Supporting Information
For
A General Intramolecular Friedel-Crafts Approach to Functionalized Pyrrolo[3,2,1-*ij*]quinolin-4-ones

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1. General Methods

All reactions were carried out in pre-dried glassware from the oven and any additional moisture was removed by flame-drying the reaction vessel. Each reaction proceeded under a nitrogen atmosphere with anhydrous solvents, unless stated otherwise. Tetrahydrofuran and diethyl ether were distilled from a sodium/benzophenone ketyl under nitrogen and stored in a Schlenk flask. Benzene, toluene, 1,2-dichloroethane and dichloromethane were purified by distillation from calcium hydride. All other reagents were purchased from Acros, Sigma-Aldrich, Fluka, VWR, Merck, Alfa Aesar, TCI and Strem (for metal catalysts) and used without further purification. Compounds **9** were synthesized according to our reported protocol.[1]

Chromatographic purification was performed as flash chromatography with Silicycle silica gel (40-65μm). For quantitative flash chromatography, technical grades solvents were utilized. Analytical thin-layer chromatography (TLC) was performed on Dynamic Absorbents, Inc. silica gel F_{254} TLC glass plates. Visualization was accomplished with UV light, aqueous basic potassium permanganate ($KMnO_4$) solution, iodine, aqueous acidic dinitrophenylhydrazine (DNP) solution, aqueous acidic *p*-anisaldehyde (PAA) solution, and an ethanol solution of phosphomolybdic acid (PMA) followed by heating. Each yield refers to an isolated, analytically-pure material.

Infrared (IR) spectra were obtained using a Nicolet 4700 FTIR with an ATR attachment from SmartOrbit Thermoelectronic Corp. The IR bands are characterized as weak (w), medium (m), and strong (s). Proton and carbon nuclear magnetic resonance spectra (^1H NMR and ^{13}C NMR) were recorded on a Varian Mercury Vx 300 MHz spectrometer, Varian Mercury Vx 400 MHz spectrometer or Bruker 400 MHz spectrometer with solvent resonances as the internal standard (^1H NMR: $CDCl_3$ at 7.26 ppm; ^{13}C NMR: $CDCl_3$ at 77.0 ppm). ^1H NMR data are reported as follows: chemical shift (ppm), multiplicity (s = singlet, d = doublet, dd = doublet of doublets, dt = doublet of triplets, ddd = doublet of doublet of doublets, t = triplet, m = multiplet, br = broad), coupling constants (Hz), and integration. Mass spectra

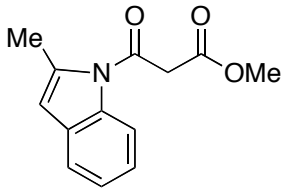
were obtained using a MicroMass Autospec M. The accurate mass analyses were run in EI mode at a mass resolution of 10,000 using PFK (perfluorokerosene) as an internal calibrant.

Diastereomeric ratios for cyclized products **11** were determined by ^{1}H NMR based on comparing the signal ratios of the benzylic protons (~4.0-5.0 ppm) for the two diastereomeric protons. These assignments are based on the coupling constants. A single observable diastereomer corresponds to >99:1 *dr*.

2. Experimental Procedures

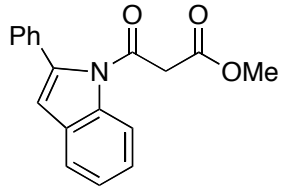
A. Synthesis of β-amide esters

Sodium hydride (1.2 equiv.) was suspended in THF and cooled to 0 °C. In a separate flask, the desired *N*-heterocycle (1.0 equiv.) was dissolved in THF and syringed into the reaction vessel. After 30 min, methyl-3-chloro-3-oxopropanoate (1.25 equiv.) was added quickly. The reaction was stirred for 14 h at room temperature. The reaction mixture was quenched with water. The organic layer was separated, and the aqueous layer was extracted three times with EtOAc. The combined organic layers were washed with brine, dried with anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was purified by silica gel flash chromatography for product isolation.



9a

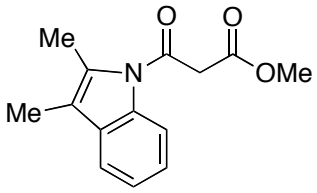
Methyl 3-(2-methyl-1*H*-indol-1-yl)-3-oxopropanoate (9a): The general procedure was followed using sodium hydride (1.91 g, 47.7 mmol), 2-methyl-1*H*-indole (5.00 g, 38.2 mmol), methyl-3-chloro-3-oxopropanoate (4.91 mL, 45.7 mmol), and THF (125 mL). After 14 h, the reaction was quenched, and column chromatography afforded **9a** as a brick red solid (6.05 g, 69%). (R_f 0.40, 30% EtOAc/Hex) [**m.p.** 74-76°C] **^{1}H NMR** (400 MHz, $CDCl_3$) δ ppm 7.87 - 7.92 (m, 1H), 7.43 - 7.48 (m, 1H), 7.21 - 7.28 (m, 2H), 6.39 (s, 1H), 4.07 (s, 2H), 3.81 (s, 3H), 2.61 (d, *J* = 1.12 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ ppm 166.9, 165.9, 137.0, 136.3, 129.8, 123.9, 123.6, 120.0, 114.9, 110.6, 52.7, 45.6, 17.3. **IR**: 3022.4 (w), 2953.1 (w), 1733.8 (s), 1700.1 (s), 1684.4 (s), 1606.3 (m), 1588.1 (m), 1526.9 (m), 1450.5 (m), 1374.4 (m), 1300.3 (m), 1235.7 (s), 1162.5 (m), 1085.2 (w), 758.3 (s), 668.5 (w), 649.4 (w) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 231.0895, Obs. 231.0894.



9b

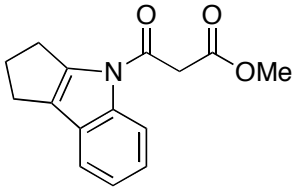
Methyl 3-oxo-3-(2-phenyl-1*H*-indol-1-yl)propanoate (9b): The general procedure was followed using sodium hydride (0.414 g, 17.3 mmol), 2-phenyl-1*H*-indole (3.00 g, 15.5 mmol), methyl-3-chloro-3-oxopropanoate (2.0 mL, 18.7 mmol), and THF (140 mL). After 5 h, the reaction was quenched, and column chromatography afforded **9b** as a orange oil (1.28 g, 28%). (R_f 0.48, 30% EtOAc/Hex) **^{1}H NMR** (400 MHz, $CDCl_3$) δ ppm 8.40 (qd, *J* = 0.84, 8.28 Hz, 1H), 7.55 - 7.59 (m, 1H), 7.44 - 7.49 (m, 5H), 7.36 -

7.41 (m, 1H), 7.29 - 7.34 (m, 1H), 6.64 (d, *J* = 0.69 Hz, 1H), 3.63 (s, 3H), 3.39 (s, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ ppm 166.8, 166.7, 139.0, 137.9, 133.3, 129.1, 129.0, 129.0, 125.5, 124.2, 120.5, 120.4, 116.3, 112.5, 52.4, 45.9. **IR**: 3029.5 (w), 2958.2 (s), 2925.4 (s), 2852.3 (s), 1745.4 (s), 1715.4 (s), 1604.8 (w), 1470.7 (m), 1451.8 (w), 1406.9 (m), 1359.4 (m), 1341.8 (w), 1301.7 (m), 1253.6 (w), 1205.1 (m), 1155.5 (m), 1117.3 (w), 1076.5 (m), 1056.7 (w), 1020.7 (w), 970.1 (m), 919.4 (w), 821.1 (w), 747.8 (m), 700.7 (w) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 293.1052, Obs. 293.1053.



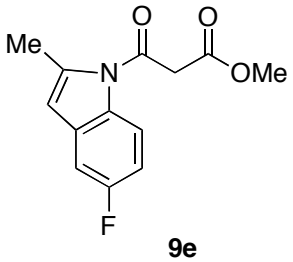
9c

Methyl 3-(2,3-dimethyl-1*H*-indol-1-yl)-3-oxopropanoate (9c): The general procedure was followed using sodium hydride (0.330 g, 8.26 mmol), 2,3-dimethyl-1*H*-indole (1.00 g, 6.89 mmol), methyl-3-chloro-3-oxopropanoate (0.921 mL, 8.61 mmol), and THF (35 mL). After 14 h, the reaction was quenched, and column chromatography afforded **9c** as a pale yellow solid (1.05 g, 62%). (R_f 0.26, 20% EtOAc/Hex) [**m.p.** 75-77°C] **^{1}H NMR** (300 MHz, $CDCl_3$) δ ppm 7.77-7.84 (m, 1H), 7.26-7.32 (m, 1H), 7.17 (m, 2H), 3.88 (s, 2H), 3.72 (s, 3H), 2.34 (s, 3H), 2.03 (s, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ ppm 8.2, 13.5, 45.2, 52.1, 114.5, 115.8, 117.7, 122.9, 123.6, 130.8, 131.6, 135.0, 165.3, 166.7. **IR**: 2989.2 (w), 2959.3 (w), 2925.6 (w), 1741.6 (s), 1681.3 (s), 1615.5 (w), 1449.6 (m), 1433.5 (m), 1361.6 (s), 1329.6 (m), 1259.5 (s), 1229.0 (w), 1162.2 (s), 1127.1 (m), 1100.2 (w), 1069.9 (w), 1019.5 (s), 928.9 (m), 833.2 (w), 759.5 (s), 691.3 (m), 613.1 (m) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 245.1052, Obs. 245.1053.



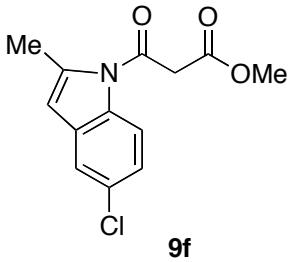
9d

Methyl 3-(2,3-dihydrocyclopenta[*b*]indol-4(1*H*)-yl)-3-oxopropanoate (9d): The general procedure was followed using sodium hydride (0.764 g, 19.1 mmol), 1,2,3,4-tetrahydrocyclopenta[*b*]indole (2.5 g, 15.9 mmol), methyl-3-chloro-3-oxopropanoate (2.1 mL, 19.1 mmol), and THF (45 mL). After 16 h, the reaction was quenched, and column chromatography afforded **9d** as a brick red solid (3.47 g, 85%). (R_f 0.30, 25% EtOAc/Hex) [**m.p.** 120-122°C] **^{1}H NMR** (300 MHz, $CDCl_3$) δ ppm 8.41 (d, *J* = 5.46 Hz, 1H), 7.31 - 7.37 (m, 1H), 7.21 - 7.30 (m, 2H), 3.86 (s, 2H), 3.79 (s, 3H), 2.92 - 3.00 (m, 2H), 2.70 - 2.78 (m, 2H), 2.45 - 2.57 (m, 2H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ ppm 167.0, 164.3, 140.9, 128.3, 126.9, 124.7, 124.2, 123.9, 118.5, 117.3, 52.7, 43.8, 29.5, 27.6, 23.6. **IR**: 3116.2 (w), 2988.6 (w), 2947.9 (w), 2928.3 (w), 2869.6 (w), 1752.8 (s), 1693.5 (s), 1608.7 (m), 1447.9 (m), 1434.9 (s), 1379.7 (m), 1346.6 (w), 1325.2 (w), 1260.4 (s), 1161.9 (m), 1122.1 (m), 1074.0 (m), 1029.6 (m), 768.6 (s), 749.6 (m), 686.1 (m) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 257.1052, Obs. 257.1045.



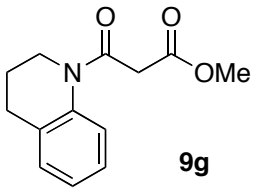
9e

Methyl 3-(5-fluoro-2-methyl-1*H*-indol-1-yl)-3-oxopropanoate (9e): The general procedure was followed using potassium hydride (0.711 g, 17.7 mmol), 5-fluoro-2-methyl-1*H*-indole (2.03 g, 13.6 mmol), methyl-3-chloro-3-oxopropanoate (1.9 mL, 17.7 mmol), and THF (18 mL). After 14 h, the reaction was quenched, and column chromatography afforded **9e** as a red solid (0.805 g, 24%). (R_f 0.20, 20% EtOAc/Hex) [**m.p.** 80-82°C] **^{1}H NMR** (300 MHz, $CDCl_3$) δ ppm 7.96 (dd, *J* = 4.45, 9.11 Hz, 1H), 7.08 (dd, *J* = 2.60, 8.54 Hz, 1H), 6.95 (dt, *J* = 2.64, 9.09 Hz, 1H), 6.35 (s, 1H), 4.03 (s, 2H), 3.80 (s, 3H), 2.59 (s, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ ppm 166.8, 165.6, 161.2, 158.0, 138.1, 133.0, 130.9, 130.8, 116.5 and 116.4 (doublet), 111.6, 111.3, 110.6 and 110.5 (doublet), 105.8, 105.4, 52.8, 45.4, 17.3. **IR**: 3013.0 (w), 2956.9 (w), 1752.2 (s), 1682.4 (s), 1603.6 (m), 1476.2 (m), 1438.8 (m), 1376.3 (m), 1301.9 (w), 1259.5 (w), 1187.8 (m), 1157.8 (s), 1129.8 (m), 1000.5 (m), 958.5 (m), 870.7 (m), 797.1 (m), 780.4 (m), 668.4 (m) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 249.0801, Obs. 249.0809.



9f

Methyl 3-(5-chloro-2-methyl-1*H*-indol-1-yl)-3-oxopropanoate (9f): The general procedure was followed using sodium hydride (0.2908 g, 7.27 mmol), 5-chloro-2-methyl-1*H*-indole (1.00 g, 6.06 mmol), methyl-3-chloro-3-oxopropanoate (0.811 mL, 7.57 mmol), and THF (30 mL). After 16 h, the reaction was quenched, and column chromatography afforded **9f** as a reddish brown solid (0.114 g, 11%). (R_f 0.25, 20% EtOAc/Hex) [**m.p.** 46-48°C] **^{1}H NMR** (300 MHz, $CDCl_3$) δ ppm 7.88 (dd, *J* = 0.44, 8.90 Hz, 1H), 7.35 (d, *J* = 2.05 Hz, 1H), 7.16 (dd, *J* = 2.09, 8.90 Hz, 1H), 6.27 (d, *J* = 0.62 Hz, 1H), 3.99 (s, 2H), 3.79 (s, 3H), 2.55 (s, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ ppm 166.6, 165.6, 137.8, 134.9, 130.9, 129.1, 123.9, 119.4, 116.3, 109.9, 52.7, 45.3, 17.2. **IR**: 2993.0 (w), 2953.8 (w), 1746.0 (s), 1697.9 (s), 1594.7 (m), 1446.3 (s), 1362.6 (s), 1334.8 (m), 1309.2 (m), 1254.0 (s), 1159.8 (s), 1075.0 (w), 1041.4 (w), 1000.9 (w), 919.6 (w), 807.6 (w), 723.6 (w) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 265.0501, Obs. 265.0499.



9g

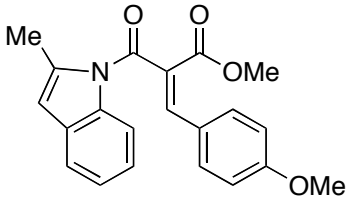
Methyl 3-(3,4-dihydroquinolin-1(2*H*)-yl)-3-oxopropanoate (9g): A mixture of potassium carbonate (6.23 g, 44.0 mmol) and 1,2,3,4-tetrahydroquinoline (3.0 g, 22.5 mmol), methyl-3-chloro-3-oxopropanoate (2.7 mL, 24.8 mmol) and acetonitrile (60 mL) were heated to reflux. After 14 h, the reaction mixture was cooled, filtered and dried *in vacuo.* The residue was dissolved in EtOAc/Hex (1:2.5). The organic layer

was separated, and the aqueous layer was extracted three times with EtOAc. The combined organic layers were dried with anhydrous sodium sulfate, filtered, and concentrated. Column chromatography afforded **9g** as a reddish orange oil (3.76 g, 72%). (R_f 0.30, 25% EtOAc/Hex) **^{1}H NMR** (300 MHz, $CDCl_3$) δ ppm 7.07 - 7.19 (m, 4H), 3.74 - 3.83 (m, 2H), 3.65 (s, 3H), 3.58 (s, 2H), 2.69 (t, *J*=6.49 Hz, 2H), 1.94 (quin, *J*=6.65 Hz, 2H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ ppm 168.1, 165.5, 128.4, 126.3, 125.8, 123.9, 52.2, 42.7, 41.4, 26.4, 23.7. **IR**: 3004.1 (w), 2951.0 (w), 2889.1 (w), 1739.3 (s), 1651.4 (s), 1603.6 (w), 1580.9 (w), 1491.8 (s), 1435.5 (w), 1386.9 (m), 1326.1 (w), 1201.5 (m), 1155.7 (m), 1074.0 (w), 1019.9 (m), 949.0 (w), 763.0 (s) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 233.1052, Obs. 233.1031.

B. Preparation of Acrylates

General Method A:[2] The β-amide ester (1.0 equiv.), aldehyde (1.3 equiv.), glacial acetic acid (0.5 equiv.), and piperidine (0.1 equiv.) were heated to a reflux in benzene using a Dean-Stark trap for 14 h. After cooling the reaction mixture to room temperature, water was added to the reaction vessel, and the organic layer was collected. Subsequently, the aqueous phase was extracted with EtOAc three times. The combined organic layers were washed with 1M HCl and saturated sodium bicarbonate. The combined organic layers were dried with Na_2SO_4, filtered, concentrated, and purified by silica gel column chromatography (gradient EtOAc/Hex).

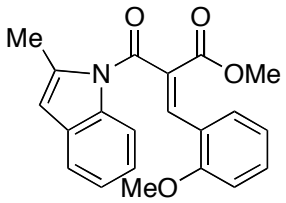
General Method B:[3] A round bottom flask was charged with the β-amide ester (1.0 equiv.) and THF (25 mL). After cooling the solution to 0 °C, titanium(IV) chloride tetrahydrofuran complex (2.0 equiv.) and CCl_4 (2.0 equiv.) were added to the reaction vessel. After 1 h at 0 °C, the aldehyde (1.0 equiv.) was added slowly, and the reaction was stirred for an hour. Then, pyridine (4.0 equiv.) was added to the solution dropwise. The reaction mixture was warmed to room temperature and allowed to stir for 14 h. The reaction was quenched with water and the organic layer was collected. The aqueous layer was extracted with ether, and the combined organic layers were washed with saturated $NaHCO_3$ and brine. The organic layer was dried with Mg_2SO_4, filtered, concentrated, and purified by silica gel column chromatography (gradient EtOAc/Hex).



(Z)-10a

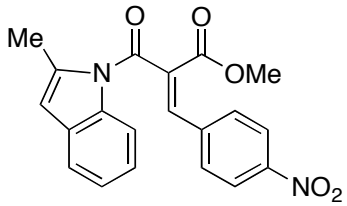
(*Z*)-Methyl 3-(4-methoxyphenyl)-2-(2-methyl-1*H*-indole-1-carbonyl)acrylate (10a): Methyl 3-(2-methyl-1*H*-indol-1-yl)-3-oxopropanoate (1.80 g, 7.78 mmol), 4-methoxybenzaldehyde (1.2 mL, 10.1 mmol), glacial acetic acid (0.262 g, 4.37 mmol), piperidine (80 µL , 0.810 mmol) and benzene (120 mL) were mixed according to general method A to afford **10a** as an orange oil (2.50 g, 92%) after 18 h. (R_f 0.24, 20% EtOAc/Hex) (*Diastereomer!*) **^{1}H NMR** (300 MHz, $CDCl_3$*)* δ ppm 8.43 (br. s., 0.81), 7.87 (s, 1.13), 7.72 (s, 0.15), 7.33 - 7.47 (m, 3.50), 7.21 - 7.30 (m, 2.08), 6.89 (d, *J* = 8.79 Hz, 0.27), 6.75 (d, *J* = 8.76 Hz, 2.10), 6.35 (s, 1.00), 3.87 (d, *J* = 0.70 Hz, 0.25), 3.83 (s, 0.26), 3.81 (s, 0.31), 3.77 (s, 2.63), 3.72 (s, 2.94), 2.48 (br. s., 2.87). **^{13}C NMR** (75 MHz, $CDCl_3$) δ ppm 166.3, 165.1, 161.8, 142.9, 142.5, 131.9, 131.4, 129.8, 125.6, 124.7, 119.6, 114.5, 114.3, 55.2, 52.7, 16.7. **IR:** 3065.3 (w), 2951.7 (w), 2939.1 (w), 1720.6 (s), 1682.4 (s), 1600.9 (s), 1511.8 (s), 1452.5 (s), 1385.8 (m), 1321.3 (m), 1290.4 (m), 1258.9 (s), 1203.7 (m),

1172.3 (s), 1123.0 (m), 1056.1 (w), 1027.6 (m), 917.2 (w), 831.4 (m), 763.9 (s), 751.0 (s), 700.6 (m) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 349.1314, Obs. 349.1319.



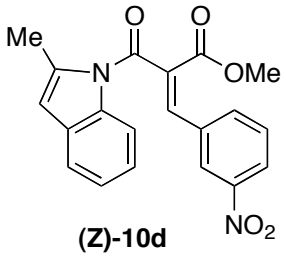
(Z)-10b

(*Z*)-Methyl 3-(2-methoxyphenyl)-2-(2-methyl-1H-indole-1-carbonyl)acrylate (10b): Methyl 3-(2-methyl-1*H*-indol-1-yl)-3-oxopropanoate (0.200 g, 0.865 mmol), 2-methoxybenzaldehyde (0.153 g, 1.12 mmol), glacial acetic acid (0.026 g, 0.433 mmol), piperidine (0.0147 g, 0.173 mmol) and benzene (20 mL) were mixed according to general method A to afford **10b** as a yellow solid (0.231 g, 75%) after 16 h. (R_f 0.69, 30% EtOAc/Hex) [**m.p.** 102-104 °C] (*Temperature for the ^{1}H NMR and ^{13}C NMR = 70 °C)* **^{1}H NMR** (400 MHz, $CDCl_3$) δ ppm 8.30 (s, 1H), 8.22 (d, *J* = 7.28 Hz, 1H), 7.43 - 7.48 (m, 1H), 7.36 - 7.42 (m, 1H), 7.28 - 7.34 (m, 1H), 7.22 - 7.28 (m, 2H), 6.87 (d, *J* = 8.34 Hz, 1H), 6.82 (t, *J* = 7.84 Hz, 1H), 6.36 (s, 1H), 3.83 (s, 3H), 3.74 (s, 3H), 2.60 (d, *J* = 0.94 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ ppm 165.9, 165.2, 158.3, 139.7, 137.1, 132.2, 130.2, 129.9, 129.2, 123.8, 123.5, 122.2, 120.9, 119.6, 115.6, 111.2, 110.0, 55.3, 52.4, 16.5. **IR**: 3051.9 (w), 3003.8 (w), 2951.9 (m), 2840.2 (m), 1713.4 (s), 1680.3 (s), 1618.7 (m), 1596.8 (s), 1574.7 (m), 1487.0 (m), 1455.4 (s), 1435.7 (s), 1383.5 (s), 1307.0 (s), 1261.0 (s), 1240.3 (s), 1194.9 (s), 1164.3 (m), 1116.8 (m), 1083.5 (m), 1050.6 (w), 1024.7 (m), 993.0 (w), 840.3 (w), 804.0 (w), 749.2 (s), 730.4 (s), 648.0 (w) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 349.1314, Obs. 349.1315.



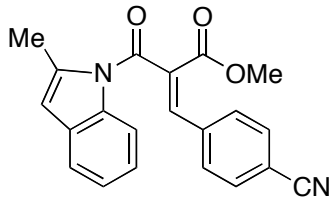
(Z)-10c

(*Z*)-Methyl 2-(2-methyl-1*H*-indole-1-carbonyl)-3-(4-nitrophenyl)acrylate (10c): Methyl 3-(2-methyl-1*H*-indol-1-yl)-3-oxopropanoate (0.300 g, 1.30 mmol), 4-nitrobenzaldehyde (0.255 mg, 1.69 mmol), glacial acetic acid (0.0357 g, 0.596 mmol), piperidine (14.0 µL, 0.130 mmol) and benzene (25 mL) were mixed according to general method A to afford **10c** as an orange solid (0.300 g, 64%) after 18 h. (R_f 0.45, 20% EtOAc/Hex) [**m.p.** 109-111 °C] (*Temperature for the ^{1}H NMR and ^{13}C NMR = 70 °C)* **^{1}H NMR** (400 MHz, $CDCl_3$) δ ppm 8.03 - 8.09 (m, 3H), 7.94 (s, 1H), 7.49 - 7.54 (m, 2H), 7.38 - 7.42 (m, 1H), 7.20 - 7.25 (m, 2H), 6.34 (d, *J* = 0.94 Hz, 1H), 3.85 (s, 3H), 2.51 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ ppm 164.4, 164.0, 148.8, 139.9, 138.5, 136.7, 132.8, 130.0, 129.9, 124.3, 124.1, 123.9, 120.0, 115.0, 111.2, 52.9, 16.4. **IR**: 3108.3 (w), 2953.4 (w), 2929.6 (w), 1726.0 (s), 1678.5 (s), 1596.9 (m), 1521.3 (s), 1456.1 (s), 1436.1 (m), 1384.8 (s), 1291.1 (s), 1256.0 (s), 1200.9 (s), 1111.8 (w), 1083.9 (w), 1027.5 (w), 992.3 (w), 852.1 (w), 825.8 (w), 749.1 (s), 691.0 (w) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 364.1059, Obs. 364.1076.



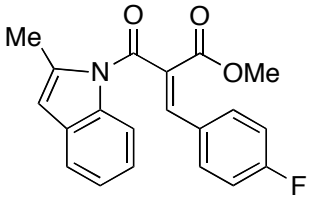
(Z)-10d

(*Z*)-Methyl 2-(2-methyl-1*H*-indole-1-carbonyl)-3-(3-nitrophenyl)acrylate (10d): Methyl 3-(2-methyl-1*H*-indol-1-yl)-3-oxopropanoate (0.350 g, 1.513 mmol), 3-nitrobenzaldehyde (0.297 mg, 1.97 mmol), glacial acetic acid (0.0417 g, 0.696 mmol), piperidine (15 µL , 0.151 mmol) and benzene (20 mL) were mixed according to general method A to afford **10d** as a pale yellow solid (0.220 g, 40 %) after 20 h. (R_f 0.40, 20% EtOAc/Hex) [**m.p.** 112-114 °C] (*Temperature for the ^{1}H NMR and ^{13}C NMR = 60 °C)* **^{1}H NMR** (400 MHz, $CDCl_3$) δ ppm 8.21 - 8.24 (m, 1H), 8.10 (ddd, *J* = 1.00, 2.16, 8.25 Hz, 1H), 8.03 (br. s., 1H), 7.94 (s, 1H), 7.63 - 7.67 (m, 1H), 7.35 - 7.41 (m, 2H), 7.18 - 7.25 (m, 2H), 6.33 (d, *J* = 0.88 Hz, 1H), 3.86 (s, 3H), 2.54 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ ppm 164.6, 164.2, 148.7, 139.9, 136.7, 134.4, 134.2, 132.1, 130.1, 130.0, 124.9, 124.3, 124.2, 124.2, 120.0, 115.1, 111.3, 53.1, 16.7. **IR**: 3083.4 (w), 2954.9 (w), 2928.5 (w), 1727.7 (s), 1678.7 (s), 1630.1 (w), 1597.5 (w), 1576.9 (w), 1532.1 (s), 1456.4 (s), 1437.6 (m), 1386.4 (s), 1351.2 (s), 1308.8 (s), 1258.6 (s), 1203.4 (s), 1086.2 (w), 1027.7 (w), 992.8 (w), 824.9 (w), 808.9 (w), 751.6 (m), 736.8 (m), 676.3 (w) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 364.1059, Obs. 364.1065.



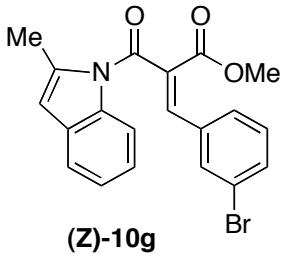
(Z)-10e

(*Z*)-Methyl 3-(4-cyanophenyl)-2-(2-methyl-1*H*-indole-1-carbonyl)acrylate (10e): Methyl 3-(2-methyl-1*H*-indol-1-yl)-3-oxopropanoate (0.350 g, 1.51 mmol), 4-cyanobenzaldehyde (0.258 mg, 1.97 mmol), glacial acetic acid (0.0417 g, 0.696 mmol), piperidine (15 µL , 0.151 mmol) and benzene (20 mL) were mixed according to general method A to afford **10e** as an off-white solid (0.325 g, 62%) after 18 h. (R_f 0.35, 20% EtOAc/Hex) [**m.p.** 122-124 °C] **^{1}H NMR** (300 MHz, $CDCl_3$) δ ppm 8.81 - 8.05 (br. s., 1H), 7.38 - 7.53 (m, 6H), 7.20 - 7.28 (m, 2H), 6.35 (s, 1H), 3.82 (s, 3H), 2.46 (br. s., 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ ppm 164.6, 164.0, 140.4, 136.4, 132.5, 131.5, 129.6, 124.2, 119.9, 117.7, 113.8, 111.2, 53.1, 16.7. **IR**: 3056.6 (w), 2954.9 (w), 2928.2 (w), 2229.5 (m), 1725.7 (s), 1677.7 (s), 1627.5 (m), 1596.9 (m), 1576.6 (m), 1504.2 (w), 1456.0 (s), 1435.5 (m), 1384.3 (s), 1303.3 (s), 1256.1 (s), 1201.7 (s), 1152.5 (w), 1117.1 (w), 1084.3 (m), 1027.4 (m), 992.3 (m), 936.0 (w), 830.2 (m), 750.5 (s) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 344.1161, Obs. 344.1169.



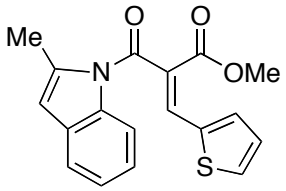
(Z)-10f

(*Z*)-Methyl 3-(4-fluorophenyl)-2-(2-methyl-1*H*-indole-1-carbonyl)acrylate (10f): Methyl 3-(2-methyl-1*H*-indol-1-yl)-3-oxopropanoate (0.350 g, 1.51 mmol), 4-fluorobenzaldehyde (0.210 mL, 2.00 mmol), glacial acetic acid (0.0417 g, 0.695 mmol), piperidine (15 µL, 0.151 mmol) and benzene (25 mL) were mixed according to general method A to afford **10f** as a yellow solid (0.293 g, 57%) after 18 h. (R_f 0.40, 20% EtOAc/Hex) [**m.p.** 76-78 °C] (*Temperature for the ^{1}H NMR and ^{13}C NMR = 60 °C)* **^{1}H NMR** (300 MHz, $CDCl_3$) δ ppm 8.20 (br. s., 1H), 7.89 (s, 1H), 7.34 - 7.45 (m, 3H), 7.17 - 7.30 (m, 2H), 6.92 (t, *J* = 8.59 Hz, 2H), 6.34 (s, 1H), 3.80 (s, 3H), 2.50 (s, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ ppm 165.7, 165.5, 164.7, 162.4, 141.8, 136.9, 131.9 and 131.7 (doublet), 129.9, 124.2 and 124.0 (doublet), 119.8, 116.4, 116.1, 115.5, 110.9, 52.7, 16.6. **IR**: 3076.2 (w), 2951.8 (w), 1724.8 (s), 1683.9 (s), 1627.8 (w), 1598.7 (s), 1508.9 (s), 1456.5 (s), 1436.7 (m), 1386.76 (s), 1301.0 (m), 1260.4 (s), 1197.7 (s), 1162.4 (s), 834.7 (m), 750.5 (s), 668.5 (m) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 337.1114, Obs. 349.1107.



(Z)-10g

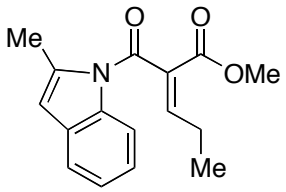
(*Z*)-Methyl 3-(3-bromophenyl)-2-(2-methyl-1*H*-indole-1-carbonyl)acrylate (10g): Methyl 3-(2-methyl-1*H*-indol-1-yl)-3-oxopropanoate (0.350 g, 1.51 mmol), 3-bromobenzaldehyde (0.230 mL, 1.97 mmol), glacial acetic acid (0.0417 g, 0.696 mmol), piperidine (15 µL , 0.151 mmol) and benzene (20 mL) were mixed according to general method A to afford **10g** as a pale gray solid (0.430 g, 72 %) after 18 h. (R_f 0.40, 20% EtOAc/Hex) [**m.p.** 107-109 °C] (*Temperature for the ^{1}H NMR and ^{13}C NMR = 60 °C)* **^{1}H NMR** (400 MHz, $CDCl_3$) δ ppm 8.08 - 8.19 (m, 1H), 7.86 (s, 1H), 7.52 - 7.56 (m, 1H), 7.39 - 7.44 (m, 2H), 7.27 - 7.33 (m, 1H), 7.21 - 7.27 (m, 2H), 7.05 - 7.11 (m, 1H), 6.35 (d, *J* = 0.75 Hz, 1H), 3.85 (s, 3H), 2.54 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ ppm 165.0, 164.4, 141.2, 136.8, 136.5, 134.5, 133.5, 132.5, 130.5, 130.3, 130.0, 127.5, 124.2, 124.0, 123.0, 119.8, 115.3, 111.0, 52.8, 16.6. **IR**: 2962.5 (w), 2926.4 (w), 1726.6 (s), 1681.3 (s), 1625.7 (m), 1596.8 (w), 1561.3 (w), 1456.4 (s), 1435.7 (m), 1384.9 (s), 1311.0 (s), 1258.9 (s), 1198.1 (s), 1076.9 (w), 1027.7 (w), 994.3 (w), 786.6 (w), 750.3 (s), 680.8 (w) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 397.0314, Obs. 397.0316.



(Z)-10h

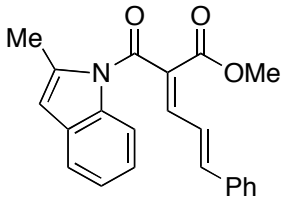
(*Z*)-Methyl 2-(2-methyl-1*H*-indole-1-carbonyl)-3-(thiophen-2-yl)acrylate (10h): Methyl 3-(2-methyl-1*H*-indol-1-yl)-3-oxopropanoate (0.350 g, 1.51 mmol), thiophene-2-carbaldehyde (0.220 mg, 1.97 mmol), glacial acetic acid (0.0417 g, 0.696 mmol), piperidine (15 µL , 0.150 mmol) and benzene (20 mL) were mixed according to general method A to afford **10h** as an off-white solid (0.396 g, 81%) after 18 h. (R_f 0.45, 20% EtOAc/Hex) [**m.p.** 153-155 °C] **^{1}H NMR** (300 MHz, $CDCl_3$) δ ppm 8.28 (br. s., 1H), 8.04 (d, *J* = 0.37 Hz, 1H), 7.42 - 7.48 (m, 1H), 7.36 (d, *J* = 5.06 Hz, 1H), 7.29 - 7.33 (m, 1H), 7.23 - 7.29 (m, 2H), 6.95 - 7.00 (m, 1H), 6.39 (s, 1H), 3.79 (s, 3H), 2.50 (s, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ ppm 165.3, 164.9, 136.9,

135.7, 135.1, 134.6, 132.5, 129.9, 128.0, 124.9, 124.2, 124.0, 119.7, 115.9, 111.0, 108.0, 52.8, 16.8. **IR**: 3104.8 (w), 2952.1 (w), 2927.5 (w), 1719.0 (s), 1675.8 (s), 1611.7 (s), 1455.9 (s), 1385.9 (s), 1342.5 (m), 1303.5 (s), 1253.5 (s), 1202.6 (s), 1086.3 (w), 1051.6 (w), 1027.9 (w), 992.9 (w), 858.1 (w), 750.5 (s), 717.4 (m) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 325.0773, Obs. 325.0780.



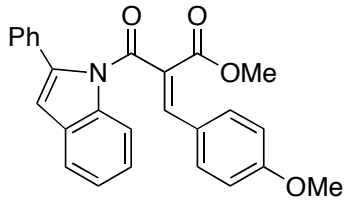
(**Z)-10i**

(*Z*)-methyl 2-(2-methyl-1*H*-indole-1-carbonyl)pent-2-enoate (10i): Methyl 3-(2-methyl-1*H*-indol-1-yl)-3-oxopropanoate (0.500 g, 2.16 mmol), propanaldehyde (0.155 mL, 2.16 mmol), $TiCl_4$•THF (1.44 g, 4.32 mmol), CCl_4 (0.418 mL, 4.32 mmol), pyridine (0.699 mL, 8.65 mmol) and THF (35 mL) were combined according to general method B to yield **10i** as a clear oil (0.421 g, 72%) after 14 h. (R_f 0.55, 20% EtOAc/Hex) **^{1}H NMR** (300 MHz, $CDCl_3$) δ ppm 8.13 (br. s., 1H), 7.50 - 7.58 (m, 1H), 7.27 - 7.36 (m, 3H), 6.48 (d, *J* = 0.77 Hz, 1H), 3.82 (s, 3H), 2.60 (s, 3H), 2.28 (quin, *J* = 7.63 Hz, 2H), 1.13 (t, *J* = 7.53 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ ppm 165.1, 164.2, 150.7, 136.8, 136.5, 131.5, 129.8, 123.8, 123.7, 119.8, 115.2, 110.4, 52.5, 23.1, 16.9, 12.3. **IR**: 2973.6 (w), 2936.1 (w), 1726.6 (s), 1685.9 (s), 1643.0 (w), 1596.5 (w), 1575.5 (w), 1456.3 (s), 1436.9 (m), 1384.1 (s), 1310.8 (s), 1289.7 (s), 1243.9 (s), 1205.4 (w), 1036.7 (w), 989.8 (w), 783.4 (w), 750.3 (s) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 271.1208, Obs. 271.1218.



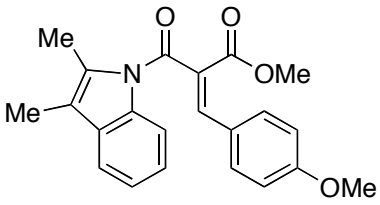
(**Z)-10j**

(2*Z*, 4*E*)-Methyl 2-(2-methyl-1*H*-indole-1-carbonyl)-5-phenylpenta-2,4-dienoate (10j): Methyl 3-(2-methyl-1*H*-indol-1-yl)-3-oxopropanoate (0.300 g, 1.30 mmol), cinnamaldehyde (0.21 mL, 1.69 mmol), glacial acetic acid (0.0357 g, 0.596 mmol), piperidine (13.97 µL, 0.1297 mmol) and benzene (25 mL) were mixed according to general method A to afford **10j** as a reddish orange oil (0.256 g, 57%) after 20 h. (R_f 0.48, 20% EtOAc/Hex) **^{1}H NMR** (300 MHz, $CDCl_3$) δ ppm 8.10 - 8.18 (m, 1H), 7.76 (d, *J* = 11.73 Hz, 1H), 7.45 - 7.51 (m, 1H), 7.34 - 7.42 (m, 2H), 7.23 - 7.34 (m, 5H), 7.12 (d, *J* = 15.39 Hz, 1H), 6.82 - 6.95 (m, 1H), 6.42 (s, 1H), 3.74 (s, 3H), 2.54 (d, *J* = 0.99 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ ppm 165.1, 164.8, 145.4, 145.0, 136.7, 136.6, 135.1, 130.0, 129.7, 129.7, 129.0, 128.8, 128.5, 127.8, 123.9, 123.7, 122.2, 119.8, 115.3, 110.4, 52.6, 16.7. **IR**: 3030.0 (w), 2949.9 (w), 1721.0 (s), 1682.4 (s), 1614.7 (m), 1590.8 (m), 1456.2 (s), 1435.3 (m), 1384.9 (s), 1308.3 (s), 1278.6 (s), 1237.6 (s), 1202.0 (w), 1077.2 (w), 993.8 (w), 836.1 (w), 750.7 (s), 691.5 (w) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 345.1365, Obs. 345.1383.



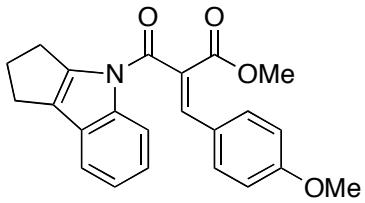
(Z)-10k

(*Z*)-Methyl 3-(4-methoxyphenyl)-2-(2-phenyl-1*H*-indole-1-carbonyl)acrylate (10k): Methyl 3-oxo-3-(2-phenyl-1*H*-indol-1-yl)propanoate (1.28 g, 4.36 mmol), 4-methoxybenzaldehyde (0.70 mL, 5.75 mmol), glacial acetic acid (0.131 g, 2.18 mmol), piperidine (50 µL , 0.506 mmol) and benzene (120 mL) were mixed according to general method A to afford **10k** as a dark brown solid (0.456 g, 25%) after 18 h. (R_f 0.37, 20% EtOAc/Hex) [**m.p.** 105-107 °C] **^{1}H NMR** (300 MHz, $CDCl_3$) δ ppm 8.63 (d, *J* = 8.17 Hz, 1H), 7.40 - 7.51 (m, 2H), 7.29 - 7.38 (m, 2H), 7.19 - 7.27 (m, 2H), 7.16 (s, 1H), 7.00 - 7.13 (m, 4H), 6.54 - 6.61 (m, 2H), 6.34 (s, 1H), 3.73 (s, 3H), 3.71 (s, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ ppm 166.8, 164.4, 161.5, 143.1, 139.8, 138.2, 133.4, 131.6, 129.6, 128.6, 127.8, 125.4, 125.2, 125.0, 124.3, 120.2, 116.6, 114.0, 111.9, 55.3, 52.3. **IR**: 3065.3 (w), 2951.7 (w), 2939.1 (w), 1720.6 (s), 1682.4 (s), 1600.9 (s), 1511.8 (s), 1452.5 (s), 1385.8 (m), 1321.3 (m), 1290.4 (m), 1258.9 (s), 1203.7 (m), 1172.3 (s), 1123.0 (m), 1056.1 (w), 1027.6 (m), 917.2 (w), 831.4 (m), 763.9 (s), 751.0 (s), 700.6 (m) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 411.1471, Obs. 411.1480.



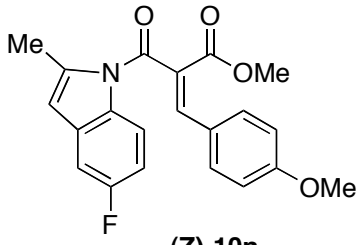
(Z)-10l

(*Z*)-Methyl 2-(2,3-dimethyl-1*H*-indole-1-carbonyl)-3-(4-methoxyphenyl)acrylate (10l): Methyl 3-(2,3-dimethyl-1*H*-indol-1-yl)-3-oxopropanoate (1.00 g, 4.08 mmol), 4-methoxybenzaldehyde (0.617 mL, 5.10 mmol), glacial acetic acid (0.112 g, 1.88 mmol), piperidine (0.0340 g, 0.407 mmol) and benzene (35 mL) were mixed according to general method A to afford **10l** as a yellow solid (1.080 g, 73%) after 18 h. (R_f 0.25, 20% EtOAc/Hex) [**m.p.** 94-96°C] **^{1}H NMR** (300 MHz, $CDCl_3$) δ ppm 8.63 (br. s, 1H), 7.87 (br. s., 1H), 7.33 - 7.48 (m, 3H), 7.28 (br. s., 2H), 6.73 (d, *J* = 8.50 Hz, 2H), 3.77 (s, 3H), 3.67 (d, *J* = 1.21 Hz, 3H), 2.25 - 2.53 (s, 3H), 2.15 (s, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ ppm 165.9, 165.2, 161.9, 142.5, 136.1, 131.8, 131.5, 126.6, 125.3, 124.3, 123.6, 117.9, 116.5, 114.6, 55.2, 52.4, 13.4, 8.6. **IR**: 3008.4 (w), 2933.3 (w), 2839.7 (w), 1721.5 (s), 1675.4 (s), 1601.6 (s), 1513.5 (s), 1458.5 (s), 1396.3 (m), 1306.8 (s), 1258.4 (s), 1203.4 (m), 1174.8 (s), 1133.5 (w), 1028.0 (w), 907.9 (w), 832.0 (w), 750.0 (s) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 363.1471, Obs. 363.1470.



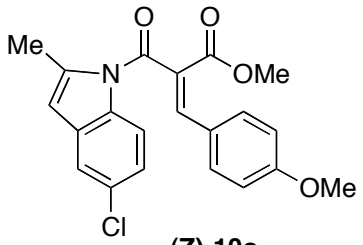
(Z)-10m

(*Z*)-Methyl 3-(4-methoxyphenyl)-2-(1,2,3,4-tetrahydrocyclopenta[*b*]indole-4-carbonyl)acrylate (10m): Methyl 3-(2,3-dihydrocyclopenta[*b*]indol-4(1*H*)-yl)-3-oxopropanoate (0.175 g, 0.681 mmol), 4-methoxybenzaldehyde (0.107 mL, 0.885 mmol), glacial acetic acid (0.0187 g, 0.313 mmol), piperidine (6.8 µL, 0.0680 mmol) and benzene (15 mL) were mixed according to general method A to afford **10m** as a white solid (0.0520 g, 20%) after 18 h. (R_f 0.40, 20% EtOAc/Hex) [**m.p.** 156-158 °C] **^{1}H NMR** (300 MHz, $CDCl_3$) δ ppm 8.64 - 8.72 (m, 1H), 7.82 (s, 1H), 7.24 - 7.44 (m, 5H), 6.75 - 6.82 (m, 2H), 3.80 (s, 3H), 3.75 (s, 3H), 2.60 - 2.80 (m, 4H), 2.25 - 2.50 (m, 2H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ ppm 165.5, 165.3, 161.8, 142.9, 141.7, 140.9, 132.0, 128.1, 127.4, 124.9, 124.5, 124.3, 124.2, 118.5, 117.7, 114.7, 55.3, 52.7, 28.9, 27.5, 23.8. **IR**: 2950.7 (w), 2857.5 (w), 1720.8 (m), 1681.7 (s), 1602.0 (s), 1513.5 (s), 1450.13 (m), 1392.8 (m), 1258.2 (s), 1173.02 (s), 1120.5 (w), 1103.5 (w), 1043.3 (w), 983.9 (w), 831.7 (w), 751.0 (m) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 375.1471, Obs. 375.1474.



(Z)-10n

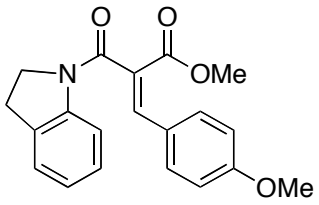
(*Z*)-Methyl 2-(5-fluoro-2-methyl-1*H*-indole-1-carbonyl)-3-(4-methoxyphenyl)acrylate (10n): Methyl 3-(5-fluoro-2-methyl-1*H*-indol-1-yl)-3-oxopropanoate (0.301 g, 1.21 mmol), 4-methoxybenzaldehyde (0.180 mL, 1.48 mmol), glacial acetic acid (0.0520 g, 0.873 mmol), piperidine (25 µL , 0.253 mmol) and benzene (30 mL) were mixed according to general method A to afford **10n** as a pale brick solid (0.382 g, 86%) after 18 h. (R_f 0.43, 30% EtOAc/Hex) [**m.p.** 95-97 °C] (*Temperature for the ^{1}H NMR and ^{13}C NMR = 60 °C)* **^{1}H NMR** (300 MHz, $CDCl_3$) δ ppm 8.25 (br s, 1H), 7.85 (s, 1H), 7.28 - 7.39 (m, 2H), 7.05 (dd, *J* = 2.57, 8.61 Hz, 1H), 6.95 (dt, *J* = 2.58, 9.12 Hz, 1H), 6.70 - 6.79 (m, 2H), 6.27 (s, 1H), 3.78 (s, 3H), 3.71 (s, 3H), 2.46 (s, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ ppm 166.0, 165.0, 162.1, 161.5, 158.3, 143.0, 133.3, 131.8, 131.1 and 131.0 (doublet), 125.8, 124.9, 116.9, 114.6, 111.5, 111.2, 110.3, 105.5, 105.2, 55.2, 52.5, 16.5. **IR**: 2948.8 (w), 2903.6 (w), 1720.7 (m), 1685.4 (m), 1602.5 (s), 1513.7 (s), 1472.7 (m), 1448.5 (m), 1389.2 (m), 1301.5 (m), 1274.6 (s), 1260.5 (s), 1176.5 (s), 995.0 (w), 957.3 (w), 832.8 (w), 764.3 (s), 750.0 (s) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 367.1220, Obs. 367.1226.



(Z)-10o

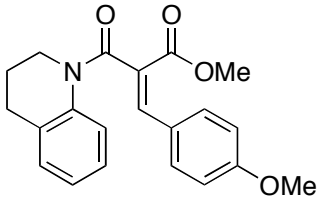
(*Z*)-Methyl 2-(5-chloro-2-methyl-1*H*-indole-1-carbonyl)-3-(4-methoxyphenyl)acrylate (10o): Methyl 3-(5-chloro-2-methyl-1*H*-indol-1-yl)-3-oxopropanoate (0.565 g, 2.14 mmol), 4-methoxybenzaldehyde (0.337 mL, 2.77 mmol), glacial acetic acid (0.0589 g, 0.982 mmol), piperidine (21 µL , 0.214 mmol) and benzene (30 mL) were mixed according to general method A to afford **10o** as an off-white solid (0.300 g, 37%) after 20 h. (R_f 0.40, 20% EtOAc/Hex) [**m.p.** 111-113 °C] (*Temperature for the ^{1}H NMR and ^{13}C NMR = 60 °C)* **^{1}H NMR** (300 MHz, $CDCl_3$) δ ppm 8.20 (d, *J* = 8.54 Hz, 1H), 7.86 (s, 1H), 7.30 - 7.40 (m, 3H), 7.20 (dd, *J* = 2.11, 8.85 Hz, 1H), 6.72 - 6.79 (m, 2H), 6.26 (s, 1H), 3.78 (s, 3H), 3.72 (s, 3H), 2.47 (s, 3H). **^{13}C**

NMR (75 MHz, $CDCl_3$) δ ppm 166.1, 164.9, 162.1, 143.2, 138.0, 135.4, 131.8, 131.2, 129.5, 125.8, 124.9, 124.1, 119.3, 116.7, 114.7, 109.8, 55.2, 52.5, 16.5. **IR**: 2952.3 (w), 2839.8 (w), 2360.1 (m), 2342.4 (m), 1718.7 (s), 1683.4 (s), 1597.9 (s), 1512.5 (s), 1442.9 (s), 1385.6 (s), 1345.9 (w), 1294.5 (m), 1255.6 (s), 1201.3 (m), 1171.5 (s), 1124.2 (w), 1072.5 (w), 1021.4 (w), 995.1 (w), 914.0 (w), 829.9 (m), 800.5 (w), 732.0 (w), 668.6 (w) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 383.0924, Obs. 383.0922.



12a

Methyl 2-(indoline-1-carbonyl)-3-(4-methoxyphenyl)acrylate (12a): Methyl 3-(indolin-1-yl)-3-oxopropanoate (0.350 g, 1.60 mmol), 4-methoxybenzaldehyde (0.252 mL, 2.08 mmol), glacial acetic acid (0.0440 g, 0.734 mmol), piperidine (16 µL, 0.160 mmol) and benzene (30 mL) were mixed according to general method A to afford **12a** as an orange oil (0.390 g, 72%) after 20 h. (R_f 0.35, 20% EtOAc/Hex) (*Diastereomers!)* **^{1}H NMR** (300 MHz, $CDCl_3$) δ ppm 8.41 (d, *J* = 8.03 Hz, 1.00), 7.82 (s, 0.20), 7.70 (s, 1.14), 7.43 - 7.54 (m, 2.47), 7.28 - 7.33 (m, 0.81), 7.16 - 7.22 (m, 1.27), 7.01 - 7.13 (m, 1.44), 6.80 - 6.88 (m, 2.53), 4.36 - 4.50 (m, 0.18), 4.18 - 4.31 (m, 0.20), 3.94 (br.s., 1.16), 3.83 (s, 3.16), 3.61 - 3.80 (m, 3.87), 3.71 - 3.76 (m, 1.78), 2.92 - 3.22 (m, 2.49). **^{13}C NMR** (101 MHz, $CDCl_3$) δ ppm 165.4, 165.0, 161.9, 142.6, 142.4, 140.6, 132.0, 131.8, 127.7, 126.0, 125.8, 124.6, 124.4, 117.5, 114.8, 114.5, 55.3, 52.4, 48.4, 28.0. **IR**: 3004.8 (w), 2951.7 (w), 2839.7 (w), 1716.9 (s), 1645.7 (s), 1598.9 (s), 1512.6 (s), 1482.3 (s), 1413.3 (m), 1255.3 (s), 1174.5 (s), 1126.4 (m), 1057.9 (m), 1027.4 (m), 832.3 (m), 755.8 (s), 668.5 (m) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 337.1314, Obs. 337.1319.

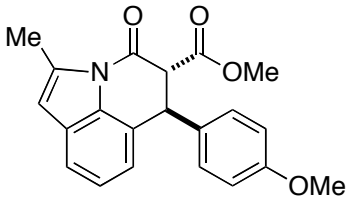


(Z)-12b

(*Z*)-Methyl 3-(4-methoxyphenyl)-2-(1,2,3,4-tetrahydroquinoline-1-carbonyl)acrylate (12b): Methyl 3-(3,4-dihydroquinolin-1(2*H*)-yl)-3-oxopropanoate (0.3500 g, 1.5010 mmol), 4-methoxybenzaldehyde (0.2370 mL, 1.9510 mmol), glacial acetic acid (0.0414 g, 0.6900 mmol), piperidine (14.80 µL, 0.1501 mmol) and benzene (30 mL) were mixed according to general method A to afford **12b** as a orange oil (0.4230 g, *Crude* = 80%) after 15 h. (R_f 0.35, 20% EtOAc/Hex) **HRMS (ESI)** M/Z+ Calc. 351.1471, Obs. 351.1499.

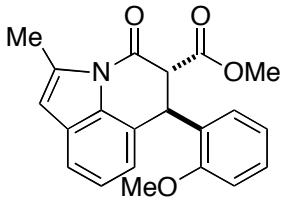
C. $In(OTf)_3$-Catalyzed Cyclizations

General Procedure: To a mixture of $In(OTf)_3$ (0.10-0.15 equiv.) in 1,2-DCE (or toluene) heated to a reflux, dissolved **10 (or 12)** (1.0 equiv) was syringed into the reaction vessel. The reaction was monitored by TLC and quenched with water. The phases were separated, and the product was extracted from the aqueous phase with DCM. The combined organic layers were washed with brine, dried over Mg_2SO_4, filtered, and concentrated for column chromatography using silica gel.



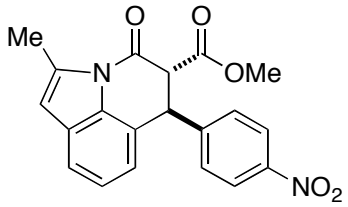
11a

Methyl 6-(4-methoxyphenyl)-2-methyl-4-oxo-5,6-dihydro-4*H*-pyrrolo[3,2,1-*ij*]quinoline-5-carboxylate (11a): Methyl 3-(4-methoxyphenyl)-2-(2-methyl-1*H*-indole-1-carbonyl)acrylate (0.258 g, 0.739 mmol), $In(OTf)_3$ (0.0428 g, 0.0760 mmol) and 1,2-DCE (13 mL) were combined according to the general procedure to afford **11a** as a brown solid (0.161 g, 63%) after 3 h. (R_f 0.35, 20% EtOAc/Hex) [**m.p.** 122-124 °C] *Diastereomeric ratio*: (50:1). **^{1}H NMR** (300 MHz, $CDCl_3$) δ ppm 7.31 - 7.37 (m, 1H), 7.14 - 7.20 (m, 2H), 7.08 - 7.13 (m, 1H), 6.84 - 6.92 (m, 2H), 6.71 (d, *J* = 7.48 Hz, 1H), 6.41 (d, *J* = 1.25 Hz, 1H), 4.96 (d, *J* = 10.85 Hz, 1H), 4.19 (d, *J* = 10.88 Hz, 1H), 3.81 (s, 3H), 3.68 (s, 3H), 2.71 (d, *J* = 1.03 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ ppm 169.0, 164.0, 159.0, 137.2, 134.9, 130.9, 129.6, 127.4, 124.0, 122.7, 121.0, 118.4, 114.3, 109.4, 58.8, 55.2, 52.6, 45.3, 15.2. **IR**: 2954.7 (w), 2922.5 (w), 2850.5 (w), 1749.6 (s), 1709.3 (s), 1611.6 (w), 1513.5 (s), 1443.5 (s), 1381.8 (s), 1340.9 (s), 1252.6 (s), 1178.9 (w), 1153.2 (m), 1032.8 (m), 818.6 (m), 764.7 (m), 749.1 (s) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 349.1314, Obs. 349.1310.



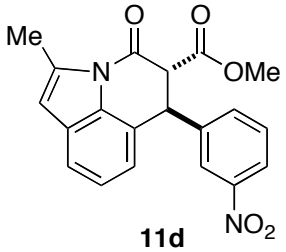
11b

Methyl 6-(2-methoxyphenyl)-2-methyl-4-oxo-5,6-dihydro-4H-pyrrolo[3,2,1-ij]quinoline-5-carboxylate (11b): Methyl 3-(2-methoxyphenyl)-2-(2-methyl-1H-indole-1-carbonyl)acrylate (0.060 g, 0.172 mmol), $In(OTf)_3$ (0.0145 g, 0.0250 mmol) and toluene (4 mL) were combined according to the general procedure to afford **11b** as a clear oil (0.0522 g, 87% for combined *cis* and *trans* isomers) after 3 h. (R_f 0.35, 20% EtOAc/Hex) *Diastereomeric ratio*: (3.7:1). (*trans*-Diastereomer chemical shifts reported) **^{1}H NMR** (400 MHz, $CDCl_3$) δ ppm 7.33 (d, *J* = 7.72 Hz, 1H), 7.23 - 7.29 (m, 1H), 7.11 (t, *J* = 7.62 Hz, 1H), 6.83 - 6.94 (m, 3H), 6.76 (d, *J* = 7.47 Hz, 1H), 6.40 (d, *J* = 1.13 Hz, 1H), 5.26 (d, *J* = 7.72 Hz, 1H), 4.41 (d, *J* = 7.65 Hz, 1H), 3.75 (s, 3H), 3.67 (s, 3H), 2.72 (d, *J* = 0.94 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ ppm 169.4, 164.4, 157.3, 137.2, 135.1, 129.7, 129.0, 127.7, 127.4, 124.0, 121.8, 120.9, 120.6, 118.1, 111.2, 109.2, 56.2, 55.4, 52.7, 41.8, 15.2. **IR**: 3065.0 (w), 3032.3 (w), 3003.1 (m), 2954.0 (m), 2839.0 (m), 1745.7 (s), 1707.2 (s), 1627.5 (w), 1600.6 (w), 1586.6 (w), 1573.3 (w), 1493.3 (m), 1443.7 (m), 1380.9 (m), 1338.7 (m), 1287.0 (m), 1210.6 (s), 1154.0 (m), 1119.0 (m), 1047.7 (w), 1026.1 (m), 967.6 (m), 911.8 (m), 820.2 (w), 748.8 (m), 732.9 (m) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 349.1314, Obs. 349.1328.



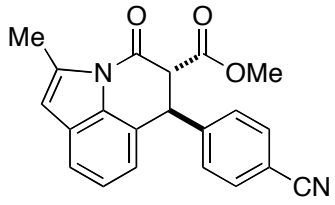
11c

***trans*-Methyl 2-methyl-6-(4-nitrophenyl)-4-oxo-5,6-dihydro-4*H*-pyrrolo[3,2,1-*ij*]quinoline-5-carboxylate (11c):** Methyl 2-(2-methyl-1*H*-indole-1-carbonyl)-3-(4-nitrophenyl)acrylate (0.070 g, 0.1922 mmol), $In(OTf)_3$ (0.0162 g, 0.0288 mmol) and toluene (4 mL) were combined according to the general procedure to afford **11c** as an orange oil (0.0548 g, 78% for combined *cis* and *trans* isomers) after 14 h. (R_f 0.40, 20% EtOAc/Hex) *Diastereomeric ratio*: (2.4:1) (*trans*-Diastereomer chemical shifts reported) **^{1}H NMR** (300 MHz, $CDCl_3$) δ ppm 8.19 - 8.28 (m, 2H), 7.42 - 7.50 (m, 2H), 7.39 (d, *J* = 7.77 Hz, 1H), 7.15 (t, *J* = 7.64 Hz, 1H), 6.64 (d, *J* = 7.48 Hz, 1H), 6.45 (d, *J* = 1.21 Hz, 1H), 5.16 (d, *J* = 10.44 Hz, 1H), 4.21 (d, *J* = 10.44 Hz, 1H), 3.69 (s, 3H), 2.71 (d, *J* = 1.17 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ ppm 168.4, 163.0, 147.6, 146.8, 137.6, 129.6, 127.8, 124.3, 124.2, 120.8, 120.6, 119.2, 109.6, 58.0, 53.0, 45.8, 15.2. **IR**: 3066.3 (w), 2955.2 (w), 2923.9 (w), 2850.9 (w), 1746.1 (s), 1709.3 (s), 1606.7 (w), 1519.4 (s), 1444.4 (s), 1381.0 (s), 1345.7 (s), 1285.8 (m), 1268.1 (m), 1211.2 (w), 1154.5 (s), 1109.6 (w), 1048.0 (w), 1008.1(w), 967.4 (w), 863.3 (m), 819.5 (m), 748.9 (s), 735.0 (s), 706.7 (m), 611.3 (w) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 364.1059, Obs. 364.1048.



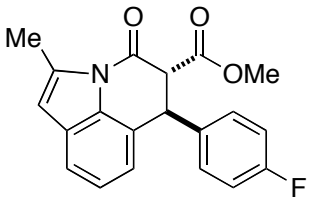
11d

Methyl 2-methyl-6-(3-nitrophenyl)-4-oxo-5,6-dihydro-4*H*-pyrrolo[3,2,1-*ij*]quinoline-5-carboxylate (11d): Methyl 2-(2-methyl-1*H*-indole-1-carbonyl)-3-(3-nitrophenyl)acrylate (0.100 g, 0.275 mmol), $In(OTf)_3$ (0.0231 g, 0.0411 mmol) and 1,2-DCE (4 mL) were combined according to the general procedure to afford **11d** as an off-white solid (0.0861 g, 86% for combined *cis* and *trans* isomers) after 13 h. (R_f 0.35, 20% EtOAc/Hex) [**m.p.** 106-108 °C] *Diastereomeric ratio*: (2.2:1). (*trans*-Diastereomer chemical shifts reported) **^{1}H NMR** (300 MHz, $CDCl_3$) δ ppm 8.19 - 8.28 (m, 2H), 7.42 - 7.50 (m, 2H), 7.39 (d, *J* = 7.77 Hz, 1H), 7.15 (t, *J* = 7.64 Hz, 1H), 6.64 (d, *J* = 7.48 Hz, 1H), 6.43 - 6.46 (m, 1H), 5.16 (d, *J* = 10.44 Hz, 1H), 4.21 (d, *J* = 10.44 Hz, 1H), 3.69 (s, 3H), 2.71 (d, *J* = 1.17 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ ppm 168.4, 163.0, 147.6, 146.8, 137.6, 129.6, 127.8, 125.0, 124.3, 124.2, 120.8, 120.6, 119.2, 109.6, 58.0, 53.0, 45.8, 15.2. **IR**: 3066.6 (w), 2955.3 (w), 2923.4 (w), 1746.0 (s), 1708.6 (s), 1530.3 (s), 1444.0 (s), 1380.6 (s), 1346.0 (s), 1286.5 (m), 1267.1 (m), 1154.2 (s), 1052.1 (w), 1003.0 (w), 966.4 (w), 904.1 (w), 817.3 (m), 738.9 (s), 709.5 (m), 613.4 (m) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 364.1059, Obs. 364.1055.



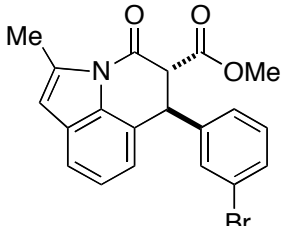
11e

Methyl 6-(4-cyanophenyl)-2-methyl-4-oxo-5,6-dihydro-4*H*-pyrrolo[3,2,1-*ij*]quinoline-5-carboxylate (11e): Methyl 3-(4-cyanophenyl)-2-(2-methyl-1*H*-indole-1-carbonyl)acrylate (0.0900 g, 0.263 mmol), $In(OTf)_3$ (0.0220 g, 0.0392 mmol) and 1,2-DCE (4 mL) were combined according to the general procedure to afford **11e** as a pale orange solid (0.0704 g, 78% for combined *cis* and *trans* isomers) after 14 h. (R_f 0.35, 20% EtOAc/Hex) [**m.p.** 155-157 °C] *Diastereomeric ratio*: (1.85:1). (*trans*-diastereomer chemical shifts reported) **^{1}H NMR** (300 MHz, $CDCl_3$) δ ppm 7.66 (d, *J* = 8.25 Hz, 2H), 7.38 (d, *J* = 7.88 Hz, 3H), 7.14 (t, *J* = 7.62 Hz, 1H), 6.64 (d, *J* = 7.44 Hz, 1H), 6.44 (d, *J* = 0.92 Hz, 1H), 5.09 (d, *J* = 10.30 Hz, 1H), 4.18 (d, *J* = 10.30 Hz, 1H), 3.69 (s, 3H), 2.71 (s, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ ppm 168.4, 163.1, 144.8, 137.5, 132.8, 129.4, 127.7, 124.3, 120.8, 120.6, 119.1, 118.4, 112.0, 109.6, 58.0, 52.9, 46.0, 15.2. **IR**: 2955.3 (w), 2922.9 (w), 2229.2 (w), 1748.4 (s), 1712.4 (s), 1532.6 (w), 1445.2 (s), 1383.0 (s), 1344.2 (m), 1275.0 (s), 1262.2 (s), 1156.3 (m), 819.0 (w), 749.7 (s) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 344.1161, Obs. 344.1172.



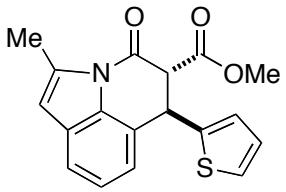
11f

Methyl 6-(4-fluorophenyl)-2-methyl-4-oxo-5,6-dihydro-4*H*-pyrrolo[3,2,1-*ij*]quinoline-5-carboxylate (11f): Methyl 3-(4-fluorophenyl)-2-(2-methyl-1*H*-indole-1-carbonyl)acrylate (0.0760 g, 0.225 mmol), $In(OTf)_3$ (0.0188 g, 0.0330 mmol) and 1,2-DCE (8 mL) were combined according to the general procedure to afford **11f** as a yellow solid (0.0716 g, 94% for combined *cis* and *trans* isomers) after 1 h. (R_f 0.68, 30% EtOAc/Hex) [**m.p.** 153-155 °C] *Diastereomeric ratio*: (2.6:1) (*trans*-Diastereomer chemical shifts reported) **^{1}H NMR** (300 MHz, $CDCl_3$) δ ppm 7.36 (d, *J* = 7.77 Hz, 1H), 7.19 - 7.25 (m, 2H), 7.13 (t, *J* = 7.62 Hz, 1H), 7.00 - 7.09 (m, 2H), 6.68 (d, *J* = 7.44 Hz, 1H), 6.42 (d, *J* = 1.17 Hz, 1H), 5.01 (d, *J* = 10.85 Hz, 1H), 4.18 (d, *J* = 10.85 Hz, 1H), 3.68 (s, 3H), 2.71 (s, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ ppm 168.8, 163.7, 137.3, 134.8, 130.2 and 130.1 (doublet), 127.5, 124.2, 122.1, 120.9, 118.7, 116.1, 115.8, 109.5, 77.2, 58.7, 52.7, 45.4, 15.2. **IR**: 3058.4 (w), 2954.5 (w), 2923.0 (w), 1746.8 (s), 1708.7 (s), 1605.3 (w), 1509.8 (s), 1443.8 (s), 1380.9 (s), 1340.0 (s), 1267.9 (m), 1224.3 (s), 1159.1 (s), 1097.6 (w), 1051.5 (w), 1010.0 (w), 967.1 (w), 818.5 (m), 748.7 (s) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 337.1114, Obs. 337.1115.



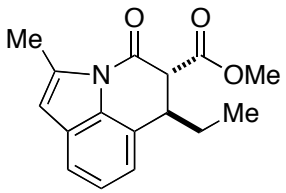
11g

Methyl 6-(3-bromophenyl)-2-methyl-4-oxo-5,6-dihydro-4*H*-pyrrolo[3,2,1-*ij*]quinoline-5-carboxylate (11g): Methyl 3-(3-bromophenyl)-2-(2-methyl-1*H*-indole-1-carbonyl)acrylate (0.100 g, 0.252 mmol), $In(OTf)_3$ (0.0213 g, 0.0377 mmol) and 1,2-DCE (4 mL) were combined according to the general procedure to afford **11g** as a pale yellow solid (0.0614 g, 61%) after 14 h. (R_f 0.40, 20% EtOAc/Hex) [**m.p.** 123-125 °C] *Diastereomeric ratio*: (8.3:1). (*trans*-Diastereomer chemical shifts reported) **^{1}H NMR** (300 MHz, $CDCl_3$) δ ppm 7.41 - 7.49 (m, 2H), 7.33 - 7.39 (m, 1H), 7.10 - 7.27 (m, 3H), 6.69 (d, *J* = 7.48 Hz, 1H), 6.43 (d, *J* = 0.92 Hz, 1H), 4.98 (d, *J* = 10.59 Hz, 1H), 4.19 (d, *J* = 10.63 Hz, 1H), 3.70 (s, 3H), 2.71 (s, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ ppm 168.7, 163.4, 141.6, 137.3, 134.8, 131.5, 131.1, 130.5, 127.5, 127.2, 124.2, 122.9, 121.4, 120.9, 118.8, 109.5, 58.3, 52.8, 45.7, 15.2. **IR**: 2961.7 (w), 2921.4 (w), 1750.2 (s), 1711.7 (s), 1570.7 (w), 1474.8 (w), 1444.8 (s), 1381.9 (s), 1341.5 (s), 1275.8 (s), 1261.6 (m), 1155.6 (m), 764.3 (s), 749.7 (s) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 397.0314, Obs. 397.0315.



11h

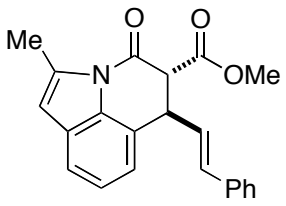
***trans*-Methyl 2-methyl-4-oxo-6-(thiophen-2-yl)-5,6-dihydro-4*H*-pyrrolo[3,2,1-*ij*]quinoline-5-carboxylate (11h):** Methyl 2-(2-methyl-1*H*-indole-1-carbonyl)-3-(thiophen-2-yl)acrylate (0.0900 g, 0.277 mmol), $In(OTf)_3$ (0.0233 g, 0.0415 mmol) and 1,2-DCE (4 mL) were combined according to the general procedure to afford **11h** as an off-white solid (0.0459 g, 51%) after 14 h. (R_f 0.40, 20% EtOAc/Hex) [**m.p.** 153-155 °C] (*Single Diastereomer*) **^{1}H NMR** (300 MHz, $CDCl_3$) δ ppm 7.37 (d, *J* = 7.70 Hz, 1H), 7.23 - 7.28 (m, 1H), 7.17 (t, *J* = 7.62 Hz, 1H), 6.91 - 6.99 (m, 3H), 6.42 (s, 1H), 5.32 (d, *J* = 9.45 Hz, 1H), 4.25 (d, *J* = 9.45 Hz, 1H), 3.72 (s, 3H), 2.70 (s, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ ppm 169.0, 163.8, 142.6, 137.8, 134.8, 128.0, 127.4, 127.0, 125.7, 124.6, 122.2, 121.3, 119.4, 109.9, 59.7, 53.3, 41.7, 15.6. **IR**: 2961.2 (w), 2927.0 (w), 1751.0 (s), 1712.1 (s), 1445.7 (s), 1382.5 (s), 1341.1 (s), 1275.5 (s), 1267.3 (m), 1156.4 (m), 1042.5 (w), 1004.0 (w), 748.9 (s), 702.5 (w) cm^{-1}**. HRMS (ESI)** M/Z+ Calc. 325.0773, Obs. 325.0754.



11i

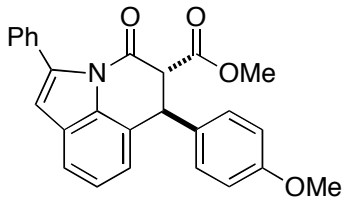
Methyl 6-ethyl-2-methyl-4-oxo-5,6-dihydro-4*H*-pyrrolo[3,2,1-*ij*]quinoline-5-carboxylate (11i): Methyl 2-(2-methyl-1*H*-indole-1-carbonyl)pent-2-enoate (0.090 g, 0.332 mmol), $In(OTf)_3$ (0.0559 g, 0.0995 mmol) and toluene (5 mL) were combined according to the general procedure to afford **11i** as a clear oil (0.0754 g, 84%) after 12 h. (R_f 0.40, 20% EtOAc/Hex) [**m.p.** 108-110 °C] *Diastereomeric ratio*: (25:1). (*trans*-Diastereomer chemical shifts reported) **^{1}H NMR** (300 MHz, $CDCl_3$) δ ppm 7.33 (d, *J* = 7.48 Hz, 1H), 7.18 (t, *J* = 7.55 Hz, 1H), 7.06 (d, *J* = 6.00 Hz, 1H), 6.35 - 6.39 (m, 1H), 3.86 (d, *J* = 4.69 Hz, 1H), 3.61 - 3.71 (m, 4H), 2.66 - 2.73 (m, 3H), 1.78 - 1.95 (m, 1H), 1.61 - 1.77 (m, 1H), 0.95 (t, *J* = 7.40 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ ppm 169.6, 164.3, 136.9, 134.6, 127.5, 123.7, 121.7, 120.6, 118.1, 109.2, 55.8, 52.8, 41.5, 27.2, 15.1, 10.6. **IR**: 2963.1 (w), 2927.6 (w) 1743.0 (w), 1715.0 (s), 1629.2 (s),1573.2 (w), 1447.6 (w),

1381.4 (s), 1328.6 (m), 1274.9 (m), 1259.8 (m), 1194.6 (w), 1160.3 (w), 821.1 (w), 763.4 (m), 750.0 (s) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 271.1208, Obs. 271.1208.



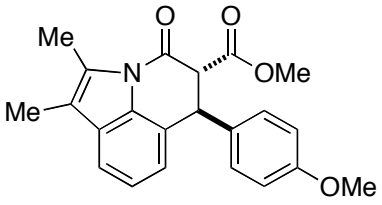
11j

(*E*)-methyl 2-methyl-4-oxo-6-styryl-5,6-dihydro-4*H*-pyrrolo[3,2,1-*ij*]quinoline-5-carboxylate (11j): Methyl 2-(2-methyl-1*H*-indole-1-carbonyl)-5-phenylpenta-2,4-dienoate (0.0700 g, 0.203 mmol), $In(OTf)_3$ (0.0341 g, 0.0608 mmol) and toluene (4 mL) were combined according to the general procedure to afford **11j** as a pale yellow solid (0.0457 g, 65%) after 14 h. (R_f 0.35, 20% EtOAc/Hex) [**m.p.** 98-100 °C] *Diastereomeric ratio*: (20:1). (*trans*-Diastereomer chemical shifts reported) **^{1}H NMR** (300 MHz, $CDCl_3$) δ ppm 7.26 - 7.43 (m, 6H), 7.21 (t, *J* = 7.57 Hz, 1H), 7.08 (d, *J* = 7.40 Hz, 1H), 6.63 (d, *J* = 15.68 Hz, 1H), 6.40 (d, *J* = 1.14 Hz, 1H), 6.24 (dd, *J* = 8.65, 15.68 Hz, 1H), 4.58 (t, *J* = 9.44 Hz, 1H), 4.00 (d, *J* = 10.22 Hz, 1H), 3.78 (s, 3H), 2.70 (d, *J* = 1.10 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ ppm 169.0, 164.0, 137.2, 136.2, 134.6, 134.5, 128.6, 128.0, 127.6, 126.5, 126.4, 124.1, 121.0, 120.5, 118.8, 109.3, 56.7, 52.8, 43.9, 15.2. **IR**: 3026.9 (w), 2952.8 (w), 2922.4 (w), 1749.6 (s), 1709.8 (s), 1444.3 (s), 1380.9 (s), 1340.2 (m), 1276.0 (m), 1260.8 (m), 1200.1 (w), 1151.3 (m), 968.1 (w), 813.1 (w), 749.0 (s), 695.0 (m)cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 345.1365, Obs. 345.1360.



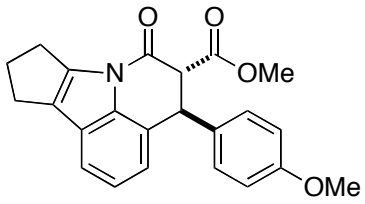
11k

Methyl 6-(4-methoxyphenyl)-4-oxo-2-phenyl-5,6-dihydro-4*H*-pyrrolo[3,2,1-*ij*]quinoline-5-carboxylate (11k): Methyl 3-(4-methoxyphenyl)-2-(2-phenyl-1*H*-indole-1-carbonyl)acrylate (0.160 g, 0.390 mmol), $In(OTf)_3$ (0.0223 g, 0.0400 mmol) and 1,2-DCE (13 mL) were combined according to the general procedure to afford **11k** as a reddish orange solid (0.155 g, 97%) after 3 h. (R_f 0.33, 20% EtOAc/Hex) [**m.p.** 108-110 °C] *Diastereomeric ratio*: (17.3:1). (*trans*-Diastereomer chemical shifts reported) **^{1}H NMR** (300 MHz, $CDCl_3$) δ ppm 8.16 - 8.22 (m, 1H), 7.71 - 7.76 (m, 1H), 7.16 - 7.45 (m, 7H), 7.02 - 7.09 (m, 2H), 6.71 - 6.77 (m, 2H), 5.19 (d, *J* = 4.40 Hz, 1H), 4.05 (d, *J* = 4.43 Hz, 1H), 3.86 (s, 3H), 3.73 (s, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ ppm 168.0, 164.9, 158.9, 139.4, 134.4, 132.0, 131.7, 130.5, 128.4, 128.4, 128.4, 127.0, 124.9, 124.5, 120.2, 116.7, 114.3, 114.1, 63.0, 55.2, 53.3, 42.7. **IR**: 3056.9 (w), 2953.2 (w), 2837.9 (w), 1730.5 (s), 1610.2 (s), 1511.9 (s), 1454.6 (s), 1392.4 (s), 1345.1 (m), 1305.2 (m), 1246.7 (s), 1145.4 (s), 1103.0 (w), 1029.6 (s), 830.8 (m), 748.6 (s), 699.8 (s), 628.6 (m) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 411.1471, Obs. 411.1470.



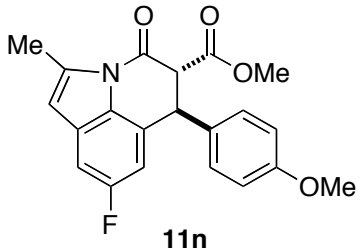
11l

Methyl 6-(4-methoxyphenyl)-1,2-dimethyl-4-oxo-5,6-dihydro-4*H*-pyrrolo[3,2,1-*ij*]quinoline-5-carboxylate (11l): Methyl 2-(2,3-dimethyl-1*H*-indole-1-carbonyl)-3-(4-methoxyphenyl)acrylate (0.0900 g, 0.2476 mmol), $In(OTf)_3$ (0.0208 g, 0.0371 mmol) and DCE (5 mL) were combined according to the general procedure to afford **11l** as a clear oil (0.7740 g, 86%) after 4 h. R_f 0.40 (20% EtOAc/Hex). *Diastereomeric ratio*: (Single Diastereomer observed) **^{1}H NMR** (400 MHz, $CDCl_3$) δ ppm 7.31 (d, *J* = 7.72 Hz, 1H), 7.12 - 7.19 (m, 3H), 6.85 - 6.89 (m, 2H), 6.72 (d, *J* = 7.47 Hz, 1H), 4.94 (d, *J* = 10.60 Hz, 1H), 4.17 (d, *J* = 10.60 Hz, 1H), 3.81 (s, 3H), 3.68 (s, 3H), 2.64 (d, *J* = 0.82 Hz, 3H), 2.22 (d, *J* = 0.88 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ ppm 169.2, 163.6, 159.0, 134.0, 132.3, 131.2, 129.5, 129.1, 123.9, 122.5, 121.1, 116.9, 116.7, 114.3, 58.9, 55.2, 52.6, 45.2, 12.4, 8.6. **IR**: 3035.9 (w), 2999.5 (w), 2953.5 (m), 2924.2 (m), 2837.8 (m), 1747.0 (s), 1700.9 (s), 1627.9 (w), 1610.8 (m), 1585.2 (w), 1512.4 (s), 1452.4 (s), 1378.4 (s), 1353.9 (s), 1338.2 (s), 1286.7 (m), 1250.0 (s), 1211.6 (m), 1178.1 (m), 1155.5 (s), 1136.1 (w), 1112.2 (w), 1030.9 (m), 980.7 (w), 912.6 (w), 850.7 (w), 822.5 (w), 792.4 (w), 768.1 (w), 746.4 (m), 731.9 (m), 610.7 (w) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 363.1471, Obs. 363.1465.



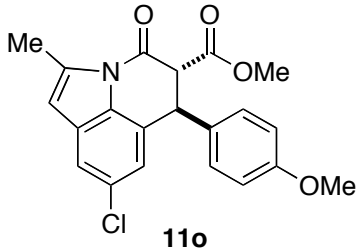
11m

***trans*-Methyl 4-(4-methoxyphenyl)-6-oxo-4,5,6,8,9,10-hexahydrocyclopenta[4,5]pyrrolo[3,2,1-*ij*]quinoline-5-carboxylate (11m):** Methyl 3-(4-methoxyphenyl)-2-(1,2,3,4-tetrahydrocyclopenta[*b*]indole-4-carbonyl)acrylate (0.0450 g, 0.120 mmol), $In(OTf)_3$ (0.0101 g, 0.0178 mmol) and toluene (3 mL) were combined according to the general procedure to afford **11m** as a white solid (0.0369 g, 82%) after 12 h. (R_f 0.40, 20% EtOAc/Hex) [**m.p.** 140-142 °C] (*Single Diastereomer observed*) **^{1}H NMR** (300 MHz, $CDCl_3$) δ ppm 7.39 - 7.47 (m, 2H), 7.13 - 7.29 (m, 2H), 7.03 - 7.09 (m, 1H), 6.76 - 6.82 (m, 2H), 3.91 (s, 1H), 3.89 (d, *J* = 0.40 Hz, 3H), 3.75 (s, 3H), 3.74 (s, 1H), 2.49 (td, *J* = 5.46, 13.07 Hz, 1H), 2.29 - 2.41 (m, 1H), 2.03 - 2.27 (m, 2H), 1.75 - 1.87 (m, 2H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ ppm 170.4, 169.2, 160.6, 147.1, 141.0, 140.0, 133.9, 128.1, 126.3, 125.9, 123.3, 117.4, 114.6, 109.6, 91.5, 67.9, 59.9, 55.5, 53.0, 51.7, 37.7, 36.3, 26.3. **IR**: 3059.4 (w), 2953.0 (w), 2867.2 (w), 1737.7 (s), 1708.1 (s), 1601.1 (m), 1579.5 (w), 1487.4 (m), 1474.2 (m), 1480.2 (m), 1352.9 (m), 1331.6 (m), 1304.1 (m), 1272.9 (s), 1227.6 (s), 1174.0 (m), 1156.2 (m), 1111.4 (w), 1096.0 (w), 1029.9 (s), 863.2 (w), 844.6 (w), 821.6 (w), 752.2 (s), 734.8 (s), 712.7 (m) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 375.1471, Obs. 375.1476.



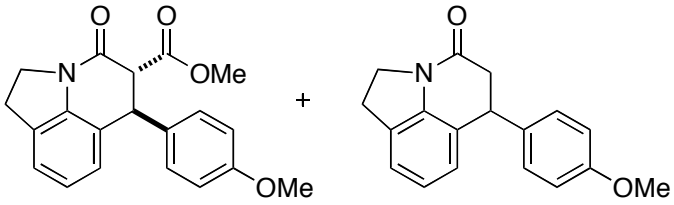
11n

***trans*-Methyl 8-fluoro-6-(4-methoxyphenyl)-2-methyl-4-oxo-5,6-dihydro-4*H*-pyrrolo[3,2,1-*ij*]quinoline-5-carboxylate (11n):** Methyl 2-(5-fluoro-2-methyl-1*H*-indole-1-carbonyl)-3-(4-methoxyphenyl)acrylate (0.0750 g, 0.204 mmol), $In(OTf)_3$ (0.0180 g, 0.0320 mmol) and 1,2-DCE (7 mL) were combined according to the general procedure to afford **11n** as a yellow solid (0.660 g, 88%) after 12 h. (R_f 0.40, 20% EtOAc/Hex) [**m.p.** 106-108 °C] *(Single Diastereomer Observed)* **^{1}H NMR** (400 MHz, $CDCl_3$) δ ppm 7.13 - 7.18 (m, 2H), 7.01 (ddd, *J* = 0.63, 2.21, 8.96 Hz, 1H), 6.86 - 6.91 (m, 2H), 6.43 - 6.48 (m, 1H), 6.37 - 6.39 (m, 1H), 4.92 (d, *J* = 10.79 Hz, 1H), 4.17 (d, *J* = 10.85 Hz, 1H), 3.81 (s, 3H), 3.68 (s, 3H), 2.70 (d, *J* = 1.19 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ ppm 168.8, 163.7, 161.7, 159.3, 159.2, 138.7, 131.1, 130.3, 129.5, 128.1 and 128.0 (doublet), 124.0 and 123.9 (doublet), 114.4, 109.2, 109.2, 109.1, 108.9, 104.7, 104.4, 58.5, 55.2, 52.7, 45.3, 15.2. **IR**: 3001.9 (w), 2954.8 (w), 2838.9 (w), 1747.3 (s), 1709.5 (s), 1632.7 (m), 1610.7 (m), 1513.3 (s), 1479.6 (s), 1435.4 (s), 1381.4 (s), 1327.8 (m), 1254.6 (s), 1210.5 (s), 1156.7 (s), 1112.8 (s), 1031.8 (s), 961.2 (m), 852.7 (s), 832.2 (s), 741.1 (s), 714.0 (m), 619.6 (m) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 367.1220, Obs. 367.1227.



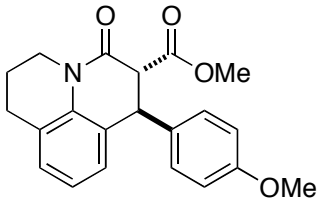
11o

Methyl 8-chloro-6-(4-methoxyphenyl)-2-methyl-4-oxo-5,6-dihydro-4*H*-pyrrolo[3,2,1-*ij*]quinoline-5-carboxylate (11o): Methyl 2-(5-chloro-2-methyl-1*H*-indole-1-carbonyl)-3-(4-methoxyphenyl)acrylate (0.0100 g, 0.261 mmol), $In(OTf)_3$ (0.0220 g, 0.0391 mmol) and toluene (5 mL) were combined according to the general procedure to afford **11o** as a white solid (0.0900 g, 90%) after 4 h. (R_f 0.45, 20% EtOAc/Hex) [**m.p.** 132-134 °C] (Single Diastereomer observed) **^{1}H NMR** (300 MHz, $CDCl_3$) δ ppm 7.29 - 7.33 (m, 1H), 7.11 - 7.18 (m, 2H), 6.85 - 6.93 (m, 2H), 6.68 (d, *J* = 1.10 Hz, 1H), 6.35 (d, *J* = 1.14 Hz, 1H), 4.91 (d, *J* = 10.74 Hz, 1H), 4.16 (d, *J* = 10.77 Hz, 1H), 3.81 (s, 3H), 3.67 (s, 3H), 2.69 (s, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ ppm 168.6, 163.7, 159.2, 138.5, 133.2, 130.1, 129.7, 129.5, 128.4, 123.9, 121.1, 118.3, 114.4, 108.6, 58.5, 55.2, 52.7, 45.2, 15.2. **IR** 2954.9 (w), 2838.0 (w), 1747.7 (s), 1710.5 (s), 1611.3 (m), 1513.0 (s) 1462.4 (m), 1427.7 (m), 1371.8 (s), 1251.1 (s), 1210.2 (m), 1178.1 (m), 1152.2 (s), 1031.1 (m), 886.2 (m), 858.6 (m), 829.4 (m), 763.7 (w), 737.9 (s), 701.6 (m) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 383.0924, Obs. 383.0923.



13 **15**

Methyl 6-(4-methoxyphenyl)-4-oxo-2,4,5,6-tetrahydro-1*H*-pyrrolo[3,2,1-*ij*]quinoline-5-carboxylate (12): Methyl 2-(indoline-1-carbonyl)-3-(4-methoxyphenyl)acrylate (0.0750 g, 0.222 mmol), $In(OTf)_3$ (0.0187 g, 0.0333 mmol) and toluene (5 mL) were combined according to the general procedure to afford **12** as an off-white solid (0.0606 g, 81%) after 42 h. (R*f* 0.40, 20% EtOAc/Hex) [**m.p.** 69-71 °C] (Mixture of *Single Diastereomer and Decarboxylated Product*) **^{1}H NMR** (300 MHz, $CDCl_3$) δ ppm 7.06 - 7.17 (m, 7.16), 6.84 - 6.93 (m, 7.14), 6.64 - 6.75 (m, 2.38), 4.66 (d, *J* = 10.77 Hz, 1.00), 4.27 (dd, *J* = 7.05, 9.69 Hz, 1.36), 4.02 - 4.22 (m, 5.13), 3.77 - 3.87 (m, 8.16), 3.65 (s, 3.16), 3.17 - 3.33 (m, 5.05), 2.77 - 2.98 (m, 2.87). **^{13}C NMR** (75 MHz, $CDCl_3$) δ ppm 169.7, 166.9, 162.9, 158.9, 158.6, 141.0, 140.1, 133.7, 131.2, 129.3, 128.9, 128.8, 125.6, 125.4, 124.0, 123.9, 123.6, 123.5, 122.6, 114.3, 114.2, 57.0, 55.2, 55.2, 52.5, 45.5, 45.2, 45.0, 41.4, 40.2, 27.9. **IR**: 3035.8 (w), 2951.3 (w), 2837.7 (w), 1745.8 (m), 1668.2 (s), 1595.3 (m), 1512.9 (s), 1480.9(s), 1468.7 (m), 1396.6 (m), 1353.3 (w), 1250.1 (s), 1178.6 (w), 1153.9 (w), 1031.8 (w), 834.7 (w), 764.9 (m), 747.5 (m) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 337.1314, Obs. 337.1313.



14

***trans*-Methyl 4-(4-methoxyphenyl)-3-oxo-1,2,3,5,6,7-hexahydropyrido[3,2,1-*ij*]quinoline-2-carboxylate (14):** Methyl 3-(4-methoxyphenyl)-2-(1,2,3,4-tetrahydroquinoline-1-carbonyl)acrylate (0.075 g, 0.213 mmol), $In(OTf)_3$ (0.0179 g, 0.0320 mmol) and toluene (5 mL) were combined according to the general procedure to afford **14** as a colorless oil (0.0622 g, 83%) after 42 h. (R_f 0.40, 20% EtOAc/Hex) (*Single Diastereomer observed*) **^{1}H NMR** (400 MHz, $CDCl_3$) δ ppm 7.07 - 7.12 (m, 2H), 7.02 - 7.06 (m, 1H), 6.83 - 6.91 (m, 3H), 6.65 - 6.69 (m, 1H), 4.53 (d, *J* = 10.04 Hz, 1H), 3.96 - 4.04 (m, 1H), 3.82 - 3.91 (m, 2H), 3.79 (s, 3H), 3.62 (s, 3H), 2.85 (t, *J* = 6.27 Hz, 2H), 1.93 - 2.03 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ ppm 169.3, 165.0, 158.8, 134.9, 131.0, 129.3, 128.5, 127.1, 126.4, 125.4, 123.0, 114.3, 55.2, 55.0, 52.5, 43.9, 41.3, 27.3, 21.3. **IR**: 3003.2 (w), 2950.0 (w), 2886.7 (w), 1746.6 (s), 1666.8 (s), 1612.8 (w), 1592.4 (w), 1513.7 (s), 1469.9 (m), 1460.9 (w), 1383.3 (m), 1274.9 (m), 1251.2 (m), 1179.0 (w), 1154.0 (w), 1031.7 (w), 764.4 (s), 749.9 (s) cm^{-1}. **HRMS (ESI)** M/Z+ Calc. 351.1471, Obs. 351.1477.

3. Control Reactions

TfOH Control Reaction:

To a mixture of TfOH (0.0010 g, 0.0068 mmol) in 1,2-DCE heated to a reflux, dissolved (*Z*)-Methyl 2-(2-methyl-1*H*-indole-1-carbonyl)-3-(4-nitrophenyl)acrylate (0.250 g, 0.6866 mmol) was syringed into the reaction vessel. The reaction mixture was stirred at reflux for 16 h. The reaction afforded only starting material as observed by crude ^{1}H NMR.

DBU Epimerization Reaction:

The diastereomeric mixture of Methyl 2-methyl-6-(4-nitrophenyl)-4-oxo-5,6-dihydro-4*H*-pyrrolo[3,2,1-*ij*]quinoline-5-carboxylate (0.06 g, 0.16478 mmol), DBU (0.0075 g, 0.0494 mmol) and 1,2-DCE (3 mL) were combined and stirred at room temperature for 14 h. The reaction afforded *trans* as a single observable diastereomer (>99:1 *dr*) from ^{1}H NMR of the crude reaction mixture.

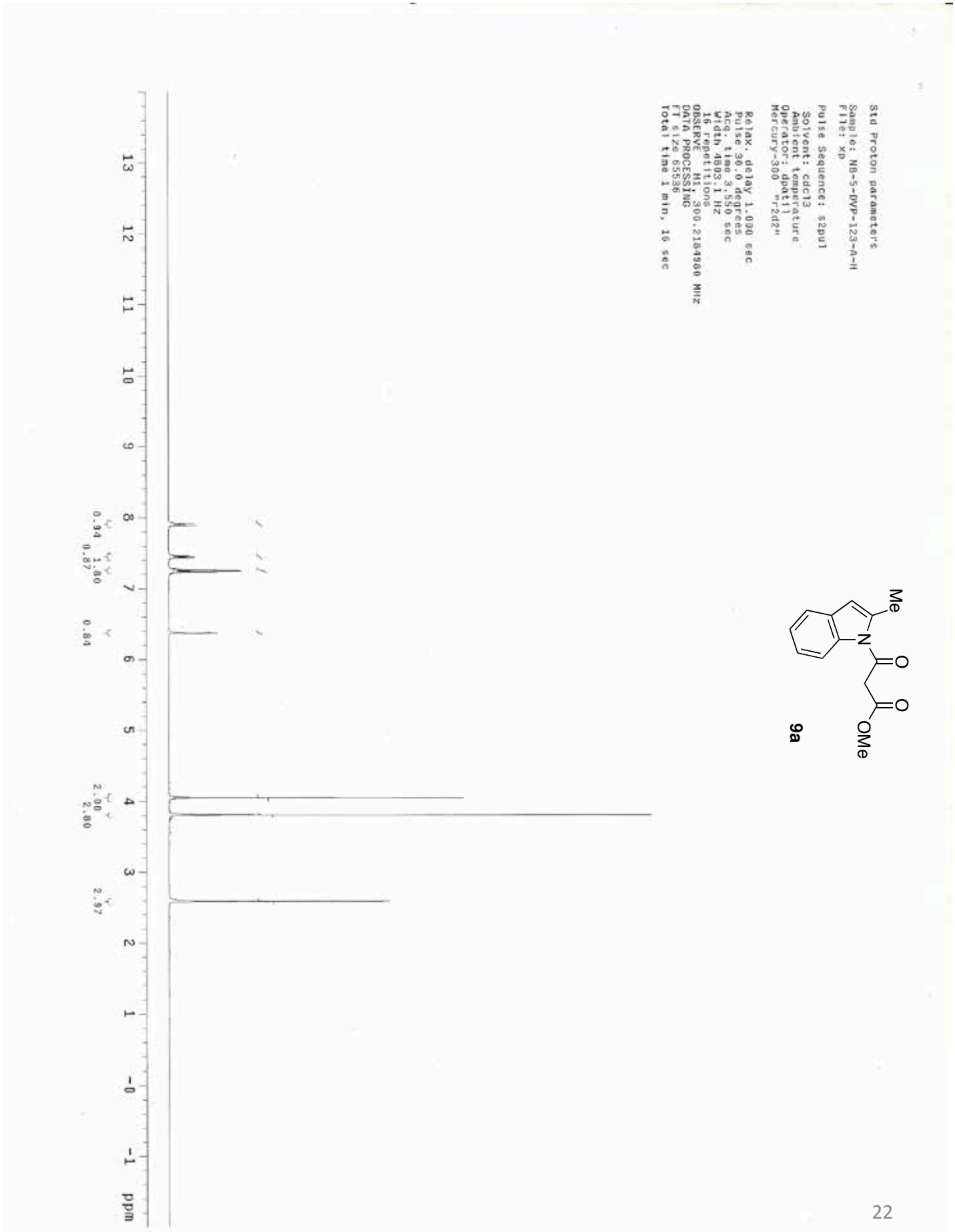
4. References

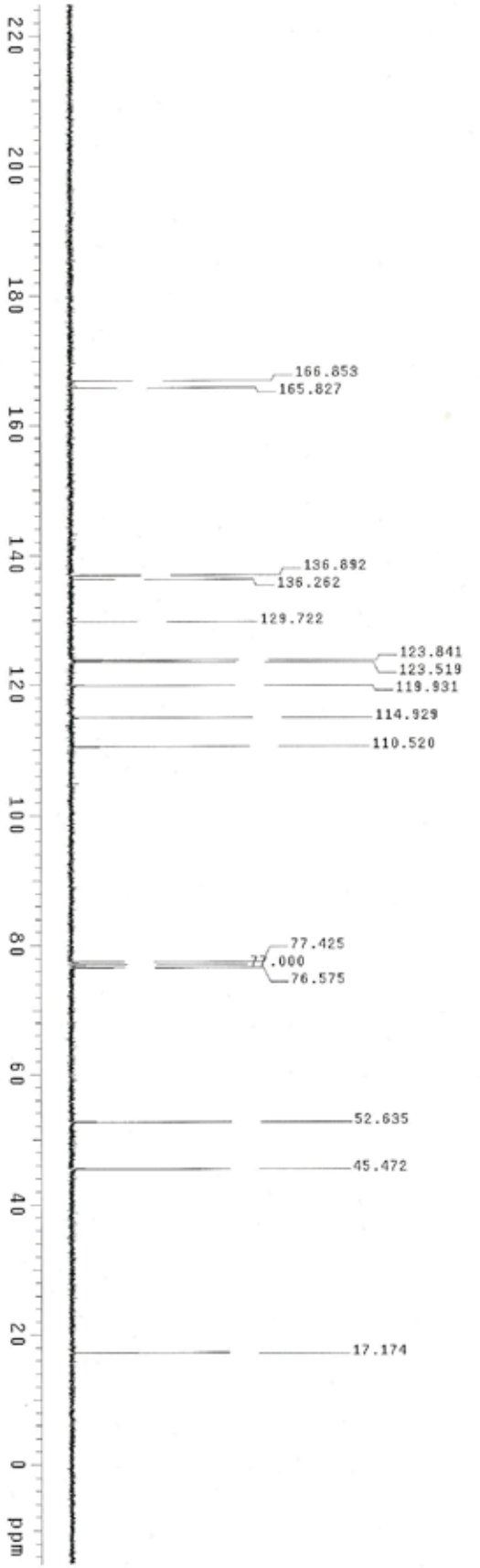
1. Patil, D. V.; Cavitt, M. A.; Grzybowski, P. L.; France, S. *Chem. Commun*. **2011**, *47*, 10278.

2. Frontier, A. J.; Vaidya, T.; Atesin, A. C.; Herrick, I. R.; Eisenberg, R. *Angew. Chem. Int. Ed*. **2010**, *49*, 3363.

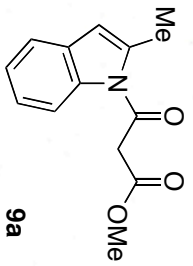
3. Aggarwal, V. K.; Beffield, A. J. *Org. Lett*. **2003**, *5*, 5075.

5. NMR Spectra (^{1}H and ^{13}C)

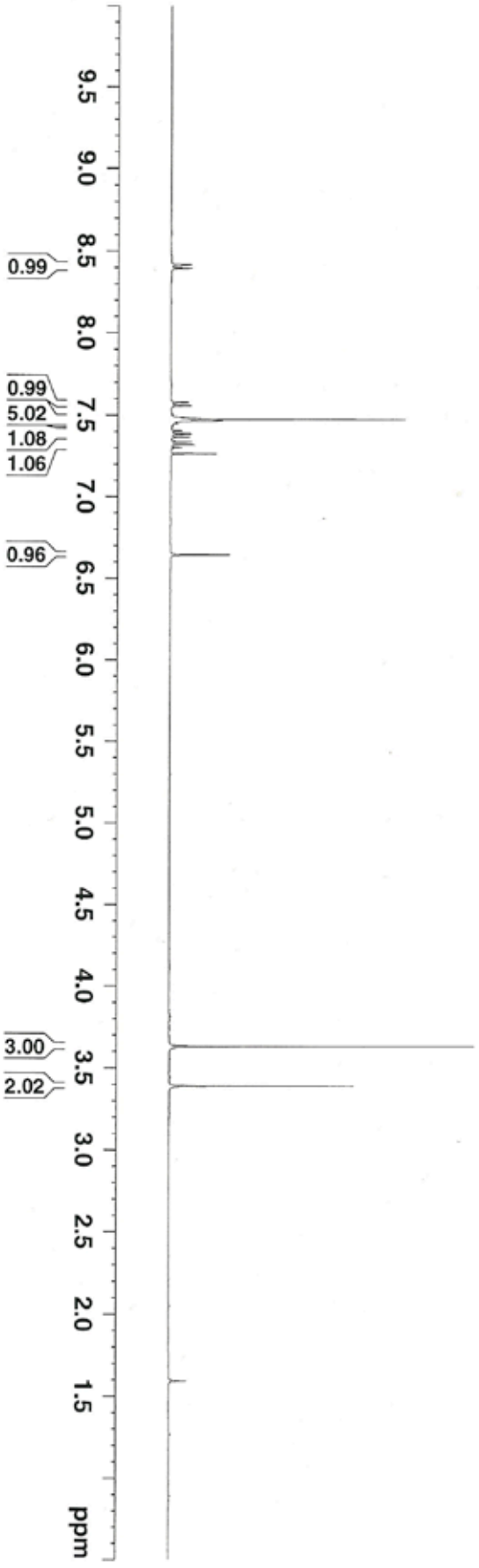

Std Proton parameters
Sample: NB-S-DVP-123-A-H
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpatil
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
16 repetitions
OBSERVE H1, 300.2184980 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 16 sec
Me
N
O
O
O
OMe
9a
0.94
1.80
0.87
0.84
2.00
2.80
2.97
13
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm

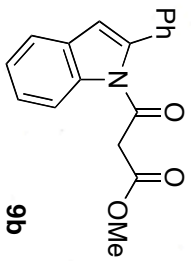



```
Std Carbon experiment

Sample: NB-5-DVP-123-A-SM-C
File: xp

Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
 Operator: dpatil
Mercury-300 "r2d2"

 Relax. delay 1.000 sec
 Pulse 30.0 degrees
 Acq. time 1.301 sec
 Width 18115.9 Hz
 64 repetitions
OBSERVE  C13, 75.4900131 MHz
DECOUPLE  H1, 300.2199481 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 65536
Total time 10 min, 45 sec
```

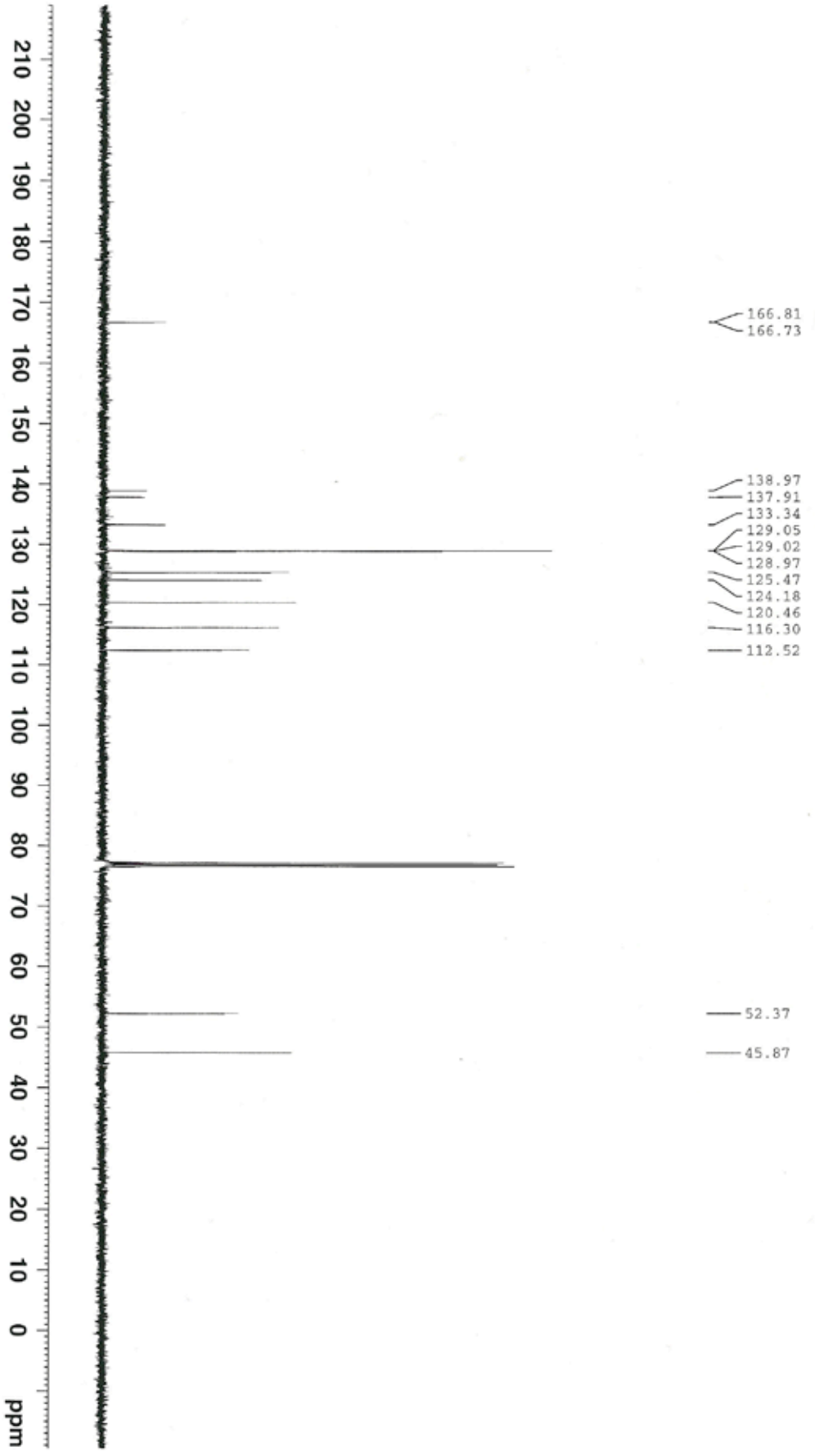


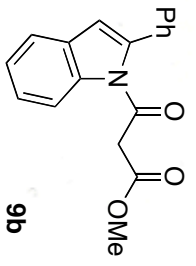
9a


9.5
9.0
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
ppm
0.99
0.99
5.02
1.08
1.06
0.96
3.00
2.02

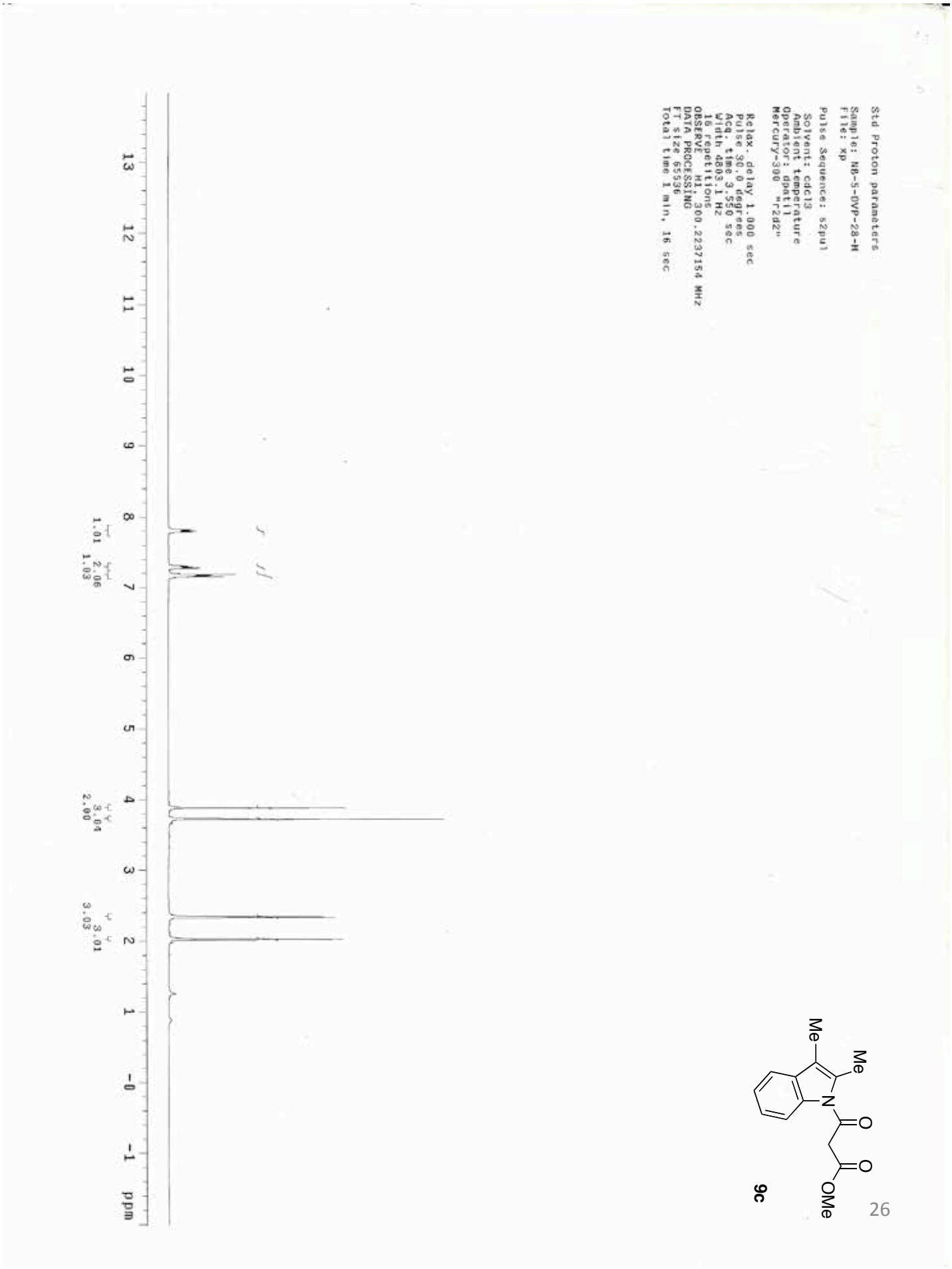

Ph
N
O
O
OMe

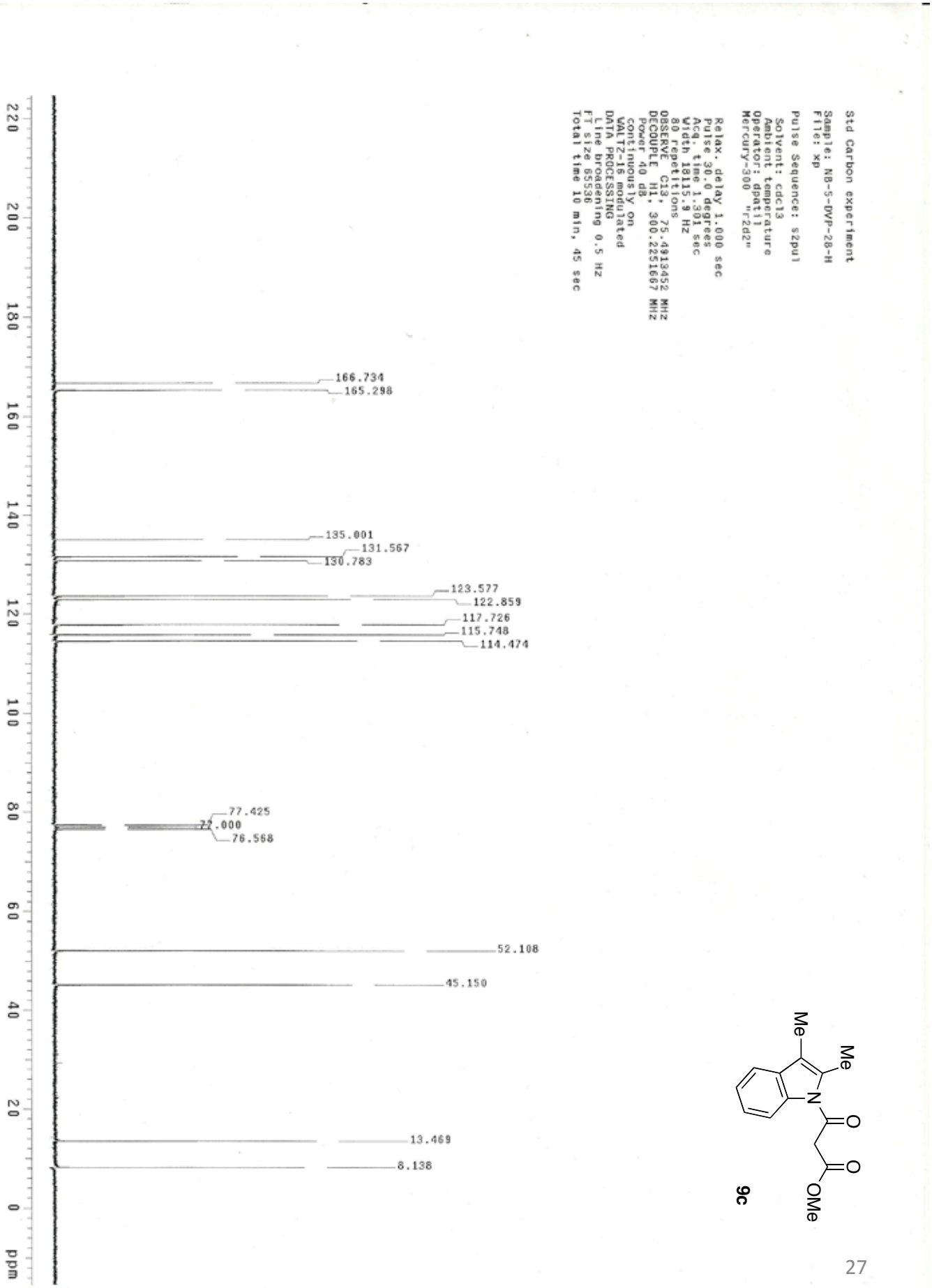
9b

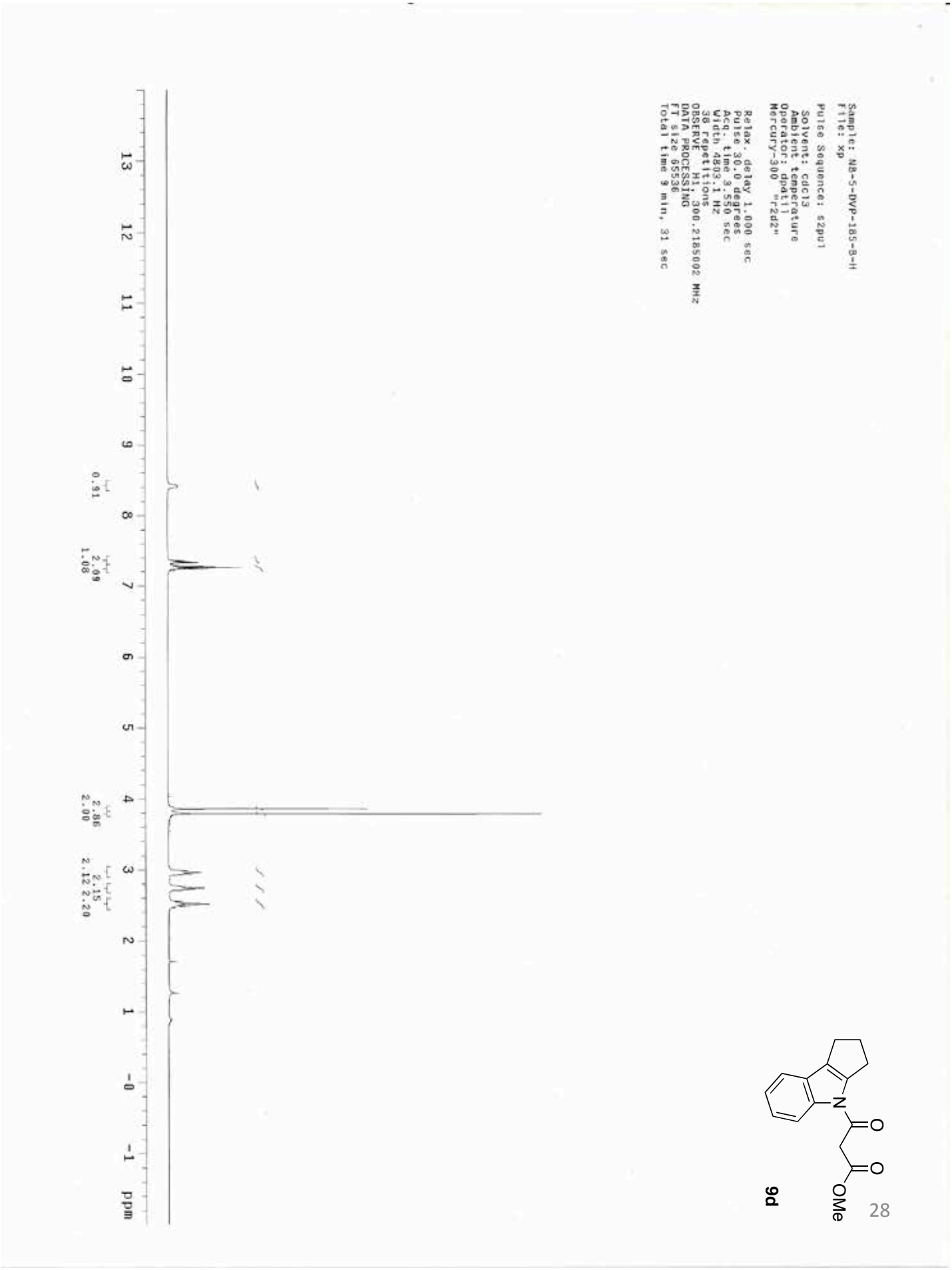

166.81
166.73
138.97
137.91
133.34
129.05
129.02
128.97
125.47
124.18
120.46
116.30
112.52
52.37
45.87
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

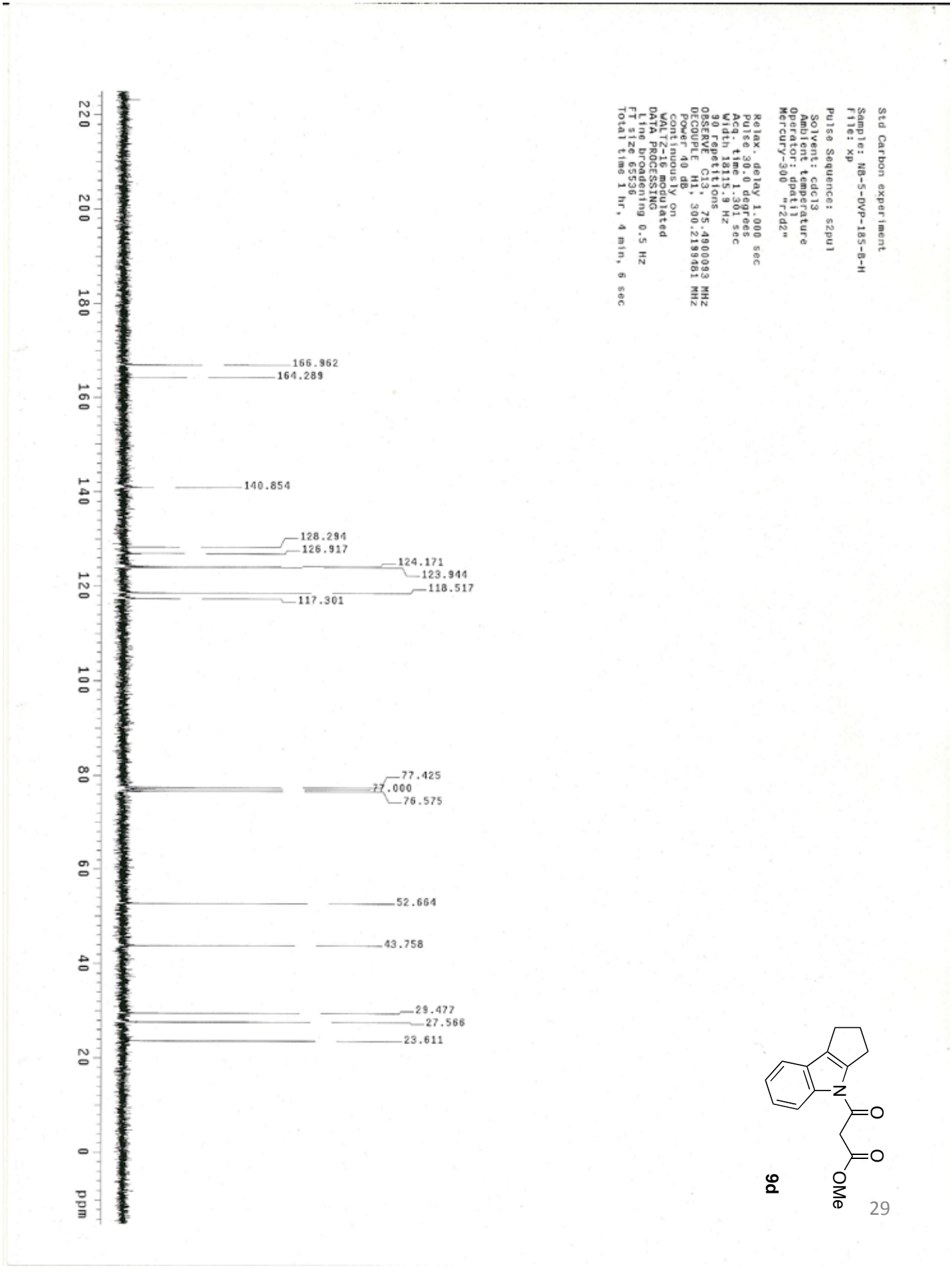

Ph
N
O
O
O
OMe

9b

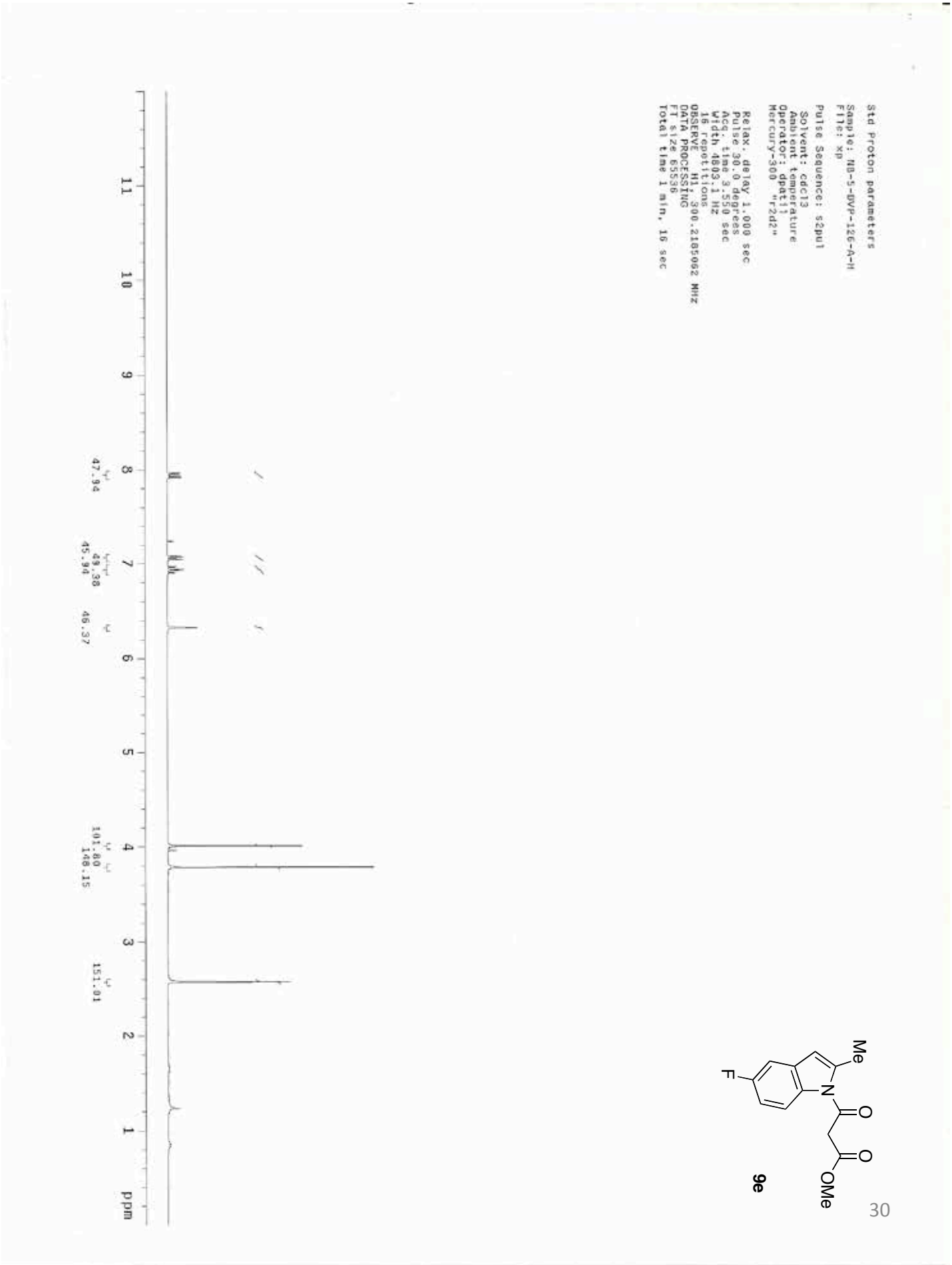

Std Proton parameters
Sample: NB-5-DVP-28-H
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpatil
Mercury-300 "r202"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
16 repetitions
OBSERVE H1, 300.2237154 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 16 sec
1.01
2.06
1.03
3.04
2.00
3.01
3.03
13
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
Me
Me
N
O
O
OMe
9c

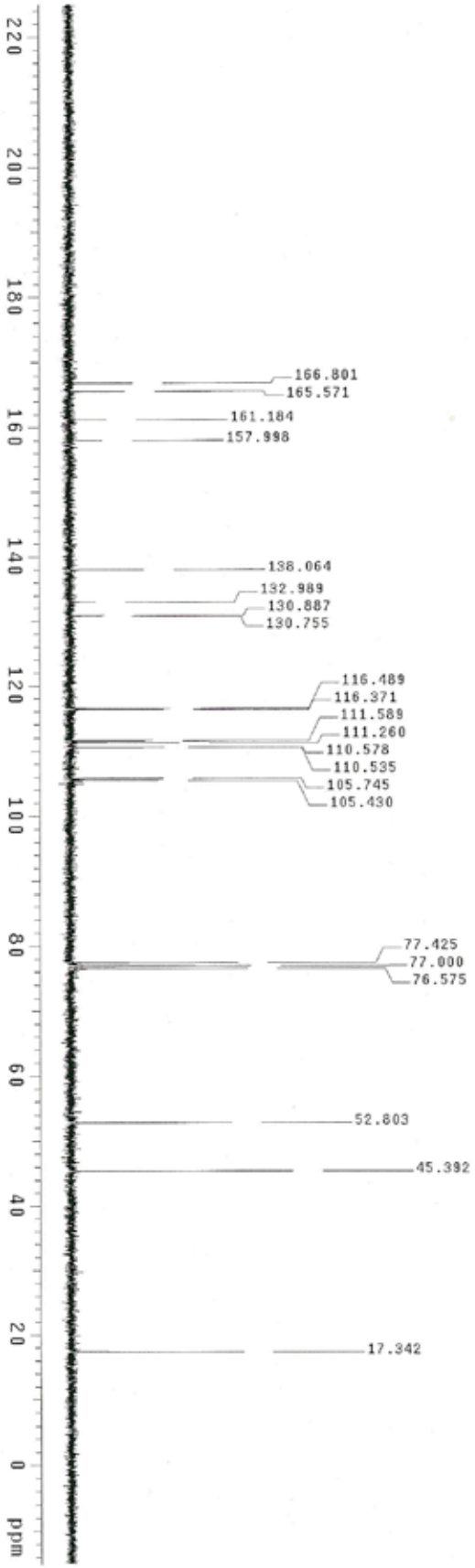

Std Carbon experiment
Sample: NB-5-DVP-28-H
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpatil
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 1.301 sec
Width 18115.9 Hz
80 repetitions
OBSERVE C13, 75.4913452 MHz
DECOUPLE H1, 300.2251667 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 10 min, 45 sec
166.734
165.298
135.001
131.567
130.783
123.577
122.859
117.726
115.748
114.474
77.425
77.000
76.568
52.108
45.150
13.469
8.138
220
200
180
160
140
120
100
80
60
40
20
0
ppm
Me
Me
N
O
O
OMe
9c


Sample: NB-5-DVP-185-B-H
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpatil
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
38 repetitions
OBSERVE H1, 300.2185002 MHz
DATA PROCESSING
FT size 65536
Total time 9 min, 31 sec
13
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
0.91
2.09
1.08
2.86
2.00
2.15
2.12
2.20
O
O
OMe
N
9d

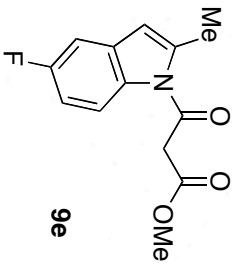

Std Carbon experiment
Sample: NB-5-DVP-185-B-H
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpatil
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 1.301 sec
Width 18115.9 Hz
90 repetitions
OBSERVE C13, 75.4900093 MHz
DECOUPLE H1, 300.2199481 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 4 min, 6 sec
166.962
164.289
140.854
128.294
126.917
124.171
123.944
118.517
117.301
77.425
77.000
76.575
52.664
43.758
29.477
27.566
23.611
220
200
180
160
140
120
100
80
60
40
20
0
ppm
N
O
O
OMe

9d

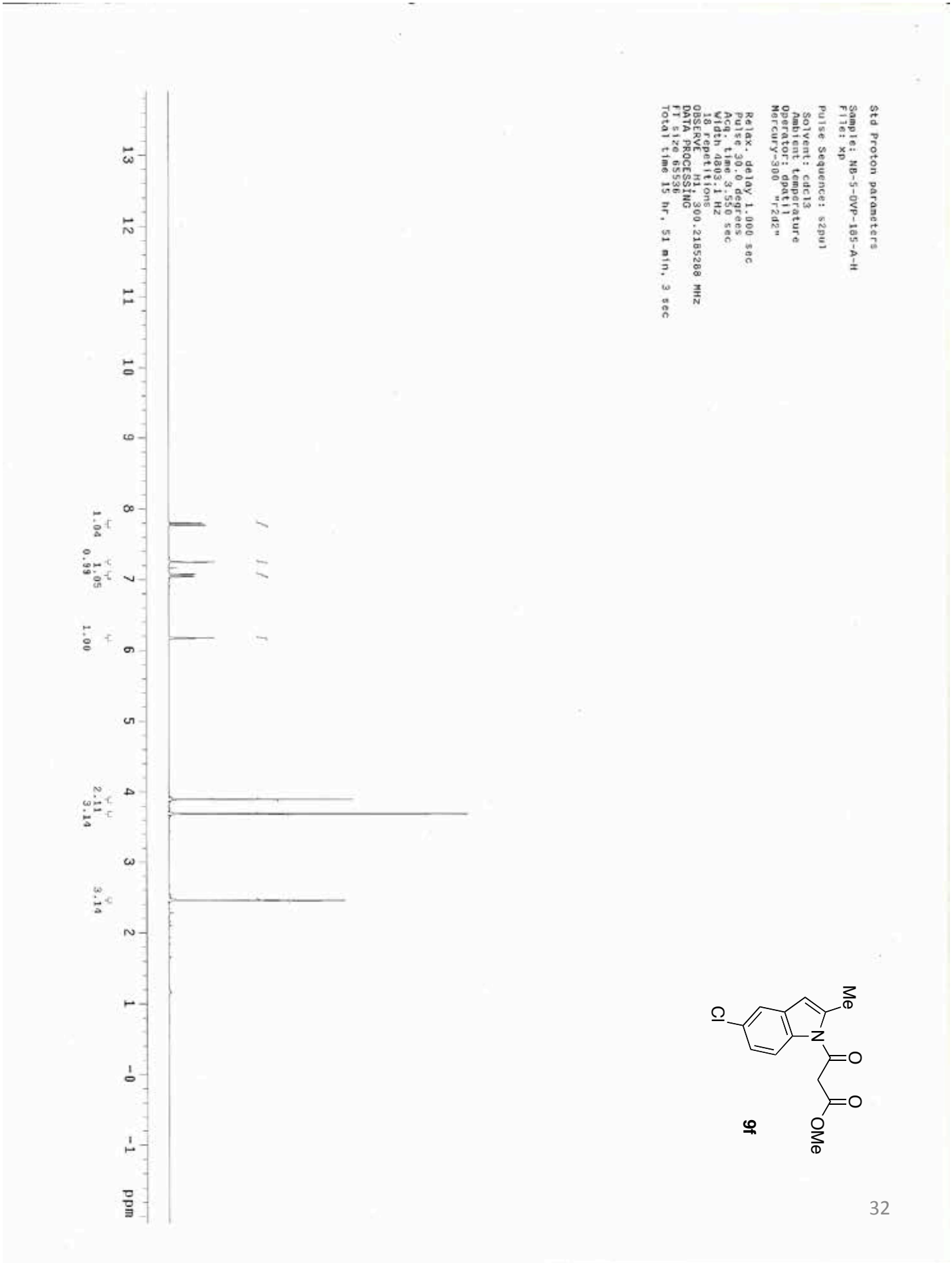

Std Proton parameters
Sample: NB-5-DVP-126-A-H
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpatil
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.2 Hz
16 repetitions
OBSERVE H1, 300.2185092 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 16 sec
47.94
49.38
45.94
46.37
101.80
148.15
151.01
11
10
9
8
7
6
5
4
3
2
1
ppm
Me
F
N
O
O
OMe
9e

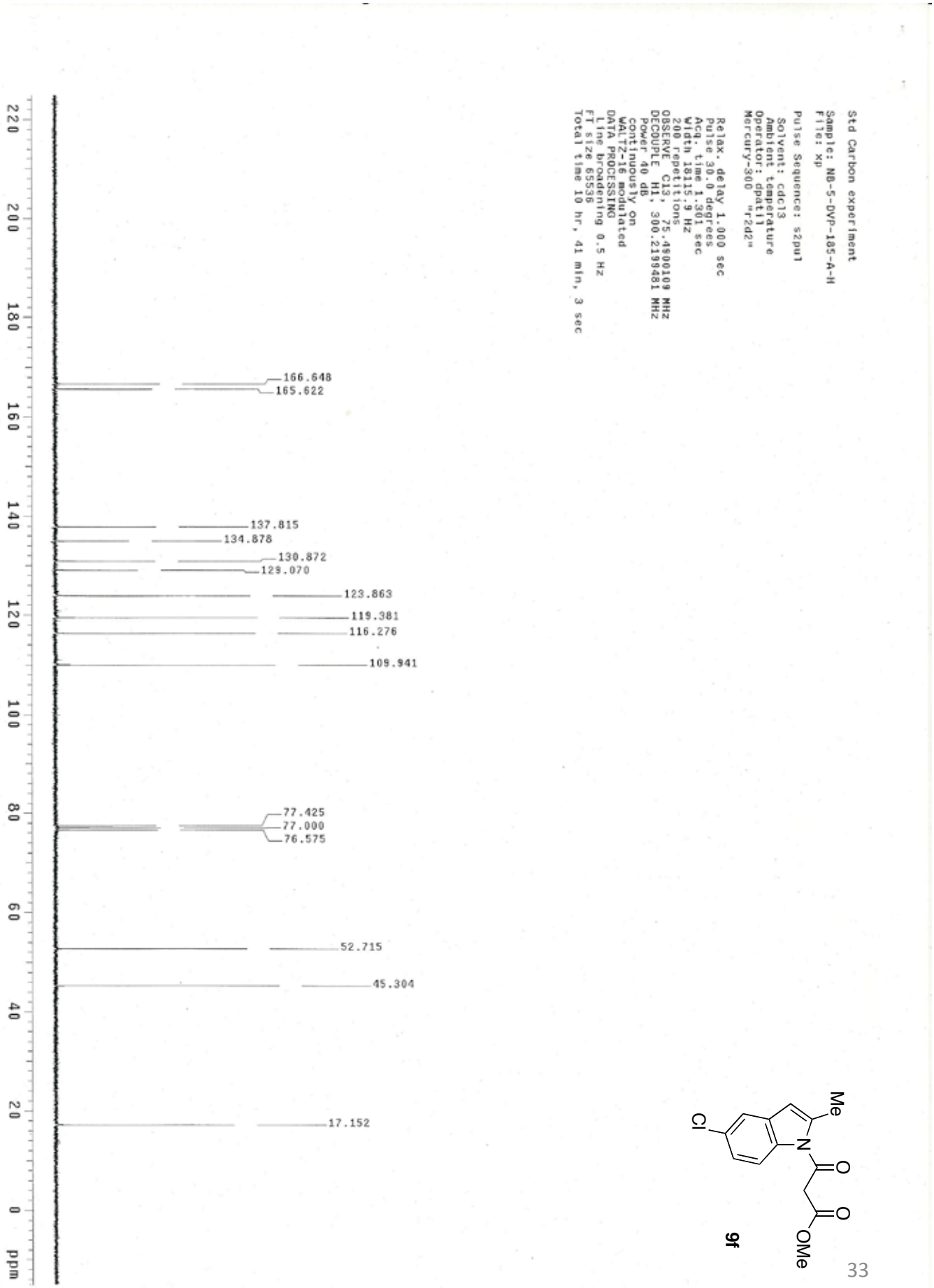



```
Std Carbon experiment

Sample: NB-5-DVP-126-A-C
File: xp

Pulse Sequence: s2pul
  Solvent: cdcl3
  Ambient temperature
Operator: dpatil
Mercury-300 "r2d2"

    Relax. delay 1.000 sec
    Pulse 30.0 degrees
    Acq. time 1.301 sec
    Width 18115.9 Hz
    144 repetitions
OBSERVE   C13,   75.4900054 MHz
DECOUPLE  H1,  300.2199481 MHz
  Power 40 dB
  continuously on
  WALTZ-16 modulated
DATA PROCESSING
  Line broadening 0.5 Hz
FT size 65536
Total time 10 min, 45 sec
```



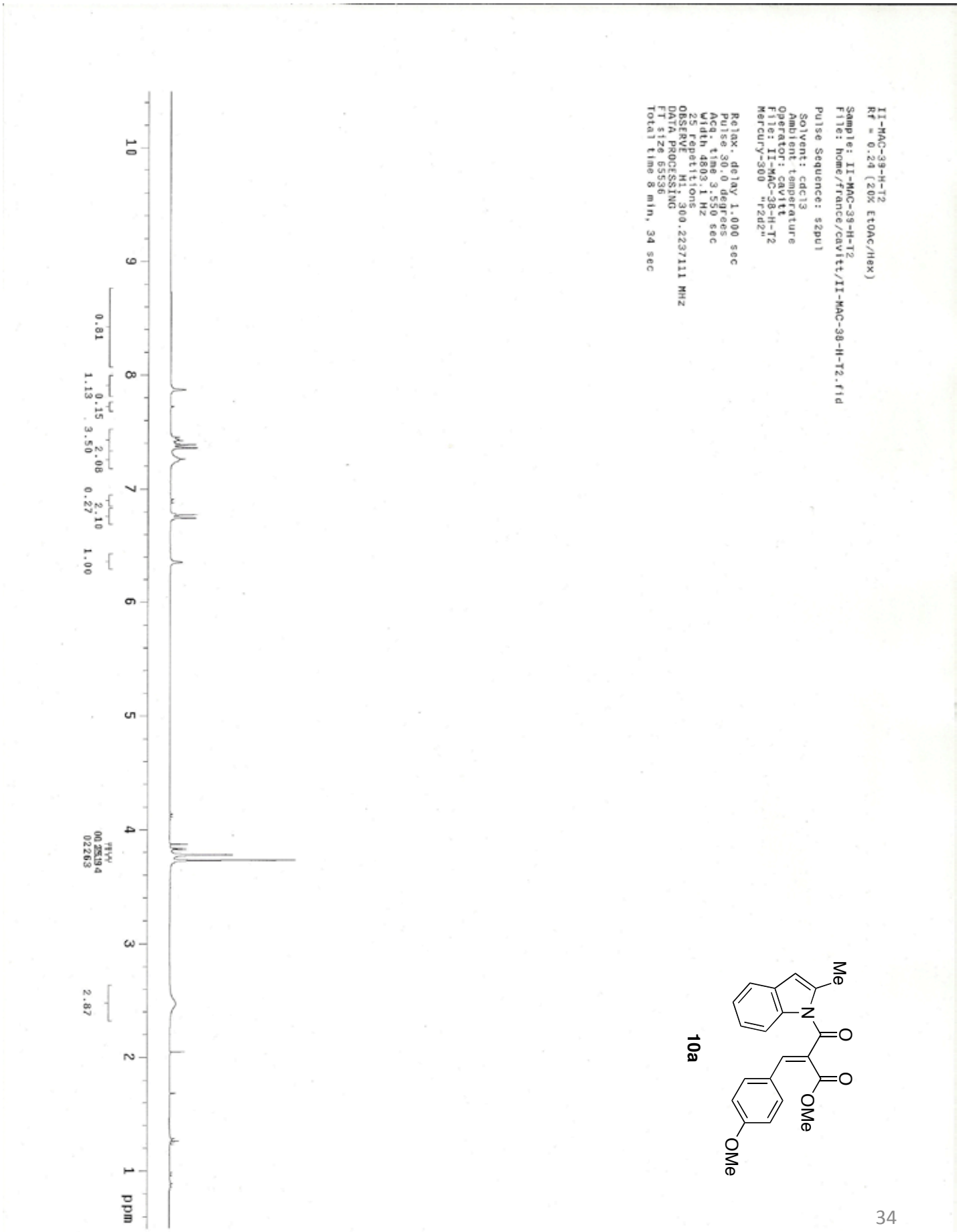
9e


Std Proton parameters
Sample: NB-5-OVP-185-A-H
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpatil
Mercury-300 "r202"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
16 repetitions
OBSERVE H1, 300.2185208 MHz
DATA PROCESSING
FT size 65536
Total time 15 hr, 51 min, 3 sec
1.04
1.05
0.99
1.00
2.11
3.14
3.14
13
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
Me
Cl
N
O
O
O
OMe
9f


Std Carbon experiment
Sample: NB-5-DVP-105-A-H
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpatil
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 1.301 sec
Width 18115.9 Hz
200 repetitions
OBSERVE C13, 75.4900109 MHz
DECOUPLE H1, 300.2199481 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 10 hr, 41 min, 3 sec
166.648
165.622
137.815
134.878
130.872
129.070
123.863
119.381
116.276
109.941
77.425
77.000
76.575
52.715
45.304
17.152
220
200
180
160
140
120
100
80
60
40
20
0
ppm

Me
Cl
N
O
O
OMe

9f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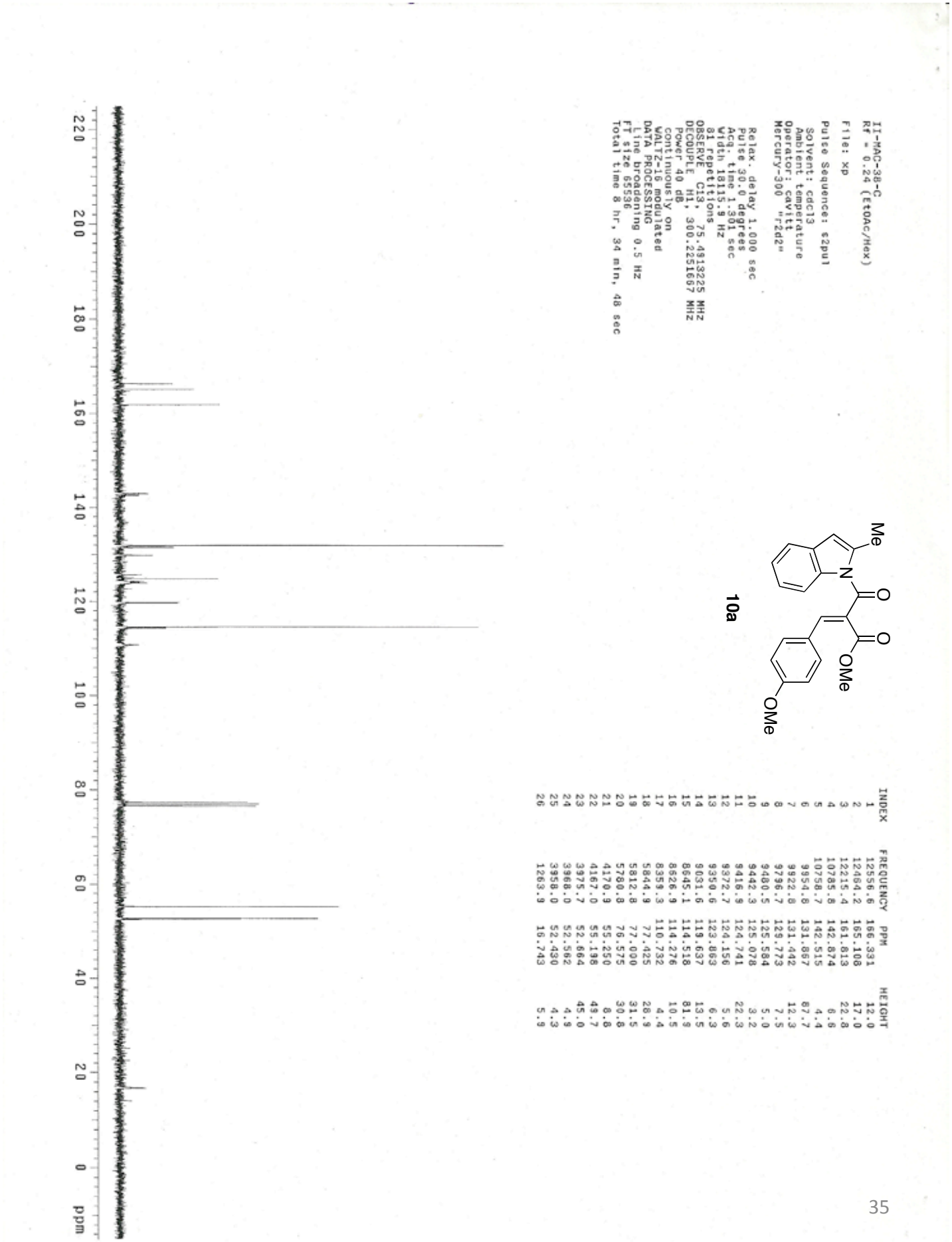
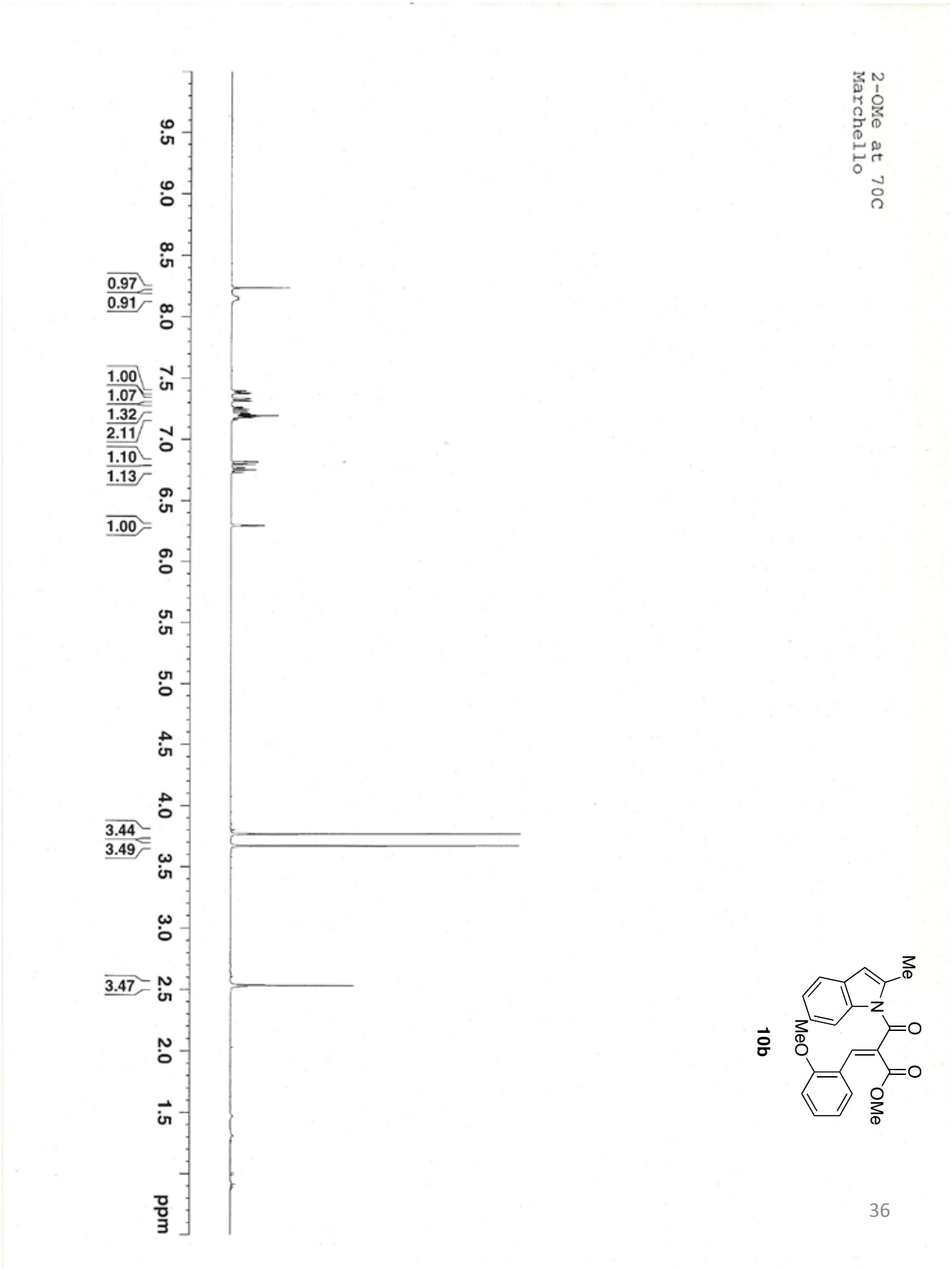
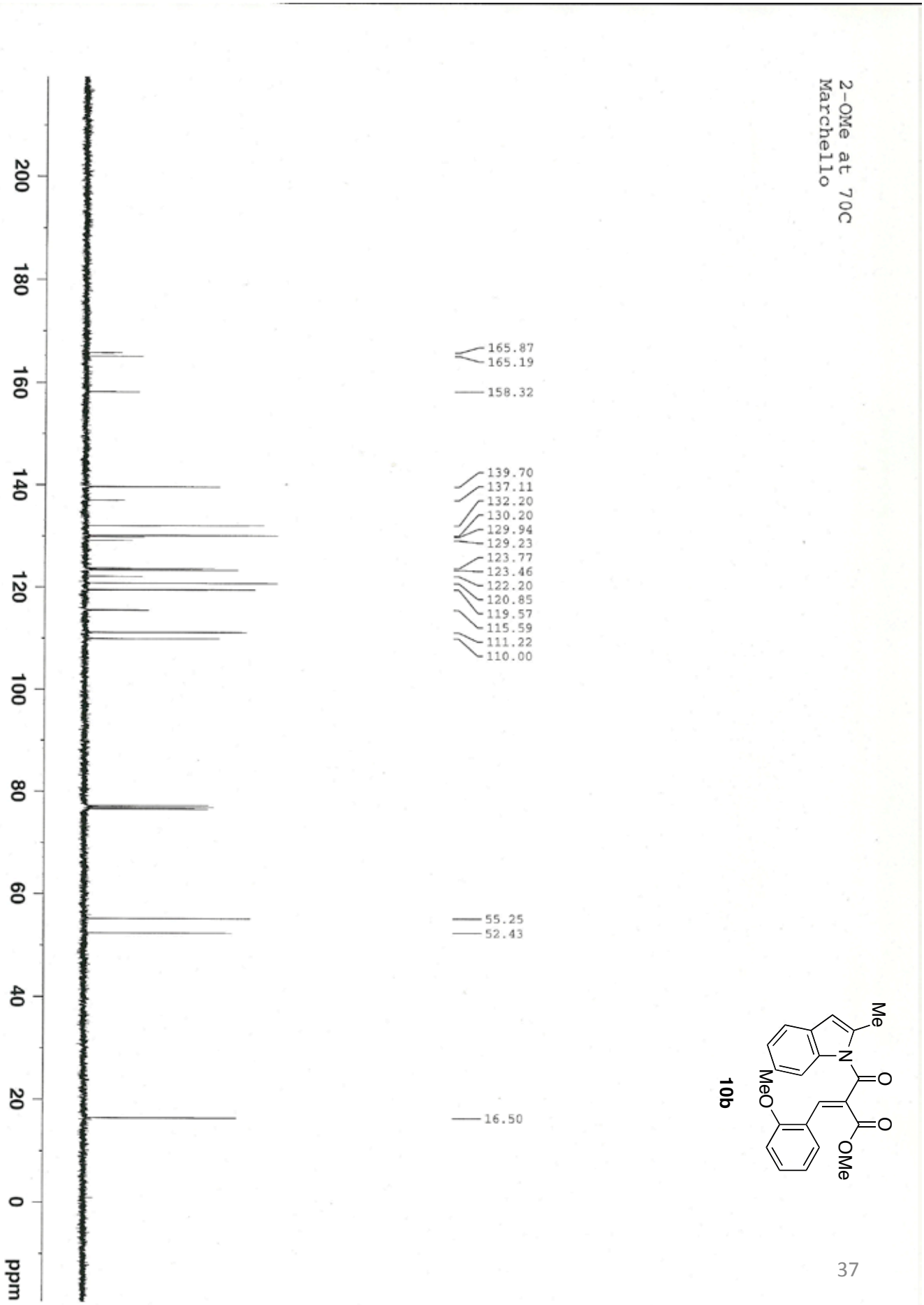
Rf = 0.24 (20% EtOAc/Hex)
Sample: II-MAC-38-H-T2
File: home/france/cavitt/II-MAC-38-H-T2.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: cavitt
File: II-MAC-38-H-T2
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
25 repetitions
OBSERVE H1, 300.2237111 MHz
DATA PROCESSING
FT size 65536
Total time 8 min, 34 sec
0.81
1.13
0.15
3.50
2.08
0.27
2.10
1.00
2.87
10
9
8
7
6
5
4
3
2
1
ppm
Me
N
O
O
OMe
OMe
10a


II-MAC-38-C
Rf = 0.24 (EtOAc/Hex)
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: cavitt
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 1.301 sec
Width 18115.9 Hz
81 repetitions
OBSERVE C13, 75.4913225 MHz
DECOUPLE H1, 300.2251667 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 8 hr, 34 min, 48 sec
Me
N
O
O
OMe
OMe
10a
INDEX FREQUENCY PPM HEIGHT
1 12556.6 166.331 12.0
2 12464.2 165.108 17.0
3 12215.4 161.813 22.8
4 10785.8 142.874 6.6
5 10758.7 142.515 4.4
6 9954.8 131.867 87.7
7 9922.8 131.442 12.3
8 9796.7 129.773 7.5
9 9680.5 125.584 5.0
10 9442.3 125.078 3.2
11 9416.9 124.741 22.3
12 9372.7 124.156 5.6
13 9350.6 123.863 6.3
14 9031.6 119.637 13.5
15 8645.1 114.518 81.9
16 8626.9 114.276 10.5
17 8359.3 110.732 4.4
18 5844.9 77.425 28.9
19 5812.8 77.000 31.5
20 5780.8 76.575 30.8
21 4170.9 55.250 8.8
22 4167.0 55.198 49.7
23 3975.7 52.664 45.0
24 3968.0 52.562 4.9
25 3958.0 52.430 4.3
26 1263.9 16.743 5.9
220
200
180
160
140
120
100
80
60
40
20
0
ppm

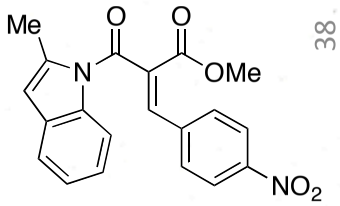

2-OMe at 70C
Marchello
0.97
0.91
1.00
1.07
1.32
2.11
1.10
1.13
1.00
3.44
3.49
3.47
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm
Me
N
O
O
MeO
OMe

10b

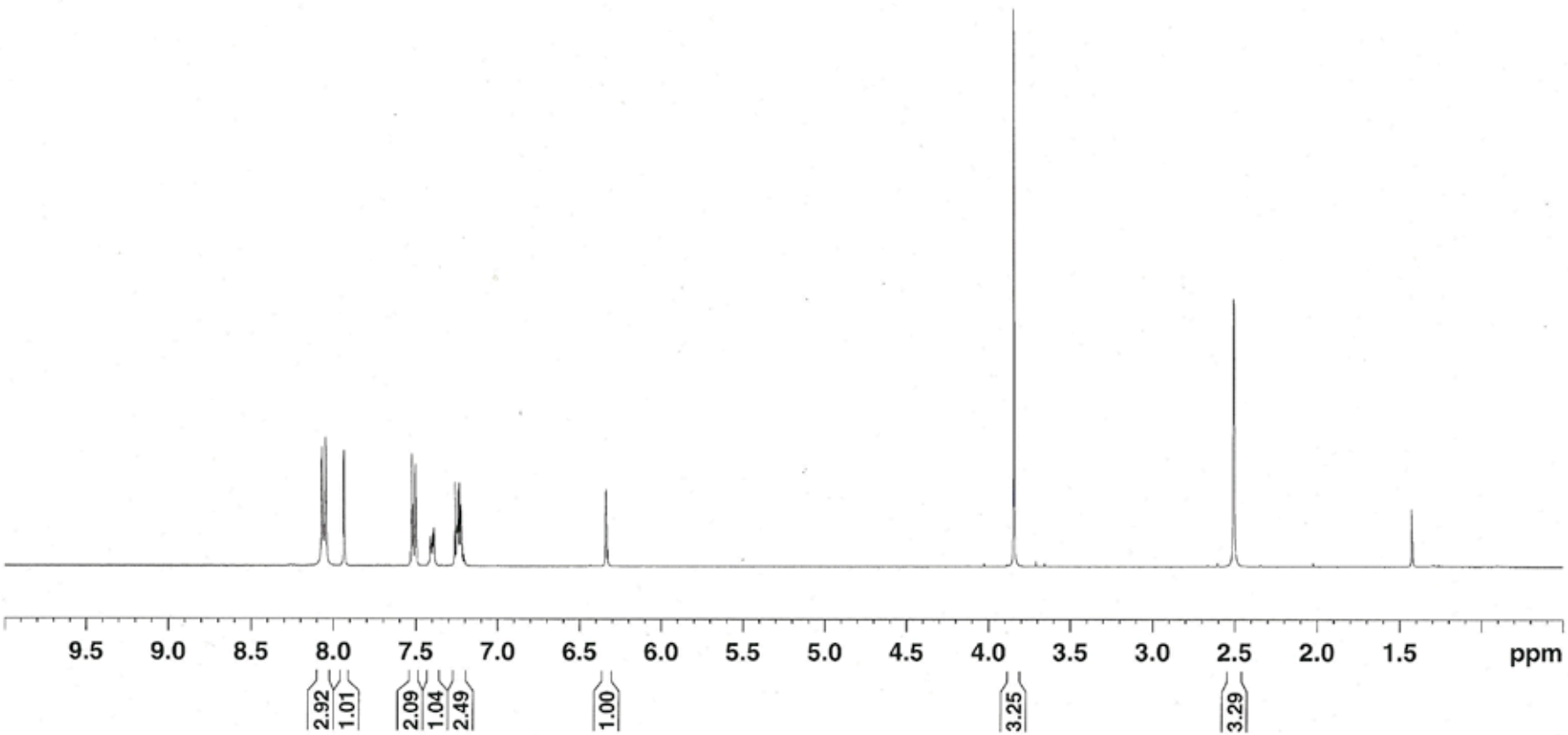

2-OMe at 70C
Marchello
165.87
165.19
158.32
139.70
137.11
132.20
130.20
129.94
129.23
123.77
123.46
122.20
120.85
119.57
115.59
111.22
110.00
55.25
52.43
16.50
200
180
160
140
120
100
80
60
40
20
0
ppm
Me
N
O
O
MeO
OMe

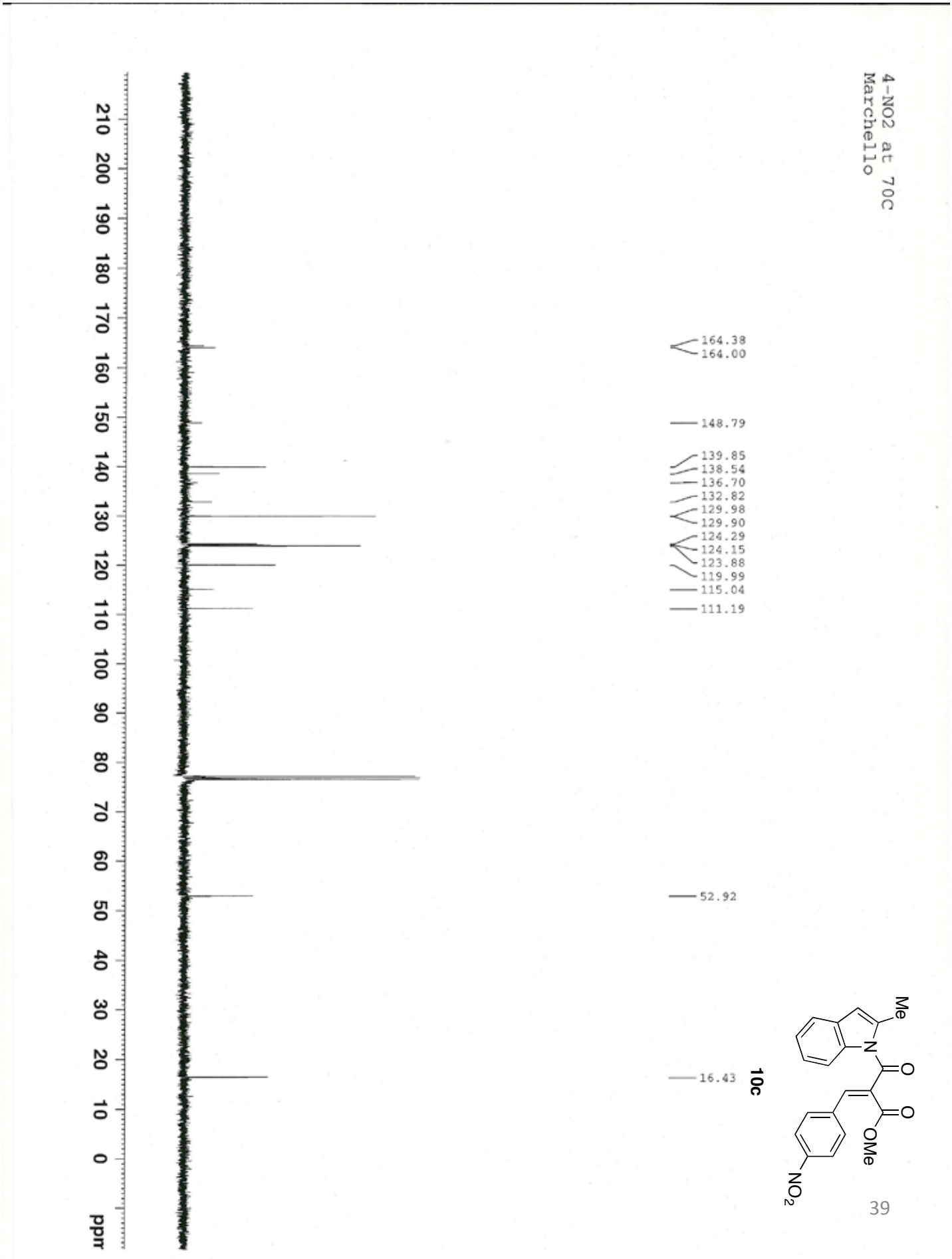
10b

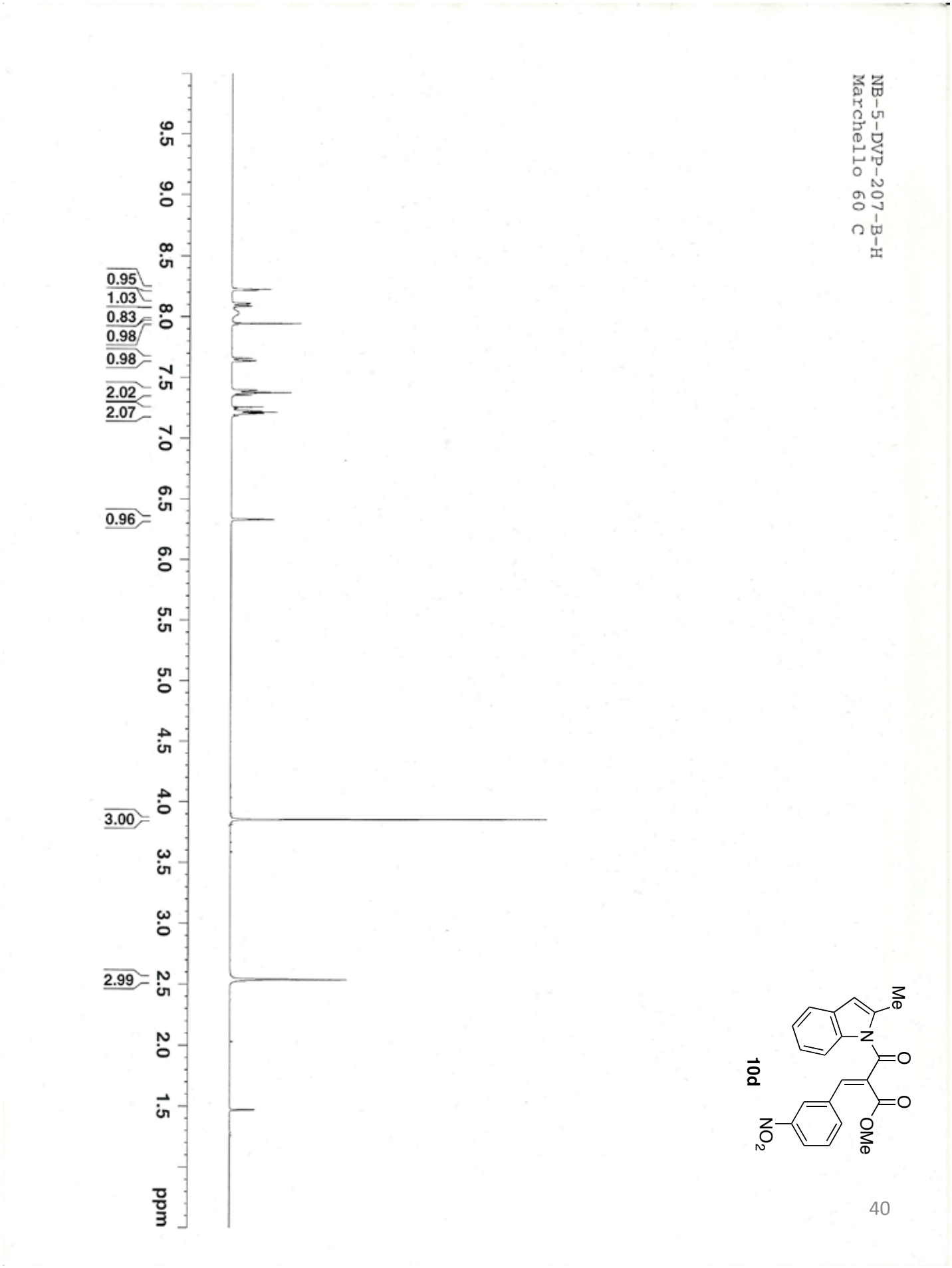
4-NO2 at 70C
Marchello

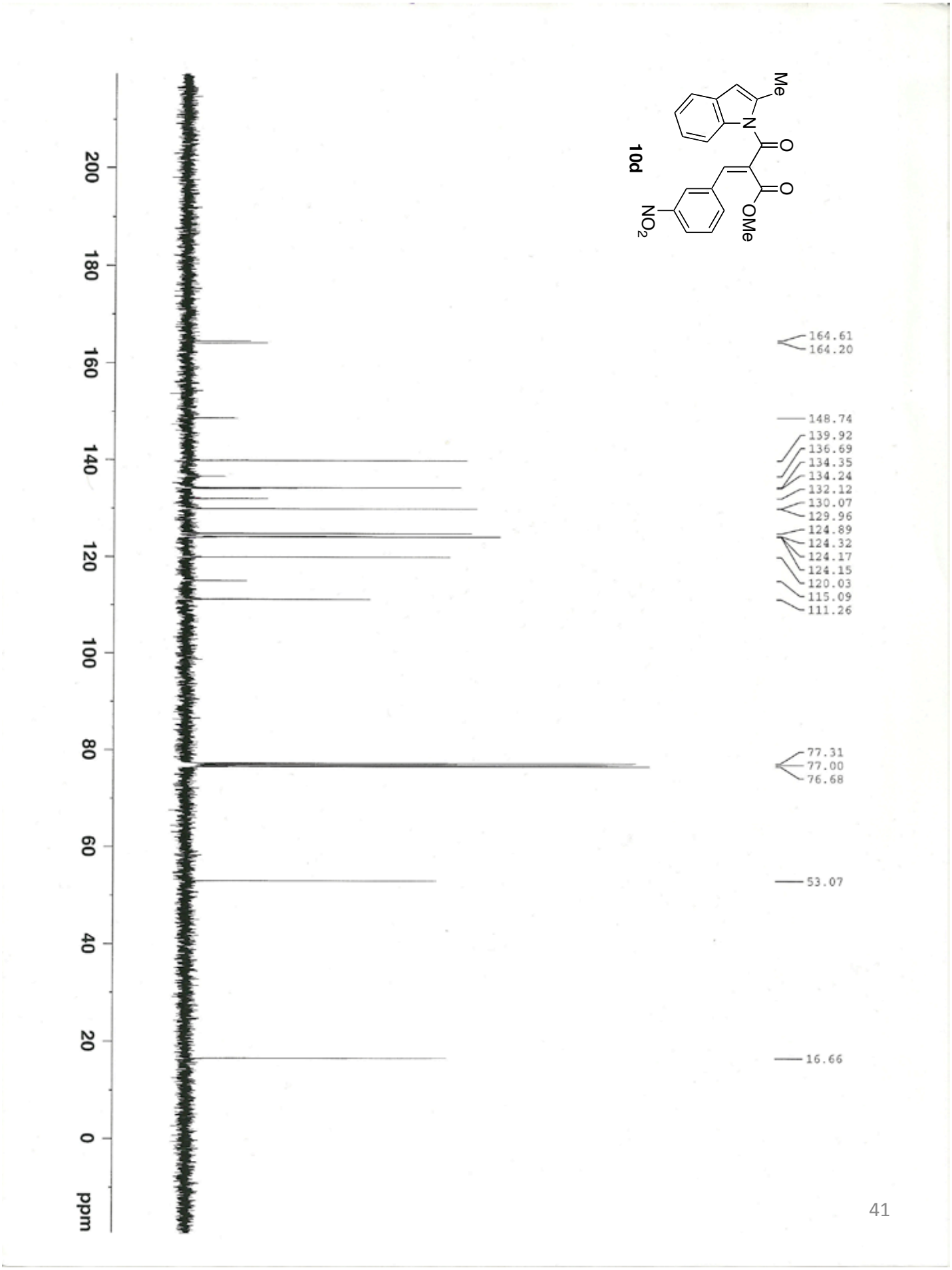


10c




4-NO2 at 70C
Marchello
164.38
164.00
148.79
139.85
138.54
136.70
132.82
129.98
129.90
124.29
124.15
123.88
119.99
115.04
111.19
52.92
16.43
10c
Me
N
O
O
OMe
NO2
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


NB-5-DVP-207-B-H
Marchello 60 C
0.95
1.03
0.83
0.98
0.98
2.02
2.07
0.96
3.00
2.99
9.5
9.0
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
ppm
Me
N
O
O
OMe
NO2
10d

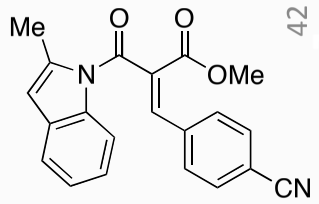

10d
Me
N
O
O
O
OMe
NO2
164.61
164.20
148.74
139.92
136.69
134.35
134.24
132.12
130.07
129.96
124.89
124.32
124.17
124.15
120.03
115.09
111.26
77.31
77.00
76.68
53.07
16.66
200
180
160
140
120
100
80
60
40
20
0
ppm

Std Proton parameters

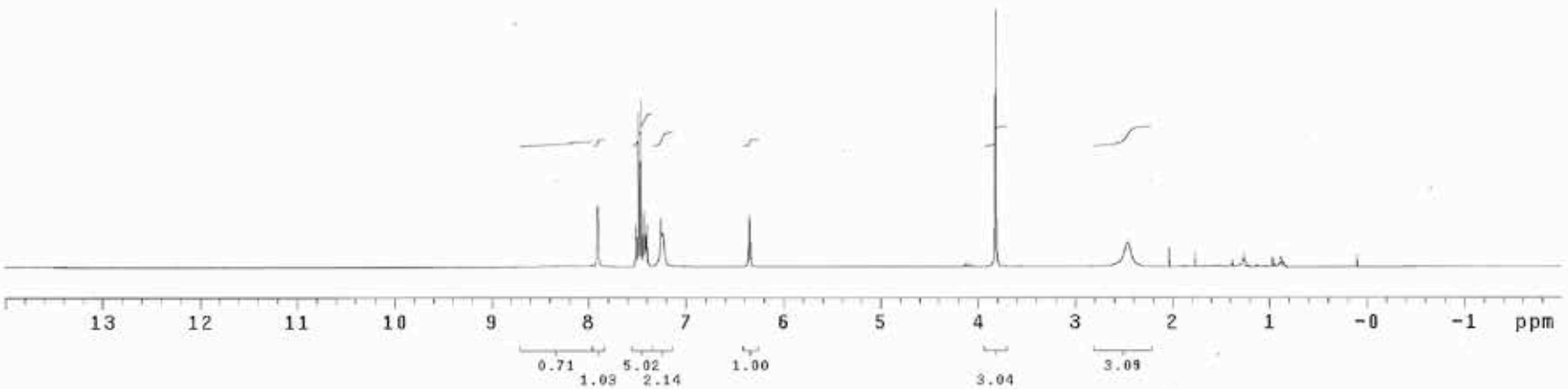
Sample: NB-5-DVP-207-A-H
File: home/france/dpatil/NB-5-DVP-207-A-H.fid

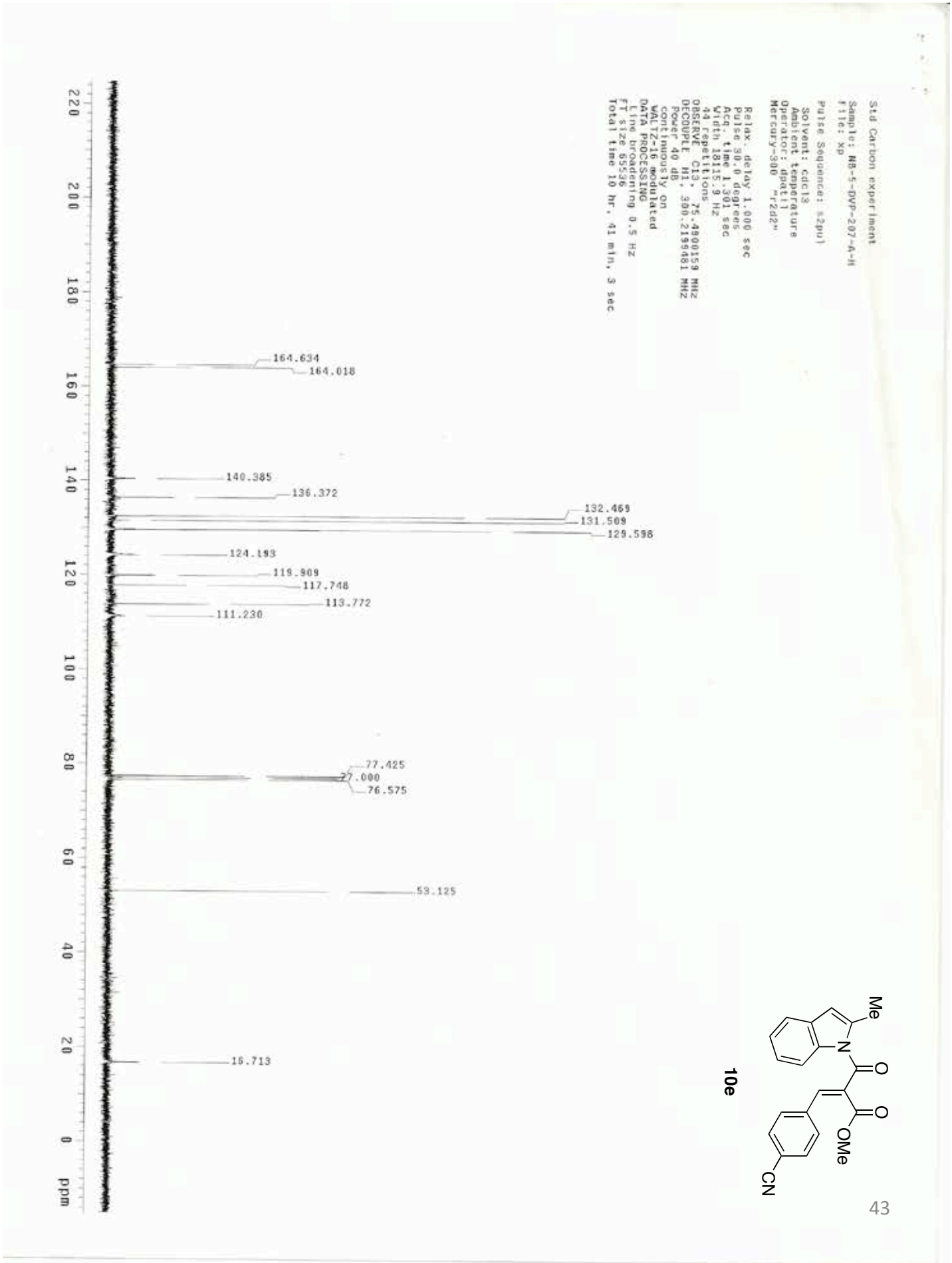
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpatil
File: NB-5-DVP-207-A-H
Mercury-300 "r2d2"

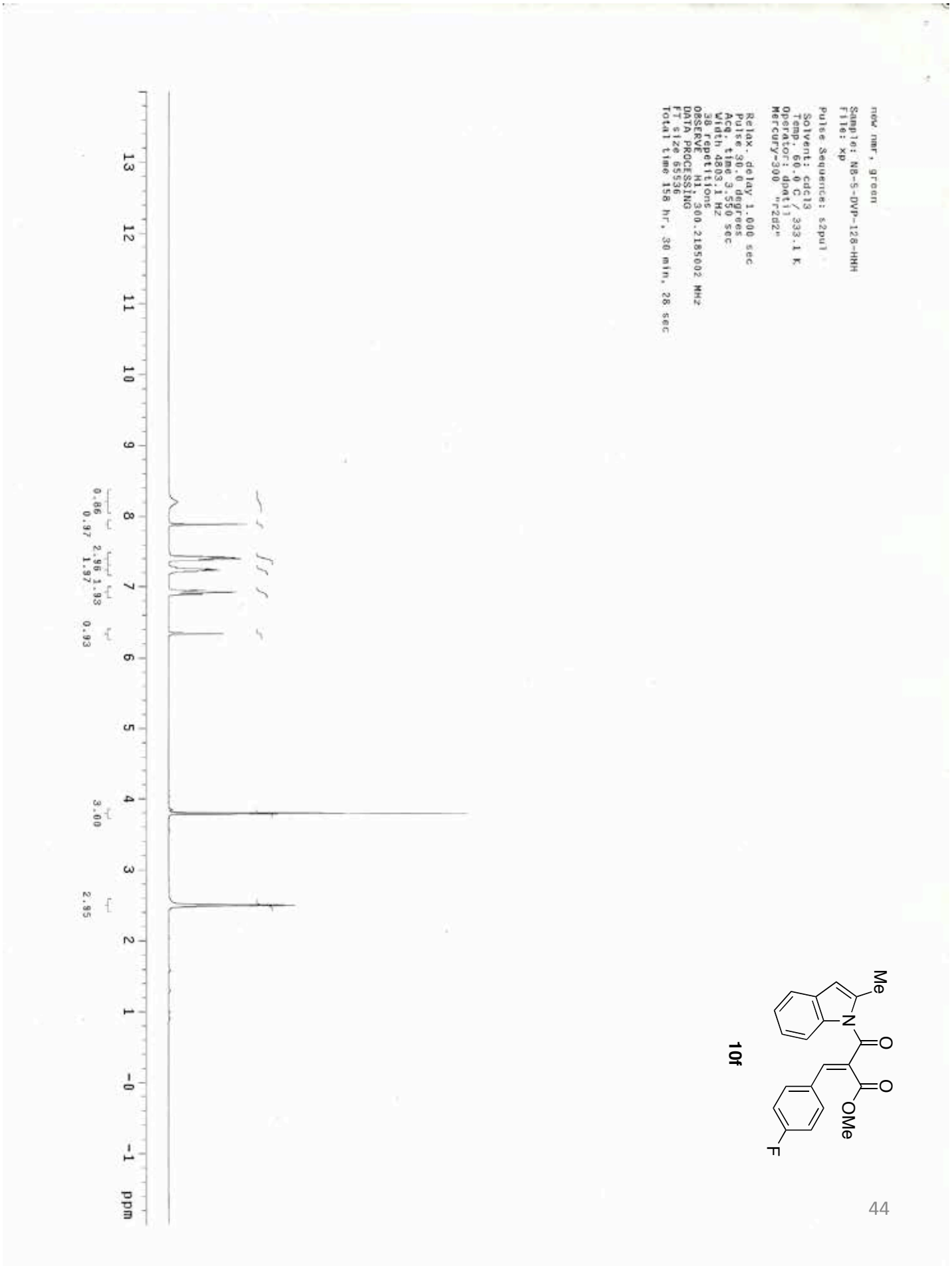
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
22 repetitions
OBSERVE H1, 300.2184986 MHz
DATA PROCESSING
FT size 65536
Total time 15 hr, 51 min, 3 sec

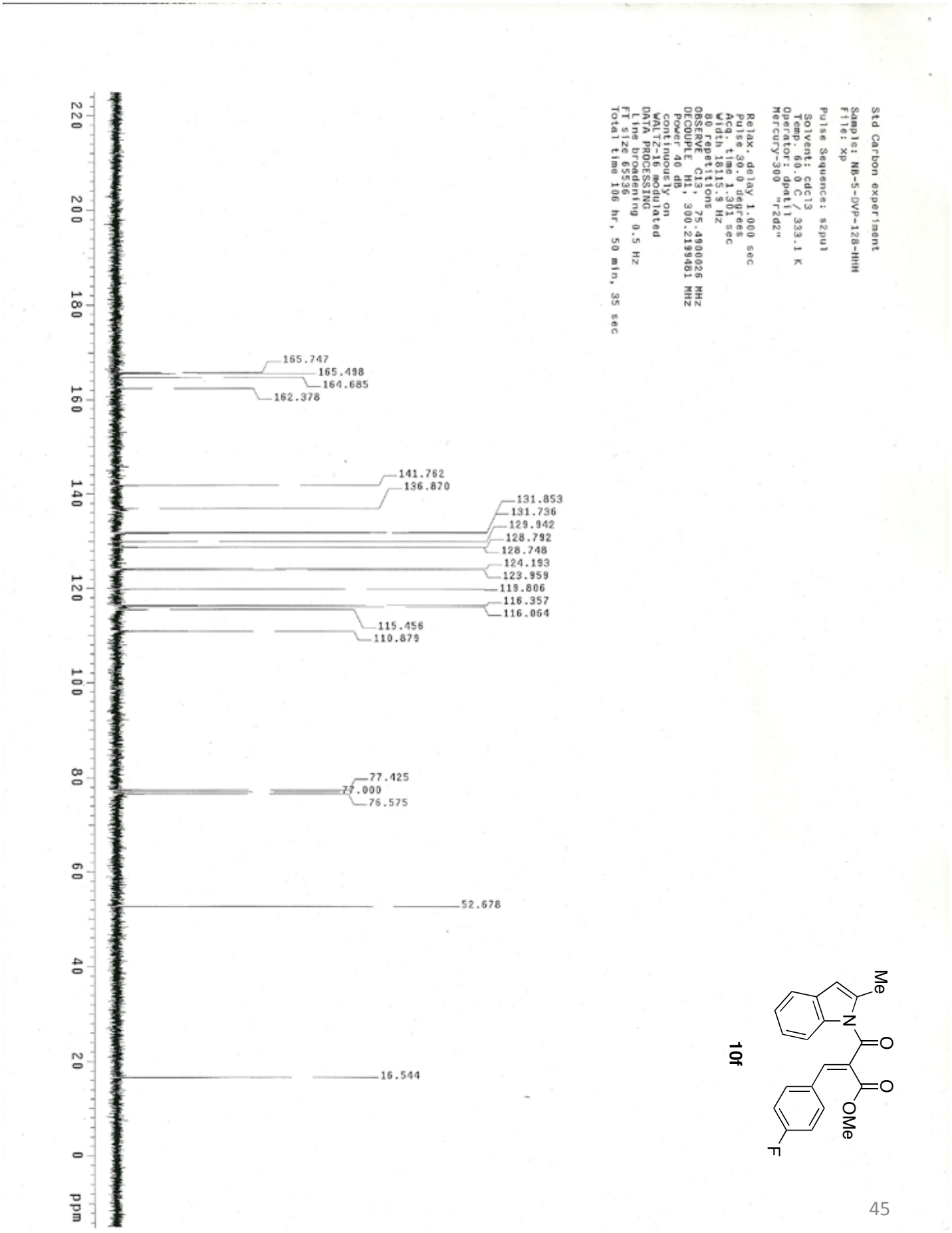


10e



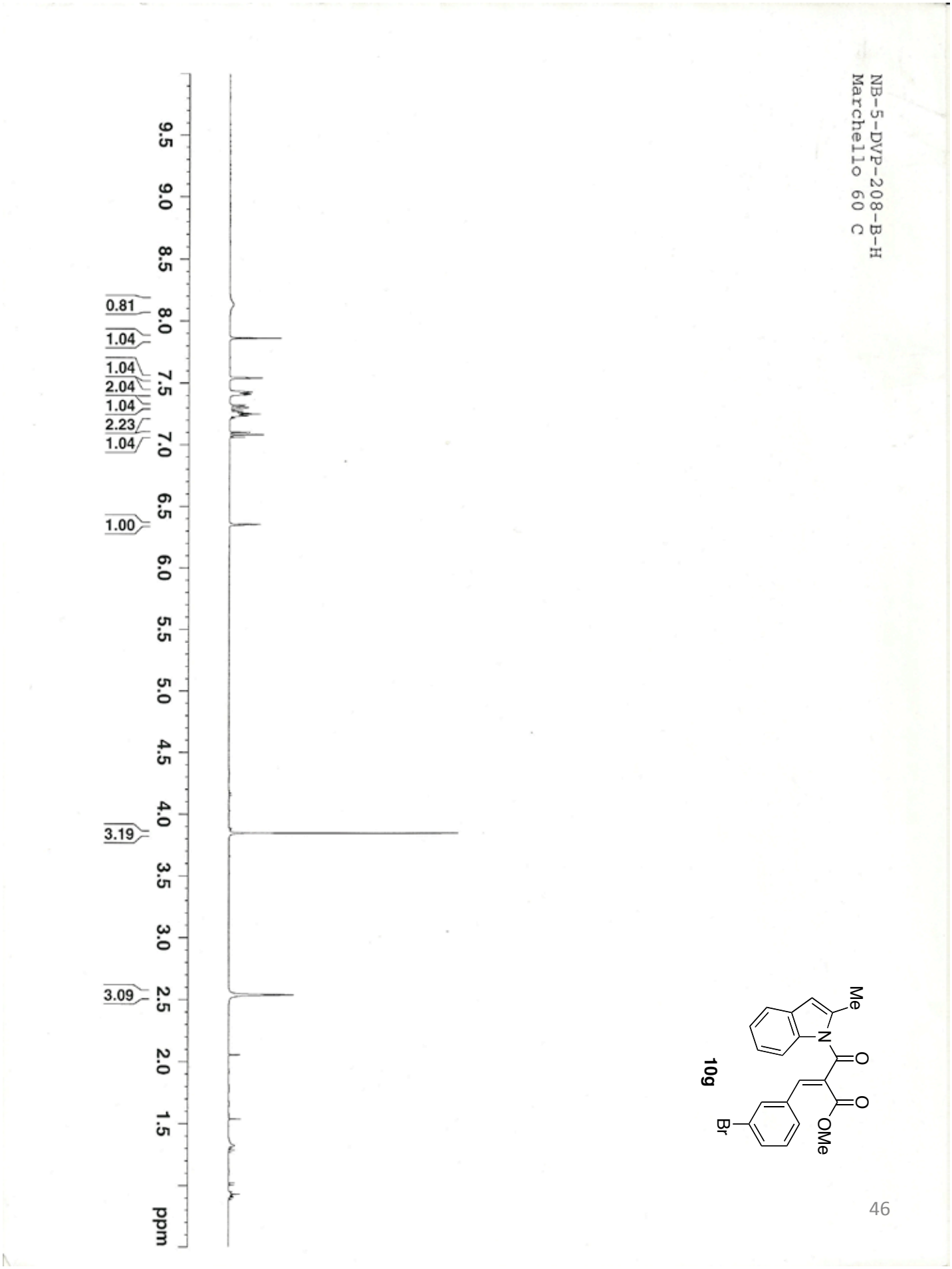

Std Carbon experiment
Sample: NB-5-OVP-207-A-H
file: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpatil
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 1.301 sec
Width 18115.9 Hz
44 repetitions
OBSERVE C13, 75.4900159 MHz
DECOUPLE H1, 300.2199481 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 10 hr, 41 min, 3 sec
164.634
164.018
140.385
136.372
132.469
131.509
129.598
124.193
119.909
117.748
113.772
111.230
77.425
77.000
76.575
53.125
15.713
ppm
Me
N
O
O
OMe
CN
10e


new nmr, green
Sample: NB-5-DVP-128-HHH
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 60.0 C / 333.1 K
Operator: dpatil
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
38 repetitions
OBSERVE H1, 300.2185002 MHz
DATA PROCESSING
FT size 65536
Total time 158 hr, 30 min, 28 sec
13 12 11 10 9 8 7 6 5 4 3 2 1 -0 -1 ppm
0.86
0.97
2.96
1.93
1.97
0.93
3.00
2.95
Me
N
O
O
OMe
F
10f

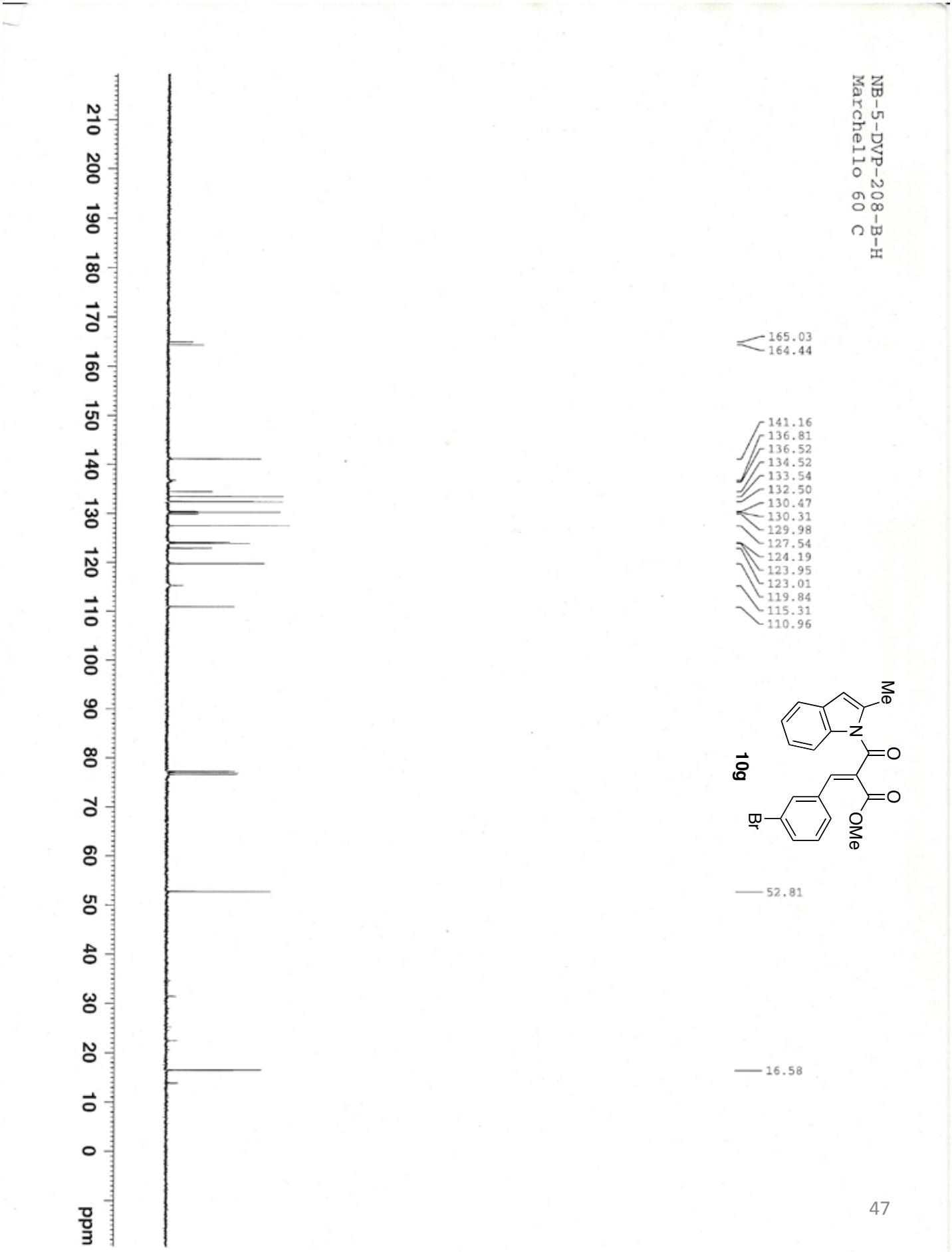

Std Carbon experiment
Sample: NB-5-DVP-128-HHH
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 60.0 C / 333.1 K
Operator: dpatil
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 1.301 sec
Width 18115.9 Hz
80 repetitions
OBSERVE C13, 75.4900028 MHz
DECOUPLE H1, 300.2199461 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 106 hr, 50 min, 35 sec
165.747
165.498
164.685
162.378
141.762
136.870
131.853
131.736
129.942
128.792
128.748
124.193
123.959
119.806
116.357
116.064
115.456
110.879
77.425
77.000
76.575
52.678
16.544
220
200
180
160
140
120
100
80
60
40
20
0
ppm

Me
N
O
O
OMe
F

10f


NB-5-DVP-208-B-H
Marchello 60 C
0.81
1.04
1.04
2.04
1.04
2.23
1.04
1.00
3.19
3.09
9.5
9.0
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
ppm
Me
N
O
O
OMe
Br

10g

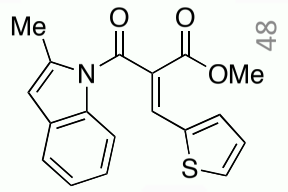

NB-5-DVP-208-B-H
Marchello 60 C
165.03
164.44
141.16
136.81
136.52
134.52
133.54
132.50
130.47
130.31
129.98
127.54
124.19
123.95
123.01
119.84
115.31
110.96
52.81
16.58
Me
N
O
O
OMe
Br
10g
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Std Proton parameters

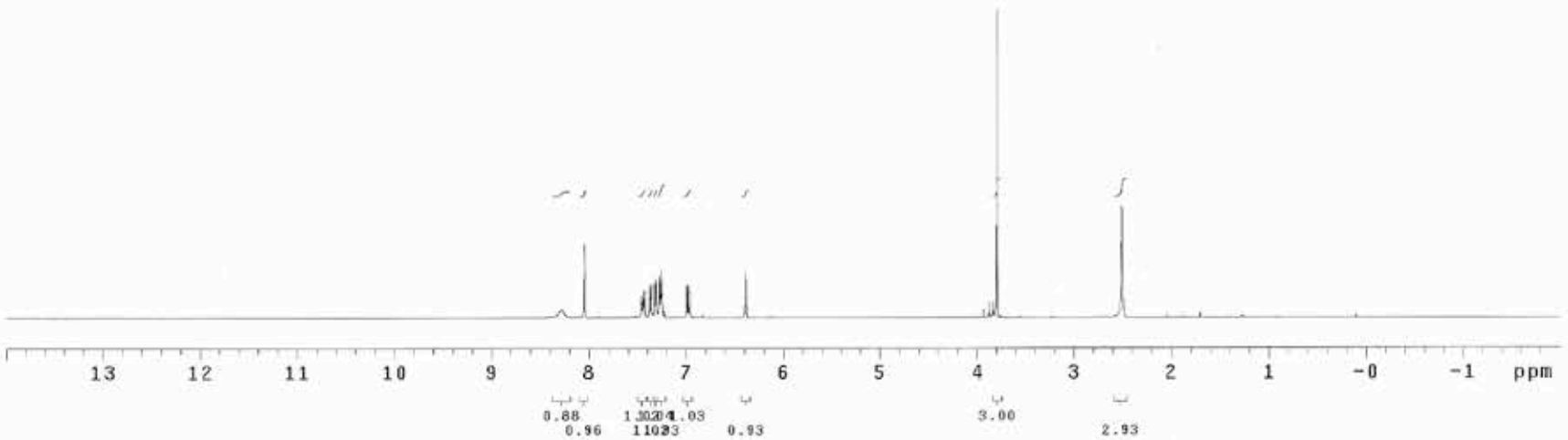
Sample: NB-5-DVP-208-A-H
File: xp

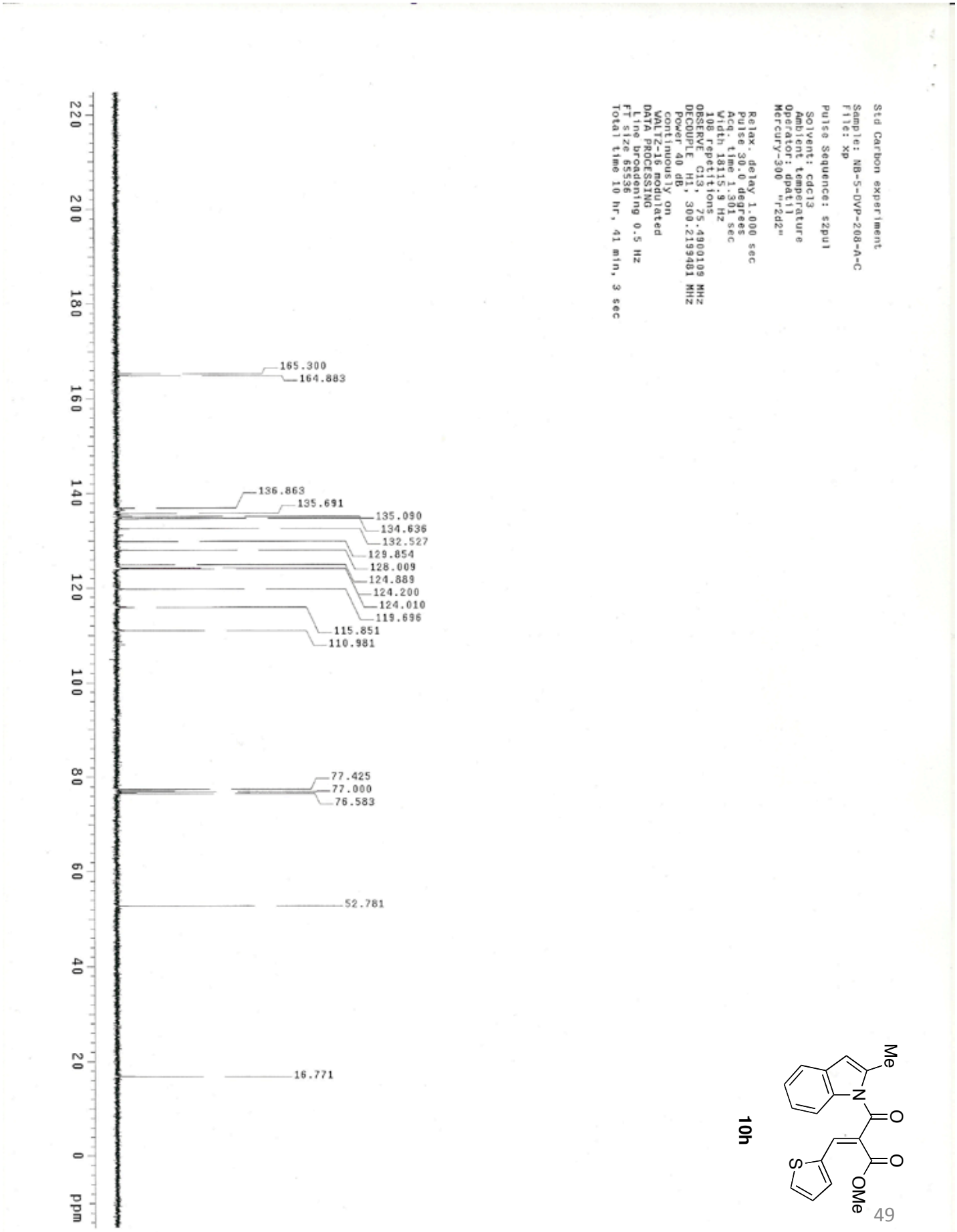
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpatil
Mercury-300 "r2d2"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
36 repetitions
OBSERVE H1, 300.2185002 MHz
DATA PROCESSING
FT size 65536
Total time 15 hr, 51 min, 3 sec

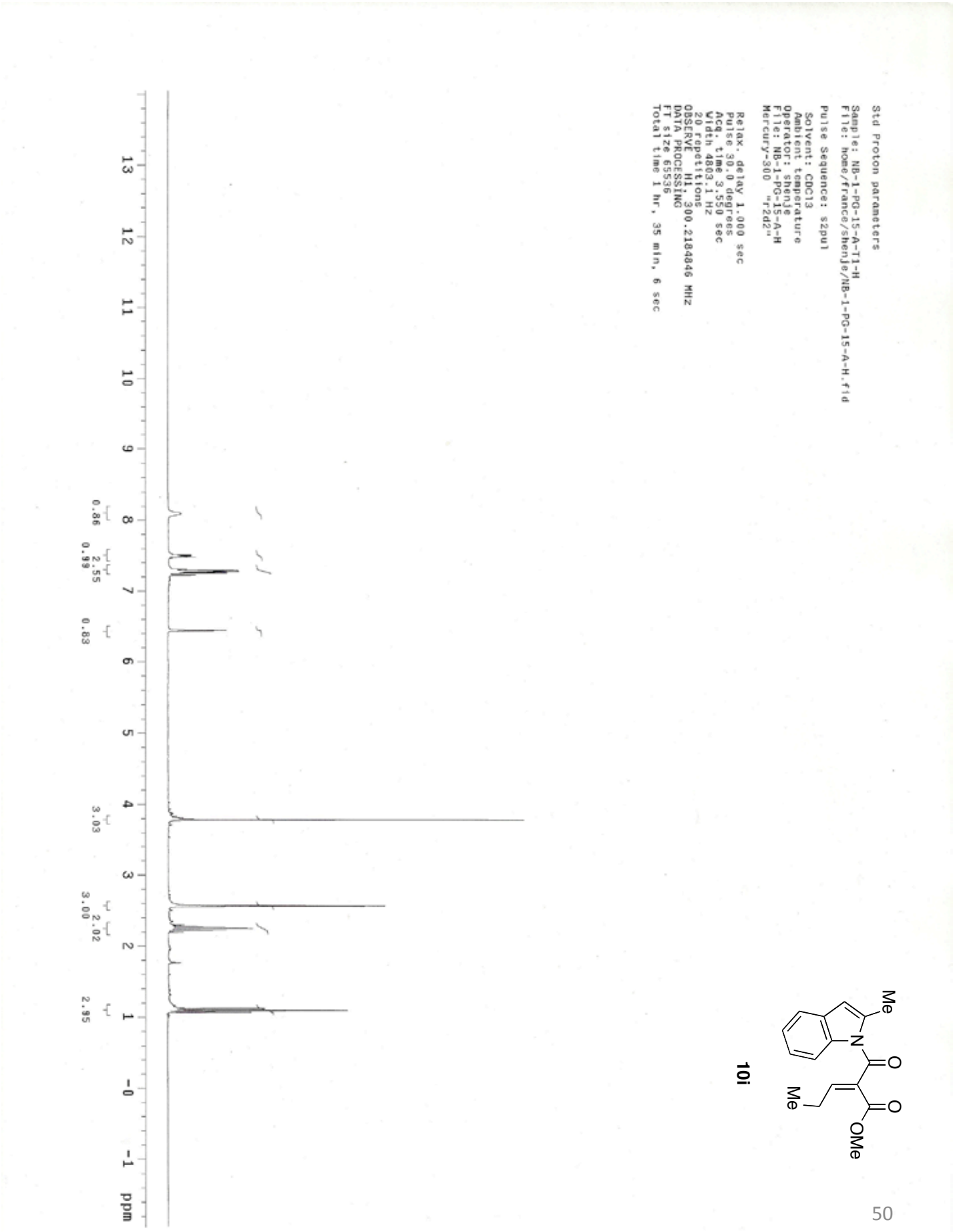


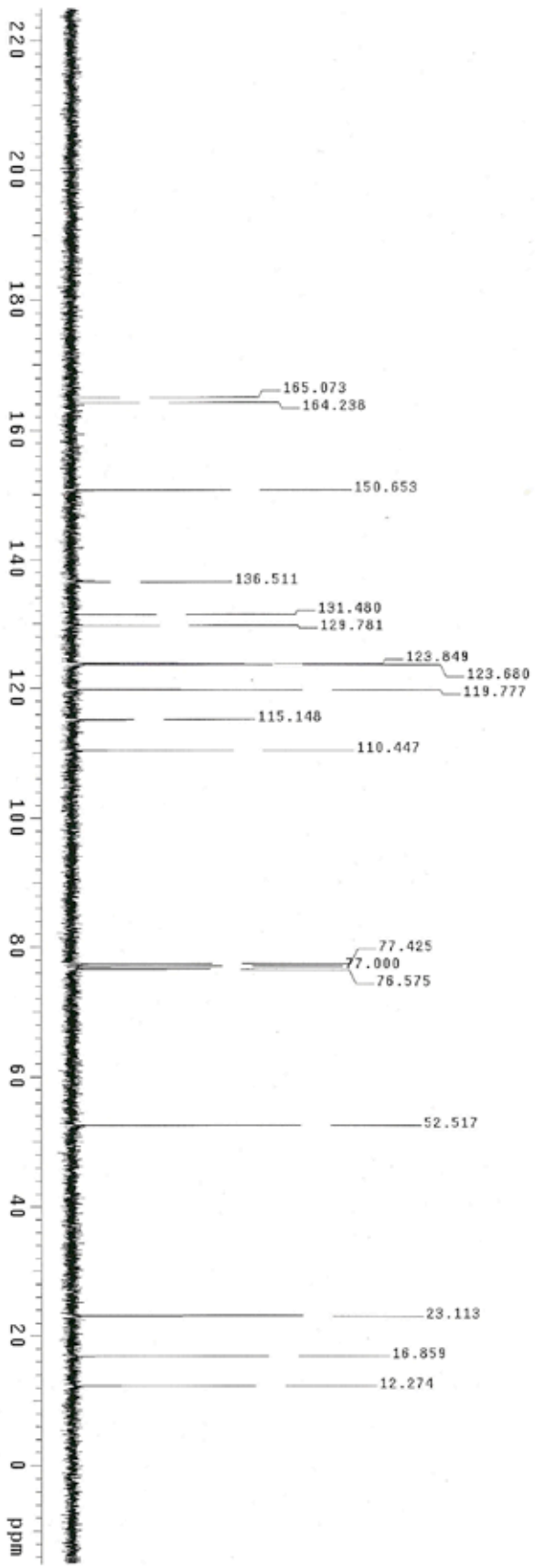
10h





10h

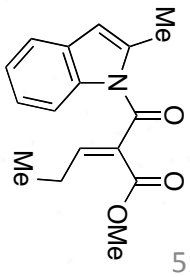

Std Proton parameters
Sample: NB-1-PG-15-A-T1-H
File: home/france/shenje/NB-1-PG-15-A-H.fid
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Operator: shenje
File: NB-1-PG-15-A-H
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
20 repetitions
OBSERVE H1, 300.2184846 MHz
DATA PROCESSING
FT size 65536
Total time 1 hr, 35 min, 6 sec
13
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
0.86
2.55
0.99
0.83
3.03
2.02
3.00
2.95
Me
N
O
O
O
Me
OMe
10i



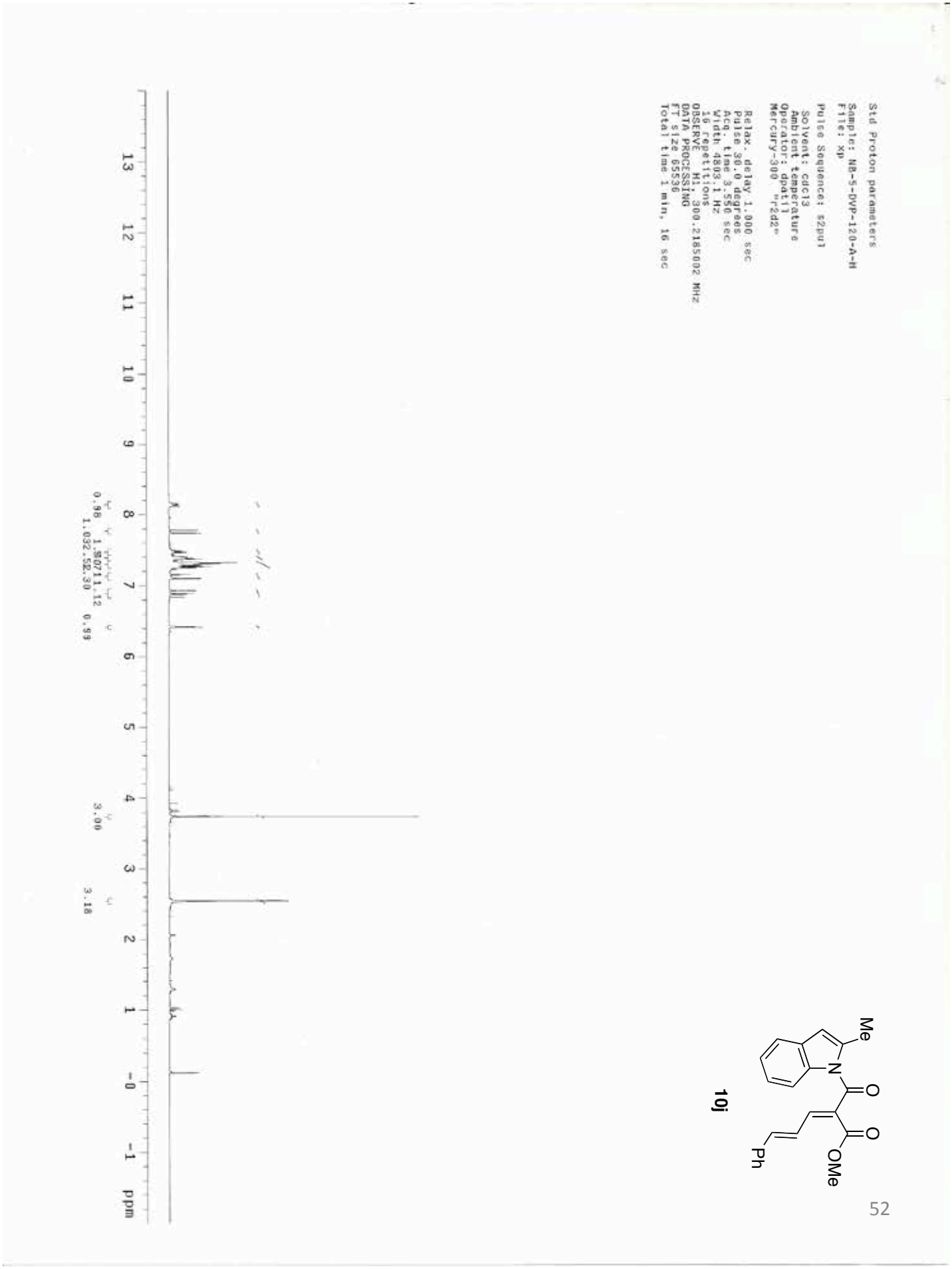
Std Carbon experiment
Sample: NB-1-PG-15-A-T1-H
File: xp

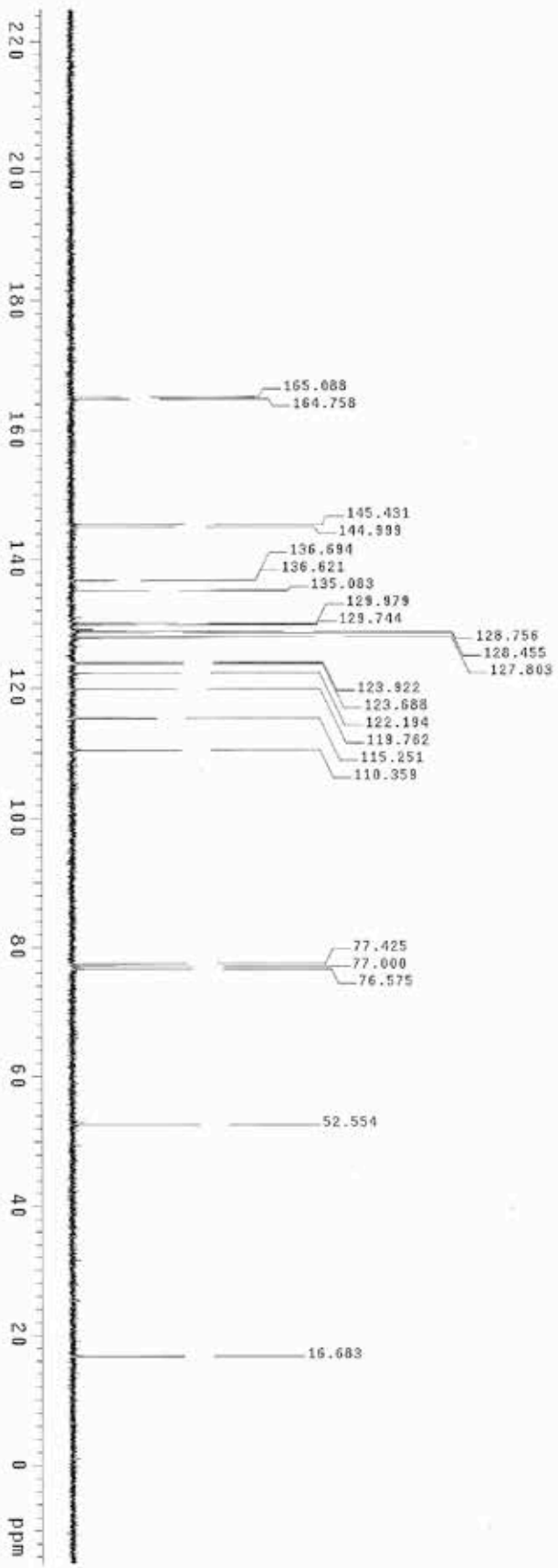
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Operator: shenje
Mercury-300 "r2d2"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 1.301 sec
Width 18115.9 Hz
90 repetitions
OBSERVE C13, 75.4900071 MHz
DECOUPLE H1, 300.2199481 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 4 min, 6 sec



10i


Std Proton parameters
Sample: NB-S-DVP-120-A-H
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpatil
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
16 repetitions
OBSERVE H1, 300.2185002 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 16 sec
13
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
0.98
1.00
1.032.52.30
0.99
3.00
3.18
Me
N
O
O
O
OMe
Ph
10j

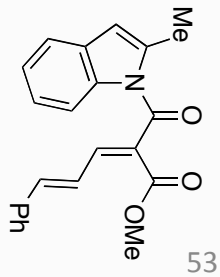


Std Carbon experiment

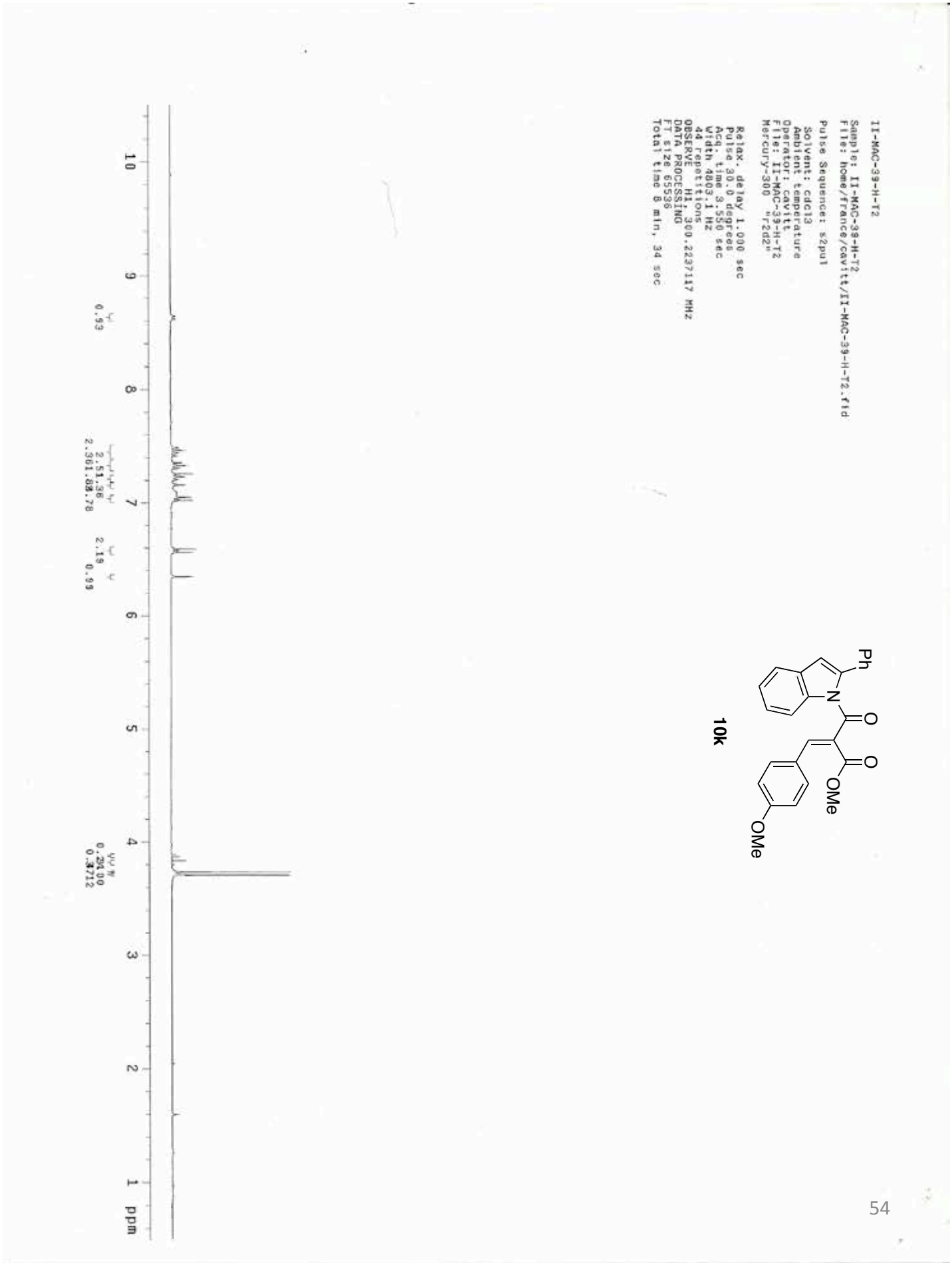
Sample: NB-5-DVP-120-A-H
File: xp

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpatil
Mercury-300 "r2d2"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 1.301 sec
Width 18115.9 Hz
96 repetitions
OBSERVE C13, 75.4900109 MHz
DECOUPLE H1, 300.2199481 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 10 min, 45 sec



10j

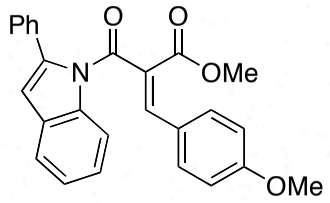

II-MAC-39-H-T2
Sample: II-MAC-39-H-T2
File: home/france/cavitt/II-MAC-39-H-T2.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: cavitt
File: II-MAC-39-H-T2
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
44 repetitions
OBSERVE H1, 300.2237117 MHz
DATA PROCESSING
FT size 65536
Total time 8 min, 34 sec
Ph
N
O
O
OMe
OMe
10k
10
9
8
7
6
5
4
3
2
1
ppm
0.93
2.51
2.36
2.30
1.88
1.78
2.19
0.99
0.29
0.37
00
12

II-MAC-39-C

Sample: II-MAC-39-C
File: xp

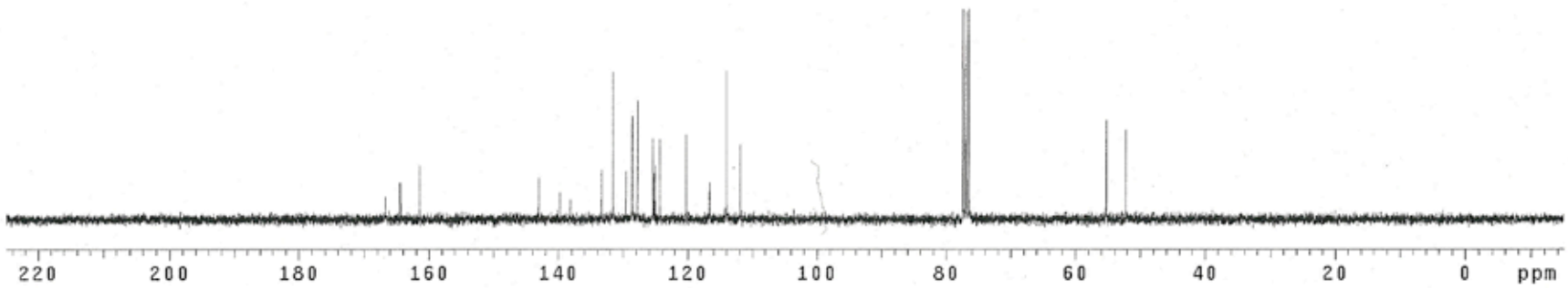
Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
Operator: cavitt
Mercury-300 "r2d2"

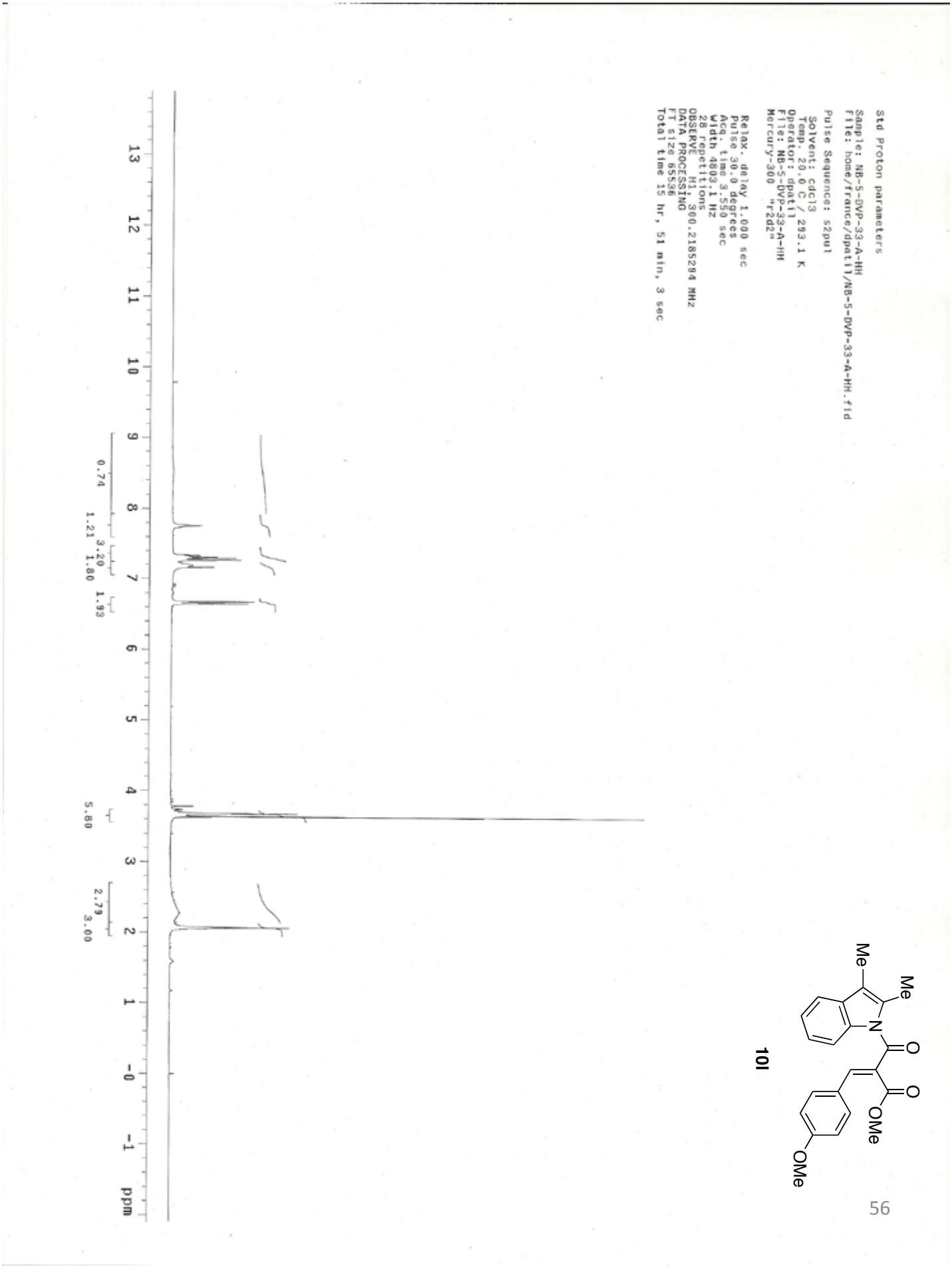
 Relax. delay 1.000 sec
 Pulse 30.0 degrees
 Acq. time 1.301 sec
 Width 18115.9 Hz
 232 repetitions
OBSERVE C13, 75.4913151 MHz
DECOUPLE H1, 300.2251667 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 0.5 Hz
FT size 65536
Total time 858 hr, 47 sec

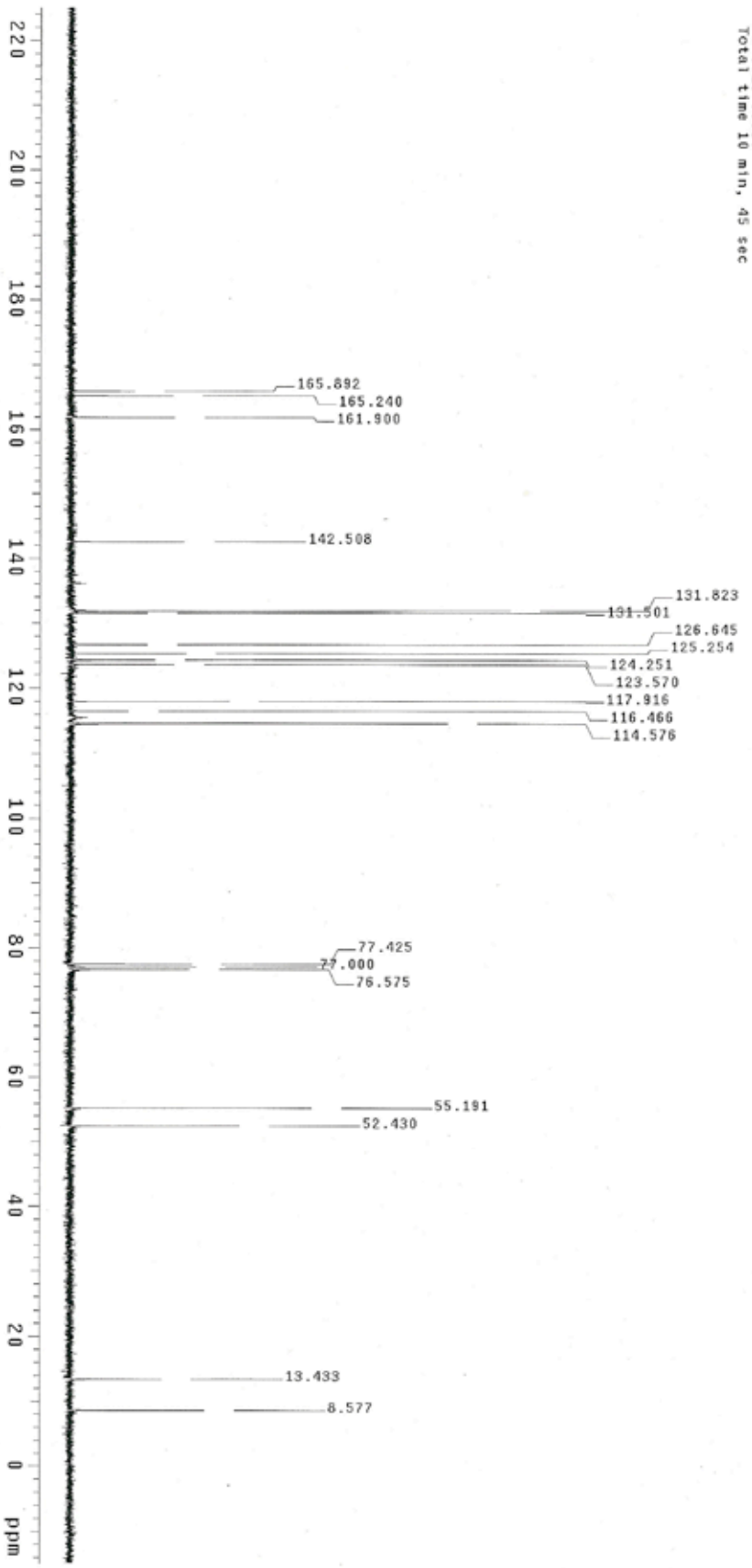


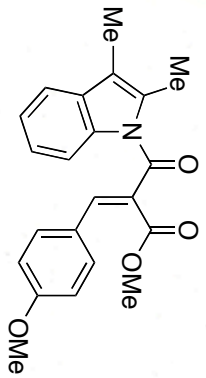
10k

INDEX	FREQUENCY	PPM	HEIGHT
1	12588.9	166.759	3.8
2	12411.4	164.408	6.1
3	12189.1	161.464	8.9
4	10799.3	143.053	6.9
5	10553.8	139.802	4.5
6	10433.3	138.205	3.3
7	10069.5	133.386	8.2
8	9932.4	131.570	24.4
9	9782.0	129.578	8.0
10	9706.8	128.582	17.1
11	9644.9	127.762	19.7
12	9464.1	125.367	13.4
13	9452.0	125.206	7.6
14	9438.1	125.023	8.9
15	9383.4	124.298	13.2
16	9077.1	120.241	14.0
17	8804.6	116.630	6.1
18	8604.4	113.979	24.6
19	8447.4	111.899	12.4
20	5844.6	77.421	34.8
21	5812.5	76.996	34.4
22	5781.0	76.578	34.8
23	4171.6	55.260	16.4
24	3943.9	52.243	14.7

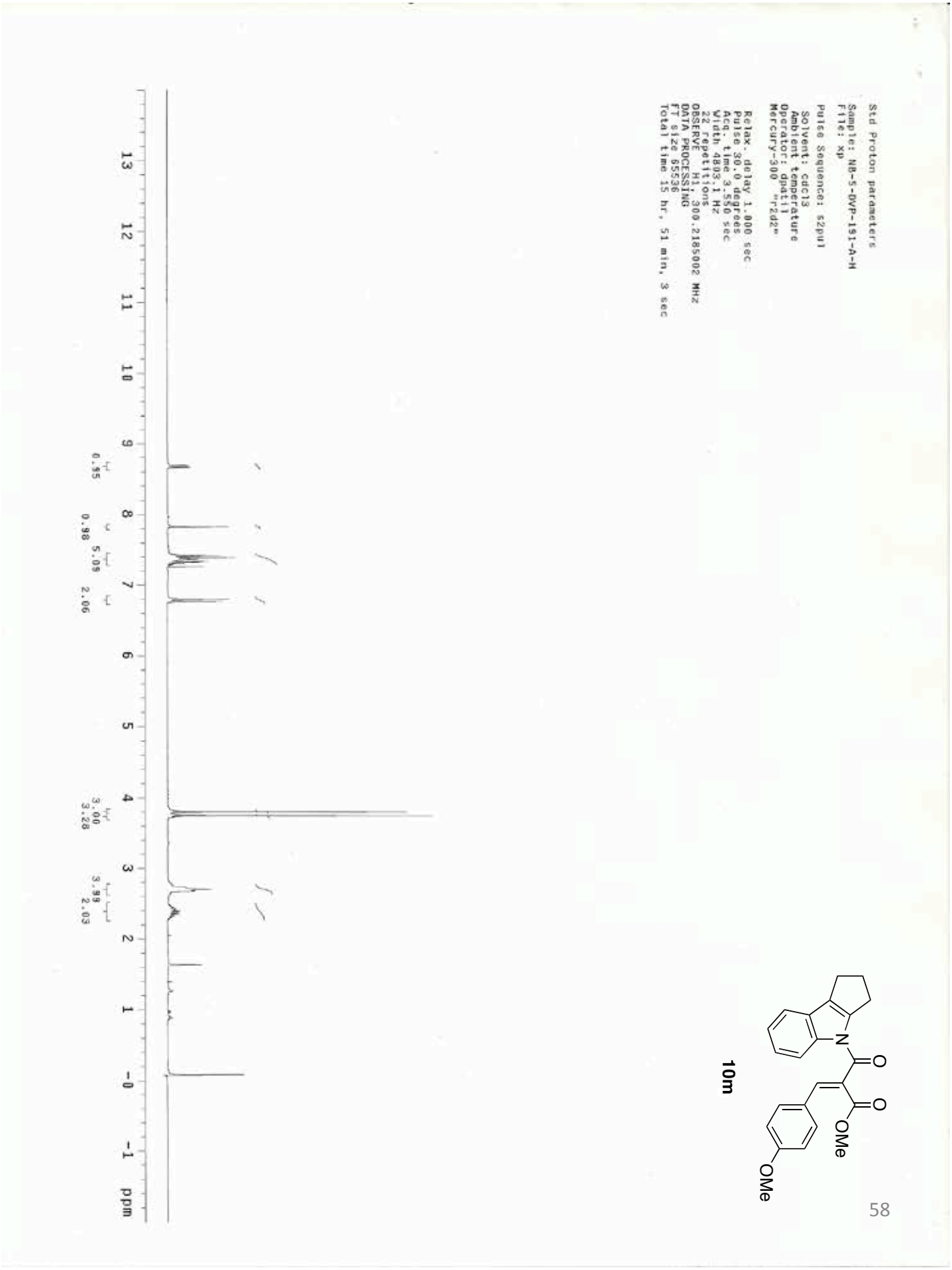



Std Proton parameters
Sample: NB-5-DVP-33-A-HH
File: home/france/dpatil/NB-5-DVP-33-A-HH.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp: 20.0 C / 293.1 K
Operator: dpatil
File: NB-5-DVP-33-A-HH
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
28 repetitions
OBSERVE H1, 300.2185294 MHz
DATA PROCESSING
FT size 65536
Total time 15 hr, 51 min, 3 sec
13
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
0.74
3.20
1.93
1.21
1.80
5.80
2.79
3.00
Me
Me
N
O
O
OMe
OMe
10l

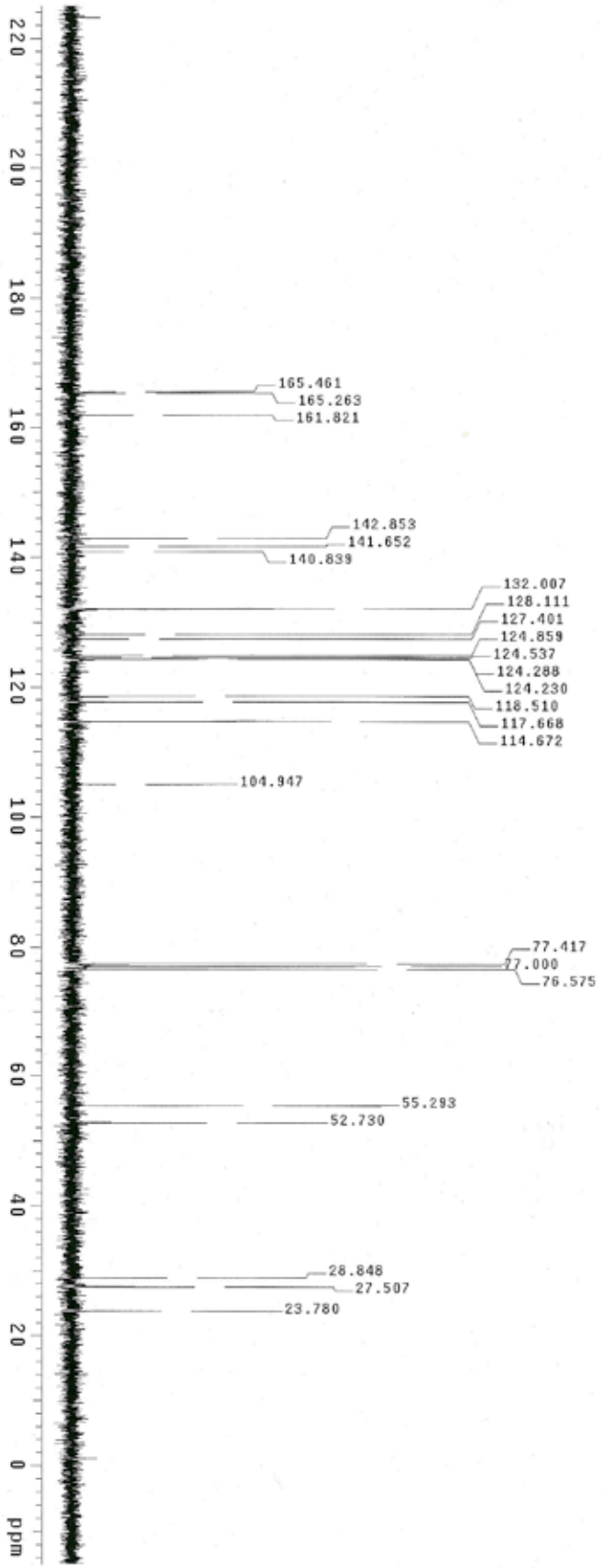

Std Carbon experiment
Sample: NB-5-DVP-33-A-H
File: home/france/dpatil/NB-5-DVP-33-A-C.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 60.0 C / 333.1 K
Operator: dpatil
File: NB-5-DVP-33-A-C
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 1.301 sec
Width 18115.9 Hz
112 repetitions
OBSERVE C13, 75.4913196 MHz
DECOUPLE H1, 300.2251667 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 10 min, 45 sec
165.892
165.240
161.900
142.508
131.823
131.501
126.645
125.254
124.251
123.570
117.916
116.466
114.576
77.425
77.000
76.575
55.191
52.430
13.433
8.577
220
200
180
160
140
120
100
80
60
40
20
0
ppm

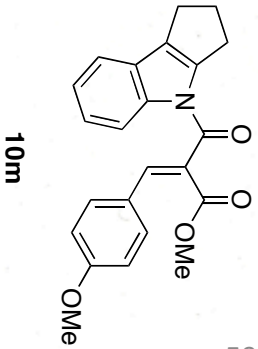

Me
Me
N
O
O
OMe
OMe

10l


Std Proton parameters
Sample: NB-S-DVP-191-A-H
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpatil
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
22 repetitions
OBSERVE H1, 300.2185002 MHz
DATA PROCESSING
FT size 65536
Total time 15 hr, 51 min, 3 sec
0.95
0.98
5.09
2.06
3.00
3.28
3.99
2.03
13
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
N
O
O
OMe
OMe

10m


Std Carbon experiment
Sample: NB-5-DVP-191-A-H
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpatil
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 1.301 sec
Width 18115.9 Hz
86 repetitions
OBSERVE C13, 75.4900059 MHz
DECOUPLE H1, 300.2199481 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 10 hr, 41 min, 3 sec
165.461
165.263
161.821
142.853
141.652
140.839
132.007
128.111
127.401
124.859
124.537
124.288
124.230
118.510
117.668
114.672
104.947
77.417
77.000
76.575
55.293
52.730
28.848
27.507
23.780
220
200
180
160
140
120
100
80
60
40
20
0
ppm


N
O
O
OMe
OMe

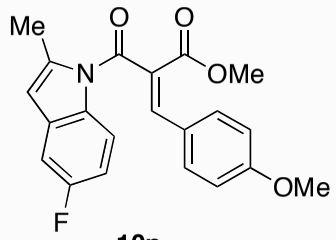
10m

Std Proton parameters

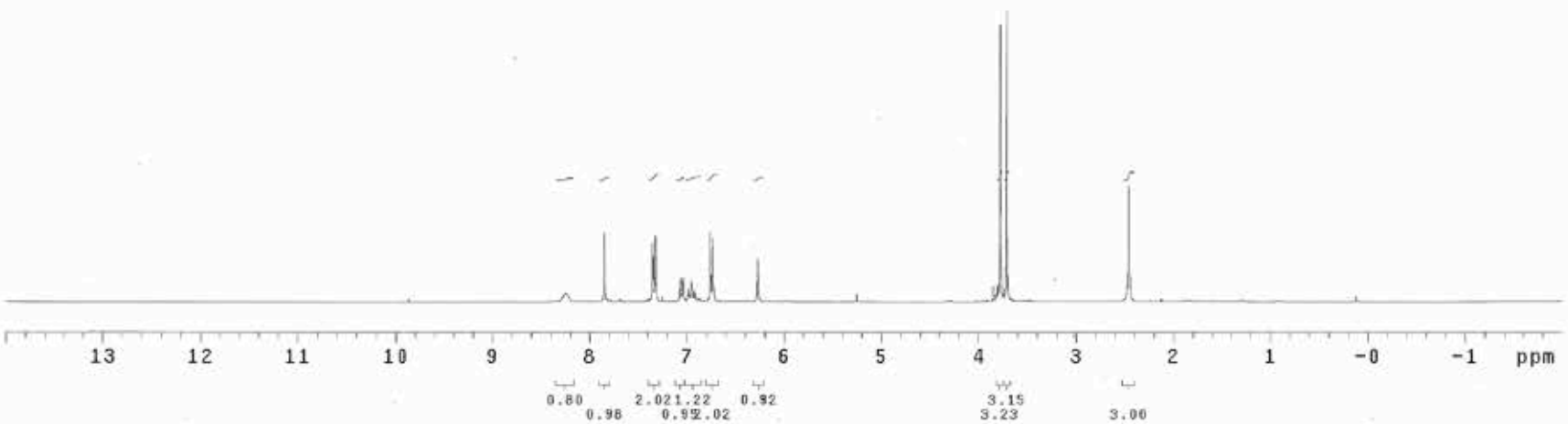
Sample: N8-5-DVP-140-8-MH
File: xp

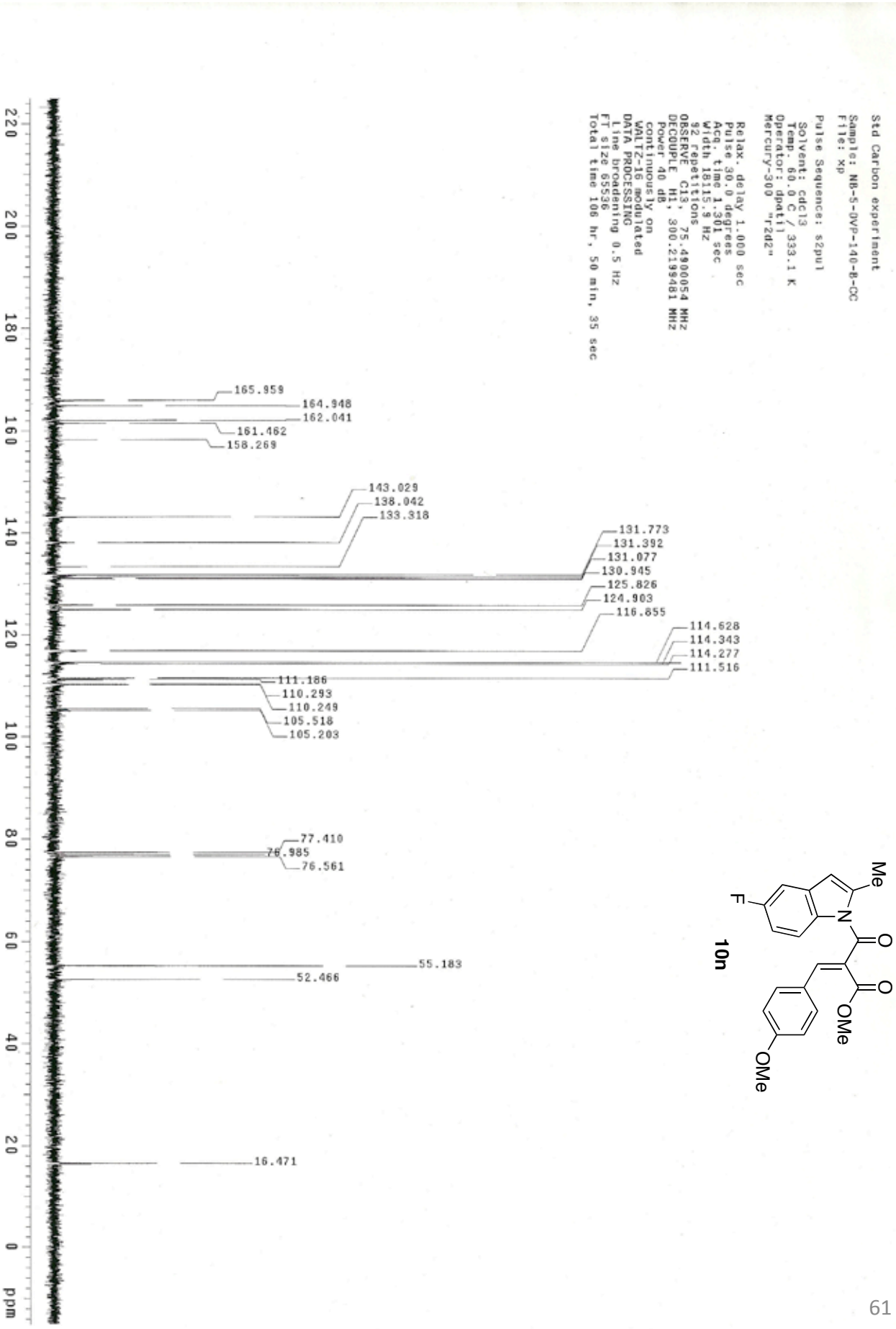
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 60.0 C / 333.1 K
Operator: dpatil
Mercury-300 "r2d2"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
22 repetitions
OBSERVE H1, 300.2185002 MHz
DATA PROCESSING
FT size 65536
Total time 158 hr, 30 min, 28 sec



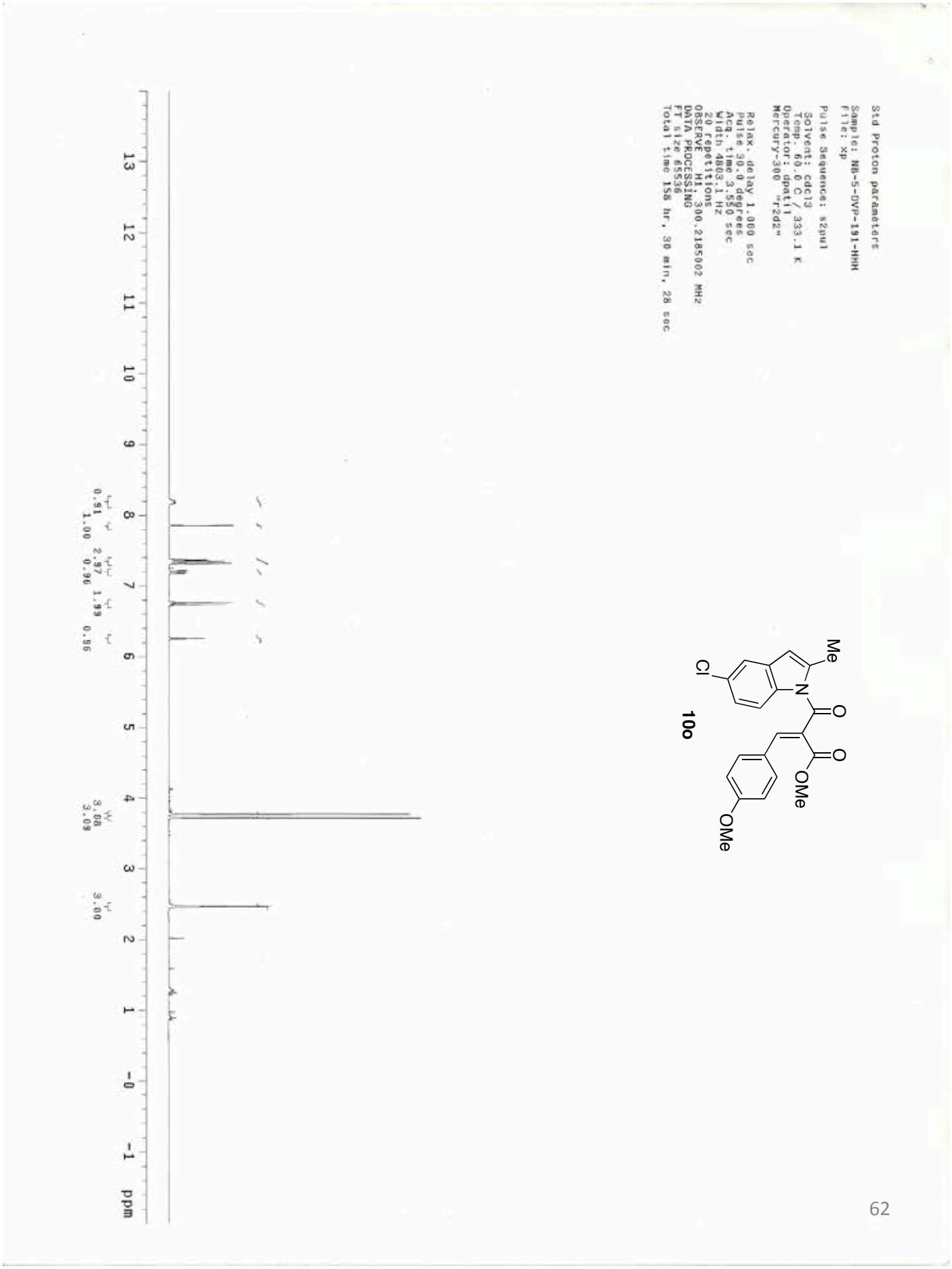
10n

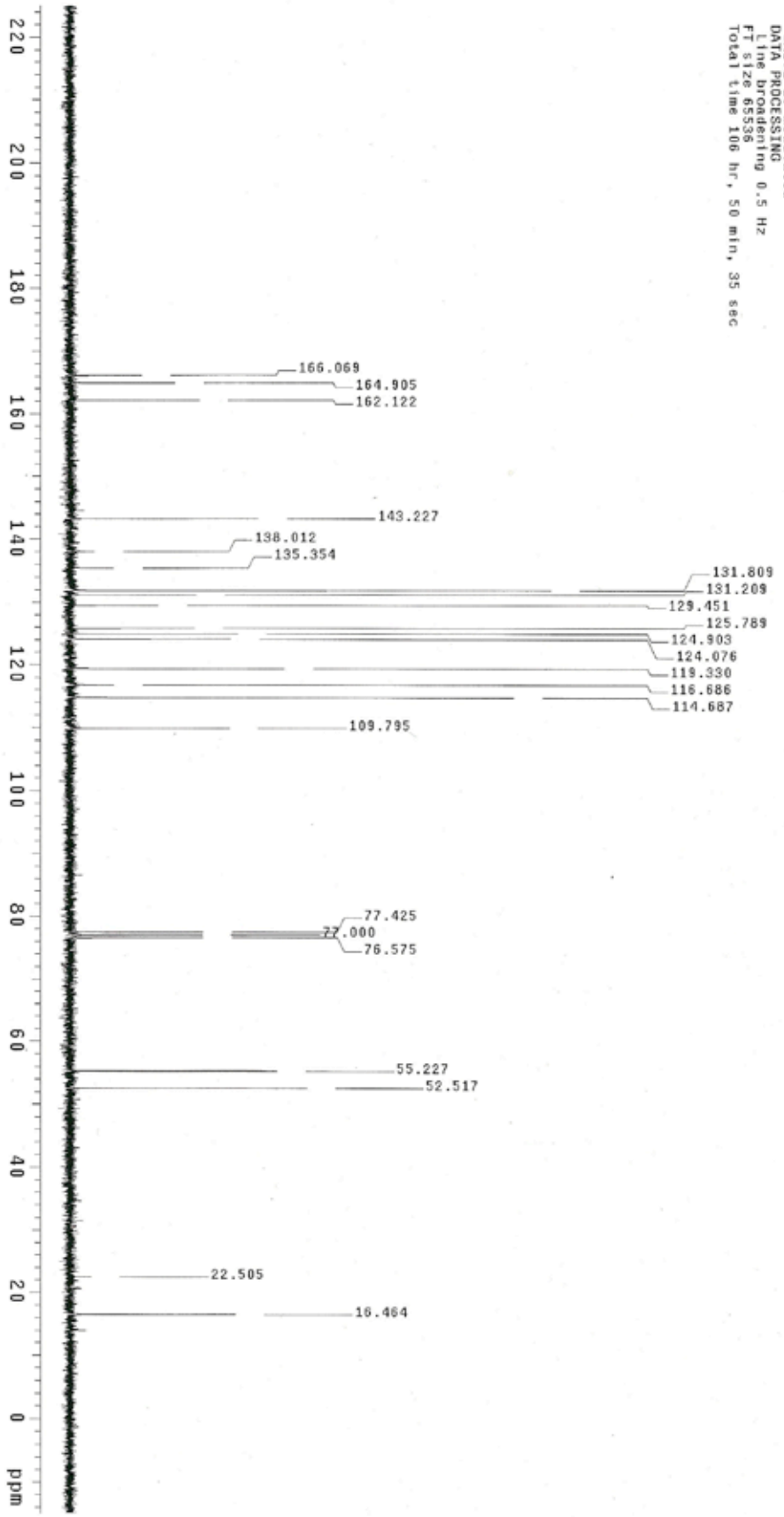


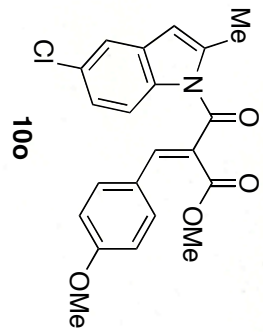

Std Carbon experiment
Sample: NB-5-OVP-140-8-CC
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Temp: 60.0 C / 333.1 K
Operator: dpatil
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 1.301 sec
Width 18115.9 Hz
32 repetitions
OBSERVE C13, 75.4900054 MHz
DECOUPLE H1, 300.2199481 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 106 hr, 50 min, 35 sec
165.959
164.948
162.041
161.462
158.269
143.029
138.042
133.818
131.773
131.392
131.077
130.945
125.826
124.903
116.855
114.628
114.343
114.277
111.516
111.186
110.293
110.249
105.518
105.203
77.410
76.985
76.561
55.183
52.466
16.471
220
200
180
160
140
120
100
80
60
40
20
0
ppm

Me
F
N
O
O
OMe
OMe

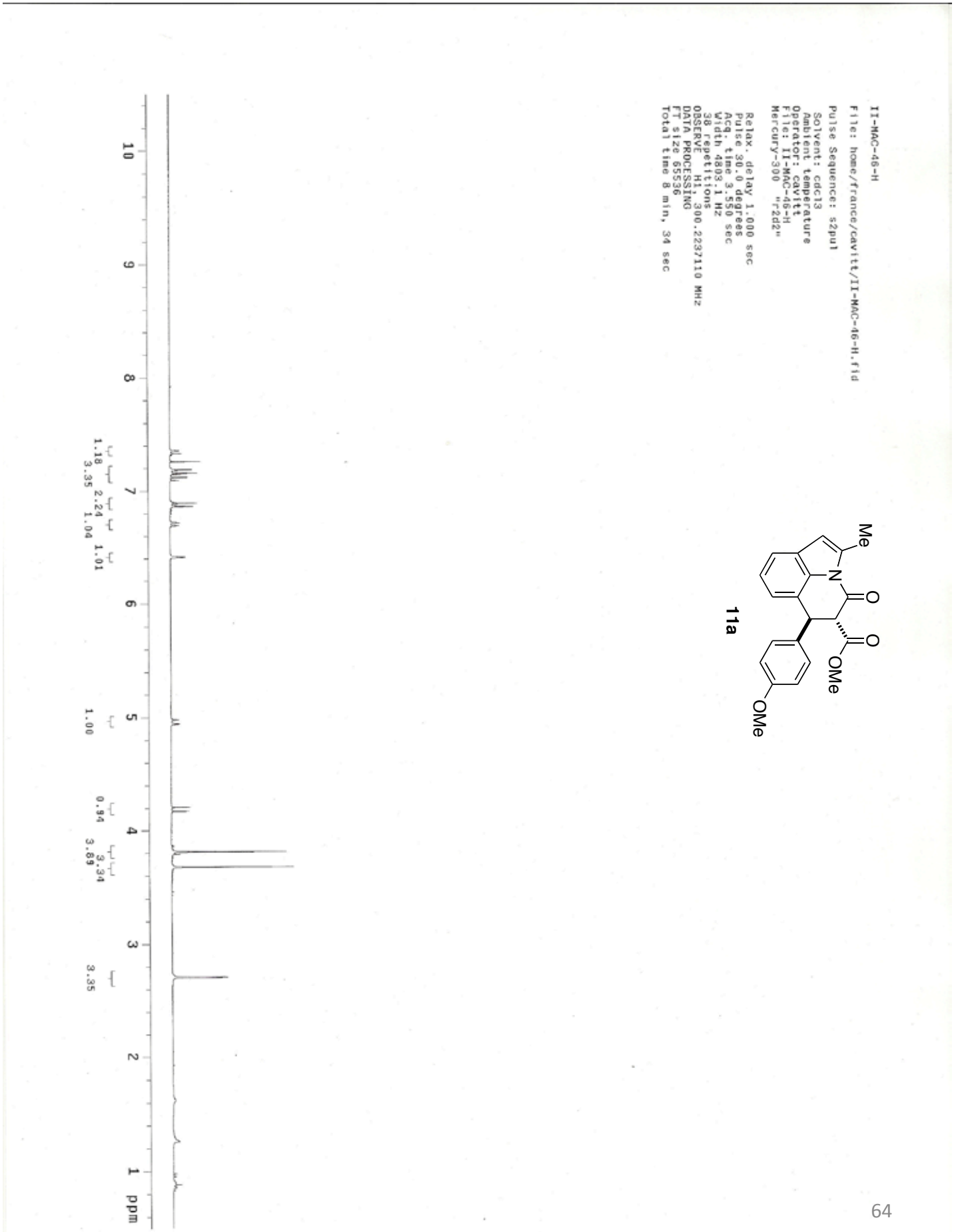
10n


Std Proton parameters
Sample: MB-5-DVP-191-HHH
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 60.0 C / 333.1 K
Operator: dpatil
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
20 repetitions
OBSERVE H1, 300.2185002 MHz
DATA PROCESSING
FT size 65536
Total time 15h hr, 30 min, 28 sec
Me
Cl
N
O
O
OMe
OMe
10o
13
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
0.91
1.00
2.97
0.96
1.99
0.96
3.08
3.03
3.00


Std Carbon experiment
Sample: NB-5-DVP-191-C-HHH
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 60.0 C / 333.1 K
Operator: dpatil
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 1.301 sec
Width 18115.9 Hz
66 repetitions
OBSERVE C13, 75.4900026 MHz
DECOUPLE H1, 300.2199481 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 106 hr, 50 min, 35 sec
166.069
164.905
162.122
143.227
138.012
135.354
131.809
131.209
129.451
125.789
124.903
124.076
119.330
116.686
114.687
109.795
77.425
77.000
76.575
55.227
52.517
22.505
16.464
220
200
180
160
140
120
100
80
60
40
20
0
ppm


Me
Cl
N
O
O
O
OMe
OMe

10o

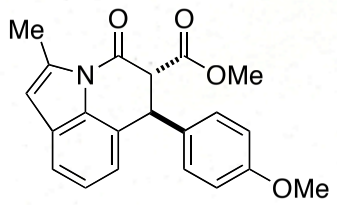

II-MAC-46-H
File: home/france/cavitt/II-MAC-46-H.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: cavitt
File: II-MAC-46-H
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
38 repetitions
OBSERVE H1, 300.2237110 MHz
DATA PROCESSING
FT size 65536
Total time 8 min, 34 sec
Me
N
O
O
OMe
OMe
11a
10
9
8
7
6
5
4
3
2
1
ppm
1.18
3.35
2.24
1.04
1.01
1.00
0.94
3.89
3.34
3.35

II-MAC-46-C

File: xp

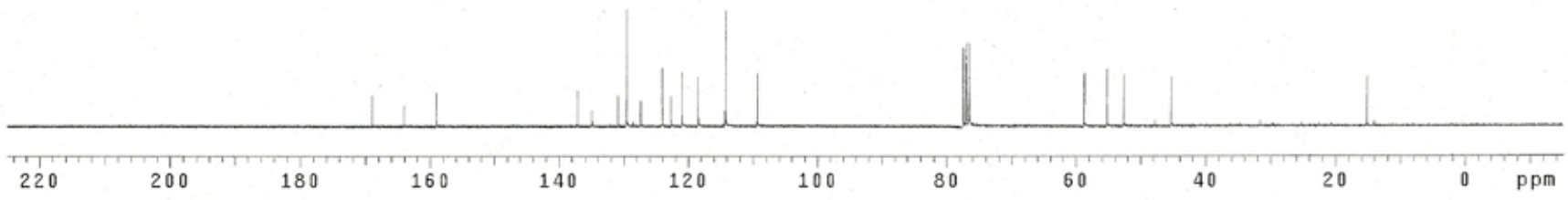
Pulse Sequence: s2pul
 Solvent: cdcl3
 Temp. 20.0 C / 293.1 K
Operator: cavitt
Mercury-300 "r2d2"

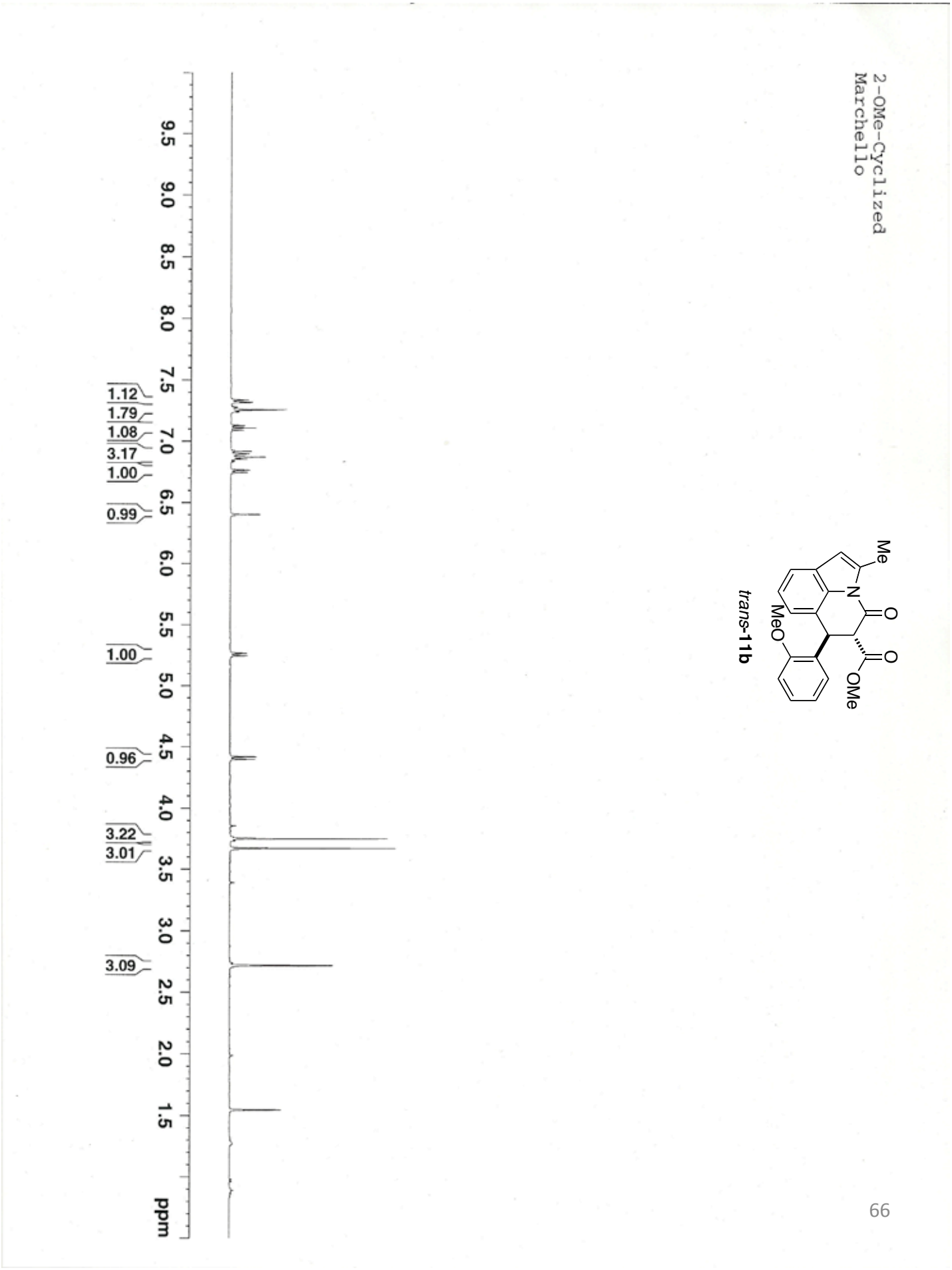
 Relax. delay 1.000 sec
 Pulse 30.0 degrees
 Acq. time 1.301 sec
 Width 18115.9 Hz
 1427 repetitions
OBSERVE C13, 75.4813159 MHz
DECOUPLE H1, 300.2251667 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 0.5 Hz
FT size 65536
Total time 858 hr, 47 sec

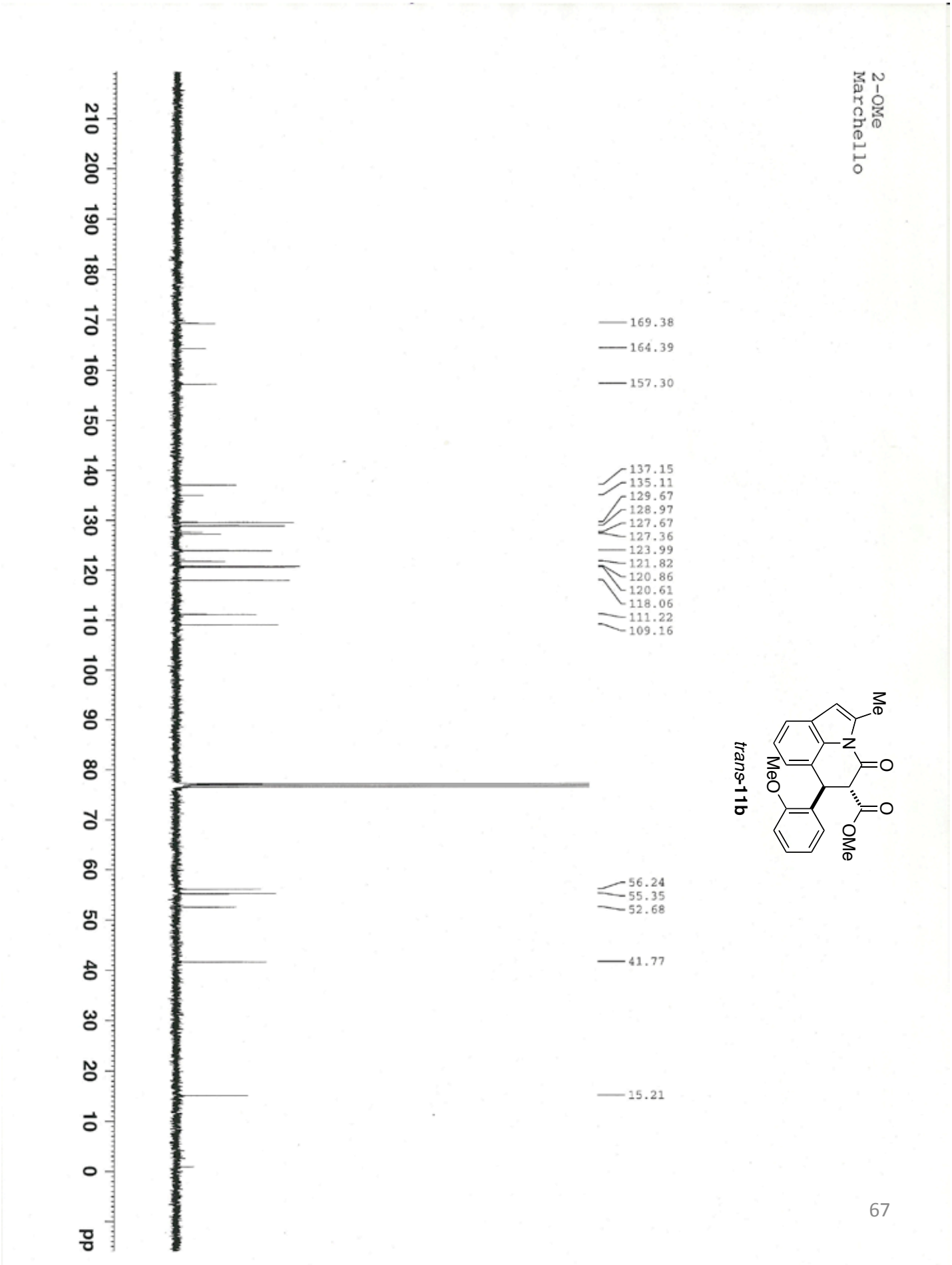


11a

INDEX	FREQUENCY	PPM	HEIGHT
1	12758.3	169.004	5.1
2	12383.5	164.039	3.4
3	12005.9	159.037	5.6
4	10355.6	137.177	5.8
5	10181.5	134.870	2.7
6	9882.9	130.915	5.0
7	9780.7	129.560	19.3
8	9617.0	127.392	4.2
9	9364.4	124.046	9.5
10	9260.4	122.669	4.9
11	9131.6	120.962	8.8
12	8941.4	118.443	8.1
13	8625.2	114.254	18.9
14	8255.9	109.362	8.7
15	5844.9	77.425	12.8
16	5812.8	77.000	13.6
17	5780.8	76.575	13.5
18	4436.8	58.772	8.7
19	4167.5	55.205	9.4
20	3973.5	52.635	8.5
21	3421.7	45.326	8.1
22	1145.6	15.176	8.1




2-OMe-Cyclized
Marchello
Me
N
O
O
MeO
OMe
trans-11b
9.5
9.0
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
ppm
1.12
1.79
1.08
3.17
1.00
0.99
1.00
0.96
3.22
3.01
3.09


2-OMe
Marchello
169.38
164.39
157.30
137.15
135.11
129.67
128.97
127.67
127.36
123.99
121.82
120.86
120.61
118.06
111.22
109.16
56.24
55.35
52.68
41.77
15.21
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm
Me
N
O
O
MeO
OMe

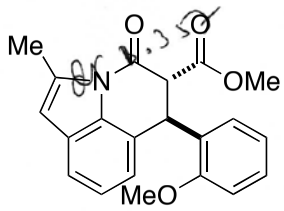
trans-**11b**

2-methoxy benzaldehyde derived !!

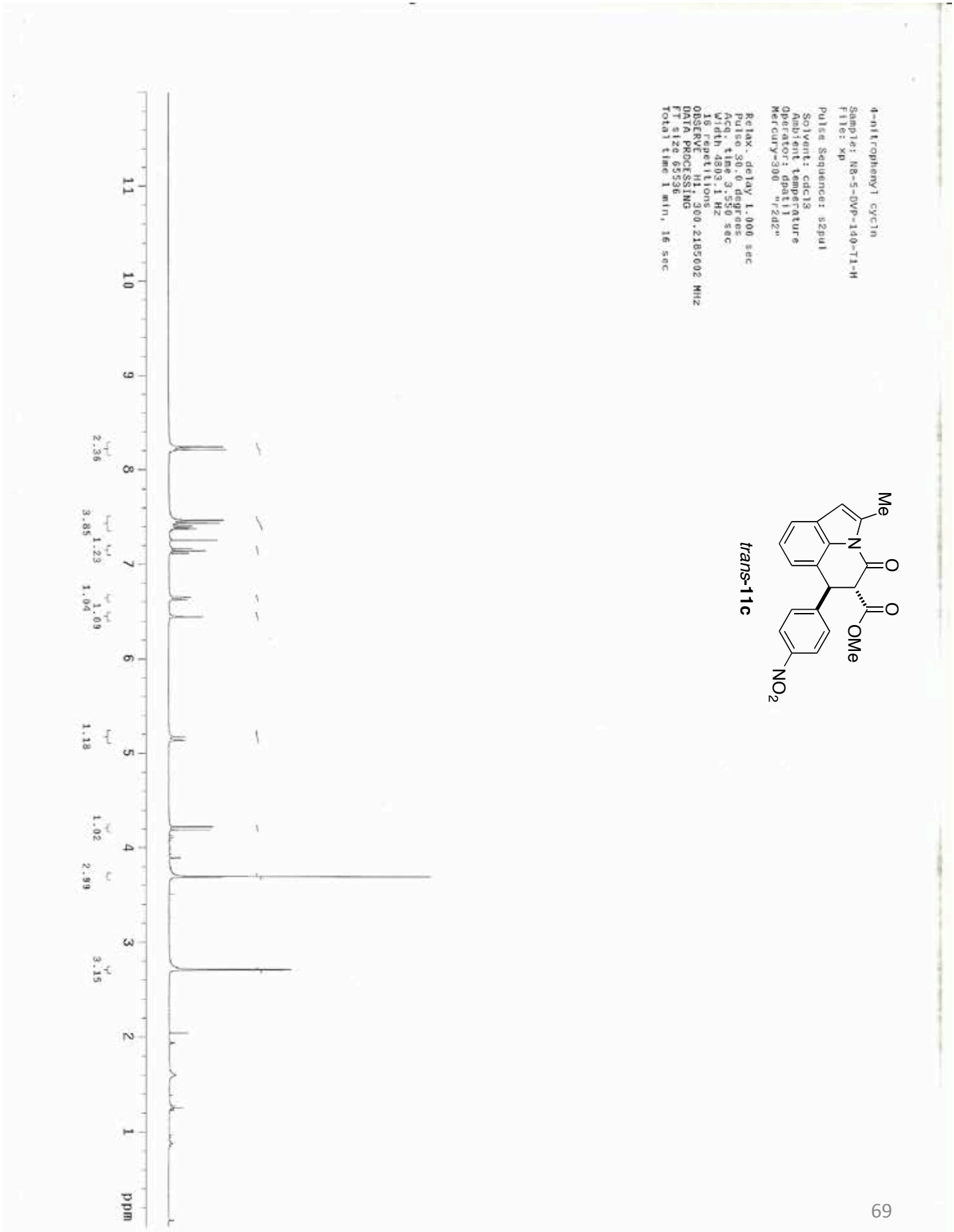
Sample: NB-1-PG-20-D-crude
File: xp

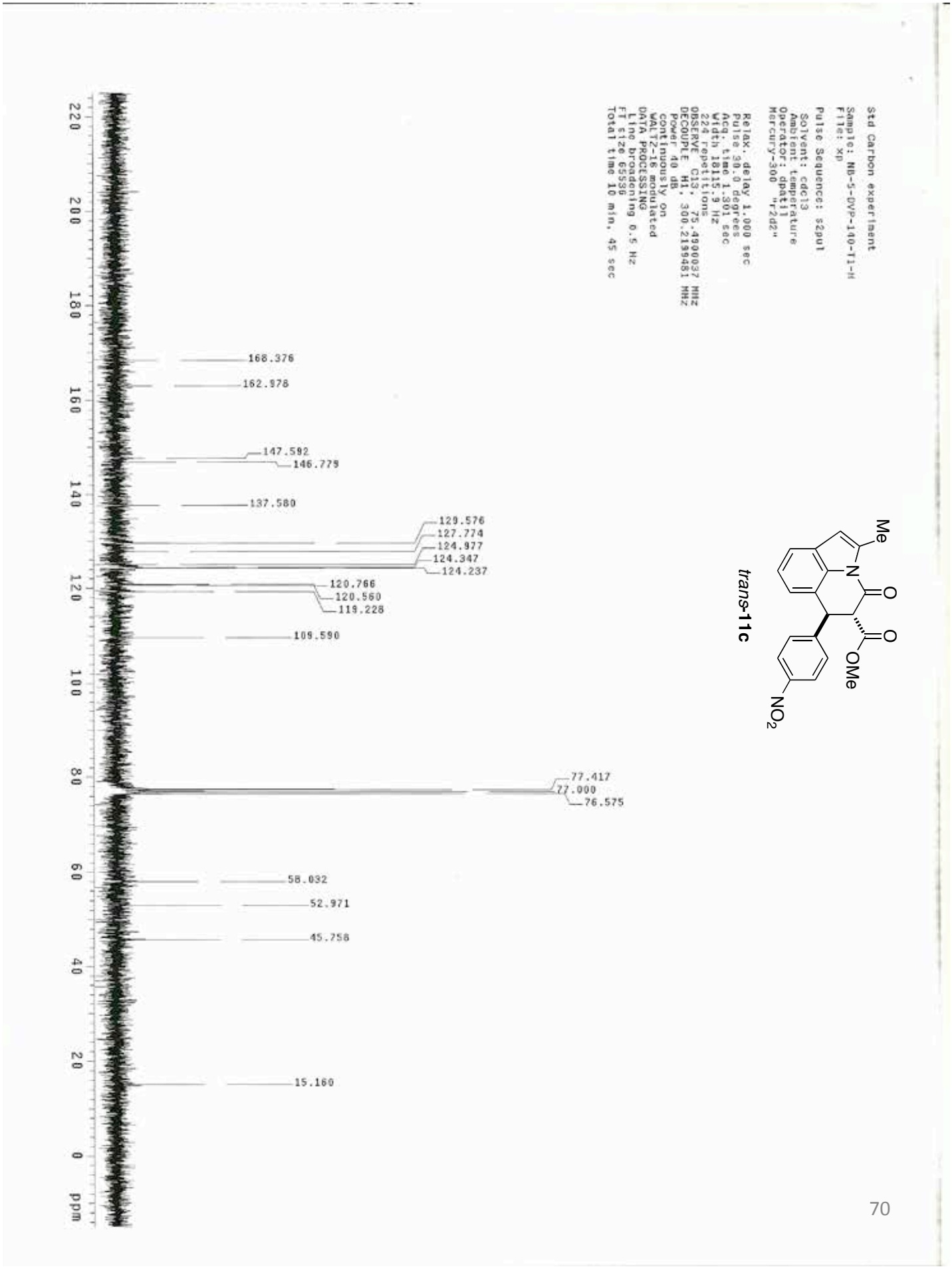
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpatil
Mercury-300 "r2d2"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
48 repetitions
OBSERVE H1, 300.2185210 MHz
DATA PROCESSING
FT size 65536
Total time 15 hr, 51 min, 3 sec

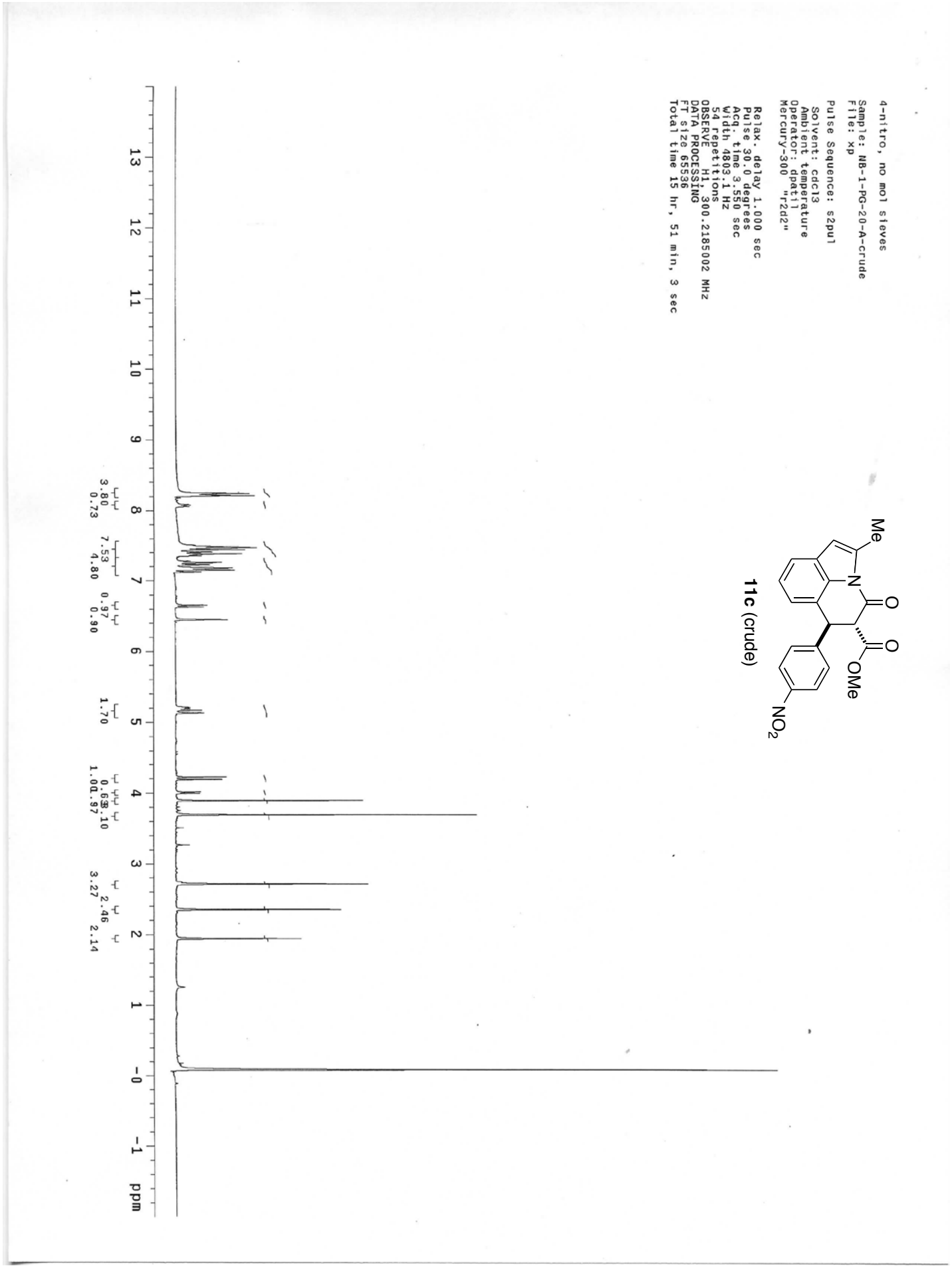


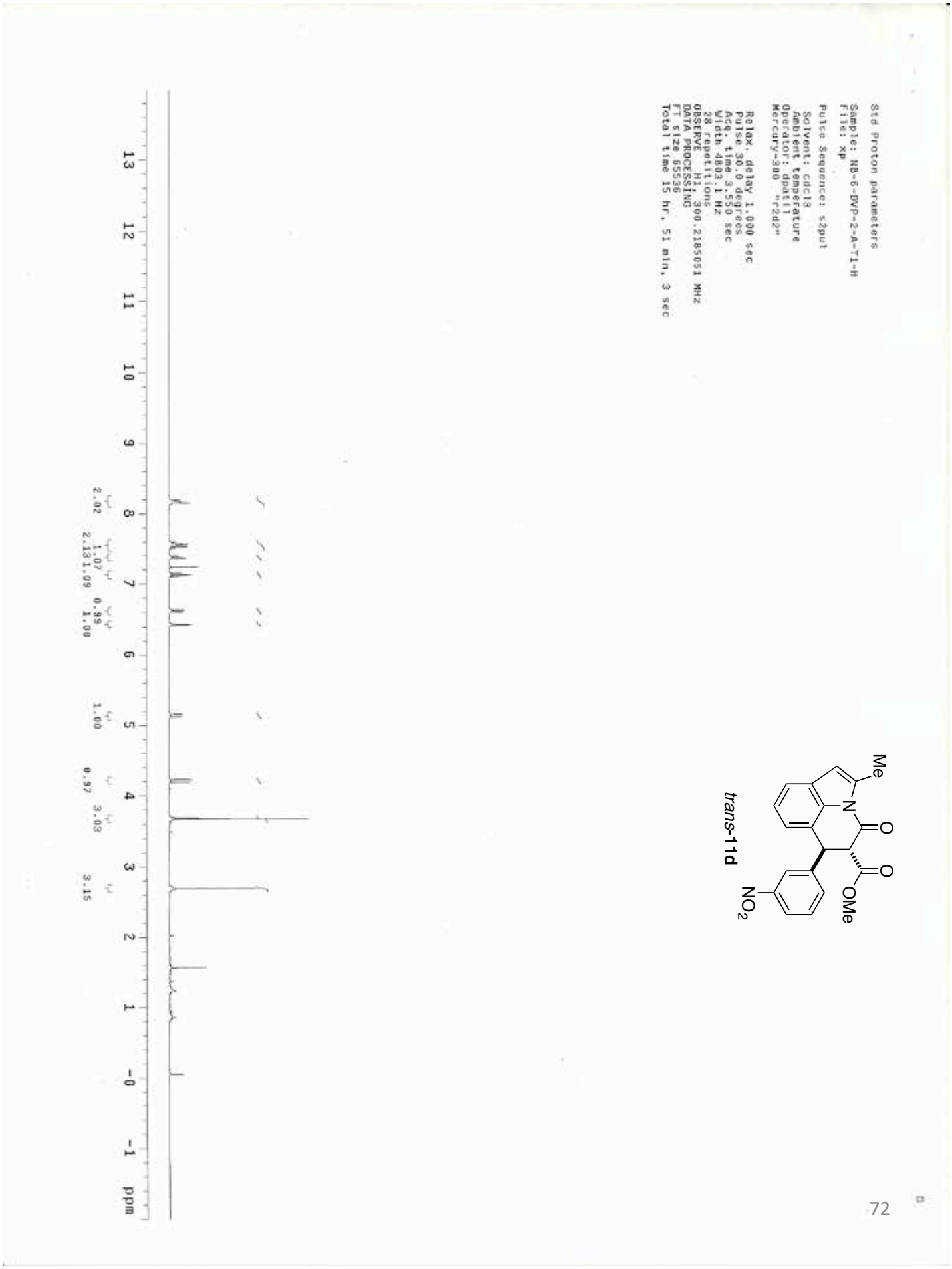
11b (crude)

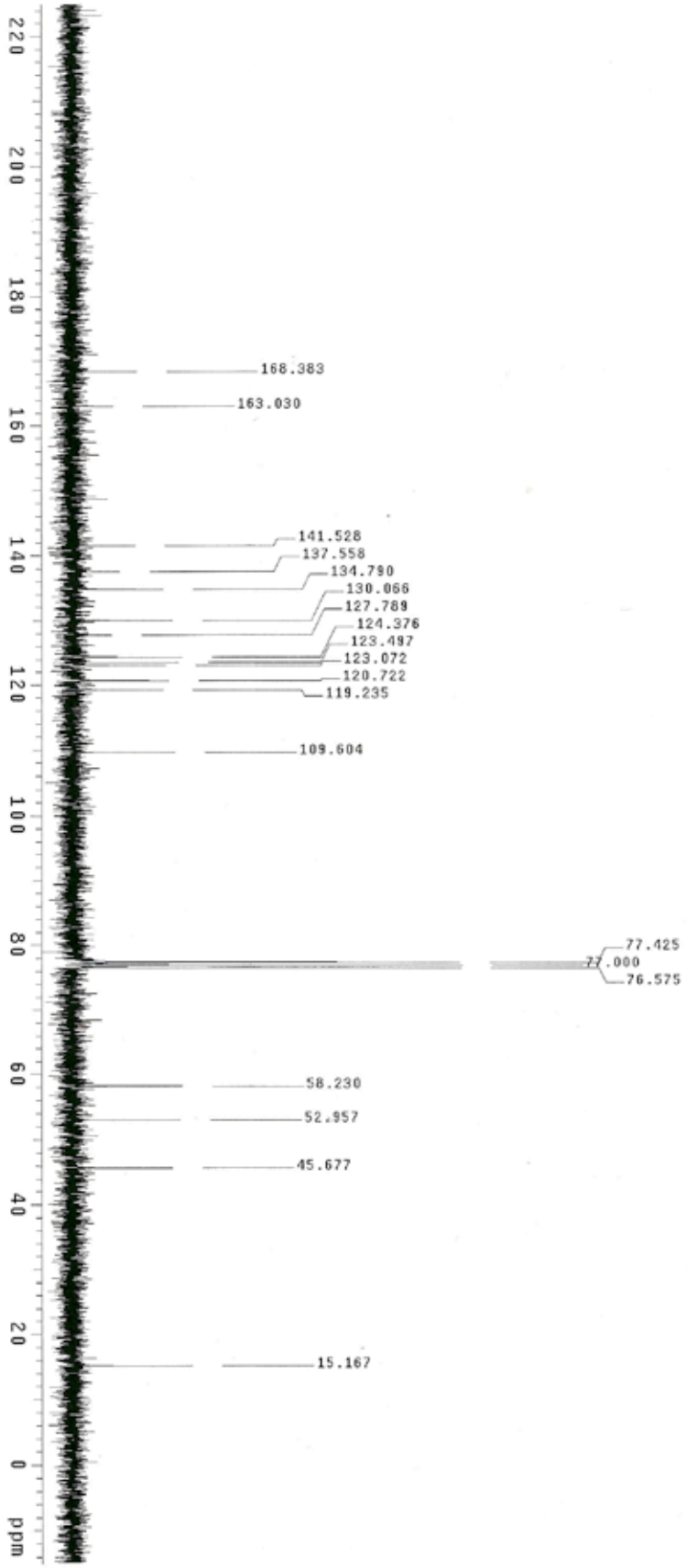

4-nitrophenyl cyclo
Sample: NB-5-DVP-140-T1-H
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpatil
Mercury-300 "r242"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
16 repetitions
OBSERVE H1, 300.2185002 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 16 sec
Me
N
O
O
OMe
NO2
trans-11c
11
10
9
8
7
6
5
4
3
2
1
ppm
2.36
3.85
1.23
1.04
1.09
1.18
1.02
2.99
3.15

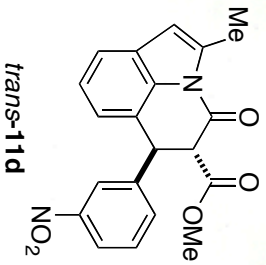

Std Carbon experiment
Sample: NB-5-DVP-140-T1-M
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpatil
Mercury-300 "r242"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 1.301 sec
Width 18115.9 Hz
224 repetitions
OBSERVE C13, 75.4900037 MHz
DECOUPLE H1, 300.2199481 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 10 min, 45 sec
168.376
162.978
147.592
146.779
137.580
129.576
127.774
124.977
124.347
124.237
120.766
120.560
119.228
109.590
77.417
77.000
76.575
58.032
52.971
45.758
15.160
220
200
180
160
140
120
100
80
60
40
20
0
ppm
Me
N
O
O
OMe
NO2

trans-**11c**




Std Proton parameters
Sample: NB-6-DVP-2-A-T1-H
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpatil
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
28 repetitions
OBSERVE H1, 300.2185051 MHz
DATA PROCESSING
FT size 65536
Total time 15 hr, 51 min, 3 sec
Me
N
O
O
OMe
NO2
trans-11d
13
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
2.02
1.07
2.13
1.09
0.99
1.00
1.00
0.97
3.03
3.15


Std Carbon experiment
Sample: NB-6-DVP-2-A-T1-C
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpatil
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 1.301 sec
Width 18115.9 Hz
96 repetitions
OBSERVE C13, 75.4909937 MHz
DECOUPLE H1, 300.2199481 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 10 hr, 41 min, 3 sec
168.383
163.030
141.528
137.558
134.790
130.066
127.789
124.376
123.497
123.072
120.722
119.235
109.604
77.425
77.000
76.575
58.230
52.957
45.677
15.167
220
200
180
160
140
120
100
80
60
40
20
0
ppm


Me
N
O
O
O
OMe
NO2

trans-**11d**

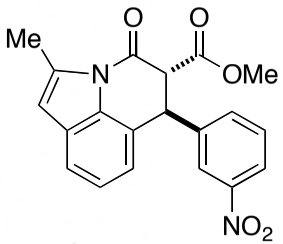
3-NO_2 benzaldehyde derived !!

Std Proton parameters

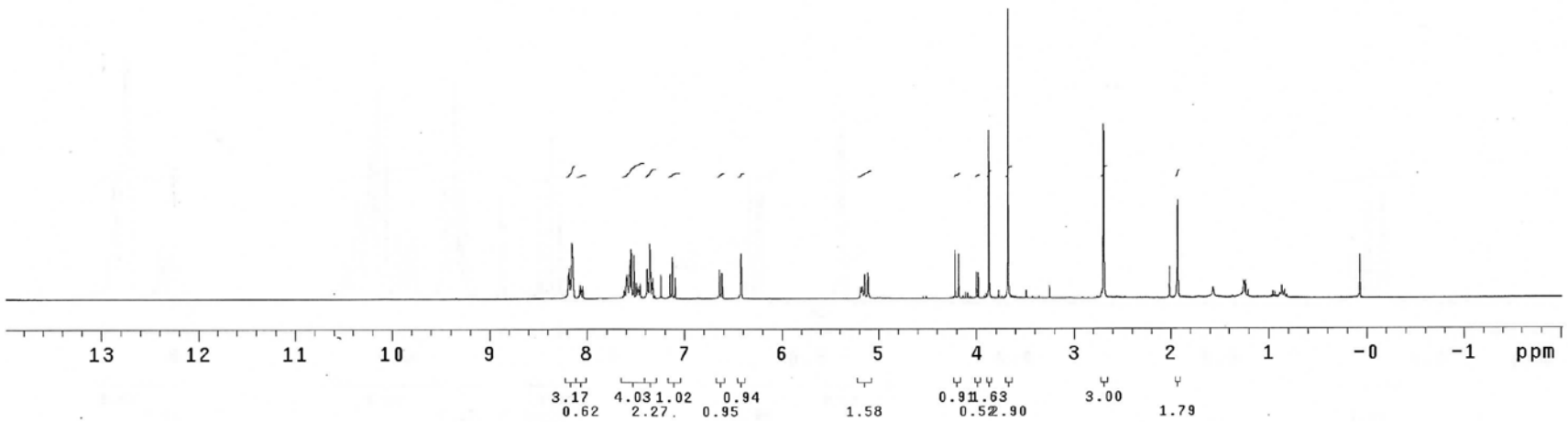
Sample: NB-6-DVP-2-A-HHH
File: xp

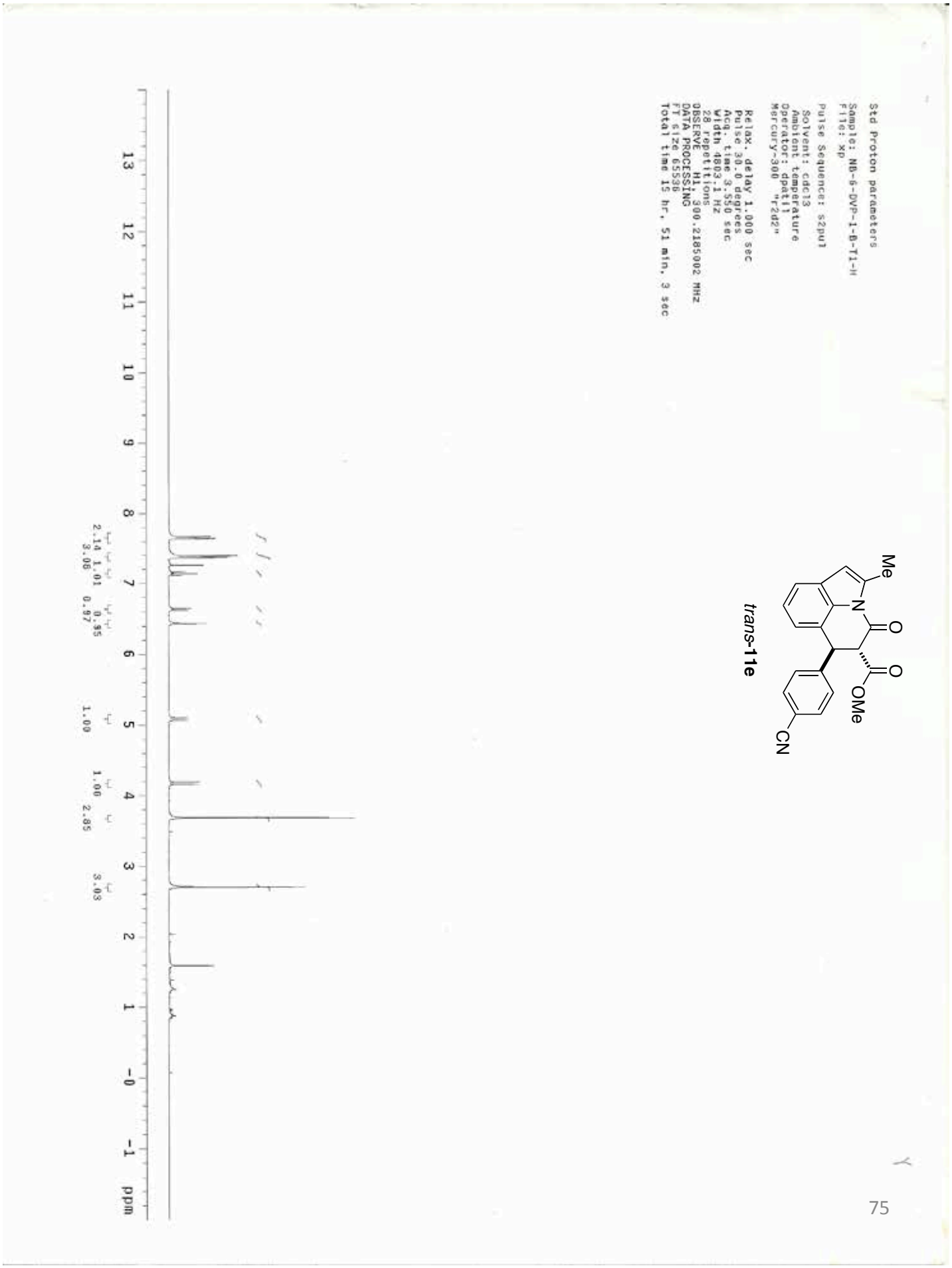
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 40.0 C / 313.1 K
Operator: dpatil
Mercury-300 "r2d2"

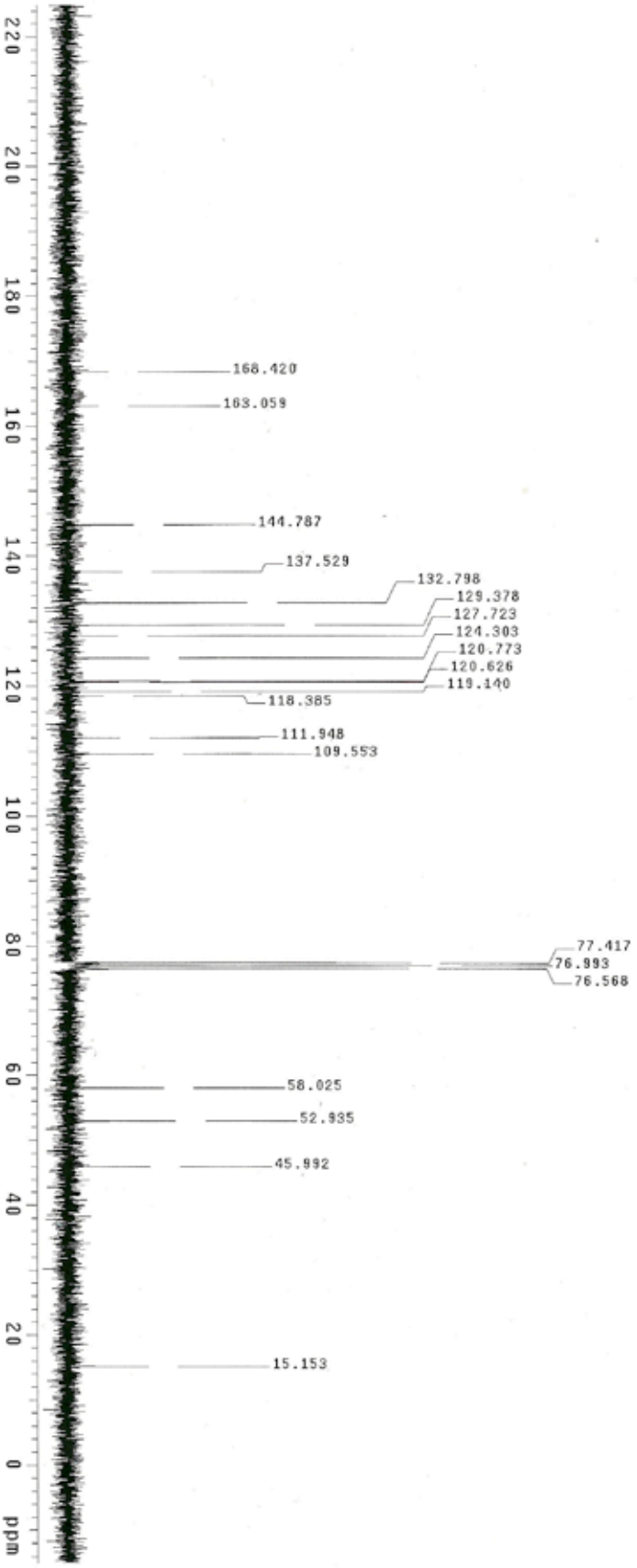
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
40 repetitions
OBSERVE H1, 300.2185051 MHz
DATA PROCESSING
FT size 65536
Total time 15 hr, 51 min, 3 sec

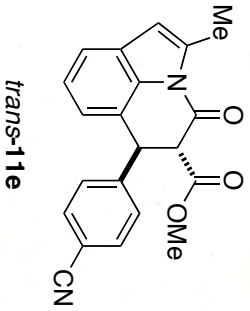


11d (crude)




Std Proton parameters
Sample: NB-6-DVP-1-B-T1-H
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpatil
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
28 repetitions
OBSERVE H1, 300.2185002 MHz
DATA PROCESSING
FT size 65536
Total time 15 hr, 51 min, 3 sec
Me
N
O
O
O
OMe
CN
trans-11e
13
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
2.14
1.01
3.06
0.95
0.97
1.09
1.00
2.85
3.03


Std Carbon experiment
Sample: NB-6-DVP-1-B-T1-H
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpatil
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 1.301 sec
Width 18115.9 Hz
64 repetitions
OBSERVE C13, 75.4800943 MHz
DECOUPLE H1, 300.2199481 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 10 hr, 41 min, 3 sec
168.420
163.059
144.787
137.529
132.798
129.378
127.723
124.303
120.773
120.626
119.140
118.385
111.948
109.553
77.417
76.993
76.568
58.025
52.935
45.992
15.153
220
200
180
160
140
120
100
80
60
40
20
0
ppm


Me
N
O
O
OMe
CN

trans-**11e**

4-CN benzaldehyde derived

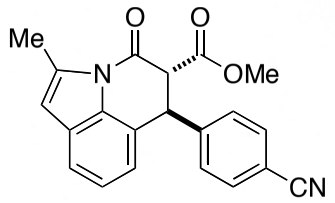
new proton

Sample: NB-6-DVP-1-B-HHH
File: xp

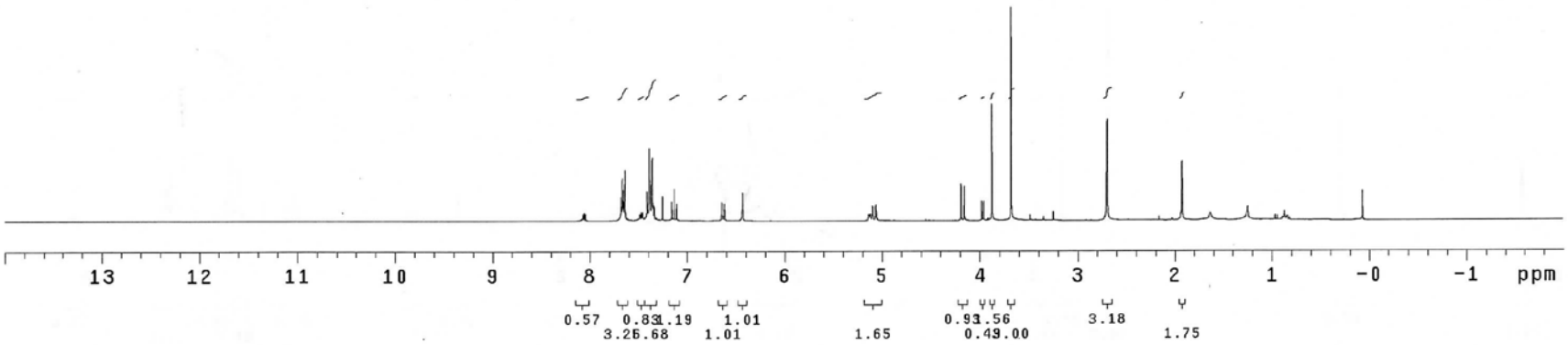
Pulse Sequence: s2pul

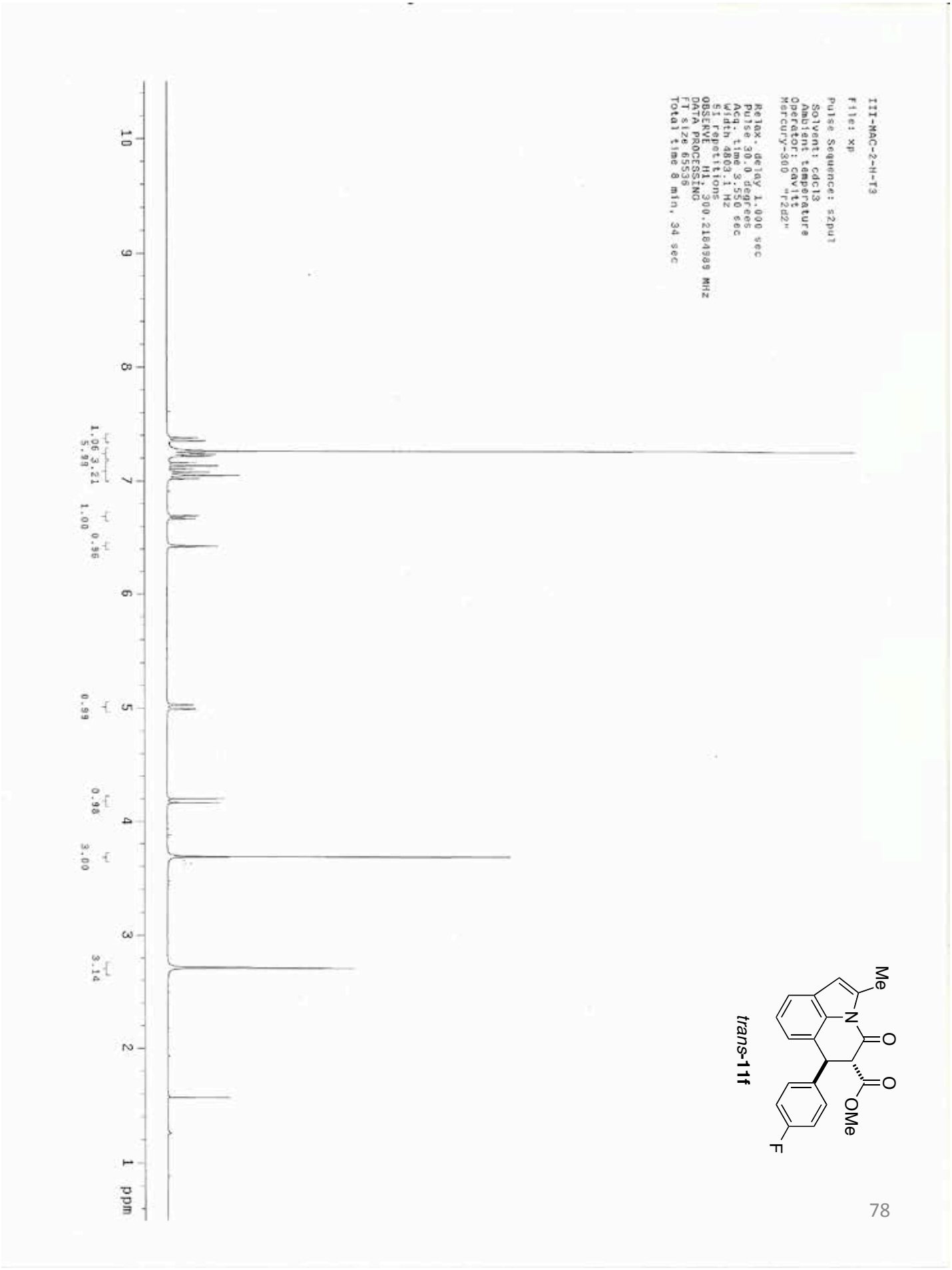
Solvent: cdcl3
Temp. 21.0 C / 294.1 K
Operator: dpatil
Mercury-300 "r2d2"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
80 repetitions
OBSERVE H1, 300.2185002 MHz
DATA PROCESSING
FT size 65536
Total time 1 hr, 18 min, 43 sec

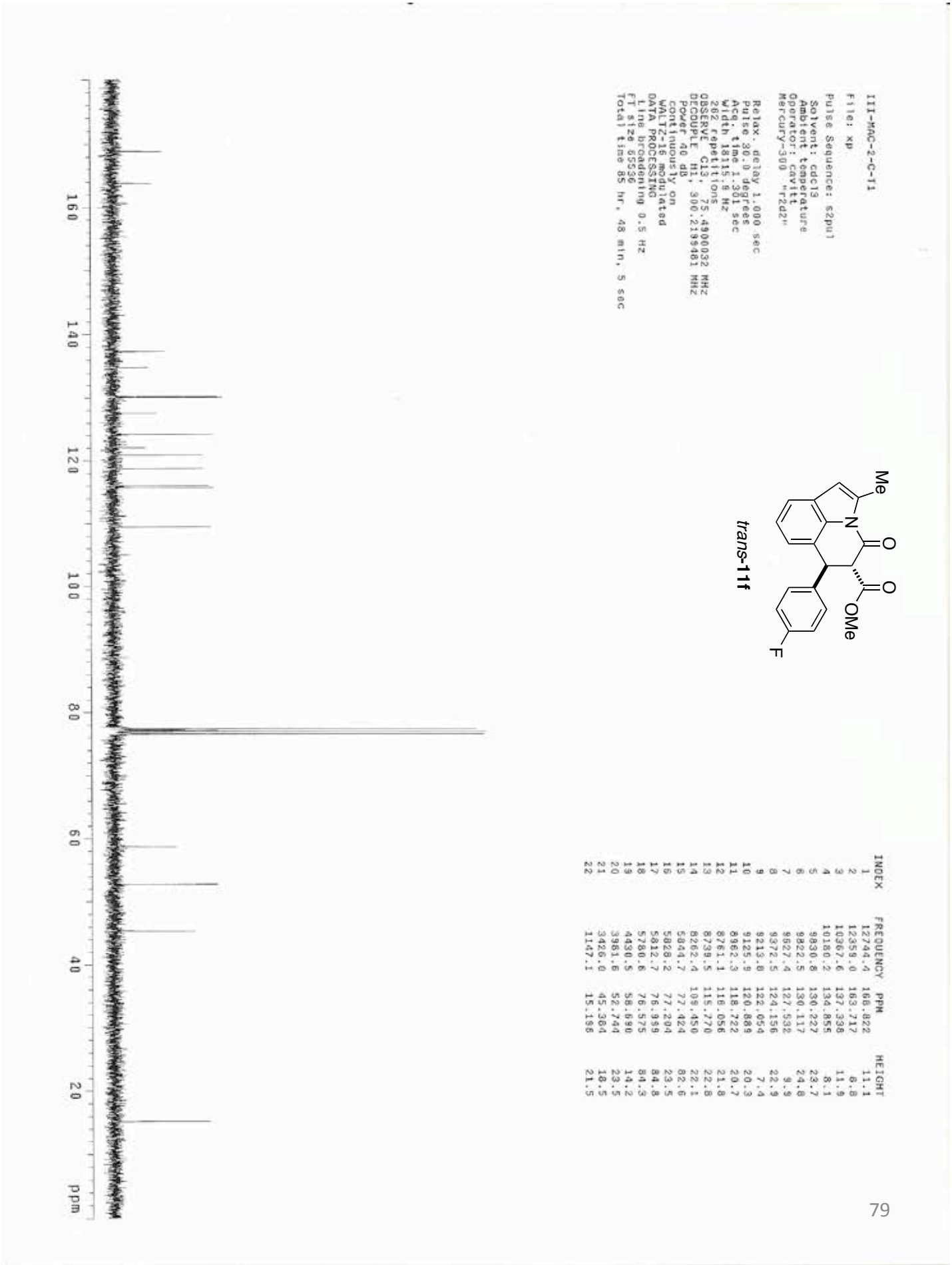


11e (crude)




III-MAC-2-H-T3
file: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: cavitt
Mercury-300 "r202"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
width 4803.1 Hz
51 repetitions
OBSERVE H1, 300.2184989 MHz
DATA PROCESSING
FT size 65536
Total time 8 min, 34 sec
1.06
3.21
5.99
1.00
0.96
0.99
0.98
3.00
3.14
10
9
8
7
6
5
4
3
2
1
ppm
Me
N
O
O
OMe
F

trans-**11f**


III-MAC-2-C-T1
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: cavitt
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 1.301 sec
Width 4815.8 Hz
262 repetitions
OBSERVE C13, 75.4900032 MHz
DECOUPLE H1, 300.2199481 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 85 hr, 48 min, 5 sec
INDEX FREQUENCY PPM HEIGHT
1 12744.4 166.822 11.1
2 12359.0 163.712 8.8
3 10367.6 137.338 11.9
4 10180.2 134.855 8.1
5 9830.8 130.227 23.7
6 9822.5 130.117 24.8
7 9627.4 127.532 9.9
8 9372.5 124.156 22.9
9 9213.8 122.054 7.4
10 9125.9 120.889 20.3
11 8962.3 118.722 20.7
12 8761.1 116.056 21.8
13 8739.5 115.770 22.8
14 8262.4 109.450 22.1
15 5844.7 77.424 82.6
16 5828.2 77.204 23.5
17 5812.7 76.999 84.8
18 5780.6 76.575 84.3
19 4430.5 58.690 14.2
20 3961.6 52.744 23.5
21 3426.0 45.384 10.5
22 1147.1 15.196 21.5
Me
N
O
O
OMe
F
trans-11f
160
140
120
100
80
60
40
20
ppm

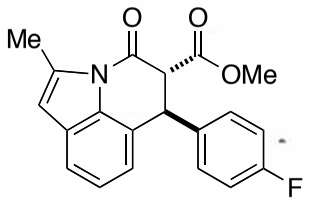
4-F benzaldehyde derived !!

III-MAC-2-H-T1

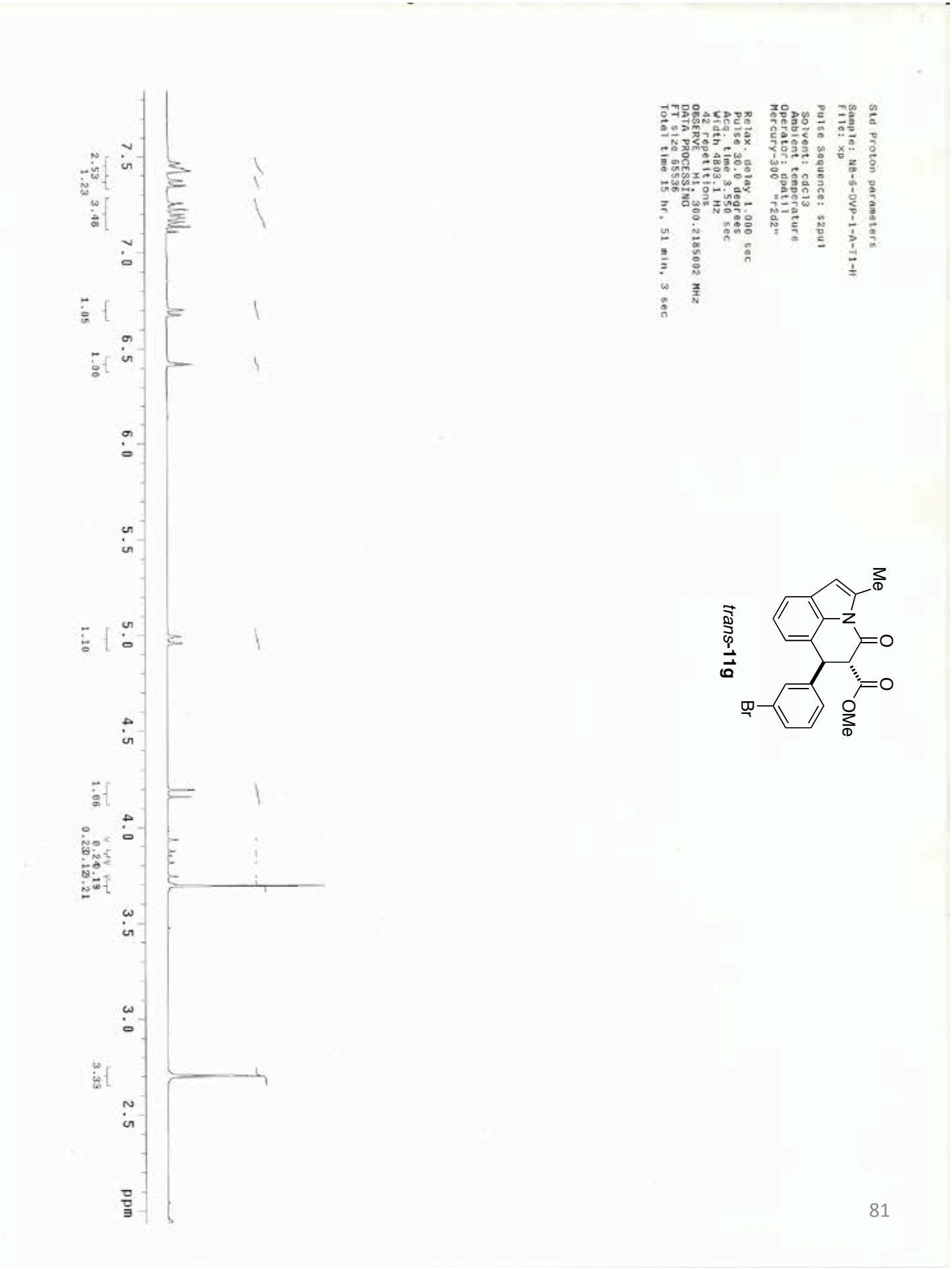
File: xp

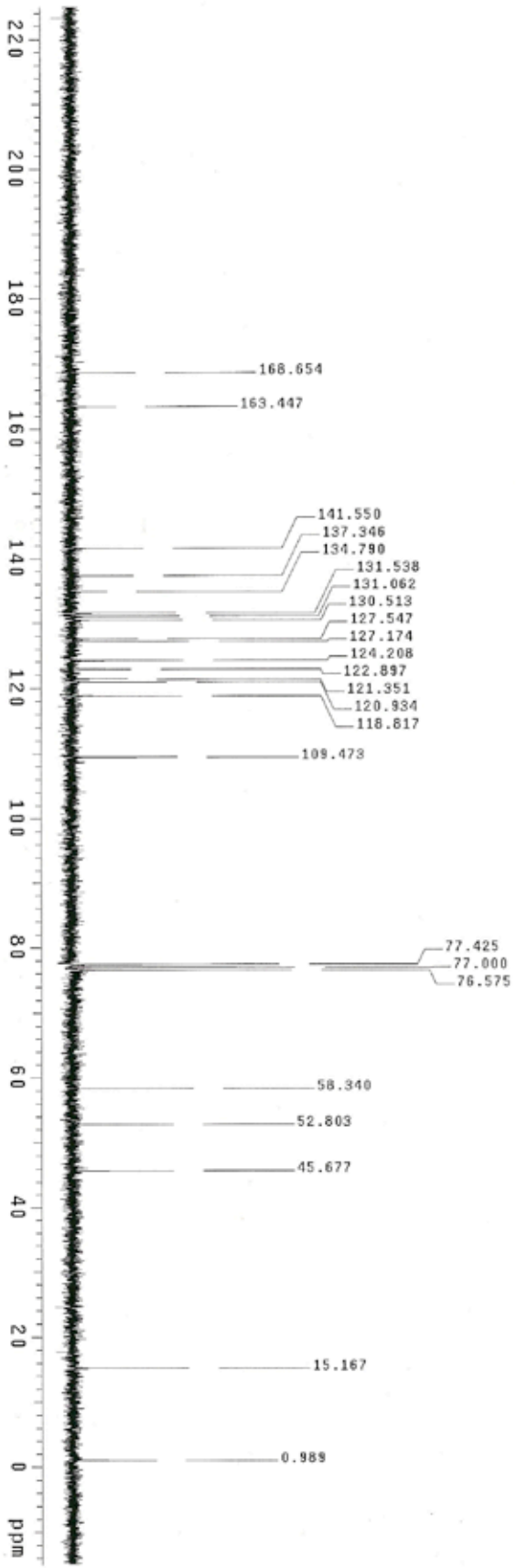
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: cavitt
Mercury-300 "r2d2"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
23 repetitions
OBSERVE H1, 300.218[illegible] MHz
DATA PROCESSING
FT size 65536
Total time 8 min, 34 sec

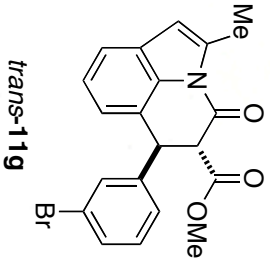


11f (crude)


Std proton parameters
Sample: NB-6-DVP-1-A-T1-H
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpatil
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
42 repetitions
OBSERVE H1, 300.2185002 MHz
DATA PROCESSING
FT size 65536
Total time 15 hr, 51 min, 3 sec
Me
N
O
O
OMe
Br
trans-11g
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
ppm
2.53
3.48
1.23
1.05
1.00
1.10
1.06
0.24
0.19
0.23
0.12
0.21
3.33




```
Std Carbon experiment

Sample: NB-6-DVP-1-A-T1-H
File: xp

Pulse Sequence: s2pul

  Solvent: cdcl3
  Ambient temperature
Operator: dpatil
Mercury-300 "r2d2"

  Relax. delay 1.000 sec
  Pulse 30.0 degrees
  Acq. time 1.301 sec
  Width 18115.9 Hz
  62 repetitions
OBSERVE  C13, 75.4900054 MHz
DECOUPLE  H1, 300.2199481 MHz
  Power 40 dB
   continuously on
  WALTZ-16 modulated
DATA PROCESSING
  Line broadening 0.5 Hz
FT size 65536
Total time 10 hr, 41 min, 3 sec
```



trans-**11g**

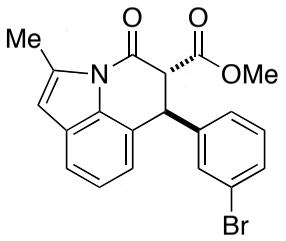
3-bromobenzaldehyde derived !!

new proton

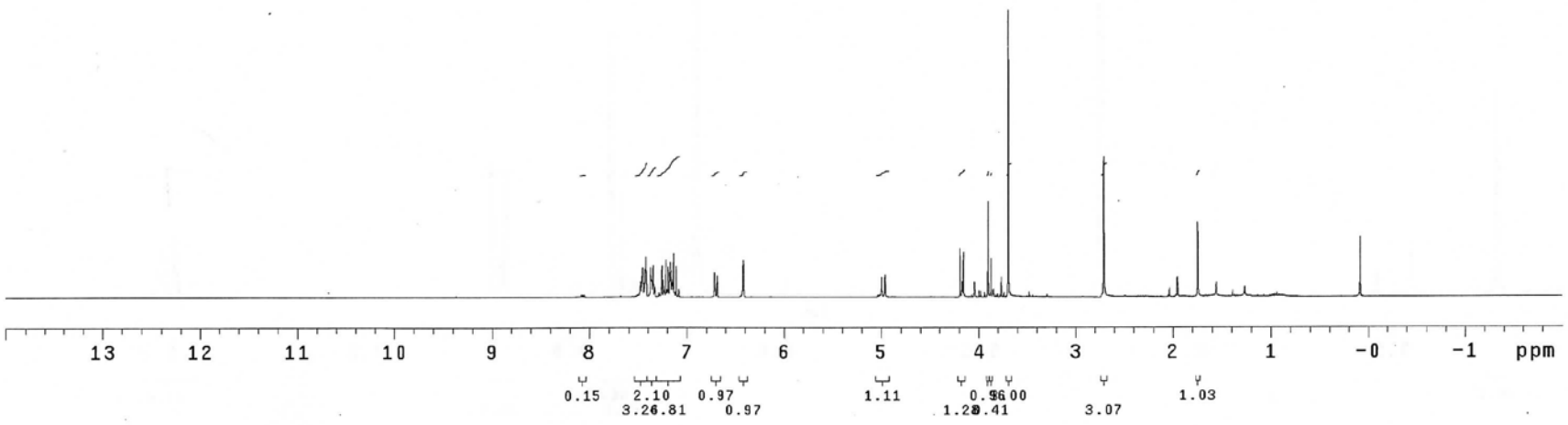
Sample: NB-6-DVP-1-A-HHH
File: xp

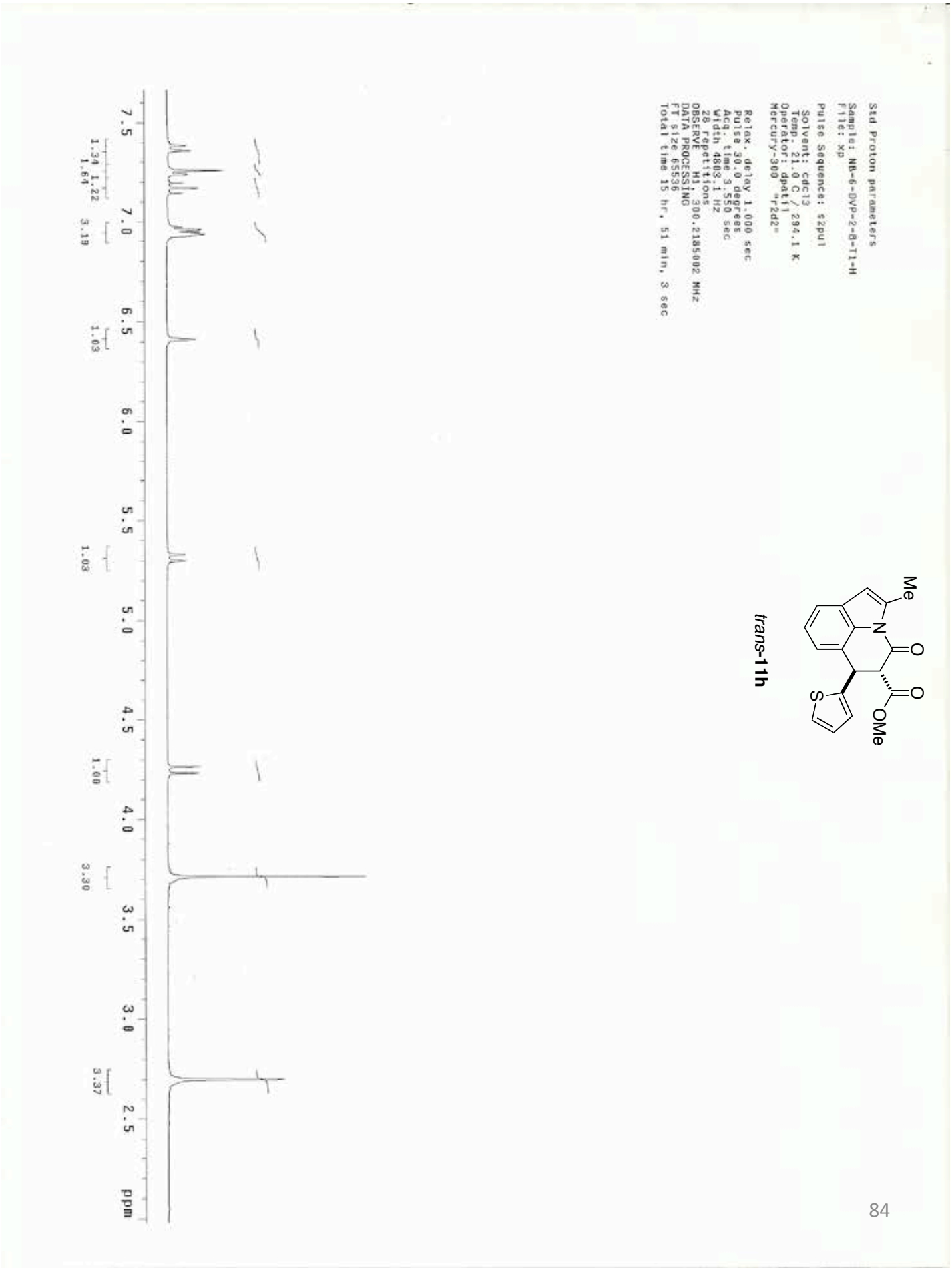
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 40.0 C / 313.1 K
Operator: dpatil
Mercury-300 "r2d2"

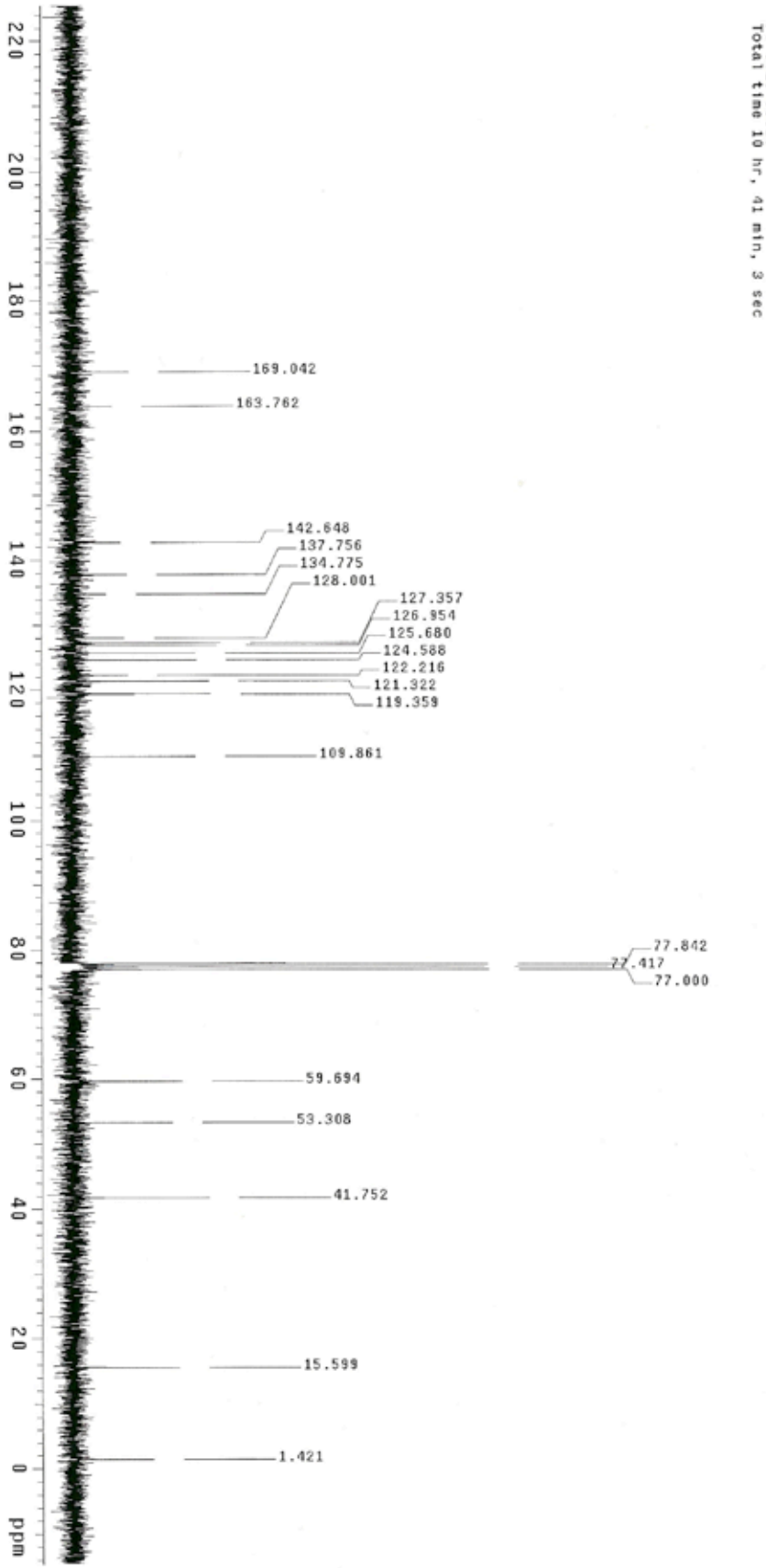
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
70 repetitions
OBSERVE H1, 300.2185002 MHz
DATA PROCESSING
FT size 65536
Total time 1 hr, 35 min, 6 sec



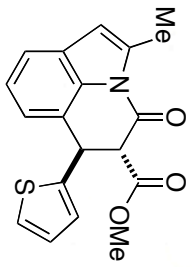
11g (crude)

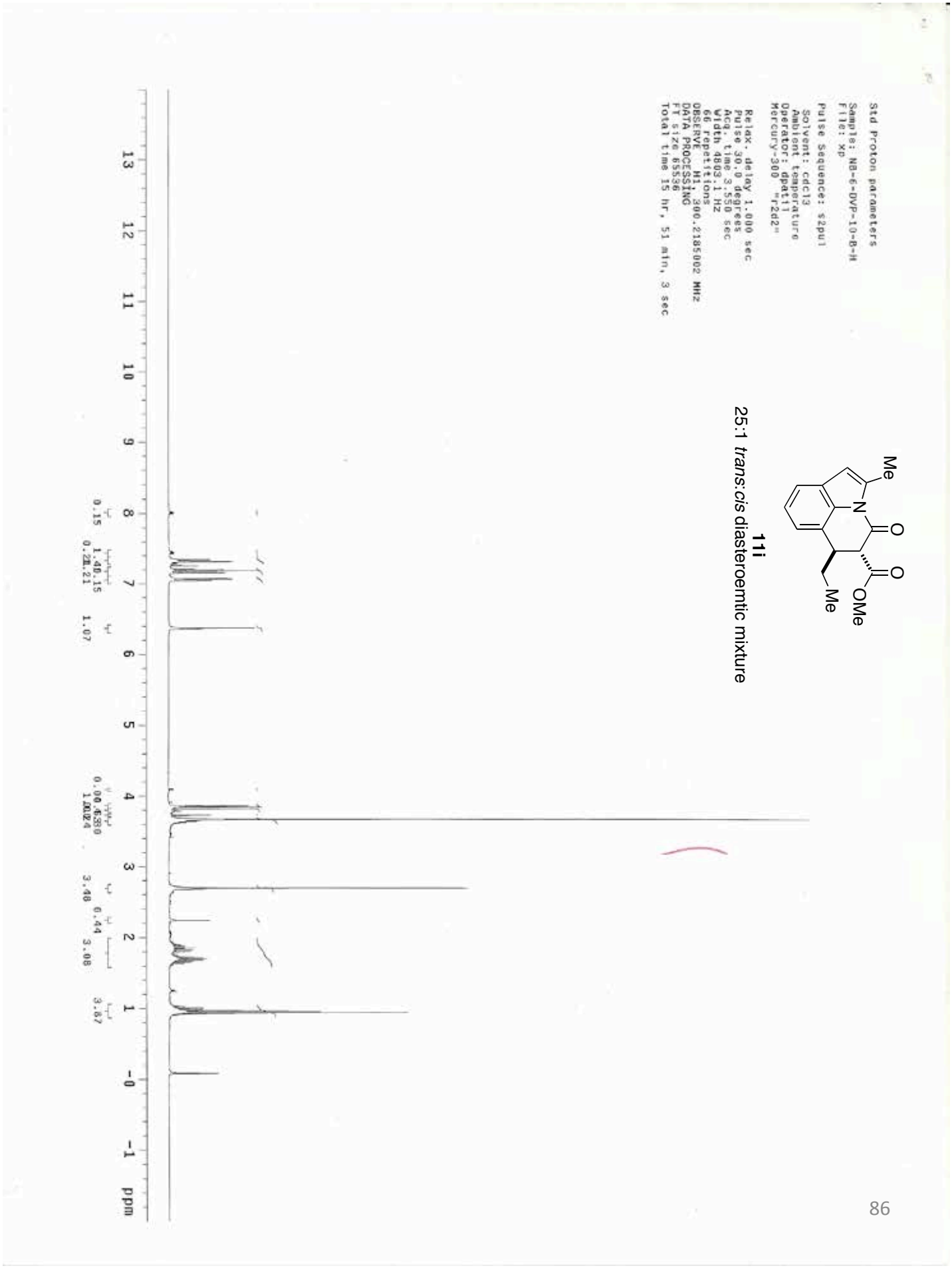


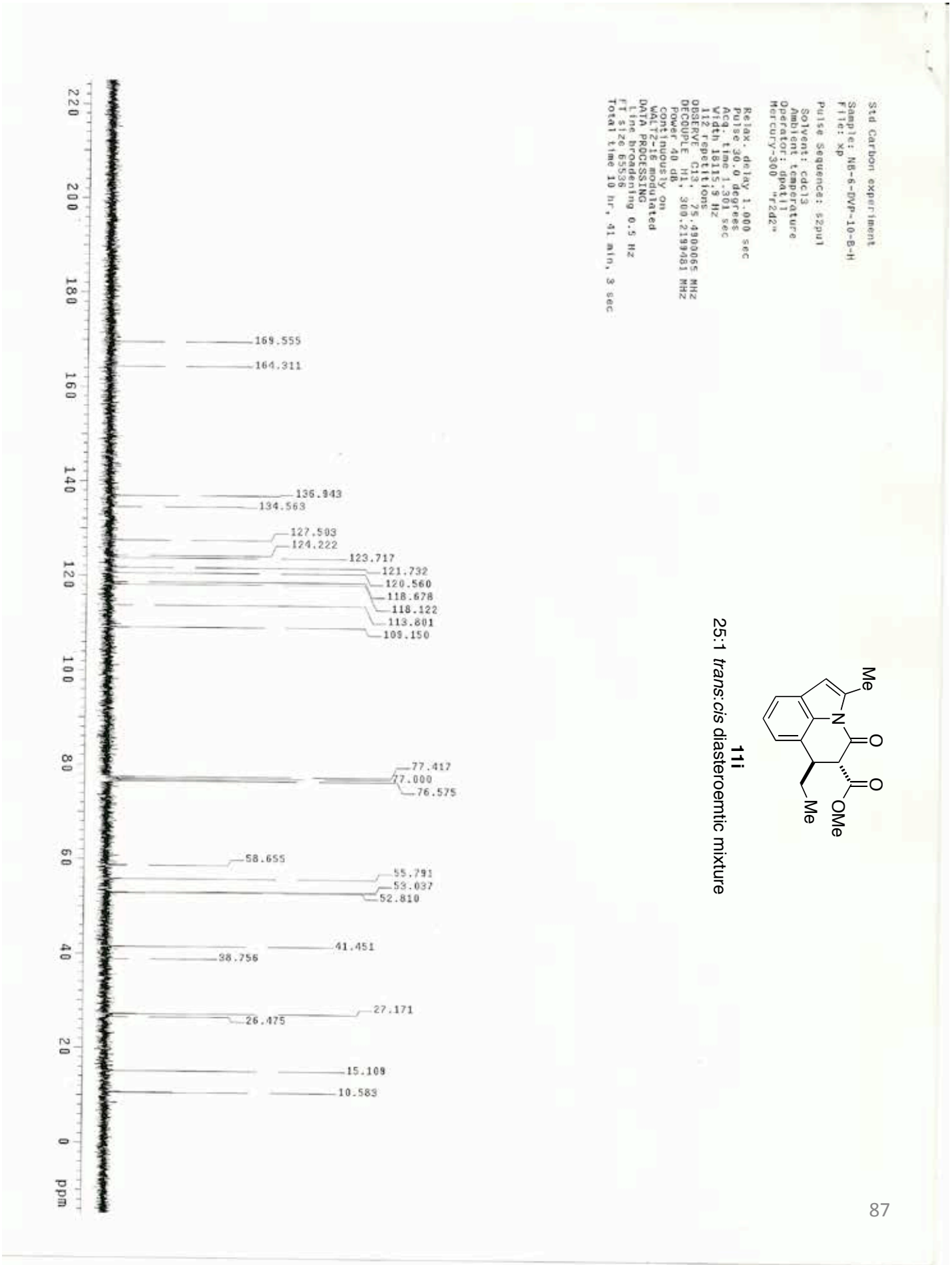

Std Proton parameters
Sample: NB-6-DVP-2-8-T1-H
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 21.0 C / 294.1 K
Operator: dpati1
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
28 repetitions
OBSERVE H1, 300.2185002 MHz
DATA PROCESSING
FT size 65536
Total time 15 hr, 51 min, 3 sec
Me
N
O
O
OMe
S
trans-11h
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
ppm
1.34
1.22
1.64
3.19
1.03
1.03
1.00
3.30
3.37


Std Carbon experiment
Sample: NB-6-DVP-2-B-TI-H
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 21.0 C / 294.1 K
Operator: dpatil
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 1.301 sec
Width 18115.9 Hz
156 repetitions
OBSERVE C13, 75.4899717 MHz
DECOUPLE H1, 300.2199481 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 10 hr, 41 min, 3 sec
169.042
163.762
142.648
137.756
134.775
128.001
127.357
126.954
125.680
124.588
122.216
121.322
119.359
109.861
77.842
77.417
77.000
59.694
53.308
41.752
15.599
1.421
220
200
180
160
140
120
100
80
60
40
20
0
ppm

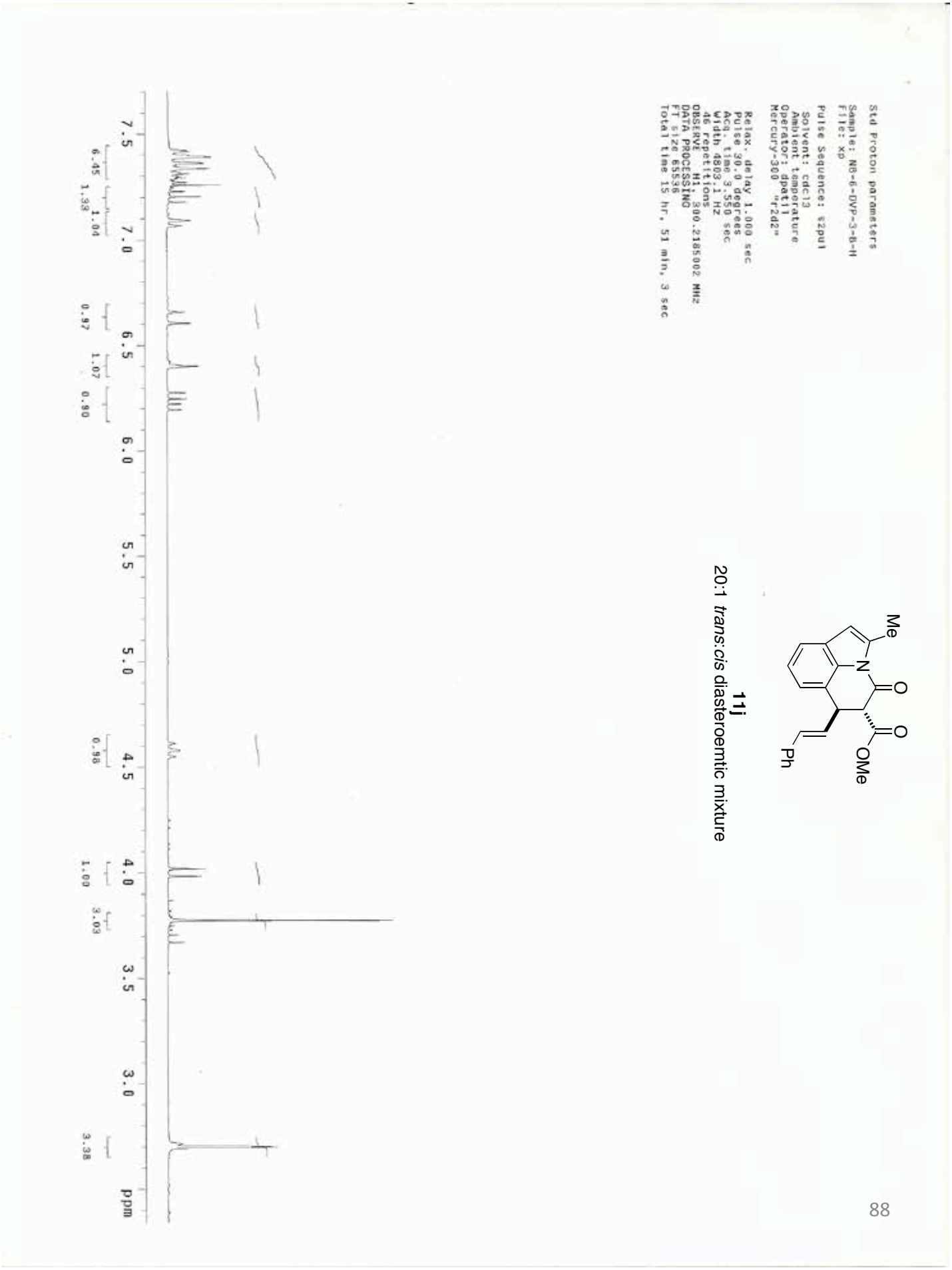
trans-**11h**

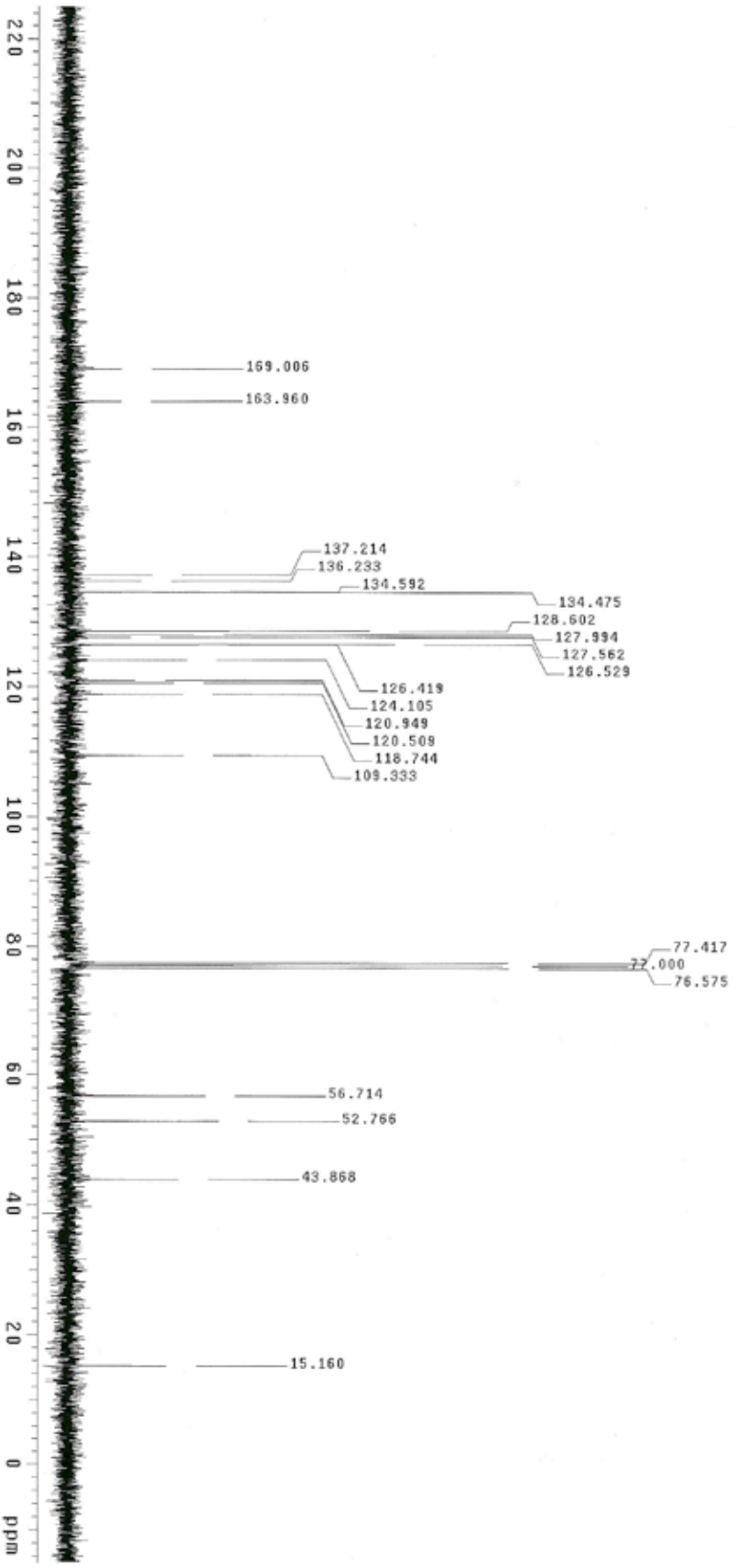

Me
N
O
O
S
OMe


Std Proton parameters
Sample: NB-6-DVP-10-B-H
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpati1
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
66 repetitions
OBSERVE H1, 300.2185002 MHz
DATA PROCESSING
FT size 65536
Total time 15 hr, 51 min, 3 sec
Me
N
O
O
OMe
Me
11i
25:1 trans:cis diasteroemtic mixture
13
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
0.15
1.40
0.21
1.07
0.00
1.00
3.48
0.44
3.08
3.87

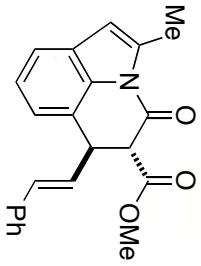


11i
25:1 *trans*:*cis* diasteroemtic mixture


Std Proton parameters
Sample: NB-6-DVP-3-B-H
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpatil
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
46 repetitions
OBSERVE H1 300.2185002 MHz
DATA PROCESSING
FT size 65536
Total time 15 hr, 51 min, 3 sec
Me
N
O
O
OMe
Ph
11j
20:1 trans:cis diasteroemtic mixture
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
ppm
6.45
1.04
1.33
0.97
1.07
0.90
0.98
1.00
3.03
3.38



11j
20:1 *trans:cis* diasteroemtic mixture

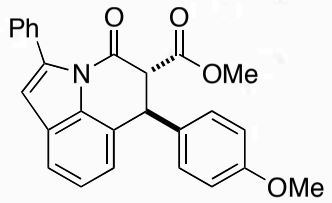


II-MAC-4S-H

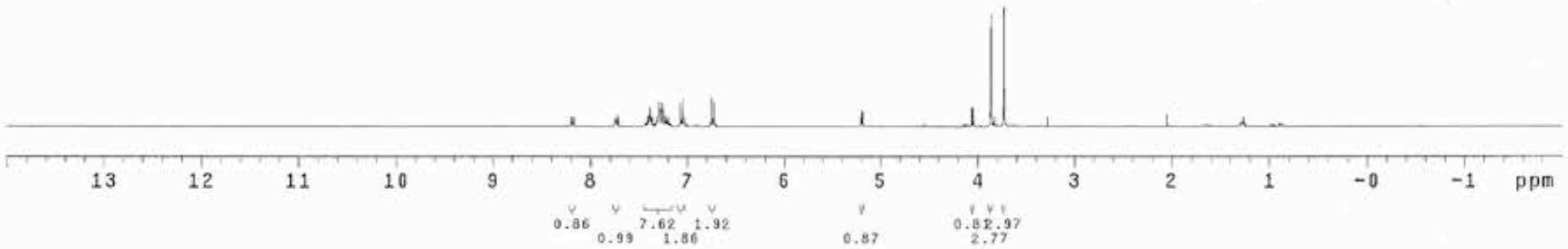
File: xp

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: cavitt
Mercury-300 "r2d2"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
39 repetitions
OBSERVE H1, 300.2297117 MHz
DATA PROCESSING
FT size 65536
Total time 8 min, 34 sec



trans-**11k**

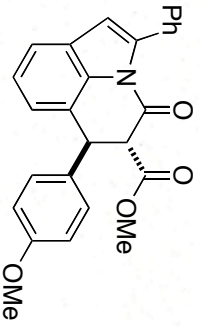


II-MAC-45-C

File: xp

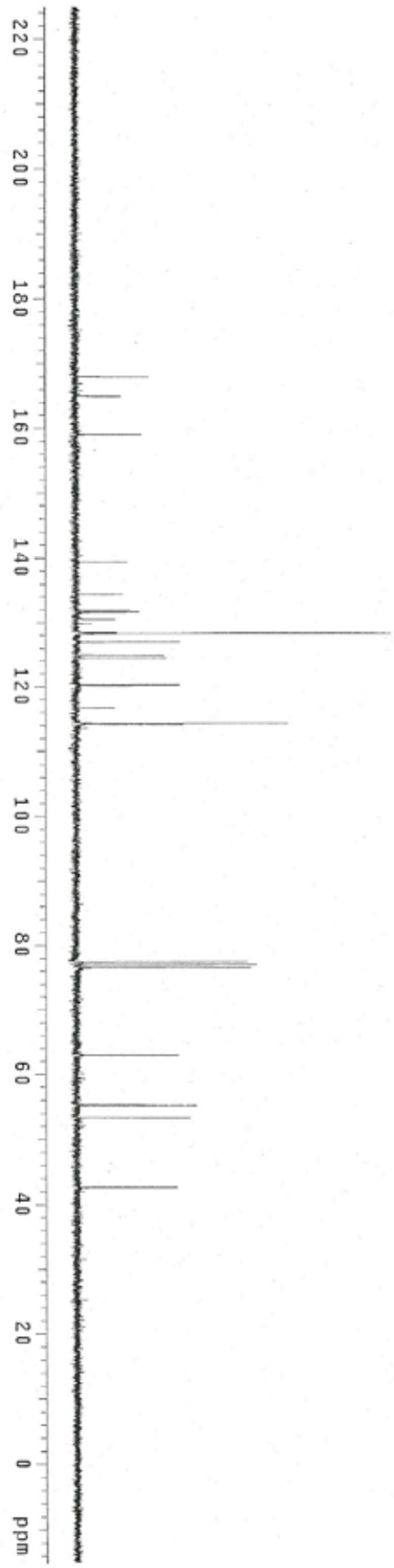
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: cavitt
Mercury-300 "f2d2"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 1.301 sec
Width 18115.9 Hz
252 repetitions
OBSERVE C13, 75.4913166 MHz
DECOUPLE H1, 300.2251867 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 858 hr, 47 sec

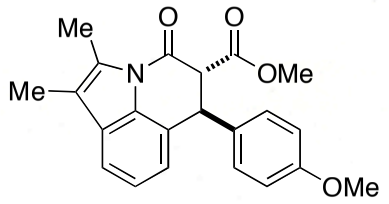


trans-**11k**

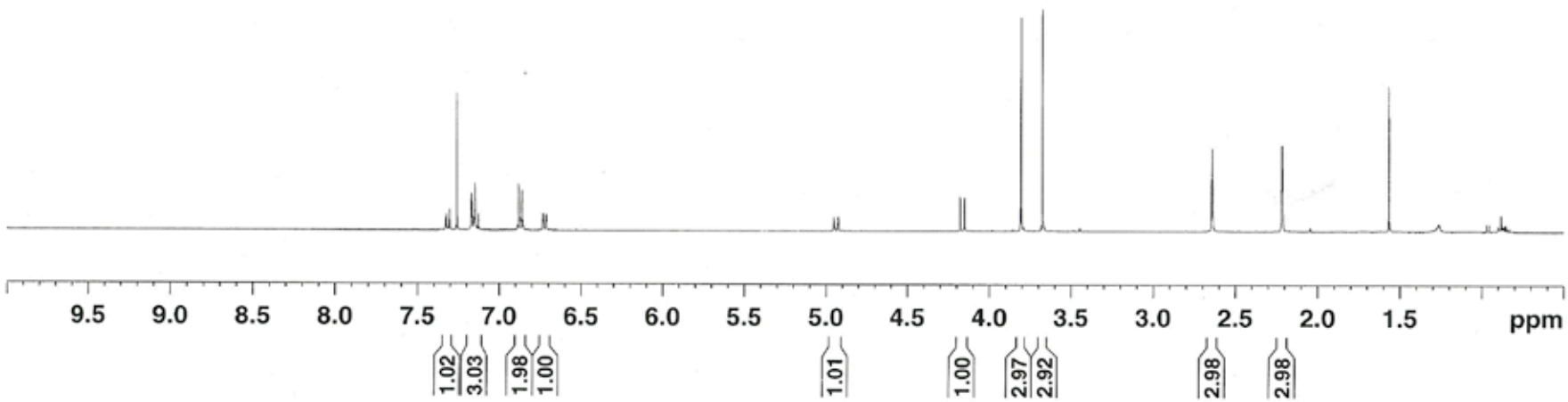
INDEX	FREQUENCY	PPM	HEIGHT
1	12679.7	167.962	12.2
2	12516.0	165.794	1.2
3	12451.9	164.945	7.5
4	11999.1	158.947	10.3
5	10520.2	139.357	8.6
6	10146.0	134.399	7.8
7	9904.1	131.990	9.0
8	9940.6	131.682	10.4
9	9928.1	131.514	2.3
10	9852.9	130.518	6.4
11	9798.8	129.800	2.6
12	9693.2	128.401	39.1
13	9689.8	128.357	52.0
14	9677.1	128.189	3.2
15	9584.8	126.966	17.2
16	9428.9	124.900	14.5
17	9395.7	124.461	14.9
18	9074.5	120.206	17.1
19	8808.0	116.676	6.5
20	8627.3	114.282	35.0
21	8616.2	114.135	17.8
22	8573.1	113.564	2.0
23	5856.4	77.577	-1.3
24	5844.7	77.423	28.2
25	5812.7	76.998	29.8
26	5780.6	76.573	28.8
27	4754.0	62.974	16.9
28	4529.5	60.000	1.3
29	4478.6	59.327	1.5
30	4165.7	55.182	19.9
31	4159.6	55.101	1.9
32	4023.1	53.292	18.8
33	3991.9	52.084	1.5
34	3222.6	42.688	16.6
35	3174.5	42.051	1.4
36	2381.1	31.542	1.5
37	1904.5	25.229	1.8
38	1586.1	21.010	1.3

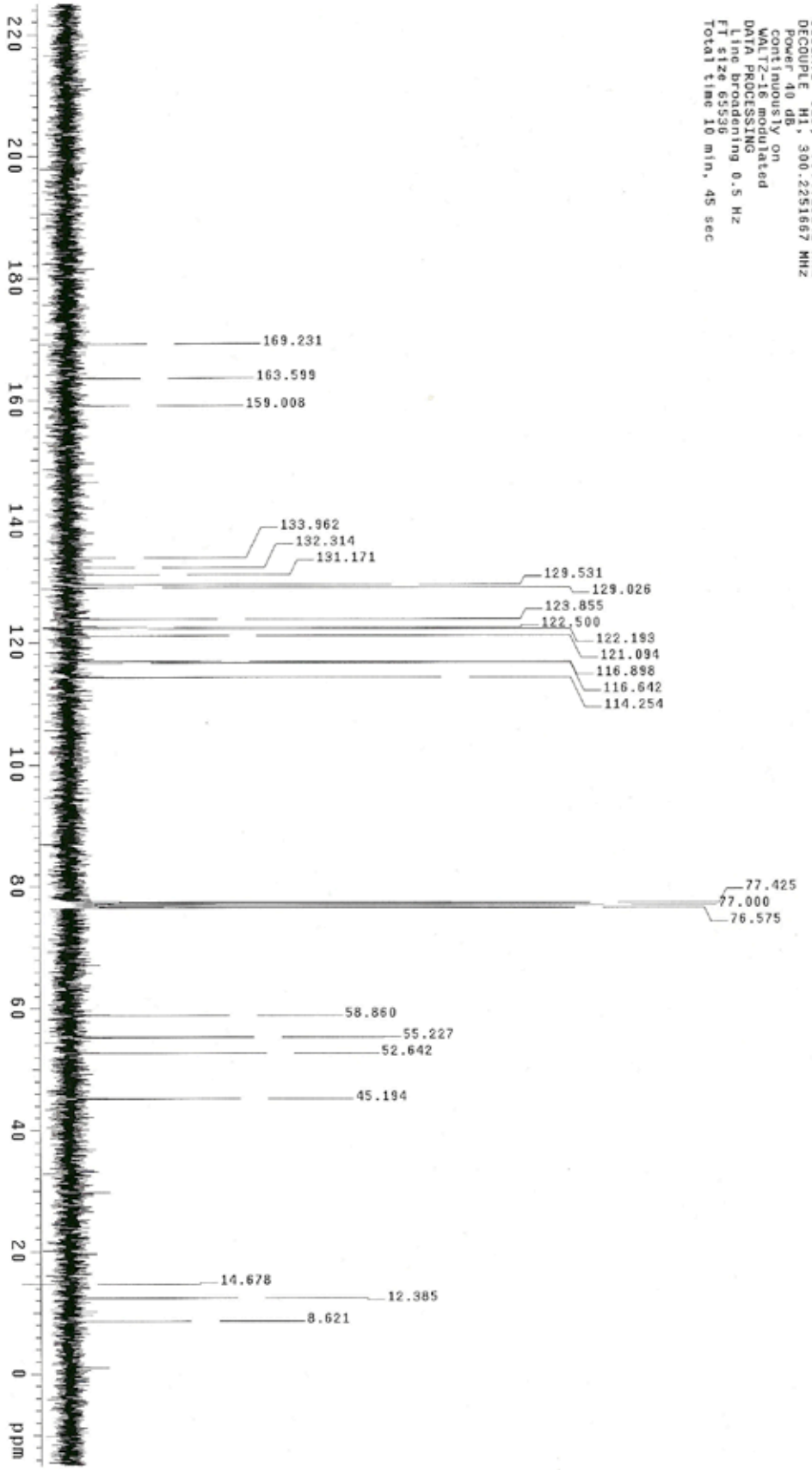


2,3-Me,Me-Cyclized-2-Substituted-Paper
Marchello

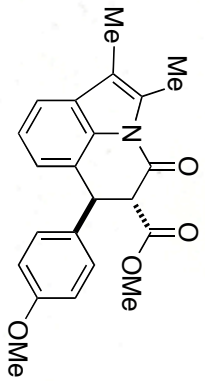


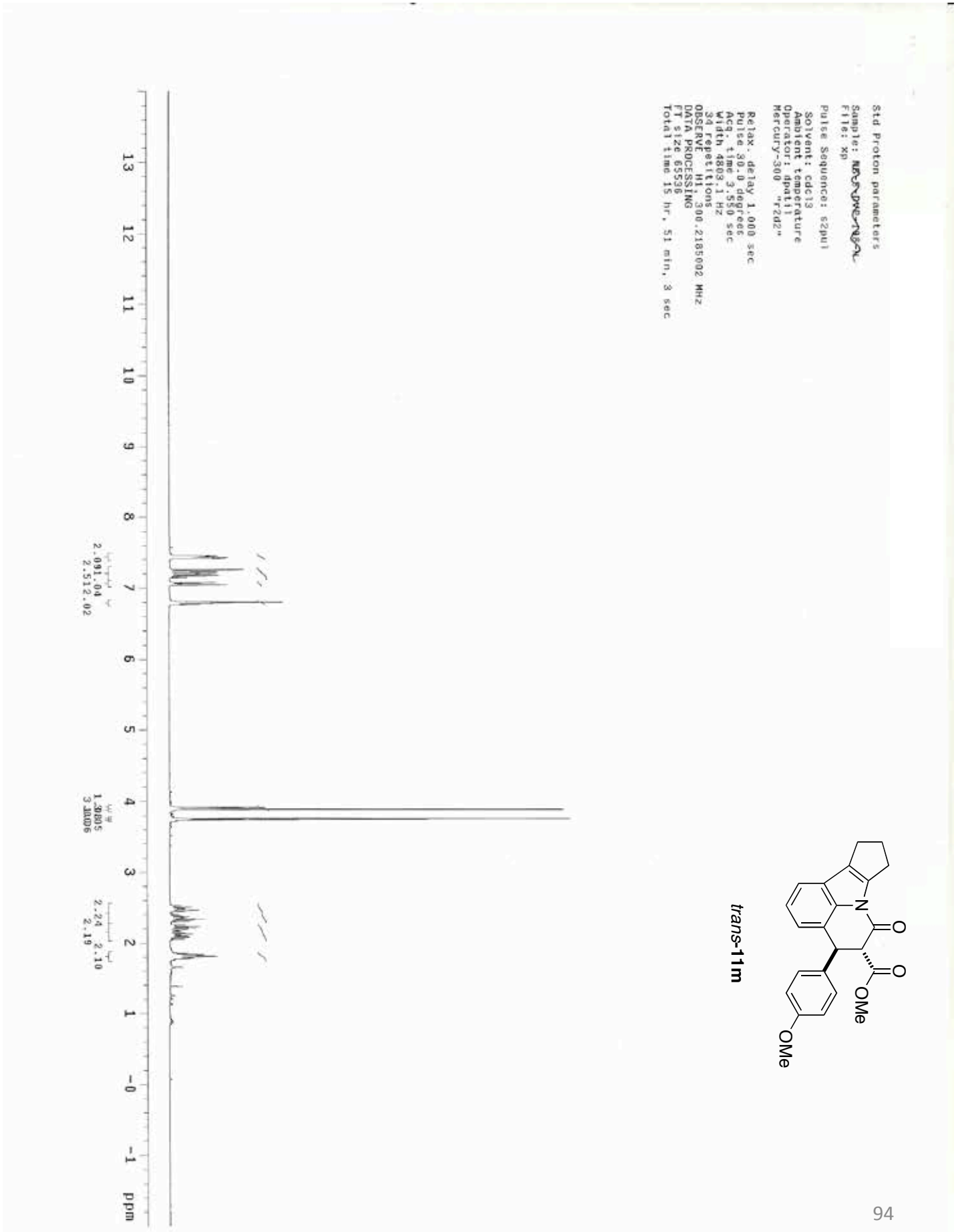
trans-**11l**



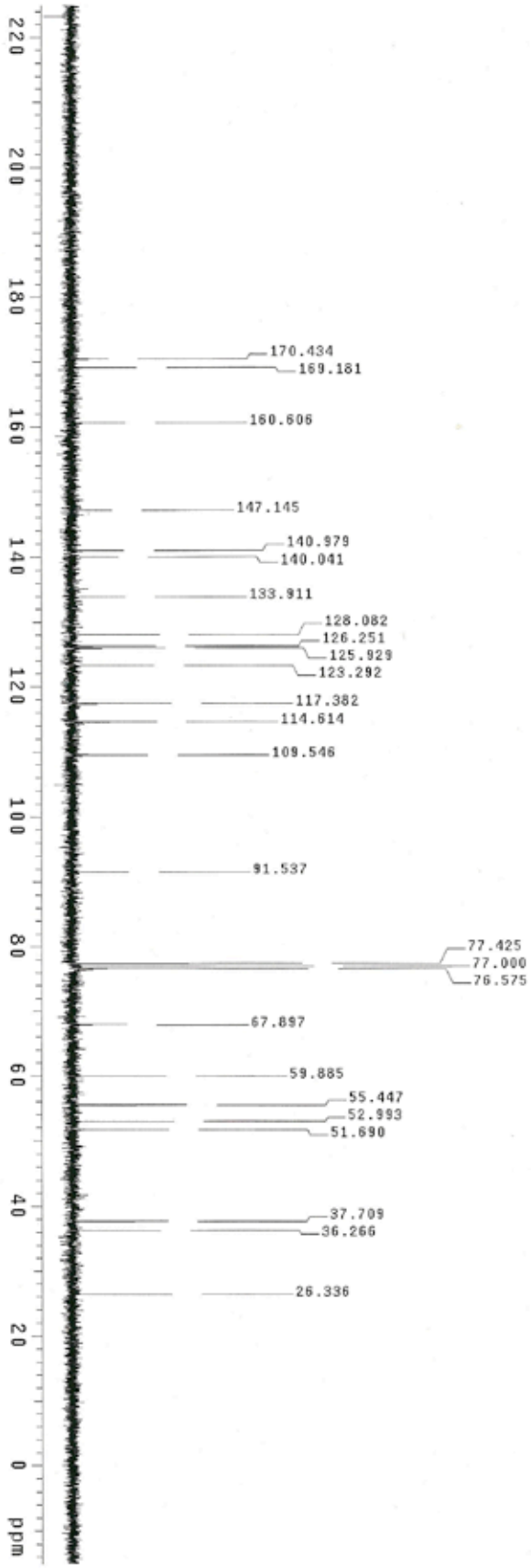

Std Carbon experiment
Sample: NB-S-DVP-34-H
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpatil
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 1.301 sec
Width 18115.9 Hz
256 repetitions
OBSERVE C13, 75.491142 MHz
DECOUPLE H1, 300.2251667 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 10 min, 45 sec
169.231
163.599
159.008
133.962
132.314
131.171
129.531
129.026
123.855
122.500
122.193
121.094
116.898
116.642
114.254
77.425
77.000
76.575
58.860
55.227
52.642
45.194
14.678
12.385
8.621
220
200
180
160
140
120
100
80
60
40
20
0
ppm

trans-**11l**


Me
Me
N
O
O
OMe
OMe


Std Proton parameters
Sample: [redacted]
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpatil
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
32 repetitions
OBSERVE H1, 300.2185002 MHz
DATA PROCESSING
FT size 65536
Total time 15 hr, 51 min, 3 sec
2.091.04
2.512.02
1.9805
3.0006
2.24
2.19
2.10
13
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
N
O
O
OMe
OMe

trans-**11m**



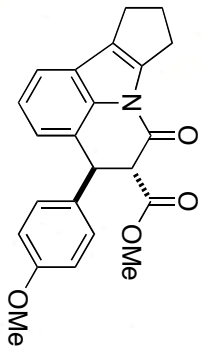
Std Carbon experiment

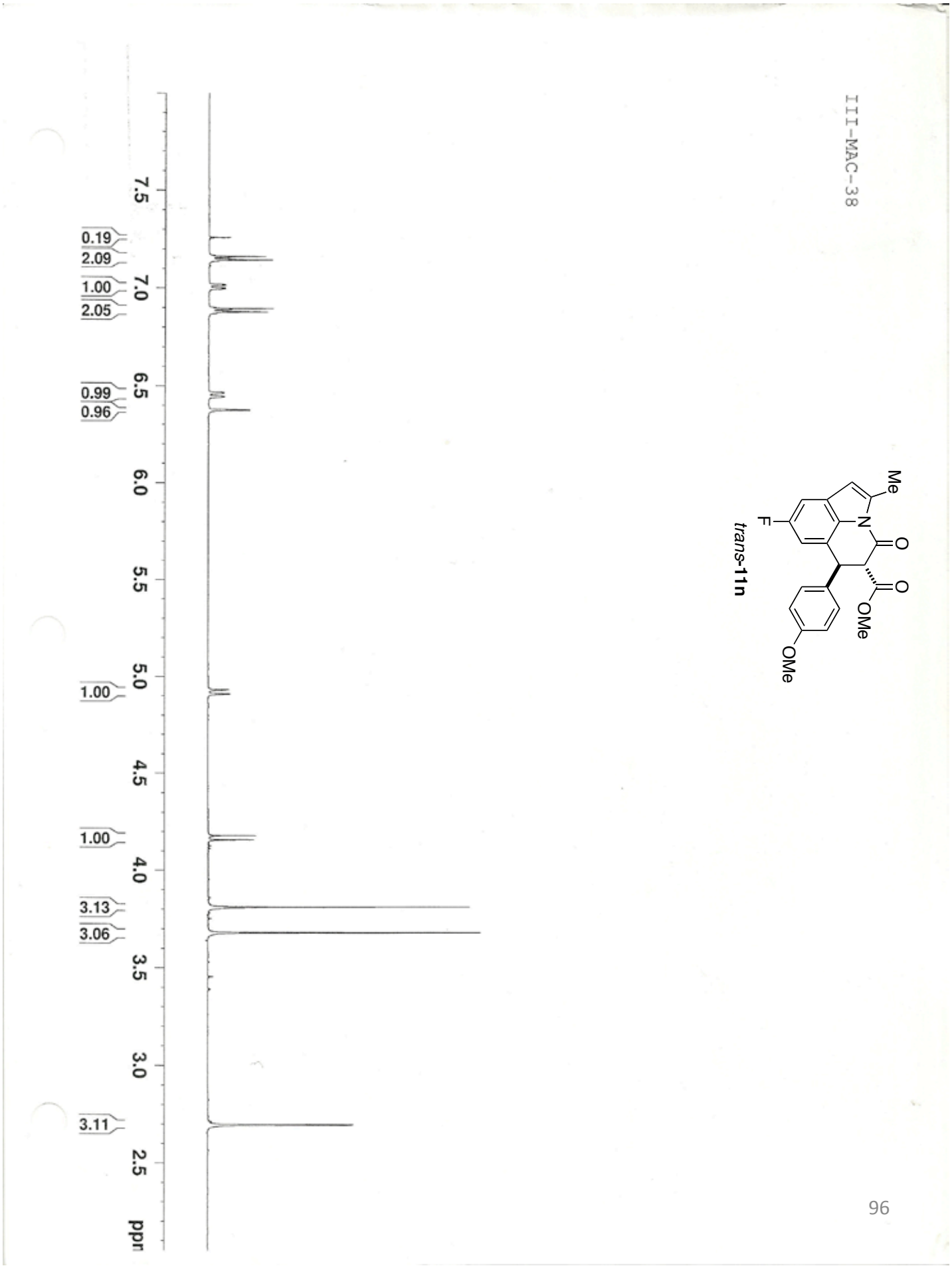
Sample: indolecyclopentyl-cyclo-H
File: xp

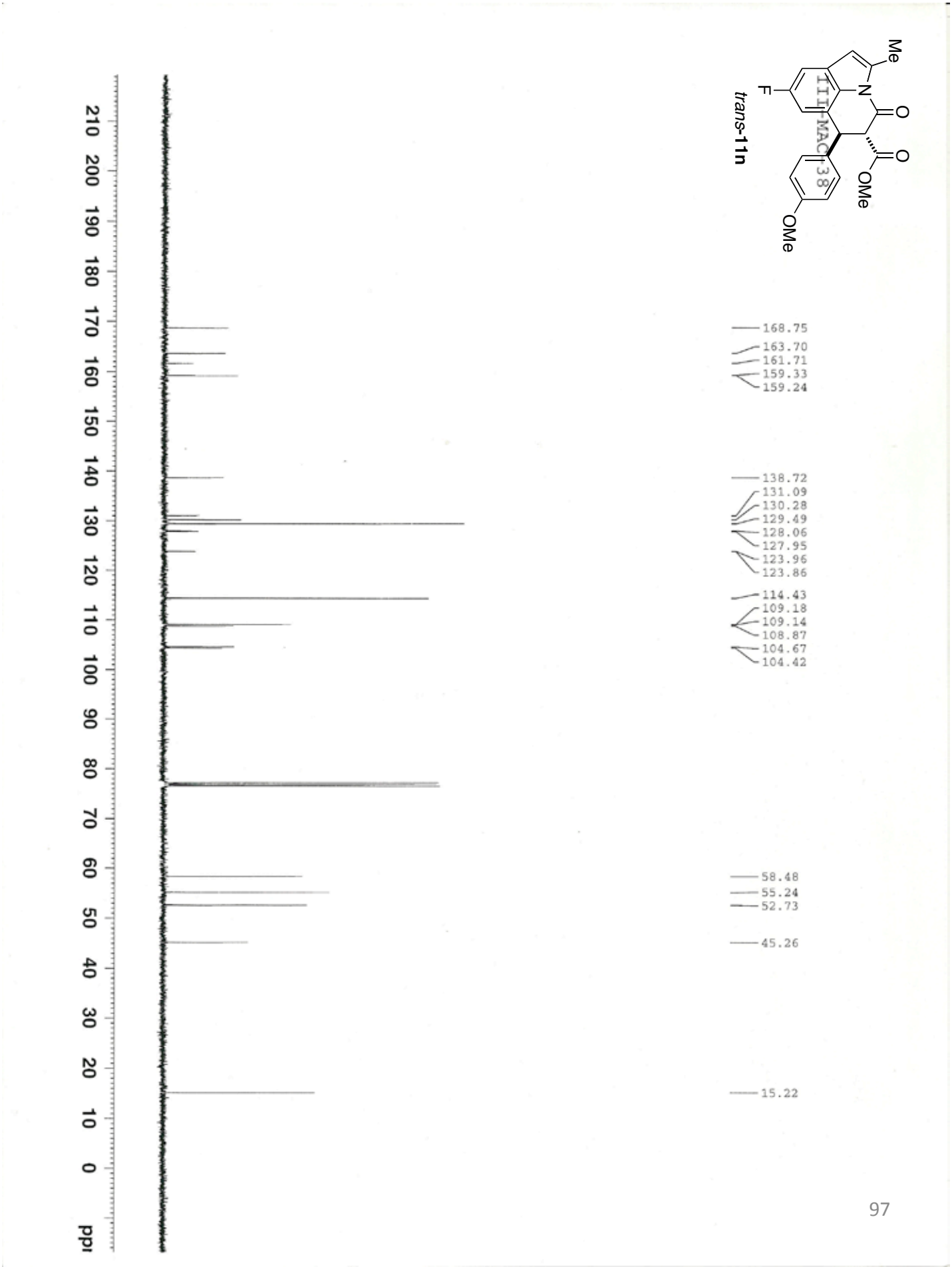
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpatil
Mercury-300 "r2d2"

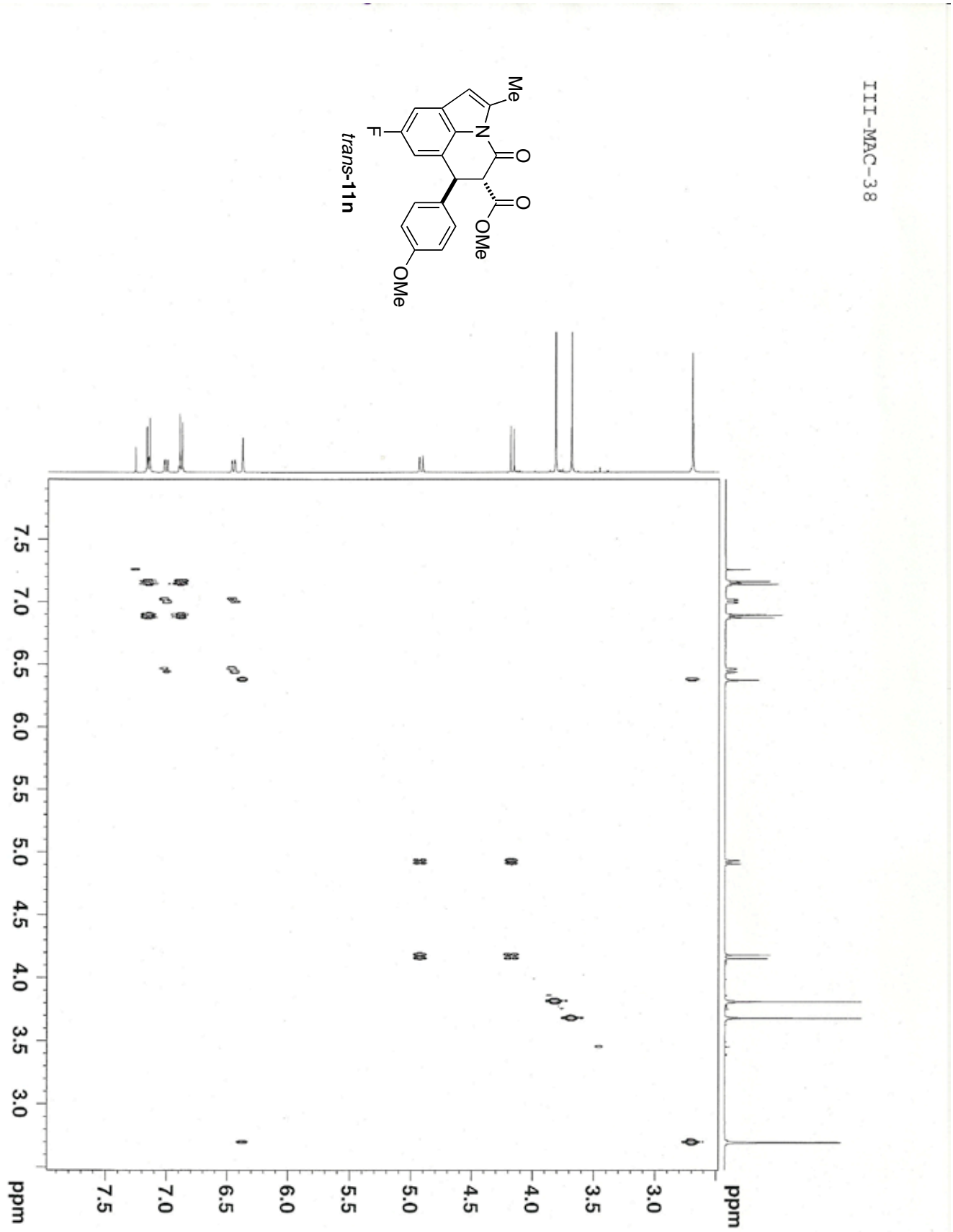
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 1.301 sec
Width 18115.9 Hz
80 repetitions
OBSERVE C13, 75.4900048 MHz
DECOUPLE H1, 300.2199481 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 10 hr, 41 min, 3 sec

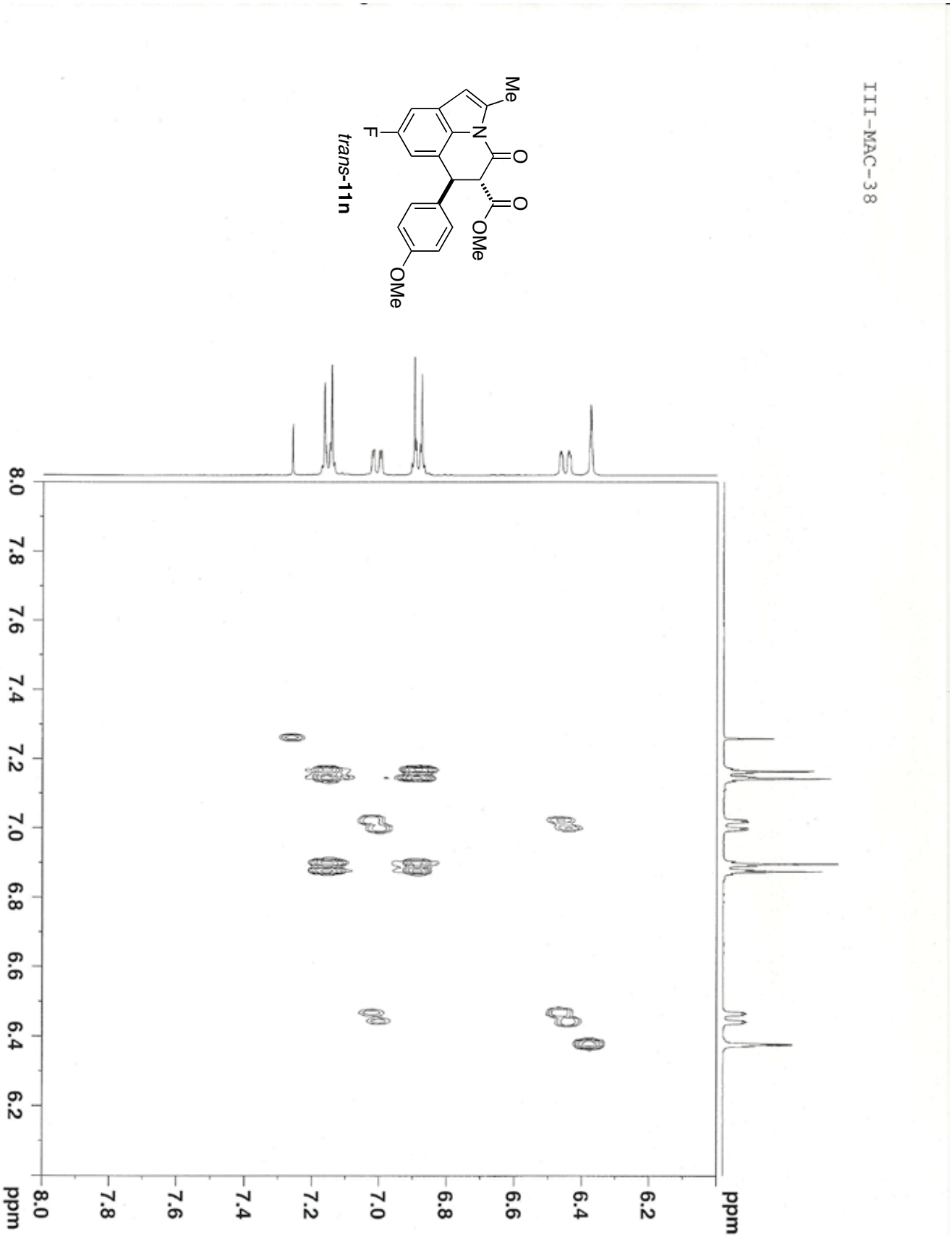
trans-**11m**

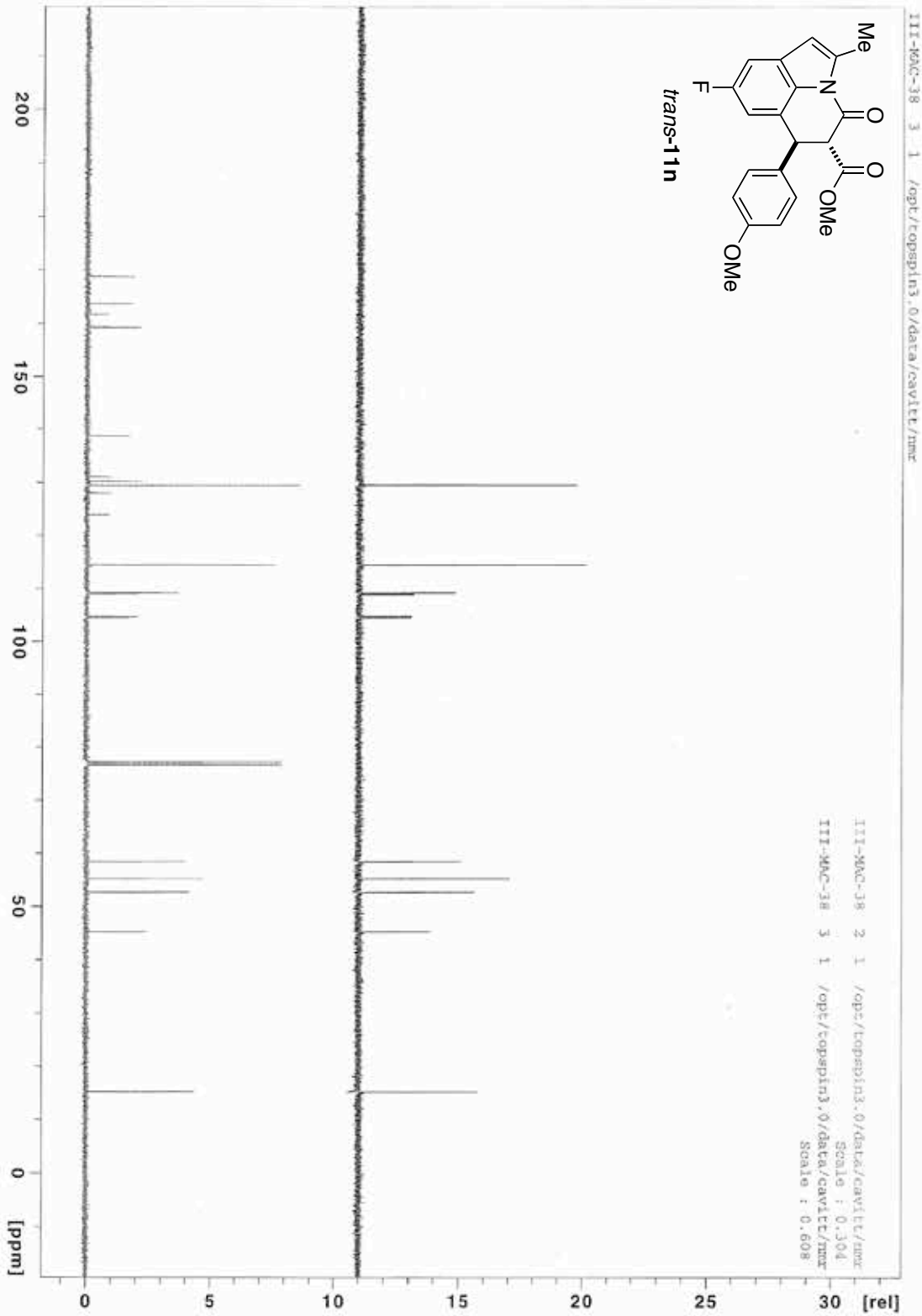


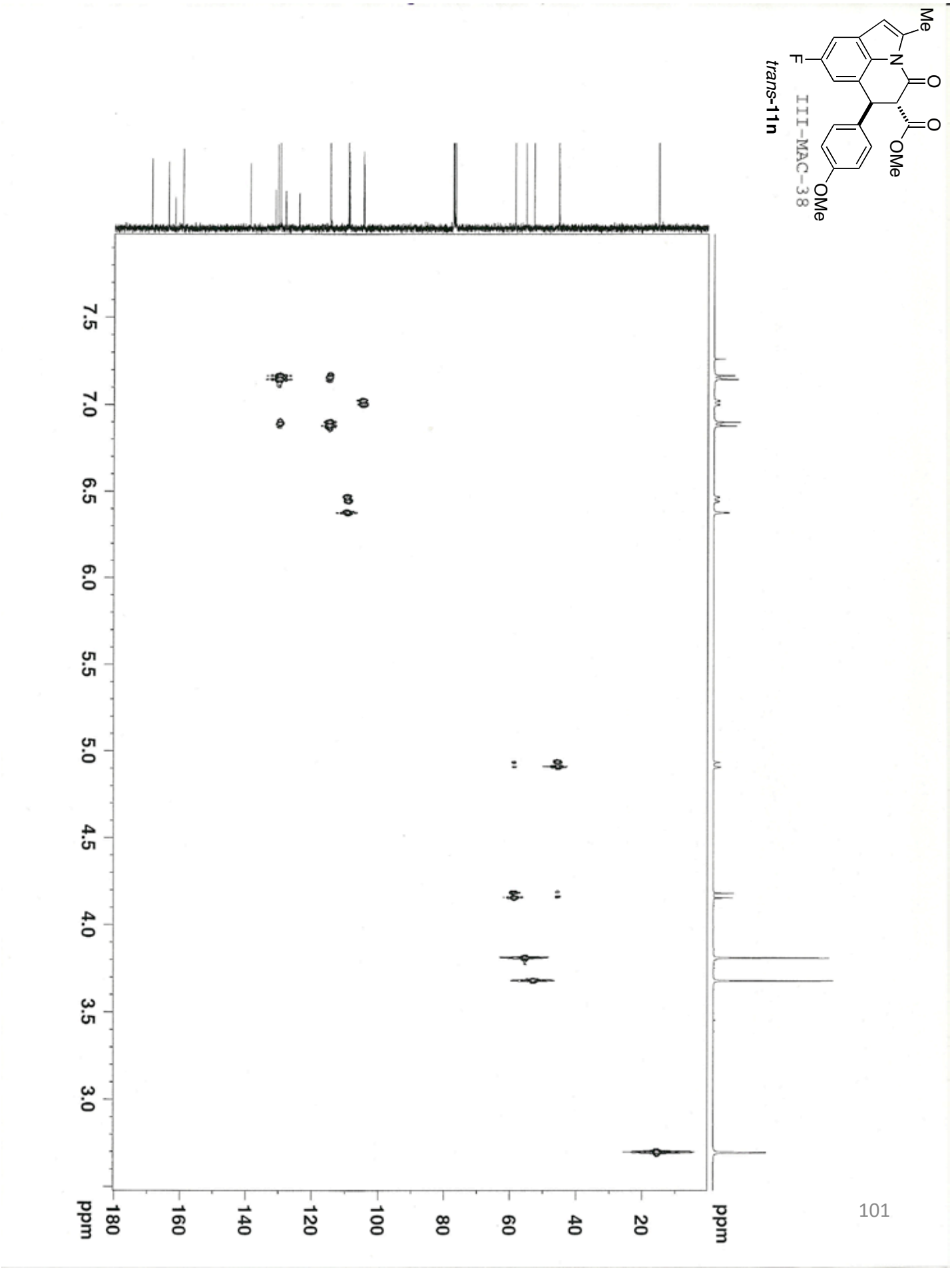

III-MAC-38
Me
F
N
O
O
OMe
OMe
trans-11n
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
ppm
0.19
2.09
1.00
2.05
0.99
0.96
1.00
1.00
3.13
3.06
3.11

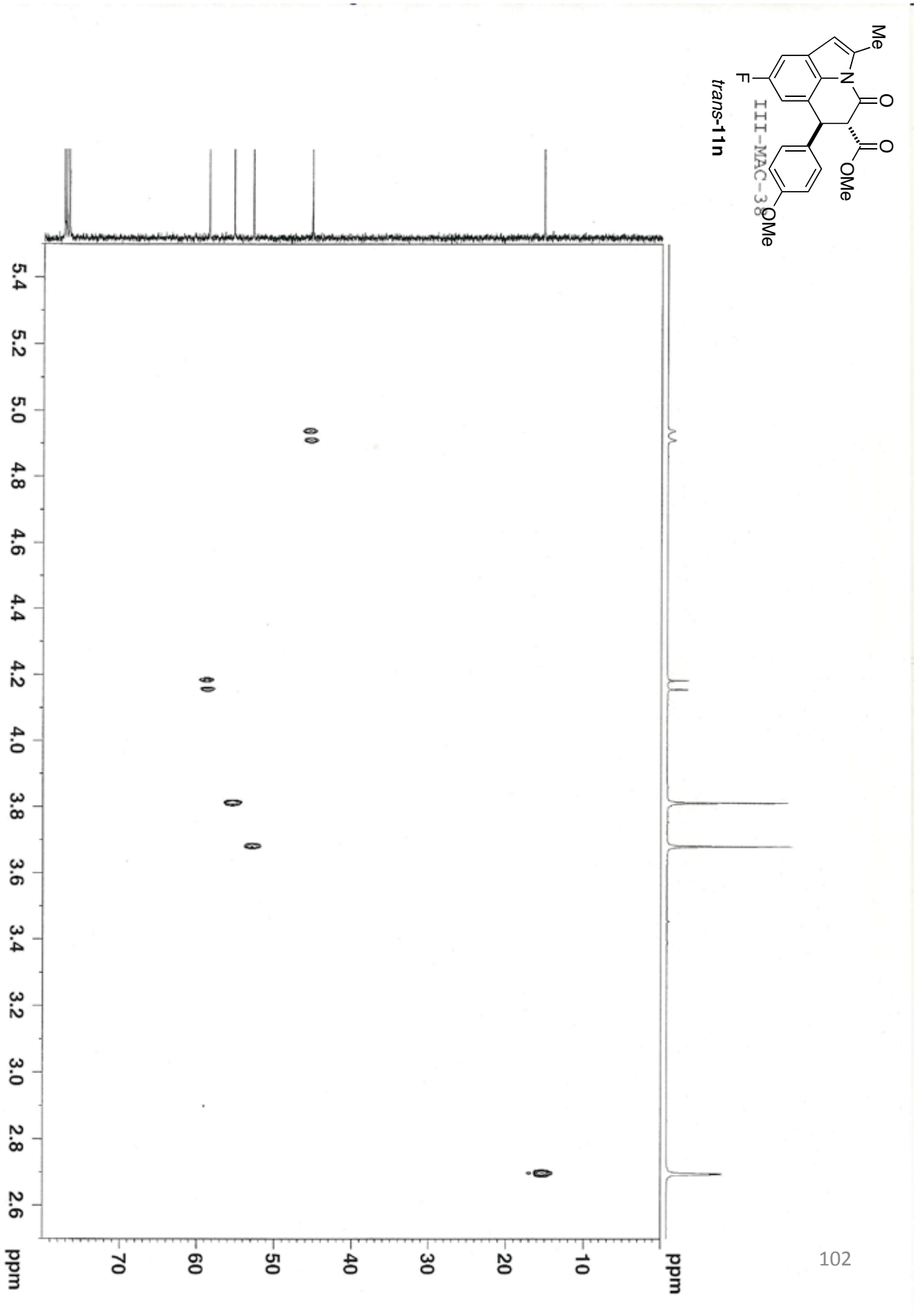

Me
F
N
O
O
OMe
OMe
trans-11n
168.75
163.70
161.71
159.33
159.24
138.72
131.09
130.28
129.49
128.06
127.95
123.96
123.86
114.43
109.18
109.14
108.87
104.67
104.42
58.48
55.24
52.73
45.26
15.22
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm

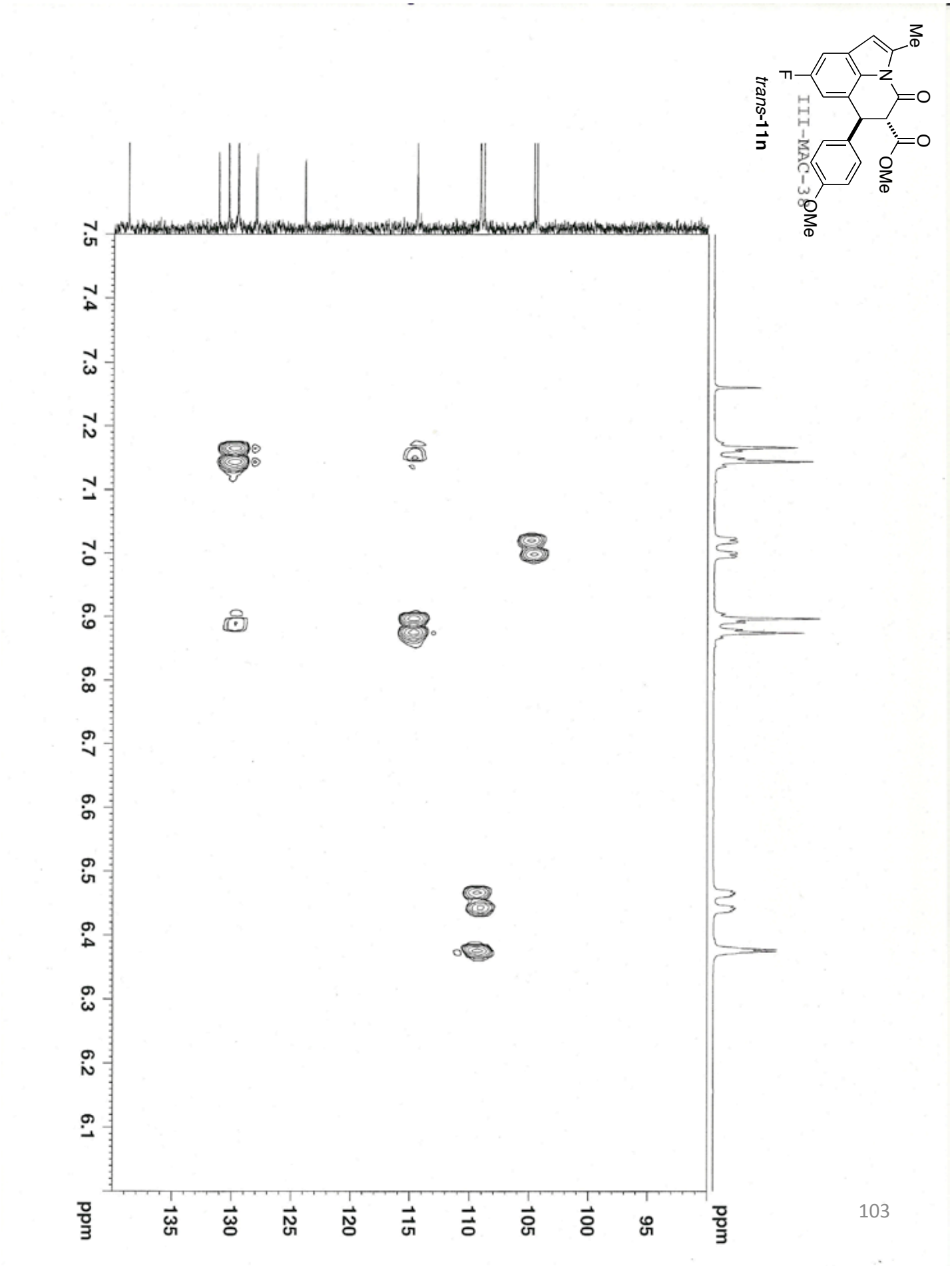

III-MAC-38
Me
F
N
O
O
OMe
OMe
trans-11n
ppm
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0

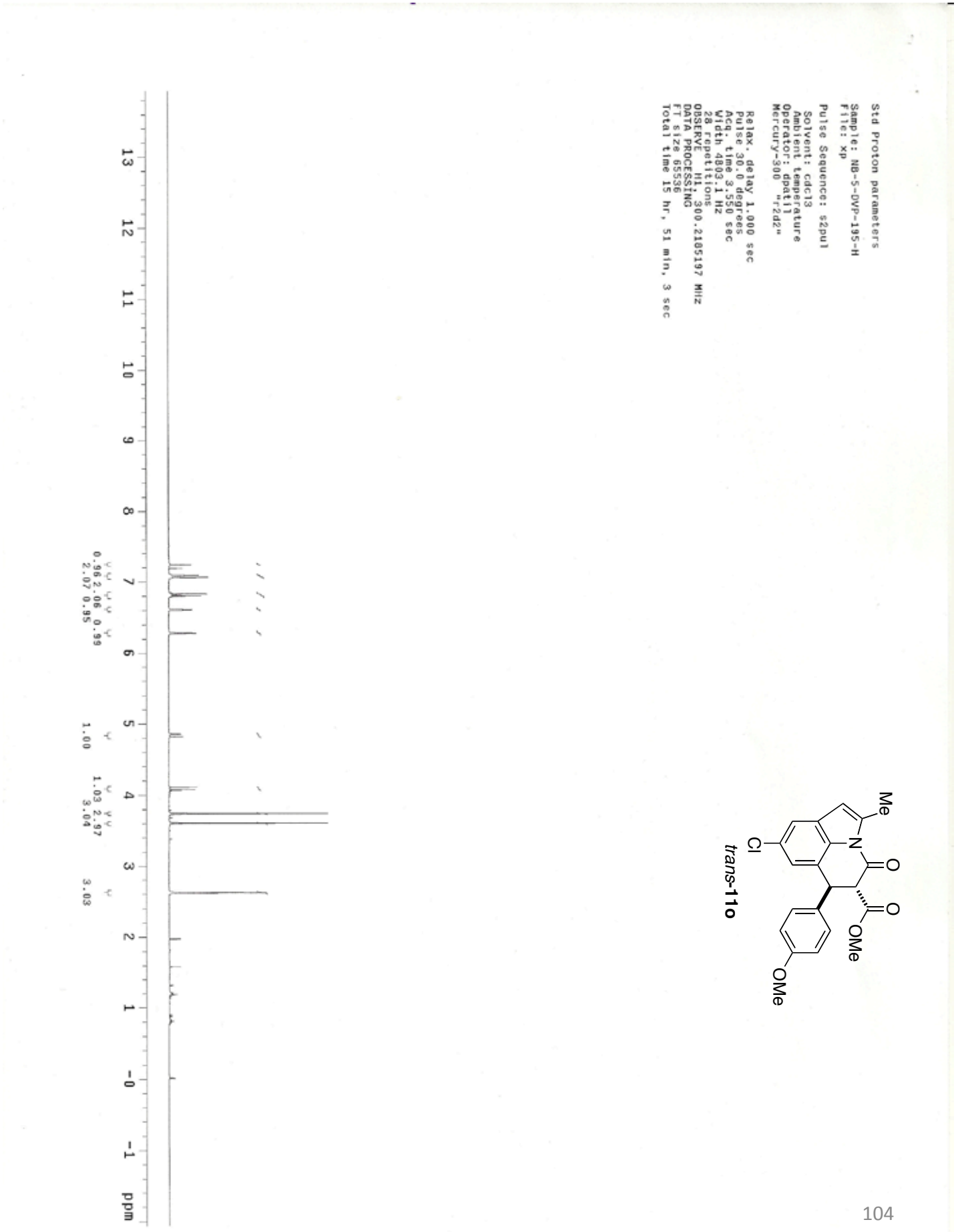

III-MAC-38
Me
F
N
O
O
OMe
OMe
trans-11n
8.0
7.8
7.6
7.4
7.2
7.0
6.8
6.6
6.4
6.2
ppm

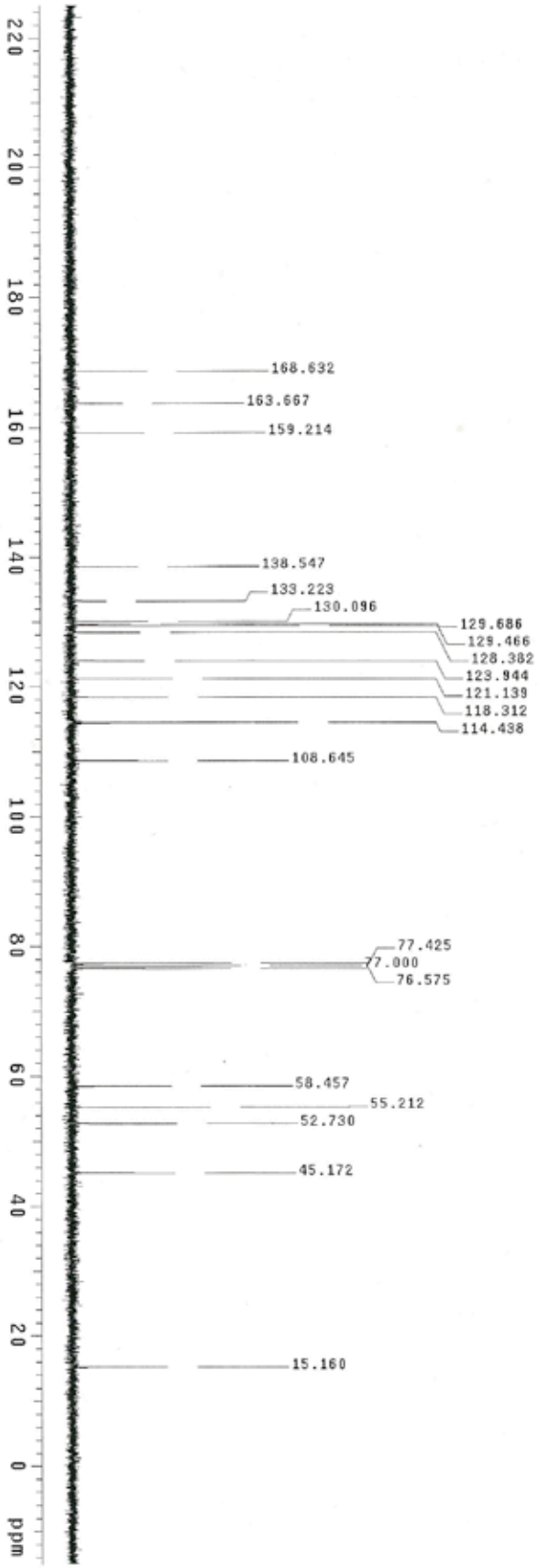

III-MAC-38 3 1 /opt/topspin3.0/data/cavitt/nmr
Me
N
O
O
OMe
F
OMe
trans-11n
III-MAC-38 2 1 /opt/topspin3.0/data/cavitt/nmr
Scale : 0.304
III-MAC-38 3 1 /opt/topspin3.0/data/cavitt/nmr
Scale : 0.608
200
150
100
50
0
[ppm]
0
5
10
15
20
25
30
[rel]


Me
N
O
O
OMe
F
OMe
III-MAC-38
trans-11n
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
ppm
180
160
140
120
100
80
60
40
20
ppm

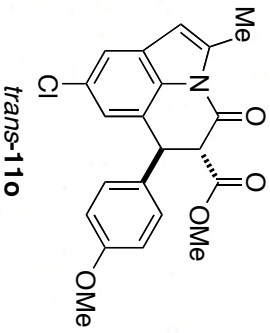



Me
N
O
O
OMe
F
OMe
III-MAC-3
trans-11n
7.5
7.4
7.3
7.2
7.1
7.0
6.9
6.8
6.7
6.6
6.5
6.4
6.3
6.2
6.1
ppm
135
130
125
120
115
110
105
100
95
ppm

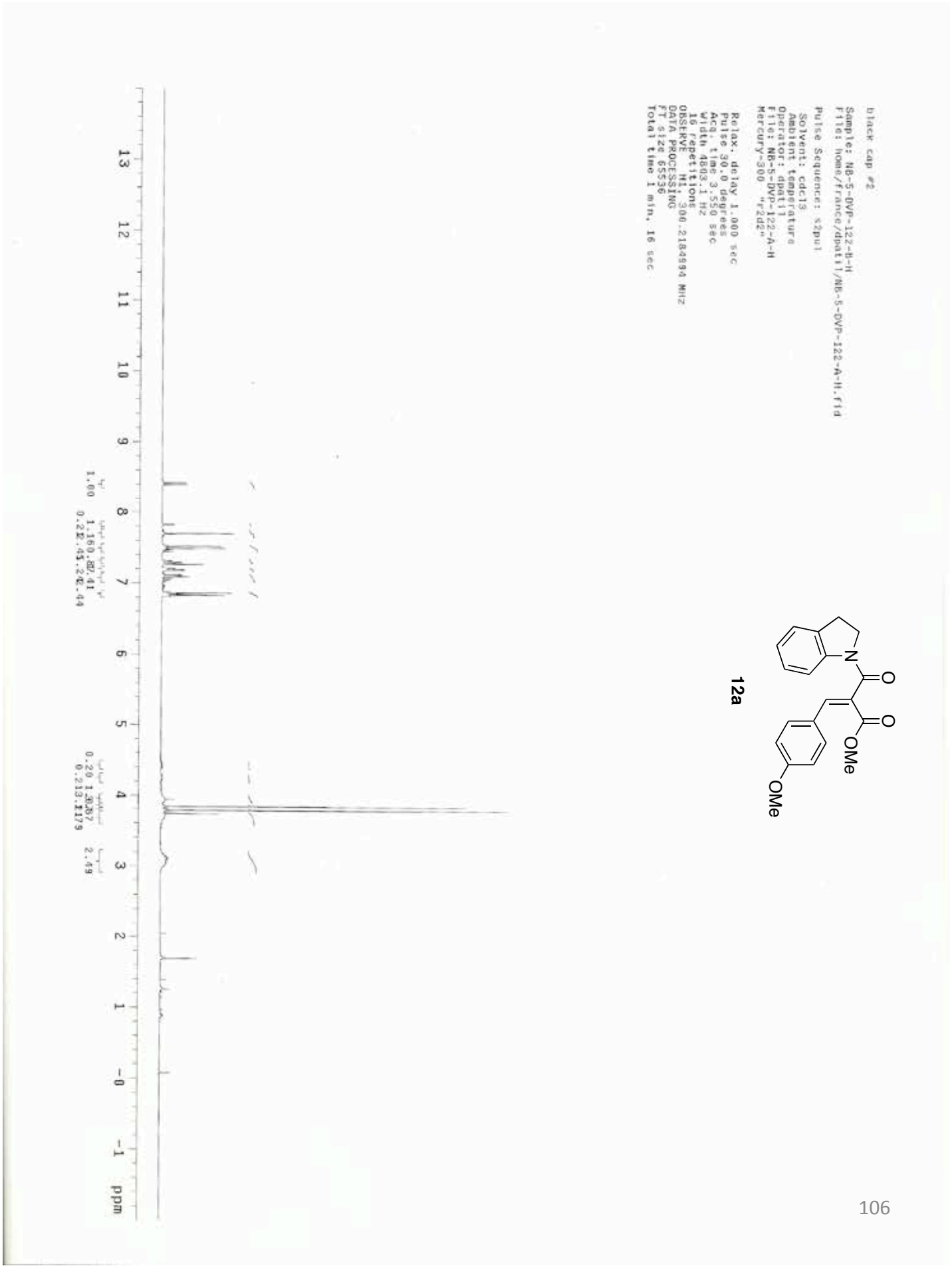

Std Proton parameters
Sample: NB-5-DVP-195-H
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpati1
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
28 repetitions
OBSERVE H1, 300.2185197 MHz
DATA PROCESSING
FT size 65536
Total time 15 hr, 51 min, 3 sec
Me
N
O
O
OMe
OMe
Cl
trans-11o
0.96 2.06 0.99
2.07 0.95
1.00
1.03 2.97
3.04
3.03
13
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm

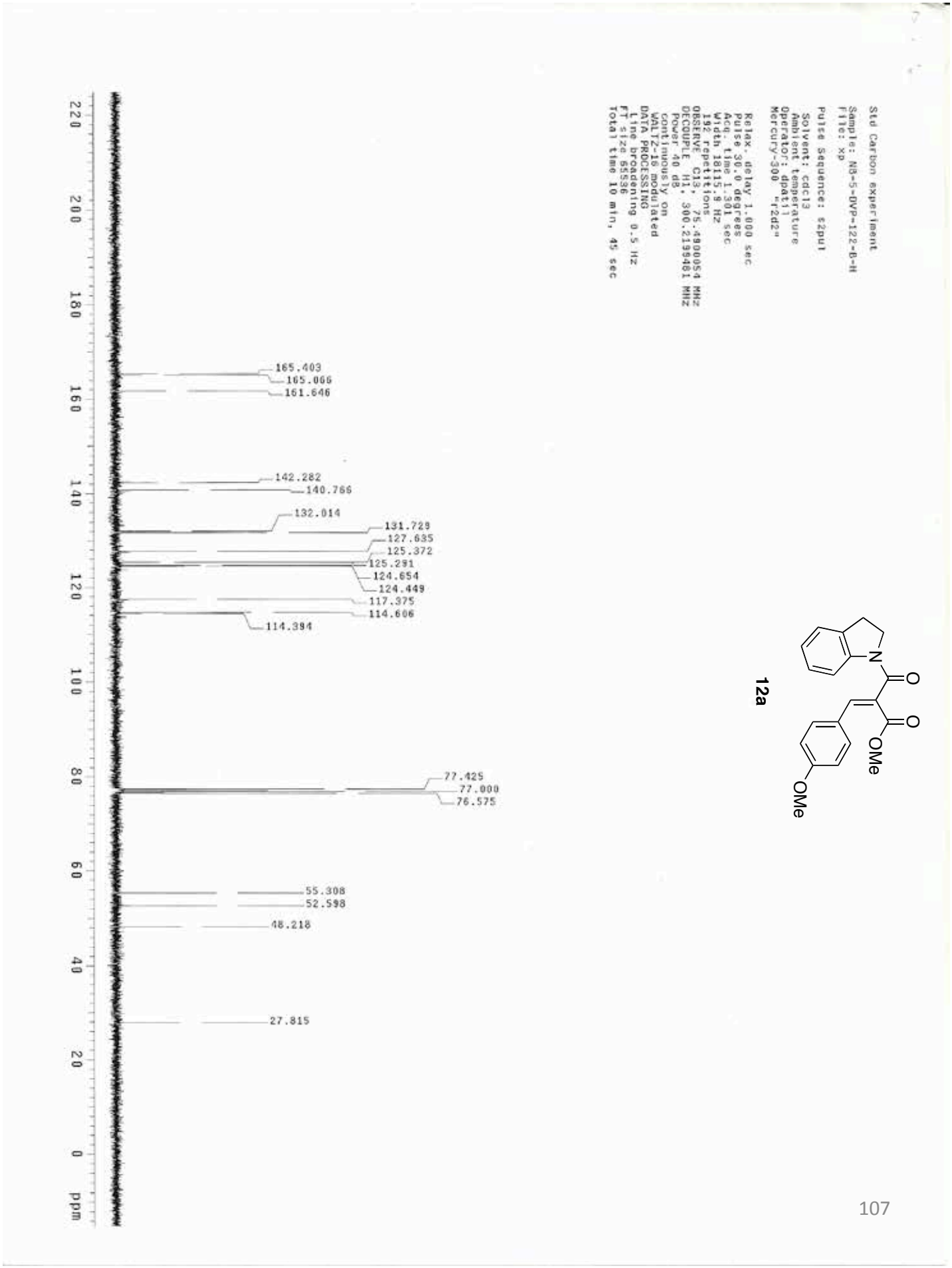



```
Std Carbon experiment

Sample: NB-5-DVP-195-H
File: xp

Pulse Sequence: s2pul

 Solvent: cdcl3
 Ambient temperature
Operator: dpatil
Mercury-300 "r2d2"

 Relax. delay 1.000 sec
 Pulse 30.0 degrees
 Acq. time 1.301 sec
 Width 18115.9 Hz
  58 repetitions
OBSERVE  C13, 75.4900059 MHz
DECOUPLE  H1, 300.2199481 MHz
 Power 40 dB
  continuously on
  WALTZ-16 modulated
DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 65536
Total time 10 hr, 41 min, 3 sec
```



trans-**11o**


black cap #2
Sample: NB-5-DVP-122-B-H
File: home/franco/dpatil/NB-5-DVP-122-A-H.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpatil
File: NB-5-DVP-122-A-H
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 3.550 sec
Width 4803.1 Hz
16 repetitions
OBSERVE H1, 300.2184994 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 16 sec
O
O
OMe
OMe
N
12a
13
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
1.00
1.16
0.22
0.20
0.21
2.49

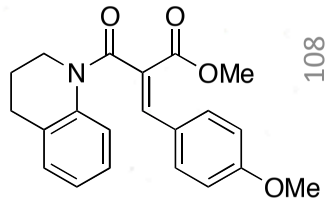

Std Carbon experiment
Sample: NB-5-DVP-122-B-H
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpatil
Mercury-300 "r2d2"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 1.301 sec
Width 18115.9 Hz
192 repetitions
OBSERVE C13, 75.4900054 MHz
DECOUPLE H1, 300.2199481 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 10 min, 45 sec
165.403
165.066
161.646
142.282
140.766
132.014
131.729
127.635
125.372
125.291
124.654
124.449
117.375
114.606
114.394
77.425
77.000
76.575
55.308
52.598
48.218
27.815
220
200
180
160
140
120
100
80
60
40
20
0
ppm
12a
N
O
O
OMe
OMe

7b

File: xp

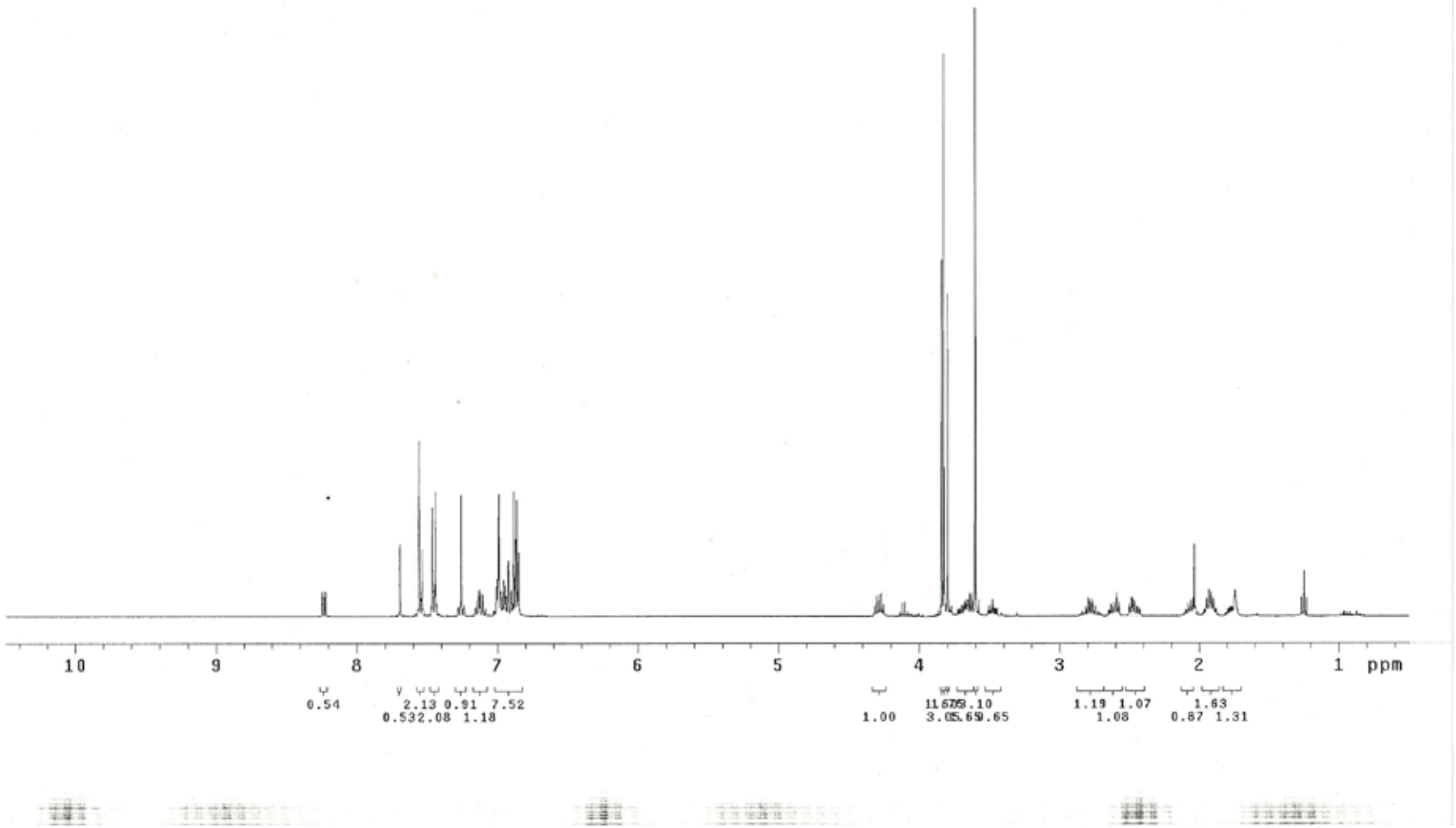
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Operator: cavitt
Mercury-400BB "amidala"

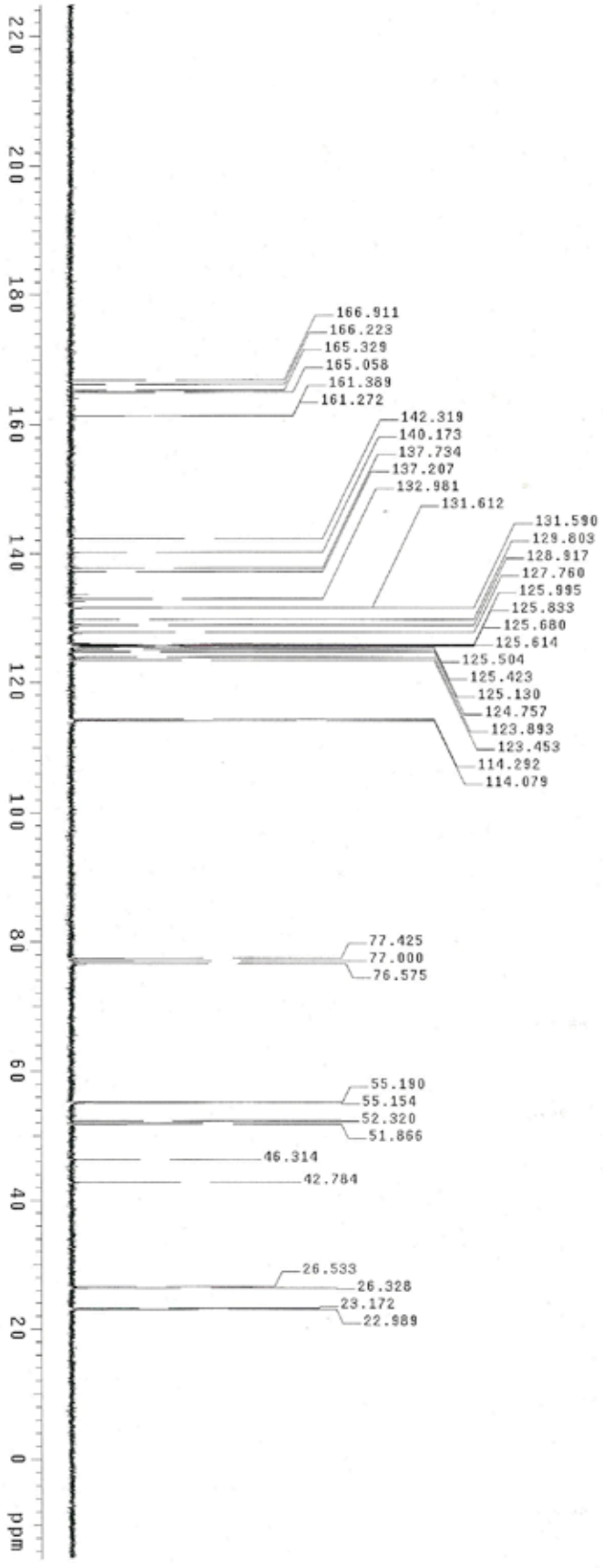
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 2.659 sec
Width 6398.0 Hz
47 repetitions
OBSERVE H1, 399.9551931 MHz
DATA PROCESSING
FT size 65536
Total time 7 min, 5 sec



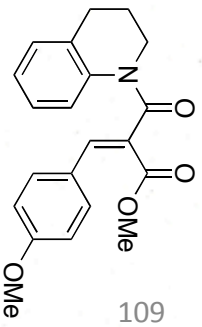
trans-**12b**

(crude mixture)



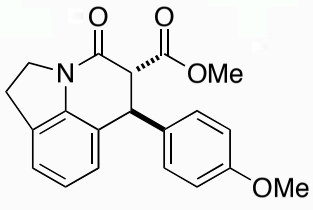



```
Std Carbon experiment

Sample: NB-5-DVP-122-B-H
File: xp

Pulse Sequence: s2pul

  Solvent: cdcl3
  Ambient temperature
Operator: dpatil
Mercury-300 "r2d2"

  Relax. delay 1.000 sec
  Pulse 30.0 degrees
  Acq. time 1.301 sec
  Width 18115.9 Hz
  128 repetitions
OBSERVE   C13, 75.4900170 MHz
DECOUPLE  H1, 300.2199481 MHz
  Power 40 dB
  continuously on
  WALTZ-16 modulated
DATA PROCESSING
  Line broadening 0.5 Hz
  FT size 65536
Total time 10 min, 45 sec
```



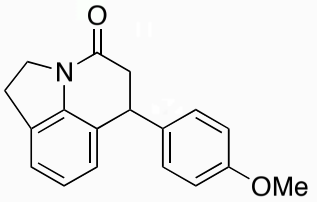
trans-**12b**
(crude mixture)

```
Std Proton parameters

File: home/france/dpatil/NB-5-DVP-142-A-HH.fid

Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
Operator: dpatil
File: NB-5-DVP-142-A-HH
Mercury-300  "r2d2"

 Relax. delay 1.000 sec
 Pulse 30.0 degrees
 Acq. time 3.550 sec
 Width 4803.1 Hz
 16 repetitions
OBSERVE    H1, 300.2184988 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 16 sec
```

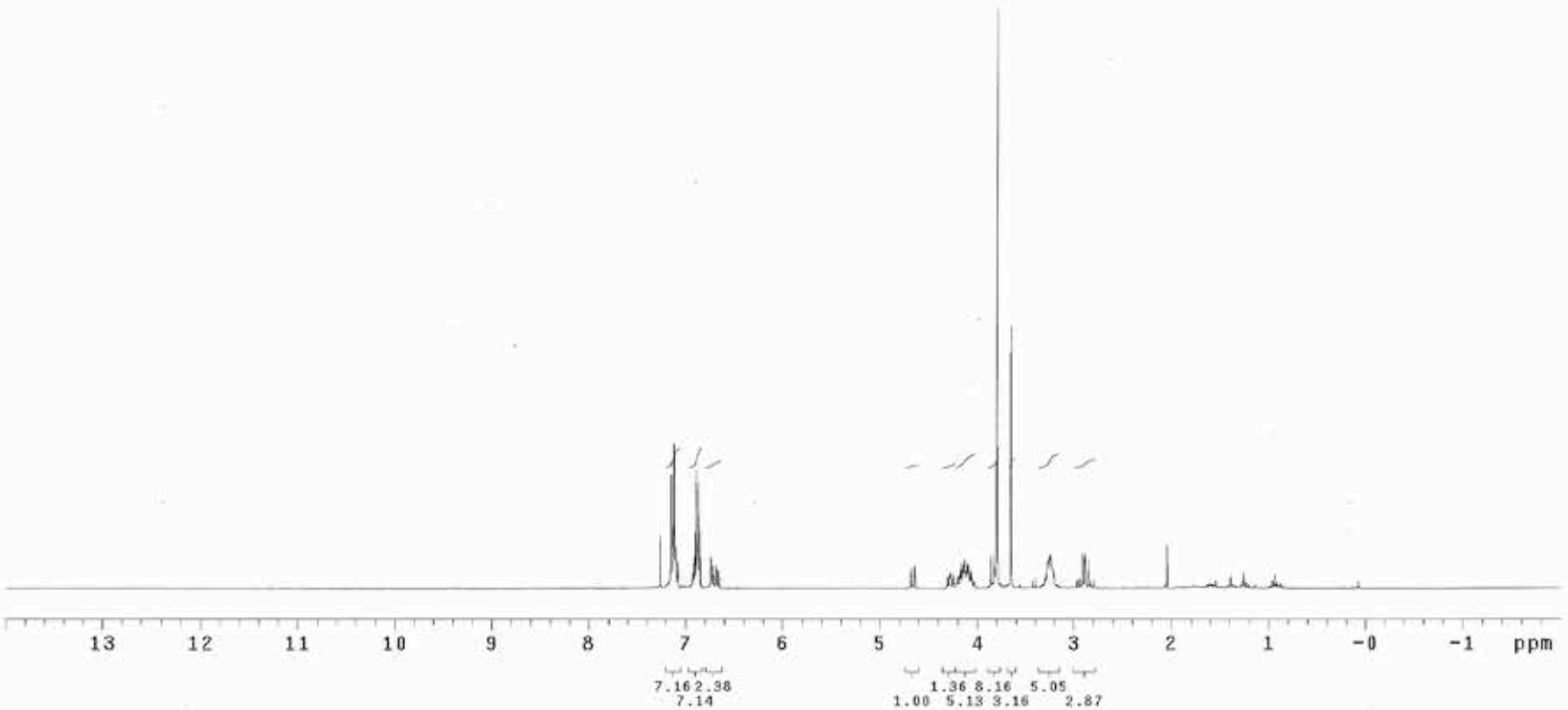


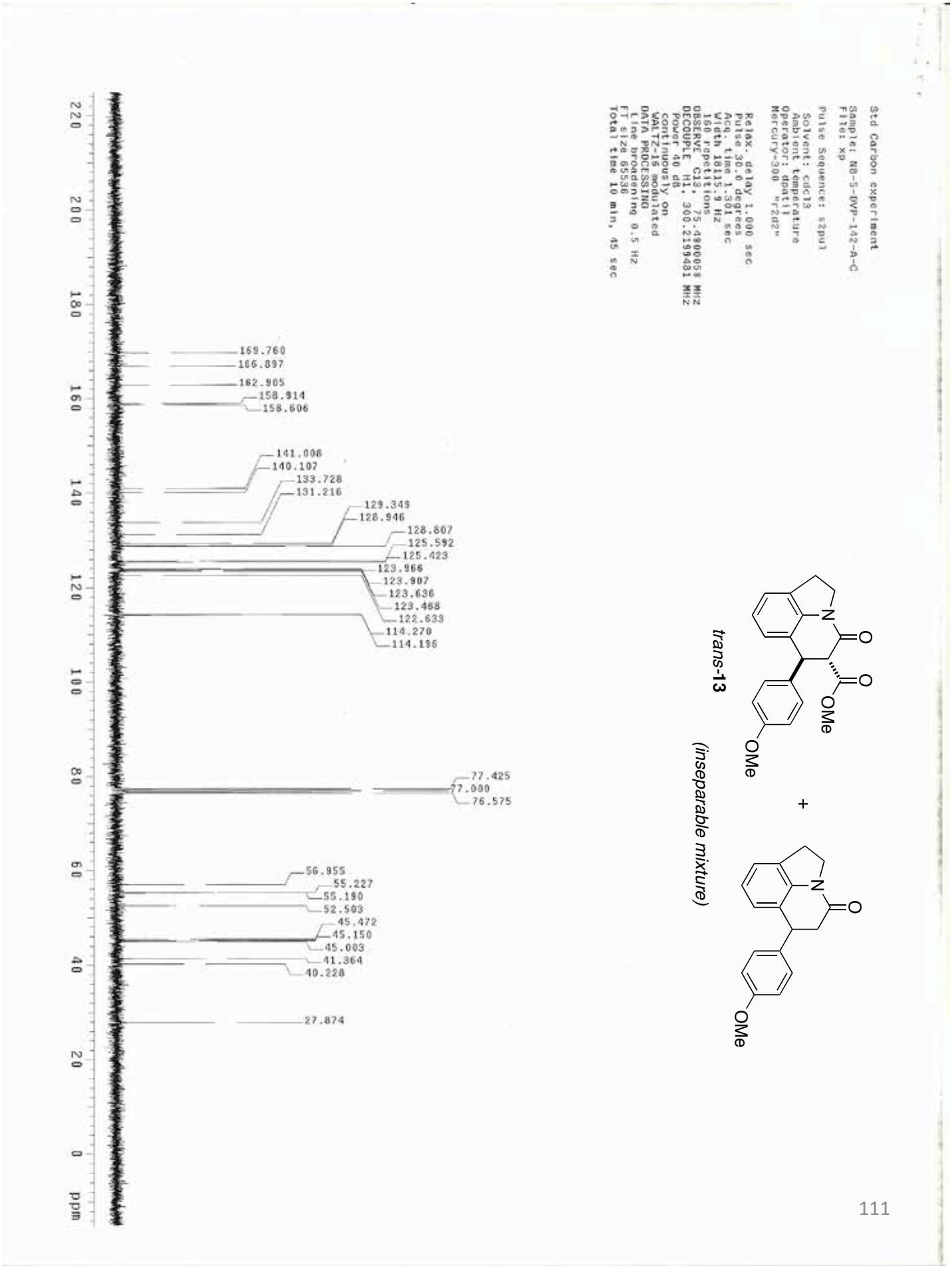
trans-**13**

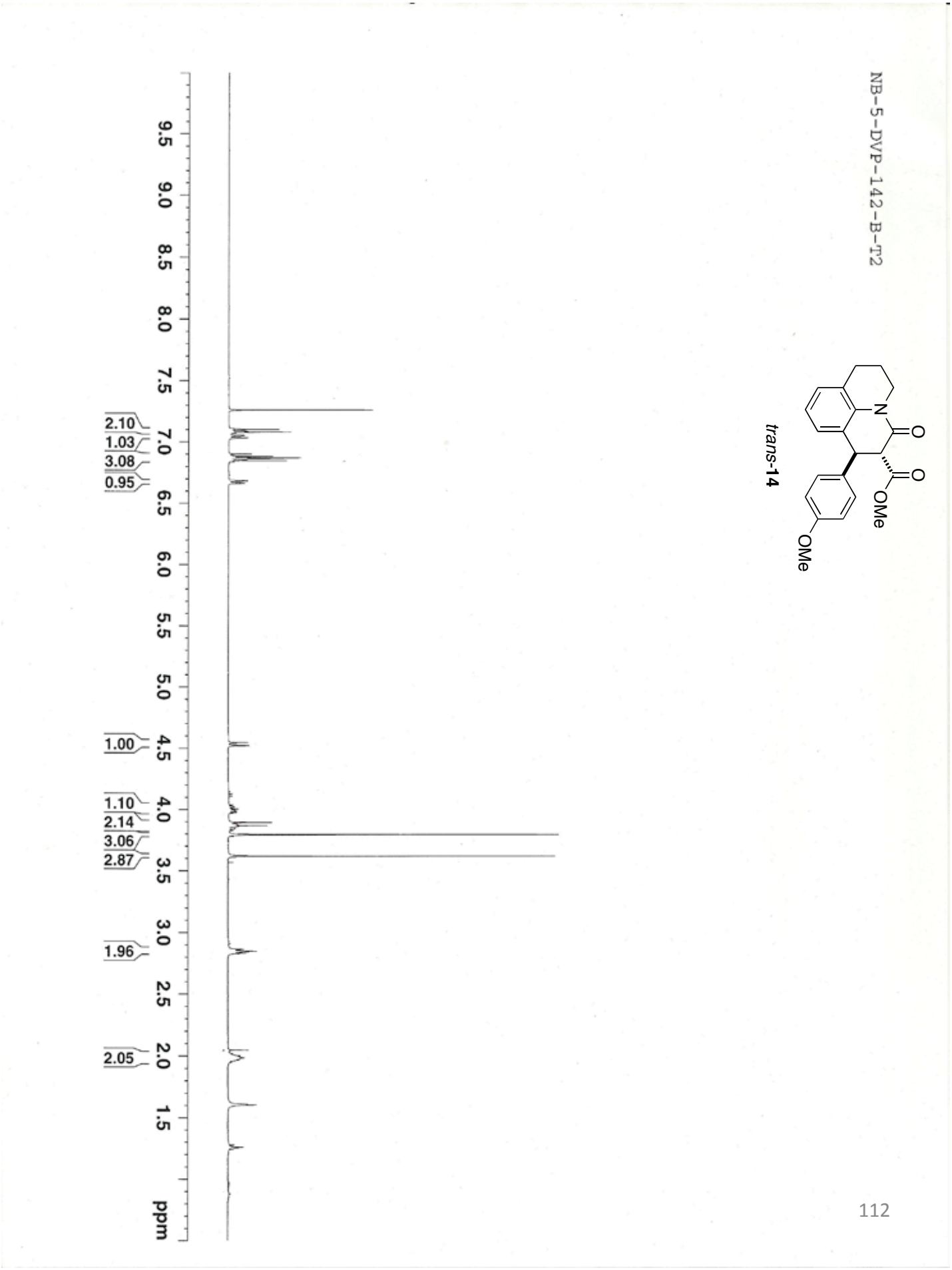
+

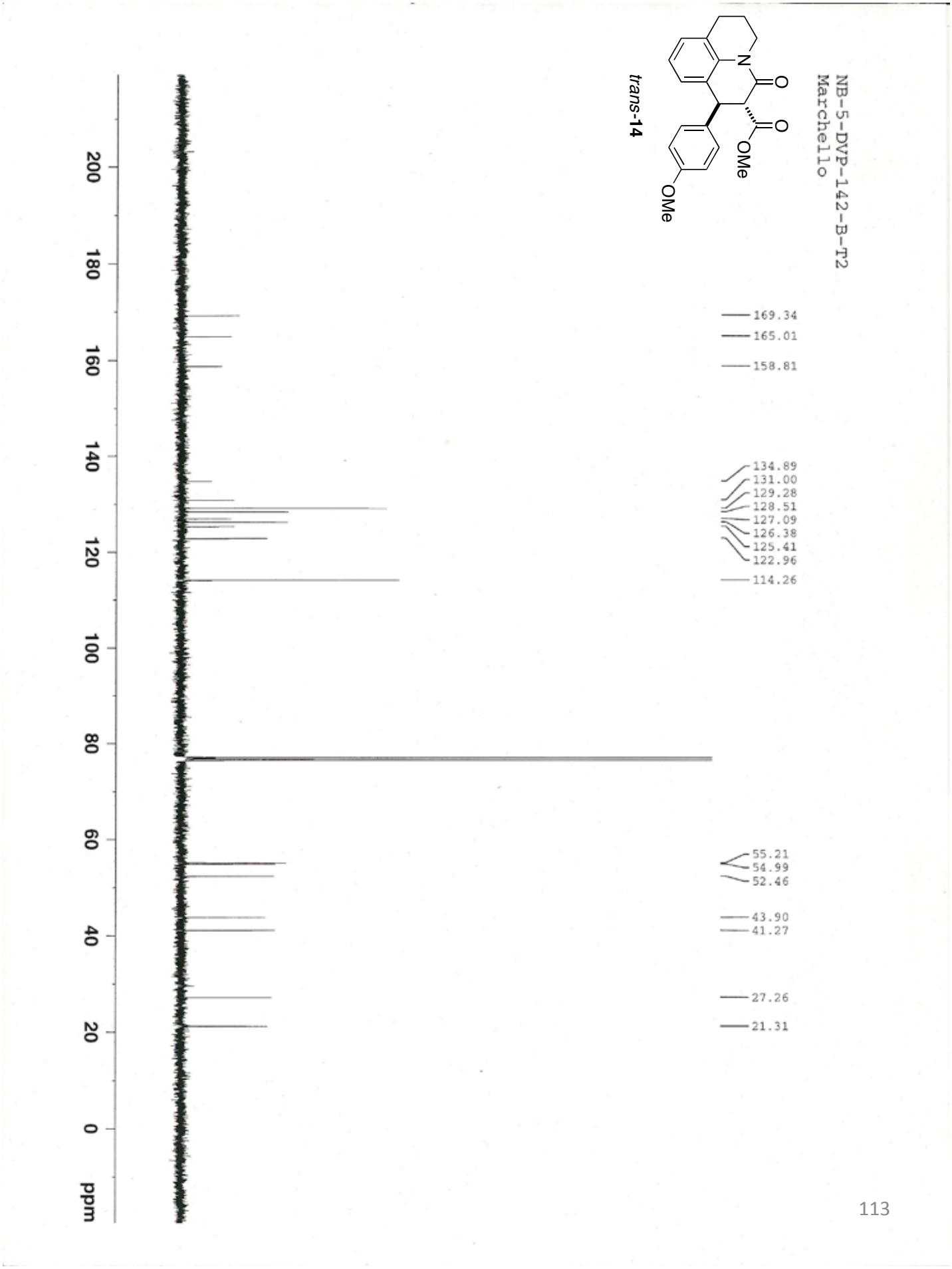


(inseparable mixture)




Std Carbon experiment
Sample: NB-5-DVP-142-A-C
File: xp
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: dpatil
Mercury-300 "r202"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 1.301 sec
Width 18115.9 Hz
160 repetitions
OBSERVE C13, 75.4900058 MHz
DECOUPLE H1, 300.2199401 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 10 min, 45 sec
169.760
166.897
162.905
158.914
158.606
141.008
140.107
133.728
131.216
129.348
128.946
128.807
125.592
125.423
123.966
123.907
123.636
123.468
122.633
114.270
114.196
77.425
77.000
76.575
56.955
55.227
55.190
52.503
45.472
45.150
45.003
41.364
40.228
27.874
220
200
180
160
140
120
100
80
60
40
20
0
ppm
N
O
O
OMe
OMe
+
N
O
OMe
trans-13
(inseparable mixture)


NB-5-DVP-142-B-T2
trans-14
N
O
O
OMe
OMe
9.5
9.0
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
ppm
2.10
1.03
3.08
0.95
1.00
1.10
2.14
3.06
2.87
1.96
2.05


NB-5-DVP-142-B-T2
Marchello
trans-14
OMe
OMe
169.34
165.01
158.81
134.89
131.00
129.28
128.51
127.09
126.38
125.41
122.96
114.26
55.21
54.99
52.46
43.90
41.27
27.26
21.31
200
180
160
140
120
100
80
60
40
20
0
ppm