyl acetate = 2:1) to provide azetidinones **5a-5s**, **13a-13h**, **15a-15f** and **17a-17b** (yields: 71-96%).

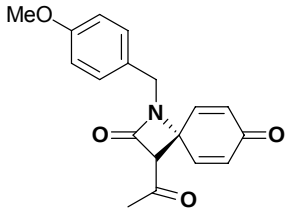


5a

3-acetyl-1-benzyl-1-azaspiro[3.5]nona-5,8-diene-2,7-dione

$C_{17}H_{15}NO_3$ Mol. Wt.: 281.31

5a: yellow syrup, Yield: 85%. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.24-7.03 (5H, *m*), 6.65 (1H, *dd*, *J* = 2.1, 10.0 Hz), 6.49 (1H, *dd*, *J* = 3.0, 10.0 Hz), 6.18-6.10 (2H, *m*), 4.27 (1H, *s*), 4.23 (1H, *d*, *J* = 15.1 Hz), 4.18 (1H, *d*, *J* = 15.1 Hz), 2.16 (3H, *s*). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 197.94, 184.34, 161.48, 146.09, 144.69, 134.92, 131.75, 131.62, 128.91, 128.82, 128.39, 70.78, 58.23, 45.59, 30.70. **EIMS** *m*/*z* (%) : 281 (M^+, 6%), 254 (3), 251 (4), 238 (8), 226 (4), 199 (18), 198 (12), 162 (3), 133 (4), 121 (12), 91 (100), 77 (6), 65 (12). **HRMS** *m*/*z* Found: 281.1056, Calcd. for $C_{17}H_{15}NO_3$ $(M)^+$: 281.1052.

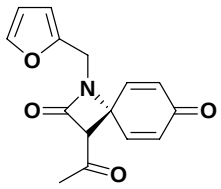


5b

3-acetyl-1-(4-methoxybenzyl)-1-azaspiro[3.5]nona-5,8-diene-2,7-dione

$C_{18}H_{17}NO_4$ Mol. Wt.: 311.31

5b: yellow syrup, Yield: 82%. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.08 (2H, *dd*, *J* = 1.9, 8.4 Hz), 6.79 (2H, *dd*, *J* = 1.9, 8.4 Hz), 6.73 (1H, *dd*, *J* = 3.0, 10.2 Hz), 6.58 (1H, *dd*, *J* = 3.0, 10.0 Hz), 6.27 (1H, *dd*, *J* = 2.1, 10.0 Hz), 6.24 (1H, *dd*, *J* = 2.1, 10.2 Hz), 4.34 (1H, *s*), 4.26 (1H, *d*, *J* = 15.0 Hz), 4.23 (1H, *dd*, *J* = 15.0 Hz), 3.77 (3H, *s*), 2.24 (3H, *s*). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 197.97, 184.35, 161.30, 159.63, 146.23, 144.82, 131.61, 131.46, 130.30, 126.97, 114.12, 70.64, 58.08, 55.25, 44.96, 30.67. **EIMS** *m*/*z* (%) : 313 (M^++2, 35%), 311 (M^+, 52%), 271 (12), 268 (16), 240 (6), 229 (51), 227 (56), 212 (14), 204 (13), 183 (16), 176 (12), 163 (65), 162 (59), 149 (53), 136 (66), 121 (100), 120 (81), 105 (36), 91 (74), 77 (90), 65 (51). **HRMS** *m*/*z* Found: 311.1151, Calcd. for $C_{18}H_{17}NO_4$ $(M)^+$: 311.1158.

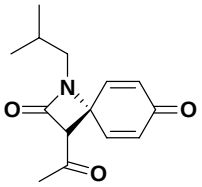


5c

3-acetyl-1-(furan-2-ylmethyl)-1-azaspiro[3.5]nona-5,8-diene-2,7-dione

$C_{15}H_{13}NO_4$ Mol. Wt.: 271.27

5c: pale yellow oil, Yield: 80%. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.30 (1H, *d*, *J* = 3.5 Hz), 6.77 (1H, *dd*, *J* = 2.8, 10.1 Hz), 6.63 (1H, *dd*, *J* = 2.9, 10.0 Hz), 6.38-6.22 (3H, *m*), 6.18 (1H, *d*, *J* = 2.5 Hz), 4.37 (1H, *d*, *J* = 15.6 Hz), 4.34 (1H, *s*), 4.29 (1H, *d*, *J* = 15.6 Hz), 2.26 (3H, *s*). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 197.86, 184.38, 161.24, 147.42, 145.63, 144.23, 142.68, 131.66, 131.49, 110.82, 110.34, 70.59, 57.94, 37.47, 30.62. **EIMS** *m*/*z* (%) : 273 (M^++2, 46%), 271 (M^+, 52%), 243 (14), 230 (30), 228 (37), 204 (48), 188 (77), 172 (21), 161 (48), 149 (79), 148 (77), 133 (81), 120 (82), 105 (48), 96 (40), 91 (49), 81 (100), 77 (65), 68 (44). **HRMS** *m*/*z* Found: 271.0844, Calcd. for $C_{15}H_{13}NO_4$ $(M)^+$: 271.0845.

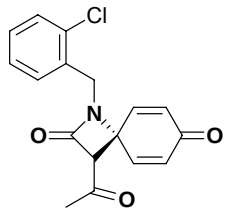


5d

3-acetyl-1-isobutyl-1-azaspiro[3.5]nona-5,8-diene-2,7-dione

$C_{14}H_{17}NO_3$ Mol. Wt.: 247.29

5d: brown oil, Yield: 84%. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 6.90 (1H, *dd*, *J* = 3.2, 10.6 Hz), 6.80 (1H, *dd*, *J* = 2.7, 10.0 Hz), 6.40-6.30 (2H, *m*), 4.36 (1H, *s*), 2.88-2.70 (2H, *m*), 2.19 (3H, *s*), 1.80-1.63 (1H, *m*), 0.82 (6H, *d*, *J* = 6.6 Hz). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 198.20, 184.22, 162.29, 146.49, 145.09, 132.09, 70.44, 57.96, 49.44, 30.63, 27.79, 20.07. **EIMS** *m*/*z* (%) : 247 (M^+, 13%), 205 (11), 204 (16), 176 (8), 168 (13), 165 (37), 148 (12), 134 (15), 122 (100), 121 (32), 105 (17), 84 (16), 69 (22), 57 (30). **HRMS** *m*/*z* Found: 247.1213, Calcd. for $C_{14}H_{17}NO_3$ $(M)^+$: 247.1208.

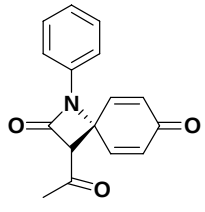


5e

3-acetyl-1-(2-chlorobenzyl)-1-azaspiro[3.5]nona-5,8-diene-2,7-dione

$C_{17}H_{14}ClNO_3$ Mol. Wt.: 315.75

5e: pale yellow syrup, Yield: 87%. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.28-7.04 (4H, *m*), 6.66 (1H, *dd*, *J* = 3.0, 10.0 Hz), 6.53 (1H, *dd*, *J* = 3.0, 10.0 Hz), 6.14 (1H, *dd*, *J* = 1.8, 10.2 Hz), 6.10 (1H, *dd*, *J* = 1.8, 10.2 Hz), 4.43 (1H, *d*, *J* = 15.0 Hz), 4.35 (1H, *d*, *J* = 15.0 Hz), 4.30 (1H, *s*), 2.17 (3H, *s*). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 198.02, 184.27, 161.35, 145.79, 144.38, 134.06, 132.13, 131.78, 131.60, 131.52, 130.19, 129.78, 127.21, 70.67, 58.12, 43.12, 30.74. **EIMS** *m*/*z* (%) : 317 (M^+, 9%), 315 (M^+, 16%), 282 (9), 280 (10), 235 (26), 233 (54), 232 (27), 190 (11), 167 (10), 148 (7), 140 (12), 127 (51), 125 (100), 108 (33), 89 (27), 87 (28), 77 (16). **HRMS** *m*/*z* Found: 315.0662, Calcd. for $C_{17}H_{14}NO_3Cl$ $(M)^+$: 315.0662.

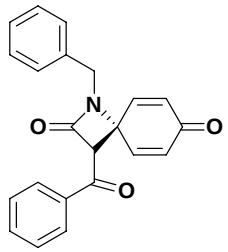


5f

3-acetyl-1-phenyl-1-azaspiro[3.5]nona-5,8-diene-2,7-dione

$C_{16}H_{13}NO_3$ Mol. Wt.: 267.28

5f: pale yellow powder, m.p.: 132-133 ºC. Yield: 85%. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.40-7.22 (4H, *m*), 7.20-7.08 (2H, *m*), 7.01 (1H, *dd*, *J* = 3.0, 10.2 Hz), 6.59 (1H, *dd*, *J* = 2.1, 10.2 Hz), 6.57 (1H, *dd*, *J* = 2.1, 10.2 Hz), 4.49 (1H, *s*), 2.33 (3H, *s*). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 197.29, 184.00, 158.91, 146.80, 145.54, 137.17, 132.73, 132.62, 129.42, 125.35, 116.62, 70.38, 58.59, 30.94. **EIMS** *m*/*z* (%) : 269 (M^++2, 16%), 267 (M^+, 50%), 227 (28), 225 (37), 212 (30), 197 (32), 183 (45), 182 (34), 167 (23), 154 (46), 148 (26), 134 (29), 133 (57), 119 (100), 105 (36), 103 (44), 93 (58), 91 (66), 77 (78), 69 (27). **HRMS** *m*/*z* Found: 267.0896, Calcd. for $C_{16}H_{13}NO_3$ $(M)^+$: 267.0895.

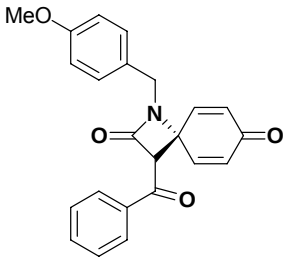


5g

3-benzoyl-1-benzyl-1-azaspiro[3.5]nona-5,8-diene-2,7-dione

$C_{22}H_{17}NO_3$ Mol. Wt.: 343.38

5g: pale yellow plates, m.p.: 130-132 ºC. Yield: 92%. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.73 (2H, *d*, *J* = 7.5 Hz), 7.56 (1H, *t*, *J* = 7.2 Hz), 7.49-7.18 (7H, *m*), 6.77 (1H, *dd*, *J* = 3.0, 10.0 Hz), 6.68 (1H, *dd*, *J* = 3.0, 10.0 Hz), 6.32 (1H, *dd*, *J* = 2.1, 10.0 Hz), 6.07 (1H, *dd*, *J* = 2.0, 10.0 Hz), 5.07 (1H, *s*), 4.38 (2H, *s*). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 190.74, 183.96, 161.44, 146.31, 144.27, 135.59, 135.04, 134.41, 132.40, 132.15, 128.97, 128.85, 128.38, 67.00, 58.44, 45.73. **EIMS** *m*/*z* (%) : 345 (M^++2, 48%), 343 (M^+, 81%), 315 (14), 302 (11), 292 (74), 291 (84), 274 (22), 263 (23), 252 (12), 238 (36), 224 (27), 211 (88), 196 (100), 183 (84), 181 (90), 168 (92), 167 (62), 154 (90), 146 (88), 133 (62), 119 (61), 106 (92), 105 (86), 92 (96), 89 (94), 78 (85), 77 (77). **HRMS** *m*/*z* Found: 343.1203, Calcd. for $C_{22}H_{17}NO_3$ $(M)^+$: 343.1208.

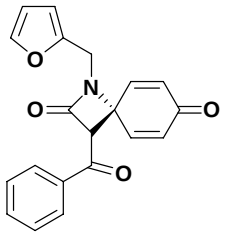


5h

3-benzoyl-1-(4-methoxybenzyl)-1-azaspiro[3.5]nona-5,8-diene-2,7-dione

$C_{23}H_{19}NO_4$ Mol. Wt.: 373.38

5h: brown syrup, Yield: 90%. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.62 (2H, *d*, *J* = 8.4 Hz), 7.60-7.15 (3H, *m*), 7.04 (2H, *d*, *J* = 8.4 Hz), 6.82-6.51 (4H, *m*), 6.40-6.20 (1H, *m*), 6.12-5.92 (1H, *m*), 5.01 (1H, *s*), 4.24 (2H, *s*), 3.69 (3H, *s*). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 190.87, 184.06, 161.42, 159.63, 146.59, 144.49, 135.57, 134.37, 132.22, 131.94, 130.34, 128.93, 128.34, 127.05, 114.14, 66.75, 58.32, 55.25, 45.09. **EIMS** *m*/*z* (%) : 375 (M^++2, 9%), 373 (M^+, 17%), 354 (10), 333 (20), 292 (19), 291 (28), 265 (45), 255 (9), 229 (36), 227 (43), 212 (10), 183 (9), 165 (8), 146 (29), 135 (15), 121 (100), 105 (89), 91 (20), 77 (70). **HRMS** *m*/*z* Found: 373.1303, Calcd. for $C_{23}H_{19}NO_4$ $(M)^+$: 373.1314.

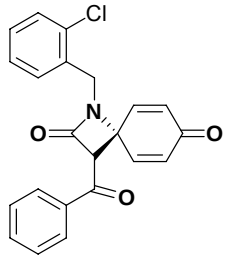


5i

3-benzoyl-1-(furan-2-ylmethyl)-1-azaspiro[3.5]nona-5,8-diene-2,7-dione

$C_{20}H_{15}NO_4$ Mol. Wt.: 333.34

5i: pale yellow solid, m.p.: 130-131 ºC. Yield: 88%. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.74 (2H, *d*, *J* = 7.2 Hz), 7.60-7.50 (1H, *m*), 7.48-7.35 (2H, *m*), 7.30 (1H, *s*), 6.81 (1H, *dt*, *J* = 2.4, 9.9 Hz), 6.74 (1H, *dd*, *J* = 2.7, 10.2 Hz), 6.36 (1H, *dt*, *J* = 1.8, 9.9 Hz), 6.27 (1H, *d*, *J* = 1.8 Hz), 6.17 (1H, *s*), 6.16-6.09 (1H, *m*), 5.05 (1H, *d*, *J* = 1.8 Hz), 4.48 (1H, *dd*, *J* = 2.7, 15.6 Hz), 4.33 (1H, *d*, *J* = 15.6 Hz). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 190.61, 184.00, 161.13, 147.55, 145.84, 143.77, 142.68, 135.57, 134.41, 132.38, 131.99, 128.96, 128.37, 110.85, 110.39, 66.81, 58.14, 37.63. **EIMS** *m*/*z* (%) : 335 (M^++2, 27%), 333 (M^+, 39%), 315 (7), 292 (37), 291 (50), 268 (24), 266 (29), 238 (34), 223 (36), 212 (67), 210 (56), 189 (67), 183 (85), 181 (60), 161 (40), 153 (82), 146 (83), 133 (45), 122 (47), 105 (100), 96 (38), 89 (48), 81 (95), 77 (96). **HRMS** *m*/*z* Found: 333.0999, Calcd. for $C_{20}H_{15}NO_4$ $(M)^+$: 333.1001.

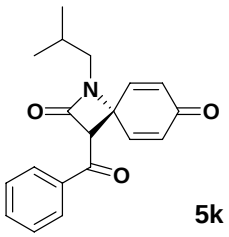


5j

3-benzoyl-1-(2-chlorobenzyl)-1-azaspiro[3.5]nona-5,8-diene-2,7-dione

$C_{22}H_{16}ClNO_3$ Mol. Wt.: 377.82

5j: Pale yellow foam, Yield: 86%. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.73 (2H, *d*, *J* = 7.5 Hz), 7.56 (1H, *t*, *J* = 7.2 Hz), 7.48-7.15 (6H, *m*), 6.81 (1H, *dd*, *J* = 3.0, 10.2 Hz), 6.72 (1H, *dd*, *J* = 3.0, 10.2 Hz), 6.30 (1H, *dd*, *J* = 1.8, 10.2 Hz), 6.07 (1H, *dd*, *J* = 1.8, 10.2 Hz), 5.09 (1H, *s*), 4.60 (1H, *d*, *J* = 15.0 Hz), 4.51 (1H, *d*, *J* = 15.0 Hz). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 190.71, 183.87, 161.35, 145.97, 143.92, 135.56, 134.42, 134.14, 132.50, 132.42, 132.18, 131.50, 130.10, 129.77, 128.96, 128.37, 127.26, 66.95, 58.36, 43.11. **EIMS** *m*/*z* (%) : 379 (M^+, 29%), 377 (M^+, 34%), 358 (8), 356 (11), 344 (22), 342 (70), 315 (11), 308 (20), 292 (93), 291 (40), 272 (18), 264 (22), 263 (20), 252 (12), 238 (40), 234 (61), 232 (72), 230 (68), 212 (52), 196 (52), 183 (70), 181 (84), 167 (63), 154 (86), 146 (100), 132 (72), 126 (64), 119 (49), 106 (77), 102 (46), 89 (65), 77 (57). **HRMS** *m*/*z* Found: 377.0821, Calcd. for $C_{22}H_{16}NO_3Cl$ $(M)^+$: 377.0819.

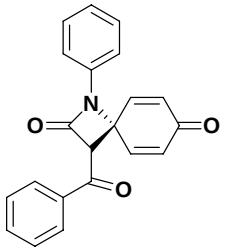


5k

3-benzoyl-1-isobutyl-1-azaspiro[3.5]nona-5,8-diene-2,7-dione

$C_{19}H_{19}NO_3$ Mol. Wt.: 309.14

5a: colorless plates, m.p.: 112-113 ºC. Yield: 96%. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.77 (2H, *dd*, *J* = 1.3, 7.9 Hz), 7.59 (1H, *t*, *J* = 7.4 Hz), 7.44 (2H, *dd*, *J* = 7.4, 7.9 Hz), 7.04 (1H, *dd*, *J* = 2.9, 10.0 Hz), 6.92 (1H, *dd*, *J* = 2.9, 10.2 Hz), 6.54 (1H, *dd*, *J* = 2.0, 10.0 Hz), 6.28 (1H, *dd*, *J* = 2.0, 10.2 Hz), 5.11 (1H, *s*), 3.02 (1H, *dd*, *J* = 7.7, 14.2 Hz), 2.93 (1H, *dd*, *J* = 7.3, 14.2 Hz), 1.95-1.73 (1H, *m*), 0.94 (6H, *t*, *J* = 6.4 Hz). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 190.97, 183.96, 162.27, 146.69, 144.68, 135.64, 134.39, 132.81, 128.97, 128.39, 66.66, 58.22, 49.71, 27.97, 20.19, 20.16. **EIMS** *m*/*z* (%) : 311 (M^++2, 8%), 309 (M^+, 16%), 266 (4), 238 (7), 210 (94), 189 (3), 182 (60), 181 (22), 165 (10), 163 (12), 153 (32), 148 (19), 133 (17), 122 (22), 120 (47), 105 (100), 92 (13), 91 (11), 77 (71). **HRMS** *m*/*z* Found: 309.1365, Calcd. for $C_{19}H_{19}NO_3$ $(M)^+$: 309.1365.

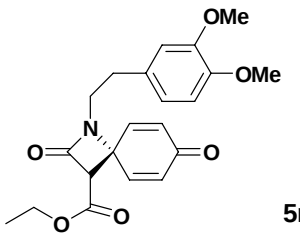


5l

3-benzoyl-1-phenyl-1-azaspiro[3.5]nona-5,8-diene-2,7-dione

$C_{21}H_{15}NO_4$ Mol. Wt.: 329.34

5l: Pale yellow foam, Yield: 80%. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.78 (2H, *dd*, *J* = 1.5, 7.4 Hz), 7.70-7.57 (1H, *m*), 7.53-7.24 (7H, *m*), 7.20-7.04 (2H, *m*), 6.68 (1H, *dd*, *J* = 1.8, 9.9 Hz), 6.37 (1H, *dd*, *J* = 2.1, 10.2 Hz), 5.24 (1H, *s*). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 190.38, 183.62, 158.99, 147.21, 145.18, 137.28, 135.56, 134.68, 133.46, 133.04, 129.42, 129.11, 128.42, 125.28, 116.61, 66.28, 58.60. **EIMS** *m*/*z* (%) : 330 (M^++1, 39%), 329 (M^+, 47%), 315 (25), 301 (29), 291 (41), 272 (22), 260 (10), 251 (25), 238 (68), 224 (45), 212 (75), 210 (86), 196 (38), 185 (77), 182 (87), 154 (73), 152 (53), 134 (42), 129 (61), 119 (61), 105 (100), 93 (64), 91 (58), 77 (91). **HRMS** *m*/*z* Found: 329.1050, Calcd. for $C_{21}H_{15}NO_3$ $(M)^+$: 329.1052.

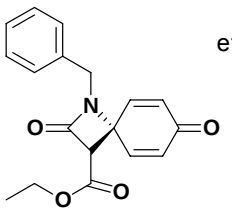


5m

ethyl 1-(3,4-dimethoxyphenethyl)-2,7-dioxo-1-azaspiro[3.5]nona-5,8-diene-3-carboxylate

$C_{21}H_{23}NO_6$ Mol. Wt.: 385.41

5m: (ref. 3a) Pale yellow syrup, Yield: 71%. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 6.75-6.59 (4H, *m*), 6.47 (1H, *dd*, *J* = 3.0, 9.9 Hz), 6.30-6.21 (2H, *m*), 4.12 (2H, *q*, *J* = 7.2 Hz), 4.03 (1H, *s*), 3.81 (3H, *s*), 3.80 (3H, *s*), 3.31 (2H, *t*, *J* = 7.2 Hz), 2.79 (2H, *t*, *J* = 7.2 Hz), 1.18 (3H, *t*, *J* = 7.2 Hz). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 183.94, 164.65, 160.81, 148.97, 147.89, 146.13, 143.67, 132.85, 131.95, 129.93, 120.68, 111.93, 111.34, 62.95, 62.00, 57.25, 55.85, 55.80, 43.29, 33.85, 13.98. **EIMS** *m*/*z* (%) : 386 (M^++1, 4%), 385 (M^+, 17%), 271 (4), 240 (3), 223 (11), 207 (2), 180 (4), 164 (84), 151 (100), 149 (7), 133 (9), 122 (6), 107 (5), 77 (3). **HRMS** *m*/*z* Found: 408.1423, Calcd. for $C_{21}H_{23}NO_6Na$ $(M+Na)^+$: 408.1423.

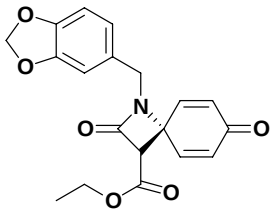


5n

ethyl 1-benzyl-2,7-dioxo-1-azaspiro[3.5]nona-5,8-diene-3-carboxylate

$C_{18}H_{17}NO_4$ Mol. Wt.: 311.33

5n: (ref. 3a) Dark green syrup, Yield: 74%. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.28-7.11 (5H, *m*), 6.77 (1H, *dd*, *J* = 2.7, 10.5 Hz), 6.59 (1H, *dd*, *J* = 2.7, 10.5 Hz), 6.22-6.14 (2H, *m*), 4.29 (2H, *s*), 4.15 (2H, *q*, *J* = 7.2 Hz), 4.14 (1H, *s*), 1.19 (3H, *t*, *J* = 7.2 Hz). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 184.14, 164.87, 160.44, 146.21, 143.63, 135.00, 132.85, 131.81, 129.02, 128.95, 128.52, 63.63, 62.29, 57.66, 45.78, 14.18. **EIMS** *m*/*z* (%) : 312 (M^++1, 2%), 311 (M^+, 10%), 265 (16), 239 (11), 238 (35), 223 (7), 197 (19), 196 (14), 179 (51), 168 (5), 150 (36), 133 (52), 132 (39), 122 (8), 105 (11), 91 (100), 77 (16). **HRMS** *m*/*z* Found: 312.1240, Calcd. for $C_{18}H_{18}NO_4$ $(M+H)^+$: 312.1235.

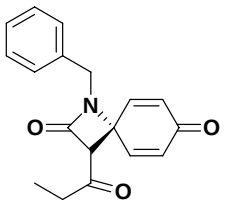


5o

ethyl 1-(benzo[*d*][1,3]dioxol-5-ylmethyl)-2,7-dioxo-1-azaspiro[3.5]nona-5,8-diene-3-carboxylate

$C_{19}H_{17}NO_6$ Mol. Wt.: 355.34

5o: (ref. 3a) Pale yellow syrup, Yield: 76%. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 6.83 (1H, *dd*, *J* = 3.0, 9.9 Hz), 6.72-6.55 (4H, *m*), 6.30-6.21 (2H, *m*), 5.93 (2H, *s*), 4.22 (2H, *s*), 4.18 (2H, *q*, *J* = 7.2 Hz), 4.11 (1H, *s*), 1.24 (3H, *t*, *J* = 7.2 Hz). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 184.18, 164.87, 160.38, 148.18, 147.82, 146.29, 143.71, 132.83, 131.77, 128.70, 122.66, 109.20, 108.40, 101.40, 63.52, 62.30, 57.59, 45.52, 14.18. **EIMS** *m*/*z* (%) : 356 (M^++1, 6%), 355 (M^+, 28%), 281 (2), 253 (3), 241 (19), 240 (12), 207 (2), 177 (27), 150 (11), 147 (8), 135 (100), 133 (12), 119 (3), 105 (2), 77 (13). **HRMS** *m*/*z* Found: 378.0954, Calcd. for $C_{19}H_{17}NO_6Na$ $(M+Na)^+$: 378.0953.

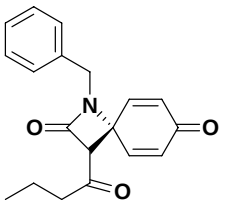


5p

1-benzyl-3-propionyl-1-azaspiro[3.5]nona-5,8-diene-2,7-dione

$C_{18}H_{17}NO_3$ Mol. Wt.: 295.33

5p: yellow oil, Yield: 82%. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.42-7.13 (5H, *m*), 6.76 (1H, *dd*, *J* = 3.0, 9.9 Hz), 6.57 (1H, *dd*, *J* = 3.0, 9.9 Hz), 6.26 (1H, *dd*, *J* = 1.8, 10.2 Hz), 6.22 (1H, *dd*, *J* = 1.8, 10.2 Hz), 4.34 (1H, *d*, *J* = 14.7 Hz), 4.33 (1H, *s*), 4.29 (1H, *d*, *J* = 14.7 Hz), 2.74-2.36 (2H, *m*), 1.04 (3H, *t*, *J* = 7.2 Hz). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 200.87, 184.38, 161.56, 146.21, 144.76, 134.98, 131.82, 131.69, 128.98, 128.89, 128.46, 70.00, 58.17, 45.68, 37.16, 7.02. **+TOF MS** *m*/*z* (%) : 318 (M^++Na, 100%), 296 (M^++H, 84%), 279 (22), 240 (5), 220 (2), 198 (11), 163 (30), 123 (19). **HRMS** *m*/*z* Found: 296.1278, Calcd. for $C_{18}H_{18}NO_3$ $(M+H)^+$: 296.1286.

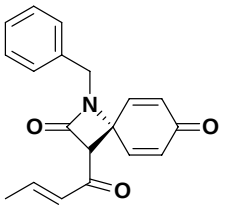


5q

1-benzyl-3-butyryl-1-azaspiro[3.5]nona-5,8-diene-2,7-dione

$C_{19}H_{19}NO_3$ Mol. Wt.: 309.36

5q: pale yellow syrup, Yield: 81%. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.42-7.14 (5H, *m*), 6.76 (1H, *dd*, *J* = 3.0, 9.9 Hz), 6.57 (1H, *dd*, *J* = 3.0, 9.9 Hz), 6.25 (1H, *dd*, *J* = 2.1, 10.0 Hz), 6.22 (1H, *dd*, *J* = 2.1, 10.0 Hz), 4.37 (2H, *s*), 4.30 (1H, *s*), 2.63-2.32 (2H, *m*), 1.69-1.51 (2H, *m*), 0.90 (3H, *t*, *J* = 7.2 Hz). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 200.44, 184.40, 161.57, 146.24, 144.80, 135.00, 131.81, 128.98, 128.89, 128.46, 70.13, 58.19, 45.65, 16.42, 13.56. **+TOF MS** *m*/*z* (%) : 332 (M^++Na, 33%), 310 (M^++H, 100%), 288 (12), 274 (20), 240 (9), 198 (22), 177 (60), 149 (29). **HRMS** *m*/*z* Found: 310.1435, Calcd. for $C_{19}H_{20}NO_3$ $(M+H)^+$: 310.1443.

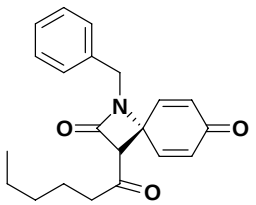


5r

(*E*)-1-benzyl-3-but-2-enoyl-1-azaspiro[3.5]nona-5,8-diene-2,7-dione

$C_{19}H_{17}NO_3$ Mol. Wt.: 307.34

5r: yellow oil, Yield: 85%. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.36-7.14 (5H, *m*), 6.89-6.70 (1H, *m*), 6.69 (1H, *d*, *J* = 3.0, 10.2 Hz), 6.63 (1H, *dd*, *J* = 3.0, 10.2 Hz), 6.27-6.20 (2H, *m*), 6.18 (1H, *dd*, *J* = 1.5, 15.6 Hz), 4.54 (1H, *s*), 4.36 (1H, *d*, *J* = 15.6 Hz), 4.28 (1H, *d*, *J* = 15.6 Hz), 1.89 (3H, *dd*, *J* = 1.5, 6.9 Hz). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 189.67, 184.26, 161.67, 147.08, 146.35, 144.73, 135.00, 132.00, 131.74, 131.06, 128.94, 128.82, 128.37, 67.86, 58.21, 45.63, 18.64. **+TOF MS** *m*/*z* (%) : 330 (M^++Na, 87%), 308 (M^++H, 100%), 279 (69), 205 (7), 198 (12), 175 (29), 160 (1), 149 (98), 133 (3), 123 (32), 119 (5). **HRMS** *m*/*z* Found: 308.1282, Calcd. for $C_{19}H_{18}NO_3$ $(M+H)^+$: 308.1286.

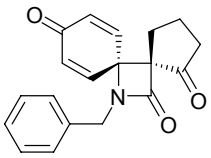


5s

1-benzyl-3-hexanoyl-1-azaspiro[3.5]nona-5,8-diene-2,7-dione

$C_{21}H_{23}NO_3$ Mol. Wt.: 337.41

5s: yellow oil, Yield: 82%. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.37-7.24 (3H, *m*), 7.22-7.13 (2H, *m*), 6.75 (1H, *dd*, *J* = 3.0, 10.2 Hz), 6.56 (1H, *dd*, *J* = 3.0, 9.9 Hz), 6.26 (1H, *dd*, *J* = 2.1, 10.2 Hz), 6.22 (1H, *dd*, *J* = 2.1, 9.9 Hz), 4.34 (1H, *d*, *J* = 15.0 Hz), 4.31 (1H, *s*), 4.27 (1H, *d*, *J* = 15.0 Hz), 2.66-2.35 (2H, *m*), 1.62-1.48 (2H, *m*), 1.39-1.19 (4H, *m*), 0.89 (3H, *t*, *J* = 7.2 Hz). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 200.59, 184.44, 161.61, 146.25, 144.85, 135.05, 131.85, 131.75, 129.04, 128.95, 128.51, 70.22, 58.25, 45.72, 43.82, 31.14, 22.65, 22.44, 13.92. **+TOF MS** *m*/*z* (%) : 360 (M^++Na, 6%), 338 (M^++H, 10%), 301 (5), 279 (3), 240 (1), 205 (4), 198 (3), 163 (1), 123 (100), 107 (2). **HRMS** *m*/*z* Found: 338.1755, Calcd. for $C_{21}H_{24}NO_3$ $(M+H)^+$: 338.1756.

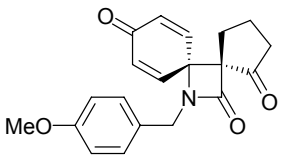


13a

azaspirocyclic compound **13a**

$C_{19}H_{17}NO_3$ Mol. Wt.: 307.34

13a: yellow syrup, Yield: 87%. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.35-7.10 (6H, *m*), 6.52 (1H, *dd*, *J* = 3.0, 10.2 Hz), 6.26 (1H, *dd*, *J* = 2.0, 10.2 Hz), 6.16 (1H, *dd*, *J* = 2.0, 10.2 Hz), 4.34 (2H, *s*), 2.53-2.30 (2H, *m*), 2.23-2.00 (3H, *m*), 1.95-1.80 (1H, *m*). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 210.76, 184.11, 156.22, 145.73, 144.98, 135.00, 132.45, 131.59, 128.86, 128.83, 128.34, 75.67, 60.71, 45.55, 38.07, 28.68, 19.59. **EIMS** *m*/*z* (%) : 309 (M^++2, 36%), 307 (M^+, 60%), 279 (11), 278 (6), 252 (48), 239 (13), 222 (6), 210 (7), 199 (59), 197 (52), 188 (8), 174 (35), 168 (12), 161 (9), 146 (21), 132 (22), 121 (29), 110 (19), 108 (15), 91 (100), 77 (23). **HRMS** *m*/*z* Found: 307.1216, Calcd. for $C_{19}H_{17}NO_3$ $(M)^+$: 307.1208.

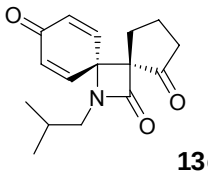


13b

azaspirocyclic compound **13b**

$C_{20}H_{19}NO_4$ Mol. Wt.: 337.37

13b: yellow powder, Yield: 85%, mp: 132-133 °C. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.10 (1H, *dd*, *J* = 2.8, 10.3 Hz), 7.03 (2H, *d*, *J* = 8.5 Hz), 6.73 (2H, *d*, *J* = 8.5 Hz), 6.46 (1H, *dd*, *J* = 2.9, 10.2 Hz), 6.18 (1H, *dd*, *J* = 1.8, 10.2 Hz), 6.08 (1H, *dd*, *J* = 1.8, 10.3 Hz), 4.21 (2H, *s*), 3.70 (3H, *s*), 2.45-2.21 (2H, *m*), 2.12-1.90 (3H, *m*), 1.82-1.70 (1H, *m*). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 210.93, 184.23, 165.15, 159.62, 145.98, 145.19, 132.23, 131.38, 130.25, 126.96, 114.12, 75.46, 60.53, 55.24, 44.93, 38.04, 28.63, 19.58. **EIMS** *m*/*z* (%) : 337 (M^+, 82%), 309 (14), 281 (13), 252 (11), 247 (22), 229 (81), 217 (21), 202 (28), 189 (13), 174 (32), 161 (25), 146 (35), 135 (27), 121 (100), 109 (42), 91 (50), 77 (61). **HRMS** *m*/*z* Found: 337.1313, Calcd. for $C_{20}H_{19}NO_4$ $(M)^+$: 337.1314.

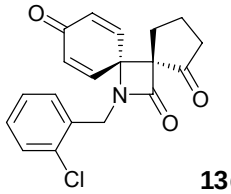


13c

azaspirocyclic compound **13c**

$C_{16}H_{19}NO_3$ Mol. Wt.: 273.33

13c: brown syrup, Yield: 82%. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.45 (1H, *dd*, *J* = 3.0, 10.2 Hz), 6.81 (1H, *dd*, *J* = 3.0, 10.2 Hz), 6.49 (1H, *dd*, *J* = 2.1, 10.2 Hz), 6.36 (1H, *dd*, *J* = 2.1, 10.2 Hz), 3.06 (1H, *dd*, *J* = 8.1, 14.1 Hz), 2.80 (1H, *dd*, *J* = 6.9, 14.1 Hz), 2.60-2.30 (2H, *m*), 2.24-1.73 (5H, *m*), 0.98 (3H, *d*, *J* = 6.9 Hz), 0.94 (3H, *d*, *J* = 6.9 Hz). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 211.17, 184.08, 165.98, 146.06, 145.33, 133.23, 131.99, 75.24, 60.52, 49.37, 38.14, 28.62, 27.94, 20.14, 20.01, 19.66. **+TOF MS** *m*/*z* (%) : 296 (M^++Na, 3%), 274 (M^++H, 7%), 175 (8), 164 (100), 133 (2), 111 (4), 108 (15). **HRMS** *m*/*z* Found: 274.1438, Calcd. for $C_{16}H_{20}NO_3$ $(M+H)^+$: 274.1443.

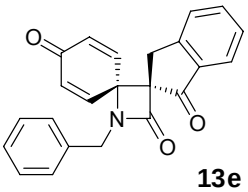


13d

azaspirocyclic compound **13d**

$C_{19}H_{16}ClNO_3$ Mol. Wt.: 341.79

13d: yellow syrup, Yield: 93%. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.51-7.10 (5H, *m*), 6.57 (1H, *dd*, *J* = 3.0, 10.2 Hz), 6.26-6.10 (2H, *m*), 4.53 (1H, *d*, *J* = 15.6 Hz), 4.47 (1H, *d*, *J* = 15.6 Hz), 2.60-2.28 (2H, *m*), 2.22-1.98 (3H, *m*), 1.92-1.79 (1H, *m*). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 210.97, 184.17, 165.28, 145.49, 144.75, 134.04, 132.43, 132.29, 131.67, 131.28, 130.07, 129.72, 127.27, 75.55, 60.61, 43.01, 38.10, 28.73, 19.64. **EIMS** *m*/*z* (%) : 343 (M^+, 14%), 341 (M^+, 35%), 324 (6), 306 (11), 288 (13), 286 (33), 273 (11), 250 (15), 233 (33), 232 (16), 216 (5), 199 (9), 176 (19), 174 (12), 146 (13), 127 (50), 125 (100), 108 (27), 99 (6), 91 (14), 89 (23), 77 (13). **HRMS** *m*/*z* Found: 341.0824, Calcd. for $C_{19}H_{16}NO_3Cl$ $(M)^+$: 341.0819.

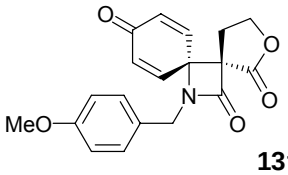


13e

azaspirocyclic compound **13e**

$C_{23}H_{17}NO_3$ Mol. Wt.: 355.39

13e: pale brown oil, Yield: 90%. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.74 (1H, *d*, *J* = 7.7 Hz), 7.62 (1H, *t*, *J* = 7.8 Hz), 7.52 (1H, *dd*, *J* = 3.0, 10.4 Hz), 7.48-7.22 (7H, *m*), 6.60 (1H, *dd*, *J* = 3.0, 10.2 Hz), 6.27 (1H, *dd*, *J* = 2.0, 10.2 Hz), 6.14 (1H, *dd*, *J* = 2.0, 10.4 Hz), 4.48 (1H, *d*, *J* = 15.0 Hz), 4.37 (1H, *d*, *J* = 15.0 Hz), 3.66 (1H, *d*, *J* = 17.8 Hz), 3.25 (1H, *d*, *J* = 17.8 Hz). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 197.99, 184.30, 165.09, 151.29, 146.28, 144.26, 136.19, 135.41, 135.02, 132.40, 131.87, 128.87, 128.37, 126.33, 124.84, 74.92, 60.88, 45.76, 31.01. **EIMS** *m*/*z* (%) : 356 (M^++1, 78%), 355 (M^+, 87%), 327 (78), 325 (52), 310 (28), 308 (20), 290 (26), 264 (45), 250 (26), 236 (68), 221 (32), 208 (30), 199 (67), 198 (64), 180 (31), 165 (40), 158 (79), 152 (29), 130 (58), 102 (71), 91 (100), 77 (38). **HRMS** *m*/*z* Found: 355.1210, Calcd. for $C_{23}H_{17}NO_3$ $(M)^+$: 355.1208.

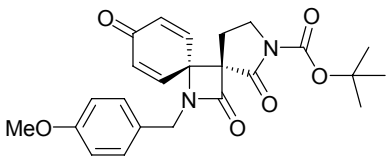


13f

azaspirocyclic compound **13f**

$C_{19}H_{17}NO_5$ Mol. Wt.: 339.34

13f: Pale yellow syrup, Yield: 87%. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.27 (1H, *dd*, *J* = 3.0, 10.2 Hz), 7.04 (2H, *dt*, *J* = 1.8, 8.7 Hz), 6.75 (2H, *dt*, *J* = 1.8, 8.7 Hz), 6.39 (1H, *dd*, *J* = 3.0, 9.9 Hz), 6.23 (1H, *dd*, *J* = 1.8, 10.2 Hz), 6.18 (1H, *dd*, *J* = 1.8, 9.9 Hz), 4.33 (1H, *ddd*, *J* = 7.5, 8.4, 9.0 Hz), 4.26 (2H, *s*), 4.13 (1H, *ddd*, *J* = 5.1, 8.4, 9.0 Hz), 3.72 (3H, *s*), 2.69 (1H, *ddd*, *J* = 5.1, 7.5, 12.6 Hz), 2.34 (1H, *ddd*, *J* = 7.2, 7.5, 12.6 Hz). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 183.81, 170.81, 162.62, 159.80, 144.61, 143.96, 132.96, 132.09, 130.35, 126.48, 114.24, 68.21, 65.83, 59.91, 55.30, 45.24, 27.25. **+TOF MS** *m*/*z* (%) : 362 (M^++Na, 70%), 321 (7), 301 (6), 274 (3), 250 (4), 228 (100), 209 (1), 198 (1), 167 (1), 135 (7), 121 (69). **HRMS** *m*/*z* Found: 362.0995, Calcd. for $C_{19}H_{17}NO_5Na$ $(M+Na)^+$: 362.1004.

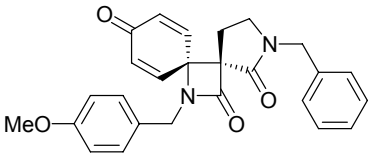


13g

azaspirocyclic compound **13g**

$C_{24}H_{26}N_2O_6$ Mol. Wt.: 438.47

13g: white plates, Yield: 84%, mp: 131-132 °C. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.45 (1H, *dd*, *J* = 3.0, 10.2 Hz), 7.11 (2H, *d*, *J* = 8.4 Hz), 6.80 (2H, *d*, *J* = 8.4 Hz), 6.45 (1H, *dd*, *J* = 3.0, 10.2 Hz), 6.27 (1H, *dd*, *J* = 2.1, 10.2 Hz), 6.21 (1H, *dd*, *J* = 2.1, 10.2 Hz), 4.34 (1H, *d*, *J* = 15.0 Hz), 4.28 (1H, *d*, *J* = 15.0 Hz), 3.86-3.70 (1H, *m*), 3.79 (3H, *s*), 3.67-3.52 (1H, *m*), 2.50 (1H, *ddd*, *J* = 5.1, 7.8, 13.5 Hz), 2.12 (1H, *ddd*, *J* = 7.5, 7.8, 13.5 Hz), 1.54 (9H, *s*). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 184.22, 163.78, 159.67, 149.34, 145.29, 144.68, 132.75, 131.76, 130.29, 126.71, 114.17, 84.03, 72.66, 60.61, 55.29, 45.05, 43.48, 27.93, 22.64. **+TOF MS** *m*/*z* (%) : 461 (M^++Na, 20%), 361 (9), 346 (11), 339 (48), 318 (25), 302 (14), 290 (7), 274 (100), 262 (2), 231 (13), 121 (49). **HRMS** *m*/*z* Found: 461.1684, Calcd. for $C_{24}H_{26}N_2O_6Na$ $(M+Na)^+$: 461.1688.

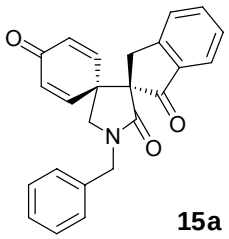


13h

azaspirocyclic compound **13h**

$C_{26}H_{24}N_2O_4$ Mol. Wt.: 428.47

13h: Pale yellow foam, Yield: 80%. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.52 (1H, *dd*, *J* = 3.0, 10.2 Hz), 7.33-7.03 (7H, *m*), 6.75 (2H, *d*, *J* = 8.7 Hz), 6.41 (1H, *dd*, *J* = 3.0, 10.2 Hz), 6.18 (1H, *dd*, *J* = 2.1, 10.2 Hz), 6.15 (1H, *dd*, *J* = 2.1, 10.2 Hz), 4.43 (1H, *d*, *J* = 15.0 Hz), 4.33 (1H, *d*, *J* = 15.0 Hz), 4.27 (1H, *s*), 3.72 (3H, *s*), 3.25-3.13 (1H, *m*), 3.09-2.98 (1H, *m*), 2.45 (1H, *ddd*, *J* = 4.8, 8.4, 13.5 Hz), 2.06 (1H, *ddd*, *J* = 5.7, 8.7, 13.5 Hz). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 184.52, 167.93, 165.15, 159.60, 146.15, 145.59, 135.15, 132.40, 131.34, 130.29, 128.89, 127.97, 127.02, 114.13, 71.62, 61.02, 55.29, 47.31, 45.03, 43.76, 22.94. **+TOF MS** *m*/*z* (%) : 451 (M^++Na, 70%), 429 (M^++H, 36%), 390 (3), 374 (3), 362 (15), 356 (2), 318 (11), 302 (2), 274 (26), 266 (38), 248 (56), 218 (9), 135 (18), 121 (65). **HRMS** *m*/*z* Found: 451.1636, Calcd. for $C_{26}H_{24}N_2O_4Na$ $(M+Na)^+$: 451.1633.



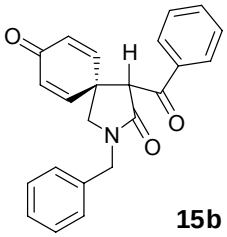
15a

azaspirocyclic compound **15a**

$C_{24}H_{19}NO_3$ Mol. Wt.: 369.41

15a: brown syrup, Yield: 87%. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.65 (1H, *d*, *J* = 6.3 Hz), 7.56 (1H, *t*, *J* = 7.5 Hz), 7.44-7.30 (7H, *m*), 6.93 (1H, *dd*, *J* = 3.0, 10.2 Hz), 6.74 (1H, *dd*, *J* = 3.0, 10.2 Hz), 6.30 (1H, *dd*, *J* = 1.8, 10.2 Hz), 6.21 (1H, *dd*, *J* = 1.8, 10.2 Hz), 4.61 (1H, *d*, *J* = 15.0 Hz), 4.52 (1H, *d*, *J* = 15.0 Hz), 4.21 (1H, *d*, *J* = 9.8 Hz), 3.45 (1H, *d*, *J* = 17.4 Hz), 2.98 (1H, *d*, *J* = 9.8 Hz), 2.68 (1H, *d*, *J* = 17.4 Hz). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 201.64, 184.69, 170.71, 154.04, 147.97, 145.59, 136.17, 135.21, 134.17, 133.14, 130.08, 128.96, 128.29, 128.12, 127.83, 126.46, 124.85, 65.24, 51.44, 48.38, 47.60, 31.71. **+TOF MS** *m*/*z* (%) : 392 (M^++Na, 24%), 370 (M^++H,

92%), 358 (100), 342 (6), 330 (5), 318 (17), 302 (10), 274 (80), 262 (4), 246 (2), 218 (1), 212 (5), 130 (11), 102 (25). **HRMS** *m/z* Found: 370.1434, Calcd. for $C_{24}H_{20}NO_3$ $(M+H)^+$: 370.1443.

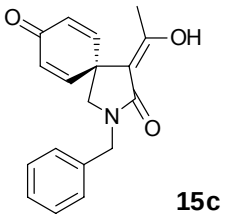


15b

azaspirocyclic compound **15a**

$C_{23}H_{19}NO_3$ Mol. Wt.: 357.41

15b: yellow plates, as a mixture of keto and enol (5%) tautomers, Yield: 90%, mp: 124-125 °C. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.86 (2H, *d*, *J* = 7.2 Hz), 7.61 (1H, *t*, *J* = 7.2 Hz), 7.60-7.18 (7H, *m*), 6.97 (1H, *dd*, *J* = 3.0, 9.9 Hz), 6.87 (1H, *dd*, *J* = 3.0, 10.2 Hz), 6.30 (1H, *dd*, *J* = 1.8, 9.9 Hz), 6.17 (1H, *dd*, *J* = 1.8, 10.2 Hz), 4.66 (1H, *d*, *J* = 14.7 Hz), 4.57 (1H, *s*), 4.54 (1H, *d*, *J* = 14.7 Hz), 3.73 (1H, *d*, *J* = 9.9 Hz), 3.18 (1H, *d*, *J* = 9.9 Hz). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 194.92, 184.47, 169.30, (150.34), 148.79, 146.80, 136.29, 135.15, 134.37, 131.13, 128.99, 128.93, 128.23, 128.13, (127.80, 127.37), 57.71, 53.26, 47.11, 44.37. **+TOF MS** *m/z* (%) : 380 (M^++Na, 45%), 358 (M^++H, 100%), 318 (13), 274 (43), 219 (10), 206 (12), 195 (9), 163 (7), 151 (28), 144 (22), 136 (44), 114 (6), 109 (9), 105 (35). **HRMS** *m/z* Found: 358.1449, Calcd. for $C_{23}H_{20}NO_3$ $(M+H)^+$: 358.1443.

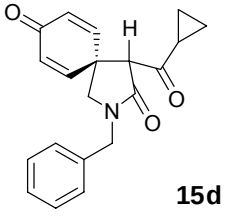


15c

azaspirocyclic compound **15c**

$C_{18}H_{17}NO_3$ Mol. Wt.: 295.33

15c: yellow powder, as a mixture of keto (38%) and enol tautomers, Yield: 76%, mp: 121-122 °C. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 12.46 (1H, *s*), 7.45-7.21 (5H, *m*), 6.83 (2H, *d*, *J* = 9.9 Hz), 6.27 (2H, *d*, *J* = 9.9 Hz), 4.53 (2H, *s*), 3.30 (2H, *s*), 1.71 (3H, *s*). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): (202.24), 184.88 (184.52), 171.65, (168.24), 167.57, 150.09, 148.43, 146.33, 135.32 (135.03), 130.95, 129.64, 128.99, 128.21, 128.13, 128.09, 99.81, 62.94, 53.43 (52.93), (47.06), 46.39, (44.04), 42.49, 32.39, 17.38. **+TOF MS** *m/z* (%) : 318 (M^++Na, 10%), 296 (M^++H, 100%), 274 (1), 262 (1), 151 (6), 120 (2). **HRMS** *m/z* Found: 296.1282, Calcd. for $C_{18}H_{18}NO_3$ $(M+H)^+$: 296.1286.

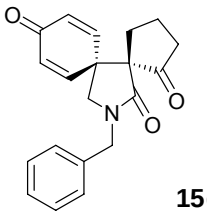


15d

azaspirocyclic compound **15d**

$C_{20}H_{19}NO_3$ Mol. Wt.: 321.37

15d: Pale yellow oil, as a mixture of keto and enol (22%) tautomers, Yield: 90%. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 12.58 (0.22 H, *s*), 7.48-7.18 (5H, *m*), 7.02-6.80 (2H, *m*), 6.31 (2H, *d*, *J* = 9.9 Hz), 4.61 (1H, *d*, *J* = 14.7 Hz), 4.52 (1H, *d*, *J* = 14.7 Hz), 3.84 (1H, *s*), 3.49 (1H, *d*, *J* = 10.2 Hz), 3.18 (1H, *d*, *J* = 10.2 Hz), 2.14-2.01 (1H, *m*), 1.28-0.92 (4H, *m*). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 204.28, (185.12), 184.60, (171.85, 171.12), 168.69, 151.33, 148.70, 146.83, (135.48), 135.12, 130.78, 129.47, 128.97, 128.21, 128.12, (128.00), 127.78, (98.44), 62.97, (53.60), 52.91, 47.02, (46.36), 44.17, (42.71), 22.71, 12.68, (11.16), (7.21). **+TOF MS** *m*/*z* (%) : 344 (M^++Na, 20%), 322 (M^++H, 100%), 302 (2), 274 (13). **HRMS** *m*/*z* Found: 322.1447, Calcd. for $C_{20}H_{20}NO_3$ $(M+H)^+$: 322.1443.

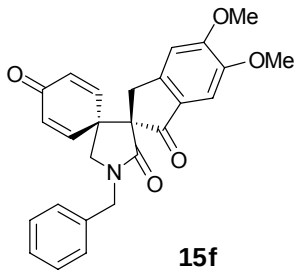


15e

azaspirocyclic compound **15e**

$C_{20}H_{19}NO_3$ Mol. Wt.: 321.37

15e: white plates, Yield: 85%, mp: 128-129 °C. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.41-7.22 (5H, *m*), 6.88 (1H, *dd*, *J* = 3.0, 10.2 Hz), 6.65 (1H, *dd*, *J* = 3.0, 10.2 Hz), 6.38 (1H, *dd*, *J* = 1.8, 10.2 Hz), 6.32 (1H, *dd*, *J* = 1.8, 10.2 Hz), 4.60 (1H, *d*, *J* = 14.7 Hz), 4.53 (1H, *d*, *J* = 14.7 Hz), 3.98 (1H, *d*, *J* = 9.6 Hz), 2.95 (1H, *d*, *J* = 9.6 Hz), 2.49-2.11 (4H, *m*), 1.92-1.78 (1H, *m*), 1.71-1.56 (1H, *m*). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 214.95, 184.57, 170.94, 147.59, 145.62, 135.25, 133.10, 129.85, 128.90, 128.16, 128.04, 64.55, 51.36, 47.89, 47.23, 38.67, 28.61, 19.76. **+TOF MS** *m*/*z* (%) : 344 (M^++Na, 8%), 322 (M^++H, 100%), 274 (2), 188 (3), 155 (1). **HRMS** *m*/*z* Found: 322.1435, Calcd. for $C_{20}H_{20}NO_3$ $(M+H)^+$: 322.1443.



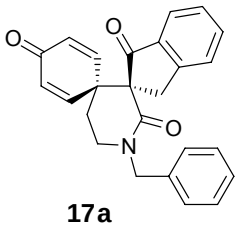
15f

azaspirocyclic compound **15f**

$C_{26}H_{23}NO_5$ Mol. Wt.: 429.46

15f: yellow plates, Yield: 85%, mp: 132-133 °C. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.46-7.30 (5H, *m*), 7.10 (1H, *s*), 7.02 (1H, *dd*, *J* = 3.0, 10.2 Hz), 6.83 (1H, *s*), 6.81 (1H, *dd*, *J* = 3.0, 10.2 Hz), 6.33 (1H, *dd*, *J* = 1.8, 10.2 Hz), 6.26 (1H, *dd*, *J* = 1.8, 10.2 Hz), 4.69 (1H, *d*, *J* = 15.0 Hz), 4.55 (1H, *d*, *J* = 15.0 Hz), 4.27 (1H, *d*, *J* = 9.9 Hz), 3.93 (3H, s), 3.88 (3H, s), 3.39 (1H, *d*, *J* = 17.1 Hz), 3.00 (1H, *d*, *J* = 9.9 Hz), 2.63 (1H, *d*, *J* = 17.1 Hz). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 199.63, 184.79, 171.08, 156.74, 149.97, 148.26, 146.00, 135.30, 132.98, 129.87, 128.91, 128.25, 128.04, 126.77, 107.23, 104.72, 77.30, 65.46, 56.38, 56.13, 51.45, 48.33, 47.53, 31.42. **+TOF MS** *m*/*z* (%) :

452 (M^++Na, 48%), 430 (M^++H, 100%), 274 (23), 242 (10), 149 (46), 136 (27), 123 (30), 118 (10), 114 (52), 109 (7), 105 (45). **HRMS** *m/z* Found: 430.1651, Calcd. for $C_{26}H_{24}NO_5$ $(M+H)^+$: 430.1654.

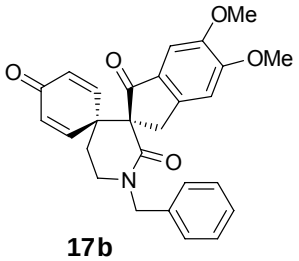


17a

azaspirocyclic compound **17a**

$C_{25}H_{21}NO_3$ Mol. Wt.: 383.44

17a: pale brown oil, Yield: 82%. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.68 (1H, *d*, *J* = 7.5 Hz), 7.59 (1H, *t*, *J* = 7.5 Hz), 7.42-7.18 (7H, *m*), 6.86 (1H, *dd*, *J* = 3.0, 10.2 Hz), 6.51 (1H, *dd*, *J* = 3.0, 10.2 Hz), 6.38 (1H, *dd*, *J* = 1.2, 10.2 Hz), 6.12 (1H, *dd*, *J* = 1.2, 10.2 Hz), 4.78 (1H, *d*, *J* = 14.7 Hz), 4.48 (1H, *d*, *J* = 14.7 Hz), 3.73 (1H, *d*, *J* = 17.1 Hz), 3.61-3.39 (3H, *m*), 2.57 (1H, *d*, *J* = 17.1 Hz), 1.52-1.39 (1H, *m*). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 202.56, 184.72, 166.55, 154.73, 148.99, 148.60, 136.37, 136.07, 134.36, 131.41, 131.13, 129.80, 128.61, 126.09, 124.92, 115.39, 59.77, 51.20, 46.35, 44.37, 35.49, 26.89. **+TOF MS** *m/z* (%) : 406 (M^++Na, 42%), 384 (M^++H, 69%), 362 (5), 348 (9), 334 (7), 318 (32), 302 (19), 274 (100), 262 (8), 228 (52), 121 (15). **HRMS** *m/z* Found: 406.1409, Calcd. for $C_{25}H_{21}NO_3Na$ $(M+Na)^+$: 406.1419.



17b

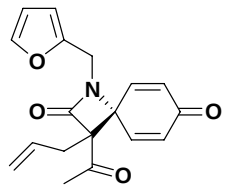
azaspirocyclic compound **17b**

$C_{27}H_{25}NO_5$ Mol. Wt.: 443.49

17b: pale yellow plates, Yield: 88%, mp: 134-135 °C. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.41-7.26 (5H, *m*), 7.13 (1H, *s*), 6.94 (1H, *dd*, *J* = 3.0, 10.2 Hz), 6.82 (1H, *s*), 6.62 (1H, *dd*, *J* = 3.0, 10.2 Hz), 6.43 (1H, *dd*, *J* = 1.2, 10.2 Hz), 6.20 (1H, *dd*, *J* = 1.2, 10.2 Hz), 4.88 (1H, *d*, *J* = 14.7 Hz), 4.53 (1H, *d*, *J* = 14.7 Hz), 3.95 (3H, *s*), 3.91 (3H, *s*), 3.67 (1H, *d*, *J* = 16.8 Hz), 3.65-3.45 (3H, *m*), 2.37 (1H, *d*, *J* = 16.8 Hz), 1.45-1.36 (1H, *m*). **^{13}C-NMR** (75MHz, $CDCl_3$), δ (ppm): 200.61, 184.82, 166.81, 156.64, 150.66, 149.77, 149.33, 148.81, 136.50, 131.25, 131.01, 128.86, 127.92, 127.70, 126.93, 106.86, 104.88, 59.95, 56.38, 56.13, 51.14, 46.30, 44.36, 35.18, 26.84. **+TOF MS** *m/z* (%) : 466 (M^++Na, 19%), 444 (M^++H, 100%), 249 (17), 158 (11), 150 (18), 125 (20), 123 (43), 118 (22), 114 (50). **HRMS** *m/z* Found: 444.1811, Calcd. for $C_{27}H_{26}NO_5$ $(M+H)^+$: 444.1810.

Preparation of 3-acetyl-3-allyl-1-benzyl-1-azaspiro[3.5]nona-5,8-diene-2,7-dione and 3-allyl-3-benzoyl-1-benzyl-1-azaspiro[3.5]nona-5,8-diene-2,7-dione by Tsuji-Trost reaction (5t and **5u):**

A mixture of compound **5c** or **5g** (135.6 mg or 171.6 mg, 0.50 mmol.), tetramethylguanidine (173 mg, 1.5 mmol, 3.0 eq.), triphenylphosphine (13 mg, 0.05 mmol, 0.1 eq.) and allylpalladium (II) chloride dimer (9.2 mg, 0.025 mmol, 0.05 eq.) in dichloromethane (15 mL) was degassed and purged with nitrogen (3 times) at 0 ºC. Allyl acetate (100 mg, 1 mmol, 2.0 eq.) in dichloromethane (1 mL) was added via syringe and the resulting mixture was then stirred at room temperature under nitrogen for 48 hours. The resulting mixture was diluted with dichloromethane (20 mL) and washed with saturated aqueous NH_4Cl solution (2 × 10 mL). The organic phase was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was chromatographed on silica gel (200-300 mesh, Petroleum ether : EtOAc = 4: 1-2:1) to afford azetidinone **5t** or **5u** (112 mg, 72% or 155 mg, 81%).

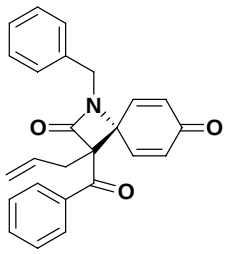


5t

3-acetyl-3-allyl-1-(furan-2-ylmethyl)-1-azaspiro[3.5]nona-5,8-diene-2,7-dione

$C_{18}H_{17}NO_4$ Mol. Wt.: 311.33

5t: Pale yellow oil. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.33 (1H, *d*, *J* = 0.6 Hz), 6.76 (1H, *dd*, *J* = 2.4, 10.2 Hz), 6.48 (1H, *dd*, *J* = 2.4, 10.2 Hz), 6.35 (1H, *dd*, *J* = 0.9, 10.2 Hz), 6.28 (1H, *s*), 6.26 (1H, *dd*, *J* = 0.9, 10.2 Hz), 6.20 (1H, *d*, *J* = 2.7 Hz), 5.90-5.70 (1H, *m*), 5.15 (2H, *d*, *J* = 12.3 Hz), 4.41 (1H, *d*, *J* = 15.9 Hz), 4.36 (1H, *d*, *J* = 15.9 Hz), 2.85 (1H, *dd*, *J* = 5.7, 14.4 Hz), 2.67 (1H, *dd*, *J* = 8.1, 14.4 Hz), 2.22 (3H, *s*). **^{13}C NMR** (75 MHz, $CDCl_3$) δ (ppm): 202.00, 183.99, 165.48, 147.54, 145.60, 143.42, 142.65, 132.84, 131.36, 130.78, 120.16, 110.85, 110.32, 79.22, 61.23, 37.10, 35.53, 29.57. **+TOF MS** *m*/*z* (%) : 334 (M^++Na, 100%), 312 (M^++H, 72%), 293 (6), 270 (3), 244 (20), 210 (2), 202 (2), 189 (12), 147 (10). **HRMS** *m*/*z* Found: 312.1239, Calcd. for $C_{18}H_{18}NO_4$ $(M+H)^+$: 312.1235.



5u

3-allyl-3-benzoyl-1-benzyl-1-azaspiro[3.5]nona-5,8-diene-2,7-dione

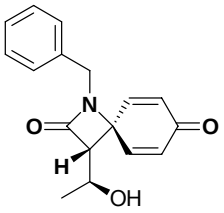
$C_{25}H_{21}NO_3$ Mol. Wt.: 383.44

5u: yellow syrup. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.69 (2H, *d*, *J* = 7.2 Hz), 7.52 (1H, *t*, *J* = 7.2 Hz), 7.42-7.15 (7H, *m*), 6.88 (1H, *dd*, *J* = 3.0, 10.5 Hz), 6.46 (1H, *dd*, *J* = 3.0, 10.5 Hz), 6.43 (1H, *dd*, *J* = 2.1, 10.2 Hz), 6.06 (1H, *dd*, *J* = 2.1, 10.2 Hz), 5.93-5.77 (1H, *m*), 4.98 (1H, *d*, *J* = 10.5 Hz), 4.78 (1H, *dd*, *J* = 1.2, 17.1 Hz), 4.45 (1H, *d*, *J* =

14.7 Hz), 4.28 (1H, *d*, *J* = 14.7 Hz), 3.02 (1H, *dd*, *J* = 6.3, 14.4 Hz), 2.89 (1H, *dd*, *J* = 8.1, 14.4 Hz). **^{13}C NMR** (75 MHz, $CDCl_3$) δ (ppm): 194.96, 183.96, 165.48, 146.74, 143.85, 135.35, 134.85, 133.72, 133.54, 131.86, 130.83, 128.94, 128.83, 128.56, 128.32, 120.29, 78.99, 61.85, 45.14, 37.39. **+TOF MS** *m*/*z* (%) : 406 (M^++Na, 54%), 384 (M^++H, 100%), 365 (4), 346 (4), 269 (10), 251 (3), 205 (5), 198 (13), 186 (4), 110 (41), 105 (20). **HRMS** *m*/*z* Found: 384.1596, Calcd. for $C_{25}H_{22}NO_3$ $(M+H)^+$: 384.1599.

Preparation of 1-benzyl-3-(1-hydroxyethyl)-1-azaspiro[3.5]nona-5,8-diene-2,7-dione (7a):

To a solution of β-lactam **5a** (281 mg, 1.0 mmol) in anhydrous ethanol and dichloromethane (20 mL, EtOH : DCM = 2:1) was added a powder of sodium borohydride (9.5 mg, 0.25 mmol, 0.25 eq.) at 0 ºC. The resulting mixture was then stirred at 0 ºC for 2 hours. After treatment with saturated aqueous NH_4Cl (2 mL) for 10 minutes, the resulting mixture was concentrated and the residue was diluted with ethyl acetate (20 mL). The resulting mixture was washed with water (2 × 5 mL) and the organic phase was dried over anhydrous Na_2SO_4. The solvent was removed and the residue was chromatographed on silica gel (200-300 mesh, Petroleum ether : EtOAc = 2:1) to afford the alcohol as a white powder (198 mg, 70%).



7a

1-benzyl-3-(1-hydroxyethyl)-1-azaspiro[3.5]nona-5,8-diene-2,7-dione

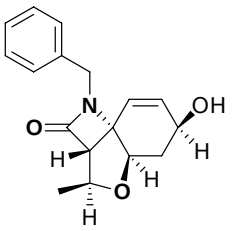
$C_{17}H_{17}NO_3$ Mol. Wt.: 283.32

7a: dr = 15 : 1 (based on ^{1}H-NMR) white plates, m.p.: 138-139 ºC. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.45-7.12 (5H, *m*), 7.06 (1H, *dd*, *J* = 3.0, 10.2 Hz), 6.59 (1H, *dd*, *J* = 3.0, 9.9 Hz), 6.28 (1H, *dd*, *J* = 2.1, 10.2 Hz), 6.17 (1H, *dd*, *J* = 2.1, 9.9 Hz), 4.41-4.21 (3H, *m*), 3.35 (1H, *d*, *J* = 7.5 Hz), 2.52 (1H, *d*, *J* = 4.8 Hz), 1.30 (3H, *d*, *J* = 6.3 Hz). **^{13}C NMR** (75 MHz, $CDCl_3$) δ (ppm): 184.85, 165.55, 148.30, 145.71, 135.47, 132.19, 130.77, 128.89, 128.76, 128.22, 68.34, 64.21, 58.96, 45.29, 21.58. **+TOF MS** *m*/*z* (%) : 306 (M^++Na, 50%), 284 (M^++H, 100%), 266 (44), 240 (2), 198 (9), 186 (3). **HRMS** *m*/*z* Found: 284.1283, Calcd. for $C_{17}H_{18}NO_3$ $(M+H)^+$: 284.1286.

Preparation of (2a,3,4a,6,8^1)-1-benzyl-6-hydroxy-3-methyl-2a,3,5,6-tetrahydro-1H-benzofuro[3-*b*]azet-2(4a*H*)-one and (2a,3,4a,6,8^1)-1-benzyl-6-hydroxy-3-phenyl-2a,3,5,6-tetrahydro-1H-benzofuro[3-*b*]azet-2(4a*H*)-one (7b and **7c):**

To a solution of β-lactam **5a** or **5g** (281 mg or 343 mg, 1.0 mmol) in anhydrous ethanol and dichloromethane (20 mL, EtOH : DCM = 2:1) was added a powder of sodium borohydride (38 mg, 1.0 mmol, 1.0 eq.) at 0 ºC. The

resulting mixture was then stirred at 0 ºC for 2 hours. After treatment with saturated aqueous NH_4Cl (2 mL) for 10 minutes, the resulting mixture was concentrated and the residue was diluted with ethyl acetate (20 mL). The resulting mixture was washed with water (2 × 5 mL) and the organic phase was dried over anhydrous Na_2SO_4. The solvent was removed and the residue was chromatographed on silica gel (200-300 mesh, Petroleum ether : EtOAc = 2:1) to afford compound **7b** or **7c** as a white powder (128 mg, 45% or 250 mg, 73%).

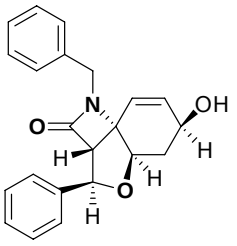


7b

(2a,3,4a,6,8^1)-1-benzyl-6-hydroxy-3-methyl-2a,3,5,6-tetrahydro-1*H*-benzofuro[3-*b*]azet-2(4a*H*)-one

$C_{17}H_{19}NO_3$ Mol. Wt.: 285.34

7b: dr > 99%, white powder, m.p.: 124-125 ºC. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.45-7.18 (5H, *m*), 6.09 (1H, *dd*, *J* = 2.4, 10.2 Hz), 5.65 (1H, *dd*, *J* = 1.5, 10.2 Hz), 4.52-4.40 (1H, *m*), 4.45 (1H, *d*, *J* = 15.0 Hz), 4.20-4.02 (1H, *m*), 4.11 (1H, *d*, *J* = 15.0 Hz), 3.81 (1H, *dd*, *J* = 5.1, 9.6 Hz), 3.26 (1H, *d*, *J* = 2.7 Hz),2.68-2.48 (1H, *m*), 2.00-1.72 (2H, *m*), 1.31 (3H, *d*, *J* = 6.6 Hz). **^{13}C NMR** (75 MHz, $CDCl_3$) δ (ppm): 167.19, 138.61, 135.58, 128.79, 128.58, 128.01, 125.33, 75.96, 74.39, 69.20, 68.64, 64.63, 44.46, 37.91, 23.27. **+TOF MS** *m*/*z* (%) : 308 (M^++Na, 59%), 286 (M^++H, 100%), 274 (20), 268 (9), 151 (1), 134 (8). **HRMS** *m*/*z* Found: 286.1435, Calcd. for $C_{17}H_{20}NO_3$ $(M+H)^+$: 286.1443.



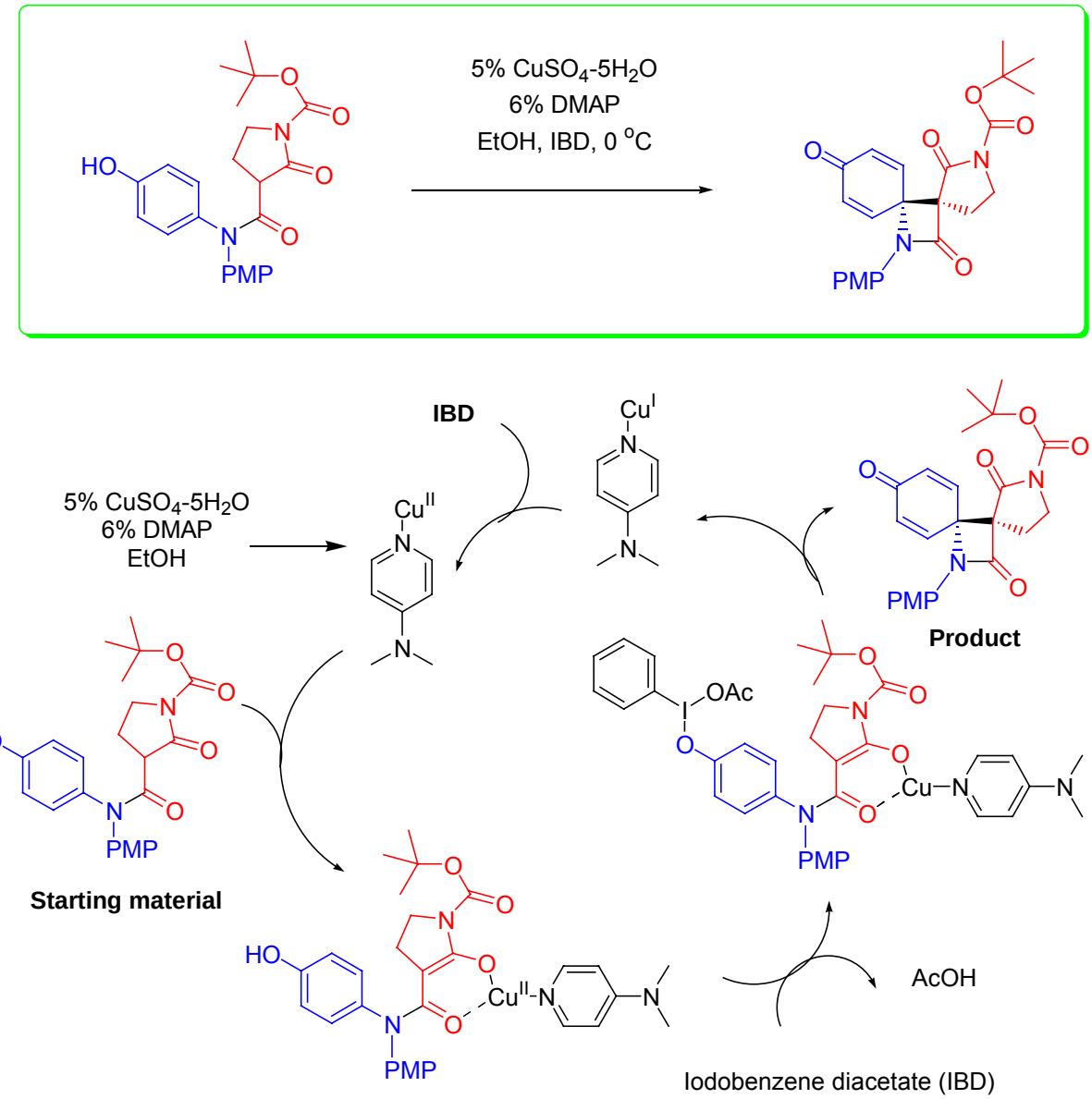
7c

(2a,3,4a,6,8^1)-1-benzyl-6-hydroxy-3-phenyl-2a,3,5,6-tetrahydro-1*H*-benzofuro[3-*b*]azet-2(4a*H*)-one

$C_{22}H_{21}NO_3$ Mol. Wt.: 347.41

7c: dr > 99%, white plates, m.p.: 127-128 ºC. **^{1}H-NMR** (300MHz, $CDCl_3$), δ (ppm): 7.44-7.22 (10H, *m*), 6.04 (1H, *dd*, *J* = 1.2, 9.9 Hz), 5.65 (1H, *dd*, *J* = 1.2, 9.9 Hz), 5.40 (1H, *d*, *J* = 2.7 Hz), 4.55 (1H, *d*, *J* = 15.0 Hz), 4.14 (1H, *d*, *J* = 15.0 Hz), 4.04 (1H, *brs*), 3.93 (1H, *dd*, *J* = 4.8, 9.9 Hz), 3.84 (1H, *d*, *J* = 2.7 Hz), 2.28-2.15 (1H, *m*), 1.88-1.75 (1H, *m*), 1.62-1.43 (1H, *m*). **^{13}C NMR** (75 MHz, $CDCl_3$) δ (ppm): 166.91, 140.86, 138.92, 135.57, 128.87, 128.63, 128.52, 128.09, 127.71, 125.79, 124.81, 78.52, 76.48, 69.11, 67.40, 64.85, 44.61, 37.28. **+TOF MS** *m*/*z* (%) : 370 (M^++Na, 100%), 348 (M^++H, 64%), 330 (1), 269 (2), 215 (11), 197 (22), 179 (4), 169 (14), 141 (1), 119 (3). **HRMS** *m*/*z* Found: 348.1593, Calcd. for $C_{22}H_{22}NO_3$ $(M+H)^+$: 348.1599.

Proposed mechanism for copper catalyzed oxidative Carbon-Carbon formation

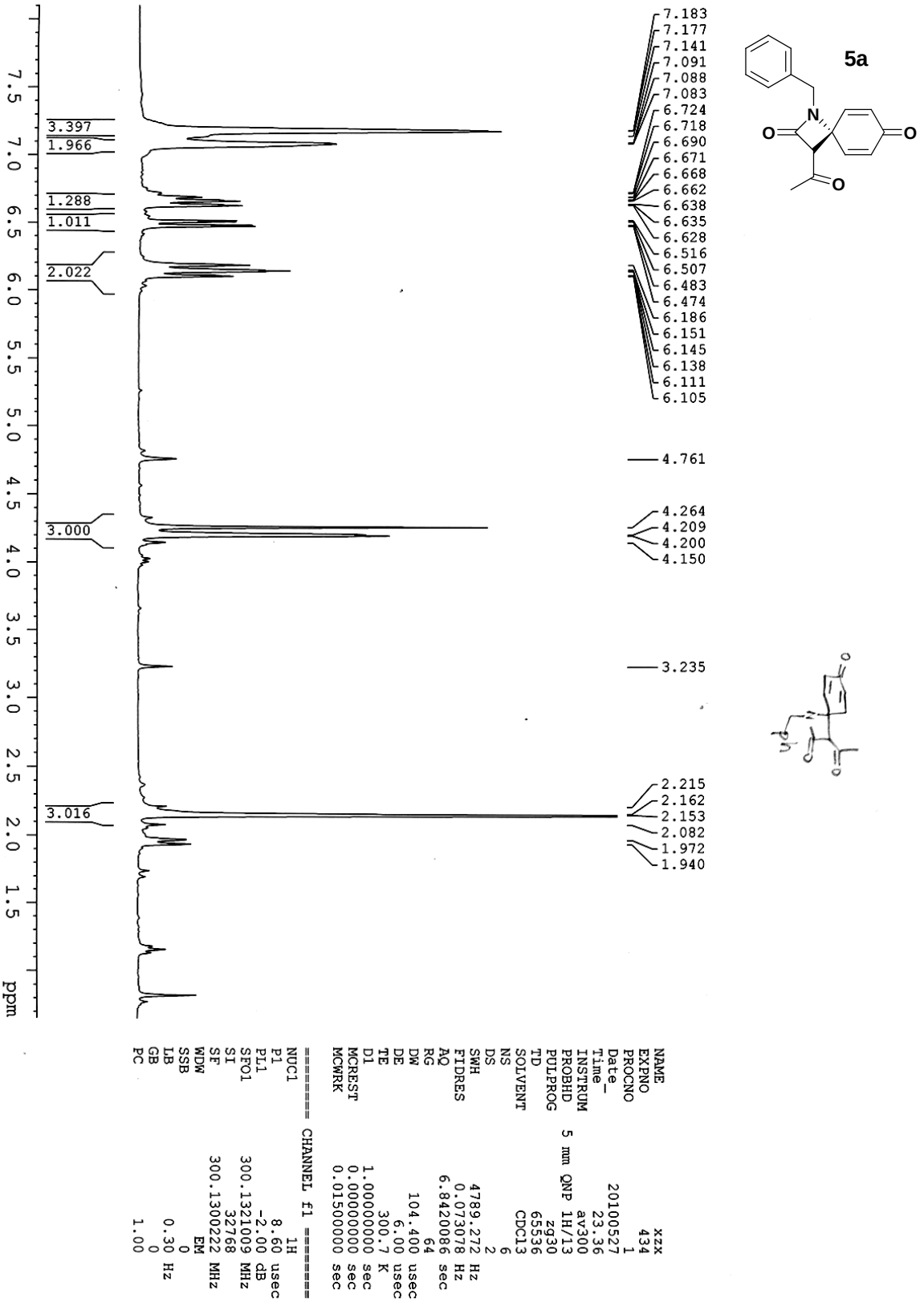


^{1}H NMR and ^{13}C NMR Spectrum for

Synthesis of Spirocyclic β-keto-Lactams: Copper Catalyzed Process

Zhongxuan Xu, Kun Huang, Tong Liu, Mingjin Xie, and Hongbin Zhang *

Key Laboratory of Medicinal Chemistry for Natural Resource, Ministry of Education, School of Chemical Science and Technology, Yunnan University, Kunming, Yunnan 650091, P. R. China Fax: 86-871-5035538. E-mail: zhanghb@ynu.edu.cn or zhang_hongbin@hotmail.com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6.638
6.635
6.628
6.516
6.507
6.483
6.474
6.186
6.151
6.145
6.138
6.111
6.105
4.761
4.264
4.209
4.200
4.150
3.235
2.215
2.162
2.153
2.082
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3.397
1.966
1.288
1.011
2.022
3.000
3.016
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FIDRES 0.073078 Hz
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MCREST 0.00000000 sec
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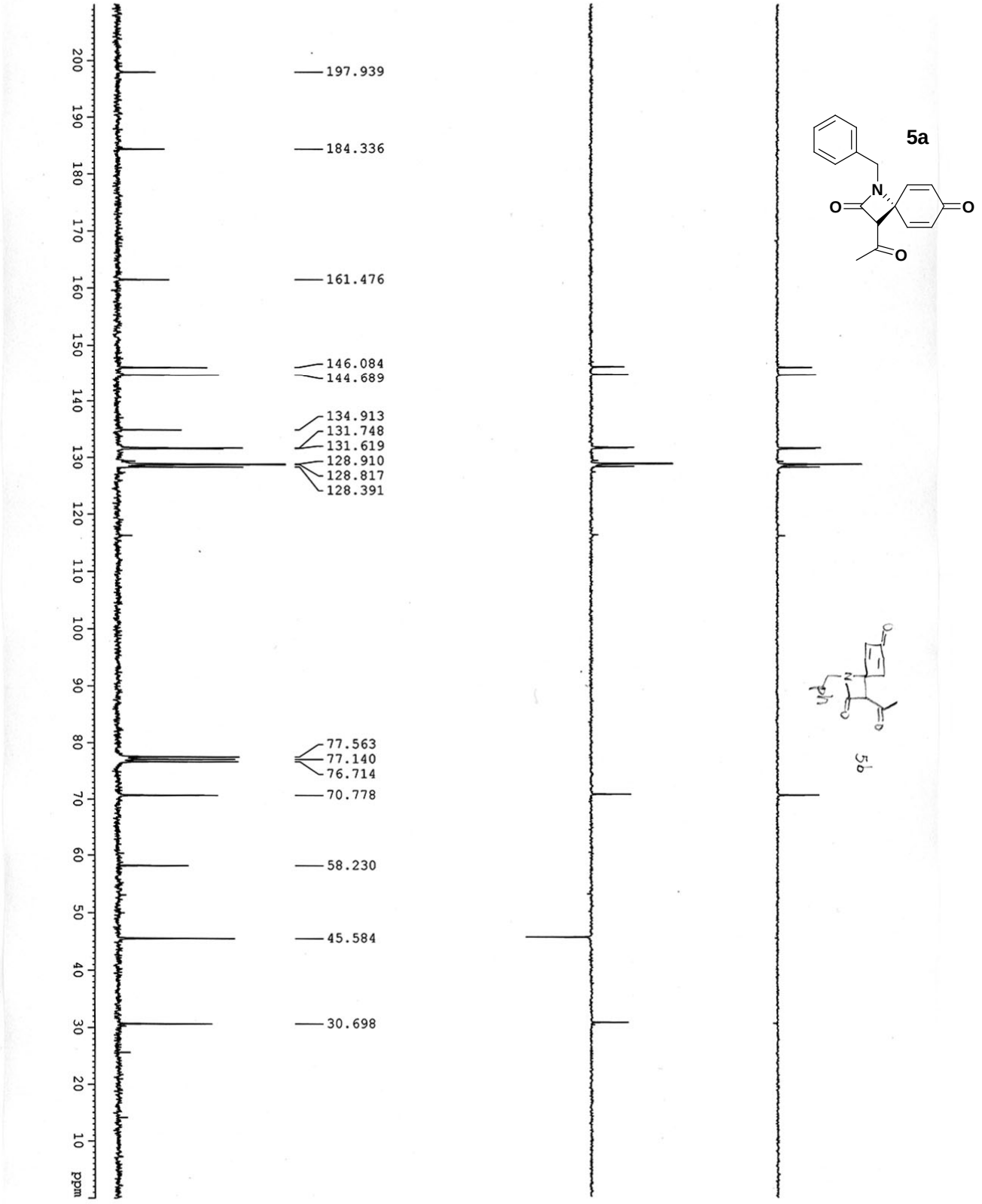
GB 0
PC 1.00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144.689
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76.714
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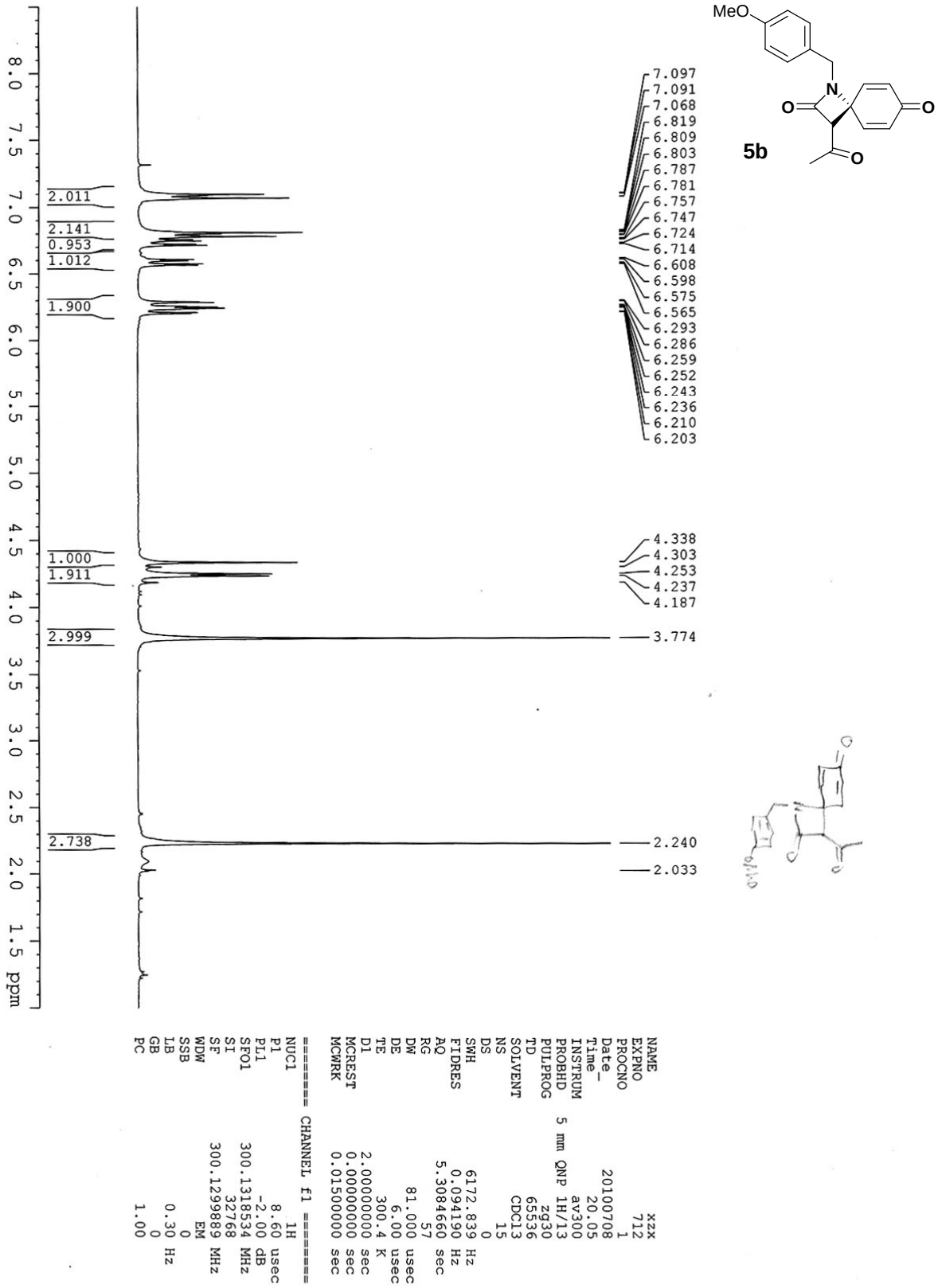
30.698
ppm
5a
5b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O
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6.757
6.747
6.724
6.714
6.608
6.598
6.575
6.565
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6.286
6.259
6.252
6.243
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6.203
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4.253
4.237
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2.011
2.141
0.953
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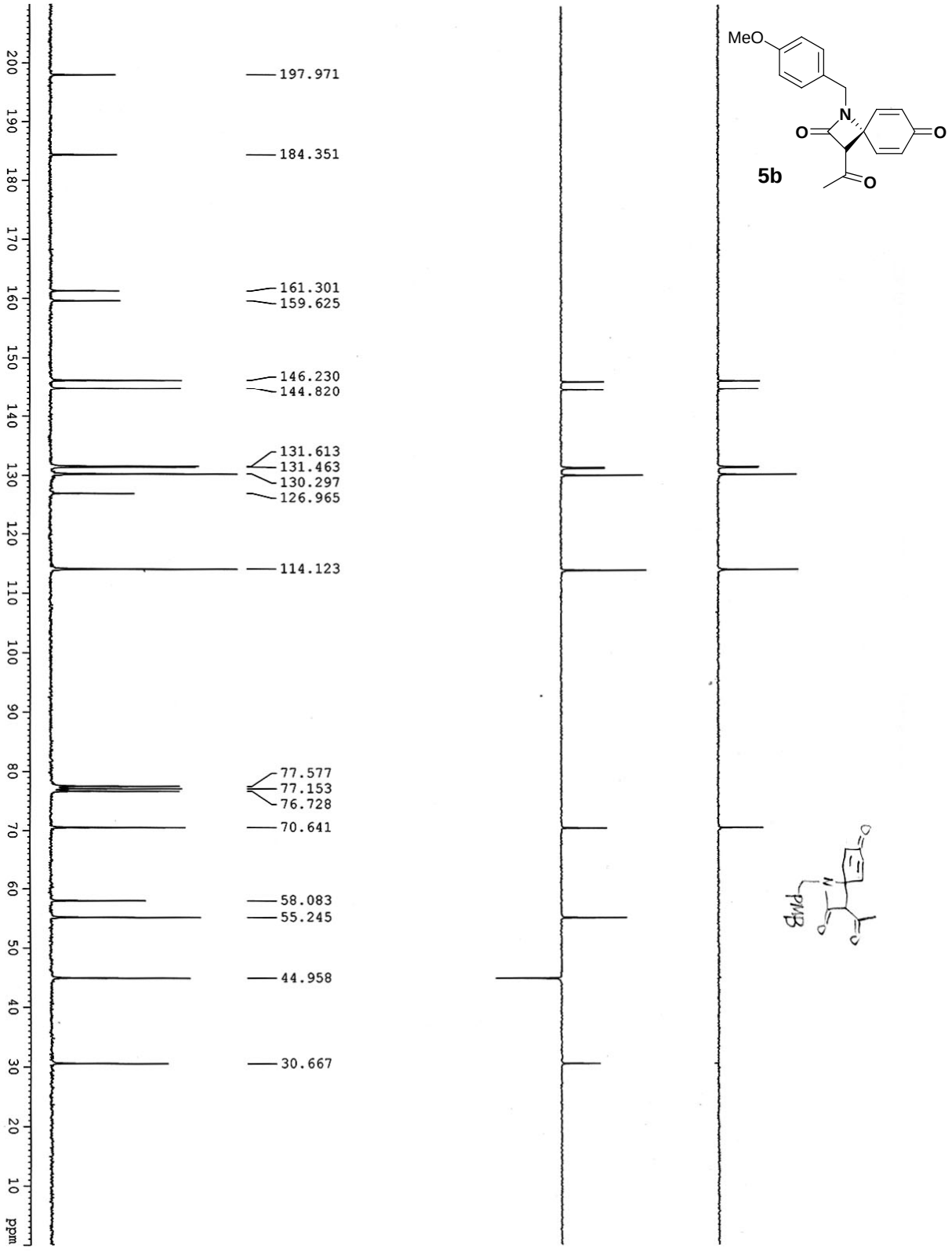
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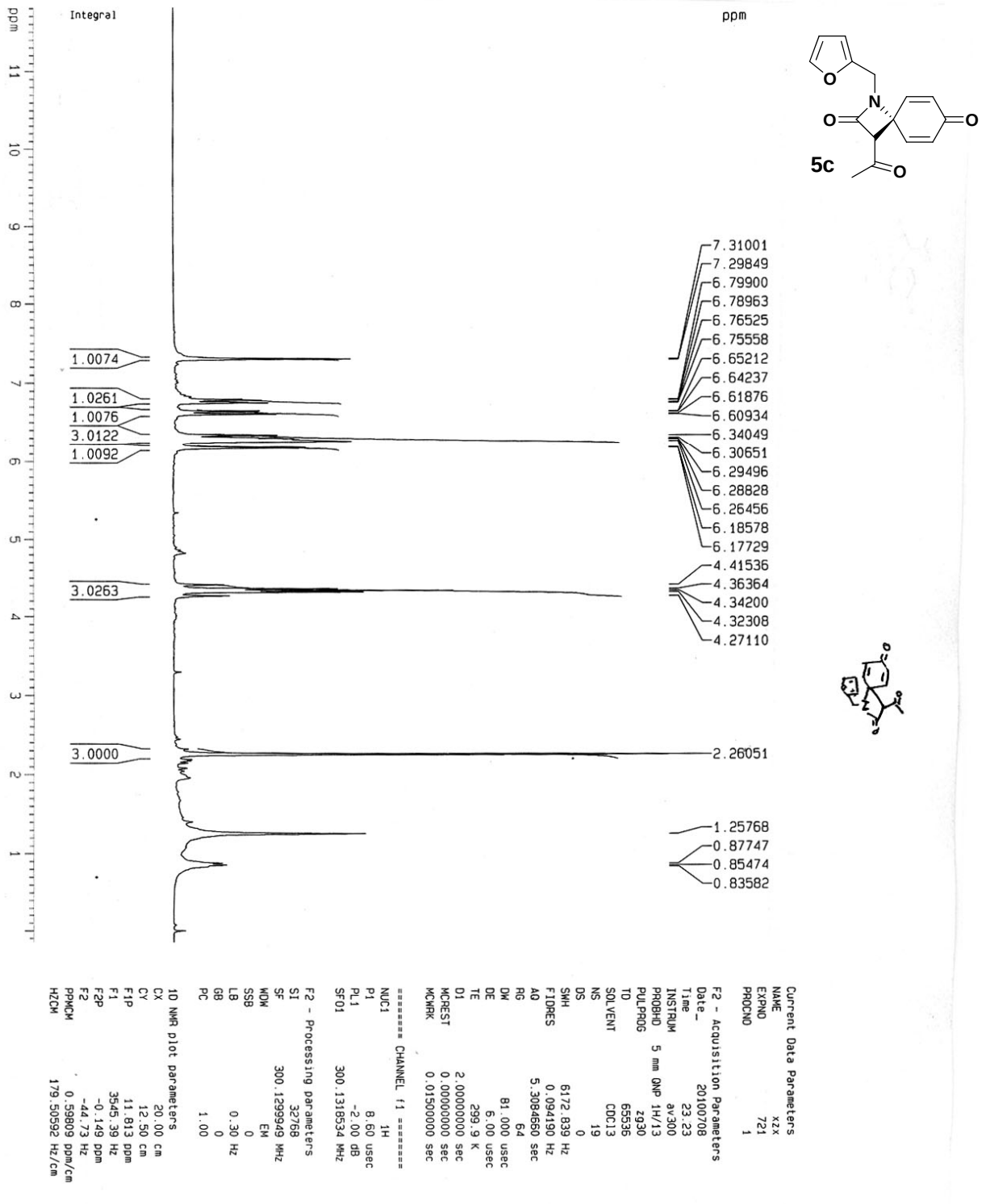
5b



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F2 - Processing parameters
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1D NMR plot parameters
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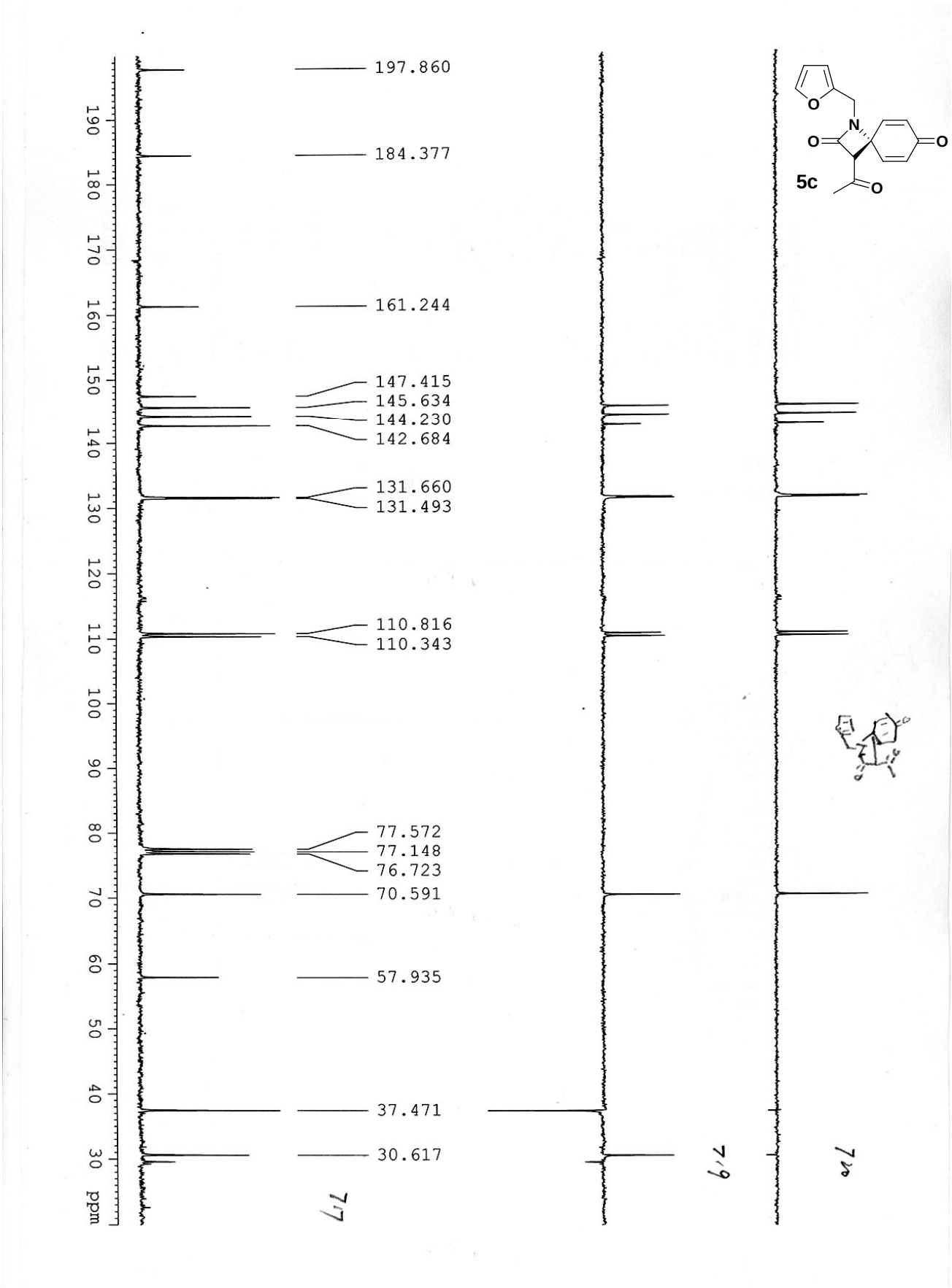
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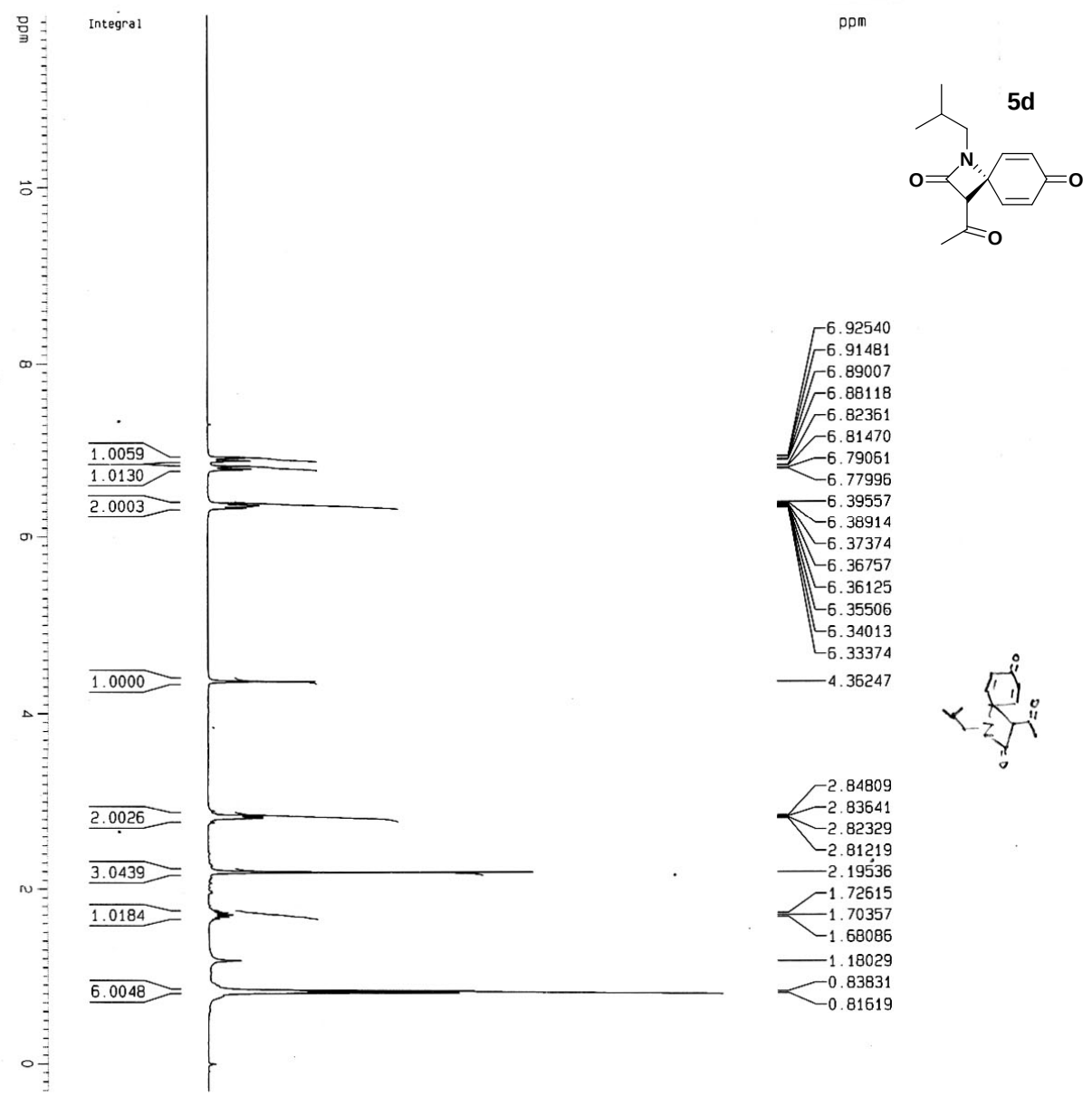


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F2 - Processing parameters
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1D NMR plot parameters
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CY                    6.00 cm
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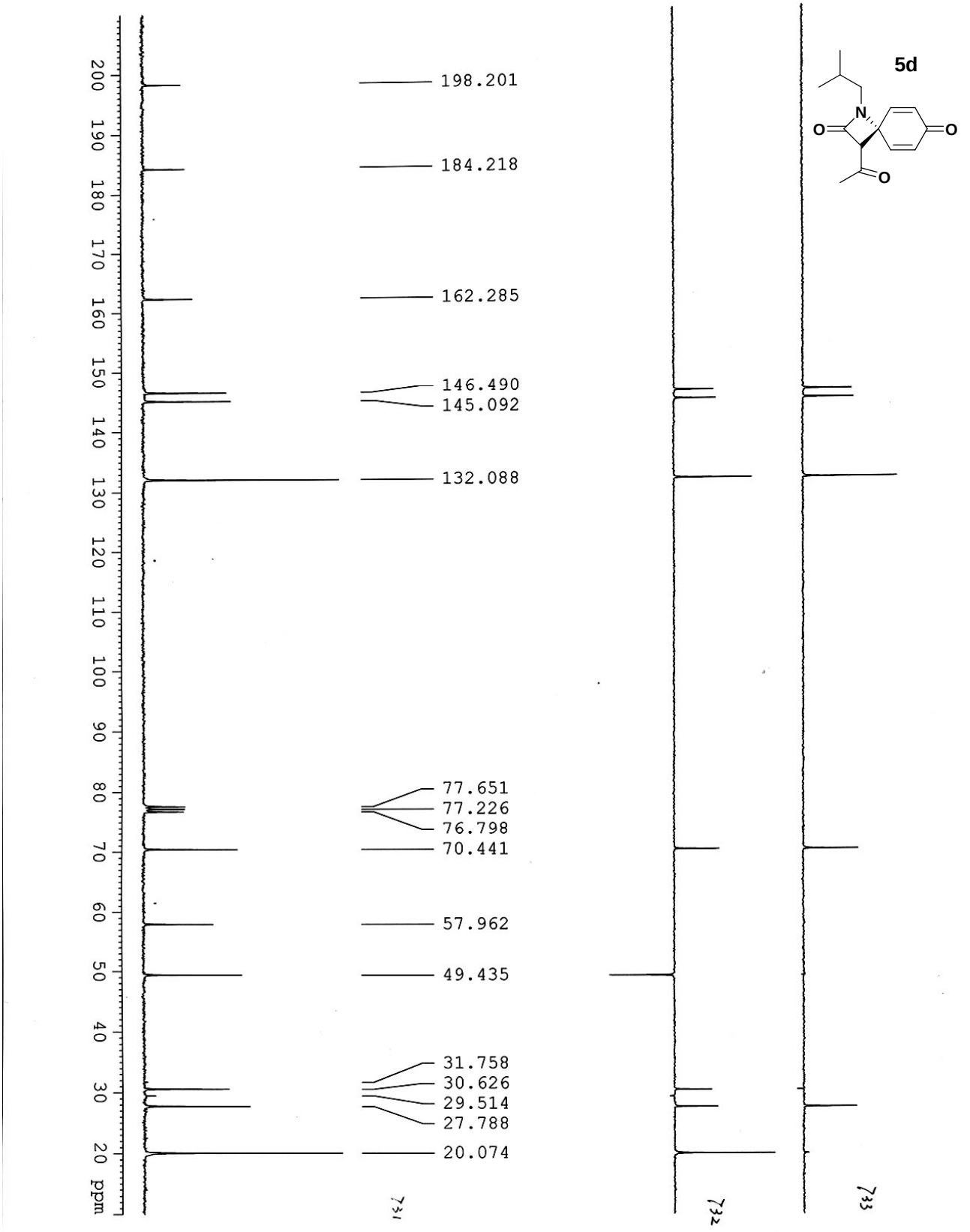
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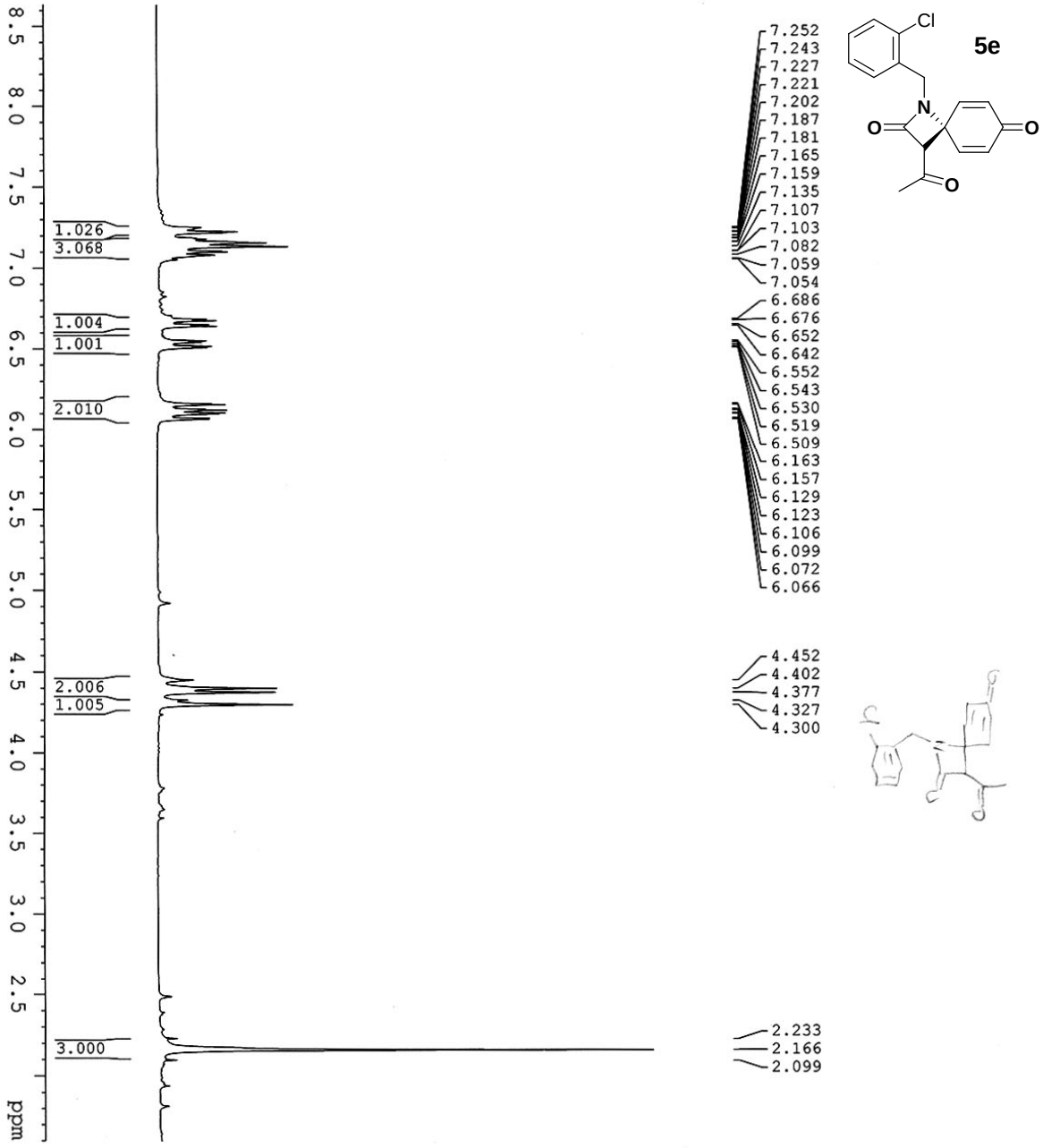
ppm
731
732
733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2.099
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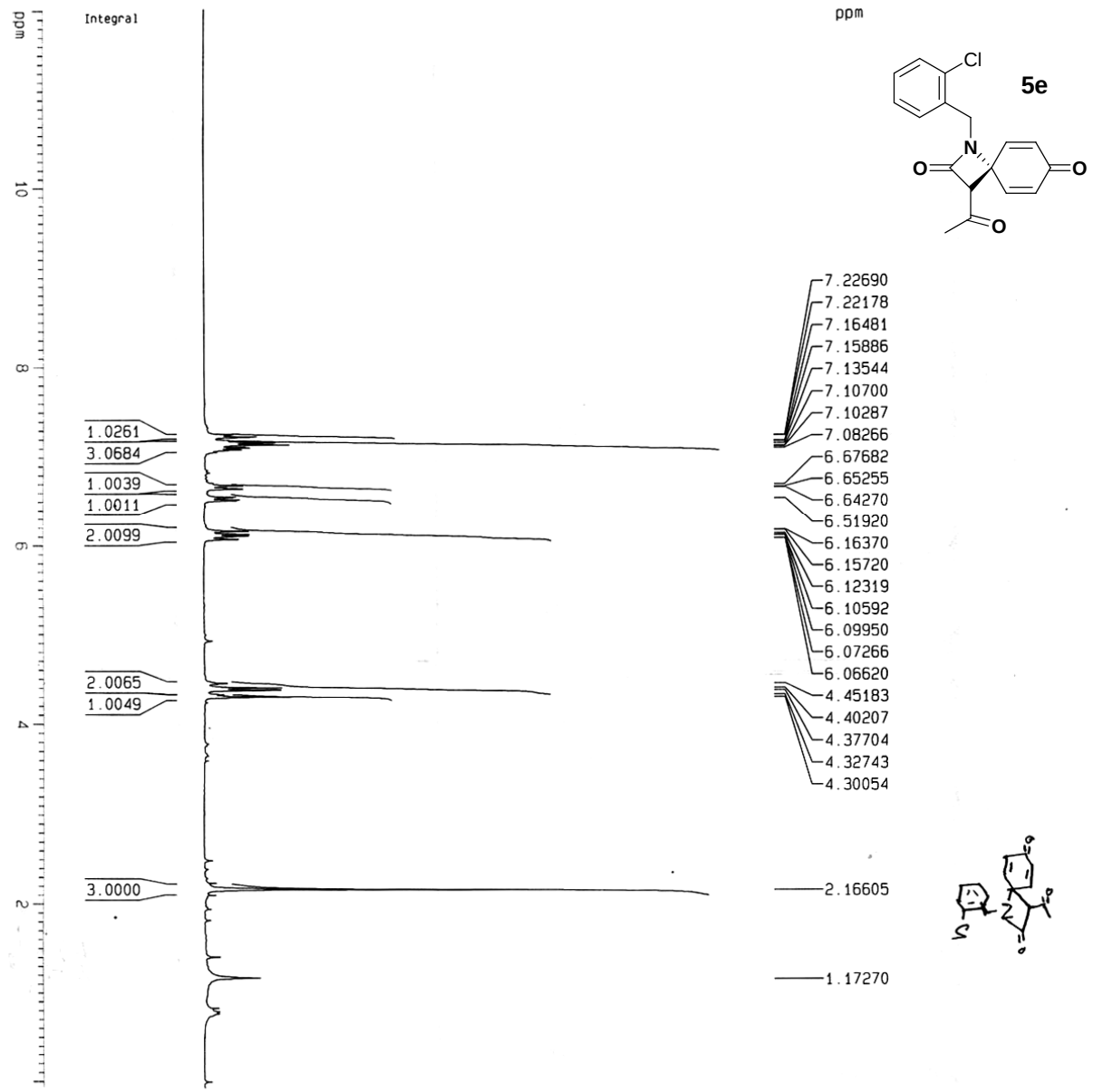


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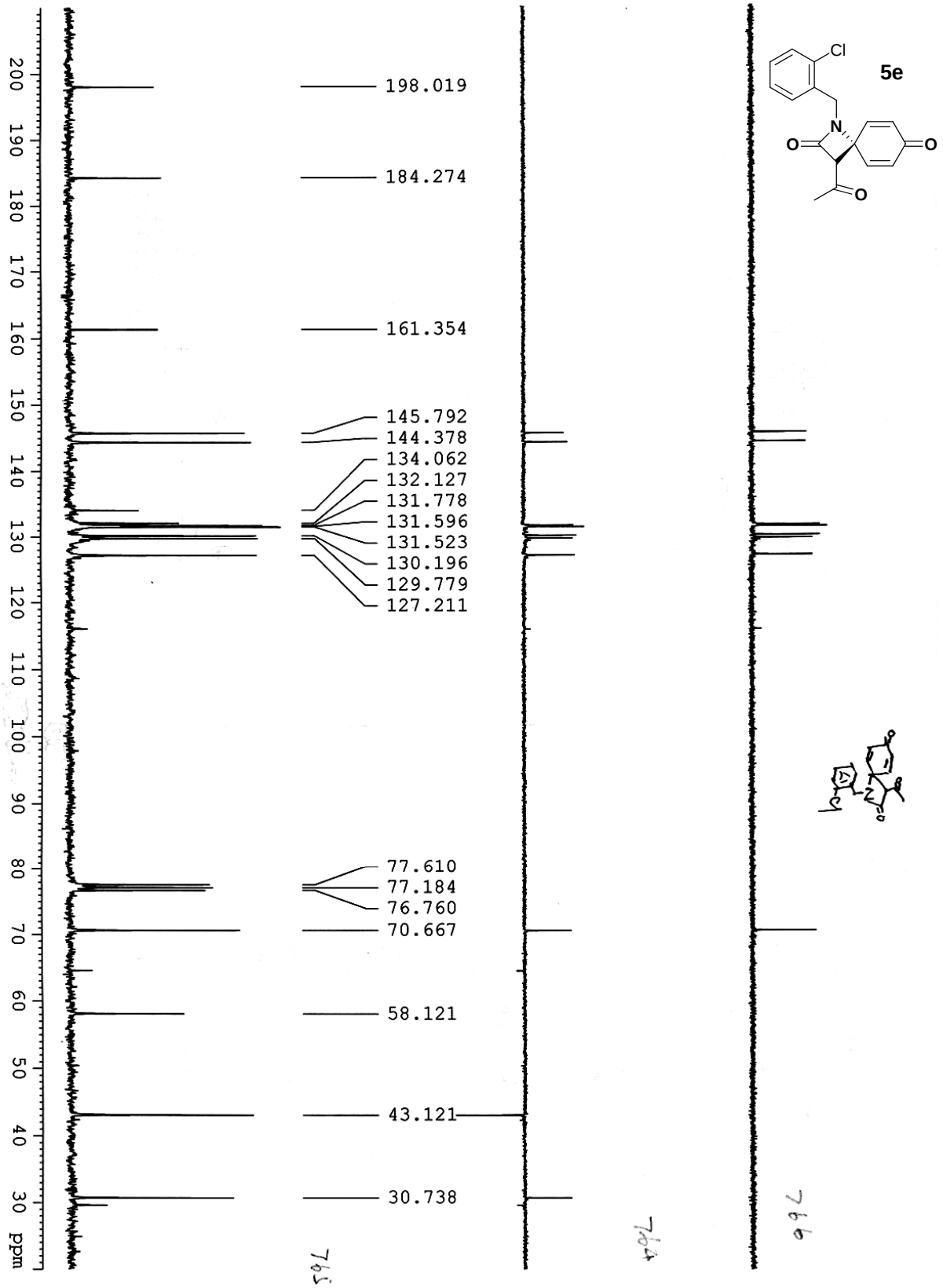
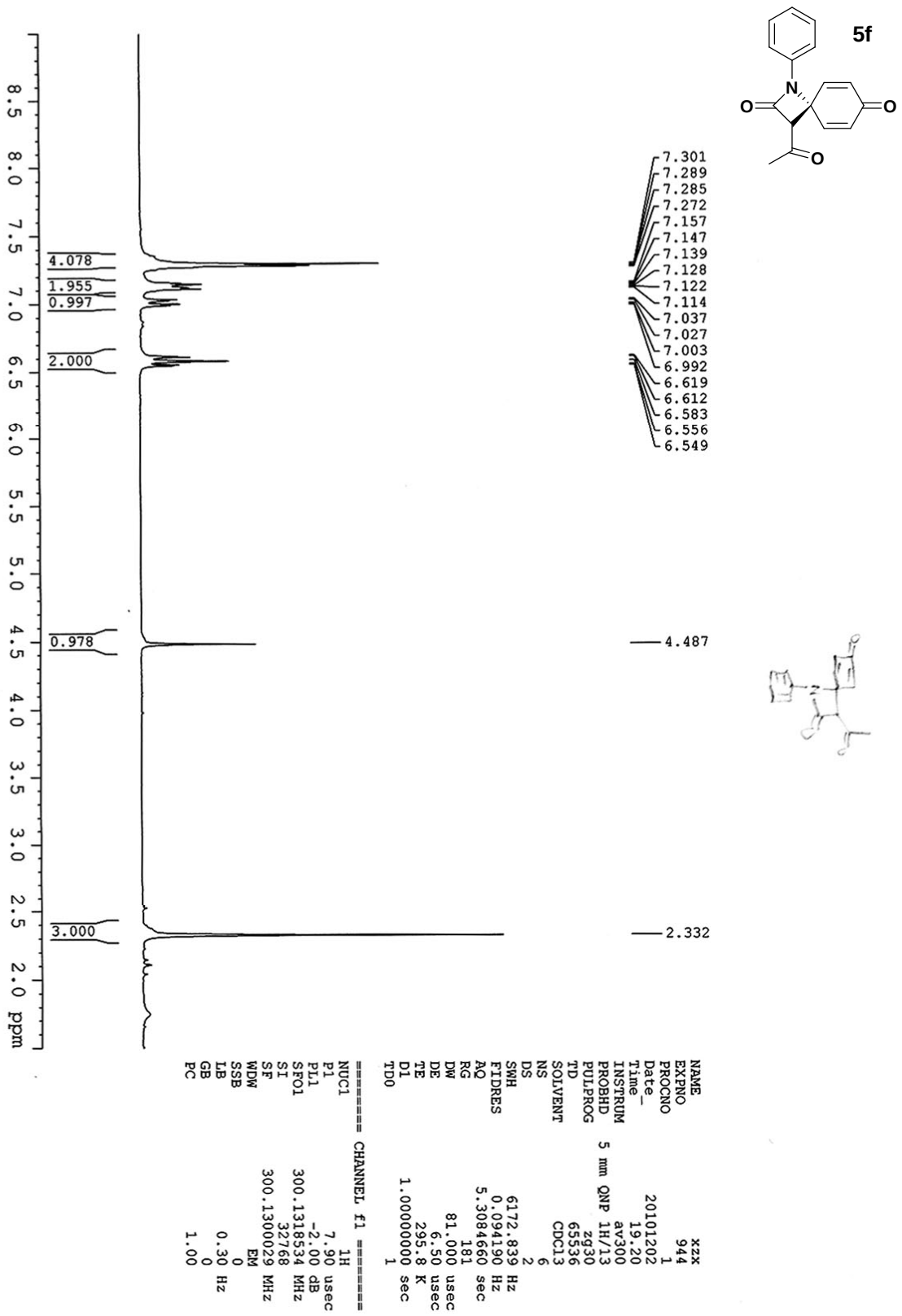
PROBHD  5 mm QNP 1H/13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    12
DS                     0
SWH              6172.839 Hz
FIDRES              0.094190 Hz
AQ                5.3084660 sec
RG                    64
DW                81.000 usec
DE                 6.00 usec
TE                 301.7 K
D1                2.00000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

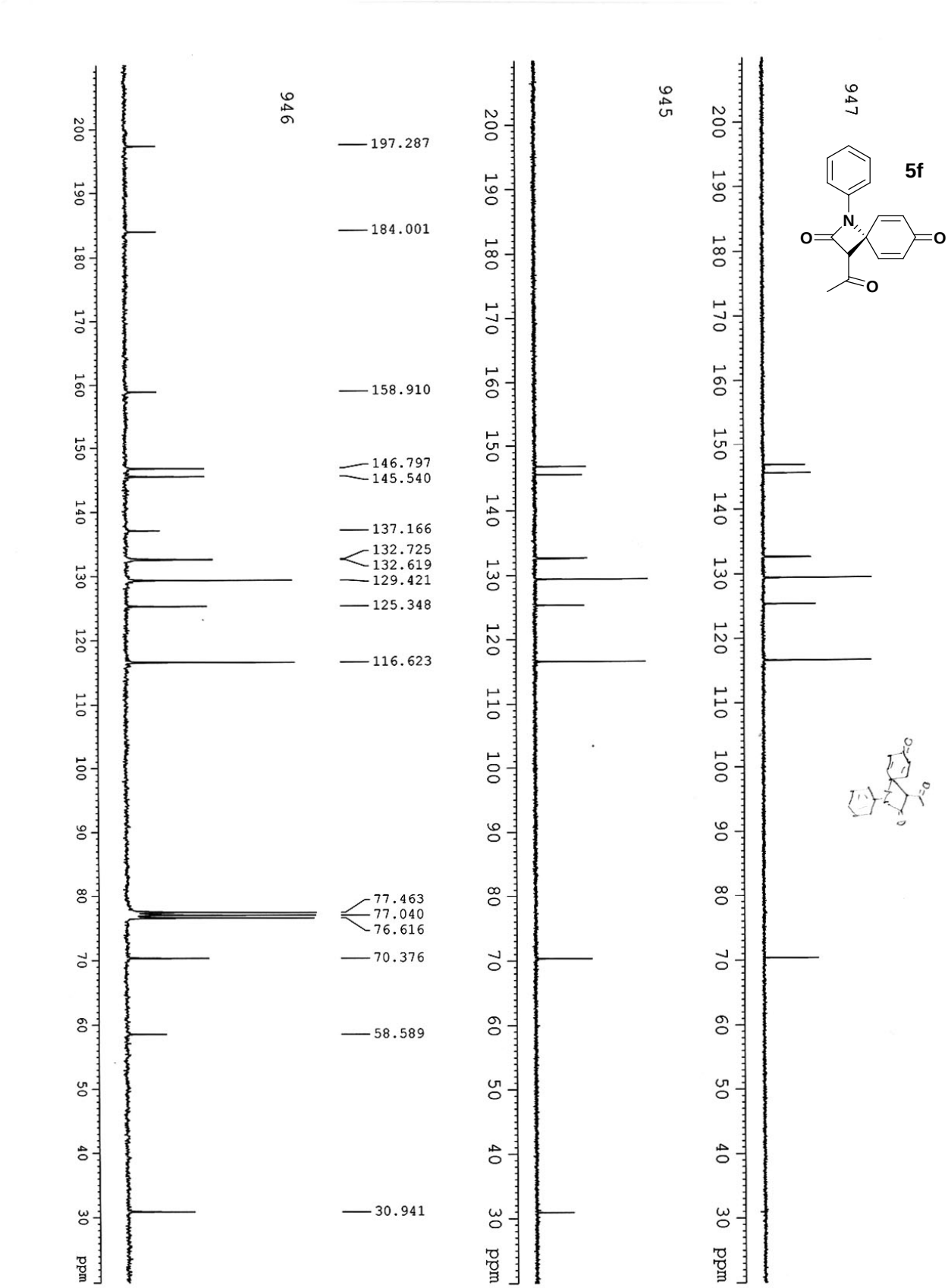
========== CHANNEL f1 ==========
NUC1                  1H
P1                 8.60 usec
PL1               -2.00 dB
SF01        300.1318534 MHz

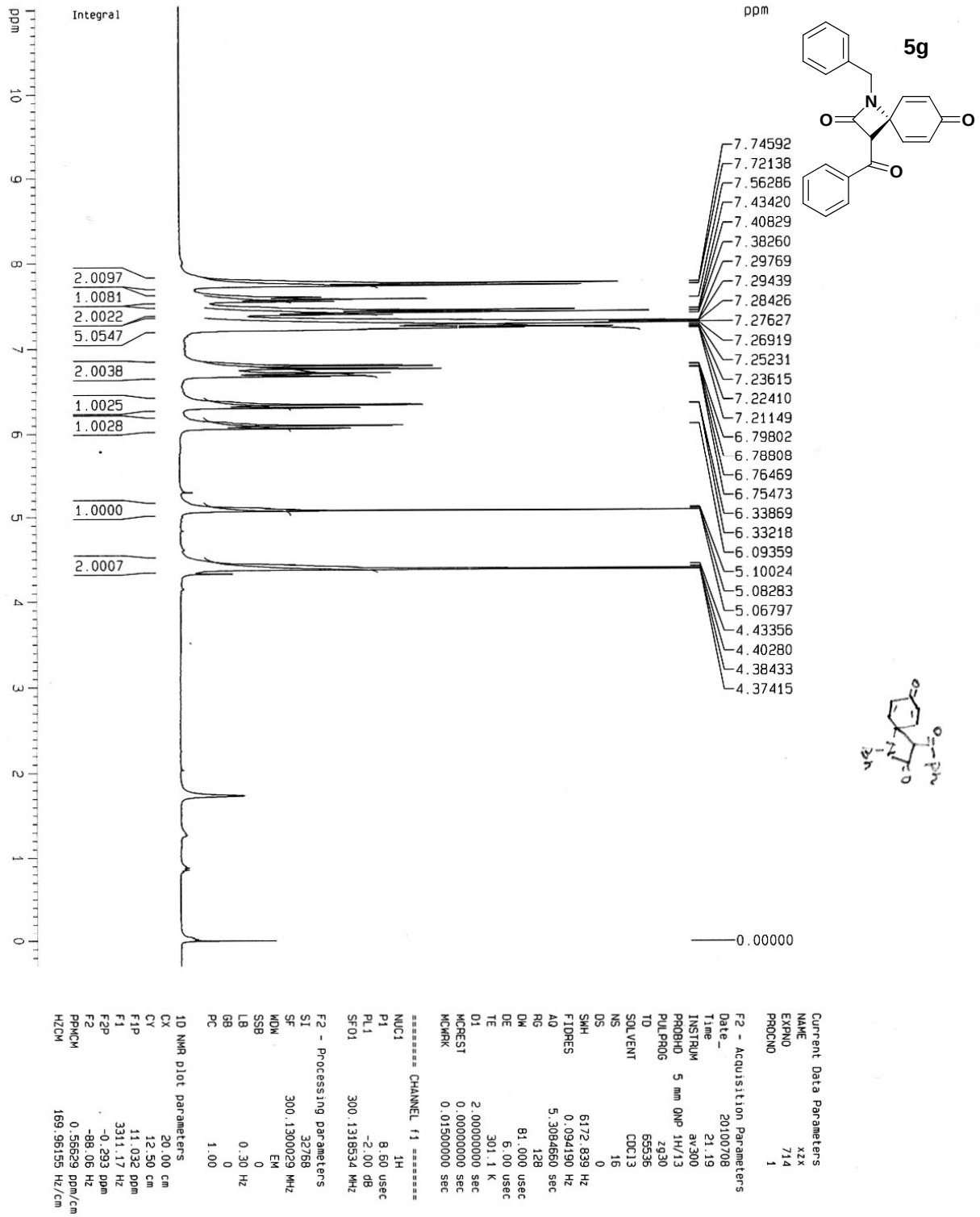
F2 - Processing parameters
SI                 32768
SF          300.1300110 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00

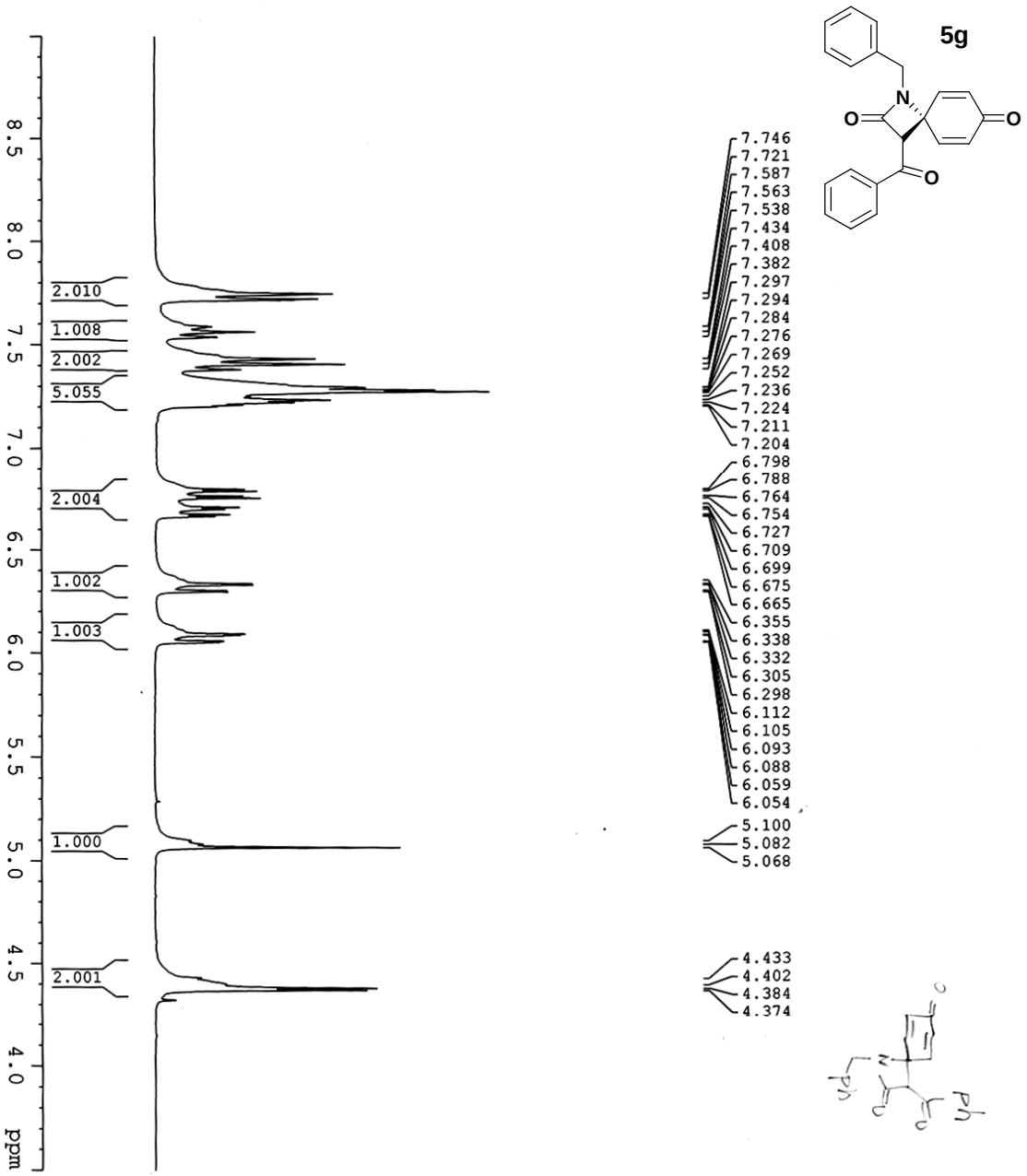
1D NMR plot parameters
CX                 20.00 cm
CY                  6.00 cm
F1P               12.007 ppm
F1                3603.68 Hz
F2P                -0.067 ppm
F2                -20.25 Hz
PPMCM             0.60373 ppm/cm
HZCM          181.19615 Hz/cm
```

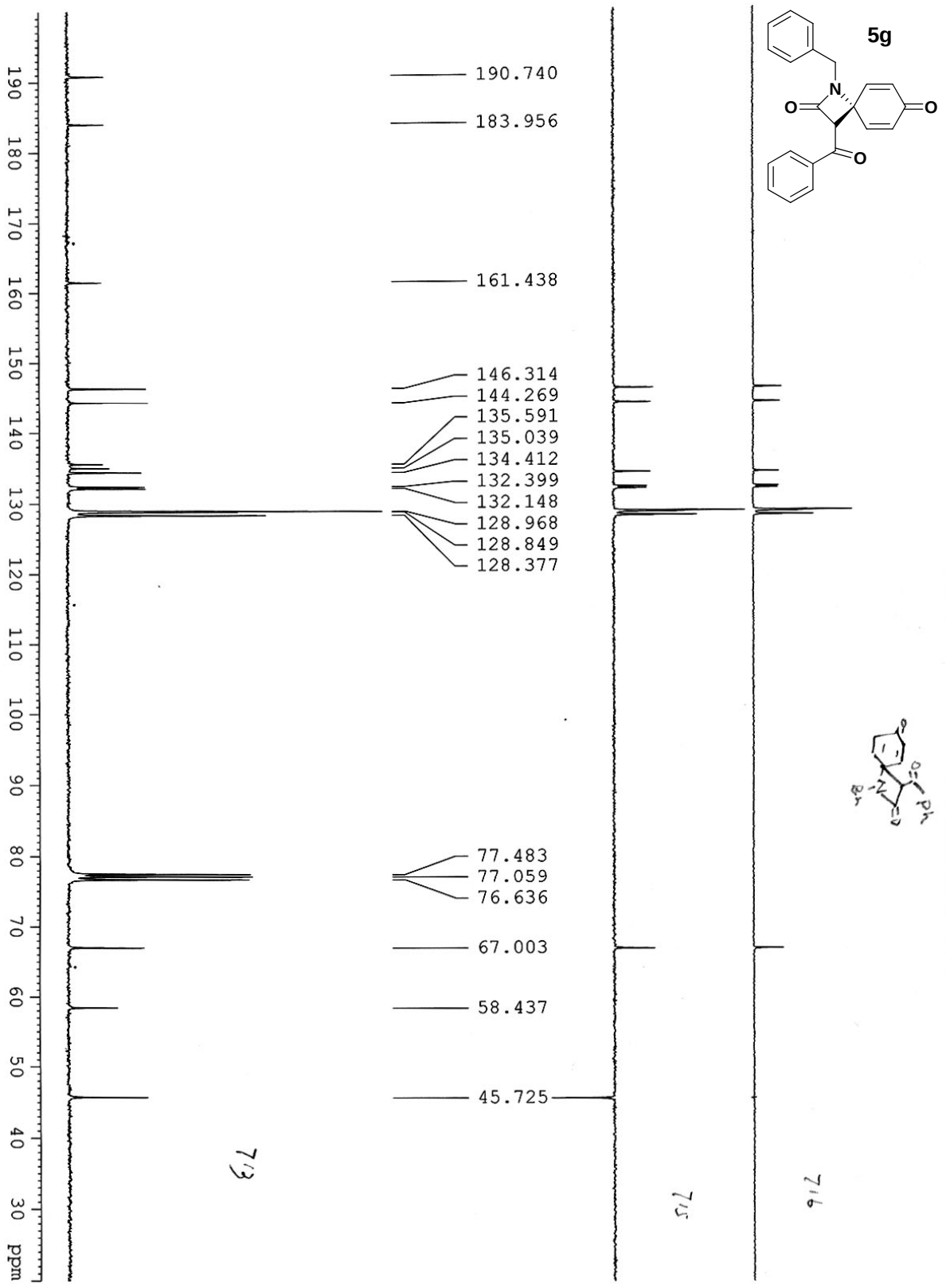

198.019
184.274
161.354
145.792
144.378
134.062
132.127
131.778
131.596
131.523
130.196
129.779
127.211
77.610
77.184
76.760
70.667
58.121
43.121
30.738
ppm
5e
Cl
N
O
O
O

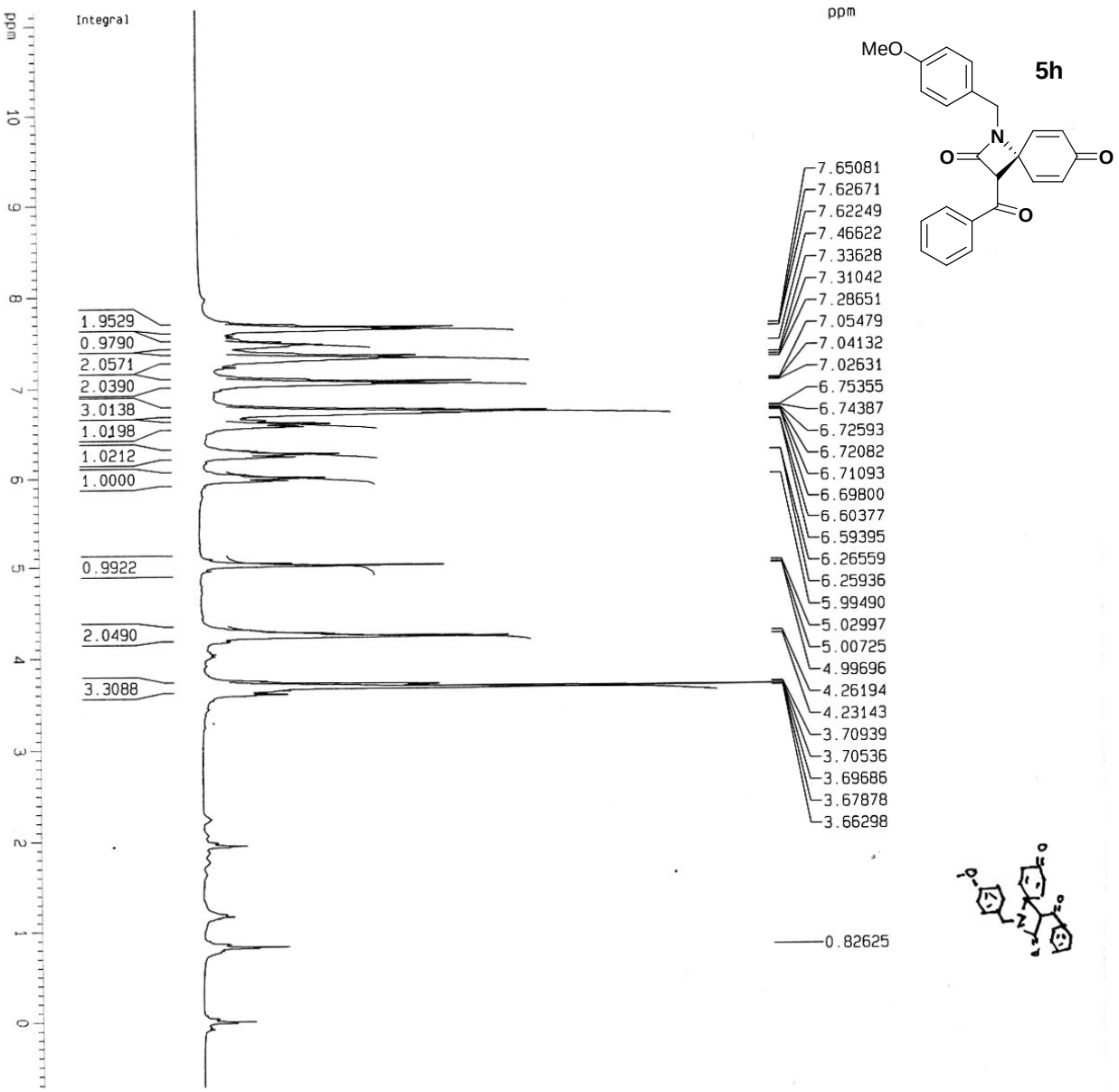

5f
7.301
7.289
7.285
7.272
7.157
7.147
7.139
7.128
7.122
7.114
7.037
7.027
7.003
6.992
6.619
6.612
6.583
6.556
6.549
4.487
2.332
4.078
1.955
0.997
2.000
0.978
3.000
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 ppm
NAME xzx
EXPNO 944
PROCNO 1
Date_ 20101202
Time_ 19.20
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 6
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 181
DW 81.000 usec
DE 6.50 usec
TE 295.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.90 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300029 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


947
945
946
5f
197.287
184.001
158.910
146.797
145.540
137.166
132.725
132.619
129.421
125.348
116.623
77.463
77.040
76.616
70.376
58.589
30.941
ppm


5g
ppm
Integral
7.74592
7.72138
7.56286
7.43420
7.40829
7.38260
7.29769
7.29439
7.28426
7.27627
7.26919
7.25231
7.23615
7.22410
7.21149
6.79802
6.78808
6.76469
6.75473
6.33869
6.33218
6.09359
5.10024
5.08283
5.06797
4.43356
4.40280
4.38433
4.37415
2.0097
1.0081
2.0022
5.0547
2.0038
1.0025
1.0028
1.0000
2.0007
0.00000
Current Data Parameters
NAME xzx
EXPNO 714
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100708
Time 21.19
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 128
DW 81.000 usec
DE 6.00 usec
TE 301.1 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.60 usec
PL1 -2.00 dB
SF01 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300029 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 11.032 ppm
F1 3311.17 Hz
F2P -0.293 ppm
F2 -88.06 Hz
PPMCM 0.56629 ppm/cm
HZCM 169.96155 Hz/cm


5g
7.746
7.721
7.587
7.563
7.538
7.434
7.408
7.382
7.297
7.294
7.284
7.276
7.269
7.252
7.236
7.224
7.211
7.204
6.798
6.788
6.764
6.754
6.727
6.709
6.699
6.675
6.665
6.355
6.338
6.332
6.305
6.298
6.112
6.105
6.093
6.088
6.059
6.054
5.100
5.082
5.068
4.433
4.402
4.384
4.374
2.010
1.008
2.002
5.055
2.004
1.002
1.003
1.000
2.001
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
ppm
NAME xzx
EXPNO 714
PROCNO 1
Date_ 20100708
Time_ 21.19
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 128
DW 81.000 usec
DE 6.00 usec
TE 301.1 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.60 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300029 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


5g
190.740
183.956
161.438
146.314
144.269
135.591
135.039
134.412
132.399
132.148
128.968
128.849
128.377
77.483
77.059
76.636
67.003
58.437
45.725
ppm



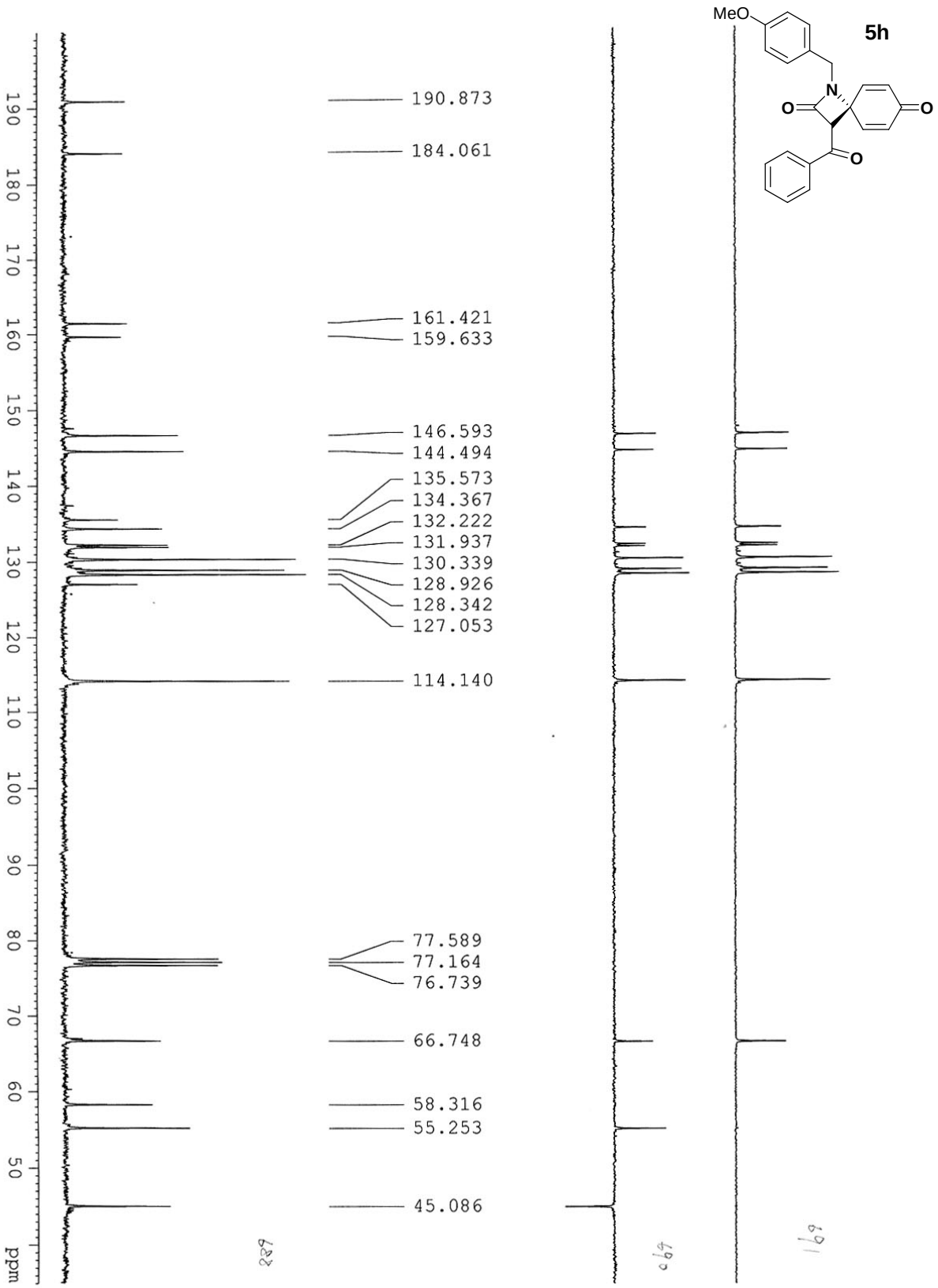

```
Current Data Parameters
NAME                  xzx
EXPNO                 687
PROCNO                  1

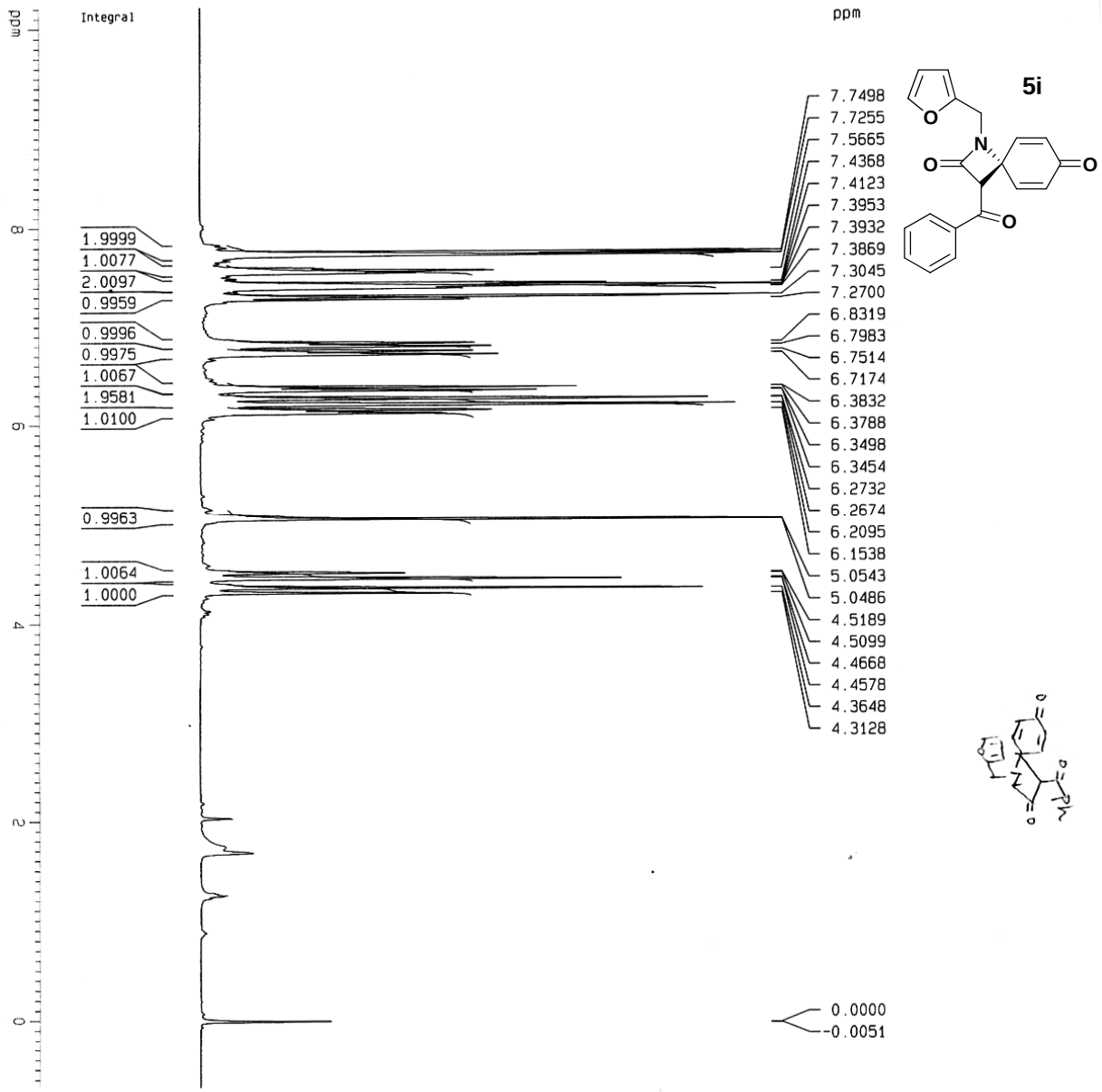
F2 - Acquisition Parameters
Date_              20100706
Time                 21.06
INSTRUM              av300
PROBHD     5 mm QNP 1H/13
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     19
DS                      0
SWH             6172.839 Hz
FIDRES          0.094190 Hz
AQ              5.3084660 sec
RG                   50.8
DW                 81.000 usec
DE                  6.00 usec
TE                 300.3 K
D1              2.00000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                  8.60 usec
PL1                -2.00 dB
SFO1          300.1318534 MHz

F2 - Processing parameters
SI                    32768
SF              300.1300191 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  20.00 cm
CY                  12.50 cm
F1P                11.169 ppm
F1                 3352.21 Hz
F2P                -0.725 ppm
F2                 -217.63 Hz
PPMCM           0.59471 ppm/cm
HZCM            178.49173 Hz/cm
```


MeO
5h
N
O
O
O
190.873
184.061
161.421
159.633
146.593
144.494
135.573
134.367
132.222
131.937
130.339
128.926
128.342
127.053
114.140
77.589
77.164
76.739
66.748
58.316
55.253
45.086
ppm



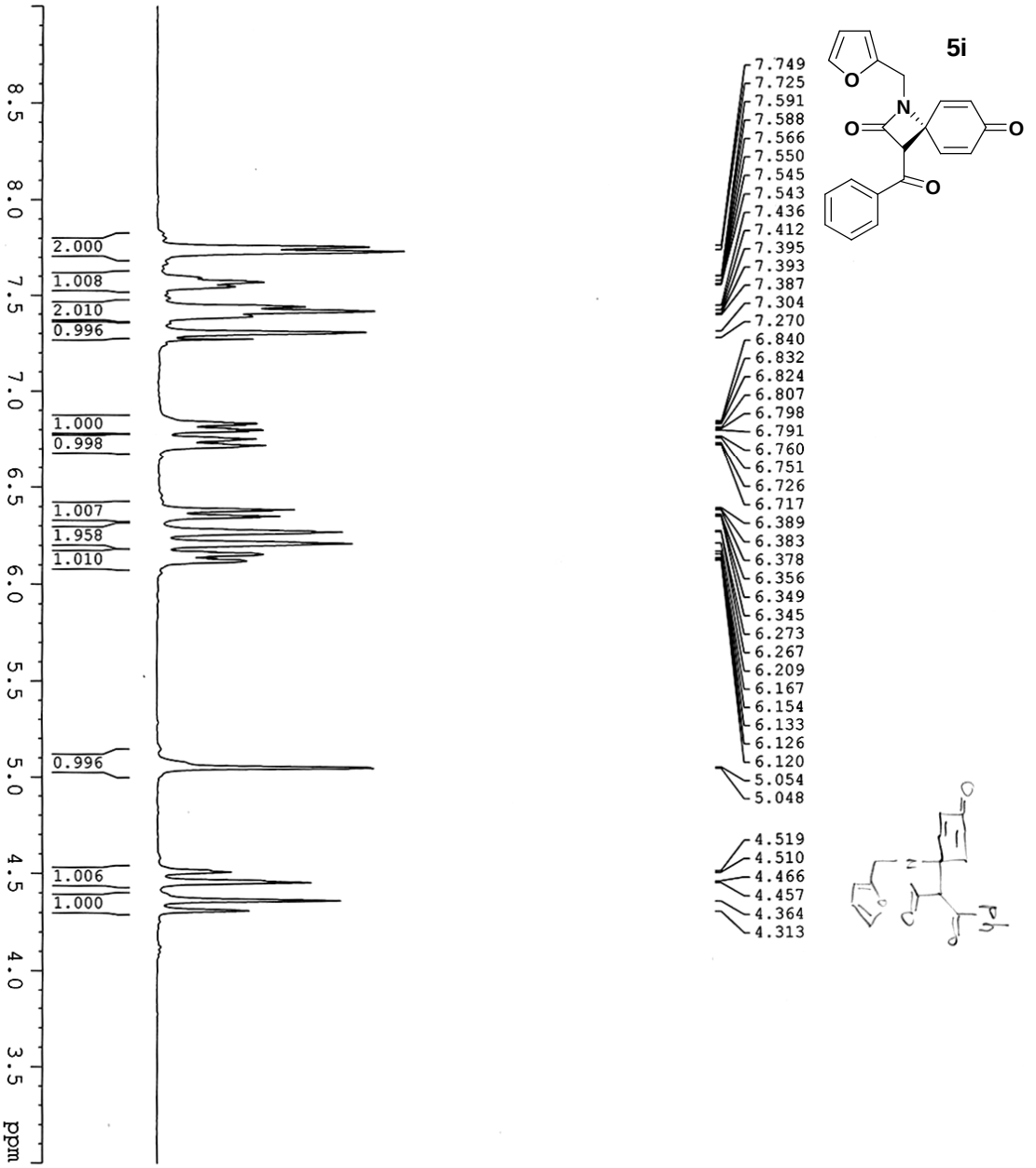
Current Data Parameters
NAME xzx
EXPNO 697
PROCNO 1

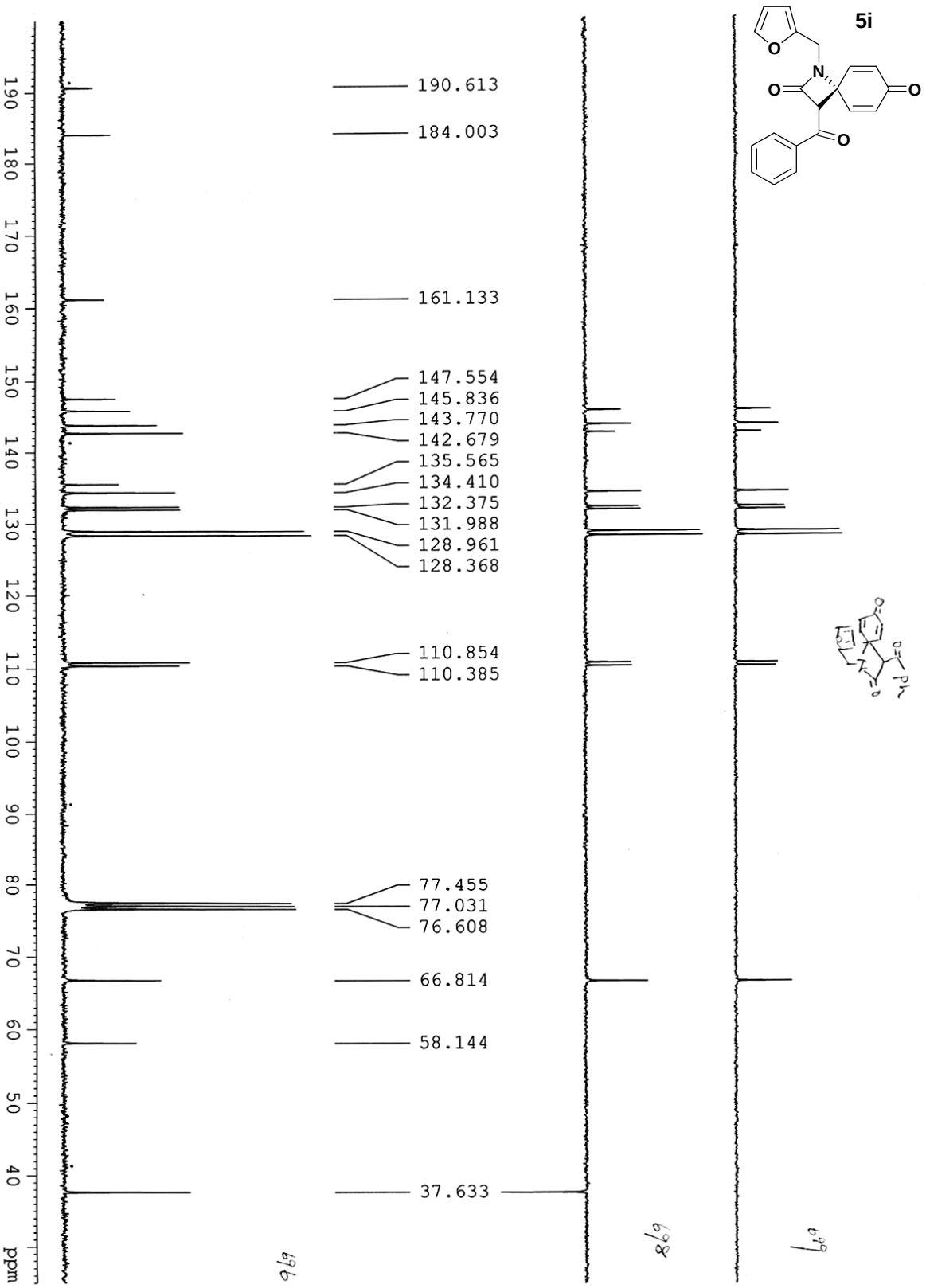
F2 - Acquisition Parameters
Date_ 20100707
Time 21.39
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 38
DS 0
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 203.2
DW 81.000 usec
DE 6.00 usec
TE 301.1 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

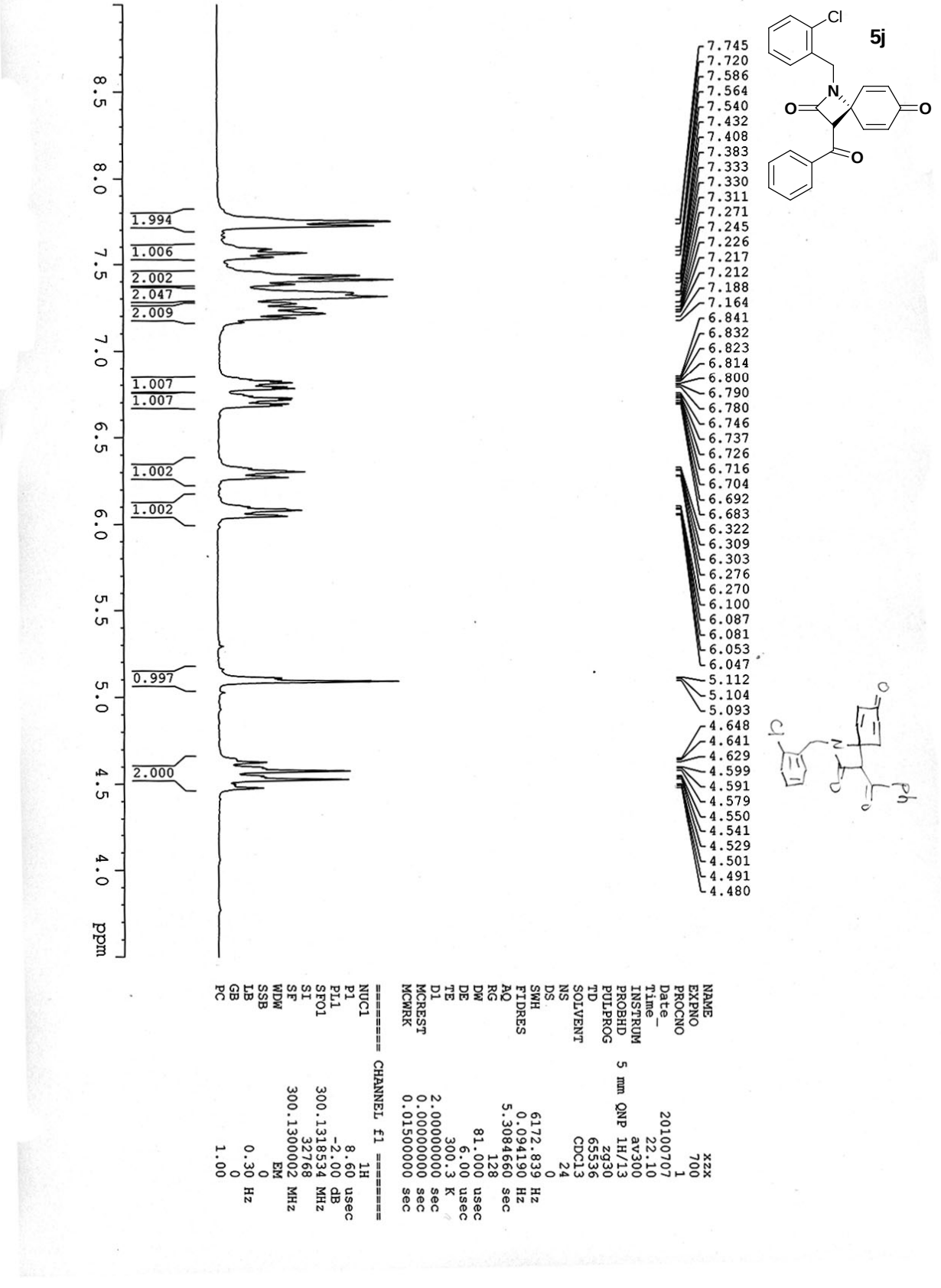
======== CHANNEL f1 ========
NUC1 1H
P1 8.60 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz

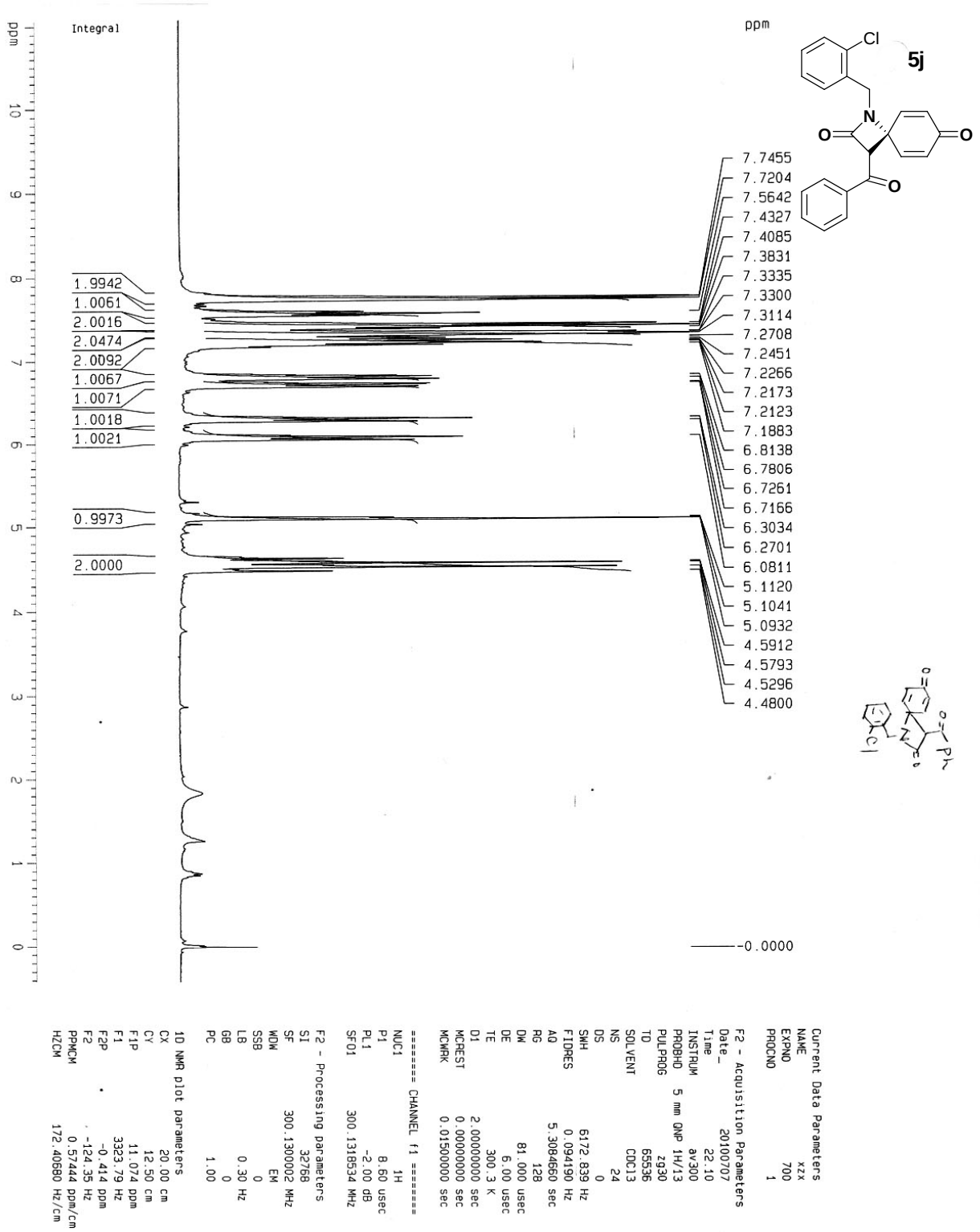
F2 - Processing parameters
SI 32768
SF 300.1300030 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

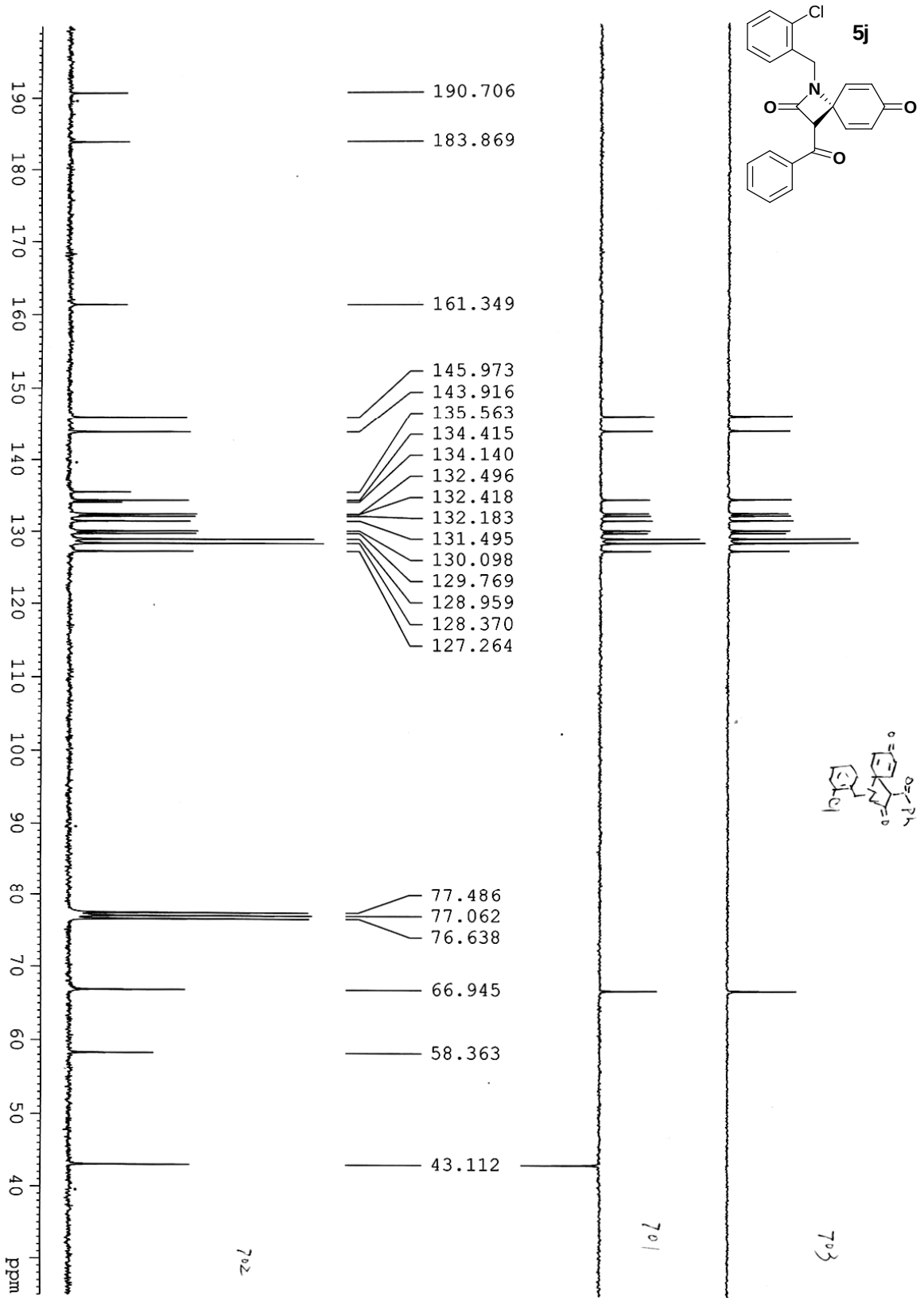
1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 10.209 ppm
F1 3063.99 Hz
F2P -0.672 ppm
F2 -201.60 Hz
PPMCM 0.54403 ppm/cm
HZCM 163.27937 Hz/cm

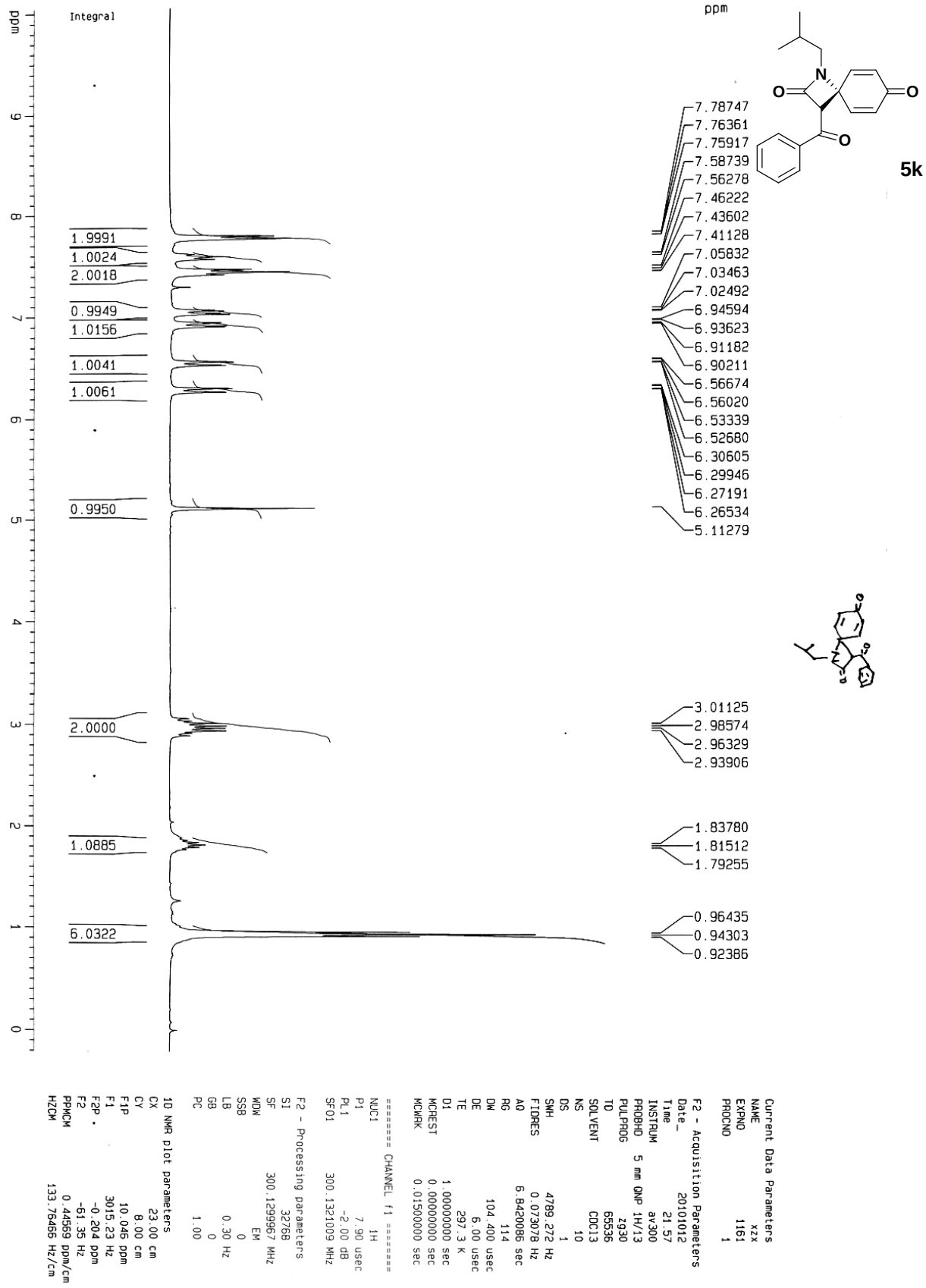

5i
7.749
7.725
7.591
7.588
7.566
7.550
7.545
7.543
7.436
7.412
7.395
7.393
7.387
7.304
7.270
6.840
6.832
6.824
6.807
6.798
6.791
6.760
6.751
6.726
6.717
6.389
6.383
6.378
6.356
6.349
6.345
6.273
6.267
6.209
6.167
6.154
6.133
6.126
6.120
5.054
5.048
4.519
4.510
4.466
4.457
4.364
4.313
2.000
1.008
2.010
0.996
1.000
0.998
1.007
1.958
1.010
0.996
1.006
1.000
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
ppm
NAME xzx
EXPNO 697
PROCNO 1
Date_ 20100707
Time_ 21.39
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 38
DS 0
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 203.2
DW 81.000 usec
DE 6.00 usec
TE 301.1 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.60 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300030 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


5i
190.613
184.003
161.133
147.554
145.836
143.770
142.679
135.565
134.410
132.375
131.988
128.961
128.368
110.854
110.385
77.455
77.031
76.608
66.814
58.144
37.633
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
ppm


5j
Cl
N
O
O
O
7.745
7.720
7.586
7.564
7.540
7.432
7.408
7.383
7.333
7.330
7.311
7.271
7.245
7.226
7.217
7.212
7.188
7.164
6.841
6.832
6.823
6.814
6.800
6.790
6.780
6.746
6.737
6.726
6.716
6.704
6.692
6.683
6.322
6.309
6.303
6.276
6.270
6.100
6.087
6.081
6.053
6.047
5.112
5.104
5.093
4.648
4.641
4.629
4.599
4.591
4.579
4.550
4.541
4.529
4.501
4.491
4.480
1.994
1.006
2.002
2.047
2.009
1.007
1.007
1.002
1.002
0.997
2.000
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
ppm
NAME xzx
EXPNO 700
PROCNO 1
Date_ 20100707
Time_ 22.10
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 24
DS 0
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 128
DW 81.000 usec
DE 6.00 usec
TE 300.3 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.60 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300002 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


5j
Cl
N
O
O
O
ppm
Integral
7.7455
7.7204
7.5642
7.4327
7.4085
7.3831
7.3335
7.3300
7.3114
7.2708
7.2451
7.2266
7.2173
7.2123
7.1883
6.8138
6.7806
6.7261
6.7166
6.3034
6.2701
6.0811
5.1120
5.1041
5.0932
4.5912
4.5793
4.5296
4.4800
1.9942
1.0061
2.0016
2.0474
2.0092
1.0067
1.0071
1.0018
1.0021
0.9973
2.0000
-0.0000
Current Data Parameters
NAME xzx
EXPNO 700
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100707
Time 22.10
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 24
DS 0
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 128
DW 81.000 usec
DE 6.00 usec
TE 300.3 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.60 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300002 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 11.074 ppm
F1 3323.79 Hz
F2P -0.414 ppm
F2 -124.35 Hz
PPMCM 0.57444 ppm/cm
HZCM 172.40680 Hz/cm


5j
Cl
N
O
O
O
190.706
183.869
161.349
145.973
143.916
135.563
134.415
134.140
132.496
132.418
132.183
131.495
130.098
129.769
128.959
128.370
127.264
77.486
77.062
76.638
66.945
58.363
43.112
ppm
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
702
701
703



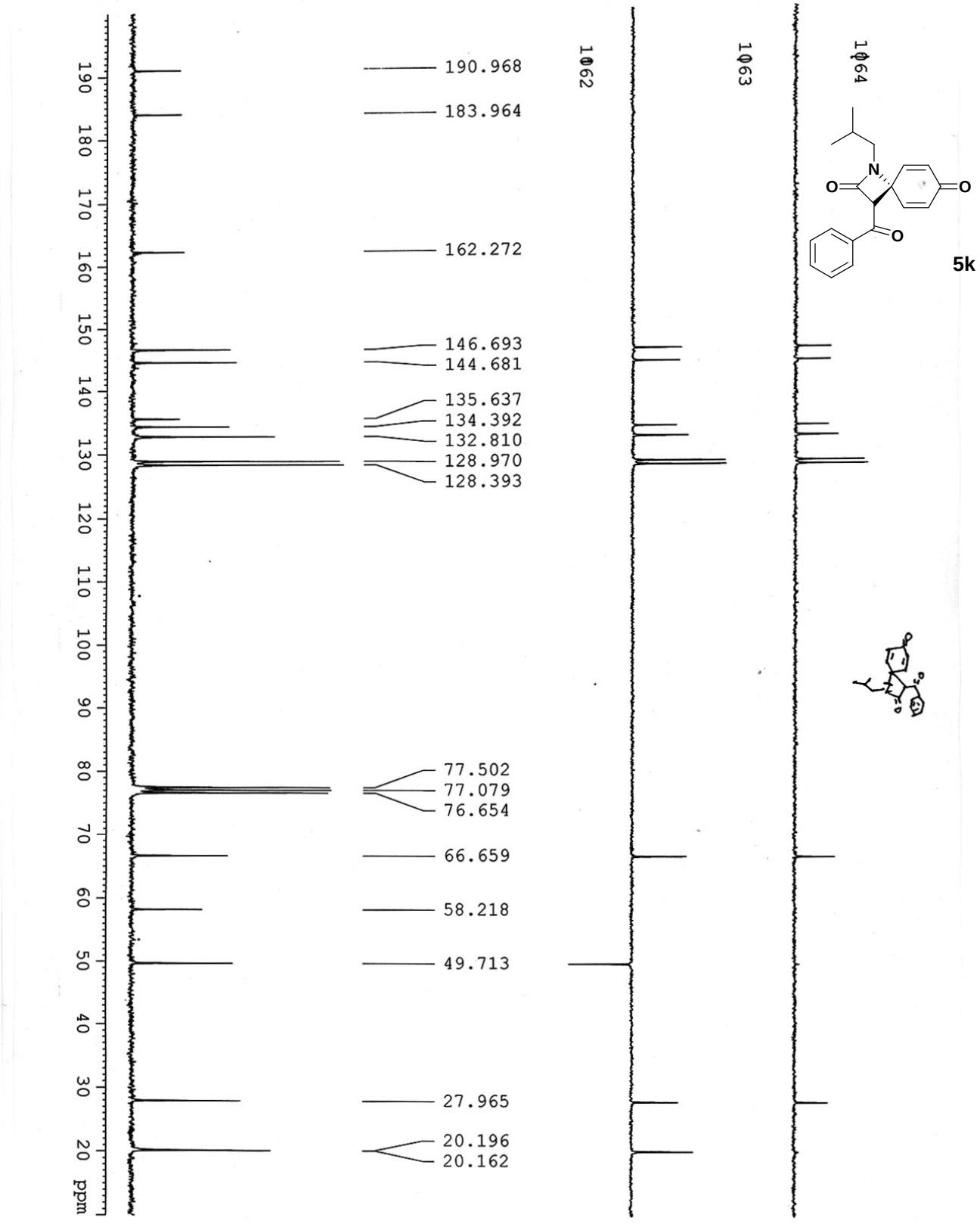
Current Data Parameters
NAME xzx
EXPNO 1161
PROCNO 1

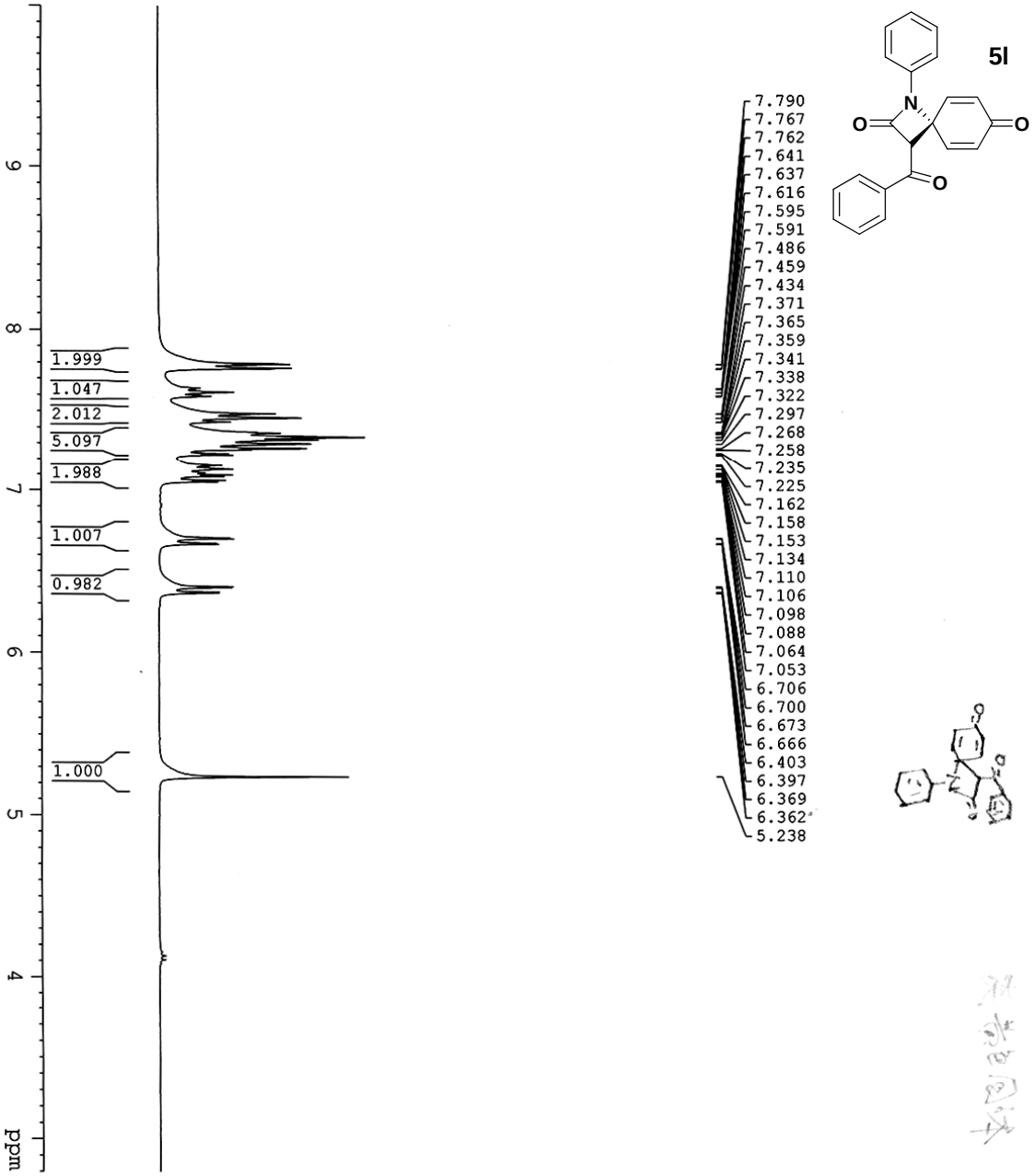
F2 - Acquisition Parameters
Date_ 20101012
Time 21.57
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 10
DS 1
SWH 4789.272 Hz
FIDRES 0.073078 Hz
AQ 6.8420086 sec
RG 114
DW 104.400 usec
DE 6.00 usec
TE 297.3 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

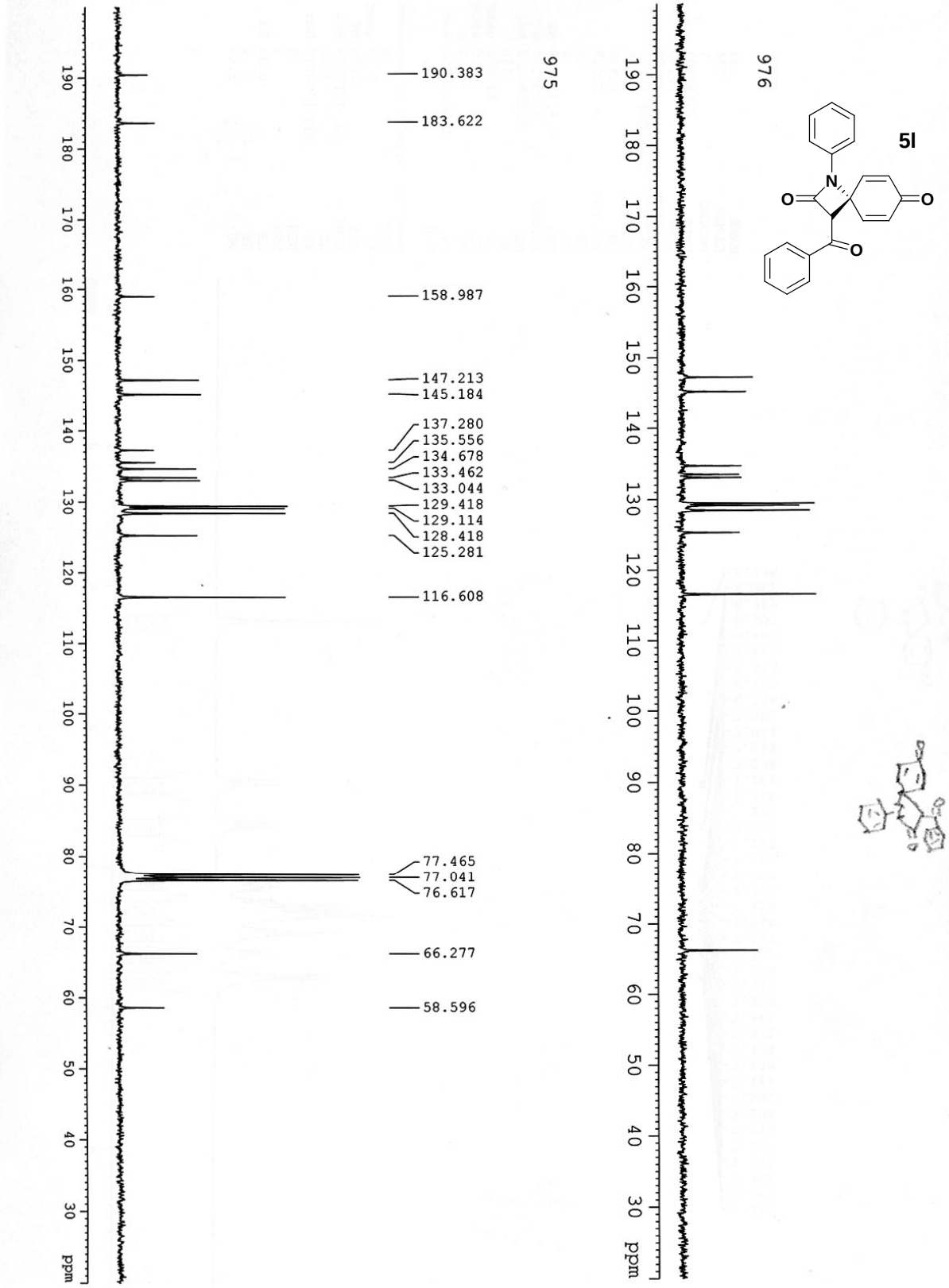
======== CHANNEL f1 ========
NUC1 1H
P1 7.90 usec
PL1 -2.00 dB
SFO1 300.1321009 MHz

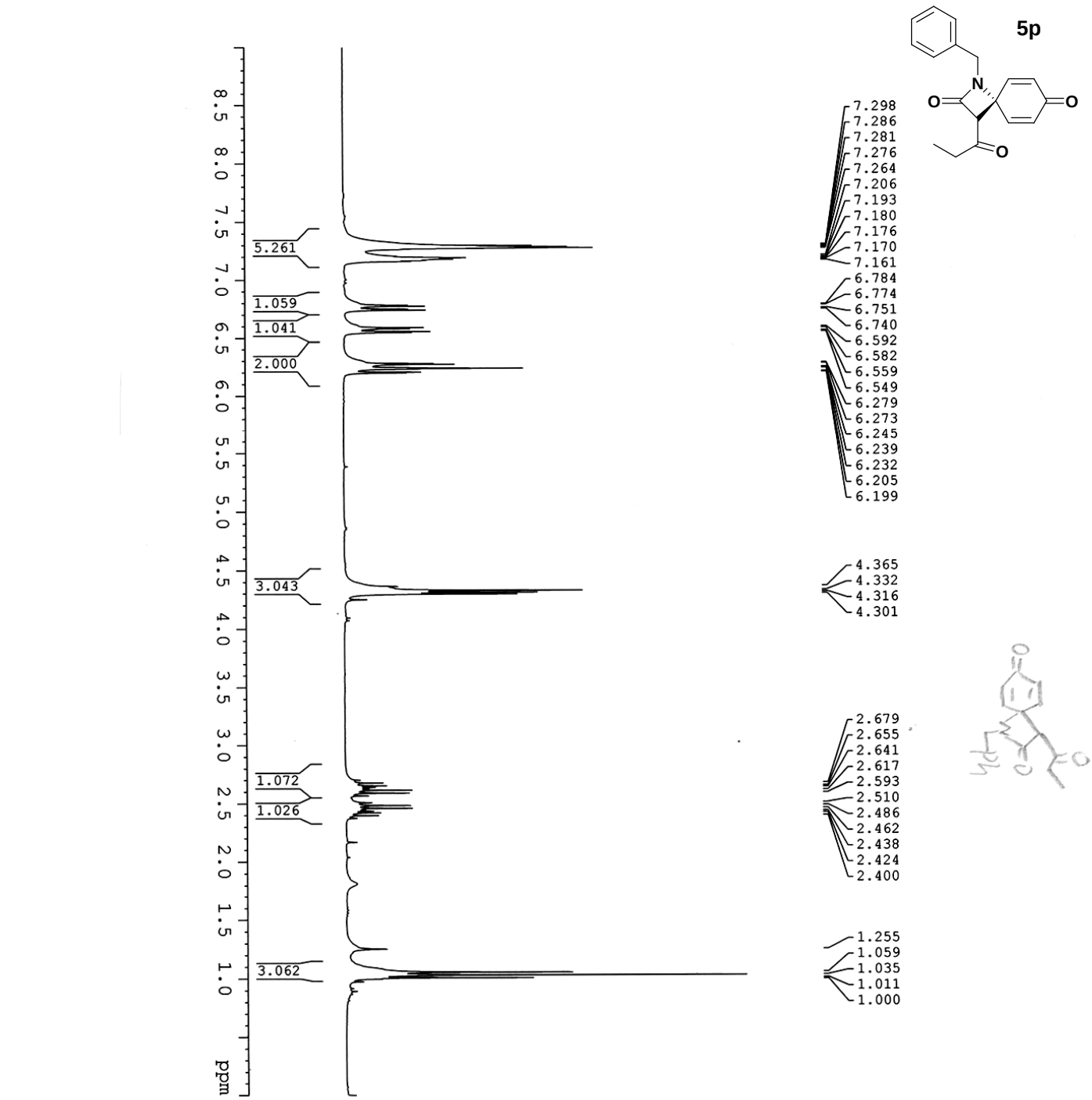
F2 - Processing parameters
SI 32768
SF 300.1299967 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

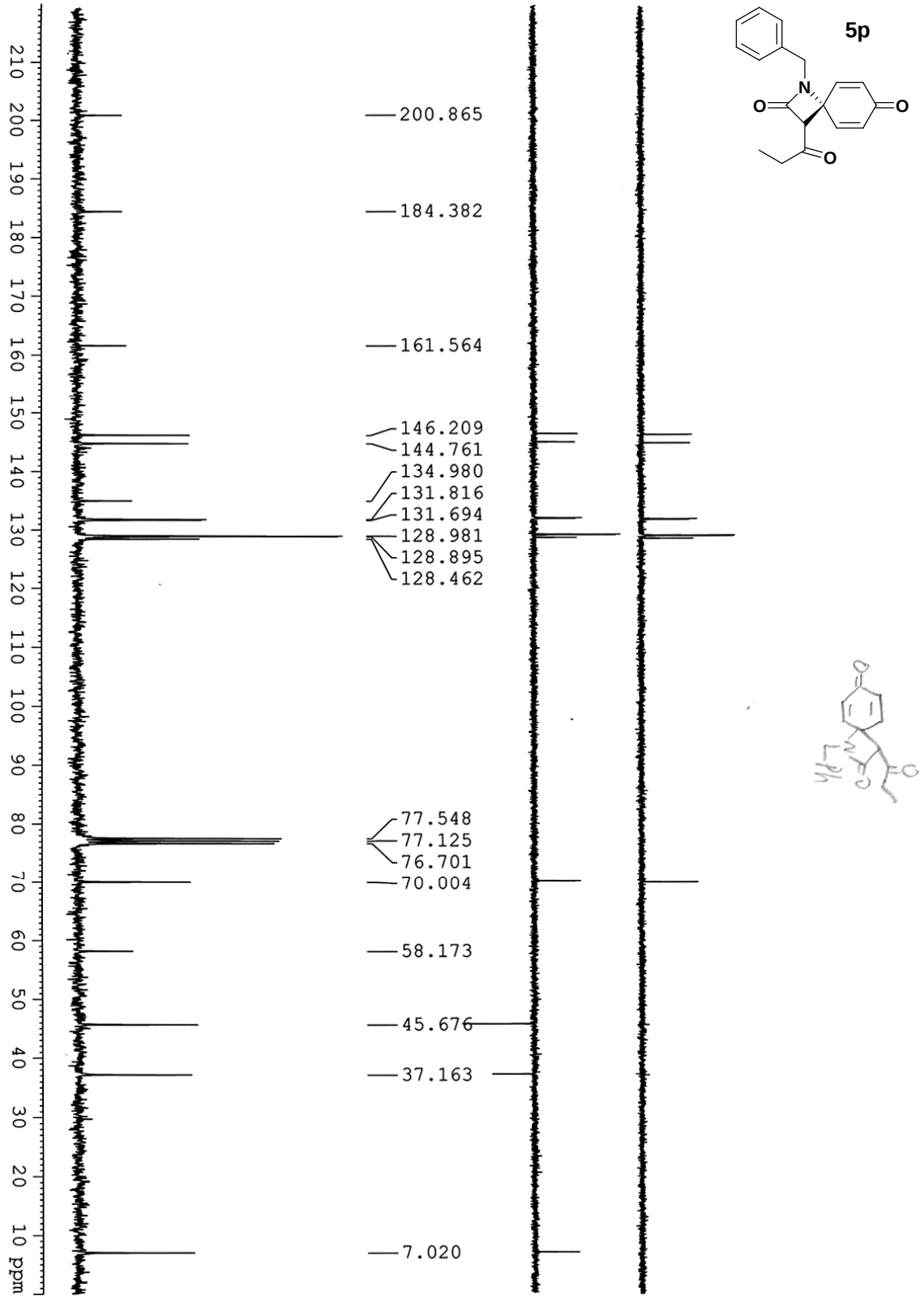
1D NMR plot parameters
CX 23.00 cm
CY 8.00 cm
F1P 10.046 ppm
F1 3015.23 Hz
F2P -0.204 ppm
F2 -61.35 Hz
PPMCM 0.44569 ppm/cm
HZCM 133.76456 Hz/cm

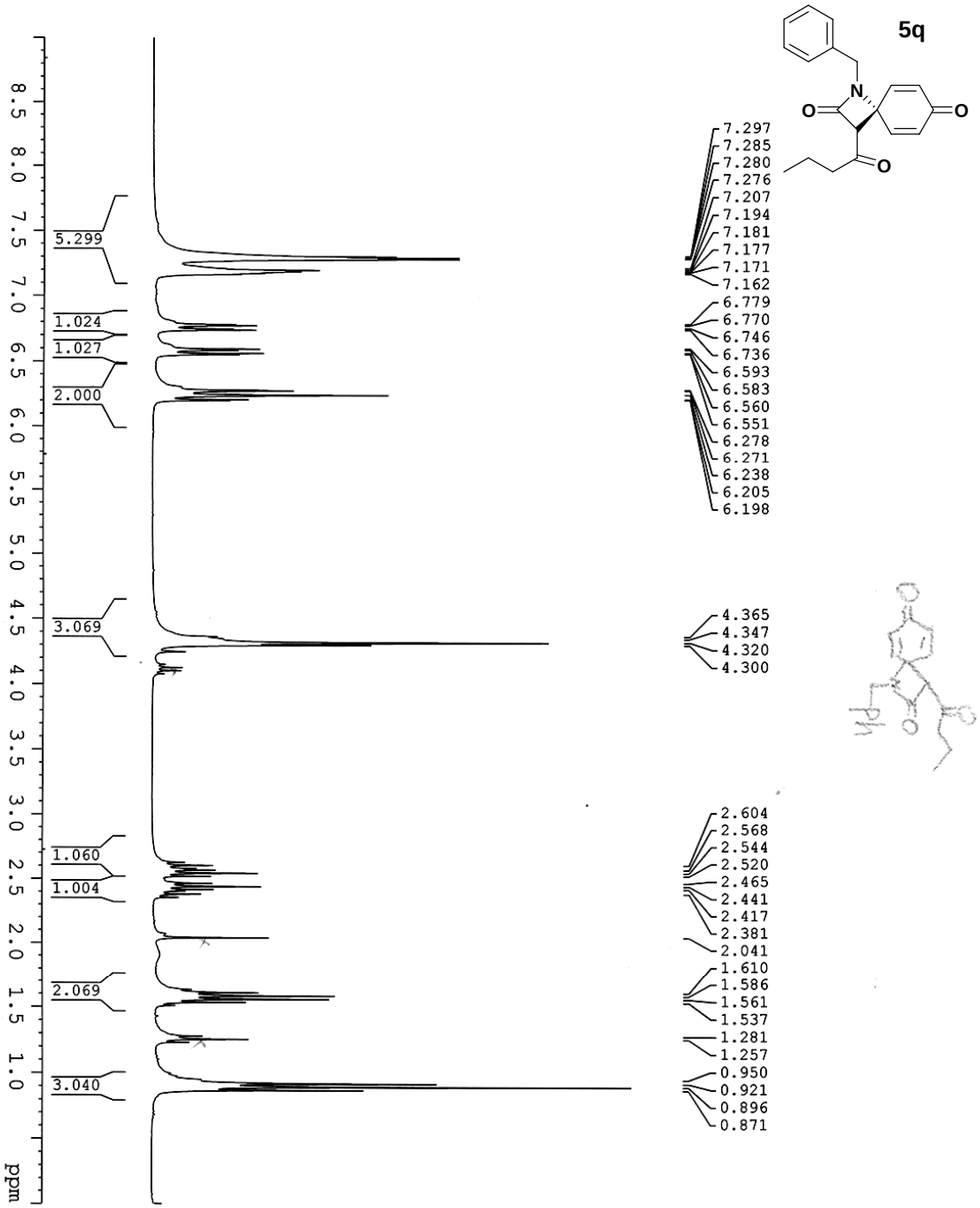

190.968
183.964
162.272
146.693
144.681
135.637
134.392
132.810
128.970
128.393
77.502
77.079
76.654
66.659
58.218
49.713
27.965
20.196
20.162
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm
1062
1063
1064
N
O
O
O
5k

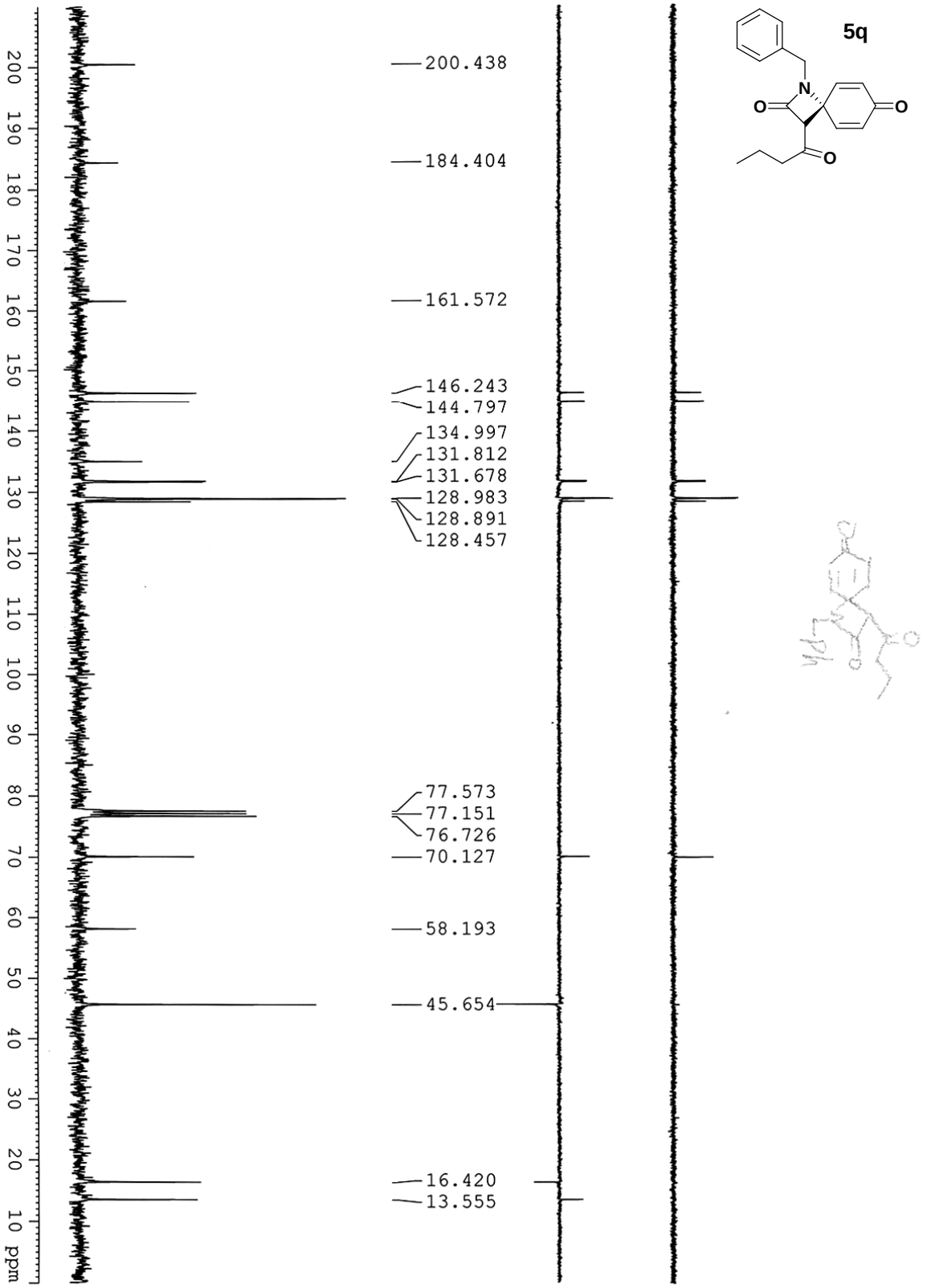

5l
7.790
7.767
7.762
7.641
7.637
7.616
7.595
7.591
7.486
7.459
7.434
7.371
7.365
7.359
7.341
7.338
7.322
7.297
7.268
7.258
7.235
7.225
7.162
7.158
7.153
7.134
7.110
7.106
7.098
7.088
7.064
7.053
6.706
6.700
6.673
6.666
6.403
6.397
6.369
6.362
5.238
1.999
1.047
2.012
5.097
1.988
1.007
0.982
1.000
ppm
NAME xzx
EXPNO 974
PROCNO 1
Date_ 20101207
Time_ 20.00
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 256
DW 81.000 usec
DE 6.50 usec
TE 293.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.90 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300040 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

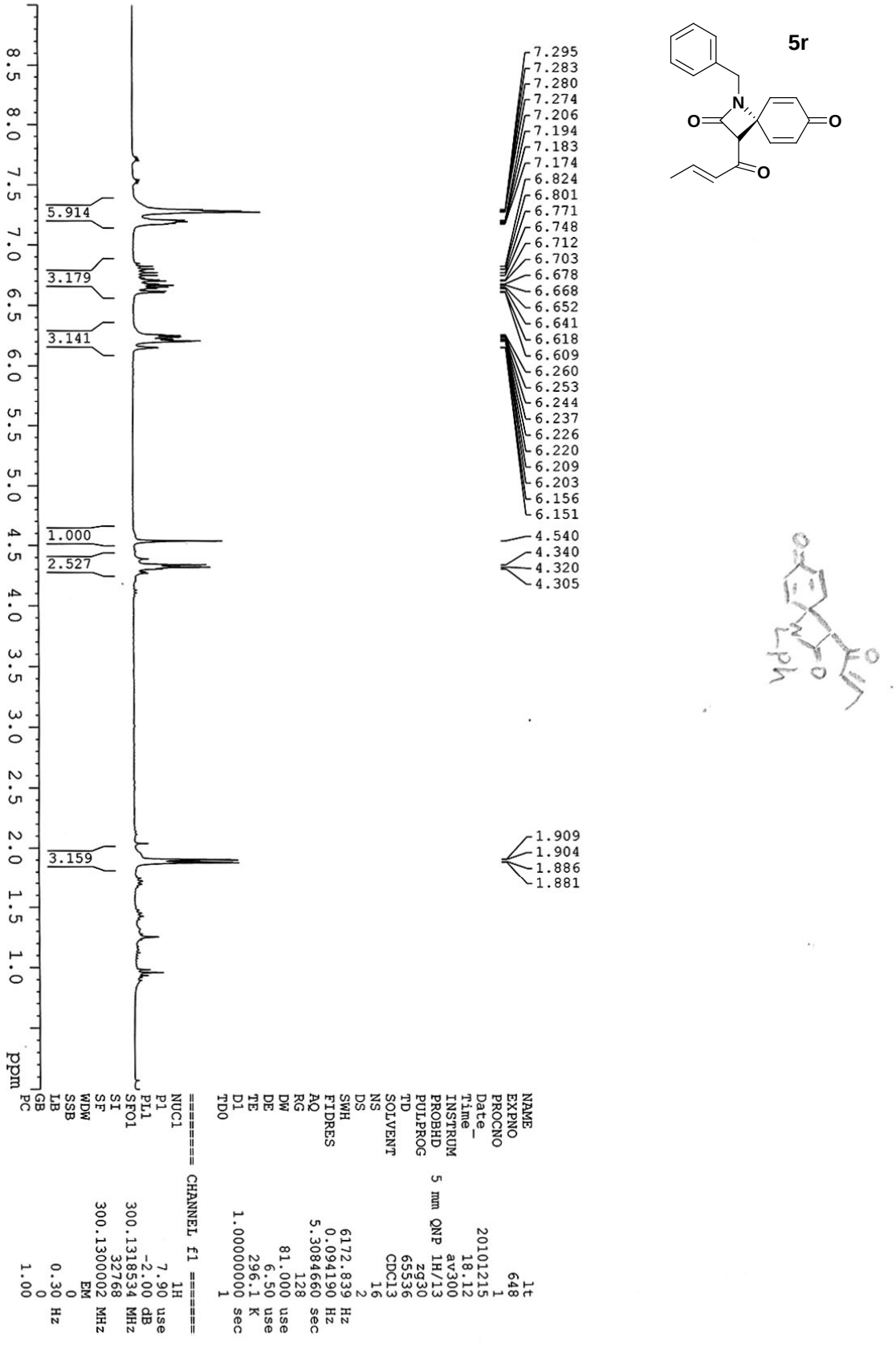

975
190.383
183.622
158.987
147.213
145.184
137.280
135.556
134.678
133.462
133.044
129.418
129.114
128.418
125.281
116.608
77.465
77.041
76.617
66.277
58.596
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 ppm
976
5l
N
O
O
O
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 ppm

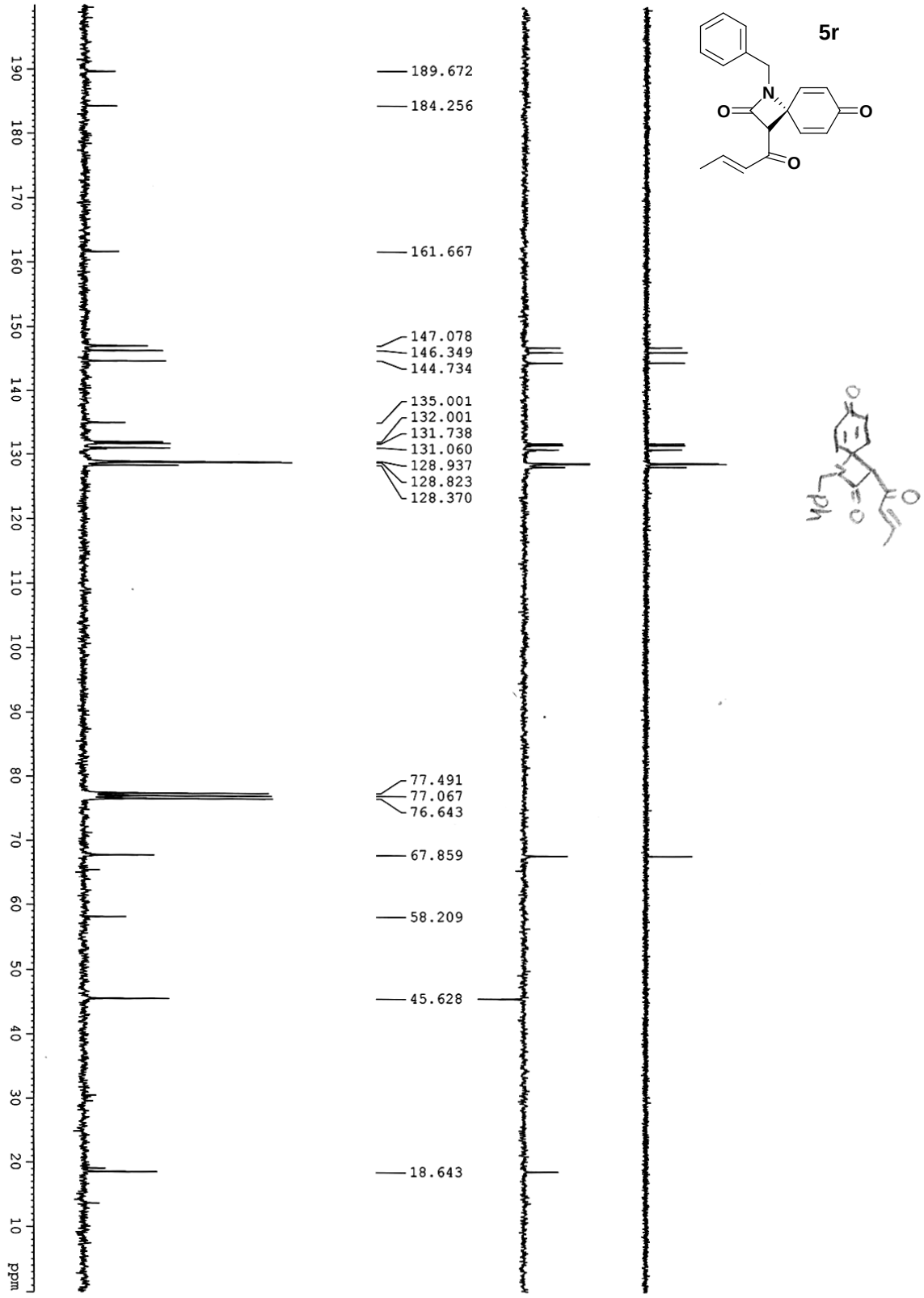

5p
7.298
7.286
7.281
7.276
7.264
7.206
7.193
7.180
7.176
7.170
7.161
6.784
6.774
6.751
6.740
6.592
6.582
6.559
6.549
6.279
6.273
6.245
6.239
6.232
6.205
6.199
4.365
4.332
4.316
4.301
2.679
2.655
2.641
2.617
2.593
2.510
2.486
2.462
2.438
2.424
2.400
1.255
1.059
1.035
1.011
1.000
5.261
1.059
1.041
2.000
3.043
1.072
1.026
3.062
ppm
NAME lt
EXPNO 617
PROCNO 1
Date_ 20101120
Time_ 21.33
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 161.3
DW 81.000 use
DE 6.50 use
TE 295.8 K
D1 1.00000000 sec
TD0 1
========== CHANNEL f1 ========
NUC1 1H
P1 7.90 use
PL1 -2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1299996 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

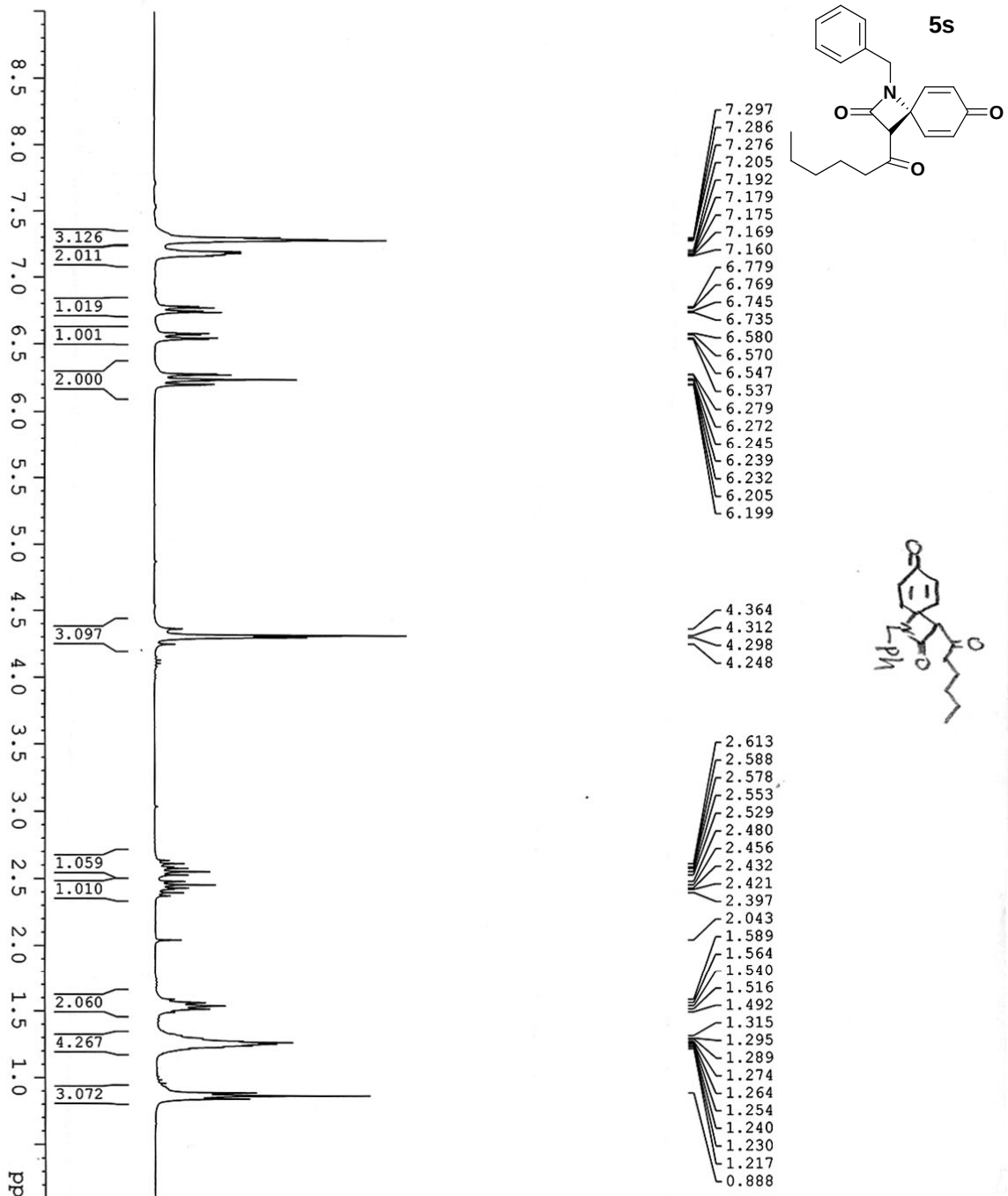

5p
200.865
184.382
161.564
146.209
144.761
134.980
131.816
131.694
128.981
128.895
128.462
77.548
77.125
76.701
70.004
58.173
45.676
37.163
7.020
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

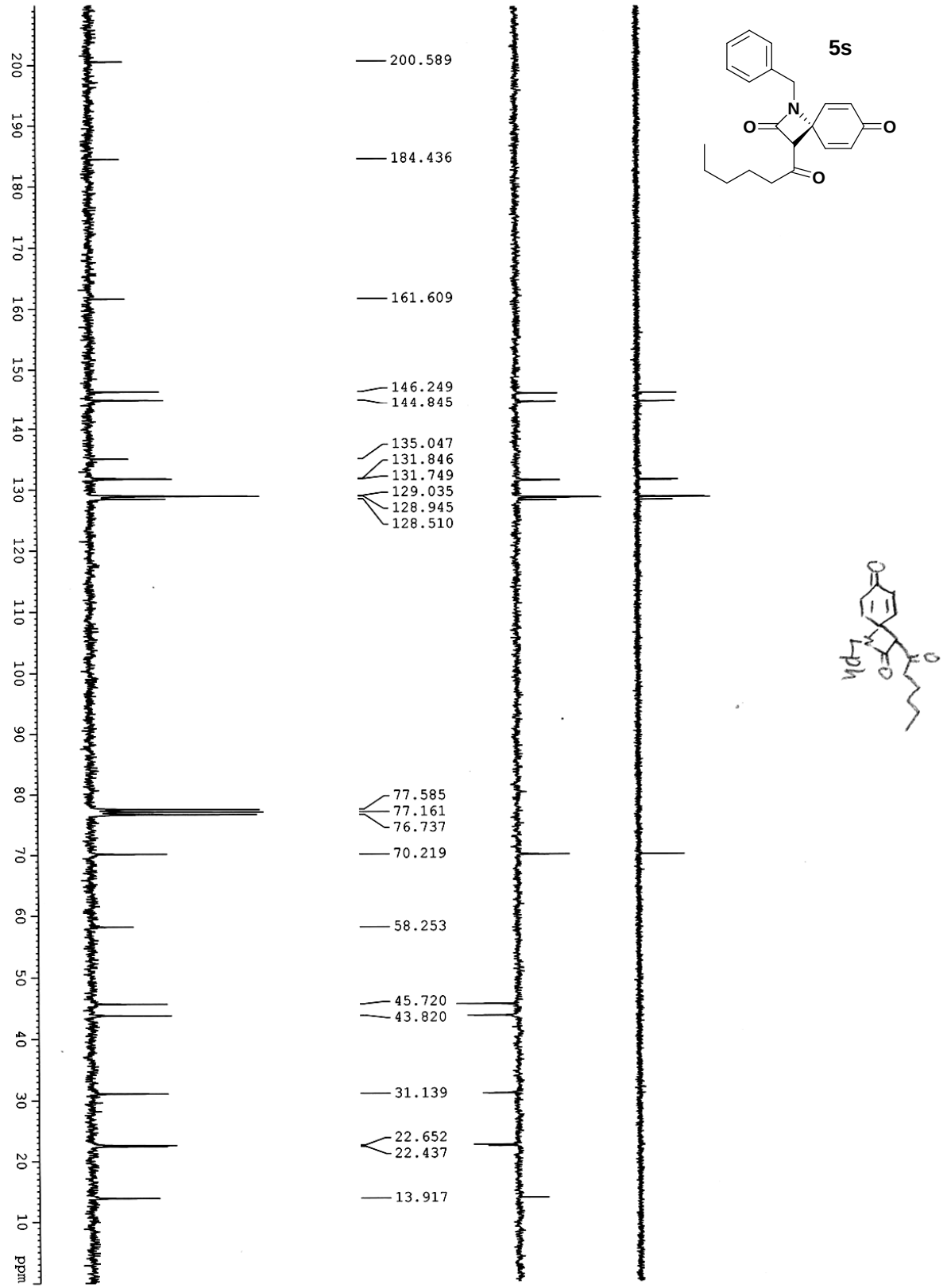

5q
7.297
7.285
7.280
7.276
7.207
7.194
7.181
7.177
7.171
7.162
6.779
6.770
6.746
6.736
6.593
6.583
6.560
6.551
6.278
6.271
6.238
6.205
6.198
4.365
4.347
4.320
4.300
2.604
2.568
2.544
2.520
2.465
2.441
2.417
2.381
2.041
1.610
1.586
1.561
1.537
1.281
1.257
0.950
0.921
0.896
0.871
5.299
1.024
1.027
2.000
3.069
1.060
1.004
2.069
3.040
ppm
NAME lt
EXPNO 602
PROCNO 1
Date_ 20101113
Time_ 22.16
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 10
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 se
RG 114
DW 81.000 us
DE 6.50 us
TE 295.4 K
D1 1.00000000 se
1
TD0
======== CHANNEL f1 ========
NUC1 1H
P1 7.90 us
PL1 -2.00 dB
SFO1 300.1318534 MH
SI 32768
SF 300.1299977 MH
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

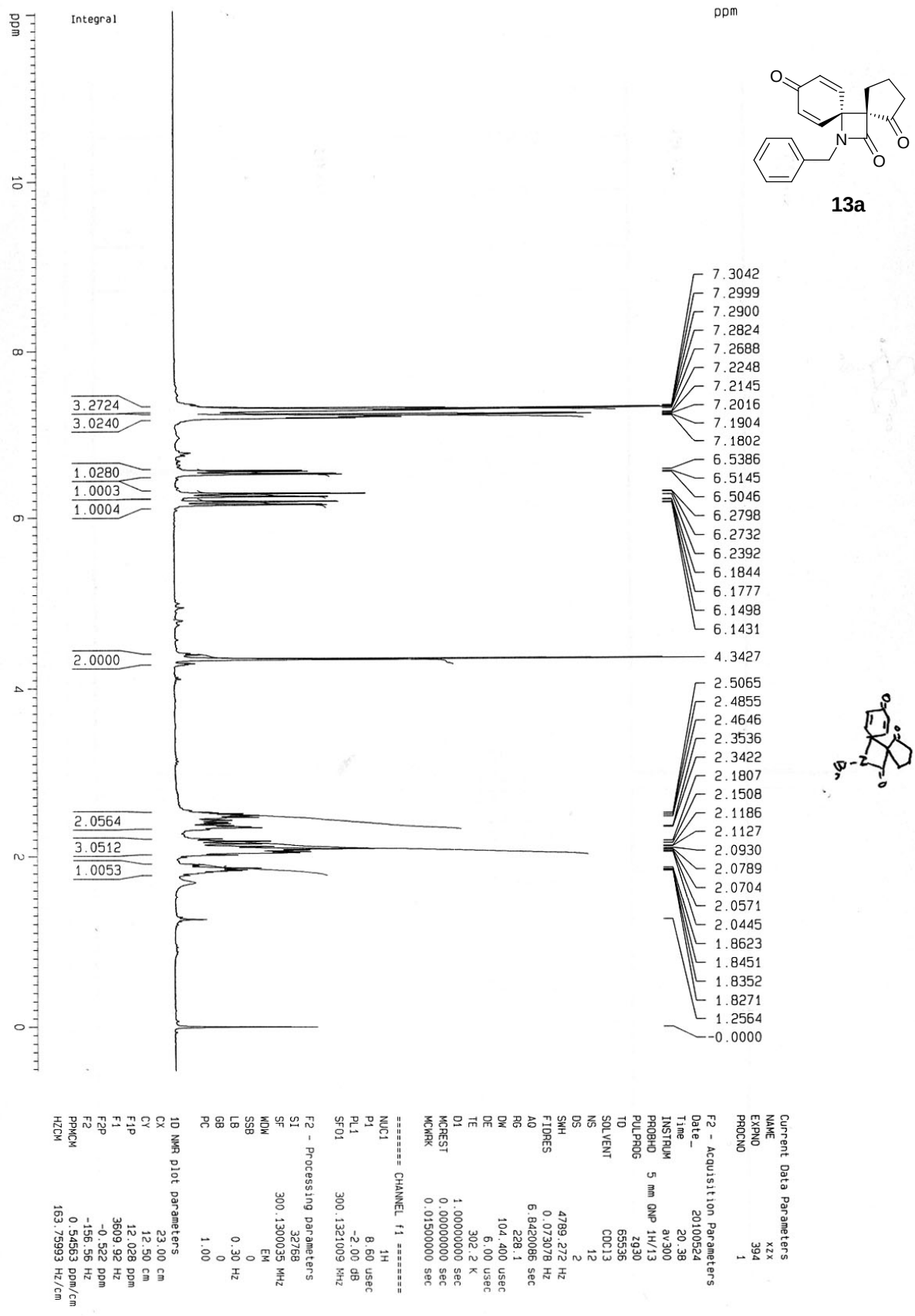

5q
200.438
184.404
161.572
146.243
144.797
134.997
131.812
131.678
128.983
128.891
128.457
77.573
77.151
76.726
70.127
58.193
45.654
16.420
13.555
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

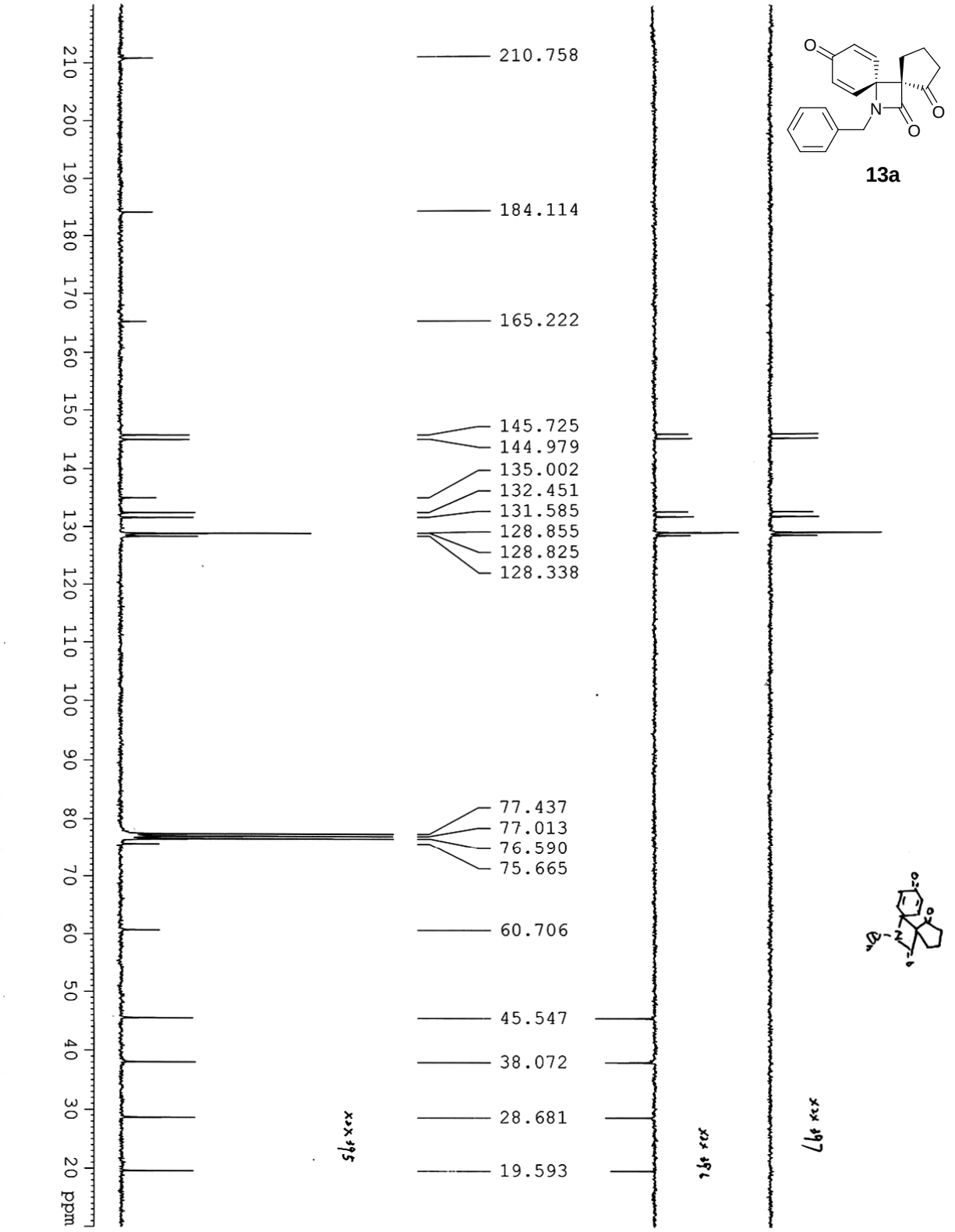

5r
7.295
7.283
7.280
7.274
7.206
7.194
7.183
7.174
6.824
6.801
6.771
6.748
6.712
6.703
6.678
6.668
6.652
6.641
6.618
6.609
6.260
6.253
6.244
6.237
6.226
6.220
6.209
6.203
6.156
6.151
4.540
4.340
4.320
4.305
1.909
1.904
1.886
1.881
5.914
3.179
3.141
1.000
2.527
3.159
ppm
NAME lt
EXPNO 648
PROCNO 1
Date_ 20101215
Time_ 18.12
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 128
DW 81.000 use
DE 6.50 use
TE 296.1 K
D1 1.00000000 sec
TDO 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.90 use
PL1 -2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300002 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


5r
189.672
184.256
161.667
147.078
146.349
144.734
135.001
132.001
131.738
131.060
128.937
128.823
128.370
77.491
77.067
76.643
67.859
58.209
45.628
18.643
ppm

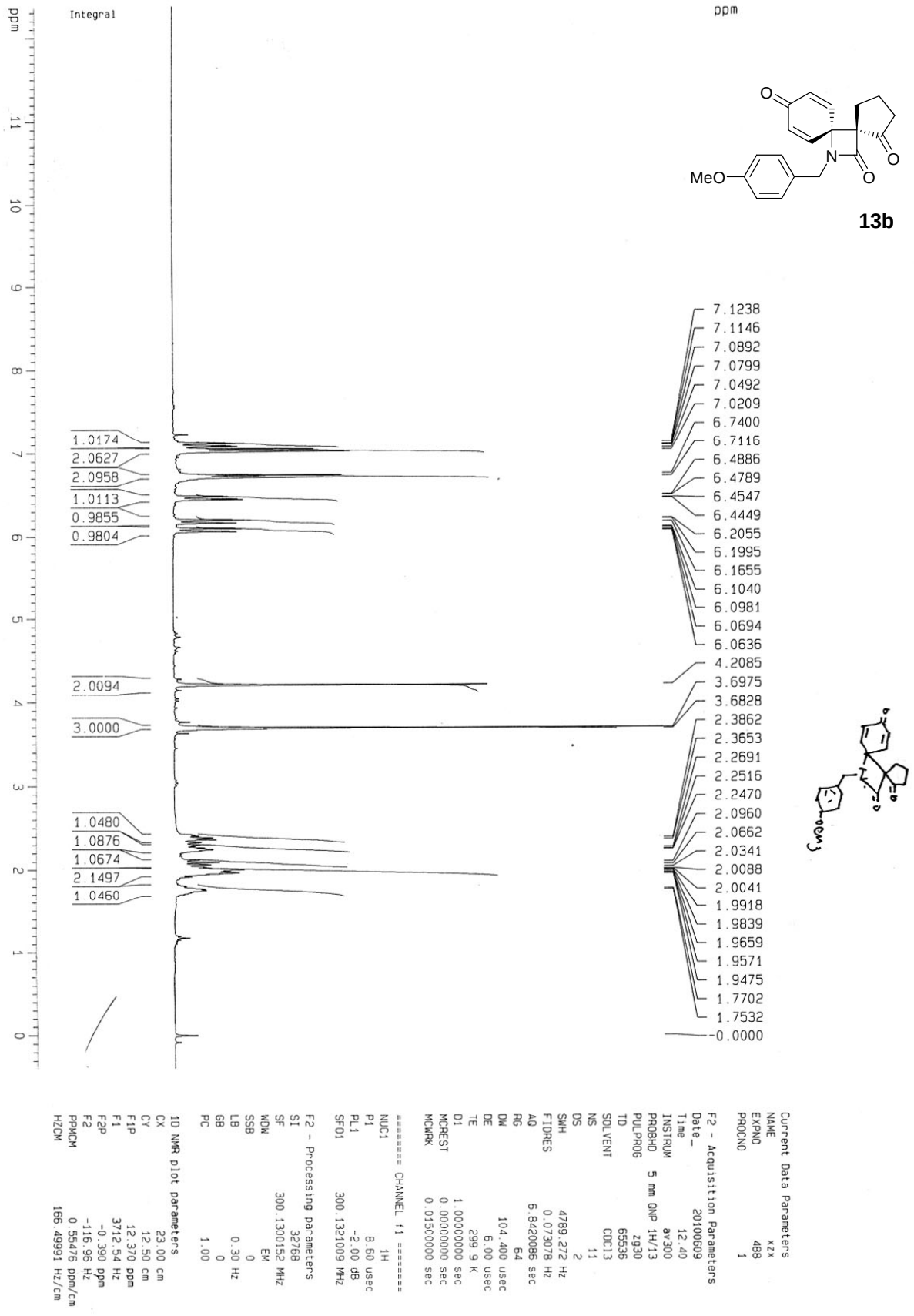

5s
N
O
O
O
7.297
7.286
7.276
7.205
7.192
7.179
7.175
7.169
7.160
6.779
6.769
6.745
6.735
6.580
6.570
6.547
6.537
6.279
6.272
6.245
6.239
6.232
6.205
6.199
4.364
4.312
4.298
4.248
2.613
2.588
2.578
2.553
2.529
2.480
2.456
2.432
2.421
2.397
2.043
1.589
1.564
1.540
1.516
1.492
1.315
1.295
1.289
1.274
1.264
1.254
1.240
1.230
1.217
0.888
3.126
2.011
1.019
1.001
2.000
3.097
1.059
1.010
2.060
4.267
3.072
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
ppm
NAME lt
EXPNO 668
PROCNO 1
Date_ 20101224
Time_ 21.44
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 se
RG 128
DW 81.000 us
DE 6.50 us
TE 295.7 K
D1 1.00000000 se
TD0 1
========== CHANNEL f1 ======
NUC1 1H
P1 7.90 us
PL1 -2.00 dB
SFO1 300.1318534 MH
SI 32768
SF 300.1300012 MH
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

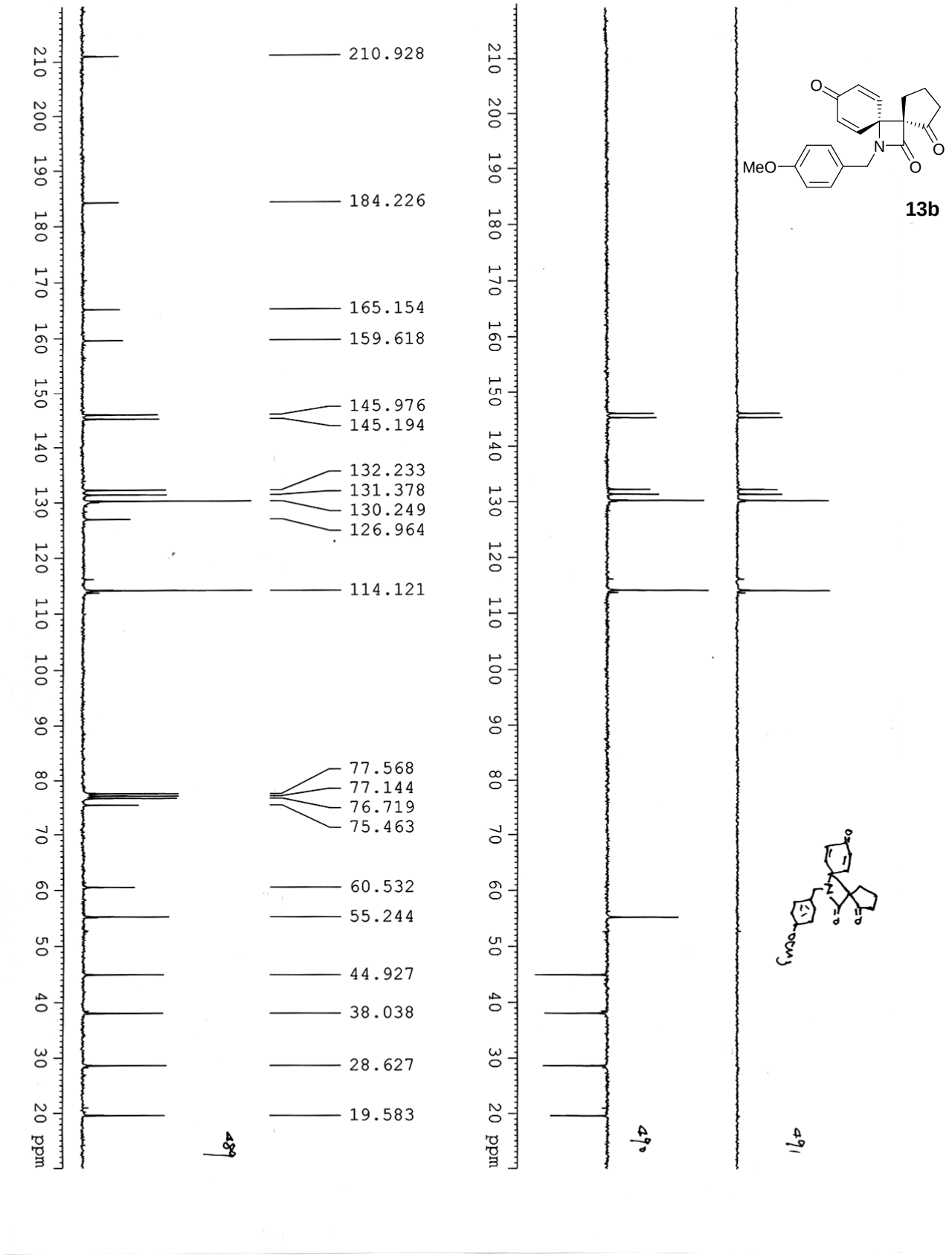

200.589
184.436
161.609
146.249
144.845
135.047
131.846
131.749
129.035
128.945
128.510
77.585
77.161
76.737
70.219
58.253
45.720
43.820
31.139
22.652
22.437
13.917
ppm
5s

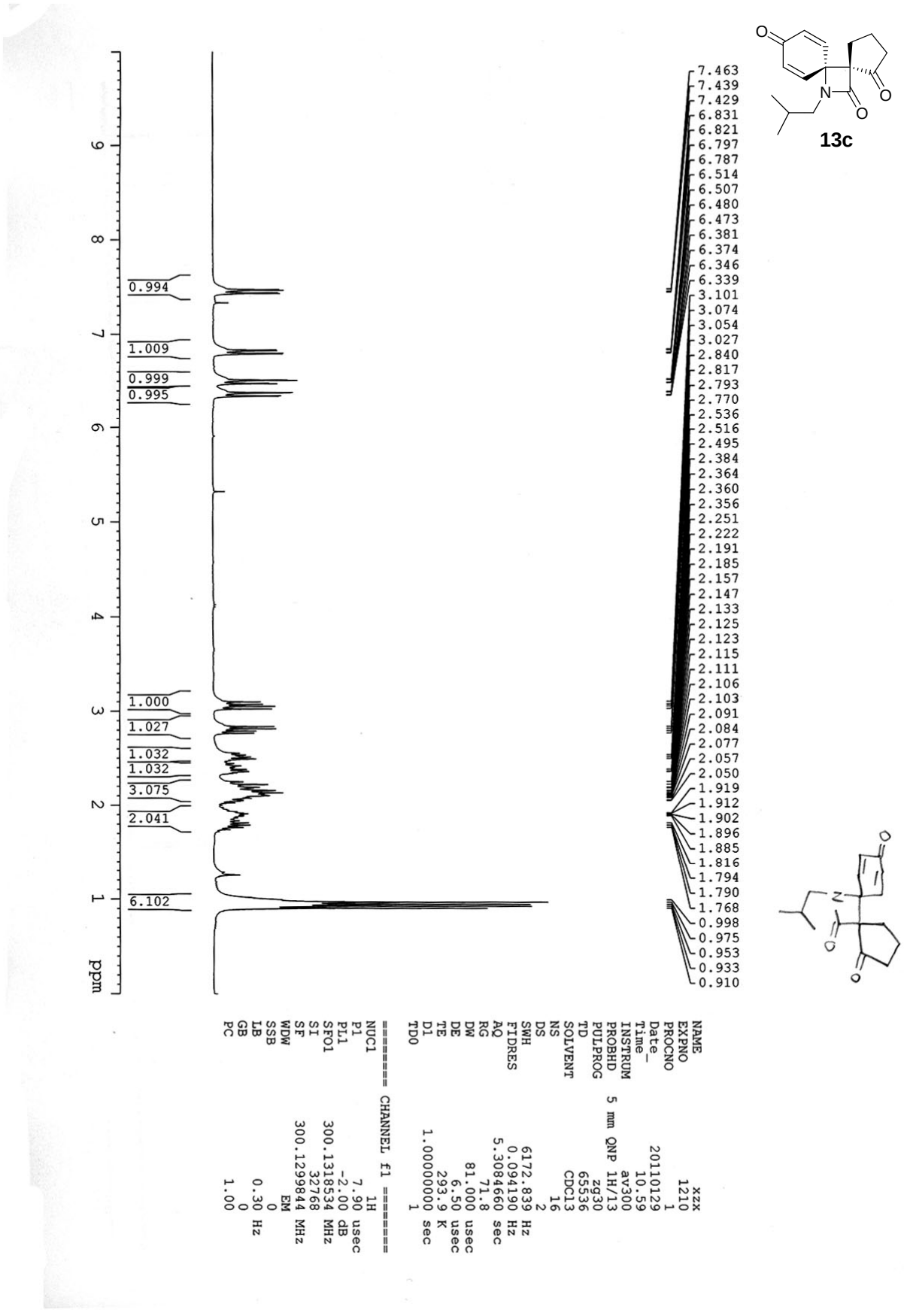

ppm
Integral
7.3042
7.2999
7.2900
7.2824
7.2688
7.2248
7.2145
7.2016
7.1904
7.1802
6.5386
6.5145
6.5046
6.2798
6.2732
6.2392
6.1844
6.1777
6.1498
6.1431
4.3427
2.5065
2.4855
2.4646
2.3536
2.3422
2.1807
2.1508
2.1186
2.1127
2.0930
2.0789
2.0704
2.0571
2.0445
1.8623
1.8451
1.8352
1.8271
1.2564
-0.0000
3.2724
3.0240
1.0280
1.0003
1.0004
2.0000
2.0564
3.0512
1.0053
O
O
N
O
13a
Current Data Parameters
NAME xzx
EXPNO 394
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100524
Time 20.38
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 12
DS 2
SWH 4789.272 Hz
FIDRES 0.073078 Hz
AQ 6.8420086 sec
RG 228.1
DW 104.400 usec
DE 6.00 usec
TE 302.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.60 usec
PL1 -2.00 dB
SF01 300.1321009 MHz
F2 - Processing parameters
SI 32768
SF 300.1300035 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 23.00 cm
CY 12.50 cm
F1P 12.028 ppm
F1 3609.92 Hz
F2P -0.522 ppm
F2 -156.56 Hz
PPMCM 0.54563 ppm/cm
HZCM 163.75993 Hz/cm

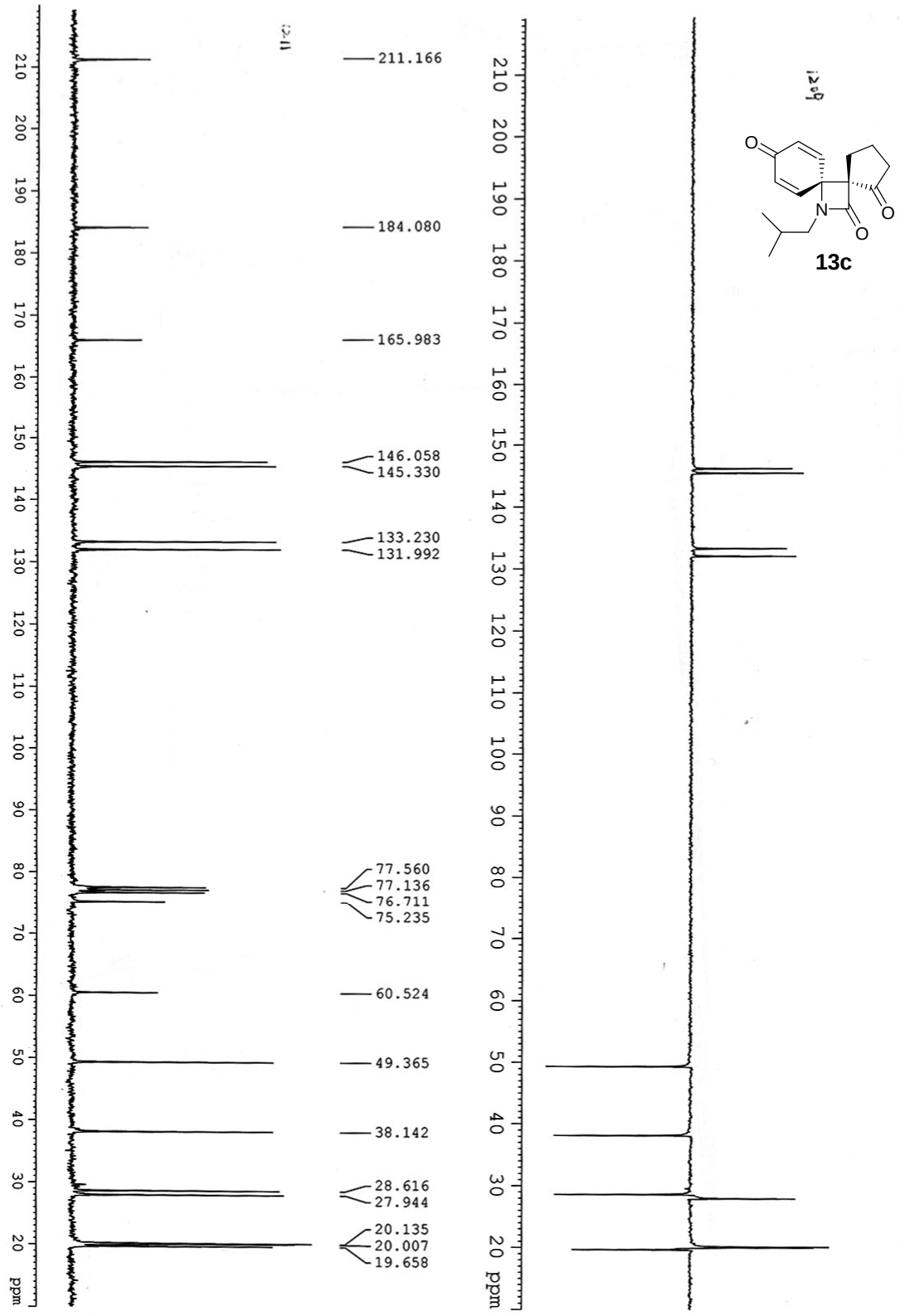

210.758
184.114
165.222
145.725
144.979
135.002
132.451
131.585
128.855
128.825
128.338
77.437
77.013
76.590
75.665
60.706
45.547
38.072
28.681
19.593
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm
O
O
N
O
O

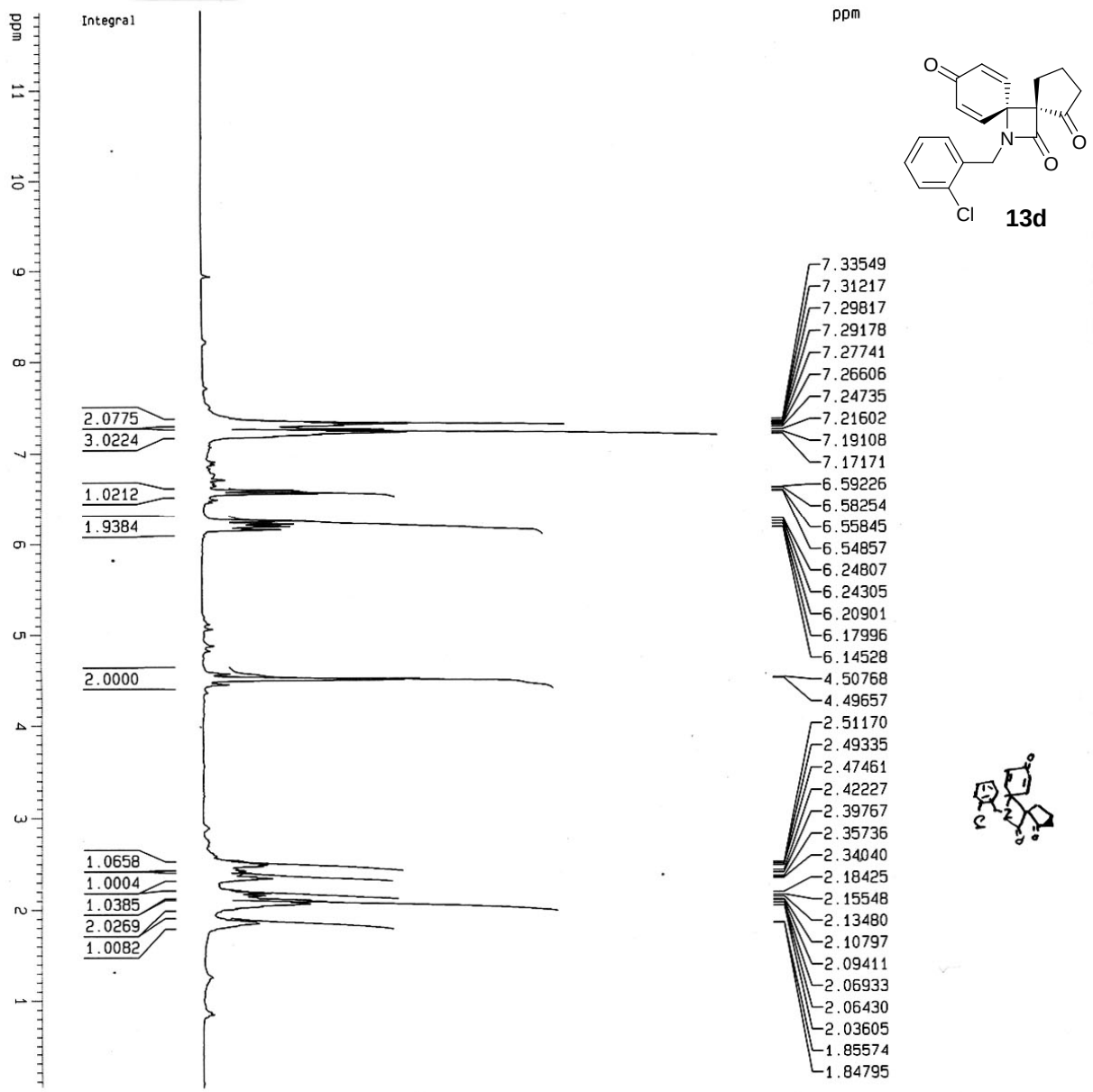
13a


ppm
Integral
MeO
13b
7.1238
7.1146
7.0892
7.0799
7.0492
7.0209
6.7400
6.7116
6.4886
6.4789
6.4547
6.4449
6.2055
6.1995
6.1655
6.1040
6.0981
6.0694
6.0636
4.2085
3.6975
3.6828
2.3862
2.3653
2.2691
2.2516
2.2470
2.0960
2.0662
2.0341
2.0088
2.0041
1.9918
1.9839
1.9659
1.9571
1.9475
1.7702
1.7532
-0.0000
1.0174
2.0627
2.0958
1.0113
0.9855
0.9804
2.0094
3.0000
1.0480
1.0876
1.0674
2.1497
1.0460
Current Data Parameters
NAME xzx
EXPNO 488
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100609
Time 12.40
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 11
DS 2
SWH 4789.272 Hz
FIDRES 0.073078 Hz
AQ 6.8420086 sec
RG 64
DW 104.400 usec
DE 6.00 usec
TE 299.9 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
========== CHANNEL f1 ==========
NUC1 1H
P1 8.60 usec
PL1 -2.00 dB
SFO1 300.1321009 MHz
F2 - Processing parameters
SI 32768
SF 300.1300152 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 23.00 cm
CY 12.50 cm
F1P 12.370 ppm
F1 3712.54 Hz
F2P -0.390 ppm
F2 -116.96 Hz
PPMCM 0.55476 ppm/cm
HZCM 166.49991 Hz/cm


MeO
O
O
O
N
13b
210.928
184.226
165.154
159.618
145.976
145.194
132.233
131.378
130.249
126.964
114.121
77.568
77.144
76.719
75.463
60.532
55.244
44.927
38.038
28.627
19.583
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm
49a
49b
49c


13c
7.463
7.439
7.429
6.831
6.821
6.797
6.787
6.514
6.507
6.480
6.473
6.381
6.374
6.346
6.339
3.101
3.074
3.054
3.027
2.840
2.817
2.793
2.770
2.536
2.516
2.495
2.384
2.364
2.360
2.356
2.251
2.222
2.191
2.185
2.157
2.147
2.133
2.125
2.123
2.115
2.111
2.106
2.103
2.091
2.084
2.077
2.057
2.050
1.919
1.912
1.902
1.896
1.885
1.816
1.794
1.790
1.768
0.998
0.975
0.953
0.933
0.910
0.994
1.009
0.999
0.995
1.000
1.027
1.032
1.032
3.075
2.041
6.102
ppm
NAME xzx
EXPNO 1210
PROCNO 1
Date_ 20110129
Time_ 10.59
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 71.8
DW 81.000 usec
DE 6.50 usec
TE 293.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.90 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1299844 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


211.166
184.080
165.983
146.058
145.330
133.230
131.992
77.560
77.136
76.711
75.235
60.524
49.365
38.142
28.616
27.944
20.135
20.007
19.658
ppm
O
N
O
O
13c



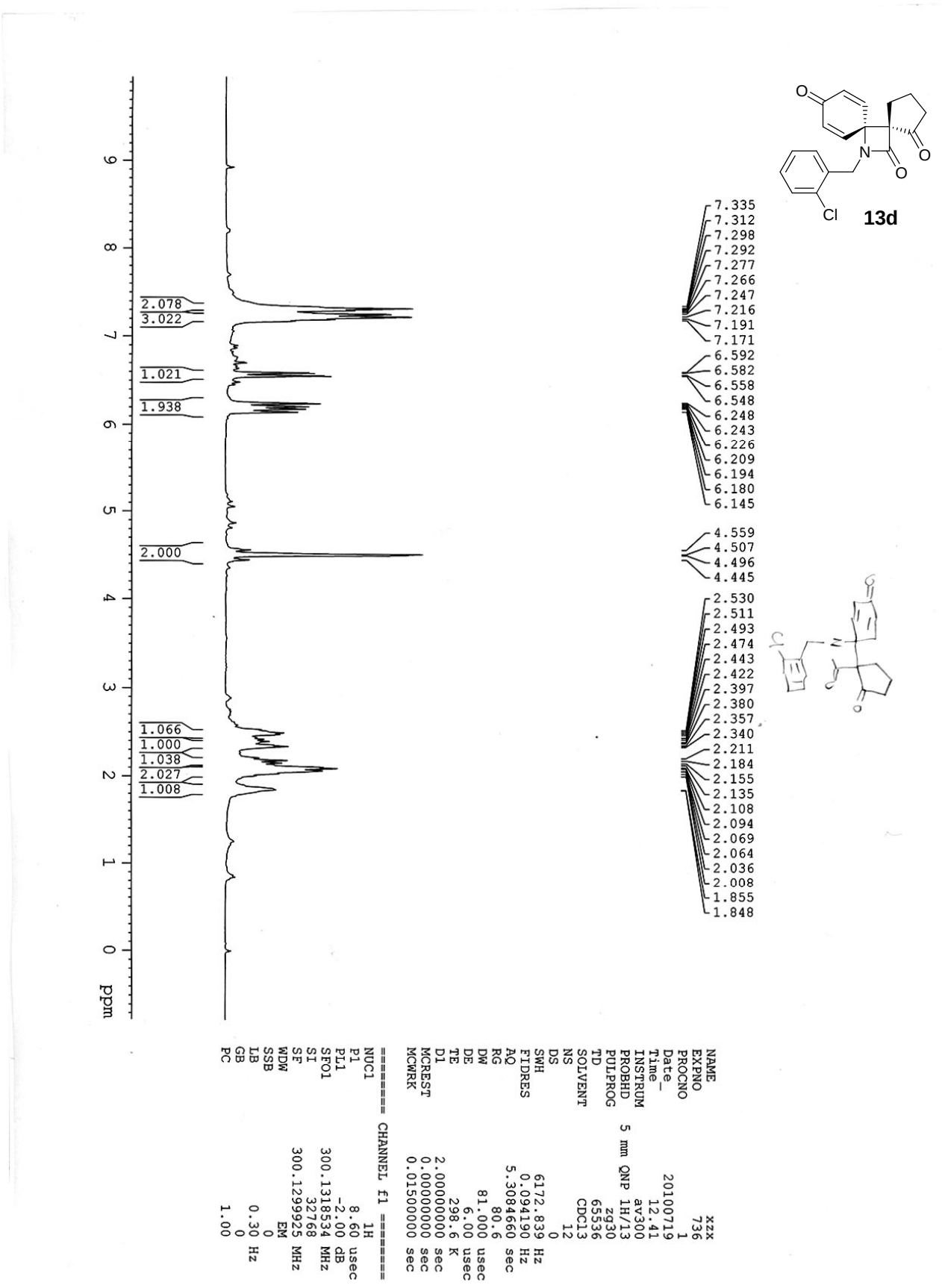

```
Current Data Parameters
NAME                  xzx
EXPNO                 736
PROCNO                1

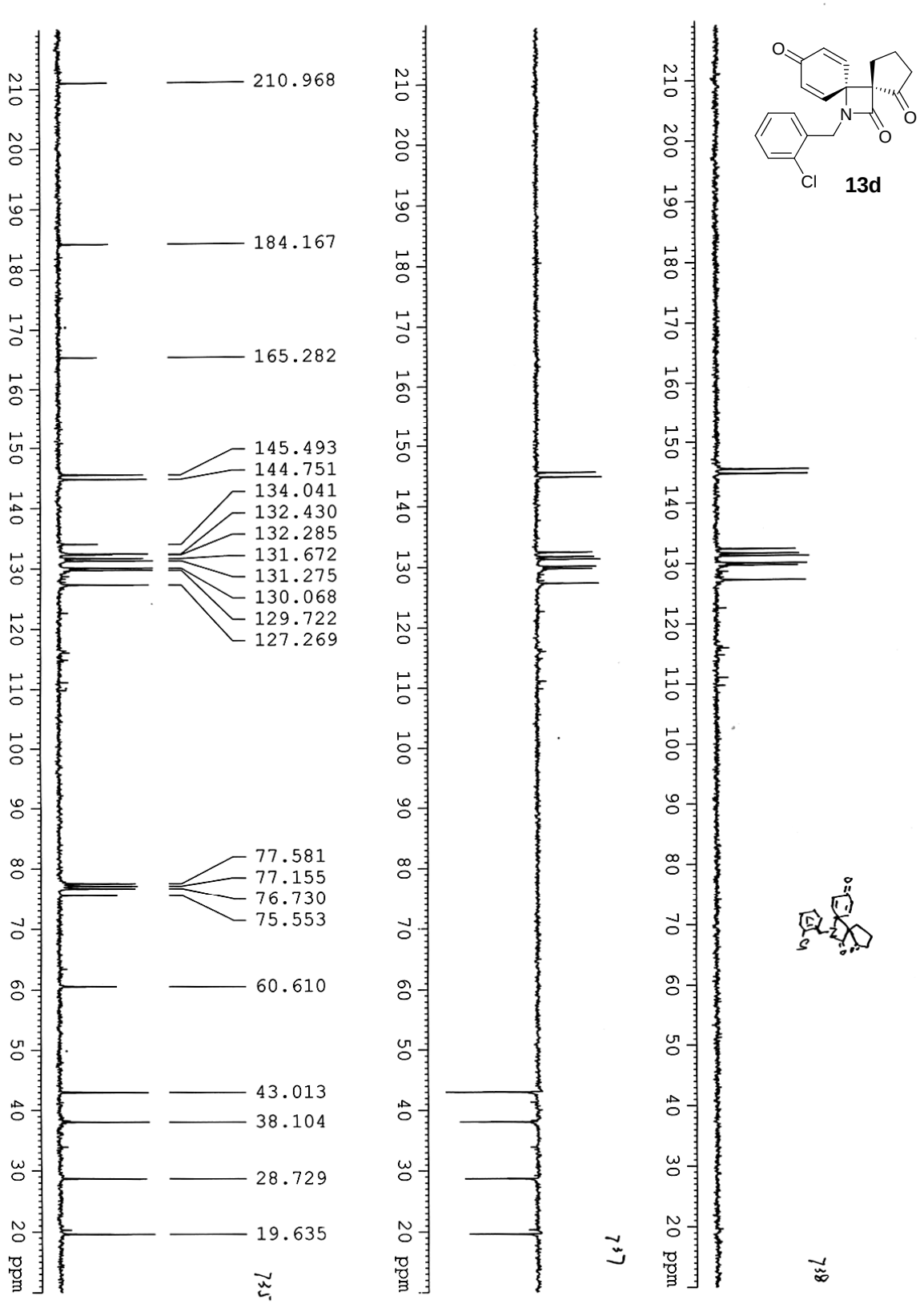
F2 - Acquisition Parameters
Date_              20100719
Time                  12.41
INSTRUM               av300
PROBHD     5 mm QNP 1H/13
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      12
DS                       0
SWH              6172.839 Hz
FIDRES           0.094190 Hz
AQ               5.3084660 sec
RG                    80.6
DW                81.000 usec
DE                 6.00 usec
TE                298.6 K
D1             2.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

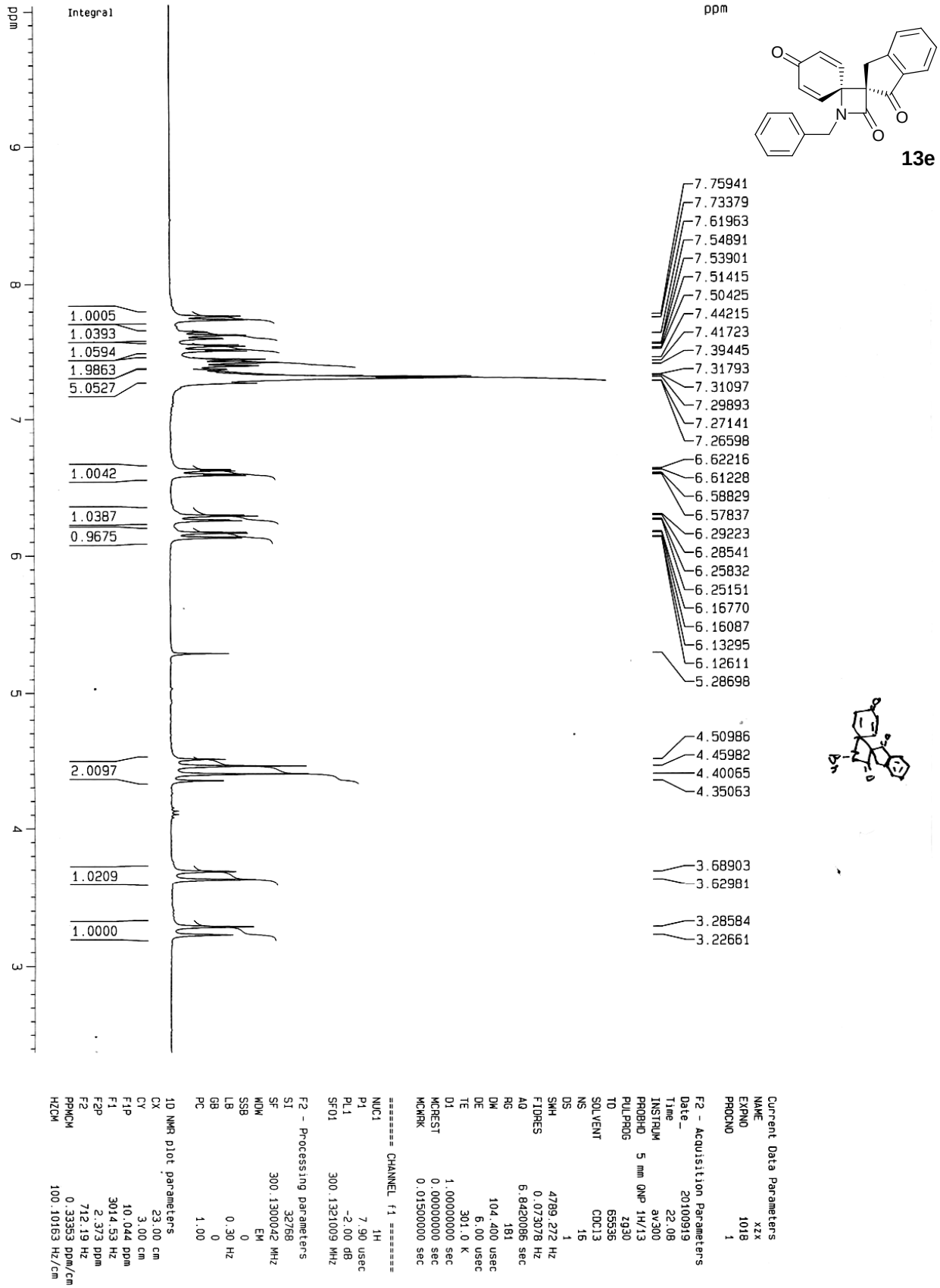
======== CHANNEL f1 ========
NUC1                  1H
P1                 8.60 usec
PL1                -2.00 dB
SF01        300.1318534 MHz

F2 - Processing parameters
SI                   32768
SF          300.1299925 MHz
MDW                     EM
SSB                      0
LB                  0.30 Hz
GB                       0
PC                    1.00

1D NMR plot parameters
CX                  20.00 cm
CY                   4.00 cm
F1P                11.850 ppm
F1               3556.39 Hz
F2P                 0.039 ppm
F2                 11.66 Hz
PPMCM          0.59053 ppm/cm
HZCM          177.23679 Hz/cm
```


13d
7.335
7.312
7.298
7.292
7.277
7.266
7.247
7.216
7.191
7.171
6.592
6.582
6.558
6.548
6.248
6.243
6.226
6.209
6.194
6.180
6.145
4.559
4.507
4.496
4.445
2.530
2.511
2.493
2.474
2.443
2.422
2.397
2.380
2.357
2.340
2.211
2.184
2.155
2.135
2.108
2.094
2.069
2.064
2.036
2.008
1.855
1.848
2.078
3.022
1.021
1.938
2.000
1.066
1.000
1.038
2.027
1.008
ppm
NAME xzx
EXPNO 736
PROCNO 1
Date_ 20100719
Time_ 12.41
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 12
DS 0
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 80.6
DW 81.000 usec
DE 6.00 usec
TE 298.6 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
========== CHANNEL f1 ========
NUC1 1H
P1 8.60 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1299925 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


O
O
N
O
Cl
13d
210.968
184.167
165.282
145.493
144.751
134.041
132.430
132.285
131.672
131.275
130.068
129.722
127.269
77.581
77.155
76.730
75.553
60.610
43.013
38.104
28.729
19.635
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm



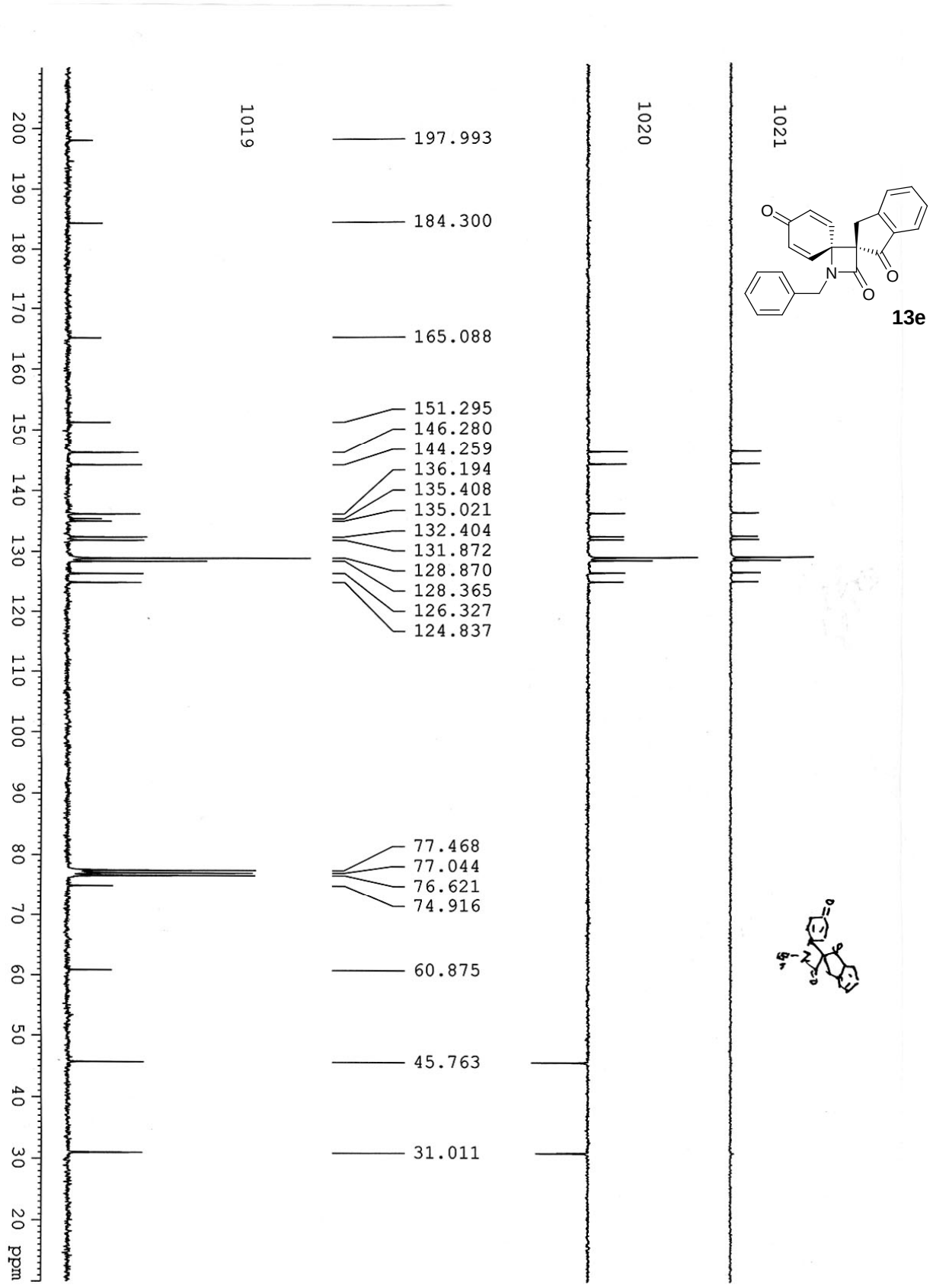
Current Data Parameters
NAME xzx
EXPNO 1018
PROCNO 1

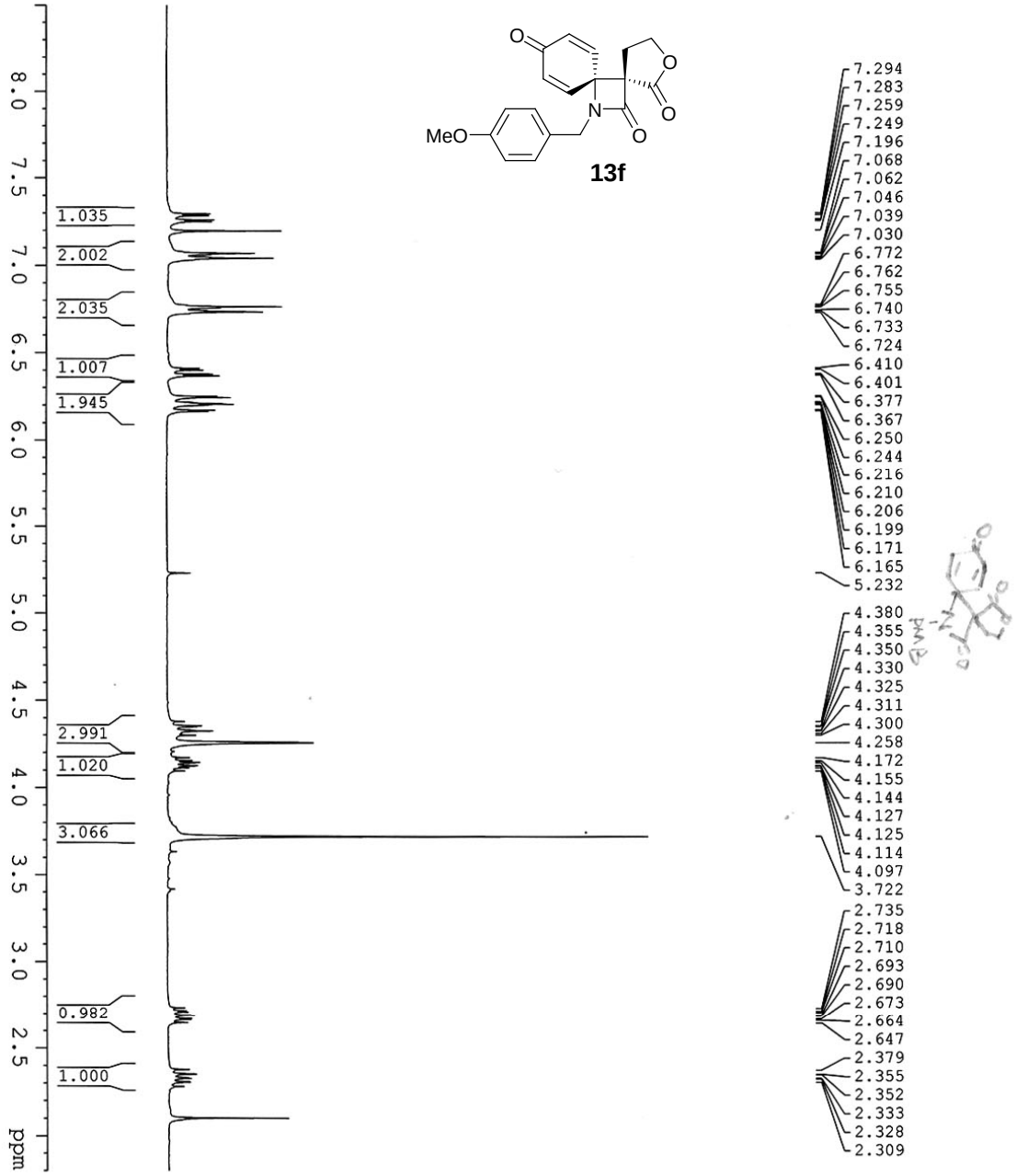
F2 - Acquisition Parameters
Date_ 20100919
Time 22.08
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 1
SWH 4789.272 Hz
FIDRES 0.073078 Hz
AQ 6.8420086 sec
RG 181
DW 104.400 usec
DE 6.00 usec
TE 301.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.90 usec
PL1 -2.00 dB
SFO1 300.1321009 MHz

F2 - Processing parameters
SI 32768
SF 300.1300042 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

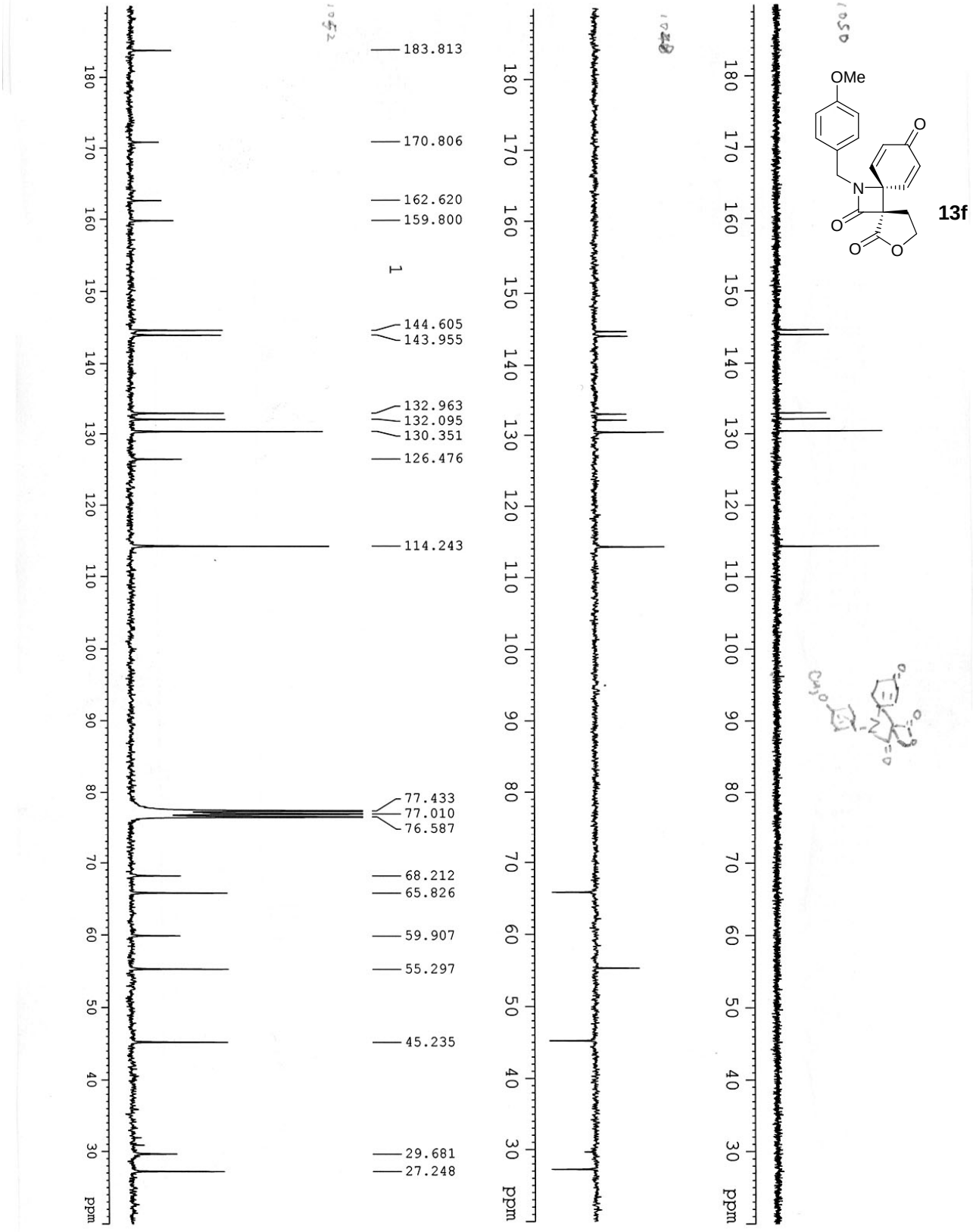
1D NMR plot parameters
CX 23.00 cm
CY 3.00 cm
F1P 10.044 ppm
F1 3014.53 Hz
F2P 2.373 ppm
F2 712.19 Hz
PPMCM 0.33353 ppm/cm
HZCM 100.10163 Hz/cm

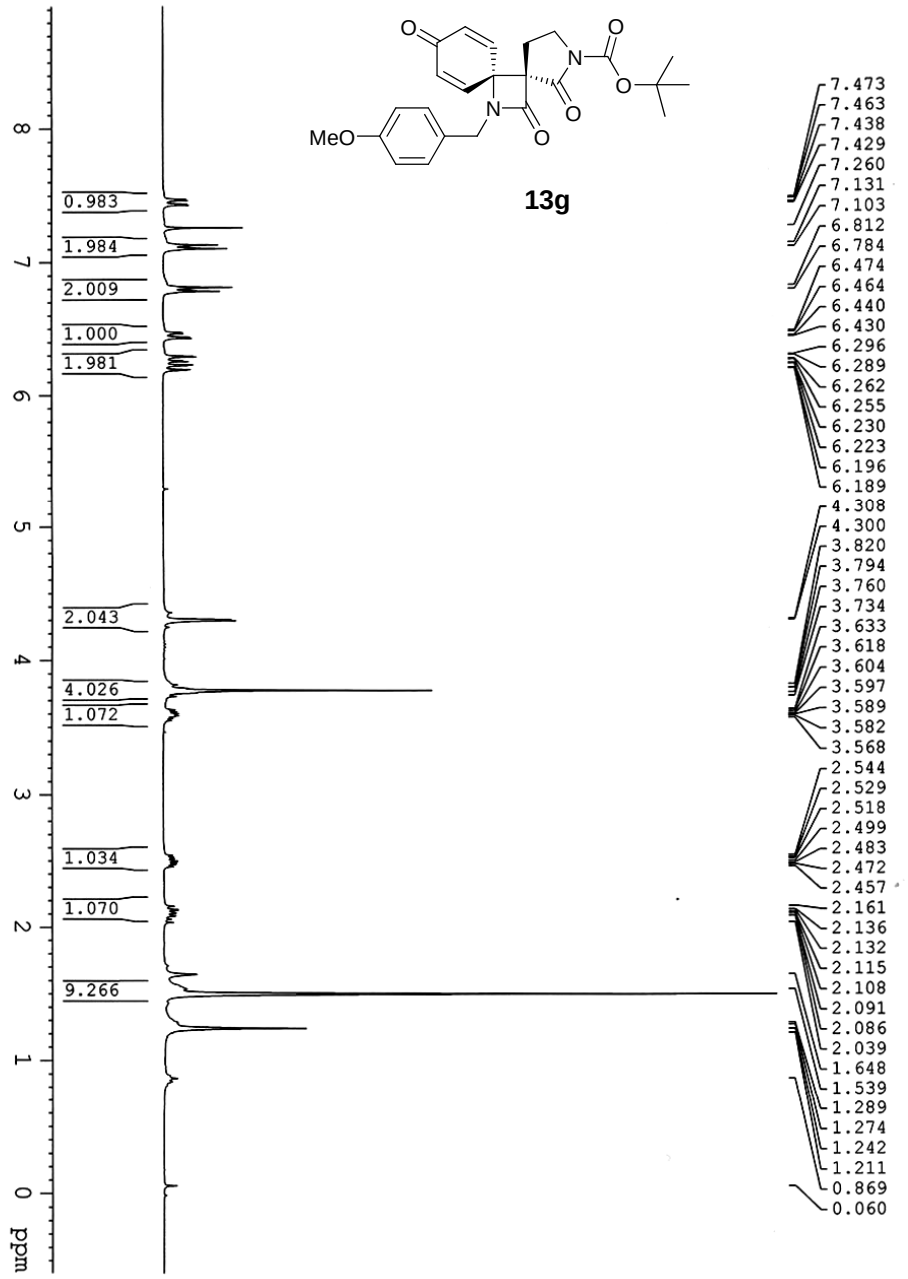

1019
1020
1021
197.993
184.300
165.088
151.295
146.280
144.259
136.194
135.408
135.021
132.404
131.872
128.870
128.365
126.327
124.837
77.468
77.044
76.621
74.916
60.875
45.763
31.011
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm
O
O
N
O
13e




```
NAME                                    xzx
EXPNO                                  1036
PROCNO                                    1
Date_                              20101223
Time_                                 17.45
INSTRUM                                av300
PROBHD     5 mm QNP 1H/13
PULPROG                                zg30
TD                                    65536
SOLVENT                               CDCl3
NS                                       16
DS                                        2
SWH                               6172.839 Hz
FIDRES                            0.094190 Hz
AQ                                5.3084660 sec
RG                                    456.1
DW                                 81.000 usec
DE                                  6.50 usec
TE                                    295.4 K
D1                                1.00000000 sec
TD0                                       1

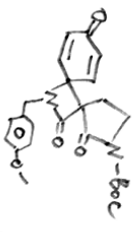
========== CHANNEL f1 ========
NUC1                                     1H
P1                                 7.90 usec
PL1                                -2.00 dB
SFO1                        300.1318534 MHz
SI                                    32768
SF                          300.1300254 MHz
WDW                                      EM
SSB                                       0
LB                                 0.30 Hz
GB                                        0
PC                                 1.00
```

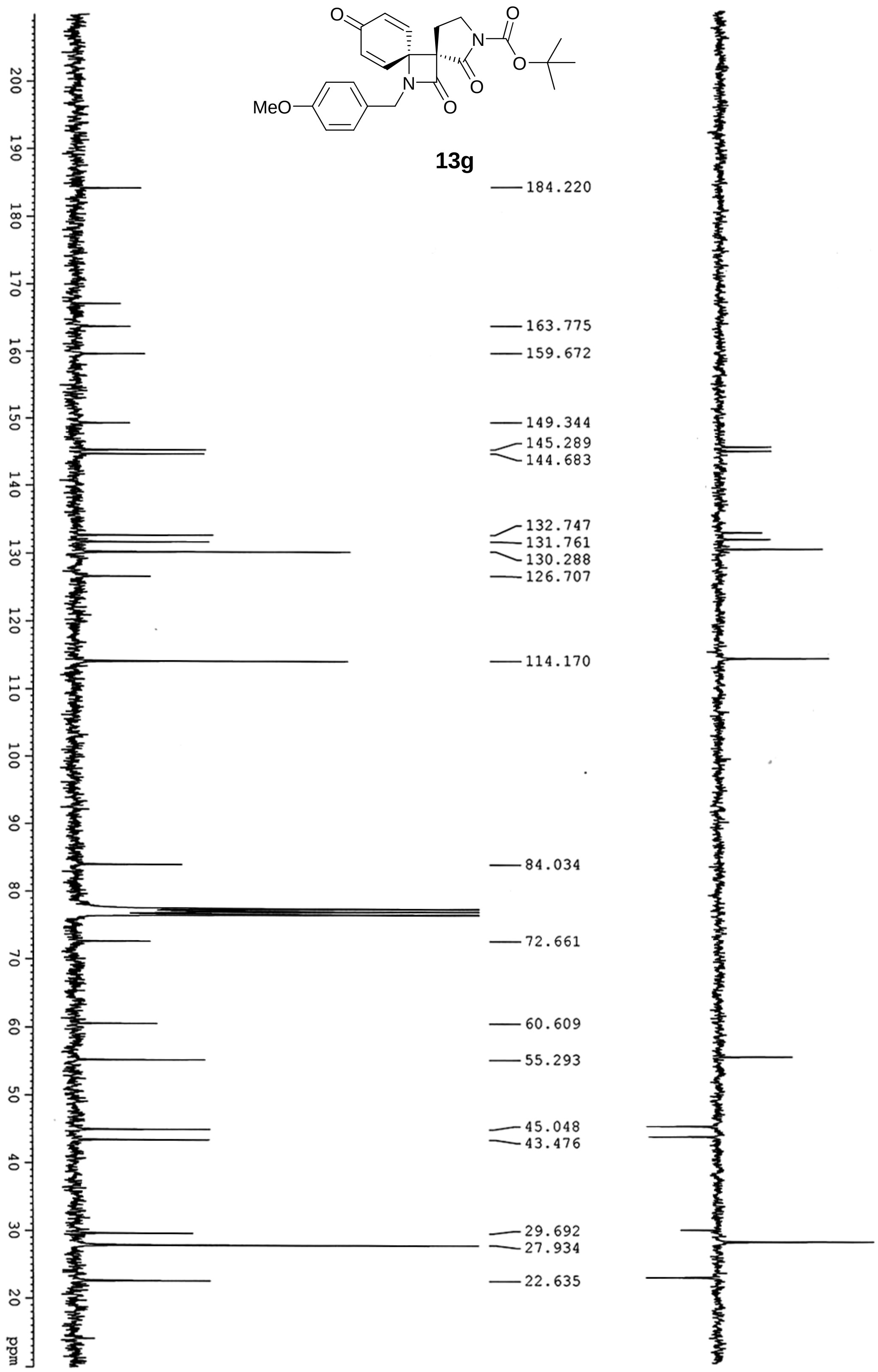

OMe
O
N
O
O
O
13f
183.813
170.806
162.620
159.800
144.605
143.955
132.963
132.095
130.351
126.476
114.243
77.433
77.010
76.587
68.212
65.826
59.907
55.297
45.235
29.681
27.248
ppm

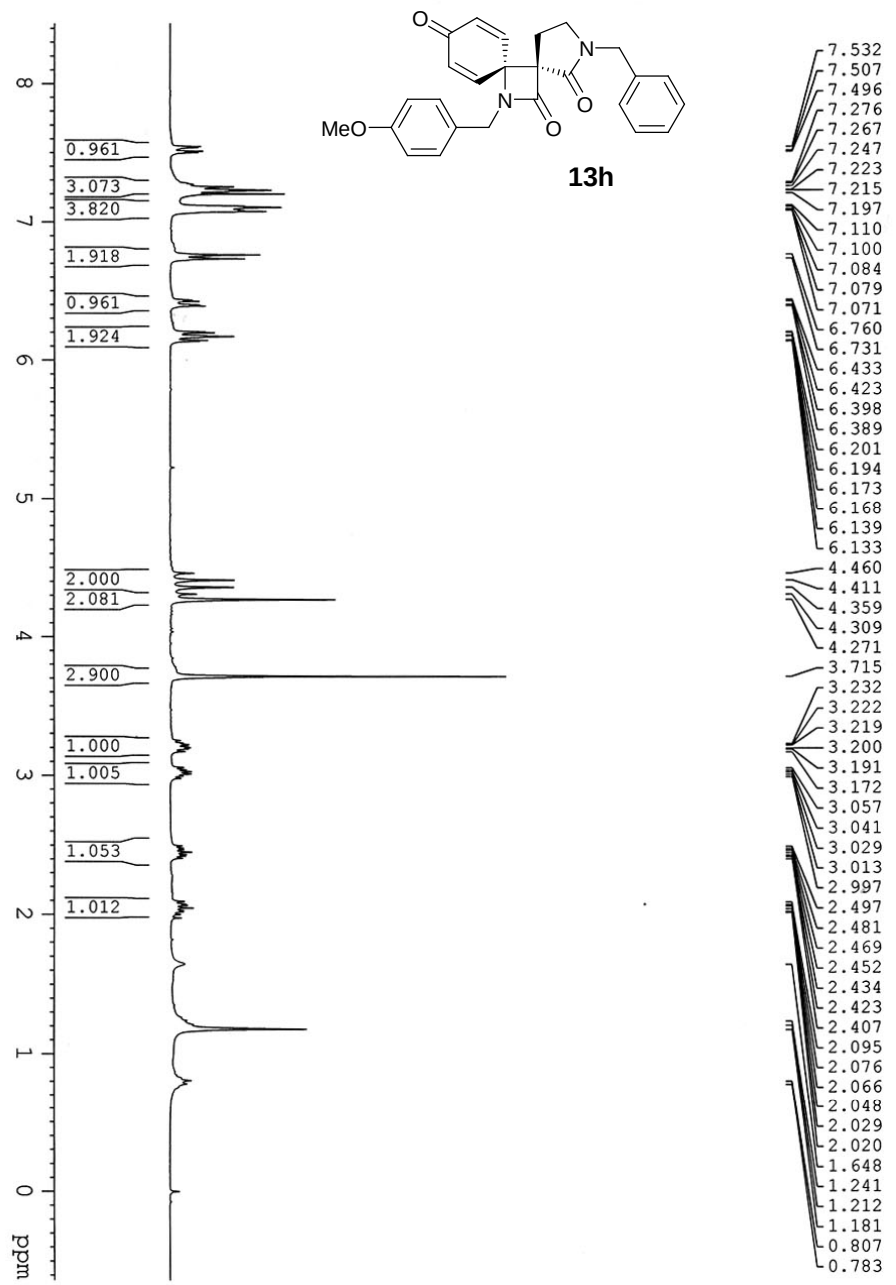


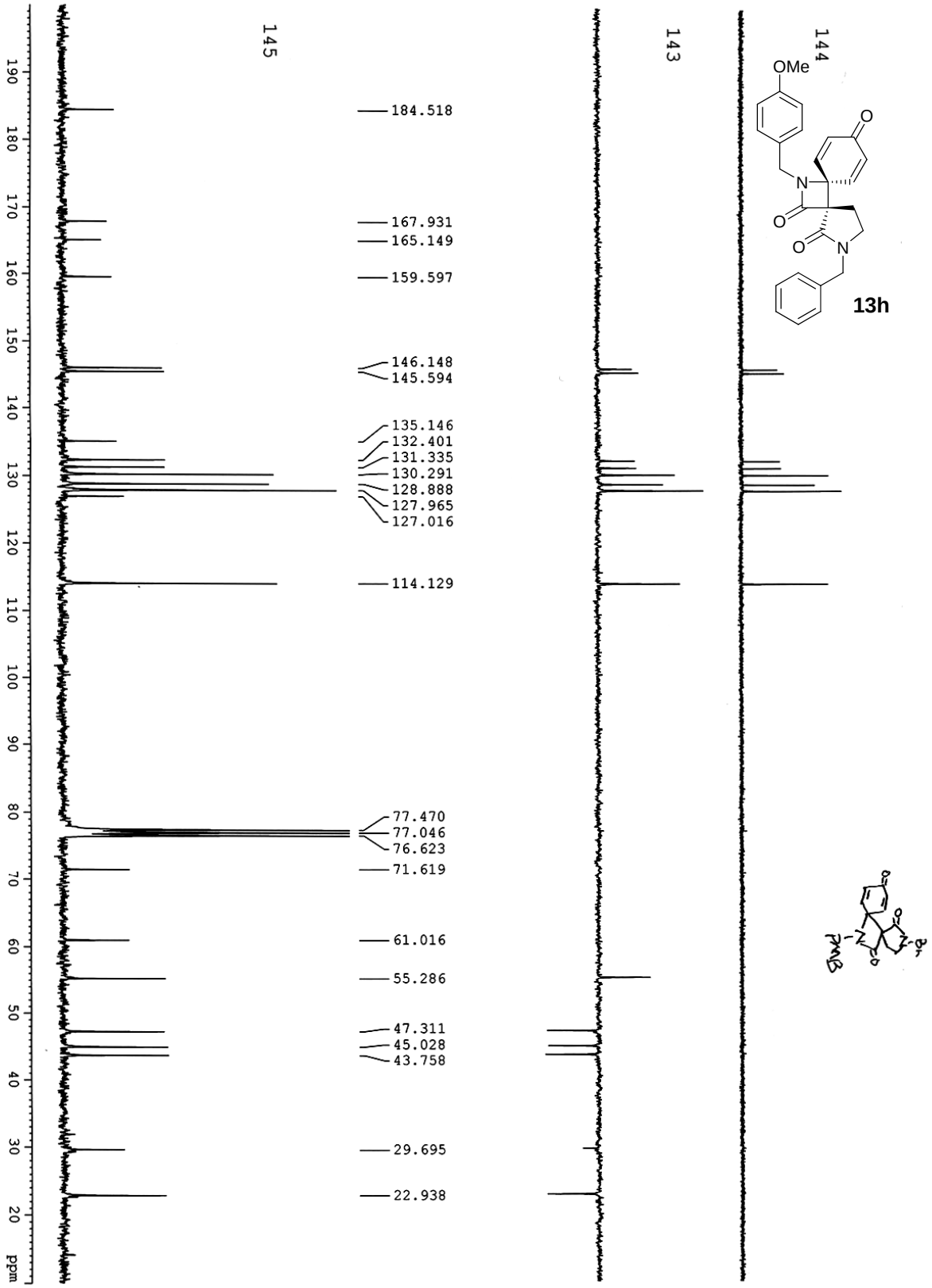

```
NAME                                   hk
EXPNO                                 114
PROCNO                                  1
Date_                            20110113
Time_                                12.18
INSTRUM                           av300
PROBHD     5 mm QNP 1H/13
PULPROG                            zg30
TD                                65536
SOLVENT                           CDCl3
NS                                   16
DS                                    2
SWH                           6172.839 Hz
FIDRES                        0.094190 Hz
AQ                            5.3084660 sec
RG                                322.5
DW                            81.000 usec
DE                             6.50 usec
TE                                291.4 K
D1                            1.00000000 sec
TD0                                   1

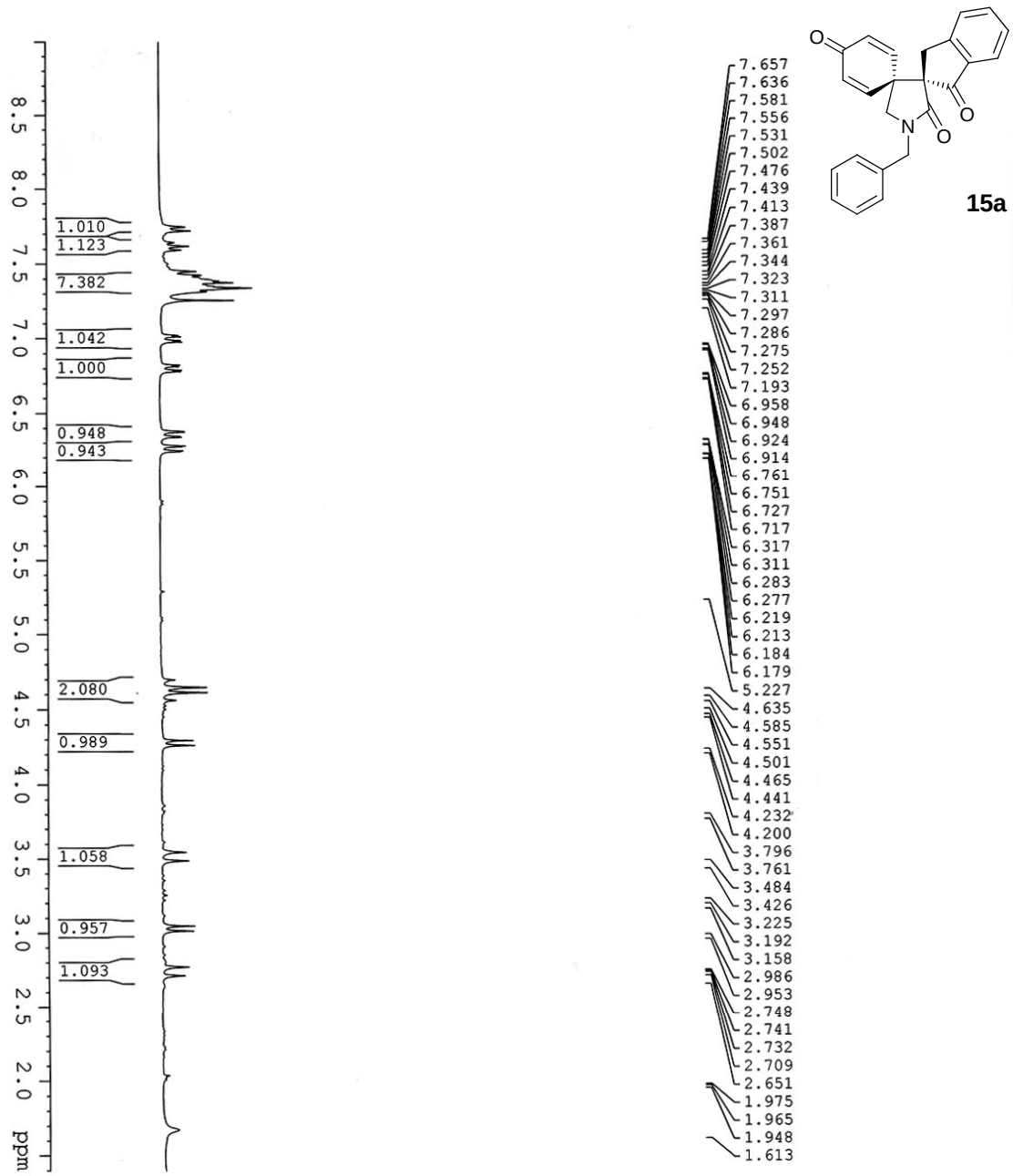
======== CHANNEL f1 ========
NUC1                                1H
P1                             7.90 usec
PL1                             -2.00 dB
SFO1                    300.1318534 MHz
SI                                32768
SF                      300.1300062 MHz
WDW                                  EM
SSB                                   0
LB                             0.30 Hz
GB                                    0
PC                                 1.00
```

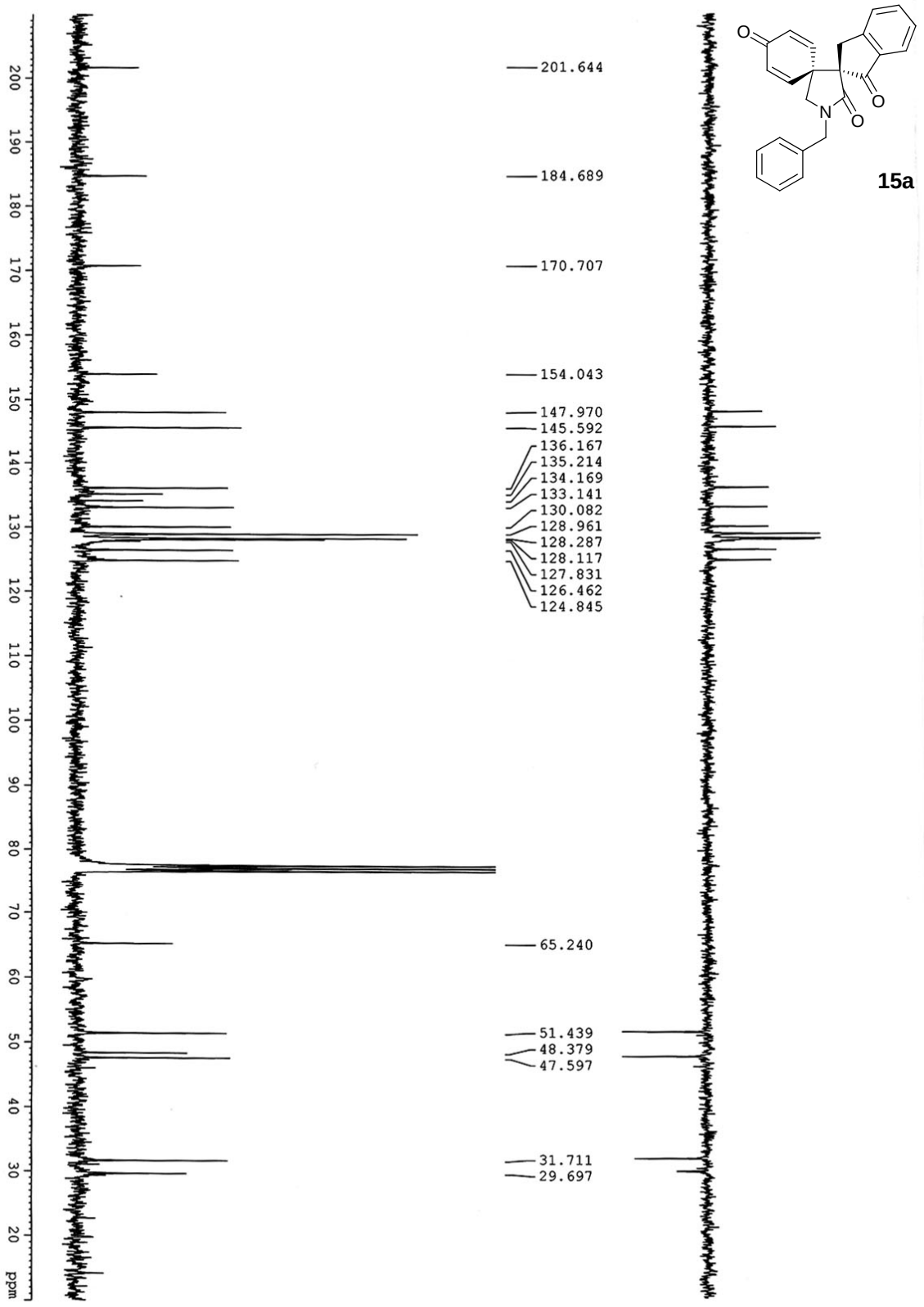


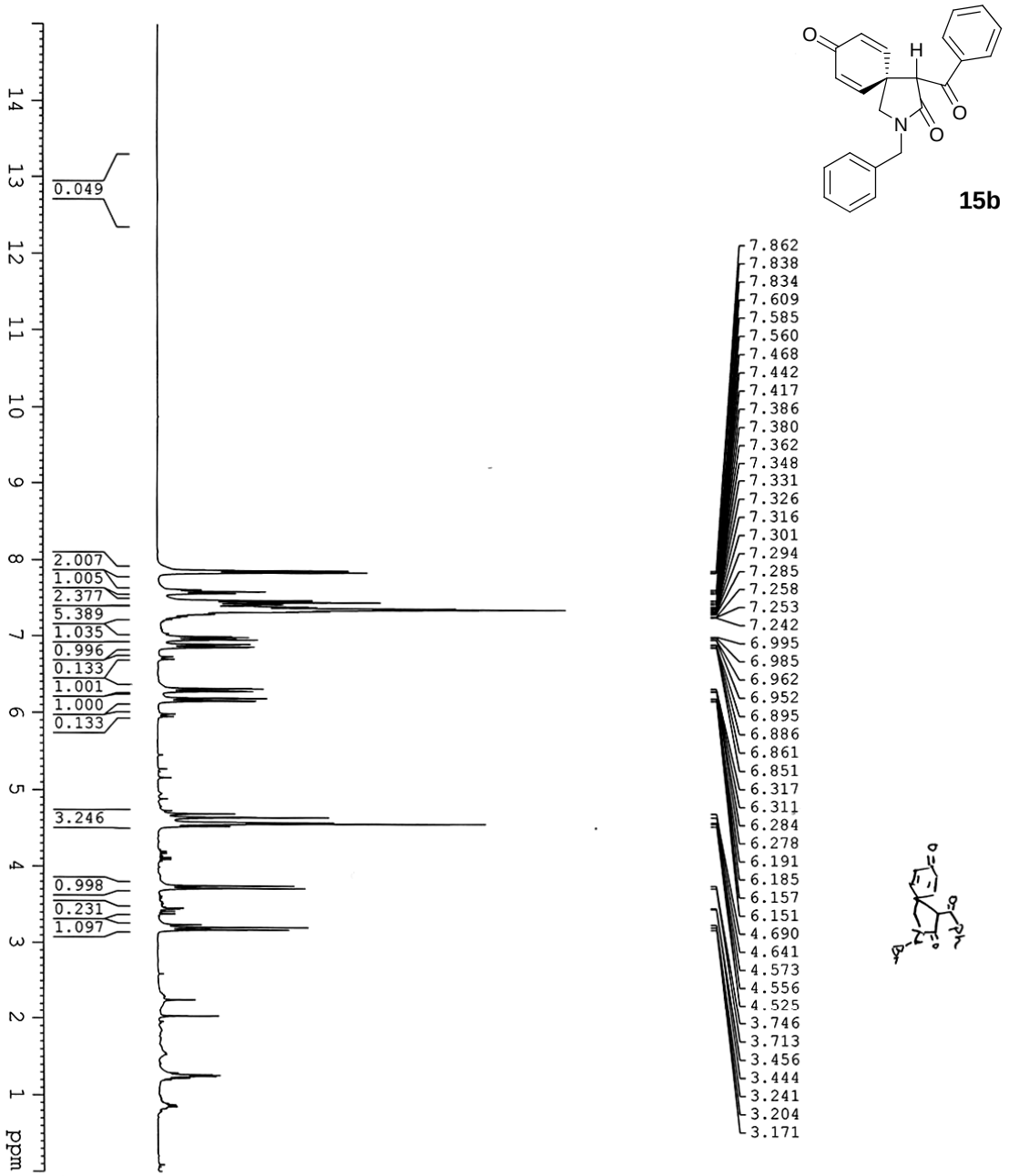

MeO
N
O
O
N
O
O
O
13g
184.220
163.775
159.672
149.344
145.289
144.683
132.747
131.761
130.288
126.707
114.170
84.034
72.661
60.609
55.293
45.048
43.476
29.692
27.934
22.635
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

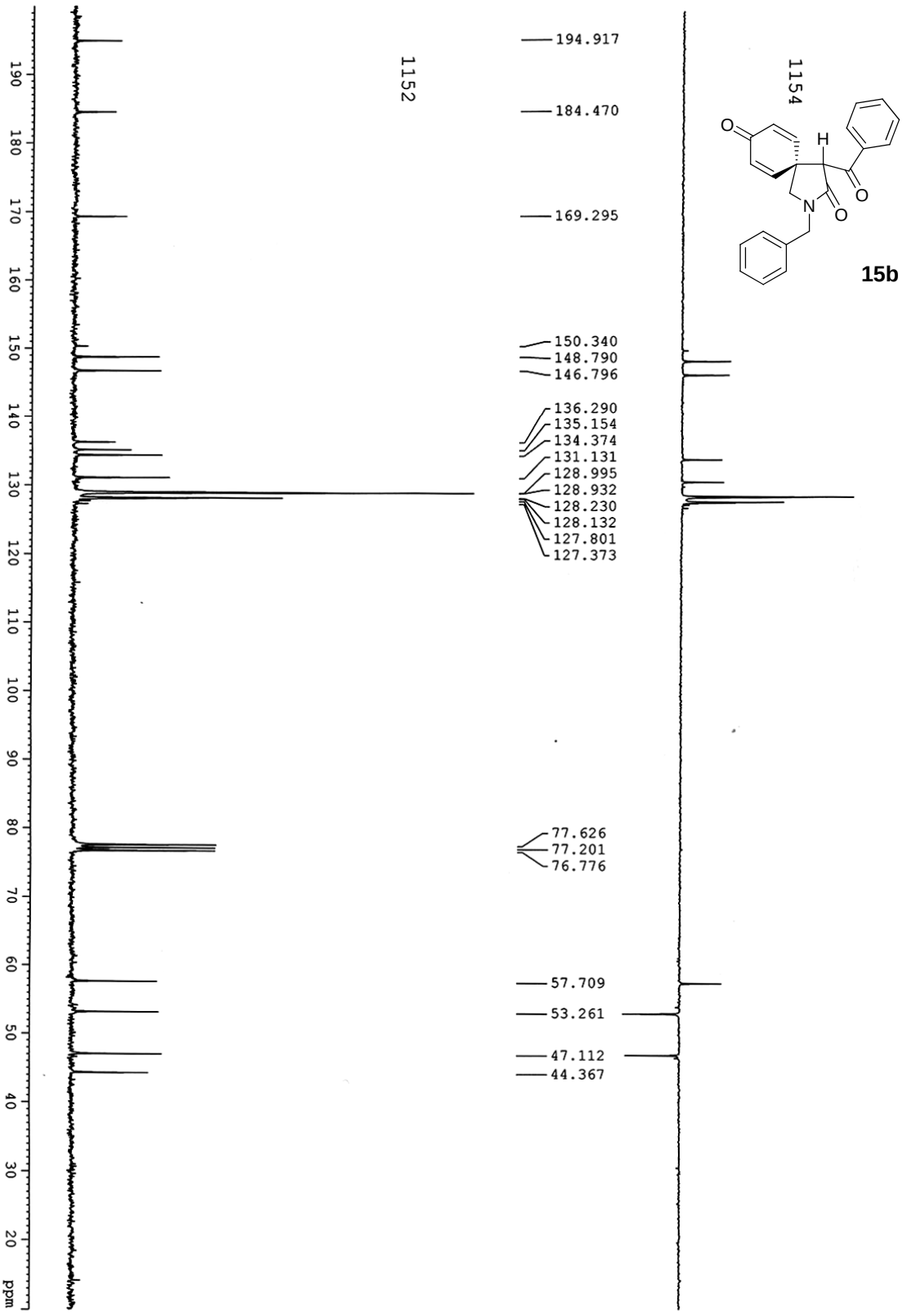

MeO
13h
0.961
3.073
3.820
1.918
0.961
1.924
2.000
2.081
2.900
1.000
1.005
1.053
1.012
ppm
7.532
7.507
7.496
7.276
7.267
7.247
7.223
7.215
7.197
7.110
7.100
7.084
7.079
7.071
6.760
6.731
6.433
6.423
6.398
6.389
6.201
6.194
6.173
6.168
6.139
6.133
4.460
4.411
4.359
4.309
4.271
3.715
3.232
3.222
3.219
3.200
3.191
3.172
3.057
3.041
3.029
3.013
2.997
2.497
2.481
2.469
2.452
2.434
2.423
2.407
2.095
2.076
2.066
2.048
2.029
2.020
1.648
1.241
1.212
1.181
0.807
0.783
NAME hk
EXPNO 142
PROCNO 1
Date_ 20110116
Time_ 10.50
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 228.1
DW 81.000 usec
DE 6.50 usec
TE 290.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.90 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300252 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

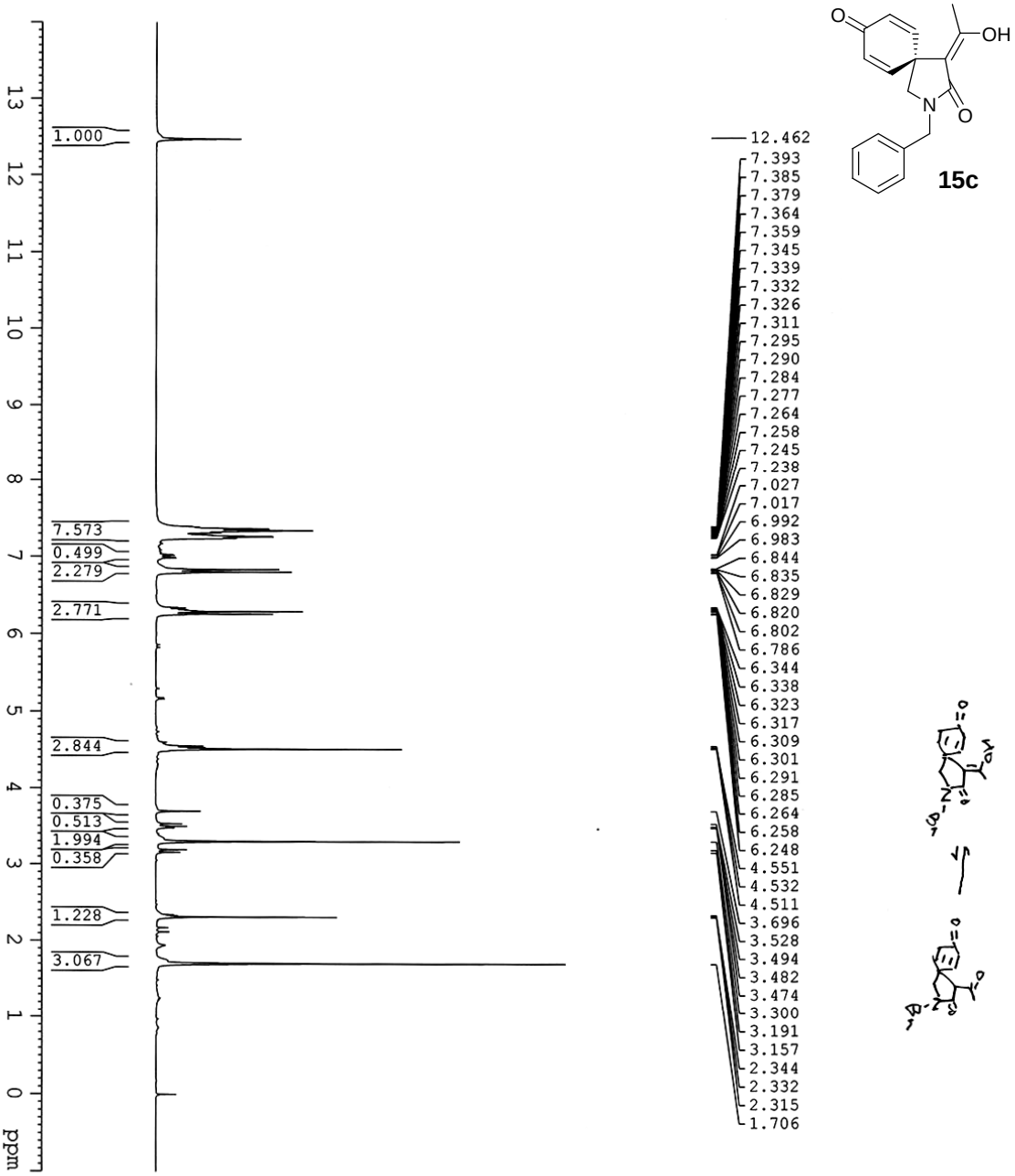

145
143
144
OMe
O
N
O
O
N
13h
184.518
167.931
165.149
159.597
146.148
145.594
135.146
132.401
131.335
130.291
128.888
127.965
127.016
114.129
77.470
77.046
76.623
71.619
61.016
55.286
47.311
45.028
43.758
29.695
22.938
ppm

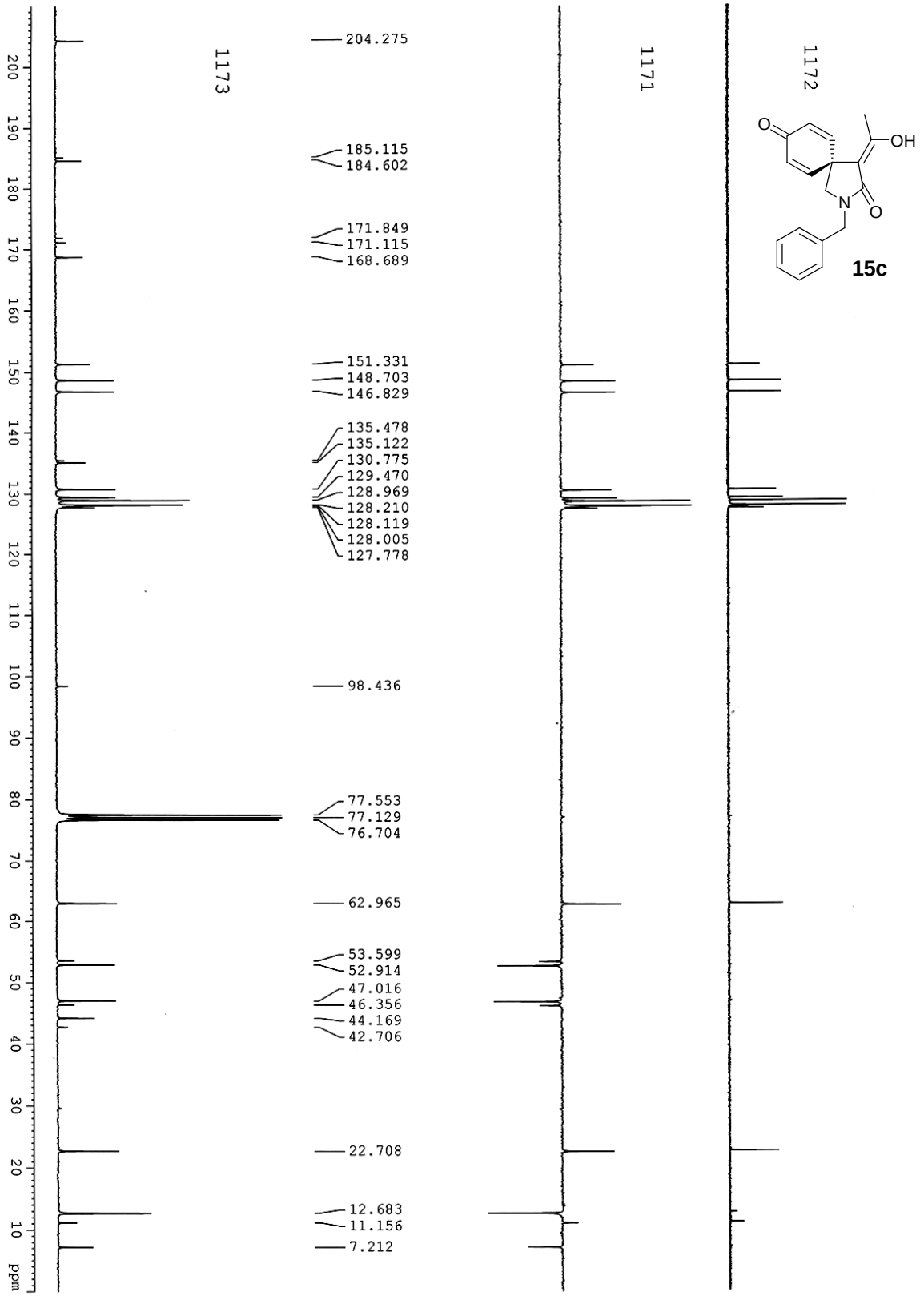

15a
7.657
7.636
7.581
7.556
7.531
7.502
7.476
7.439
7.413
7.387
7.361
7.344
7.323
7.311
7.297
7.286
7.275
7.252
7.193
6.958
6.948
6.924
6.914
6.761
6.751
6.727
6.717
6.317
6.311
6.283
6.277
6.219
6.213
6.184
6.179
5.227
4.635
4.585
4.551
4.501
4.465
4.441
4.232
4.200
3.796
3.761
3.484
3.426
3.225
3.192
3.158
2.986
2.953
2.748
2.741
2.732
2.709
2.651
1.975
1.965
1.948
1.613
1.010
1.123
7.382
1.042
1.000
0.948
0.943
2.080
0.989
1.058
0.957
1.093
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 ppm
NAME hk
EXPNO 112
PROCNO 1
Date_ 20110113
Time_ 9.07
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 256
DW 81.000 usec
DE 6.50 usec
TE 291.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.90 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300075 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

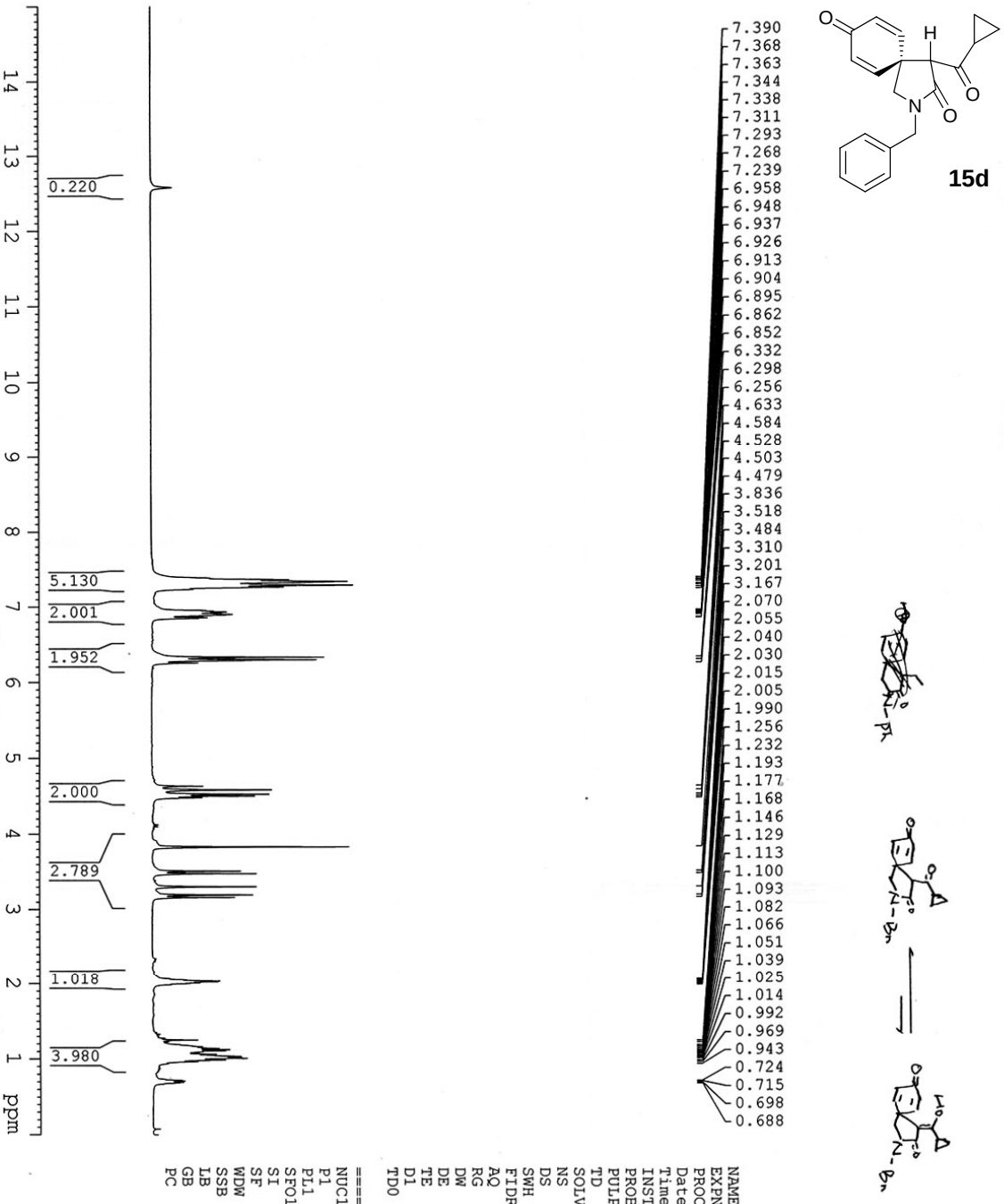

201.644
184.689
170.707
154.043
147.970
145.592
136.167
135.214
134.169
133.141
130.082
128.961
128.287
128.117
127.831
126.462
124.845
65.240
51.439
48.379
47.597
31.711
29.697
ppm
O
O
O
N
15a

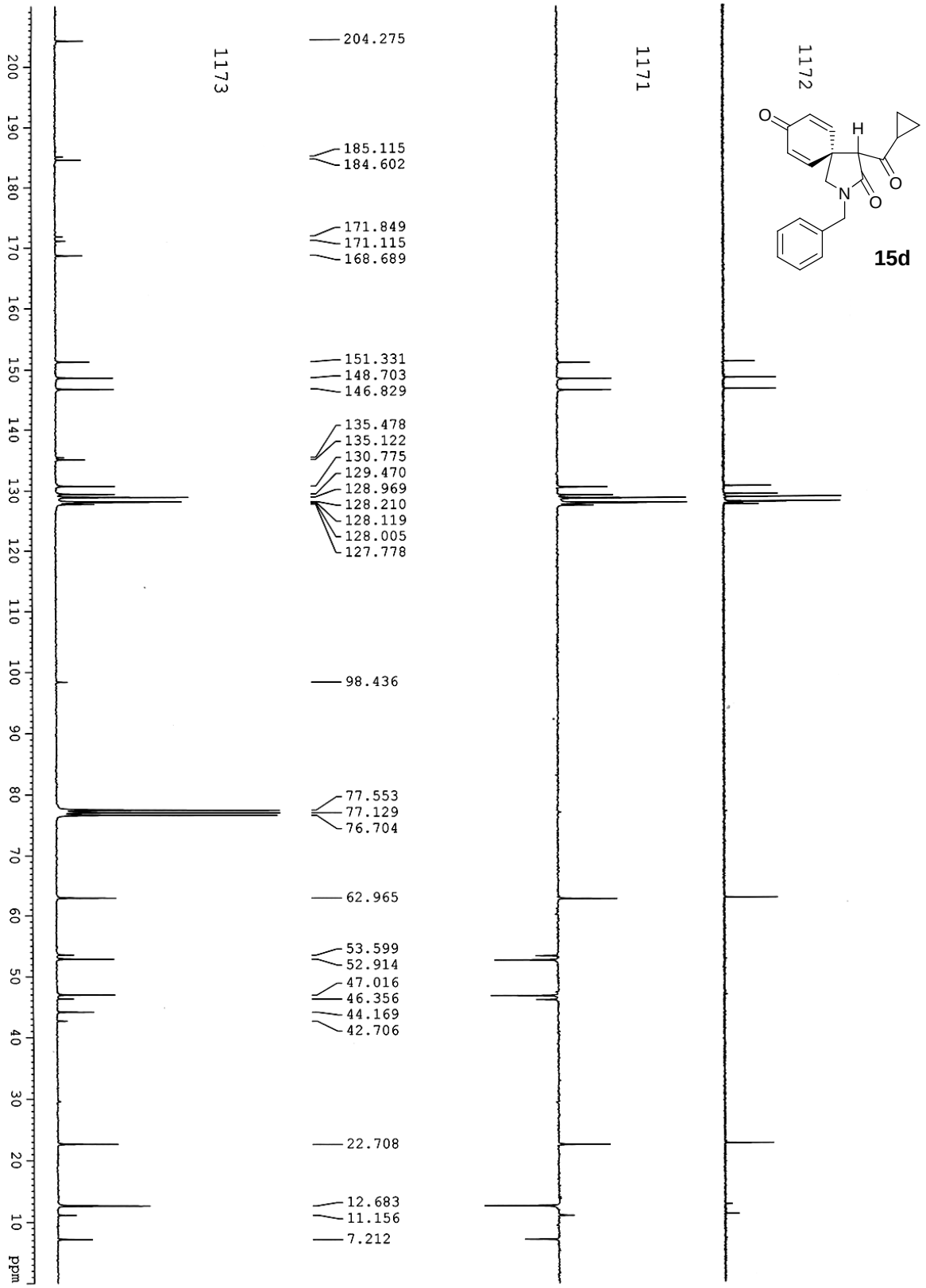

15b
7.862
7.838
7.834
7.609
7.585
7.560
7.468
7.442
7.417
7.386
7.380
7.362
7.348
7.331
7.326
7.316
7.301
7.294
7.285
7.258
7.253
7.242
6.995
6.985
6.962
6.952
6.895
6.886
6.861
6.851
6.317
6.311
6.284
6.278
6.191
6.185
6.157
6.151
4.690
4.641
4.573
4.556
4.525
3.746
3.713
3.456
3.444
3.241
3.204
3.171
0.049
2.007
1.005
2.377
5.389
1.035
0.996
0.133
1.001
1.000
0.133
3.246
0.998
0.231
1.097
ppm
NAME xzx
EXPNO 1153
PROCNO 1
Date_ 20110112
Time_ 19.44
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 71.8
DW 81.000 usec
DE 6.50 usec
TE 291.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.90 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1299986 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

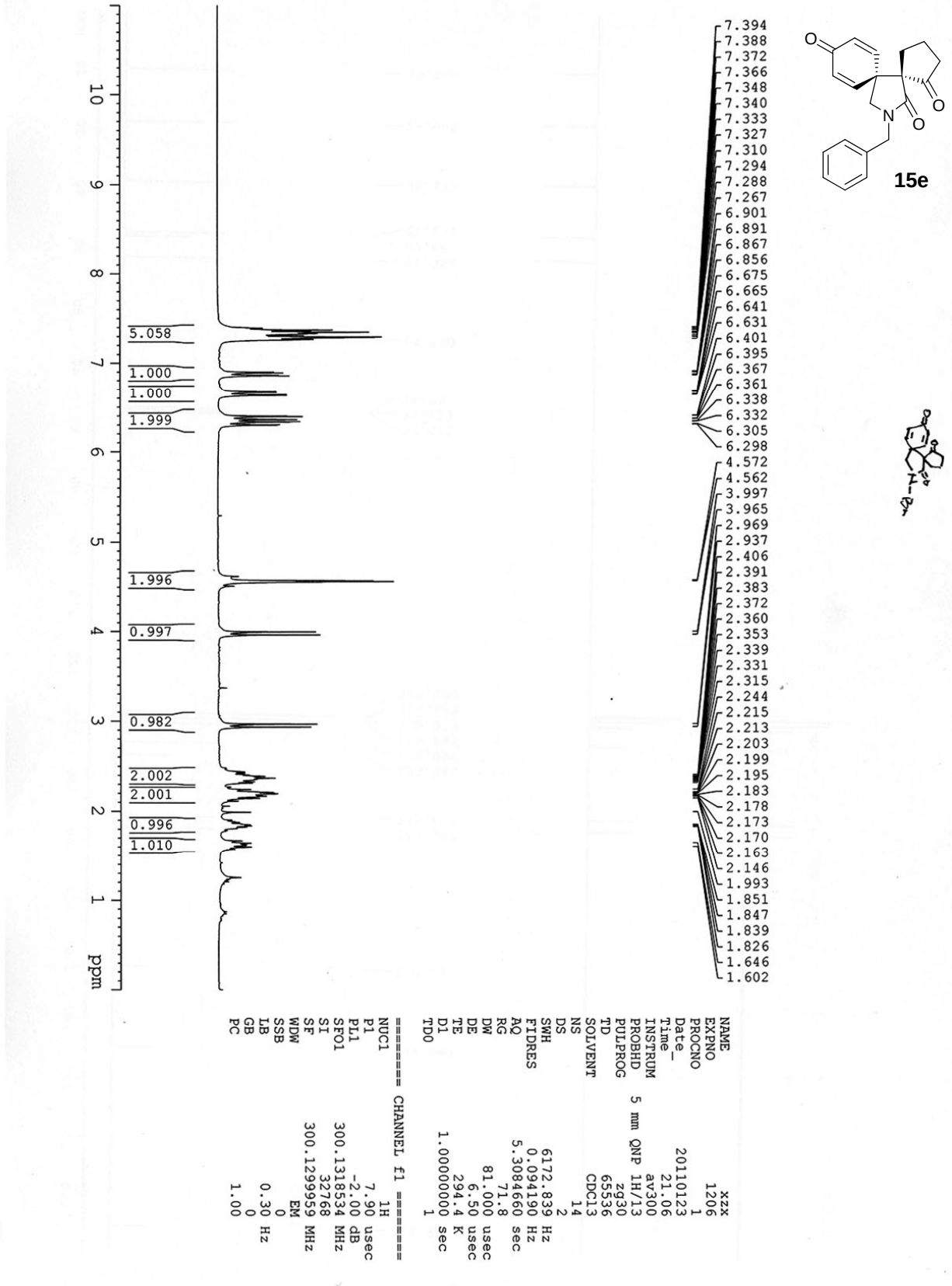

1152
1154
194.917
184.470
169.295
150.340
148.790
146.796
136.290
135.154
134.374
131.131
128.995
128.932
128.230
128.132
127.801
127.373
77.626
77.201
76.776
57.709
53.261
47.112
44.367
ppm
O
H
O
N
O
15b

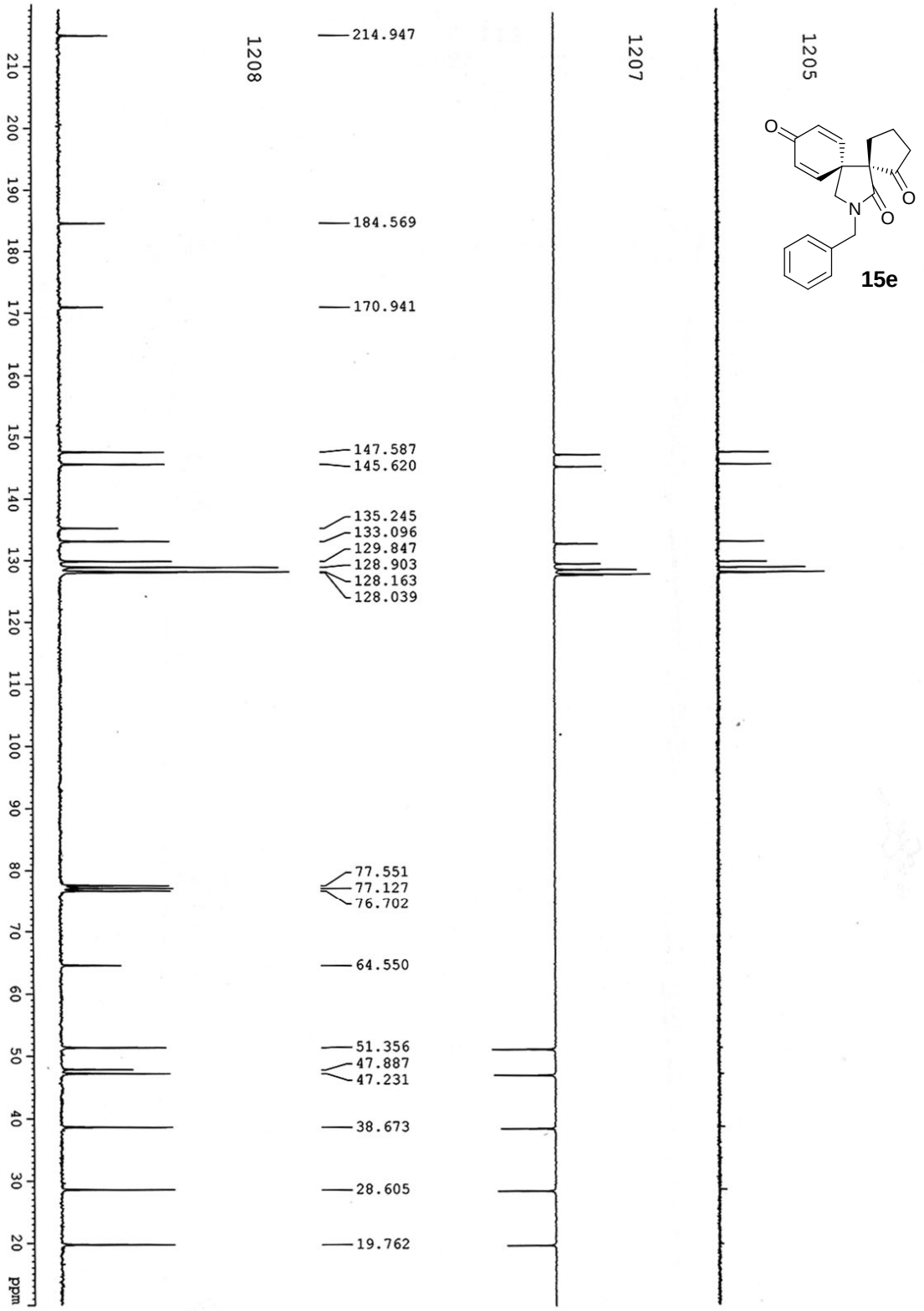

15c
12.462
7.393
7.385
7.379
7.364
7.359
7.345
7.339
7.332
7.326
7.311
7.295
7.290
7.284
7.277
7.264
7.258
7.245
7.238
7.027
7.017
6.992
6.983
6.844
6.835
6.829
6.820
6.802
6.786
6.344
6.338
6.323
6.317
6.309
6.301
6.291
6.285
6.264
6.258
6.248
4.551
4.532
4.511
3.696
3.528
3.494
3.482
3.474
3.300
3.191
3.157
2.344
2.332
2.315
1.706
1.000
7.573
0.499
2.279
2.771
2.844
0.375
0.513
1.994
0.358
1.228
3.067
13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
NAME xzx
EXPNO 1211
PROCNO 1
Date_ 20110123
Time_ 22.02
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 9
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 90.5
DW 81.000 usec
DE 6.50 usec
TE 294.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.90 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1299988 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

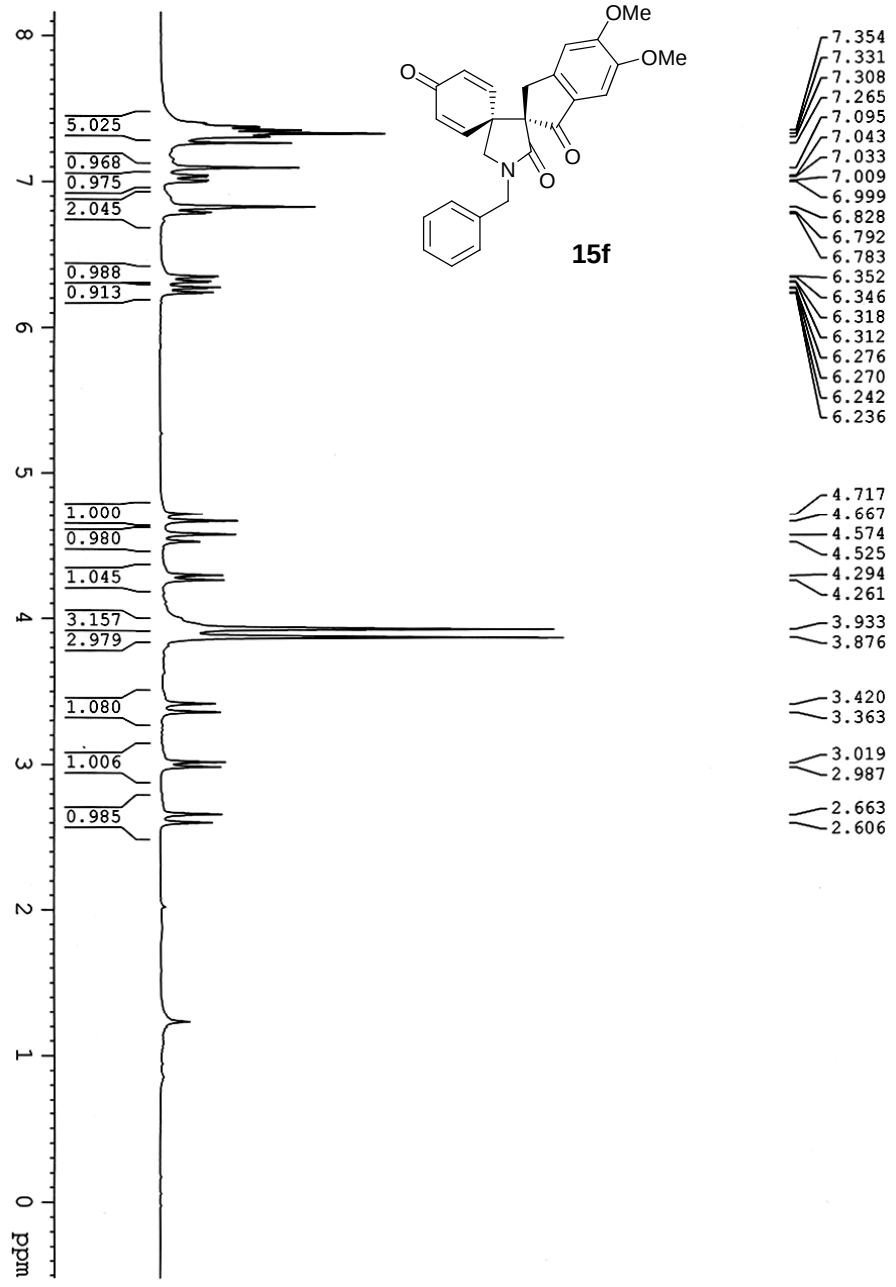

1173
1171
1172
204.275
185.115
184.602
171.849
171.115
168.689
151.331
148.703
146.829
135.478
135.122
130.775
129.470
128.969
128.210
128.119
128.005
127.778
98.436
77.553
77.129
76.704
62.965
53.599
52.914
47.016
46.356
44.169
42.706
22.708
12.683
11.156
7.212
ppm
O
OH
N
O
15c

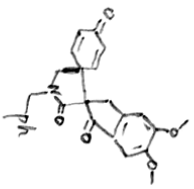

15d
7.390
7.368
7.363
7.344
7.338
7.311
7.293
7.268
7.239
6.958
6.948
6.937
6.926
6.913
6.904
6.895
6.862
6.852
6.332
6.298
6.256
4.633
4.584
4.528
4.503
4.479
3.836
3.518
3.484
3.310
3.201
3.167
2.070
2.055
2.040
2.030
2.015
2.005
1.990
1.256
1.232
1.193
1.177
1.168
1.146
1.129
1.113
1.100
1.093
1.082
1.066
1.051
1.039
1.025
1.014
0.992
0.969
0.943
0.724
0.715
0.698
0.688
0.220
5.130
2.001
1.952
2.000
2.789
1.018
3.980
ppm
NAME xzx
EXPNO 1170
PROCNO 1
Date_ 20110114
Time_ 17.56
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 90.5
DW 81.000 usec
DE 6.50 usec
TE 293.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.90 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1299962 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


1173
1171
1172
204.275
185.115
184.602
171.849
171.115
168.689
151.331
148.703
146.829
135.478
135.122
130.775
129.470
128.969
128.210
128.119
128.005
127.778
98.436
77.553
77.129
76.704
62.965
53.599
52.914
47.016
46.356
44.169
42.706
22.708
12.683
11.156
7.212
ppm
O
H
O
N
O
15d


15e
7.394
7.388
7.372
7.366
7.348
7.340
7.333
7.327
7.310
7.294
7.288
7.267
6.901
6.891
6.867
6.856
6.675
6.665
6.641
6.631
6.401
6.395
6.367
6.361
6.338
6.332
6.305
6.298
4.572
4.562
3.997
3.965
2.969
2.937
2.406
2.391
2.383
2.372
2.360
2.353
2.339
2.331
2.315
2.244
2.215
2.213
2.203
2.199
2.195
2.183
2.178
2.173
2.170
2.163
2.146
1.993
1.851
1.847
1.839
1.826
1.646
1.602
5.058
1.000
1.000
1.999
1.996
0.997
0.982
2.002
2.001
0.996
1.010
10
9
8
7
6
5
4
3
2
1
ppm
NAME xzx
EXPNO 1206
PROCNO 1
Date_ 20110123
Time_ 21.06
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 14
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 71.8
DW 81.000 usec
DE 6.50 usec
TE 294.4 K
D1 1.00000000 sec
TD0 1
========== CHANNEL f1 ========
NUC1 1H
P1 7.90 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1299959 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

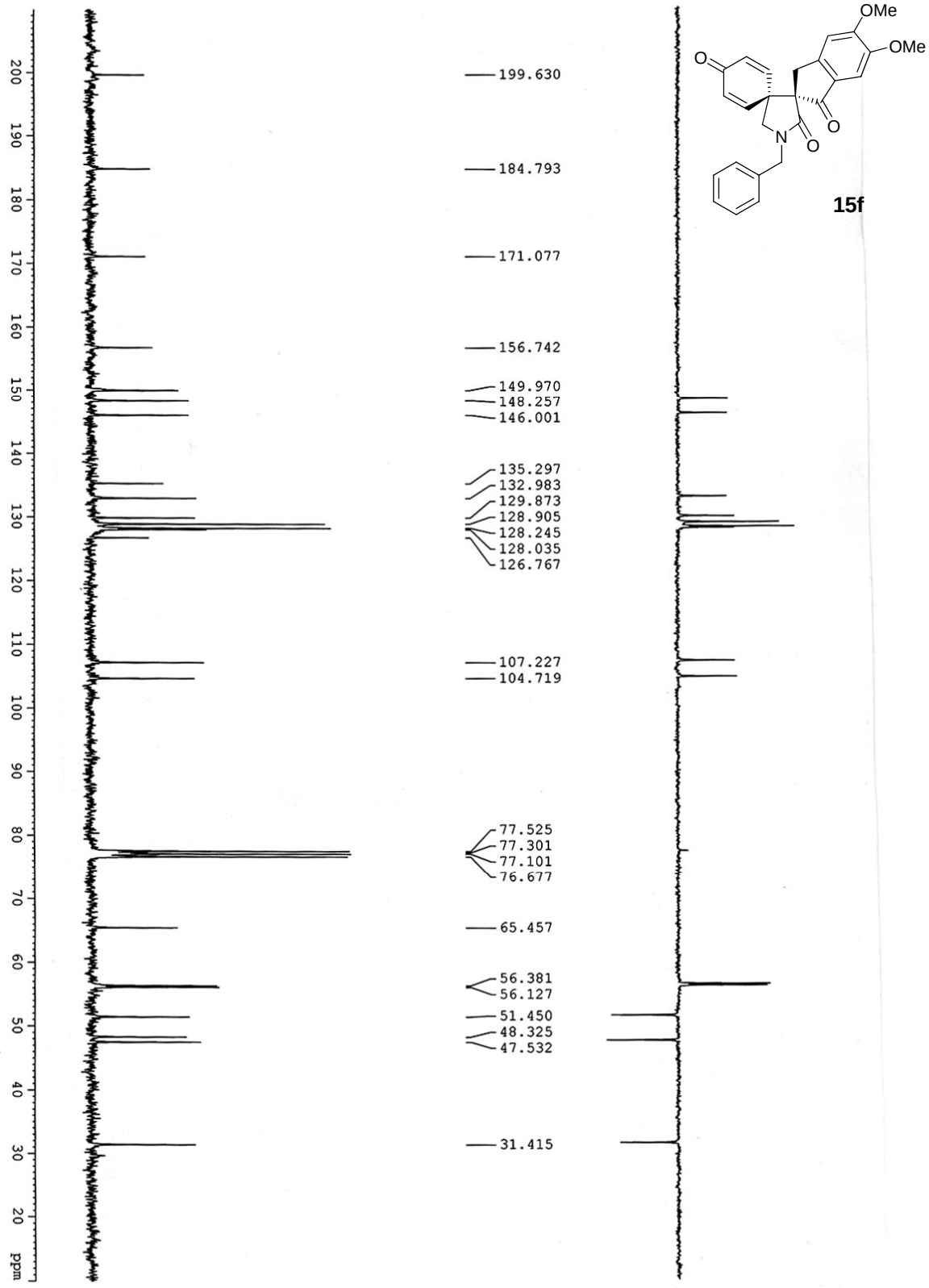

1208
1207
1205
214.947
184.569
170.941
147.587
145.620
135.245
133.096
129.847
128.903
128.163
128.039
77.551
77.127
76.702
64.550
51.356
47.887
47.231
38.673
28.605
19.762
ppm
O
O
O
N
O
15e

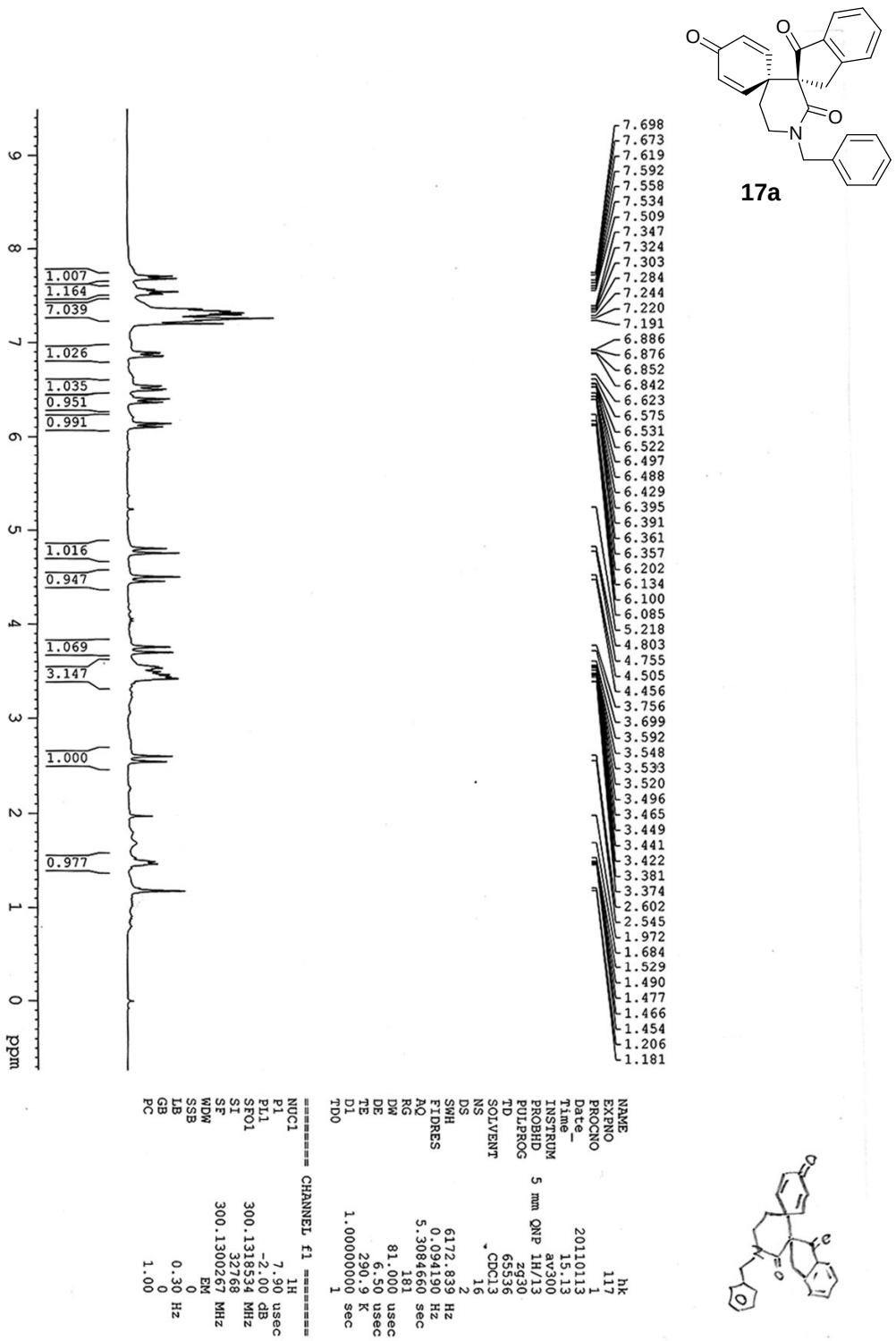


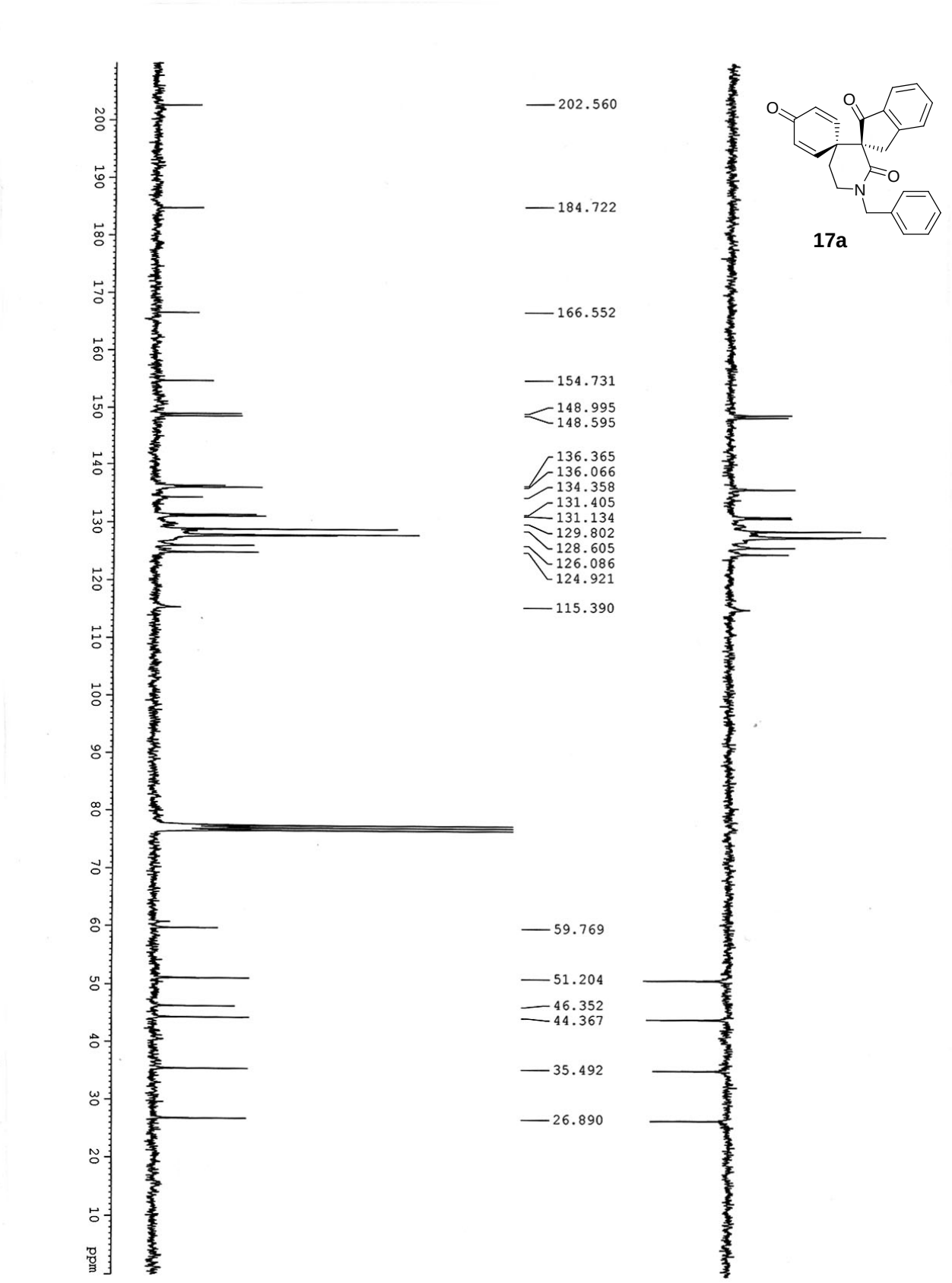


```
NAME                                    hk
EXPNO                                   99
PROCNO                                  1
Date_                       20110111
Time_                       10.00
INSTRUM                     av300
PROBHD          5 mm QNP 1H/13
PULPROG                     zg30
TD                          65536
SOLVENT                     CDCl3
NS                          16
DS                          2
SWH                         6172.839 Hz
FIDRES                      0.094190 Hz
AQ                          5.3084660 sec
RG                          114
DW                          81.000 usec
DE                          6.50 usec
TE                          293.3 K
D1                          1.00000000 sec
TD0                         1

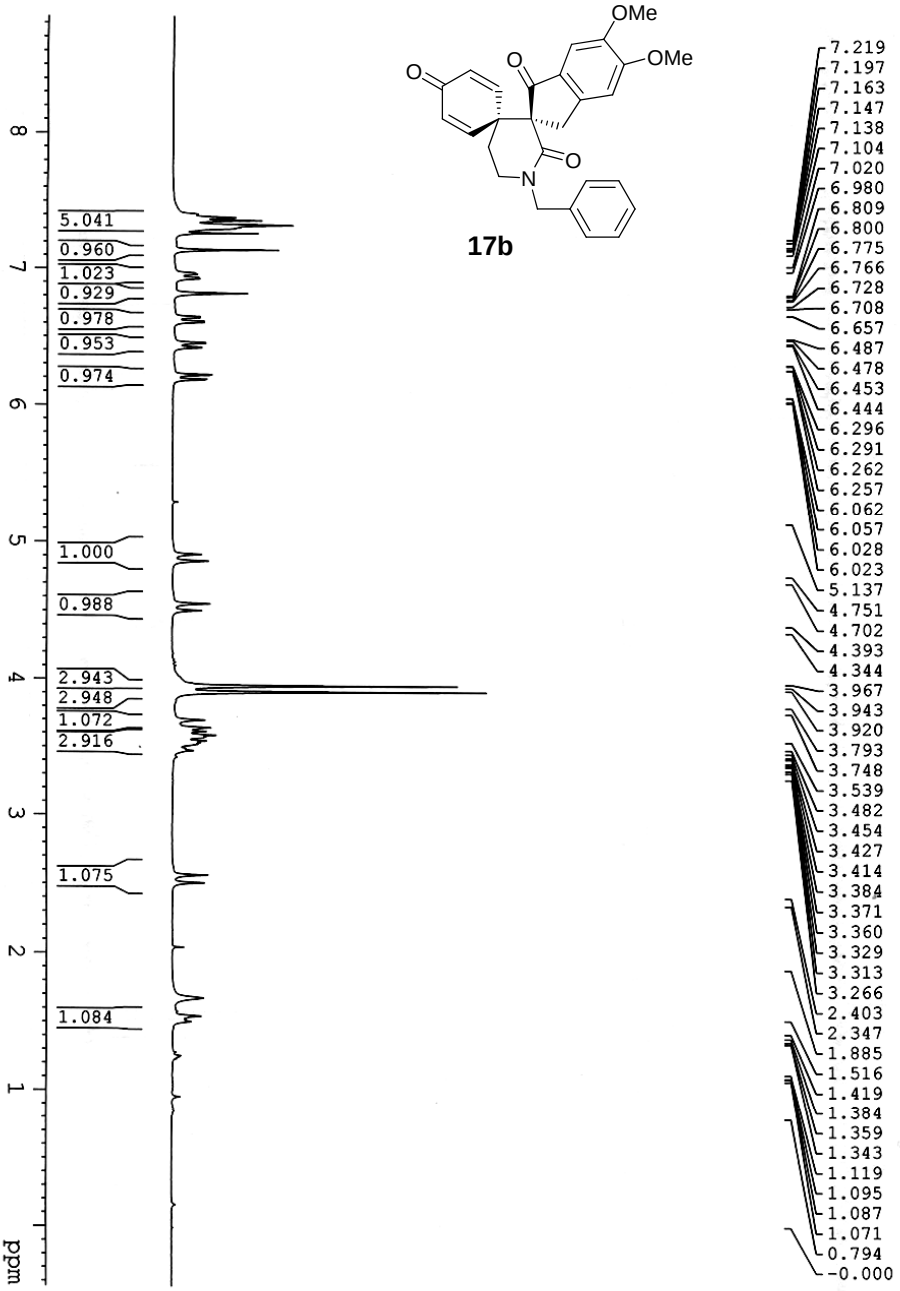
======== CHANNEL f1 ========
NUC1                        1H
P1                          7.90 usec
PL1                         -2.00 dB
SFO1                        300.1318534 MHz
SI                          32768
SF                          300.1300043 MHz
WDW                         EM
SSB                         0
LB                          0.30 Hz
GB                          0
PC                          1.00
```

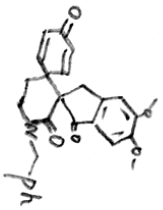

199.630
184.793
171.077
156.742
149.970
148.257
146.001
135.297
132.983
129.873
128.905
128.245
128.035
126.767
107.227
104.719
77.525
77.301
77.101
76.677
65.457
56.381
56.127
51.450
48.325
47.532
31.415
ppm
OMe
OMe
O
O
O
N
O
15f

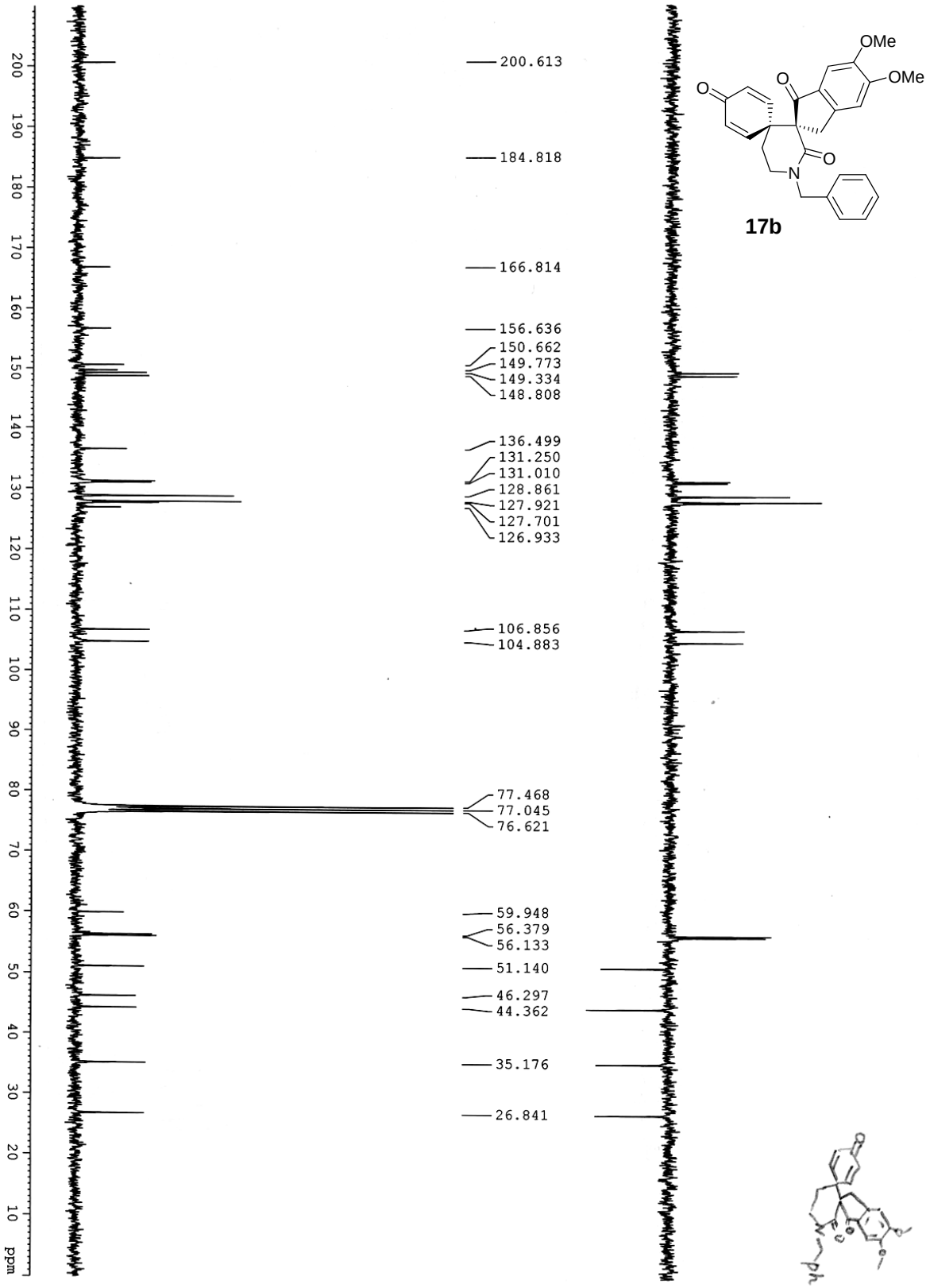

17a
7.698
7.673
7.619
7.592
7.558
7.534
7.509
7.347
7.324
7.303
7.284
7.244
7.220
7.191
6.886
6.876
6.852
6.842
6.623
6.575
6.531
6.522
6.497
6.488
6.429
6.395
6.391
6.361
6.357
6.202
6.134
6.100
6.085
5.218
4.803
4.755
4.505
4.456
3.756
3.699
3.592
3.548
3.533
3.520
3.496
3.465
3.449
3.441
3.422
3.381
3.374
2.602
2.545
1.972
1.684
1.529
1.490
1.477
1.466
1.454
1.206
1.181
1.007
1.164
7.039
1.026
1.035
0.951
0.991
1.016
0.947
1.069
3.147
1.000
0.977
ppm
NAME hk
EXPNO 117
PROCNO 1
Date_ 20110113
Time_ 15.13
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 181
DW 81.000 usec
DE 6.50 usec
TE 290.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.90 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300267 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


202.560
184.722
166.552
154.731
148.995
148.595
136.365
136.066
134.358
131.405
131.134
129.802
128.605
126.086
124.921
115.390
59.769
51.204
46.352
44.367
35.492
26.890
ppm
O
O
O
N

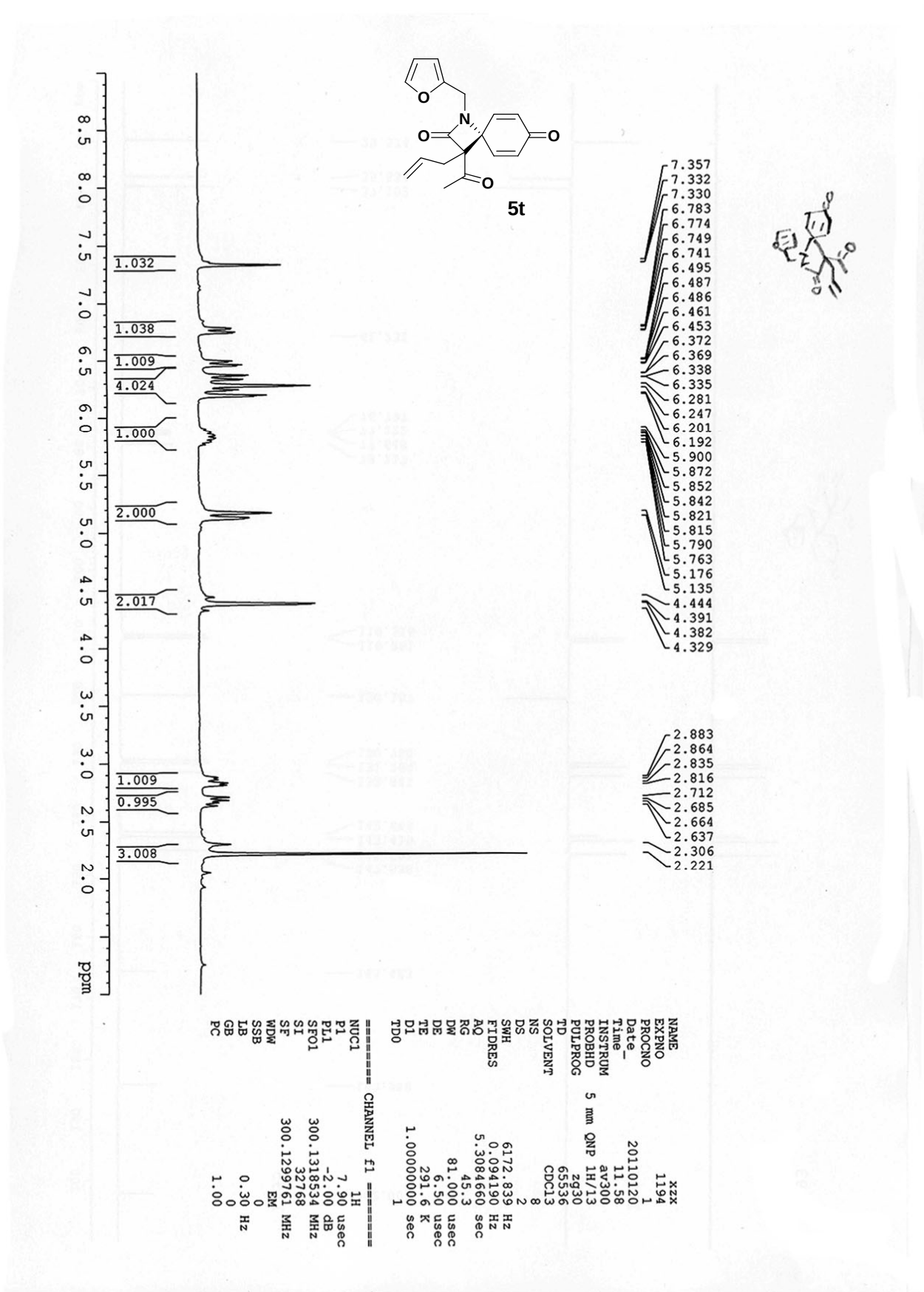
17a

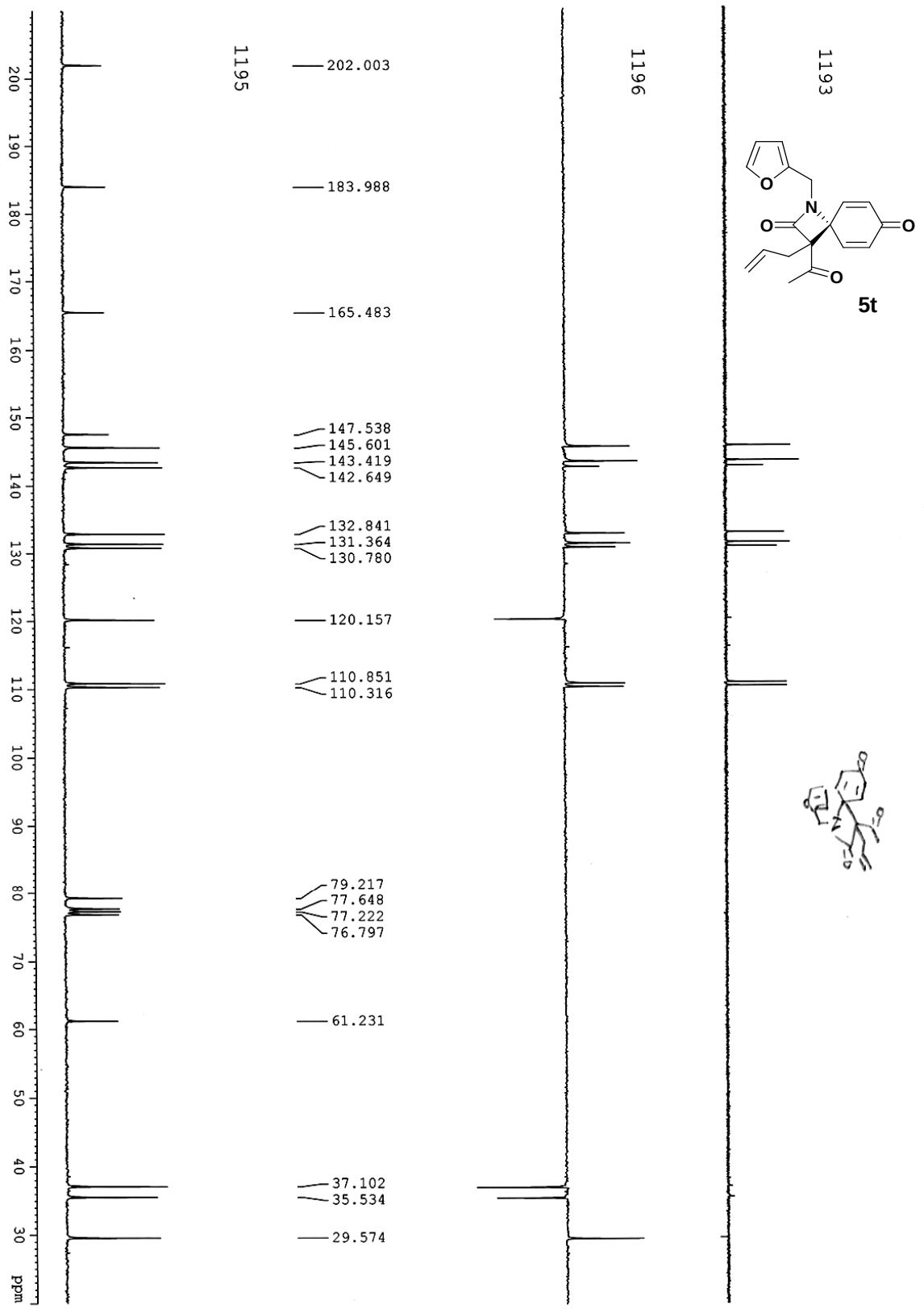

OMe
OMe
O
O
O
N
17b
7.219
7.197
7.163
7.147
7.138
7.104
7.020
6.980
6.809
6.800
6.775
6.766
6.728
6.708
6.657
6.487
6.478
6.453
6.444
6.296
6.291
6.262
6.257
6.062
6.057
6.028
6.023
5.137
4.751
4.702
4.393
4.344
3.967
3.943
3.920
3.793
3.748
3.539
3.482
3.454
3.427
3.414
3.384
3.371
3.360
3.329
3.313
3.266
2.403
2.347
1.885
1.516
1.419
1.384
1.359
1.343
1.119
1.095
1.087
1.071
0.794
-0.000
5.041
0.960
1.023
0.929
0.978
0.953
0.974
1.000
0.988
2.943
2.948
1.072
2.916
1.075
1.084
ppm
NAME hk
EXPNO 154
PROCNO 1
Date_ 20110118
Time_ 12.53
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 322.5
DW 81.000 usec
DE 6.50 usec
TE 288.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.90 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300057 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

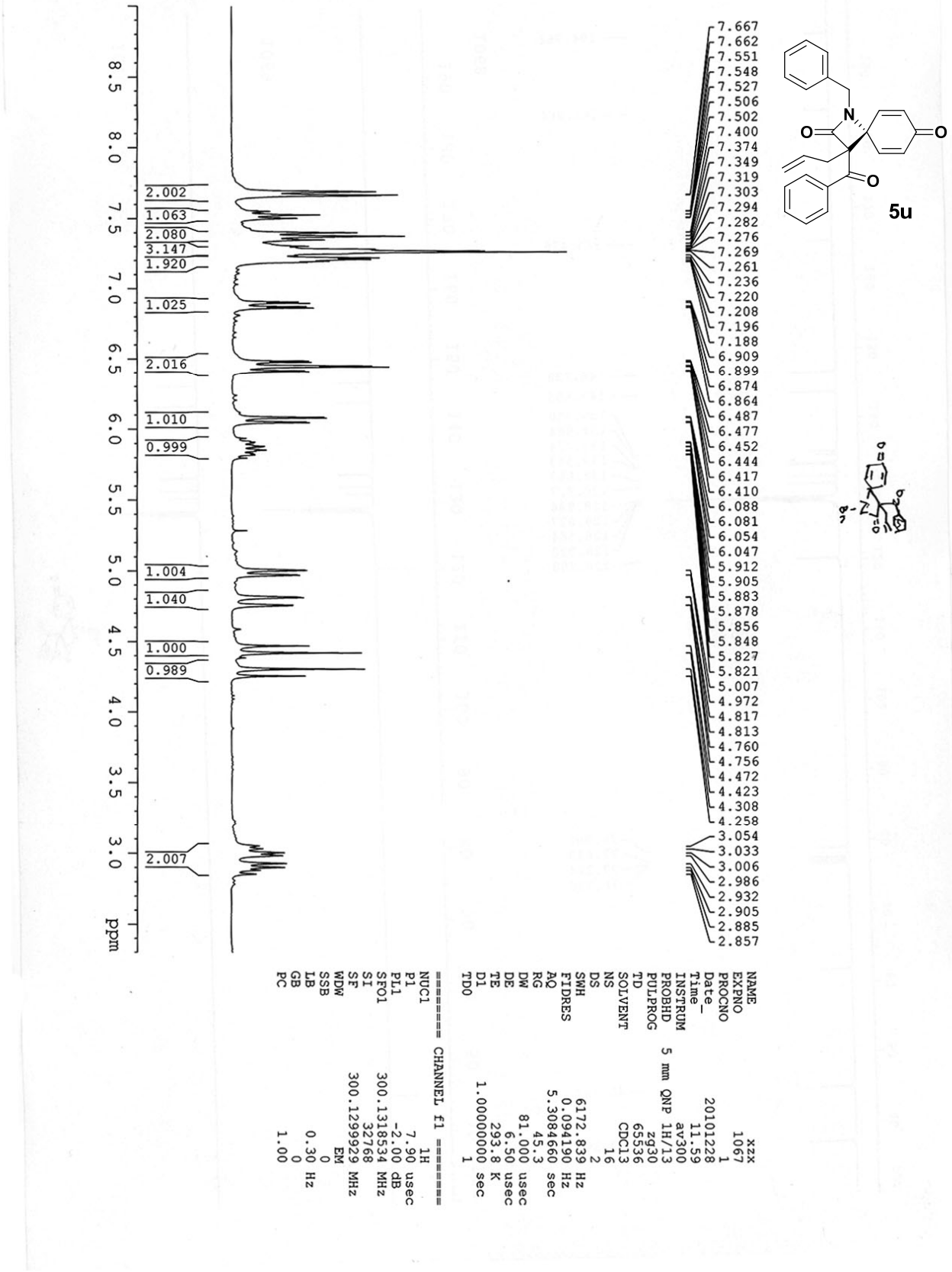


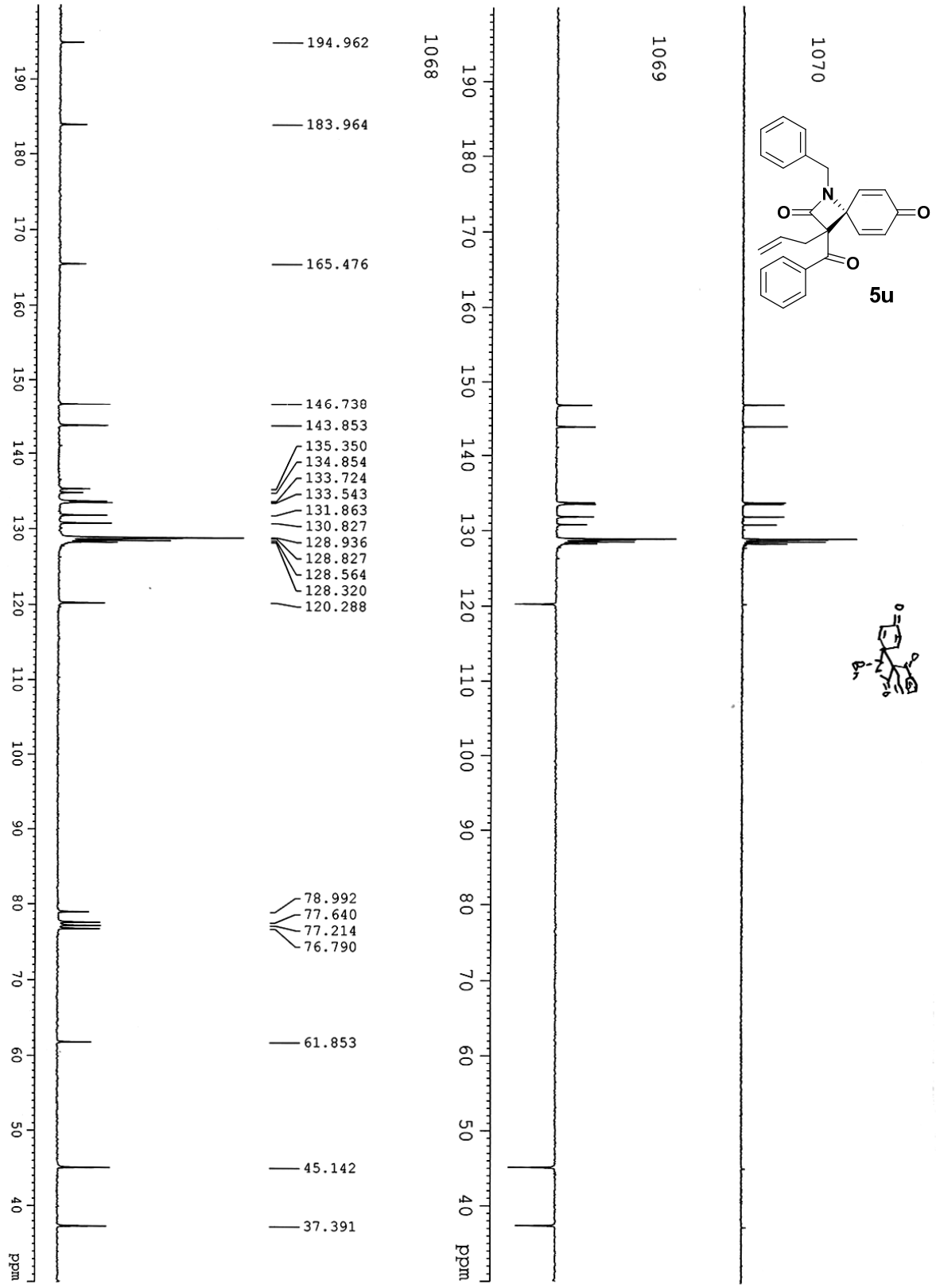

200.613
184.818
166.814
156.636
150.662
149.773
149.334
148.808
136.499
131.250
131.010
128.861
127.921
127.701
126.933
106.856
104.883
77.468
77.045
76.621
59.948
56.379
56.133
51.140
46.297
44.362
35.176
26.841
ppm
OMe
OMe
O
O
O
O
N

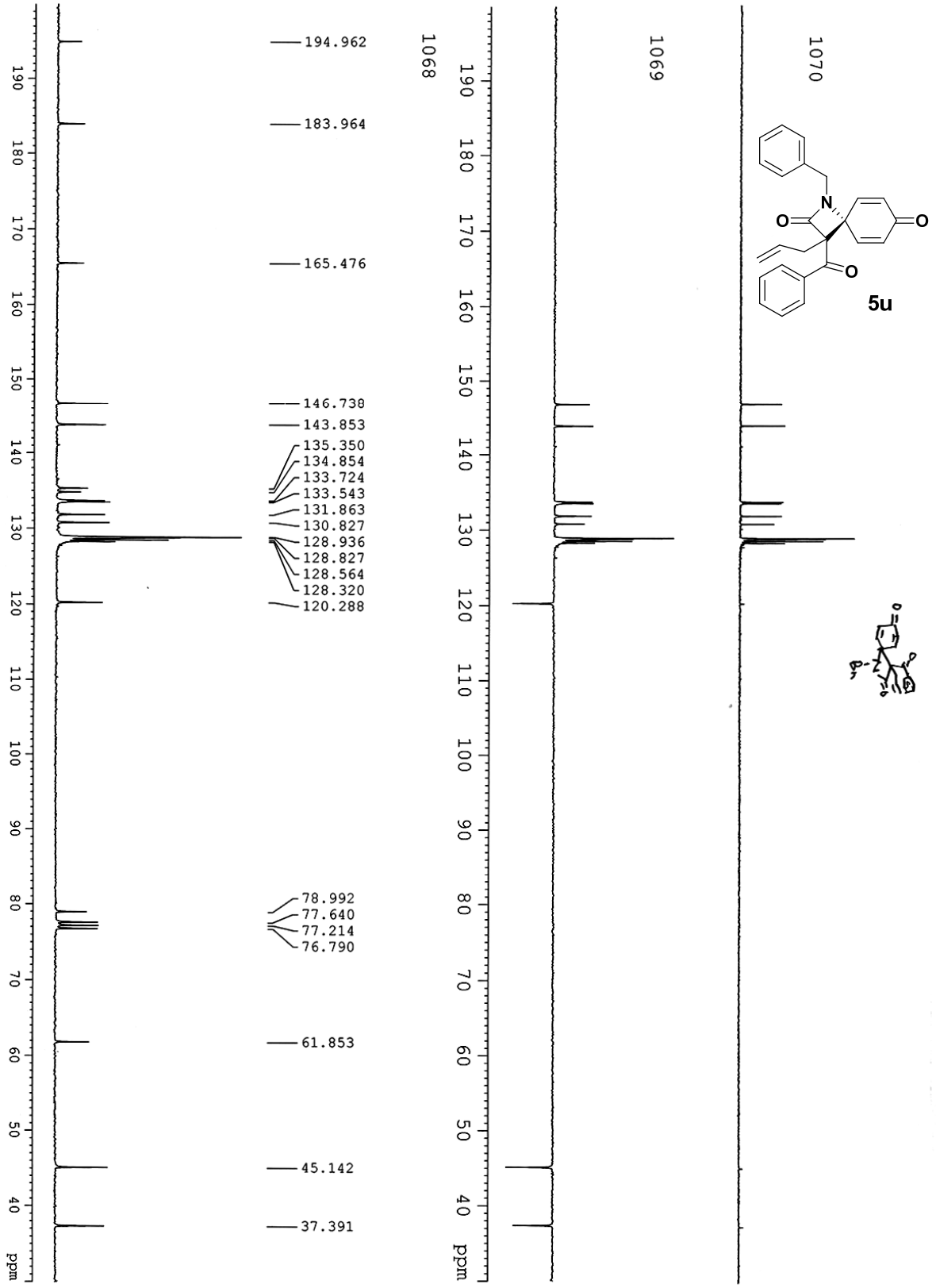
17b

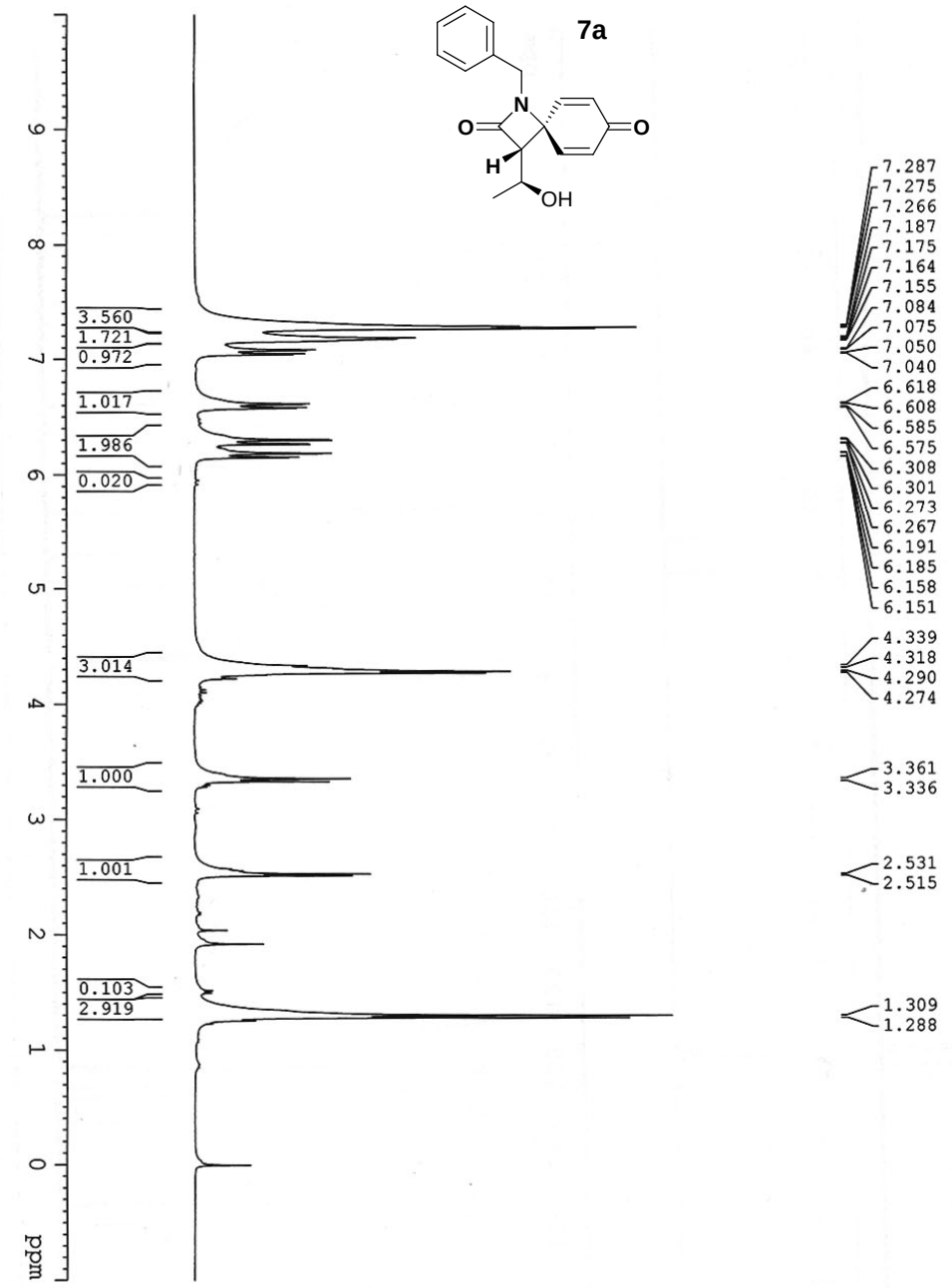

5t
7.357
7.332
7.330
6.783
6.774
6.749
6.741
6.495
6.487
6.486
6.461
6.453
6.372
6.369
6.338
6.335
6.281
6.247
6.201
6.192
5.900
5.872
5.852
5.842
5.821
5.815
5.790
5.763
5.176
5.135
4.444
4.391
4.382
4.329
2.883
2.864
2.835
2.816
2.712
2.685
2.664
2.637
2.306
2.221
1.032
1.038
1.009
4.024
1.000
2.000
2.017
1.009
0.995
3.008
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0
ppm
NAME xzx
EXPNO 1194
PROCNO 1
Date_ 20110120
Time_ 11.58
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 45.3
DW 81.000 usec
DE 6.50 usec
TE 291.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.90 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1299761 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

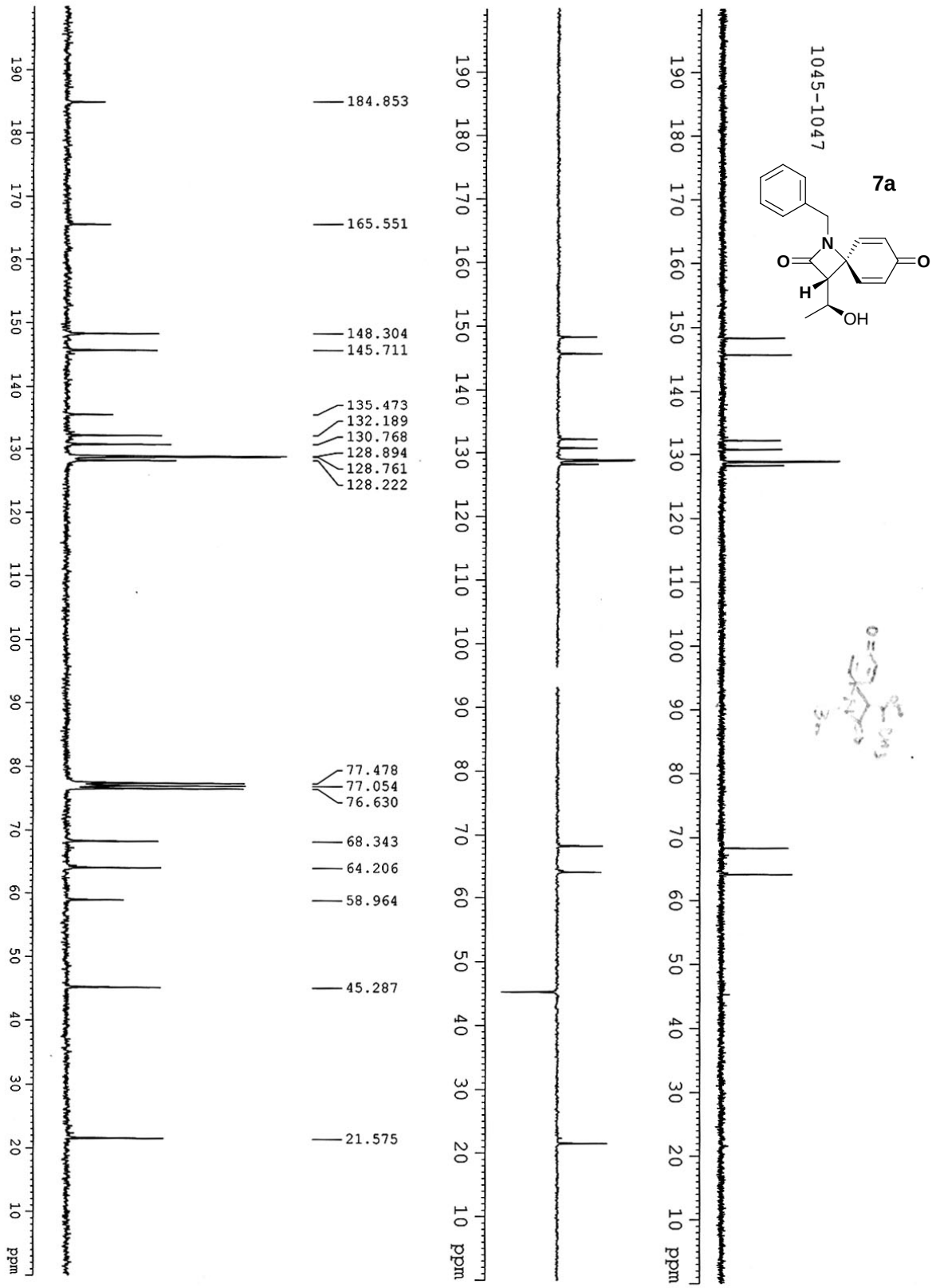

1195
1196
1193
202.003
183.988
165.483
147.538
145.601
143.419
142.649
132.841
131.364
130.780
120.157
110.851
110.316
79.217
77.648
77.222
76.797
61.231
37.102
35.534
29.574
ppm
5t

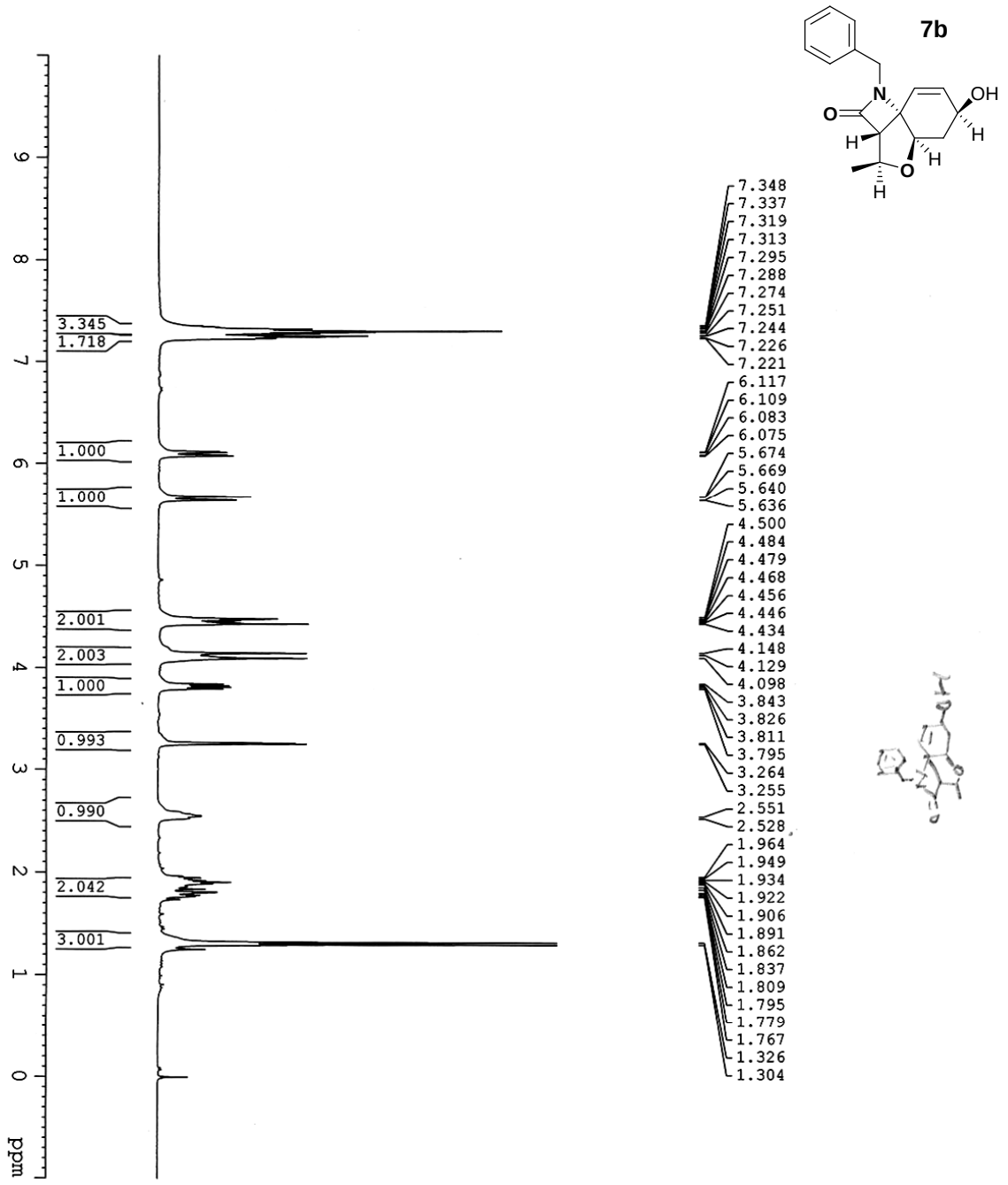

5u
7.667
7.662
7.551
7.548
7.527
7.506
7.502
7.400
7.374
7.349
7.319
7.303
7.294
7.282
7.276
7.269
7.261
7.236
7.220
7.208
7.196
7.188
6.909
6.899
6.874
6.864
6.487
6.477
6.452
6.444
6.417
6.410
6.088
6.081
6.054
6.047
5.912
5.905
5.883
5.878
5.856
5.848
5.827
5.821
5.007
4.972
4.817
4.813
4.760
4.756
4.472
4.423
4.308
4.258
3.054
3.033
3.006
2.986
2.932
2.905
2.885
2.857
2.002
1.063
2.080
3.147
1.920
1.025
2.016
1.010
0.999
1.004
1.040
1.000
0.989
2.007
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
ppm
NAME xzx
EXPNO 1067
PROCNO 1
Date_ 20101228
Time 11.59
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 45.3
DW 81.000 usec
DE 6.50 usec
TE 293.8 K
D1 1.00000000 sec
TD0 1
========== CHANNEL f1 ========
NUC1 1H
P1 7.90 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1299929 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

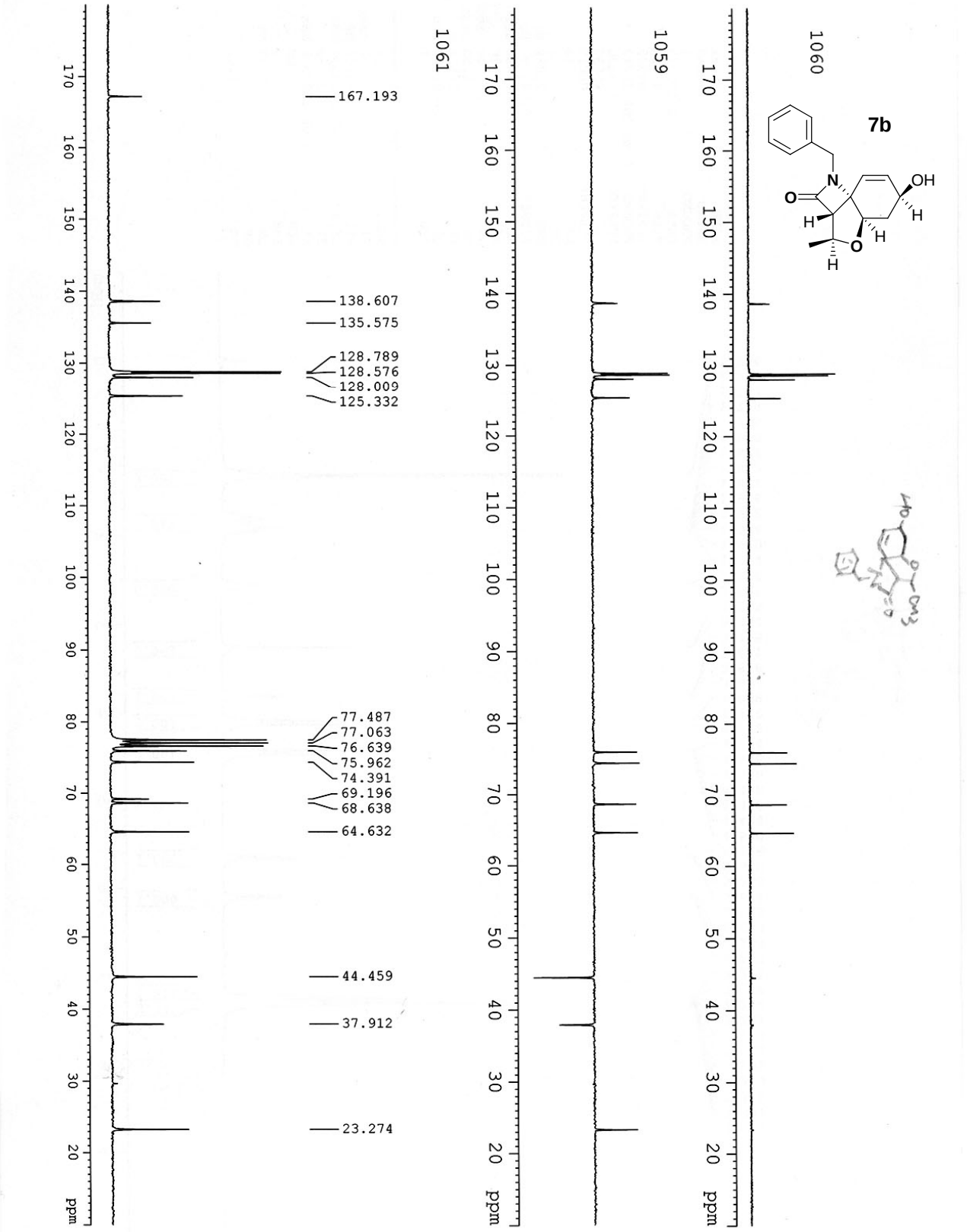

194.962
183.964
165.476
146.738
143.853
135.350
134.854
133.724
133.543
131.863
130.827
128.936
128.827
128.564
128.320
120.288
78.992
77.640
77.214
76.790
61.853
45.142
37.391
ppm
1068
1069
1070
5u

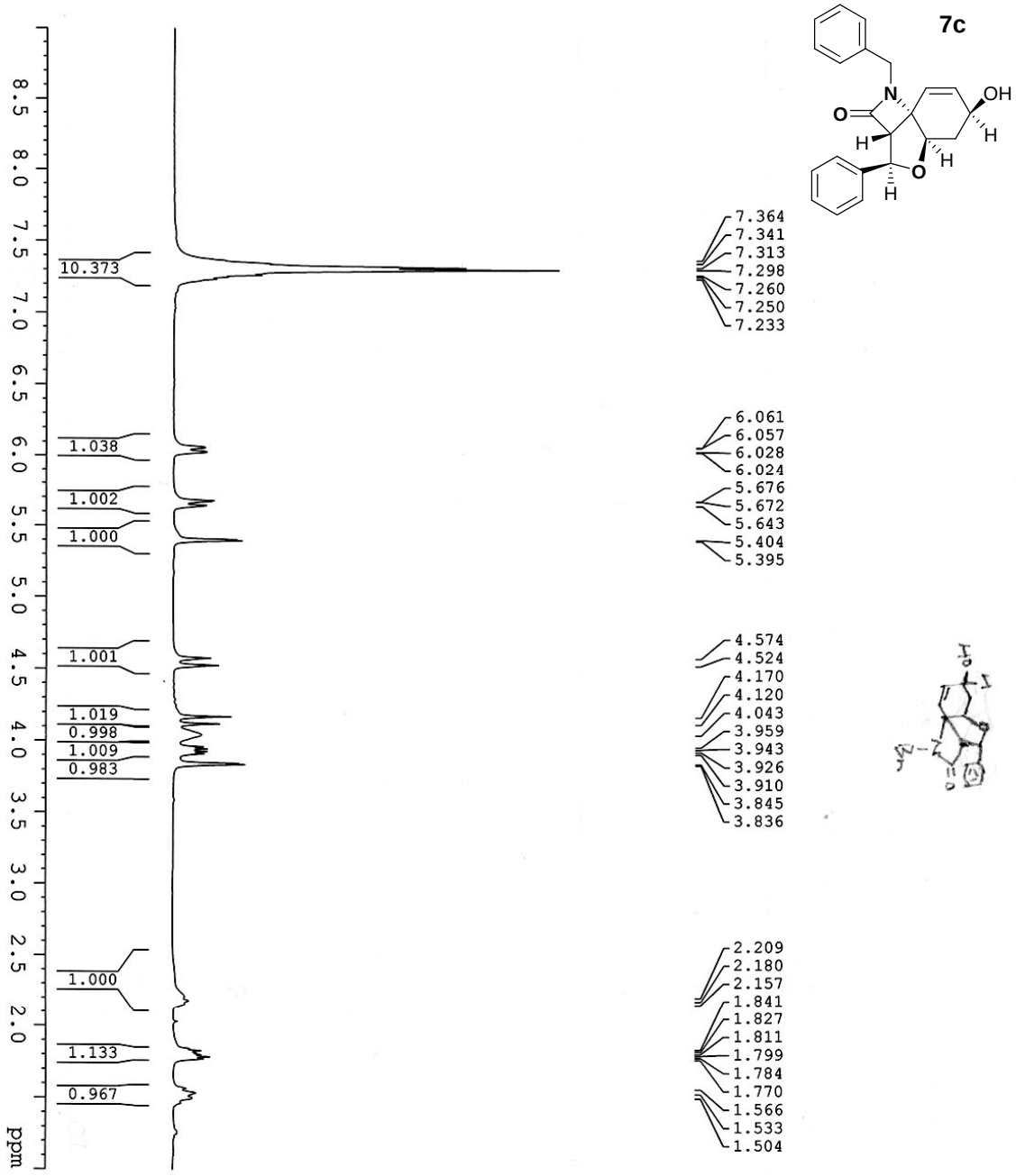

1070
1069
1068
5u
194.962
183.964
165.476
146.738
143.853
135.350
134.854
133.724
133.543
131.863
130.827
128.936
128.827
128.564
128.320
120.288
78.992
77.640
77.214
76.790
61.853
45.142
37.391
ppm

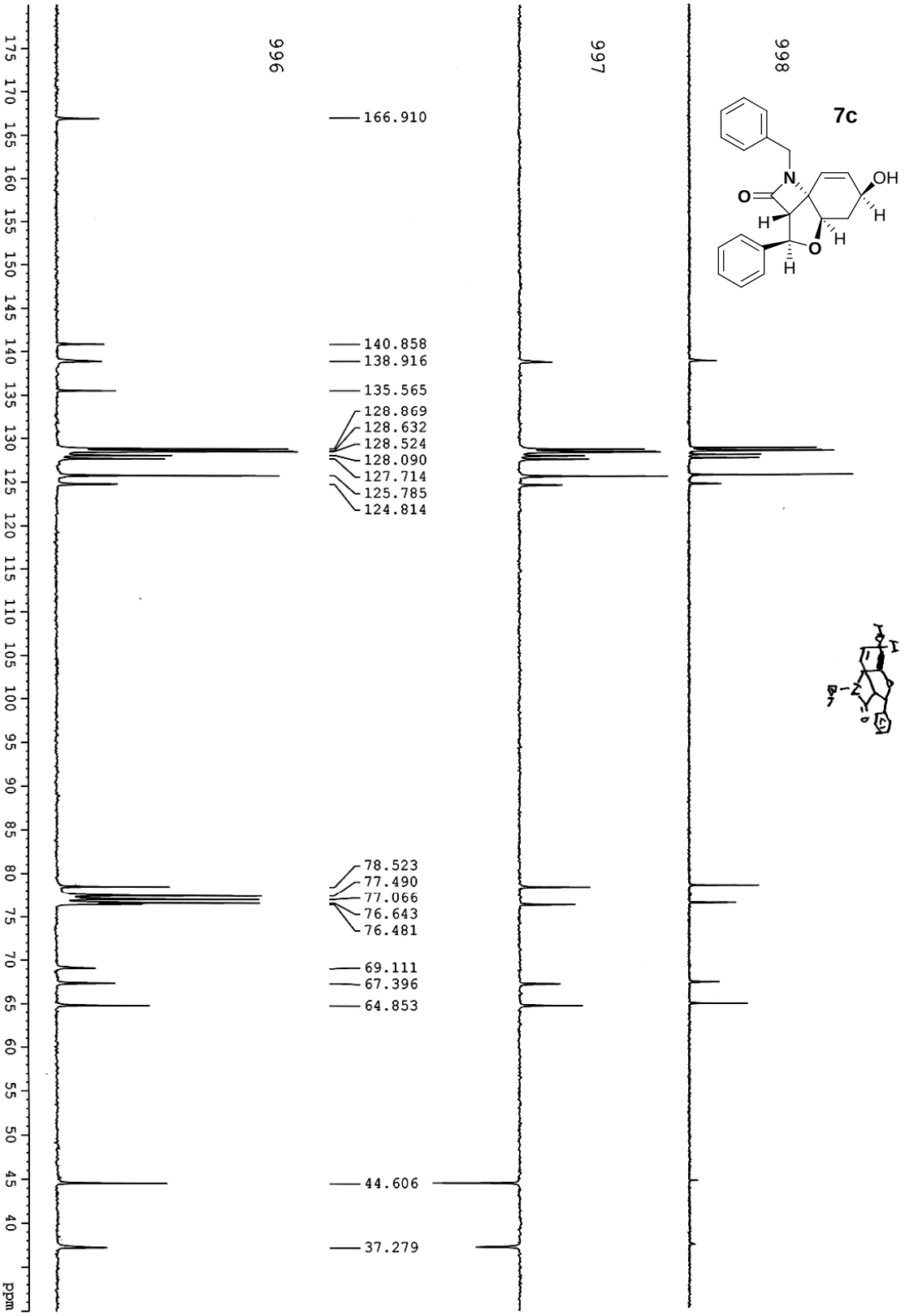

7a
N
O
O
H
OH
7.287
7.275
7.266
7.187
7.175
7.164
7.155
7.084
7.075
7.050
7.040
6.618
6.608
6.585
6.575
6.308
6.301
6.273
6.267
6.191
6.185
6.158
6.151
4.339
4.318
4.290
4.274
3.361
3.336
2.531
2.515
1.309
1.288
3.560
1.721
0.972
1.017
1.986
0.020
3.014
1.000
1.001
0.103
2.919
ppm
NAME xzx
EXPNO 1044
PROCNO 1
Date_ 20101223
Time_ 20.29
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 181
DW 81.000 usec
DE 6.50 usec
TE 295.6 K
D1 1.00000000 sec
TD0 1
========== CHANNEL f1 ========
NUC1 1H
P1 7.90 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300014 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


1045-1047
7a
N
O
O
H
OH
184.853
165.551
148.304
145.711
135.473
132.189
130.768
128.894
128.761
128.222
77.478
77.054
76.630
68.343
64.206
58.964
45.287
21.575
ppm


7b
OH
N
O
H
H
O
H
H
H
7.348
7.337
7.319
7.313
7.295
7.288
7.274
7.251
7.244
7.226
7.221
6.117
6.109
6.083
6.075
5.674
5.669
5.640
5.636
4.500
4.484
4.479
4.468
4.456
4.446
4.434
4.148
4.129
4.098
3.843
3.826
3.811
3.795
3.264
3.255
2.551
2.528
1.964
1.949
1.934
1.922
1.906
1.891
1.862
1.837
1.809
1.795
1.779
1.767
1.326
1.304
3.345
1.718
1.000
1.000
2.001
2.003
1.000
0.993
0.990
2.042
3.001
ppm
NAME xzx
EXPNO 1058
PROCNO 1
Date_ 20101225
Time 17.01
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 128
DW 81.000 usec
DE 6.50 usec
TE 295.0 K
D1 1.00000000 sec
TD0 1
========== CHANNEL f1 ==========
NUC1 1H
P1 7.90 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300018 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


7b
167.193
138.607
135.575
128.789
128.576
128.009
125.332
77.487
77.063
76.639
75.962
74.391
69.196
68.638
64.632
44.459
37.912
23.274
ppm


7c
OH
N
O
H
H
H
O
H
7.364
7.341
7.313
7.298
7.260
7.250
7.233
6.061
6.057
6.028
6.024
5.676
5.672
5.643
5.404
5.395
4.574
4.524
4.170
4.120
4.043
3.959
3.943
3.926
3.910
3.845
3.836
2.209
2.180
2.157
1.841
1.827
1.811
1.799
1.784
1.770
1.566
1.533
1.504
10.373
1.038
1.002
1.000
1.001
1.019
0.998
1.009
0.983
1.000
1.133
0.967
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0
ppm
NAME xzx
EXPNO 994
PROCNO 1
Date_ 20101212
Time_ 16.38
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 128
DW 81.000 usec
DE 6.50 usec
TE 295.4 K
D1 1.00000000 sec
TD0 1
========== CHANNEL f1 ========
NUC1 1H
P1 7.90 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
SI 32768
SF 300.1300061 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


996
997
998
166.910
140.858
138.916
135.565
128.869
128.632
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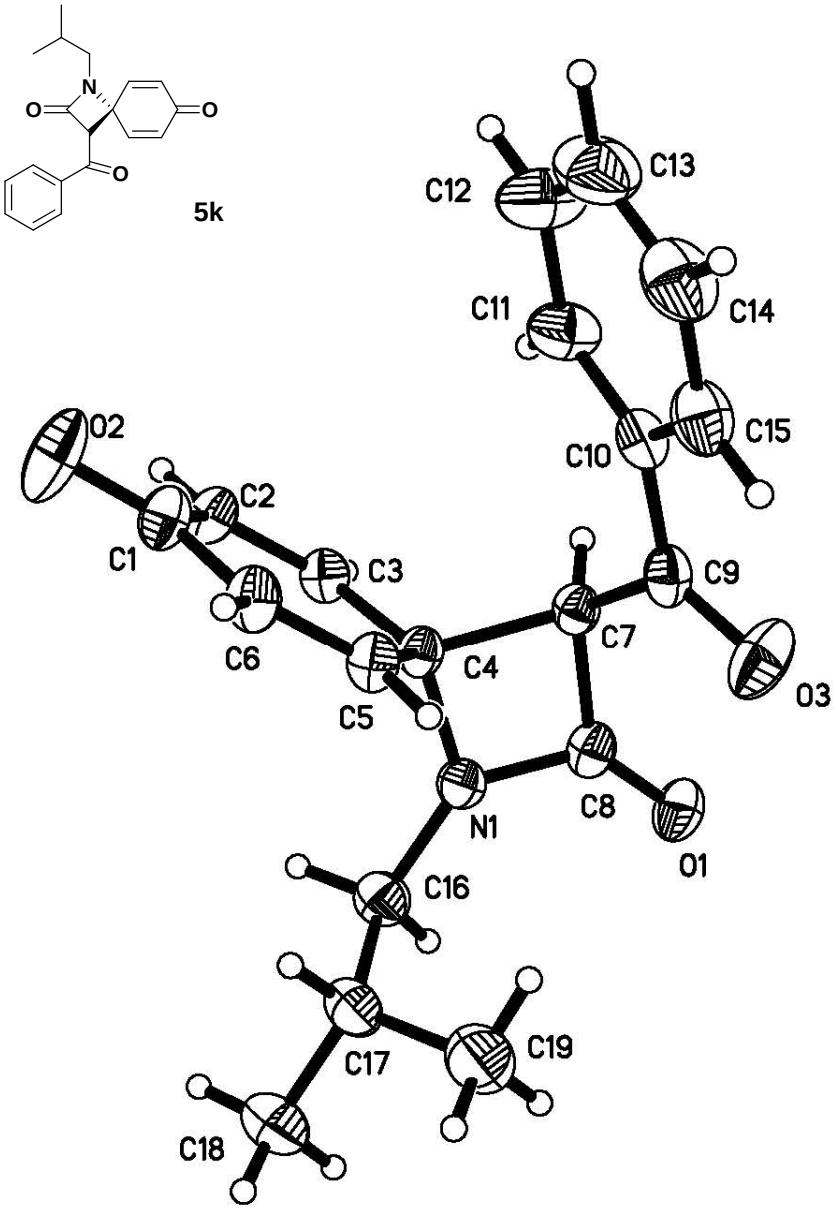
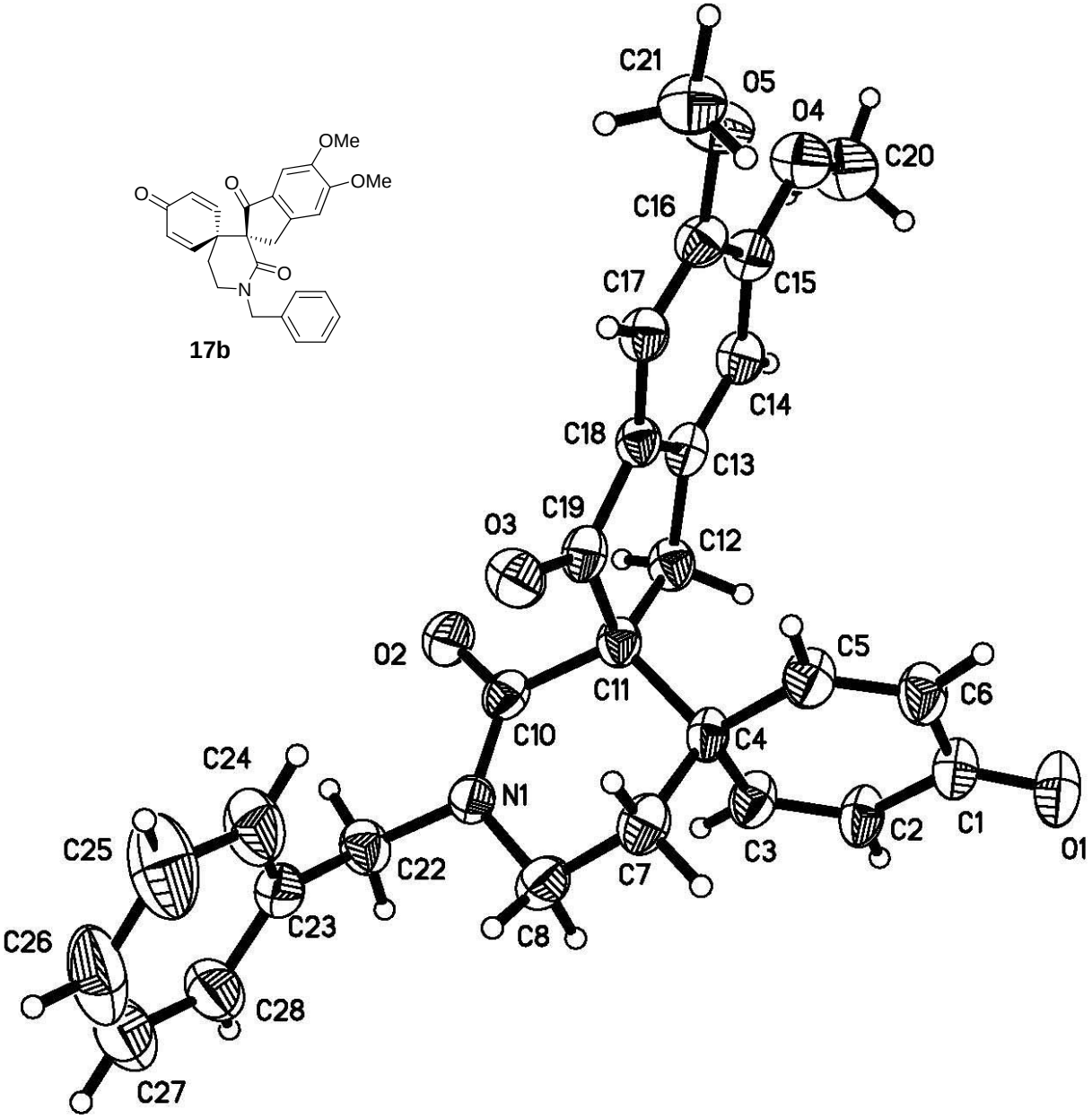
128.090
127.714
125.785
124.814
78.523
77.490
77.066
76.643
76.481
69.111
67.396
64.853
44.606
37.279
ppm
7c
N
O
OH
H
H
H
O
H


N
O
O
O
5k
C12
C13
C11
C14
C10
C15
O2
C2
C1
C3
C9
C6
C4
C7
O3
C5
N1
C8
O1
C16
C17
C19
C18

OMe
OMe
O
O
O
N

17b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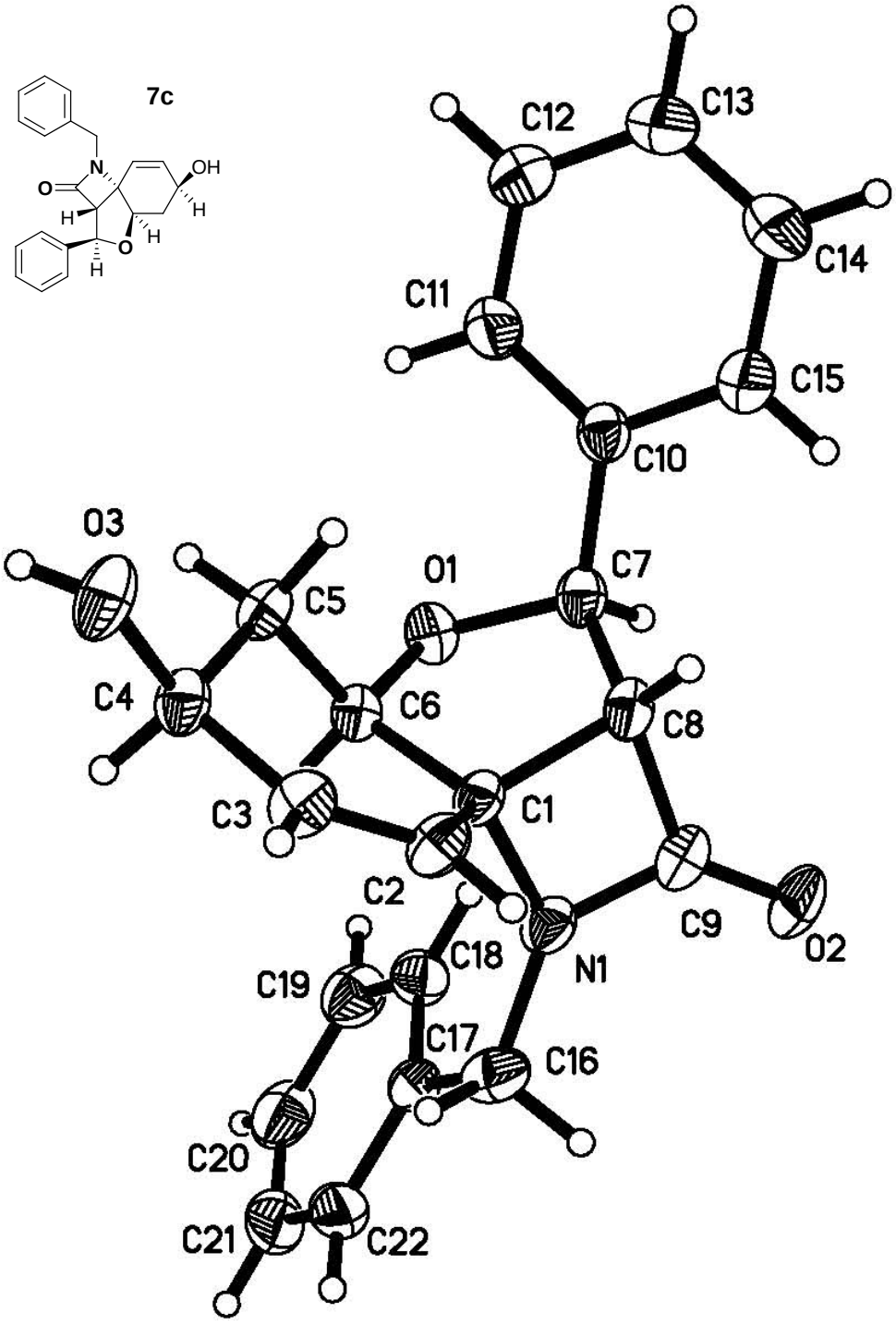
O5
O4
C20
C16
C17
C15
C14
C18
C13
C19
O3
C12
O2
C11
C5
C6
C10
C4
C24
N1
C25
C22
C7
C3
C2
C1
O1
C23
C8
C26
C28
C27


7c
O
N
OH
H
H
H
O
H
C12
C13
C14
C11
C15
C10
O3
O1
C7
C5
C4
C6
C8
C3
C1
C2
C9
O2
C18
N1
C19
C17
C16
C20
C21
C22