Silver-Catalyzed Formal Inverse Electron-Demand Diels-Alder Reaction of 1,2-Diazines and Siloxy Alkynes

Supporting Information

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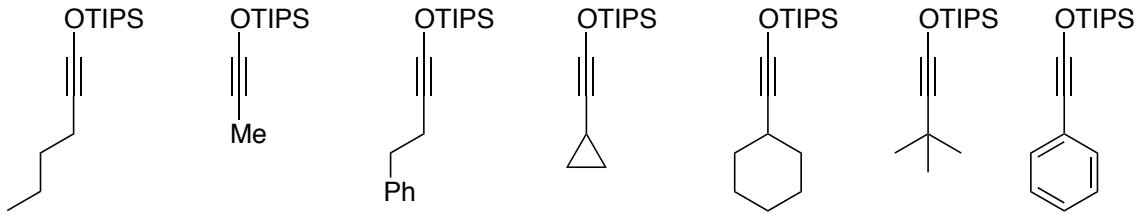
General Information. All reactions were performed using oven or flame-dried glassware under an inert atmosphere of nitrogen. Silver-catalyzed reactions were performed with the fume hood lights off, but no other precautions were taken to exclude ambient light. Reactions were monitored by thin-layer chromatography (TLC) on Whatman silica gel 60 Å F254 plates and visualized by UV and/or with $KMnO_4$ staining solution. Flash column chromatography was performed on Silicycle 40-63 μm Flash silica gel. NMR spectra were measured on Brüker DRX and DMX spectrometers at 500 MHz for ^1H spectra and 125 MHz for ^{13}C spectra and calibrated from internal standard (TMS, 0 ppm) or residual solvent signals (chloroform at 7.26 ppm, benzene at 7.16 ppm and methylene chloride at 5.32 ppm for ^1H spectra; chloroform at 77.0 ppm, benzene at 128.39 ppm and methylene chloride at 54.0 ppm for ^{13}C spectra). ^1H-NMR data are reported as follows: chemical shift (parts per million, ppm), multiplicity (s = singlet, d = doublet, t = triplet, quin = quintet, sep = septet, dd = doublet of doublets, dt = doublet of triplets, m = multiplet, br = broad, app = apparent), coupling constant (Hz) and integration. Infrared spectra were measured on a Nicolet 6700 FT-IR spectrometer on NaCl plates. Mass spectral analysis was performed by the College of Sciences Major Instrumentation Cluster at Old Dominion University (Norfolk, VA) directed by Susan Hatcher. Melting points were determined using a Thomas Hoover Uni-Melt Capillary Melting Point Apparatus and are uncorrected.

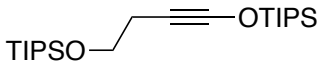
Materials. Dichloromethane (CH_2Cl_2) and tetrahydrofuran (THF) were purified by passage over activated alumina using an Innovative Technology solvent purification system. Triisopropyl trifluoromethanesulfonate (TIPSOTf) was distilled under reduced pressure over calcium hydride. Hexamethyldisilazane (HMDS) was distilled under positive pressure of nitrogen over calcium hydride. Phthalazine was purified by flash column chromatography (ethyl acetate) and stored in a desiccator. 1-chlorophthalazine was purified by flash column chromatography (gradient of EtOAc/CH_2Cl_2) prior to use. Silver trifluoromethanesulfonate (AgOTf) (99%) was purchased from Strem Chemicals, Inc. and stored in a desiccator. Silver bis(trifluoromethanesulfonyl)imide ($AgNTf_2$) (97%) was purchased from Aldrich Chemical Co. and stored in a desiccator. All other commercially available reagents were used as received unless stated otherwise.

Preparation of Siloxy Alkynes



2a **2g** **2h** **2j** **2k** **2l** **2m**

The previously known siloxy alkynes **2a**,[1] **2g**,[2] **2h**,[3] **2j**,[4] **2k**,[2] **2l**[5] and **2m**[2] were prepared according to the reported procedures.



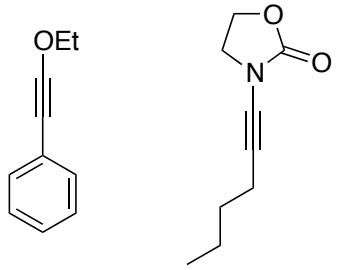
2i

A 500-mL flame-dried, three-necked, round-bottomed flask equipped with a stir bar, fitted with rubber septa and a nitrogen inlet was charged with THF (125 mL) and (but-3-yn-1-yloxy)triisopropylsilane (9.06g, 40.0 mmol). The resulting solution was cooled to -78 °C and anhydrous *t*-BuOOH (10.0 mL of a 4.40 M solution in nonane, 44.0 mmol) was added dropwise. CAUTION! SOLUTIONS OF OXIDANTS AND OXIDIZABLE SUBSTRATES ARE POTENTIALLY HAZARDOUS AND POSSIBLY SUBJECT TO VIOLENT DECOMPOSITION BY ADVENTITIOUS CATALYSIS. A syringe pump was used to add freshly prepared LiHMDS (96.0 mL of a 1M solution in THF, 96.0 mmol) to the reaction mixture over a period of 30 minutes. The resultant mixture was allowed to warm to 0 °C, and was stirred at this temperature for 2 h. The reaction mixture was then cooled to -78 °C, TIPSOTf (11.82 mL, 44.0 mmol) was added dropwise via a syringe pump over a period of 10 min, and the mixture was allowed to stir for 5 min at this temperature. The reaction vessel was transferred to a 0 °C ice water bath and was

[1] Schramm, M. P.; Shubinets, V.; Kozmin, S. A. *Org. Synth.* **2010**, *87*, 253-263.
[2] Sun, J.; Keller, V. A.; Meyer, S. T.; Kozmin, S. A. *Adv. Synth. Catal.* **2010**, *352*, 839-842.
[3] Zhang, L.; Kozmin, S. A. *J. Am. Chem. Soc.* **2004**, *126*, 10204-10205.
[4] Montavon, T. J.; Li, J.; Cabrera-Pardo, J. R.; Mrksich, M.; Kozmin, S. A. *Nat. Chem.* **2012**, *4*, 45-51.
[5] Sun, J; Meyer, S. T.; Kozmin, S. A. *Angew. Chem. Int. Ed.* **2006**, *45*, 4991-4993.

allowed to stir for an additional 30 minutes. The reaction was quenched by addition of hexanes (200 mL). The crude mixture was then transferred to a separatory funnel and was washed with saturated aqueous $NaHCO_3$ (150 mL). The organic layer was collected, and the aqueous layer was extracted with hexanes (2 x 50 mL). The combined organic layers were washed with saturated aqueous $Na_2S_2O_3$ (125 mL) and brine (100 mL). The organic layer was collected, dried with $MgSO_4$, filtered, and concentrated by rotary evaporation. Volatile impurities were removed by Kugelrohr distillation, and the resulting mixture was purified via flash chromatography over basic alumina using hexanes as the eluent, affording siloxy alkyne **2i** as a clear oil (6.62 g, 16.6 mmol, 42 % yield).

^{1}H NMR (500 MHz, C_6D_6): δ 3.83 (t, J = 7.0 Hz, 2H), 2.50 (t, J = 7.0 Hz, 2H), 1.19-1.03 (m, 42H); ^{13}C NMR (125 MHz, C_6D_6): δ 88.4, 64.4, 28.3, 22.8, 18.6, 17.9, 12.7, 12.5; IR (film): 2945, 2868, 2282, 1464, 1249, 1108, 883 cm^{-1}; HRMS (ESI) Calcd for $(C_{22}H_{46}O_2Si_2)Na^+$ $(M+Na)^+$: 421.2929, Found: 421.2922.



11 **12**

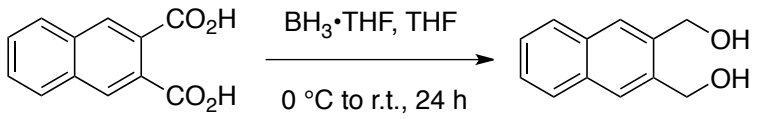
The previously known ethoxy alkyne **11**[6] and ynamide **12**[7] were prepared according to the reported procedures.

[6] Davies, P. W.; Cremonesi, A.; Dumitrescu, L. *Angew. Chem. Int. Ed.* **2011**, *50*, 8931-8935.

[7] Frederick, M. O.; Mulder, J. A.; Tracey, M. R.; Hsung, R. P.; Huang, J.; Kurtz, K. C. M.; Shen, L.; Douglas, C. J. *J. Am. Chem. Soc.* **2003**, *125*, 2368-2369.

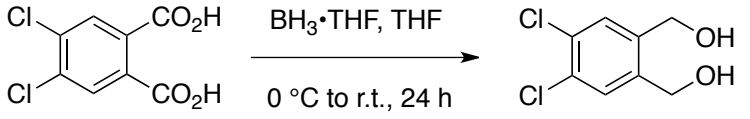
Preparation of 1,2-Diazines

General Procedure for the Reduction of Dicarboxylic Acids.



13 **14**

Naphthalene-2,3-diyldimethanol (**14**). The following procedure was adapted from the work of Weber and Wilcox.[8] A 250-mL flame-dried, three-necked, round-bottomed flask equipped with a stir bar, fitted with rubber septa and a nitrogen inlet was charged with naphthalene-2,3-dicarboxylic acid (**13**) (6.350 g, 29.4 mmol) and THF (40 mL). The resulting mixture was cooled to 0 °C and BH_3 • THF (76.4 mL of a 1 M solution, 76.4 mmol) was added dropwise via syringe pump at a rate of 0.3 mL/min. After addition of BH_3•THF was complete, the reaction mixture was warmed to room temperature and allowed to proceed for 24 h. The resulting mixture was cooled to 0 °C and quenched by dropwise addition of a 1:1 mixture of THF:H_2O (40 mL). Solid K_2CO_3 was added until the aqueous and organic layers separated. The resulting mixture was transferred to a separatory funnel; the organic layer was collected, and the aqueous layer was extracted with THF (2 x 50 mL). The organic layers were combined, dried with $MgSO_4$, filtered and concentrated via rotary evaporation. The residue was dried *in vacuo* to give 5.534 g of **14** (29.4 mmol, quantitative yield) as a white solid, which was used without further purification. Spectral data for **14** matches published reports.[8]



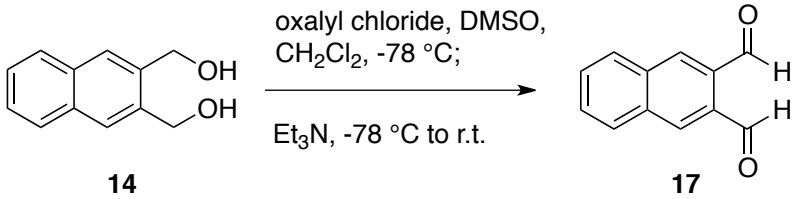
15 **16**

(4,5-Dichloro-1,2-phenylene)dimethanol (**16**). Following the general procedure for reduction of dicarboxylic acids, 9.725 g of **16** (46.97 mmol, quantitative yield) was obtained from the reduction of 4,5-dichlorophthalic acid (**15**) (11.040 g, 46.97 mmol)

[8] Wilcox, C. F. Jr.; Weber, K. A. *J. Org. Chem*. **1986**, *51*, 1088-1094.

with BH_3•THF (122.1 mL of a 1 M solution, 122.1 mmol). Spectral data for **16** matches published reports.[9]

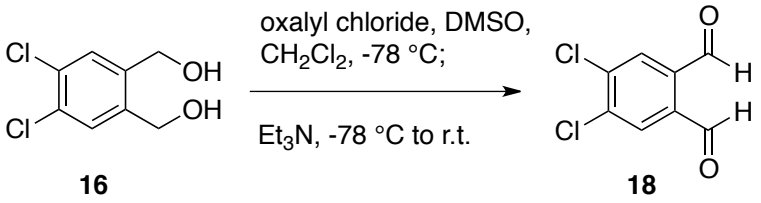
General Procedure for the Swern Oxidation



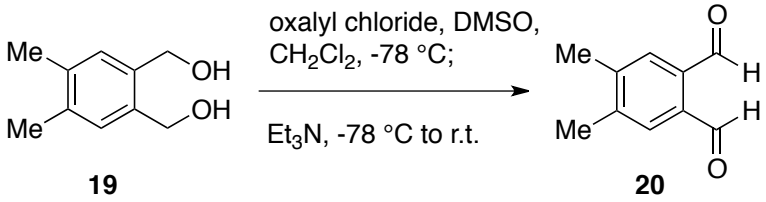
14 **17**

Naphthalene-2,3-dicarbaldehyde (**17**). The following procedure was adapted from the work of Farooq.[9] A 250-mL flame-dried, three-necked, round-bottomed flask equipped with a stir bar, fitted with rubber septa and a nitrogen inlet was charged with 100 ml of CH_2Cl_2 and oxalyl chloride (5.23 mL, 61.8 mmol), and the resulting solution was cooled to -78 °C. A solution of DMSO (8.80 mL, 123.7 mmol) in CH_2Cl_2 was added to the flask via syringe pump at a rate of 0.35 mL/min and the resulting mixture was stirred for 10 minutes after the addition of DMSO was complete. A solution of diol **14** (5.291g, 28.11 mmol) in CH_2Cl_2 was then added to the reaction mixture via syringe pump at a rate of 0.35 ml/min. The resulting mixture was stirred for 30 minutes and Et_3N (68.6 mL, 491.9 mmol) was added at a rate of 0.75 ml/min via a syringe pump. The mixture was stirred at -78 °C for 10 min, warmed to 0 °C and stirred for 2 hours. Following warming to room temperature, the reaction mixture was quenched with 200 mL of cold water and transferred to a separatory funnel. The organic layer was collected, and the aqueous layer was extracted with CH_2Cl_2 (2 x 100 mL). The combined organic layers were washed with sat. $NaHCO_3$ (1 x 100 mL), brine (1 x 200 mL), collected, dried with $MgSO_4$, filtered, and concentrated via rotary evaporation. The crude product was purified by flash chromatography (hexanes:ethyl acetate 9:1 to 4:1) to give 5.330 g of **17** (28.94 mmol, 81 % yield) as a white solid. Spectral data for **17** matches published reports.[10]

[9] Farooq, O. *Synthesis* **1994**, 1035-1036.
[10] Lin, C-H.; Lin, K-H.; Pal, B.; Tsou, L-D. *Chem*. *Commun*. **2009**, 803-805.

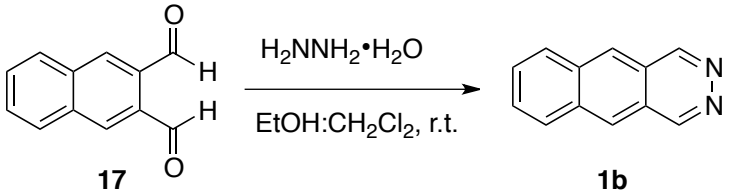


4,5-Dichlorophthalaldehyde (**18**). Following the general procedure for the Swern oxidation of diols, 2.253 g of **18** (11.10 mmol, 70 % yield) was obtained from (4,5-dichloro-1,2-phenylene)dimethanol (**16**) (3.282 g, 15.85 mmol) after purification by flash chromatography (hexanes: ethyl acetate 9:1 to 4:1). Spectral data for **18** matches published reports.[9]



4,5-Dimethylphthalaldehyde (**20**). Following the general procedure for the Swern oxidation of diols, 5.330 g of **20** (32.86 mmol, 92 % yield) was obtained from (4,5-dimethyl-1,2-phenylene)dimethanol (**19**) (5.951 g, 35.80 mmol) after purification by flash chromatography (hexanes to 9:1 hexanes:ethyl acetate). Spectral data for **20** matches published reports.[9]

General Procedure for the Synthesis of 1,2-Diazines

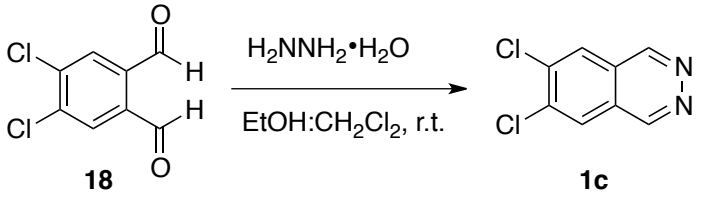


Benzo[g]phthalazine (**1b**). The following procedure was adapted from work of Sivasankaran and Zimmerman.[11] A solution of dialdehyde **17** (3.990g, 21.66 mmol) in 1:1 CH_2Cl_2:EtOH (40 mL) was added to a round-bottomed flask containing a solution of $H_2NNH_2 \bullet H_2O$ (3.16 mL, 64.99 mmol) in 33 mL of EtOH at 0 °C via syringe pump at a rate of 0.25 mL/min. The reaction was warmed to room temperature and was allowed to

[11] Sivasnkaran, R.; Zimmerman, K. International Patent WO2008/008821, **2008**.

proceed for 2 h. The reaction mixture was concentrated via rotary evaporation. Toluene (100 mL) was added to the crude material, the resulting mixture was stirred for 10 min at room temperature, and the solvent was removed by rotary evaporation. This process was repeated with CH_2Cl_2 (100 mL), and the crude residue was purified by flash chromatography (ethyl acetate to 9:1 ethyl acetate:isopropanol) to give 3.549 g of **1b** as a white solid (19.69 mmol, 91 % yield).

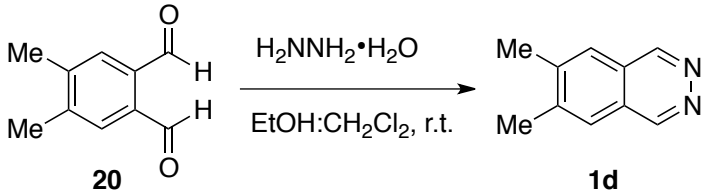
^{1}H NMR (500 MHz, $CDCl_3$): δ 9.62 (s, 2H), 8.54 (s, 2H), 8.17-8.16 (m, 2H), 7.72-7.70 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 151.7, 135.1, 129.0, 128.4, 126.5, 123.0; HRMS (ESI) Calcd for $C_{12}H_9N_2^+$ $(M+H)^+$: 181.0766, Found: 181.0769.



18 **1c**

6,7-Dichlorophthalazine (**1c**). Following the general procedure for the synthesis of 1,2-diazines, 1.637 g of **1c** (8.220 mmol, 67 % yield) was obtained as a white solid from 4,5-dichlorophthalaldehyde (**18**) (2.490 g, 12.267 mmol) and H_2NNH_2 • H_2O (1.79 mL, 36.80 mmol) after purification by flash chromatography (CH_2Cl_2 to 1:1 CH_2Cl_2: ethyl acetate).

^{1}H NMR (500 MHz, $CDCl_3$): δ 9.49 (s, 2H), 8.11 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 149.4, 137.8, 127.7, 125.2; HRMS (ESI) Calcd for $C_8H_5N_2Cl_2^+$ $(M+H)^+$: 198.9830, Found: 198.9825.

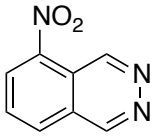


20 **1d**

6,7-Dimethylphthalazine (**1d**). Following the general procedure for synthesis of 1,2-diazines, 4.081 g of **1d** (25.80 mmol, 79 % yield) was obtained as a white solid from 4,5-dimethylphthalaldehyde (**20**) (5.323 g, 32.82 mmol) and H_2NNH_2 • H_2O (4.78 mL, 98.5

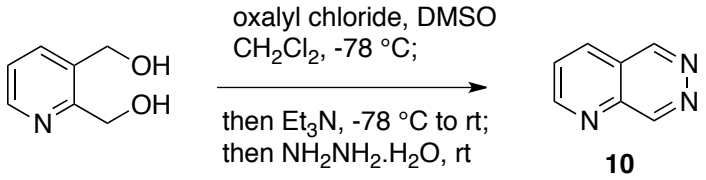
mmol) after purification by flash chromatography (ethyl acetate to 9:1 ethyl acetate:isopropanol).

^{1}H NMR (500 MHz, $CDCl_3$): δ 9.41 (s, 2H), 7.70 (s, 2H), 2.53 (s, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 150.4, 143.3, 125.7, 125.5, 20.7; HRMS (ESI) Calcd for $C_{10}H_{11}N_2^+$ $(M+H)^+$: 159.0922, Found: 159.0918.



1f

Diazine **1f** was prepared according to the reported procedure.[12]



10

Pyrido[2,3-d]pyridazine **10**[13] was prepared as follows:

A 250-mL, oven-dried, round-bottomed flask equipped with a magnetic stir bar was charged with oxalyl chloride (1.21 mL, 14.3 mmol) and 20 mL of CH_2Cl_2, and the resulting solution was cooled to -78 °C. A solution of DMSO (2.02 mL, 28.5 mmol) in CH_2Cl_2 (5 mL + 3 mL for rinsing the flask) was added to the oxalyl chloride solution dropwise over 20 min and the resulting mixture was stirred for 15 min. A solution of 2,3-bis(hydroxymethyl)pyridine[14] (901 mg, 6.5 mmol) in a mixture of 10 mL CH_2Cl_2 and 1.0 mL DMSO was then added to the reaction over 20 min. The flask of the diol was rinsed once more with a mixture of 5 mL CH_2Cl_2 and 0.5 mL DMSO. The resulting mixture was

[12] Gomtsyan, A.; Bayburt, E. K.; Schmidt, R. G.; Zheng, G. Z.; Perner, R. J.; Didomenico, S.; Koenig, J. R.; Turner, S.; Jinkerson, T.; Drizin, I.; Hannick, S. M.; Macri, B. S.; McDonald, H. A.; Honore, P.; Wismer, C. T.; Marsh, K. C.; Wetter, J.; Stewart, K. D.; Oie, T.; Jarvis, M. F.; Surowy, C. S.; Faltynek, C. R.; Lee, C. H. *J. Med. Chem.* **2005**, *48*, 744-752.

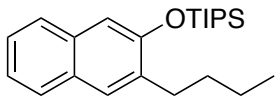
[13] Paul, D. B.; Rodda, H. J. *Aust. J. Chem.* **1968**, *21*, 1291-1310.

[14] Yoshiizumi, K.; Yamamoto, M.; Miyasaka, T.; Ito, Y.; Kumihara, H.; Sawa, M.; Kiyoi, T.; Yamamoto, T.; Nakajima, F.; Hirayama, R.; Kondo, H.; Ishibushi, E.; Ohmoto, H.; Inoue, Y.; Yoshino, K. *Bioorg. Med. Chem.* **2003**, *11*, 433-450.

stirred at -78 °C for 30 min and Et_3N (15.8 mL, 113.3 mmol) was added over 15 min. After stirring at -78 °C for an additional 5 min, the dry ice-acetone bath was replaced with ice-water bath and the reaction was allowed to proceed for 1 h at 0 °C and for 1 h at room temperature. $NH_2NH_2•H_2O$ (0.94 mL, 19.4 mmol) was added and the resulting mixture was stirred at room temperature for 12 h. It was then diluted with CH_2Cl_2, filtered through a plug of silica and washed several times with several EtOAc. The filtrate was concentrated *in vacuo* and the residue was purified by flash column chromatography (EtOAc only) to afford pure pyrido[2,3-d]pyridazine **10** (534 mg, 63%) as a yellow solid.

mp: 149-150 °C (lit.[13] mp: 154-155 °C); ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.78 (s, 1H), 9.58 (d, *J* = 1.3 Hz, 1H), 9.26 (dd, *J* = 4.3, 1.5 Hz, 1H), 8.31 (dt, *J* = 8.3, 0.7 Hz, 1H), 7.84 (dd, *J* = 8.2, 4.3 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 156.6, 153.0, 150.7, 141.9, 134.1, 127.4, 122.2.

Representative Procedure for the Cycloaddition Reaction of 1,2-Diazines and Siloxy Alkynes:



3a

An oven-dried test tube was charged with phthalazine **1a** (130 mg, 1.0 mmol), evacuated under vacuum and refilled with nitrogen three times. Another oven-dried test tube was charged with 2,2′-bipyridine **8** (8.6 mg, 0.055 mmol) and $AgNTf_2$ (19.4 mg, 0.05 mmol). It was evacuated and refilled with nitrogen three times and then 2.5 mL of anhydrous CH_2Cl_2 was added. The resulting clear, colorless solution was stirred under nitrogen for 10 min and 0.5 mL of this solution was added via syringe to the test tube containing phthalazine. An additional 0.5 mL of CH_2Cl_2 was added and the resulting clear, light yellow solution was stirred for 15 min. Siloxy alkyne **2a** (331 mg, 1.3 mmol) was weighed in a 1.0 mL syringe and added to the reaction mixture slowly as a neat liquid. The color turned yellow first, then brownish green as the reaction proceeded. The reaction was monitored by TLC and at the end of 2 h, the reaction mixture was filtered through a plug of silica gel with several CH_2Cl_2 washings. This operation removed

$AgNTf_2$ and ligand **8** and gave a mixture of the unreacted siloxy alkyne **2a** and the product. Unreacted siloxy alkyne was removed by heating in a Kugelrohr distillation apparatus under reduced pressure (125 °C; 3 mmHg) for 1 h. The resulting mixture was then purified by flash column chromatography (hexanes only) to give pure siloxy naphthalene **3a** (291 mg, 82%) as a colorless oil.

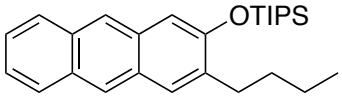
(3-butylnaphthalen-2-yloxy)triisopropylsilane 3a: colorless oil; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.69 (d, *J* = 7.9 Hz, 1H), 7.62 (d, *J* = 8.1 Hz, 1H), 7.56 (s, 1H), 7.34 (dt, *J* = 8.1, 1.2 Hz, 1H), 7.28 (dt, *J* = 7.5, 1.2 Hz, 1H), 7.09 (s, 1H), 2.77 (t, *J* = 7.9 Hz, 2H), 1.68-1.63 (m, 2H), 1.44-1.37 (m, 5H), 1.15 (d, *J* = 7.5 Hz, 18H), 0.95 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 153.0, 135.0, 133.2, 129.0, 128.2, 127.0, 126.0, 125.1, 123.4, 112.4, 32.2, 31.1, 22.8, 18.1, 14.1, 13.1; IR (film): 2945, 2867, 1631, 1599, 1499, 1466, 1258, 929, 883 cm^{-1}; HRMS (ESI) Calcd for $(C_{23}H_{36}OSi)Na^+$ $(M+Na)^+$: 379.2428, Found: 379.2430.

Alternatively, an acidic work-up can be applied to remove the unreacted siloxy alkyne in place of the Kugelrohr distillation:

The reaction was carried out according to the Representative Procedure using phthalazine (65 mg, 0.5 mmol), $AgNTf_2$ (0.005 mmol, 1 mol%), 2,2′-bipyridine **8** (0.0055 mmol, 1.1 mol%) and siloxy alkyne **2a** (165 mg, 0.65 mmol). At the end of 2 h, the reaction mixture was diluted with 0.5 mL CH_2Cl_2 and 0.5 mL MeOH. 25 μL TFA (trifluoroacetic acid) was added and the resulting solution was stirred under air for 15 min. It was then diluted with H_2O, and the aqueous phase was extracted with CH_2Cl_2 (3x10 mL). The combined organic phase was dried over $MgSO_4$, filtered and concentrated *in vacuo*. Purification by flash colum chromatography (hexanes only) gave pure siloxy naphthalene product **3a** (145 mg, 81%) as a colorless oil.

Cycloaddition of Phthalazine 1a with Siloxy Alkyne 2a on a 20 mmol Scale:

A 100-mL, oven-dried, round-bottomed flask equipped with a magnetic stir bar was charged with phthalazine **1a** (2.60 g, 20.0 mmol), 2,2′-bipyridine **8** (34.4 mg, 0.22 mmol) and $AgNTf_2$ (77.6 mg, 0.20 mmol). The flask was evacuated and refilled with nitrogen three times. Anhydrous CH_2Cl_2 (20 mL) was added and the resulting clear, light yellow solution was stirred for 15 min under nitrogen. Siloxy alkyne **2a** (6.62 g, 26.0 mmol) was weighed in a 10-mL syringe and added to the reaction mixture slowly as a neat liquid. After a few minutes, vigorous gas evolution (N_2) started and the color turned first yellow, then brownish green. The reaction was exothermic and the flask was inserted into a water bath to maintain room temperature. At the end of 3 h, the reaction mixture was filtered through silica gel (5.5 g), washed with additional CH_2Cl_2 (100 mL) and concentrated *in vacuo* to afford a yellow-orange oil. The unreacted siloxy alkyne **2a** was removed by heating in a Kugelrohr distillation apparatus under vacuum (2 mmHg; 1.5 h at 130 °C, then 4 h at 140 °C). Flash column chromatography of the resulting mixture (eluted with hexanes only) gave pure siloxy naphthalene product **3a** (5.26 g, 74%) as a very light yellow oil.

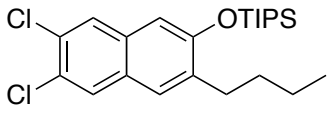


3b

(3-butylanthracen-2-yloxy)triisopropylsilane 3b: Siloxy anthracene **3b** was prepared according to the Representative Procedure using benzo[g]phthalazine **1b** (90 mg, 0.5 mmol), $AgNTf_2$ (0.01 mmol, 2 mol%), 2,2′-bipyridine **8** (0.011 mmol, 2.2 mol%), siloxy alkyne **2a** (165 mg, 0.65 mmol) and CH_2Cl_2 (2.0 mL). [The catalyst stock solution was prepared by dissolving 2,2′-bipyridine **8** (8.6 mg, 0.055 mmol) and $AgNTf_2$ (19.4 mg, 0.05 mmol) in 5.0 mL CH_2Cl_2 and 1.0 mL of this solution was added onto benzo[g]phthalazine **1b** followed by the addition of 1.0 mL more CH_2Cl_2.] At the end of 1.5 h, 1.0 mL MeOH and 25 μL TFA were added to the reaction mixture and the resulting solution was stirred for 10 min. It was then diluted with H_2O and the aqueous phase was extracted three times with CH_2Cl_2. The combined organic phase was dried over $MgSO_4$, filtered and concentrated *in vacuo*. Purification by flash column

chromatography (hexanes only) gave pure siloxy anthracene **3b** (147 mg, 72%) as a colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 8.26 (s, 1H), 8.17 (s, 1H), 7.93 (d, *J* = 8.0 Hz, 1H), 7.90 (d, *J* = 8.2 Hz, 1H), 7.72 (s, 1H), 7.40-7.34 (m, 2H), 7.21 (s, 1H), 2.82 (t, *J* = 7.8 Hz, 2H), 1.74-1.68 (m, 2H), 1.49-1.40 (m, 5H), 1.18 (d, *J* = 7.5 Hz, 18H), 0.97 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 153.0, 136.2, 132.0, 131.5, 130.4, 128.5, 128.1, 127.8, 127.5, 125.0, 124.9, 124.1, 123.3, 110.7, 32.1, 31.4, 22.8, 18.2, 14.1, 13.1; IR (film): 2945, 2866, 1634, 1459, 1279, 1214, 1147, 901, 882 cm^{-1}; HRMS (ESI) Calcd for $(C_{27}H_{38}OSi)Na^+$ $(M+Na)^+$: 429.2584, Found: 429.2581.

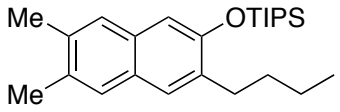


3c

(3-butyl-6,7-dichloronaphthalen-2-yloxy)triisopropylsilane 3c: Siloxy naphthalene **3c** was prepared according to the Representative Procedure using phthalazine **1c** (100 mg, 0.5 mmol), $AgNTf_2$ (0.005 mmol, 1 mol%), 2,2′-bipyridine **8** (0.0055 mmol, 1.1 mol%), siloxy alkyne **2a** (165 mg, 0.65 mmol) and CH_2Cl_2 (5.0 mL). [The catalyst stock solution was prepared by dissolving 2,2′-bipyridine **8** (8.6 mg, 0.055 mmol) and $AgNTf_2$ (19.4 mg, 0.05 mmol) in 5.0 mL CH_2Cl_2 and 0.5 mL of this solution was added onto a solution of phthalazine **1c** in 4.5 mL CH_2Cl_2.] At the end of 7 h, 2.0 mL MeOH and 25 μL TFA were added to the reaction mixture and the resulting solution was stirred for 5 min. It was then diluted with H_2O and the aqueous phase was extracted three times with CH_2Cl_2. The combined organic phase was dried over $MgSO_4$, filtered and concentrated *in vacuo*. Purification by flash column chromatography (hexanes only) gave pure siloxy naphthalene **3c** (167 mg, 78%) as a colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.77 (s, 1H), 7.73 (s, 1H), 7.45 (s, 1H), 6.98 (s, 1H), 2.75 (t, *J* = 7.8 Hz, 2H), 1.67-1.61 (m, 2H), 1.45-1.34 (m, 5H), 1.14 (d, *J* = 7.5 Hz, 18H), 0.95 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 154.0, 136.6, 132.3, 129.3, 128.0,

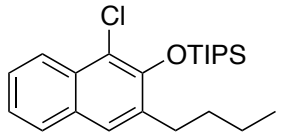
127.9, 127.2, 126.9, 111.3, 32.0, 31.1, 22.7, 18.1, 14.0, 13.1; ^{13}C NMR (125 MHz, CD_2Cl_2): δ 154.8, 137.4, 132.9, 129.5, 128.6, 128.4, 127.7, 127.5, 127.4, 111.9, 32.6, 31.6, 23.3, 18.4, 14.4, 13.6; IR (film): 2946, 2867, 1629, 1584, 1488, 1385, 1359, 1252, 933, 883, 827 cm^{-1}; HRMS (ESI) Calcd for $(C_{23}H_{34}Cl_2OSi)Na^+$ $(M+Na)^+$: 447.1648, Found: 447.1648.



3d

(3-butyl-6,7-dimethylnaphthalen-2-yloxy)triisopropylsilane 3d: Siloxy naphthalene **3d** was prepared according to the Representative Procedure using phthalazine **1d** (158 mg, 1.0 mmol), $AgNTf_2$ (0.02 mmol, 2 mol%), 2,2′-bipyridine **8** (0.022 mmol, 2.2 mol%), siloxy alkyne **2a** (331 mg, 1.3 mmol) and CH_2Cl_2 (4.0 mL). [The catalyst stock solution was prepared by dissolving 2,2′-bipyridine **8** (8.6 mg, 0.055 mmol) and $AgNTf_2$ (19.4 mg, 0.05 mmol) in 5.0 mL CH_2Cl_2 and 2.0 mL of this solution was added onto a solution of phthalazine **1d** in 2.0 mL CH_2Cl_2.] At the end of 3 h, the reaction mixture was filtered through a plug of silica gel, washed with additional CH_2Cl_2 and concentrated *in vacuo*. The resulting mixture was then heated in a Kugelrohr distillation apparatus under reduced pressure (120 °C, 3 mmHg) for 1.5 h. Purification by flash column chromatography (hexanes only) gave pure siloxy naphthalene **3d** (325 mg, 84%) as a colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.44 (s, 2H), 7.39 (s, 1H), 6.99 (s, 1H), 2.74 (t, J = 7.8 Hz, 2H), 2.37 (s, 3H), 2.36 (s, 3H), 1.67-1.61 (m, 2H), 1.44-1.34 (m, 5H), 1.14 (d, J = 7.5 Hz, 18H), 0.94 (t, J = 7.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 152.4, 134.6, 133.9, 132.7, 132.0, 127.9, 127.2, 126.5, 125.7, 111.6, 32.3, 31.1, 22.7, 20.1, 20.0, 18.1, 14.1, 13.1; IR (film): 2944, 2867, 1640, 1610, 1499, 1466, 1388, 1372, 1253, 1208, 1147, 905, 882 cm^{-1}; HRMS (ESI) Calcd for $(C_{25}H_{40}OSi)Na^+$ $(M+Na)^+$: 407.2741, Found: 407.2734.



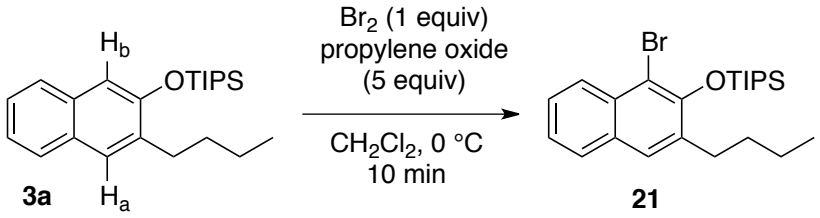
3e

(3-butyl-1-chloronaphthalen-2-yloxy)triisopropylsilane 3e: Siloxy naphthalene **3e** was prepared according to the Representative Procedure using 1-chlorophthalazine (82 mg, 0.5 mmol), $AgNTf_2$ (0.01 mmol, 2 mol%), 2,2′-bipyridine **8** (0.011 mmol, 2.2 mol%), siloxy alkyne **2a** (165 mg, 0.65 mmol) and CH_2Cl_2 (1.0 mL). [The catalyst stock solution was prepared by dissolving 2,2′-bipyridine **8** (8.6 mg, 0.055 mmol) and $AgNTf_2$ (19.4 mg, 0.05 mmol) in 5.0 mL CH_2Cl_2 and 1.0 mL of this solution was added onto 1-chlorophthalazine.] At the end of 4 h, 1.0 mL MeOH and 2 drops of TFA were added to the reaction mixture. The resulting solution was stirred for 3 min, filtered first through basic alumina and then silica gel, washed with additional CH_2Cl_2 and concentrated *in vacuo*. Purification by flash column chromatography (hexanes only) gave pure siloxy naphthalene **3e** (130 mg, 67%) as a colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 8.12 (d, J = 8.5 Hz, 1H), 7.70 (d, J = 8.1 Hz, 1H), 7.51 (s, 1H), 7.47 (t, J = 7.7 Hz, 1H), 7.36 (t, J = 7.5 Hz, 1H), 2.79 (t, J = 7.9 Hz, 2H), 1.67 (app quin, J = 7.8 Hz, 2H), 1.53-1.40 (m, 5H), 1.14 (d, J = 7.6 Hz, 18H), 0.96 (t, J = 7.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 149.8, 135.5, 130.6, 129.6, 127.2, 126.5, 126.1, 124.3, 123.6, 118.8, 32.1, 31.4, 22.7, 18.1, 14.5, 14.0; IR (film): 2946, 2867, 1596, 1441, 1362, 1256, 1151, 1115, 964, 883 cm^{-1}; HRMS (ESI) Calcd for $(C_{23}H_{35}ClOSi)H^+$ $(M+H)^+$: 391.2218, Found: 391.2217.

Discussion on the Regiochemistry of the Siloxy Naphthalene 3e: In order to determine the regiochemistry of **3e**, the corresponding brominated analog **21** was prepared by the electrophilic bromination of **3a** according to the procedure shown below. In the ^{1}H-NMR of **3a**, the two singlets at 7.56 and 7.09 ppm were assigned as the signals of H_a and H_b, respectively, based on the regular electronic considerations. The latter signal (7.09 ppm) disappeared upon bromination of **3a** supporting that the electrophilic substitution took place at the 1- position. This is in accord with the known reactivity of 2-naphthols in

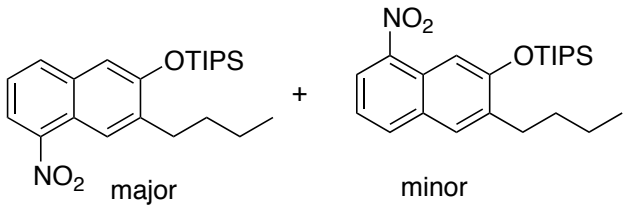
electrophilic aromatic substitution reactions. The ^{1}H- and ^{13}C-NMR spectra of **3e** and **21** exhibit close similarities in terms of the signal pattern and chemical shifts.



(1-bromo-3-butylnaphthalen-2-yloxy)triisopropylsilane 21: The following procedure was adapted from the work of Corey and co-workers.[15] To a solution of the siloxy naphthalene **3a** (40 mg, 0.11 mmol) in 0.5 mL of anhydrous CH_2Cl_2 was added propylene oxide (39 μL, 0.56 mmol). The resulting clear solution was cooled to 0 °C and 0.5 M Br_2 in CCl_4 (225 μL, 0.11 mmol) was added dropwise. The resulting light yellow solution was stirred for 10 min at the same temperature and then filtered through a pad of silica gel with several washings with hexanes. Removal of all volatiles *in vacuo* afforded the product (46 mg, 94%) as a colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 8.14 (d, J = 8.6 Hz, 1H), 7.69 (d, J = 8.1 Hz, 1H), 7.55 (s, 1H), 7.47 (dt, J = 7.6, 1.0 Hz, 1H), 7.36 (t, J = 7.5 Hz, 1H), 2.81 (t, J = 7.9 Hz, 2H), 1.71-1.64 (m, 2H), 1.54 (sept, J = 7.7 Hz, 3H), 1.43 (sext, J = 7.5 Hz, 2H), 1.15 (d, J = 7.6 Hz, 18H), 0.96 (t, J = 7.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 151.3, 135.5, 132.0, 130.0, 127.4, 127.3, 126.5, 126.4, 124.3, 112.0, 32.1, 31.6, 22.7, 18.1, 14.6, 14.1; IR (film): 2946, 2867, 1558, 1450, 1360, 1251, 1151, 1018, 954, 883 cm^{-1}; HRMS (ESI) Calcd for $(C_{23}H_{35}BrOSi)Na^{+}$ $(M+Na)^{+}$: 457.1533, Found: 457.1526.

[15] Liang, H.; Hu, L.; Corey, E. J. *Org. Lett.* **2011**, *13*, 4120-4123.



3f
1.9:1 regioisomer ratio

Prepared according to the Representative Procedure using 5-nitrophthalazine **1f** (88 mg, 0.5 mmol), $AgNTf_2$ (0.01 mmol, 2 mol%), 2,2′-bipyridine **8** (0.011 mmol, 2.2 mol%), siloxy alkyne **2a** (165 mg, 0.65 mmol) and CH_2Cl_2 (2.0 mL). [The catalyst stock solution was prepared by dissolving 2,2′-bipyridine **8** (8.6 mg, 0.055 mmol) and $AgNTf_2$ (19.4 mg, 0.05 mmol) in 5.0 mL CH_2Cl_2 and 1.0 mL of this solution was added onto a mixture of 5-nitrophthalazine **1f** and 1.0 mL CH_2Cl_2.] At the end of 4 h, the reaction mixture was filtered through a plug of silica gel, washed with additional CH_2Cl_2 and concentrated *in vacuo*. It was then heated in a Kugelrohr distillation apparatus under reduced pressure (3 mmHg; 120 °C) for 2 h. Purification by flash column chromatography (hexanes to 0.5% EtOAc in hexanes) gave siloxy naphthalene product **3f** (149 mg, 74%) as a 1.9:1 regioisomeric mixture (yellow oil).

^{13}C NMR (125 MHz, $CDCl_3$): δ 156.7, 154.0, 146.2, 144.3, 139.3, 136.6, 134.8, 134.3, 132.6, 130.4, 129.1, 125.6, 124.4, 123.7, 123.4, 121.6, 121.4, 120.6, 112.9, 108.3, 32.1, 31.8, 31.7, 30.8, 22.8, 22.7, 18.08, 18.06, 14.0, 13.0, 12.9; IR (film): 2947, 2868, 1631, 1604, 1522, 1462, 1326, 1271, 1135, 997, 882, 804 cm^{-1}; HRMS (ESI) Calcd for $(C_{23}H_{35}NO_3Si)Na^+$ $(M+Na)^+$: 424.2278, Found: 424.2277.

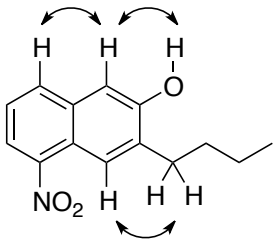
Major regioisomer:

^{1}H NMR (500 MHz, $CDCl_3$): δ 8.33 (s, 1H), 8.04 (dd, *J* = 7.6, 0.9 Hz, 1H), 7.88 (d, *J* = 8.2 Hz, 1H), 7.39 (t, *J* = 7.9 Hz, 1H), 7.18 (s, 1H), 2.83 (t, *J* = 7.8 Hz, 2H), 1.69-1.65 (m, 2H), 1.48-1.38 (m, 5H), 1.16 (d, *J* = 7.5 Hz, 18H), 0.96 (t, *J* = 7.4 Hz, 3H).

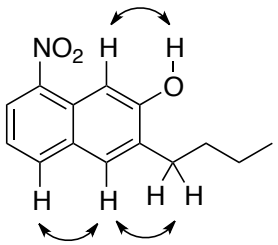
Minor regioisomer:

^{1}H NMR (500 MHz, $CDCl_3$): δ 8.26 (dd, *J* = 7.7, 1.0 Hz, 1H), 8.13 (s, 1H), 7.98 (d, *J* = 8.1 Hz, 1H), 7.67 (s, 1H), 7.33 (t, *J* = 7.9 Hz, 1H), 2.80 (t, *J* = 7.9 Hz, 2H), 1.69-1.65 (m, 2H), 1.48-1.38 (m, 5H), 1.17 (d, *J* = 7.5 Hz, 18H), 0.96 (t, *J* = 7.4 Hz, 3H).

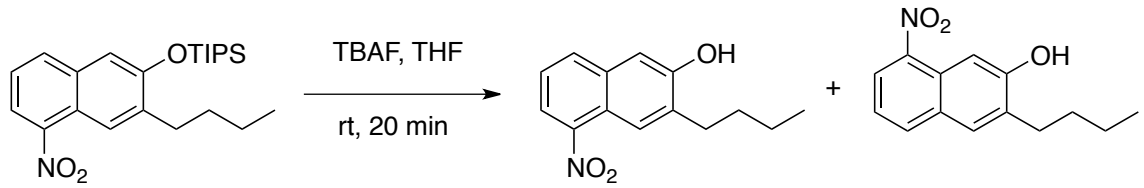
Discussion on the Regiochemistry of the Major and Minor Isomers of 3f: The regioisomeric mixture **3f** was desilylated according to the procedure shown below and the resulting 2-naphthol products **22** (major) and **23** (minor) were separated by flash column chromatography. The isolated yields for these products (62% and 33%) reflected well the regioisomer ratio (1.9:1) of **3f**. NOESY spectra of both compounds were recorded and the key NOEs shown below by the curved arrows support the structural assignments of these regioisomers.[16]



22
(major)



23
(minor)



3f
1.9:1 regiosomers

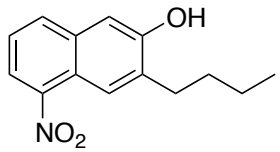
22 (major)
62%

23 (minor)
33%

To a solution of the regioisomeric mixture **3f** (119 mg, 0.3 mmol) in 5 mL of anhydrous THF was added TBAF (0.44 mL, 0.44 mmol, 1.0 M in THF) at room temperature, under nitrogen. The bright yellow solution immediately turned dark blue upon addition of TBAF. The reaction mixture was stirred for 20 min at which time TLC analysis indicated full consumption of the starting material. It was then quenched with saturated NH_4Cl solution (10 mL) and the color turned back to bright yellow. The mixture was diluted with H_2O (5 mL) and the aqueous phase was extracted with EtOAc (3x15 mL). The combined organic phase was dried over $MgSO_4$, filtered and concentrated *in vacuo* to afford a yellow oil. Purification by flash column chromatography (hexanes to 2% to 5%

[16] For the NOESY spectra of **22** and **23**, see pages S-59 and S-62, respectively.

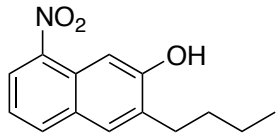
to 10% to 20% EtOAc in hexanes) gave pure 2-naphthol derivatives **22** (45 mg, 62%, major regioisomer) and **23** (24 mg, 33%, minor regioisomer) as yellow solids.



22

3-butyl-5-nitronaphthalen-2-ol 22:

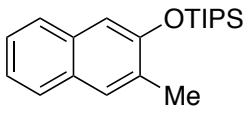
mp 108-109 °C; R_f = 0.12 (10% EtOAc in hexanes); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.34 (s, 1H), 8.05 (dd, *J* = 7.6, 1.1 Hz, 1H), 7.88 (d, *J* = 8.2 Hz, 1H), 7.40 (t, *J* = 7.9 Hz, 1H), 7.19 (s, 1H), 5.41 (s, 1H), 2.83 (t, *J* = 7.7 Hz, 2H), 1.73-1.67 (m, 2H), 1.44 (sext, *J* = 7.5 Hz, 2H), 0.97 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 153.5, 146.2, 135.3, 134.7, 132.5, 124.0, 123.8, 121.7, 120.7, 110.0, 31.7, 30.7, 22.6, 13.9; IR (film): 3388 (br s), 2951, 2861, 1630, 1606, 1516, 1462, 1307, 1186, 1123, 981 cm^{-1}; HRMS (ESI) Calcd for $(C_{14}H_{14}NO_3)^-$ $(M-H)^-$: 244.0979, Found: 244.0984.



23

3-butyl-8-nitronaphthalen-2-ol 23:

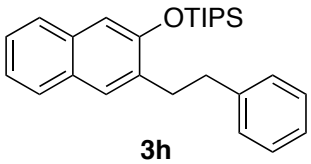
mp 129-130 °C; R_f = 0.41 (10% EtOAc in hexanes); ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.28 (dd, *J* = 7.8, 1.2 Hz, 1H), 8.13 (s, 1H), 8.01 (d, *J* = 8.1 Hz, 1H), 7.69 (s, 1H), 7.35 (t, *J* = 7.9 Hz, 1H), 6.03 (s, 1H), 2.82 (t, *J* = 7.7 Hz, 2H), 1.74-1.68 (m, 2H), 1.44 (sext, *J* = 7.6 Hz, 2H), 0.98 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 156.4, 144.1, 134.7, 133.3, 130.5, 129.5, 125.6, 124.7, 121.4, 105.4, 31.4, 30.0, 22.6, 14.0; IR (film): 3411 (br s), 2924, 2866, 1634, 1503, 1464, 1444, 1323, 1214 cm^{-1}; HRMS (ESI) Calcd for $(C_{14}H_{14}NO_3)^-$ $(M-H)^-$: 244.0979, Found: 244.0988.



3g

Triisopropyl(3-methylnaphthalen-2-yloxy)silane 3g: Siloxy naphthalene **3g** was prepared according to the Representative Procedure using phthalazine **1a** (65 mg, 0.5 mmol), $AgNTf_2$ (0.01 mmol, 2 mol%), 2,2′-bipyridine **8** (0.011 mmol, 2.2 mol%), siloxy alkyne **2g** (212 mg, 1.0 mmol) and CH_2Cl_2 (1.5 mL). [The catalyst stock solution was prepared by dissolving 2,2′-bipyridine **8** (8.6 mg, 0.055 mmol) and $AgNTf_2$ (19.4 mg, 0.05 mmol) in 5.0 mL CH_2Cl_2 and 0.5 mL of this solution was added onto a solution of phthalazine **1a** in 0.5 mL CH_2Cl_2. After 1.5 h, 0.5 mL more catalyst stock solution was added resulting in a total of 2 mol% catalyst loading.] At the end of 3 h, the reaction mixture was filtered through a plug of silica gel, washed with additional CH_2Cl_2 and concentrated *in vacuo*. The resulting mixture was then heated in a Kugelrohr distillation apparatus under reduced pressure (75 °C; 1 mmHg) for 2 h. Purification by flash column chromatography (hexanes only) gave pure siloxy naphthalene **3g** (115 mg, 73%) as a colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.67 (d, J = 8.1 Hz, 1H), 7.62 (d, J = 8.2 Hz, 1H), 7.58 (s, 1H), 7.34 (t, J = 7.5 Hz, 1H), 7.28 (dt, J = 7.4, 0.9 Hz, 1H), 7.10 (s, 1H), 2.40 (s, 3H), 1.39 (sept, J = 7.5 Hz, 3H), 1.15 (d, J = 7.5 Hz, 18H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 153.3, 133.3, 130.6, 129.1, 128.9, 126.8, 126.1, 125.1, 123.4, 112.4, 18.1, 17.8, 13.0; IR (film): 2945, 2867, 1631, 1601, 1501, 1467, 1333, 1260, 1180, 1161, 1109, 929, 881 cm^{-1}; HRMS (ESI) Calcd for $(C_{20}H_{30}OSi)H^+$ $(M+H)^+$: 315.2139, Found: 315.2143.

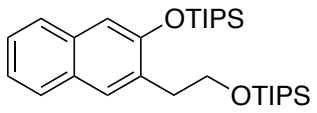


3h

Triisopropyl(3-phenethylnaphthalen-2-yloxy)silane 3h: Siloxy naphthalene **3h** was prepared according to the Representative Procedure using phthalazine **1a** (65 mg, 0.5 mmol), $AgNTf_2$ (0.01 mmol, 2 mol%), 2,2′-bipyridine **8** (0.011 mmol, 2.2 mol%), siloxy

alkyne **2h** (227 mg, 0.75 mmol) and CH_2Cl_2 (1.0 mL). [The catalyst stock solution was prepared by dissolving 2,2′-bipyridine **8** (8.6 mg, 0.055 mmol) and $AgNTf_2$ (19.4 mg, 0.05 mmol) in 5.0 mL CH_2Cl_2 and 1.0 mL of this solution was added onto phthalazine **1a**.] At the end of 1 h, the reaction mixture was filtered through a plug of silica gel, washed with additional CH_2Cl_2 and concentrated *in vacuo*. The sulting mixture was then heated in a Kugelrohr distillation apparatus under reduced pressure (1 mmHg; at 150 °C for 45 min, then 200 °C for 1.5 h). Purification by flash column chromatography (hexanes only) gave pure siloxy naphthalene **3h** (141 mg, 70%) as a colorless oil.

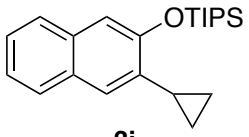
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.67 (d, J = 8.1 Hz, 1H), 7.64 (d, J = 8.2 Hz, 1H), 7.53 (s, 1H), 7.36 (dt, J = 7.5, 1.2 Hz, 1H), 7.28 (app t, J = 7.7 Hz, 3H), 7.24-7.18 (m, 3H), 7.14 (s, 1 H), 3.12-3.08 (m, 2H), 3.01-2.97 (m, 2H), 1.43 (sept, J = 7.5 Hz, 3H), 1.17 (d, J = 7.5 Hz, 18H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 152.9, 142.2, 133.8, 133.4, 129.0, 128.7, 128.5, 128.3, 127.1, 126.1, 125.8, 125.3, 123.5, 112.5, 36.5, 33.3, 18.2, 13.2; IR (film): 2945, 2866, 1631, 1600, 1499, 1466, 1388, 1363, 1335, 1258, 1180, 1104, 929, 882 cm^{-1}; HRMS (ESI) Calcd for $(C_{27}H_{36}OSi)Na^+$ $(M+Na)^+$: 427.2428, Found: 427.2434.



3i

Triisopropyl(3-(2-(triisopropylsilyloxy)ethyl)naphthalen-2-yloxy)silane 3i: Siloxy naphthalene **3i** was prepared according to the Representative Procedure using phthalazine **1a** (65 mg, 0.5 mmol), $AgNTf_2$ (0.005 mmol, 1 mol%), 2,2′-bipyridine **8** (0.0055 mmol, 1.1 mol%), siloxy alkyne **2i** (259 mg, 0.65 mmol) and CH_2Cl_2 (0.5 mL). [The catalyst stock solution was prepared by dissolving 2,2′-bipyridine **8** (8.6 mg, 0.055 mmol) and $AgNTf_2$ (19.4 mg, 0.05 mmol) in 5.0 mL CH_2Cl_2 and 0.5 mL of this solution was added onto phthalazine **1a**.] At the end of 1 h, the reaction mixture was filtered through a plug of silica gel, washed with additional CH_2Cl_2 and concentrated *in vacuo*. Purification by flash column chromatography (hexanes to 2% EtOAc in hexanes) gave pure siloxy naphthalene **3i** (204 mg, 81%) as a colorless oil.

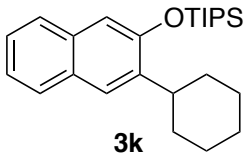
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.69 (d, *J* = 8.1 Hz, 1H), 7.65 (s, 1H), 7.62 (d, *J* = 8.1 Hz, 1H), 7.35 (dt, *J* = 7.5, 1.1 Hz, 1H), 7.28 (dt, *J* = 7.5, 1.0 Hz, 1H), 7.10 (s, 1H), 3.96 (t, *J* = 7.1 Hz, 2H), 3.05 (t, *J* = 7.1 Hz, 2H), 1.40 (sept, *J* = 7.5 Hz, 3H), 1.15 (d, *J* = 7.5 Hz, 18H), 1.08-1.01 (m, 21H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 152.9, 133.4, 131.1, 129.7, 129.0, 127.1, 126.0, 125.3, 123.4, 112.4, 63.3, 35.0, 18.1, 18.0, 13.1, 12.0; IR (film): 2944, 2866, 1633, 1600, 1500, 1466, 1387, 1258, 1180, 1158, 1110, 929, 883 cm^{-1}; HRMS (ESI) Calcd for $(C_{30}H_{52}O_2Si_2)Na^+$ $(M+Na)^+$: 523.3398, Found: 523.3379.



3j

(3-cyclopropylnaphthalen-2-yloxy)triisopropylsilane 3j: Siloxy naphthalene **3j** was prepared according to the Representative Procedure using phthalazine **1a** (65 mg, 0.5 mmol), $AgNTf_2$ (0.01 mmol, 2 mol%), 2,2′-bipyridine **8** (0.011 mmol, 2.2 mol%), siloxy alkyne **2j** (238 mg, 1.0 mmol) and CH_2Cl_2 (1.0 mL). [The catalyst stock solution was prepared by dissolving 2,2′-bipyridine **8** (8.6 mg, 0.055 mmol) and $AgNTf_2$ (19.4 mg, 0.05 mmol) in 5.0 mL CH_2Cl_2 and 1.0 mL of this solution was added onto phthalazine **1a**.] At the end of 3 h, the reaction mixture was filtered through a plug of silica gel, washed with additional CH_2Cl_2 and concentrated *in vacuo*. The resulting mixture was then heated in a Kugelrohr distillation apparatus under reduced pressure (1 mmHg; at 75 °C for 30 min, then 115 °C for 1 h). Purification by flash column chromatography (hexanes only) gave pure siloxy naphthalene **3j** (128 mg, 75%) as a colorless oil.

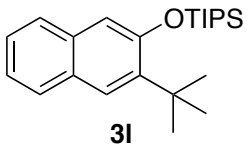
^{1}H NMR (500 MHz, CD_2Cl_2): δ 7.66 (d, *J* = 8.0 Hz, 1H), 7.62 (d, *J* = 8.0 Hz, 1H), 7.33 (dt, *J* = 7.4, 1.2 Hz, 1H), 7.29 (s, 1H), 7.26 (dt, *J* = 7.5, 1.1 Hz, 1H), 7.13 (s, 1 H), 2.34-2.29 (m, 1H), 1.43 (sept, *J* = 7.5 Hz, 3H), 1.17 (d, *J* = 7.5 Hz, 18H), 1.01-0.97 (m, 2H), 0.77-0.73 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 153.8, 135.9, 132.9, 129.0, 127.0, 126.0, 125.1, 123.6, 123.4, 112.4, 18.1, 13.1, 10.9, 8.0; IR (film): 2945, 2867, 1632, 1600, 1498, 1472, 1449, 1334, 1261, 1179, 1051, 930, 883 cm^{-1}; HRMS (ESI) Calcd for $(C_{22}H_{32}OSi)Na^+$ $(M+Na)^+$: 363.2115, Found: 363.2119.



3k

(3-cyclohexylnaphthalen-2-yloxy)triisopropylsilane 3k: Siloxy naphthalene **3k** was prepared according to the Representative Procedure using phthalazine **1a** (65 mg, 0.5 mmol), $AgNTf_2$ (0.01 mmol, 2 mol%), 2,2′-bipyridine **8** (0.011 mmol, 2.2 mol%), siloxy alkyne **2k** (210 mg, 0.75 mmol) and CH_2Cl_2 (1.0 mL). [The catalyst stock solution was prepared by dissolving 2,2′-bipyridine **8** (8.6 mg, 0.055 mmol) and $AgNTf_2$ (19.4 mg, 0.05 mmol) in 5.0 mL CH_2Cl_2 and 1.0 mL of this solution was added onto phthalazine **1a**.] At the end of 3 h, the reaction mixture was filtered through a plug of silica gel, washed with additional CH_2Cl_2 and concentrated *in vacuo*. The resulting mixture was then heated in a Kugelrohr distillation apparatus under reduced pressure (120 °C; 2 mmHg) for 1 h. Purification by flash column chromatography (hexanes only) gave pure siloxy naphthalene **3k** (158 mg, 83%) as a colorless oil, which solidified upon standing in the refrigerator to form a white solid.

mp: 65-66 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.71 (d, J = 8.1 Hz, 1H), 7.61 (d, J = 8.1 Hz, 1H), 7.59 (s, 1H), 7.34 (dt, J = 7.5, 1.2 Hz, 1H), 7.27 (dt, J = 7.5, 1.1 Hz, 1H), 7.09 (s, 1H), 3.10 (br t, 1H), 1.98 (br d, 2H), 1.87 (br s, 2H), 1.78 (br d, J = 12.7 Hz, 1H), 1.49-1.37 (m, 7H), 1.34-1.25 (m, 1H), 1.16 (d, J = 7.5 Hz, 18H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 152.4, 139.8, 132.8, 129.1, 127.2, 125.9, 125.2, 125.1, 123.3, 112.3, 37.5, 33.5, 27.2, 26.5, 18.2, 13.1; IR (film): 2926, 2866, 1631, 1599, 1497, 1466, 1389, 1252, 1179, 1101, 1001, 929, 882 cm^{-1}; HRMS (ESI) Calcd for $(C_{25}H_{38}OSi)Na^+$ $(M+Na)^+$: 405.2584, Found: 405.2575.



3l

(3-*tert*-butylnaphthalen-2-yloxy)triisopropylsilane 3l: A test tube charged with phthalazine **1a** (65 mg, 0.5 mmol), 2,2′-bipyridine **8** (8.6 mg, 0.055 mmol) and $AgNTf_2$ (19.4 mg, 0.05 mmol) was evacuated and refilled with nitrogen three times. 0.5 mL of

anhydrous CH_2Cl_2 was added and the resulting clear, light yellow solution was stirred for 15 min under nitrogen. Siloxy alkyne **2l** (255 mg, 1.0 mmol) was weighed in a 1.0-mL syringe and added to the reaction mixture slowly as a neat liquid. The reaction mixture was stirred at room temperature, under nitrogen for 22 h. It was then filtered through a plug of silica gel, washed with additional CH_2Cl_2 and concentrated *in vacuo* to afford a yellow-orange oil. Purification by flash column chromatography (hexanes only) gave pure siloxy naphthalene **3l** (120 mg, 67%) as a colorless oil, which solidified upon standing to form a white solid.

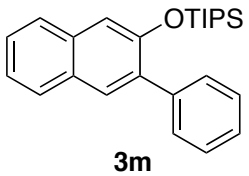
The same reaction was performed once more using phthalazine **1a** (65 mg, 0.5 mmol), 2,2′-bipyridine **8** (8.6 mg, 0.055 mmol), $AgNTf_2$ (19.4 mg, 0.05 mmol), siloxy alkyne **2l** (255 mg, 1.0 mmol) and anhydrous CH_2Cl_2 (0.5 mL) according to the procedure described above. The reaction mixture was stirred in refluxing CH_2Cl_2 (45 °C) for 7 h. It was then filtered through a plug of silica gel, washed with additional CH_2Cl_2 and concentrated *in vacuo* to afford an orange oil. Purification by flash column chromatography (hexanes only) gave pure siloxy naphthalene **3l** (142 mg, 80%) as a colorless oil, which solidified upon standing to form a white solid.

Cycloaddition of Phthalazine 1a with Siloxy Alkyne 2l on a 10 mmol Scale:

A 50-mL, oven-dried, single-necked, round-bottomed flask equipped with a magnetic stir bar was charged with phthalazine **1a** (1.30 g, 10.0 mmol), 2,2′-bipyridine **8** (171.8 mg, 1.1 mmol) and $AgNTf_2$ (388.0 mg, 1.0 mmol). The flask was evacuated under vacuum for 5 min and refilled with nitrogen. 10 mL of anhydrous CH_2Cl_2 was added and the resulting clear, light yellow solution was stirred for 15 min under nitrogen. Siloxy alkyne **2l** (3.82 g, 15.0 mmol) was weighed in a 10-mL syringe and added to the reaction mixture slowly as a neat liquid. The resulting clear, yellow solution was stirred at room temperature for 10 min. The reaction flask was then quickly fitted with a reflux condenser and inserted into a preheated oil bath at 45 °C. The reaction mixture was stirred in refluxing CH_2Cl_2, under nitrogen for 10 h during which time the color first turned green and then brown. The reaction mixture was filtered through silica gel (6 g),

washed with additional CH_2Cl_2 (120 mL) and concentrated *in vacuo* to afford an orange-brown oil. Flash column chromatography of the resulting mixture (hexanes only) gave pure siloxy naphthalene **3l** (2.97 g, 83%) as a white solid.

mp: 59-60 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.77 (d, *J* = 8.2 Hz, 1H), 7.75 (s, 1H), 7.64 (d, *J* = 8.1 Hz, 1H), 7.39 (dt, *J* = 7.4, 1.0 Hz, 1H), 7.32 (dt, *J* = 7.5, 1.0 Hz, 1H), 7.13 (s, 1 H), 1.56-1.50 (m, 12H), 1.23 (d, *J* = 7.5 Hz, 18H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 153.7, 140.9, 132.9, 128.7, 127.5, 125.7, 125.44, 125.39, 123.4, 113.2, 35.3, 29.9, 18.3, 113.4; IR (film): 2947, 2868, 1632, 1594, 1464, 1330, 1253, 1215, 1184, 1059, 926, 882, 871, 822 cm^{-1}; HRMS (ESI) Calcd for $(C_{23}H_{36}OSi)Na^+$ $(M+Na)^+$: 379.2428, Found: 379.2425.



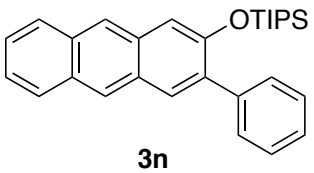
3m

Triisopropyl(3-phenylnaphthalen-2-yloxy)silane 3m: Siloxy naphthalene **3m** was prepared according to the Representative Procedure using phthalazine **1a** (130 mg, 1.0 mmol), $AgNTf_2$ (0.01 mmol, 1 mol%), 2,2′-bipyridine **8** (0.011 mmol, 1.1 mol%), siloxy alkyne **2m** (357 mg, 1.3 mmol) and CH_2Cl_2 (1.0 mL). [The catalyst stock solution was prepared by dissolving 2,2′-bipyridine **8** (8.6 mg, 0.055 mmol) and $AgNTf_2$ (19.4 mg, 0.05 mmol) in 5.0 mL CH_2Cl_2 and 1.0 mL of this solution was added onto phthalazine **1a**.] At the end of 1 h, the reaction mixture was filtered through a plug of silica gel, washed with additional CH_2Cl_2 and concentrated *in vacuo*. The resulting mixture was then heated in a Kugelrohr distillation apparatus under reduced pressure (170 °C; 1 mmHg) for 1 h. Purification by flash column chromatography (hexanes only) gave pure siloxy naphthalene **3m** (357 mg, 95%) as a light yellow oil, which solidified upon standing in the refrigerator to form a white solid.

This reaction was performed according to the above procedure using phthalazine **1a** (130 mg, 1.0 mmol), $AgNTf_2$ (0.005 mmol, 0.5 mol%), 2,2′-bipyridine **8** (0.0055 mmol, 0.55 mol%), siloxy alkyne **2m** (357 mg, 1.3 mmol) and CH_2Cl_2 (1.0 mL) (Reaction time = 3

h). Purification by flash column chromatography (hexanes only) gave pure siloxy naphthalene **3m** (347 mg, 92%) as a light yellow oil.

mp: 57-58 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.77 (d, *J* = 8.1 Hz, 1H), 7.75 (s, 1H), 7.69 (d, *J* = 8.2 Hz, 1H), 7.57 (d, *J* = 7.0 Hz, 2H), 7.43-7.39 (m, 3H), 7.33 (dt, *J* = 7.7, 1.1 Hz, 2H), 7.24 (s, 1 H), 1.20 (sept, *J* = 7.5 Hz, 3H), 0.99 (d, *J* = 7.5 Hz, 18H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 151.7, 139.1, 134.9, 133.9, 129.90, 129.85, 129.1, 127.7, 127.6, 126.9, 126.1, 126.0, 123.8, 113.8, 17.9, 12.9; IR (film): 3056, 2945, 2866, 1630, 1595, 1502, 1493, 1462, 1437, 1356, 1336, 1277, 1267, 1246, 1201, 1179, 992, 929, 882, 857 cm^{-1}; HRMS (ESI) Calcd for $(C_{25}H_{32}OSi)Na^{+}$ $(M+Na)^{+}$: 399.2115, Found: 399.2117.

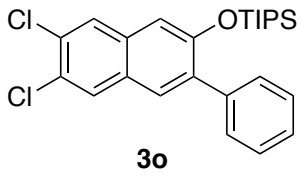


3n

Triisopropyl(3-phenylanthracen-2-yloxy)silane 3n: Siloxy anthracene **3n** was prepared according to the Representative Procedure using benzo[g]phthalazine **1b** (180 mg, 1.0 mmol), $AgNTf_2$ (0.01 mmol, 1 mol%), 2,2′-bipyridine **8** (0.011 mmol, 1.1 mol%), siloxy alkyne **2m** (357 mg, 1.3 mmol) and CH_2Cl_2 (3.0 mL). [The catalyst stock solution was prepared by dissolving 2,2′-bipyridine **8** (8.6 mg, 0.055 mmol) and $AgNTf_2$ (19.4 mg, 0.05 mmol) in 5.0 mL CH_2Cl_2 and 1.0 mL of this solution was added onto the solution of benzo[g]phthalazine **1b** in 2.0 mL CH_2Cl_2.] At the end of 3 h, the reaction mixture was filtered through a plug of silica gel, washed with additional CH_2Cl_2 and concentrated *in vacuo*. Purification by flash column chromatography (hexanes only) gave pure siloxy anthracene **3n** (356 mg, 84%) as a yellow oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 8.35 (s, 1H), 8.24 (s, 1H), 7.94 (t, *J* = 8.0 Hz, 2H), 7.92 (s, 1H), 7.61 (d, *J* = 7.2 Hz, 2H), 7.44-7.34 (m, 6H), 1.25 (sept, *J* = 7.6 Hz, 3H), 1.01 (d, *J* = 7.5 Hz, 18H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 151.6, 139.0, 136.1, 132.3, 132.0, 130.6, 129.9, 128.4, 128.2, 127.8, 127.6, 127.1, 126.1, 125.4, 124.4, 123.5, 112.2, 17.9, 12.9; ^{13}C NMR (125 MHz, CD_2Cl_2): δ 152.2, 139.6, 136.7, 132.9, 132.6, 131.2, 130.5,

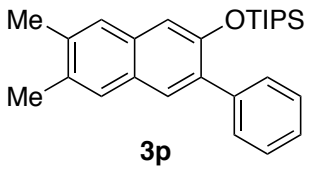
130.4, 129.0, 128.7, 128.4, 128.1, 127.7, 126.6, 126.0, 125.1, 124.0, 112.8, 18.3, 13.5; IR (film): 2944, 2866, 1629, 1457, 1436, 1293, 1212, 1175, 994, 900, 884 cm^{-1}; HRMS (ESI) Calcd for $(C_{29}H_{34}OSi)Na^+$ $(M+Na)^+$: 449.2271, Found: 449.2263.



3o

(6,7-dichloro-3-phenylnaphthalen-2-yloxy)triisopropylsilane 3o: Siloxy naphthalene **3o** was prepared according to the Representative Procedure using 6,7-dichlorophthalazine **1c** (100 mg, 0.5 mmol), $AgNTf_2$ (0.005 mmol, 1 mol%), 2,2′-bipyridine **8** (0.0055 mmol, 1.1 mol%), siloxy alkyne **2m** (178 mg, 0.65 mmol) and CH_2Cl_2 (5.0 mL). [The catalyst stock solution was prepared by dissolving 2,2′-bipyridine **8** (8.6 mg, 0.055 mmol) and $AgNTf_2$ (19.4 mg, 0.05 mmol) in 5.0 mL CH_2Cl_2 and 0.5 mL of this solution was added onto the solution of 6,7-dichlorophthalazine **1c** in 4.5 mL CH_2Cl_2.] At the end of 2 h, the reaction mixture was filtered through a plug of silica gel, washed with additional CH_2Cl_2 and concentrated *in vacuo*. Purification by flash column chromatography (hexanes only) gave pure siloxy naphthalene **3o** (190 mg, 85%) as a colorless oil.

^1H NMR (500 MHz, $CDCl_3$): δ 7.85 (s, 1H), 7.80 (s, 1H), 7.64 (s, 1H), 7.53 (d, J = 7.7 Hz, 2H), 7.41 (t, J = 7.4 Hz, 2H), 7.35 (t, J = 7.3 Hz, 1H), 7.12 (s, 1H), 1.19 (sept, J = 7.5 Hz, 3H), 0.97 (d, J = 7.5 Hz, 18H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 152.9, 138.3, 136.3, 132.9, 130.2, 129.8, 128.8, 128.5, 128.0, 127.9, 127.7, 127.3, 127.0, 112.7, 17.8, 12.9; IR (film): 2945, 2867, 1627, 1584, 1479, 1399, 1354, 1121, 973, 885, 813 cm^{-1}; HRMS (ESI) Calcd for $(C_{25}H_{30}Cl_2OSi)H^+$ $(M+H)^+$: 445.1516, Found: 445.1508.



3p

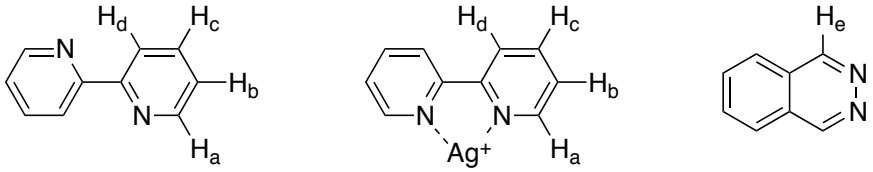
(6,7-dimethyl-3-phenylnaphthalen-2-yloxy)triisopropylsilane 3p: Siloxy naphthalene **3p** was prepared according to the Representative Procedure using 6,7-

dimethylphthalazine **1d** (158 mg, 1.0 mmol), $AgNTf_2$ (0.01 mmol, 1 mol%), 2,2′-bipyridine **8** (0.011 mmol, 1.1 mol%), siloxy alkyne **2m** (357 mg, 1.3 mmol) and CH_2Cl_2 (2.0 mL). [The catalyst stock solution was prepared by dissolving 2,2′-bipyridine **8** (8.6 mg, 0.055 mmol) and $AgNTf_2$ (19.4 mg, 0.05 mmol) in 5.0 mL CH_2Cl_2 and 1.0 mL of this solution was added onto the solution of 6,7-dimethylphthalazine **1d** in 1.0 mL CH_2Cl_2.] At the end of 6 h, the reaction mixture was filtered through a plug of silica gel, washed with additional CH_2Cl_2 and concentrated *in vacuo*. Purification by flash column chromatography (hexanes only) gave pure siloxy naphthalene **3p** (378 mg, 94%) as a colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.63 (s, 1H), 7.56 (app d, J = 7.0 Hz, 2H), 7.52 (s, 1H), 7.46 (s, 1H), 7.39 (app t, J = 7.4 Hz, 2H), 7.31 (app t, J = 7.3 Hz, 1H), 7.14 (s, 1H), 2.41 (s, 3H), 2.39 (s, 3H), 1.18 (sept, J = 7.5 Hz, 3H), 0.97 (d, J = 7.5 Hz, 18H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 151.1, 139.4, 135.7, 133.9, 133.3, 132.7, 129.9, 128.8, 128.0, 127.7, 127.1, 126.7, 125.8, 113.0, 20.2, 20.0, 17.9, 12.9; IR (film): 2943, 2866, 1609, 1463, 1411, 1371, 1267, 1201, 990, 900, 830 cm^{-1}; HRMS (ESI) Calcd for $(C_{27}H_{36}OSi)Na^+$ $(M+Na)^+$: 427.2428, Found: 427.2421.

^{1}H- and ^{13}C-NMR Studies on the Complexation of Ag^{+} to Phthalazine:

^{1}H- and ^{13}C-NMR experiments were carried out to see the effect of the complexation of $AgNTf_2$ to phthalazine in the presence of 2-2'-bipyridine. CD_2Cl_2 was used as the solvent in all experiments. First, the ^{1}H- and ^{13}C-NMR spectra of bipyridine and a 1:1 mixture of bipyridine and $AgNTf_2$ at 0.1 M concentrations were recorded (Tables 1 and 2, entries 1-2). The chemical shift of H_d of bipyridine shifted upfield from 8.43 ppm to 8.21 ppm whereas all the other signals shifted downfield upon complexation with $AgNTf_2$. This upfield shift of H_d can be attributed to the different conformations of bipyridine in the absence and presence of $AgNTf_2$ as shown below.



Then, the ^{1}H- and ^{13}C-NMR spectra of phthalazine were recorded in the presence of 0, 20, 50 and 100% $AgNTf_2$-bipyridine (1:1) (Tables 1 and 2, entries 3,4,5 and 6). In all experiments, only one set of signals was observed for each species with no line broadening, which indicated rapid complexation-decomplexation processes. Moreover, the chemical shift of H_e of phthalazine shifted gradually downfield from 9.52 to 9.69 ppm with the increasing amounts of $AgNTf_2$-bipyridine added. On the other hand, the chemical shifts of bipyridine in these experiments were very close to those found in entry 2 (Table 1) which suggested that bipyridine stayed complexed to $AgNTf_2$ in the presence of phthalazine. A similar trend was observed in ^{13}C-NMR spectra in which the chemical shift of C_1 of phthalazine shifted downfield from 151.6 to 155.0 ppm with the increasing amounts of $AgNTf_2$-bipyridine added (Table 2, entries 3,4,5 and 6). As a control experiment, the ^{1}H- and ^{13}C-NMR spectra of a 1:1 mixture of phthalazine and bipyridine (0.1 M) were recorded and the values were found to be essentially the same as those when the NMRs of both compounds were recorded separately (Tables 1 and 2, entry 8). It should also be noted that, in all experiments, the ^{1}H-NMR spectra were recorded after 10

and 45 minutes in order to see if an equilibration process would cause any change and the same spectra were obtained in each case.

Finally, ^{1}H- and ^{13}C-NMR spectra of a mixture of phthalazine and $AgNTf_2$ (1:0.2)[17] were recorded (Tables 1 and 2, entry 7). The chemical shifts of H_e and C_1 of phthalazine were found to be affected more in the absence of bipyridine (9.65 and 153.1 ppm, respectively).

Table 1. ^{1}H-NMR Studies

		^{1}H-NMR (ppm)[a]				
		Phthalazine	2-2'-Bipyridine			
		H_e	H_a	H_d	H_c	H_b
1	0.1 M bipyridine	-	8.66	8.43	7.83	7.32
2	0.1 M bipyridine+$AgNTf_2$ (1:1)	-	8.70	8.21	8.07	7.60
3	0.1 M phthalazine	9.52	-	-	-	-
4[b]	Phthalazine + bipyridine + $AgNTf_2$ (1:0.2:0.2)	9.57	8.73	8.27	8.06	7.57
5[b]	Phthalazine + bipyridine + $AgNTf_2$ (1:0.5:0.5)	9.64	8.71	8.26	-	7.58
6[b]	Phthalazine + bipyridine + $AgNTf_2$ (1:1:1)	9.69	8.69	8.24	-	7.58
7[b]	Phthalazine + $AgNTf_2$ (1:0.2)	9.65	-	-	-	-
8	0.1 M phthalazine + bipyridine (1:1)	9.51	8.65	8.43	7.82	7.31

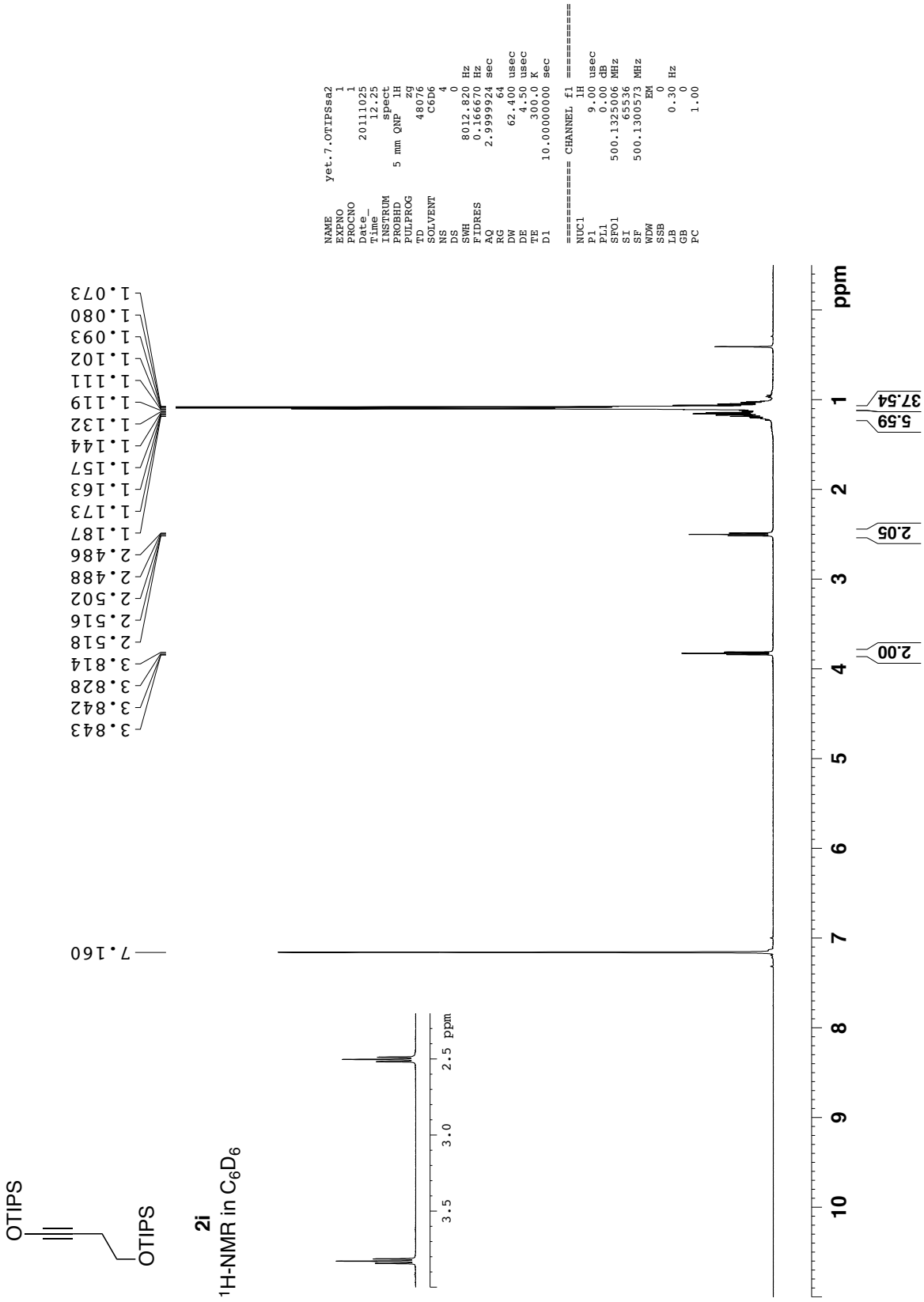
[a] Solvent (CD_2Cl_2) signal was calibrated to 5.32 ppm. [b][phthalazine] = 0.1 M.

[17] An attempt to prepare a 0.1 M, 1:1 mixture of phthalazine and $AgNTf_2$ failed due to the insolubility of the mixture.

Table 2. ^{13}C-NMR Studies

		^{13}C-NMR (ppm)[a]						
		Phthalazine		2-2'-Bipyridine				
1	0.1 M bipyridine	-	-	156.6	149.7	137.4	124.3	121.4
2	0.1 M bipyridine+$AgNTf_2$ (1:1)	-	-	151.7	151.7	140.1	126.6	122.9
3	0.1 M phthalazine	151.6	133.1	-	-	-	-	-
4[b]	Phthalazine + bipyridine + $AgNTf_2$ (1:0.2:0.2)	152.3	133.7	152.8	151.5	139.6	126.1	123.0
5[b]	Phthalazine + bipyridine + $AgNTf_2$ (1:0.5:0.5)	153.3	134.4	152.5	151.6	139.8	126.3	123.2
6[b]	Phthalazine + bipyridine + $AgNTf_2$ (1:1:1)	155.0	135.5	152.3	151.6	139.9	126.4	123.2
7[b]	Phthalazine + $AgNTf_2$ (1:0.2)	153.1	134.1	-	-	-	-	-
8	0.1 M phthalazine + bipyridine (1:1)	151.6	133.1	156.6	149.7	137.4	124.3	121.3

[a] Solvent (CD_2Cl_2) signal was calibrated to 54.0 ppm. [b][phthalazine] = 0.1 M.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OTIPS
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2.502
2.488
2.486
1.187
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1.111
1.102
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1.080
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D1 10.00000000 sec
CHANNEL f1
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P1 9.00 usec
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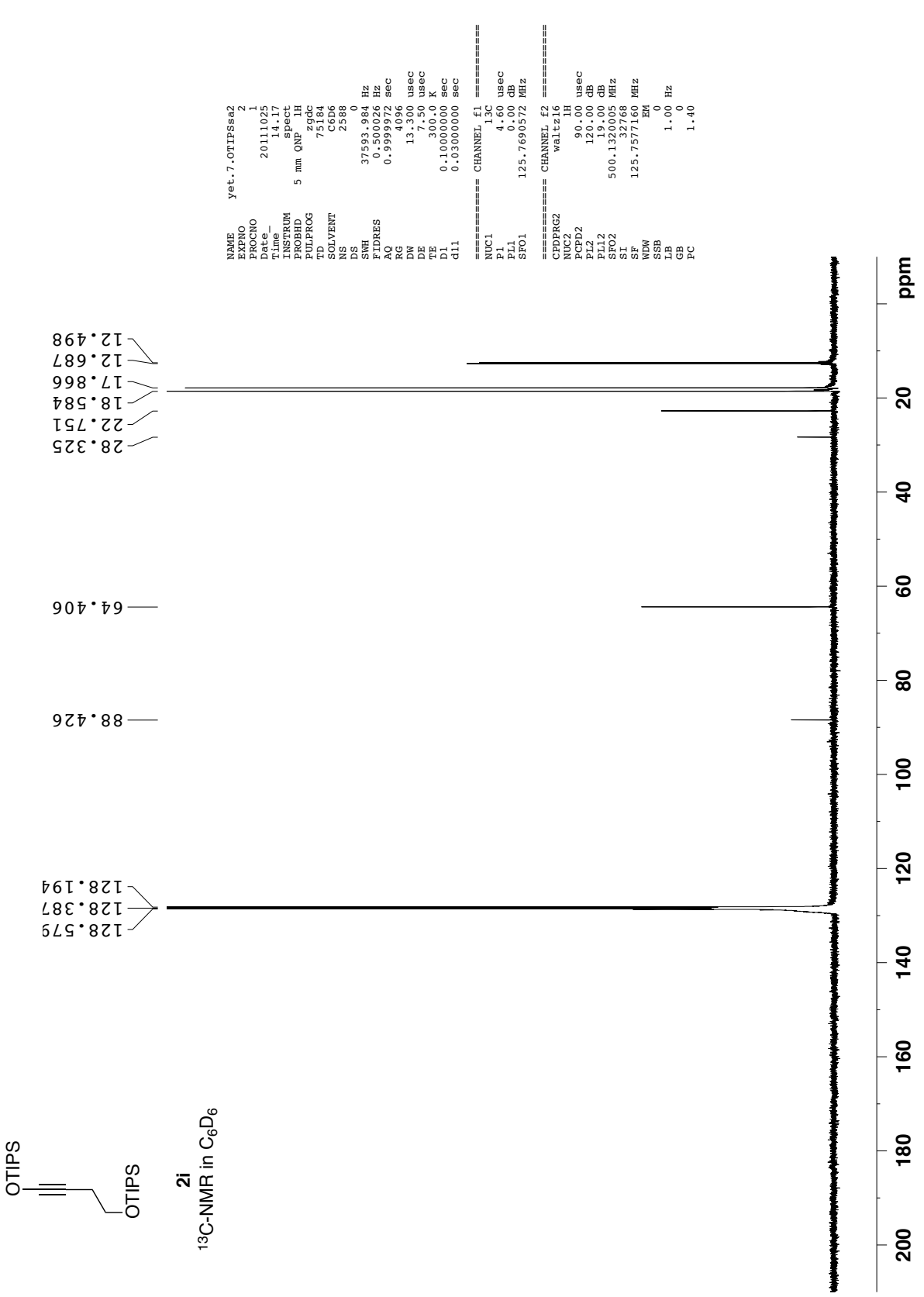
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OTIPS
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18.584
17.866
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ppm
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160
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120
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80
60
40
20
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PL1 0.00 dB
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PCPD2 90.00 usec
PL2 120.00 dB
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SI 32768
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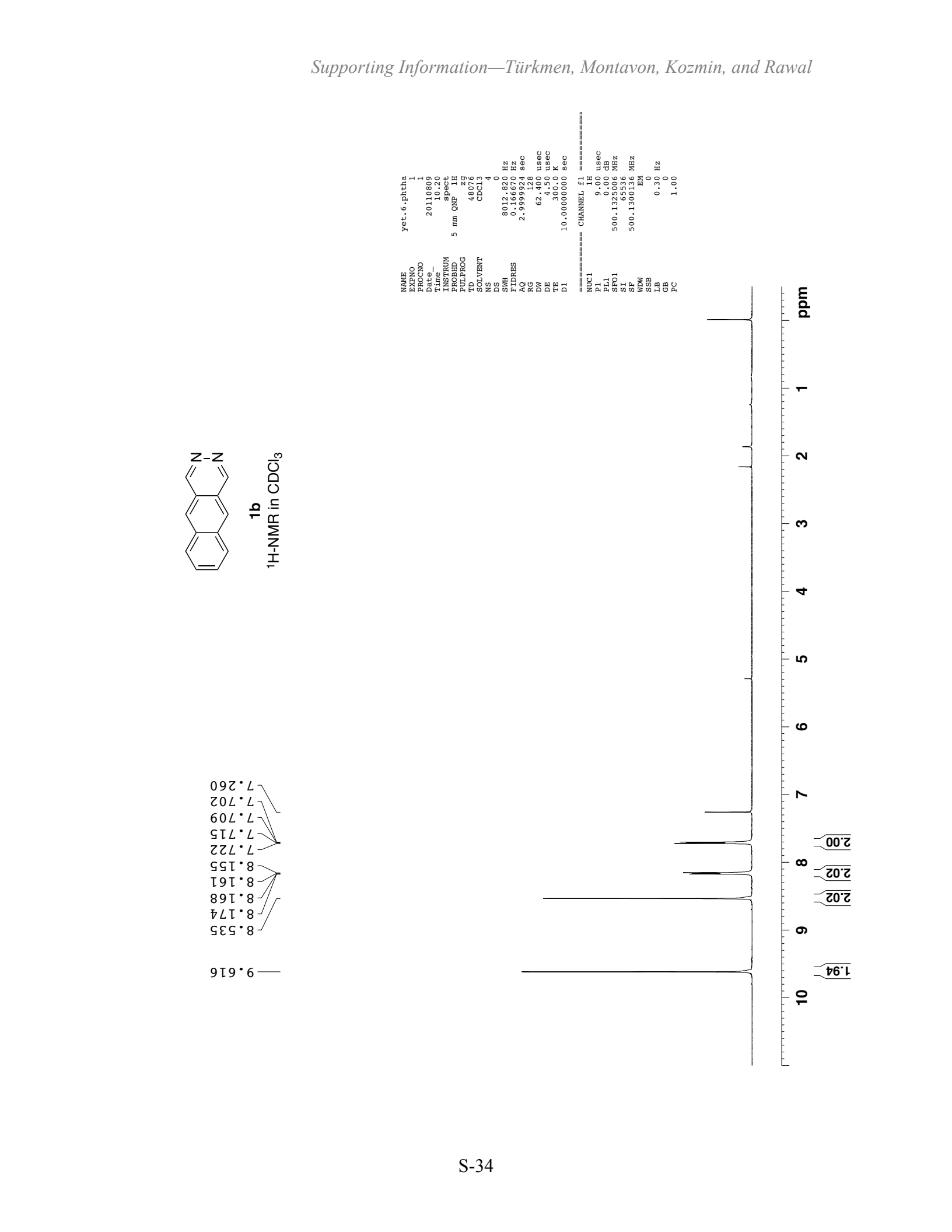
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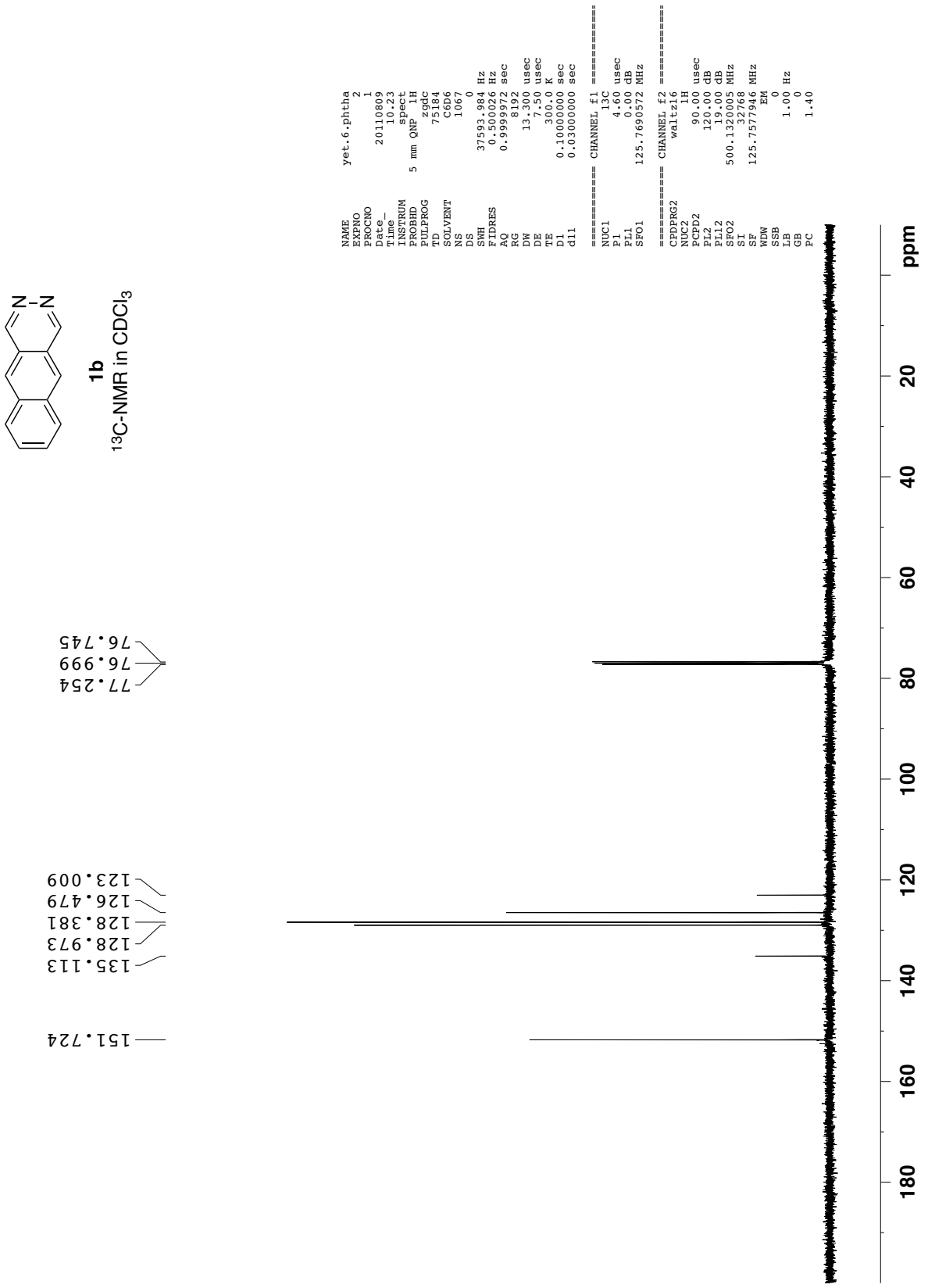
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NUC1 13C
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CHANNEL f2 waltz16
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NUC2 90.00 usec
PCPD2 120.00 dB
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PL12 500.1320005 MHz
SFO2 32768
SI 125.7577946 MHz
SF EM
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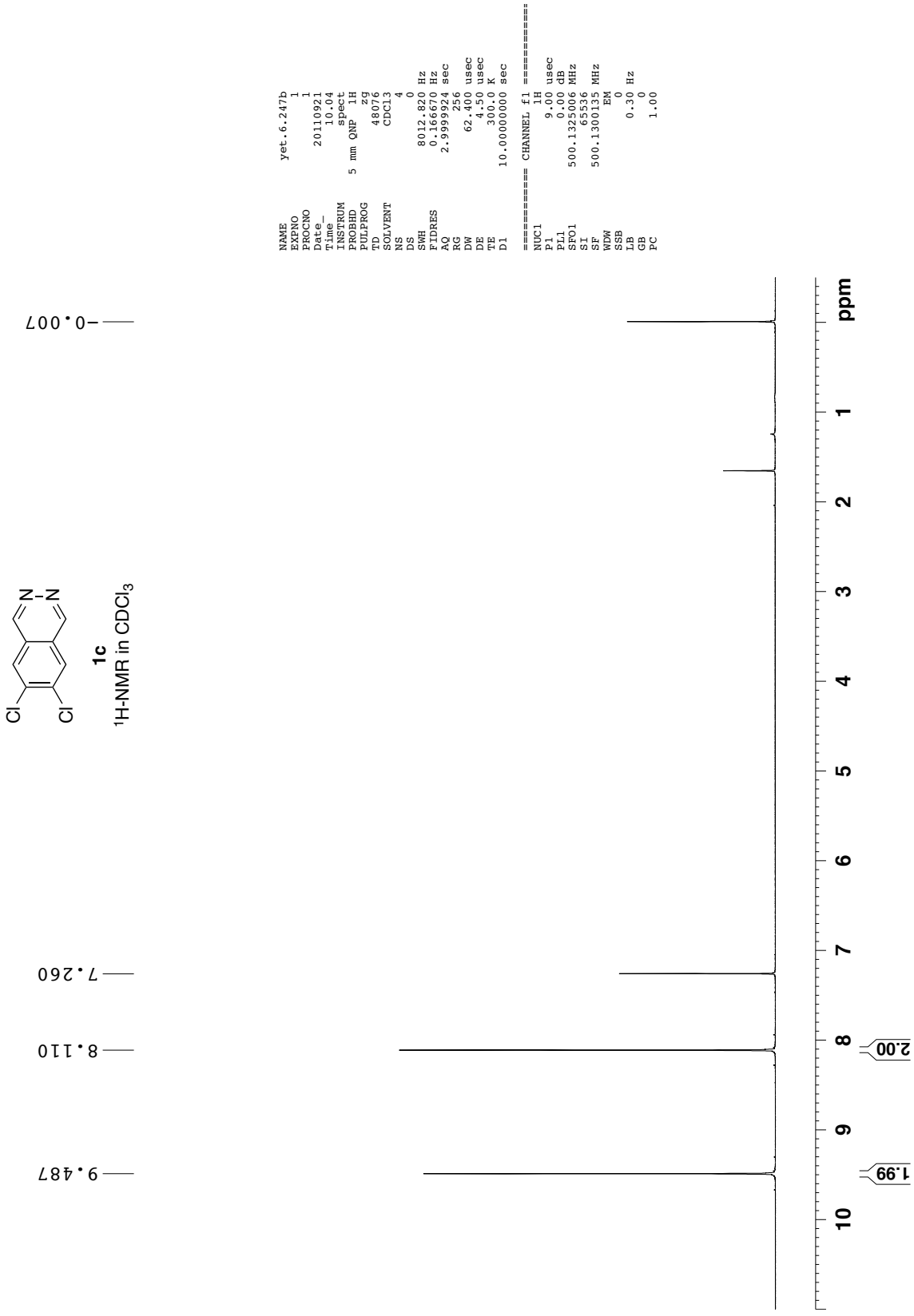
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WDW EM
SSB 0
LB 0.30 Hz
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N N
Cl Cl
1c
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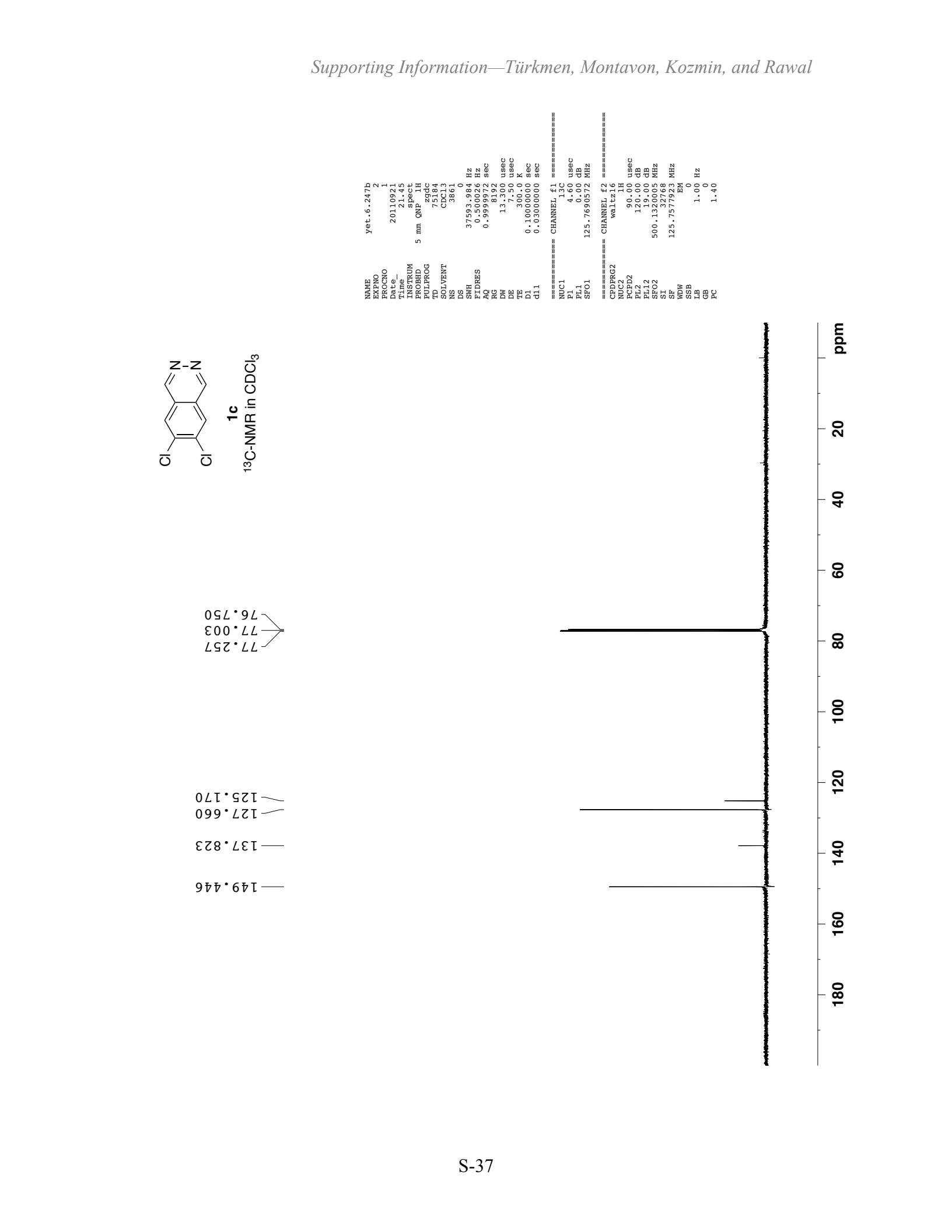
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Cl
N
N
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P1 4.60 usec
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SFO1 125.7690572 MHz
CHANNEL f2
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NUC2 1H
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PL2 120.00 dB
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SI 32768
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60
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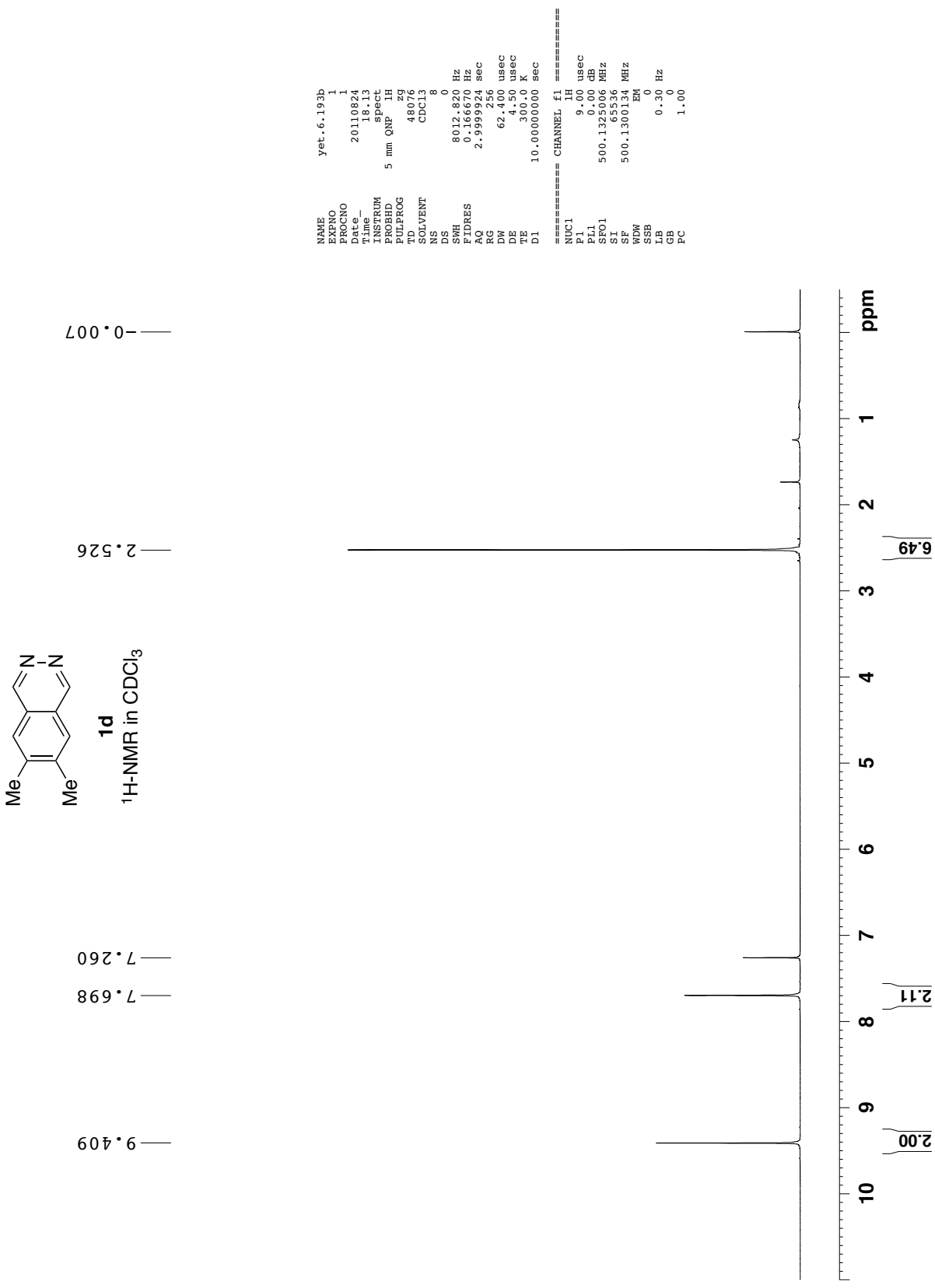
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ppm
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2
3
4
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Me
Me
1d
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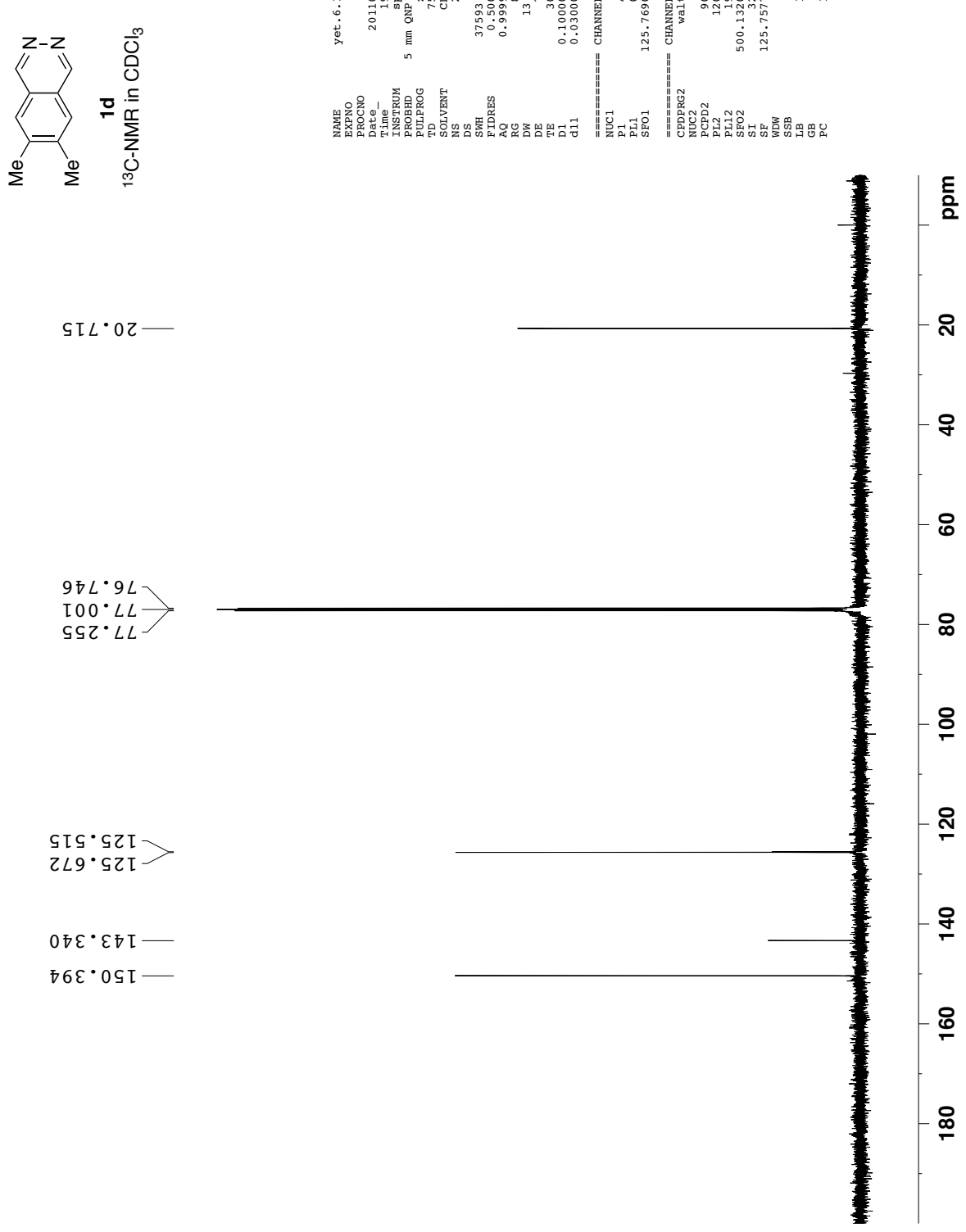
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Me
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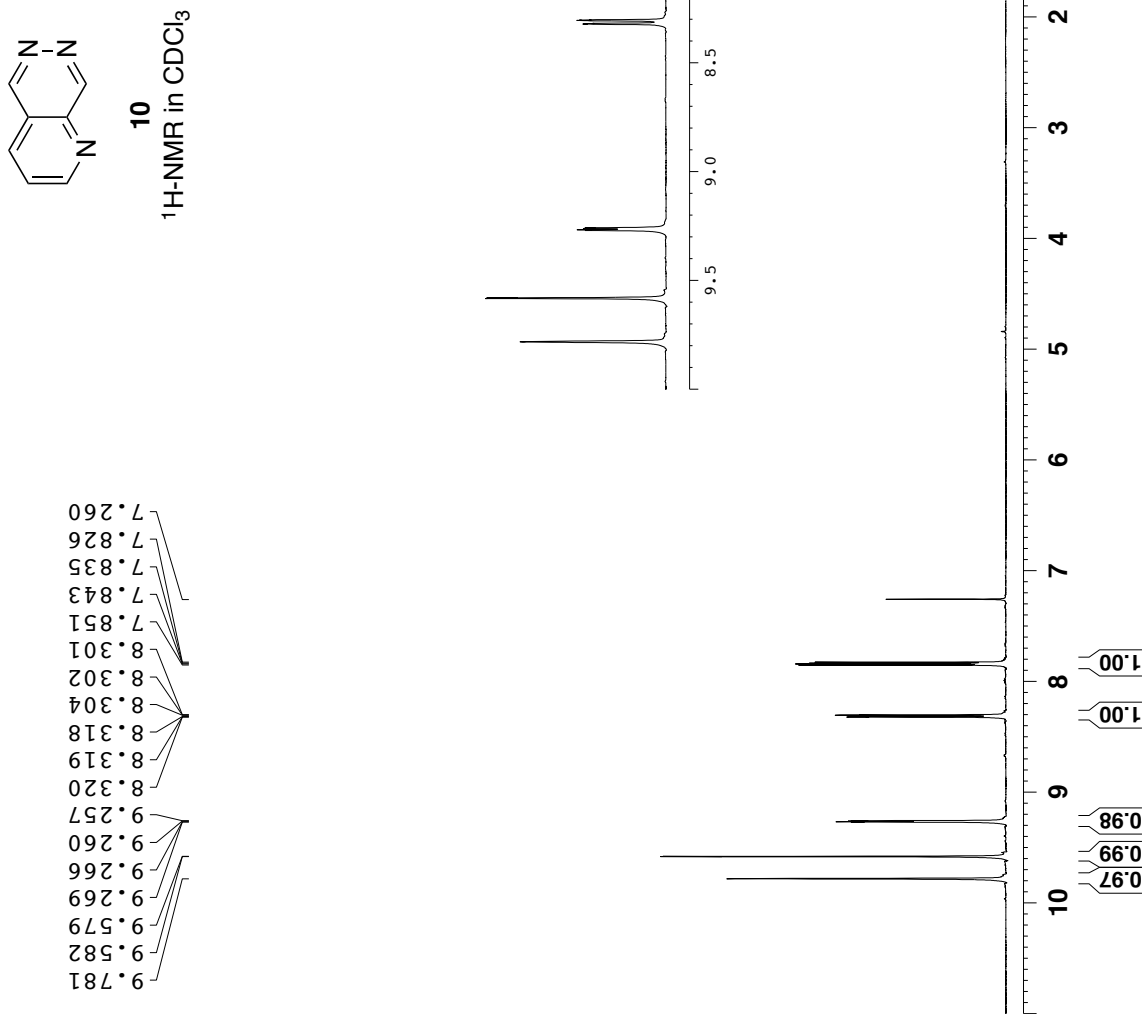
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9.257
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8.318
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8.302
8.301
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7.843
7.835
7.826
7.260
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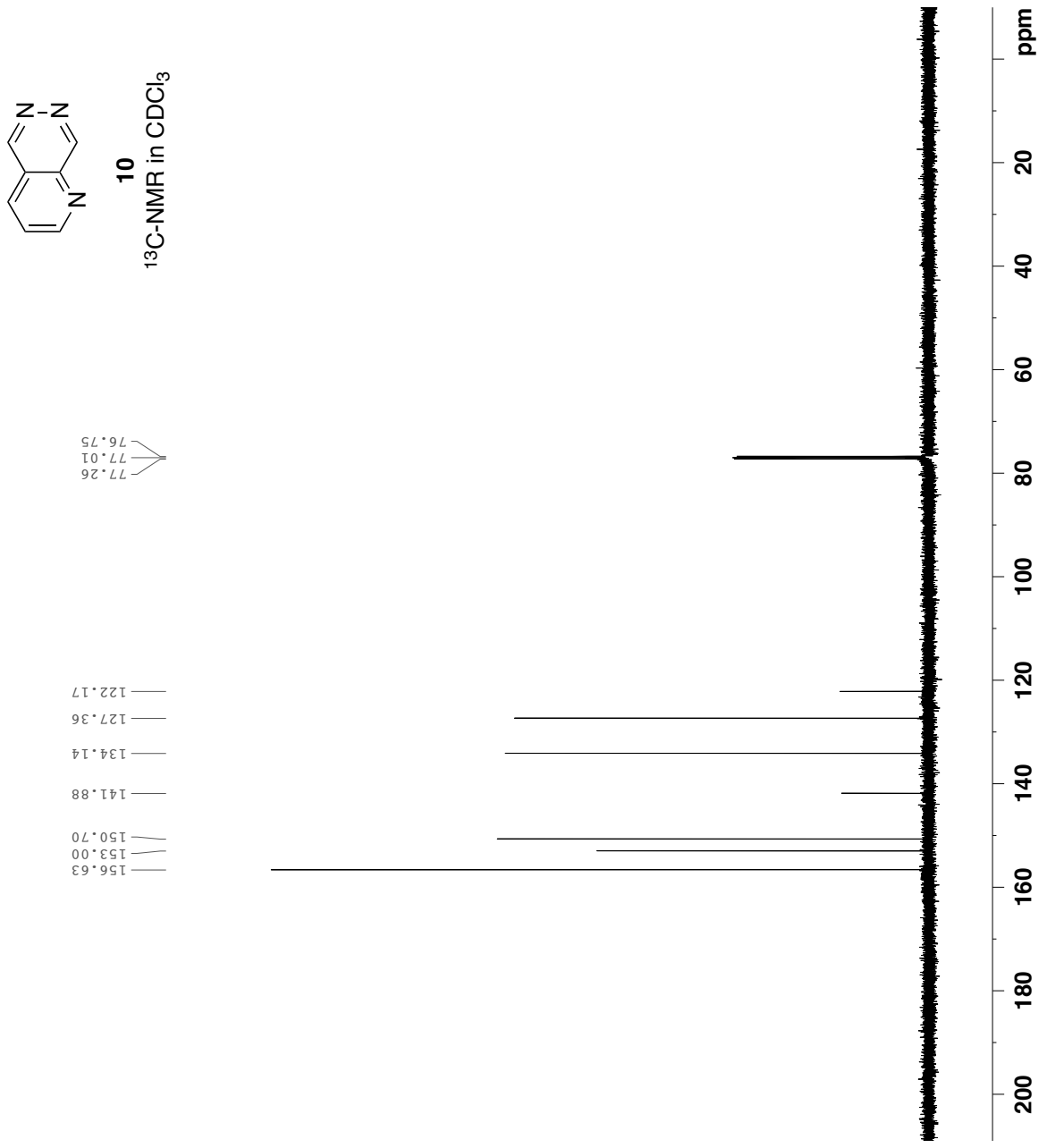
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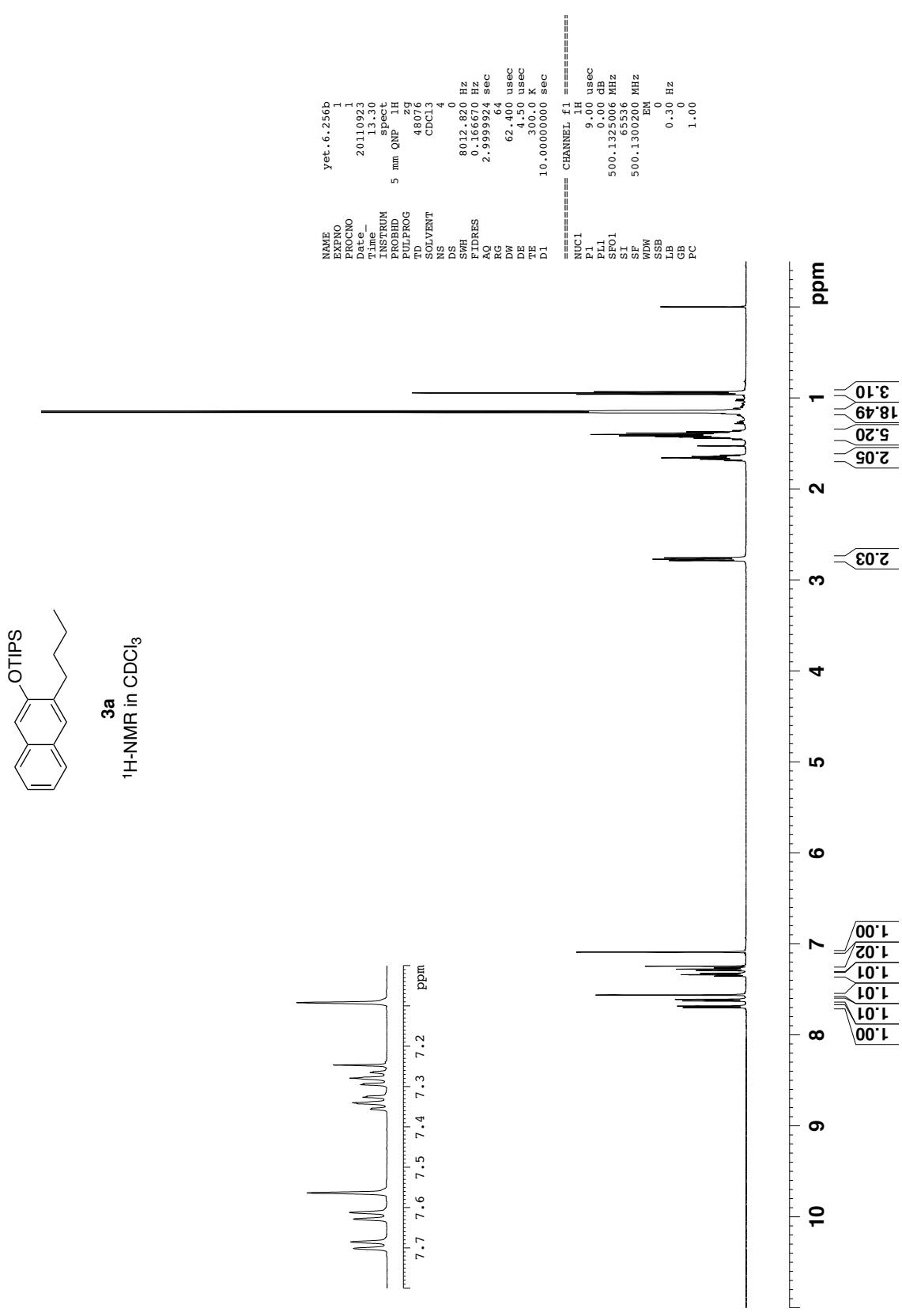
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DE 4.50 usec
TE 300.0 K
D1 10.00000000 sec
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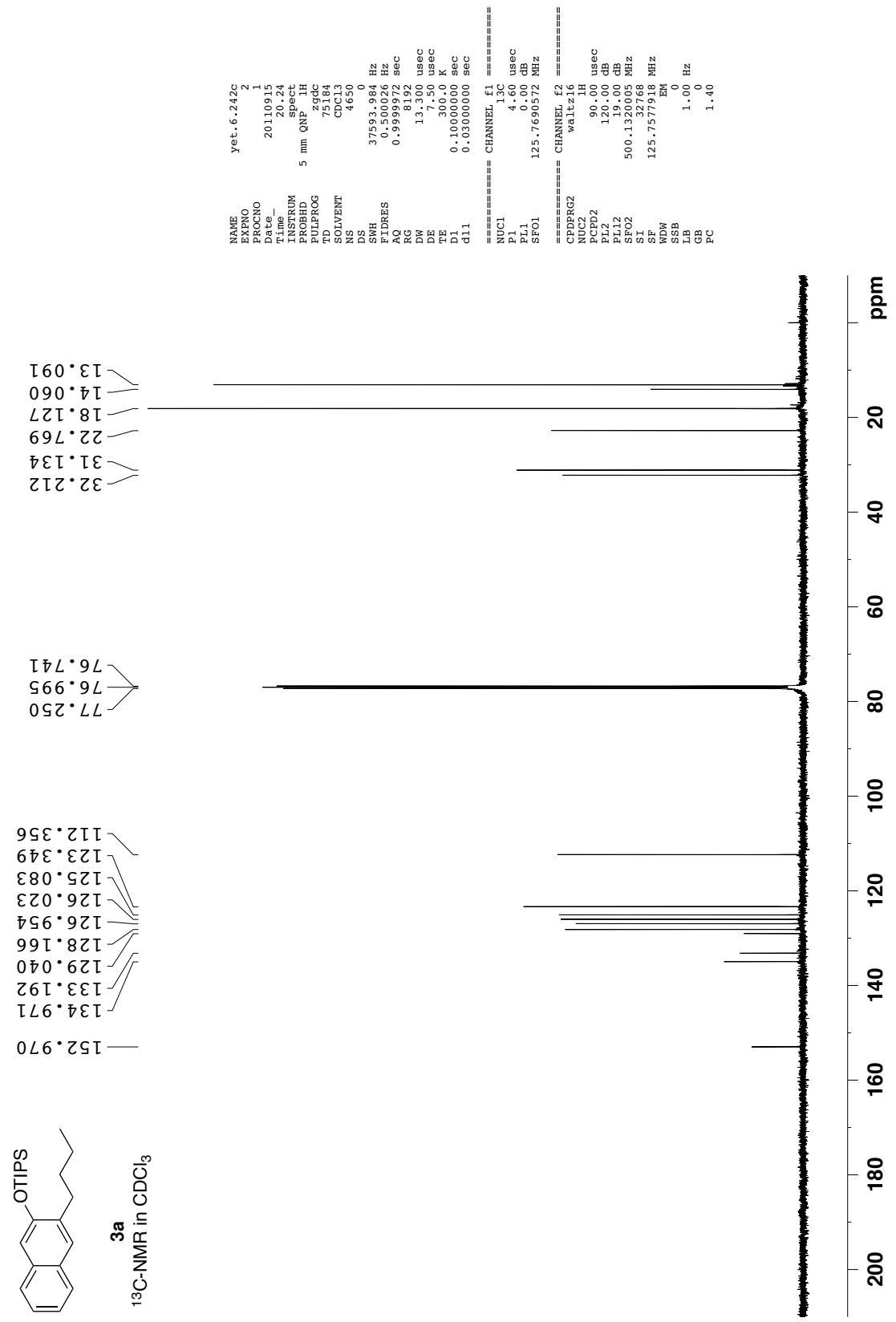


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TE                  300.0 K
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PCPD2               90.00 usec
PL2                120.00 dB
PL12                19.00 dB
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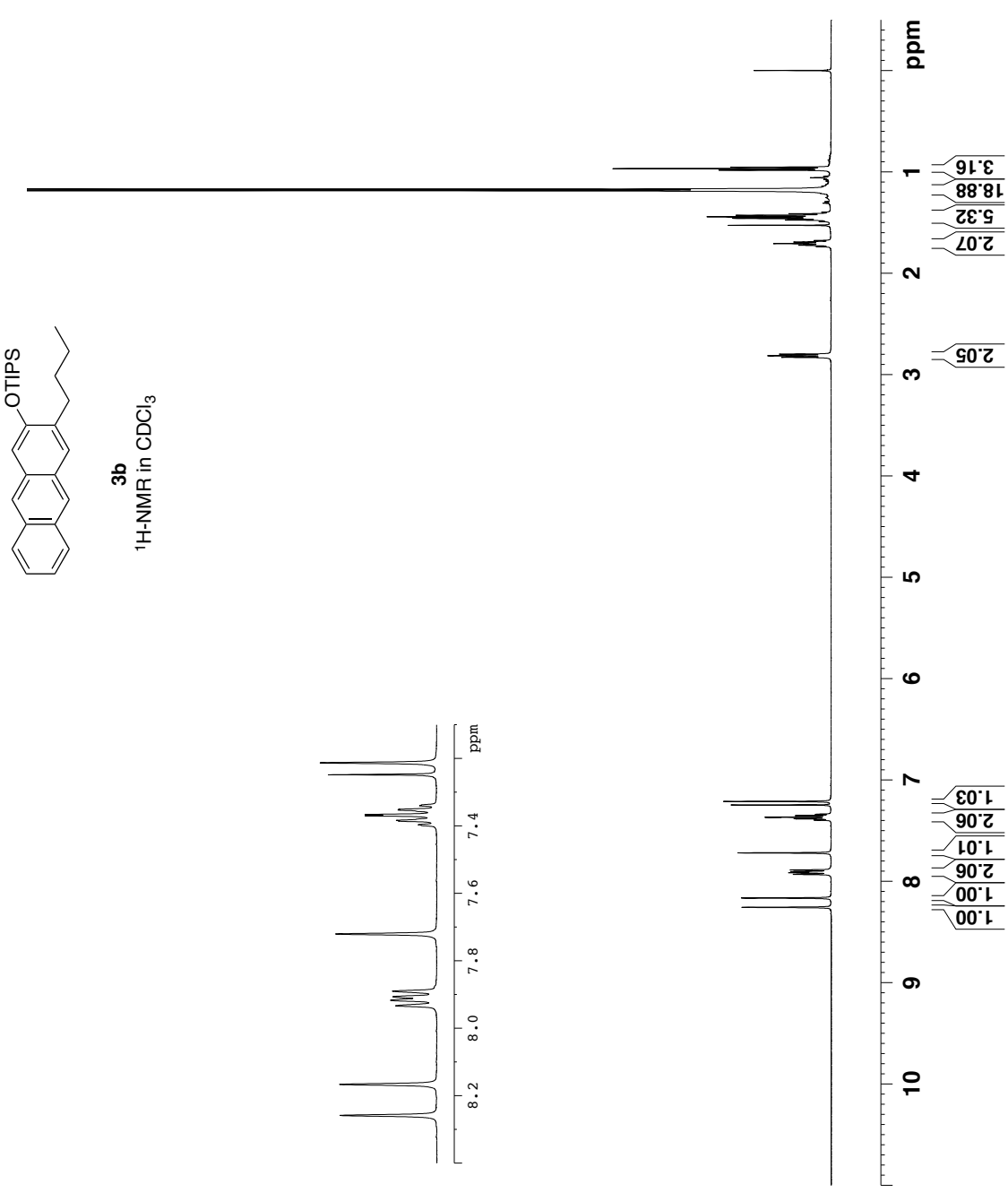
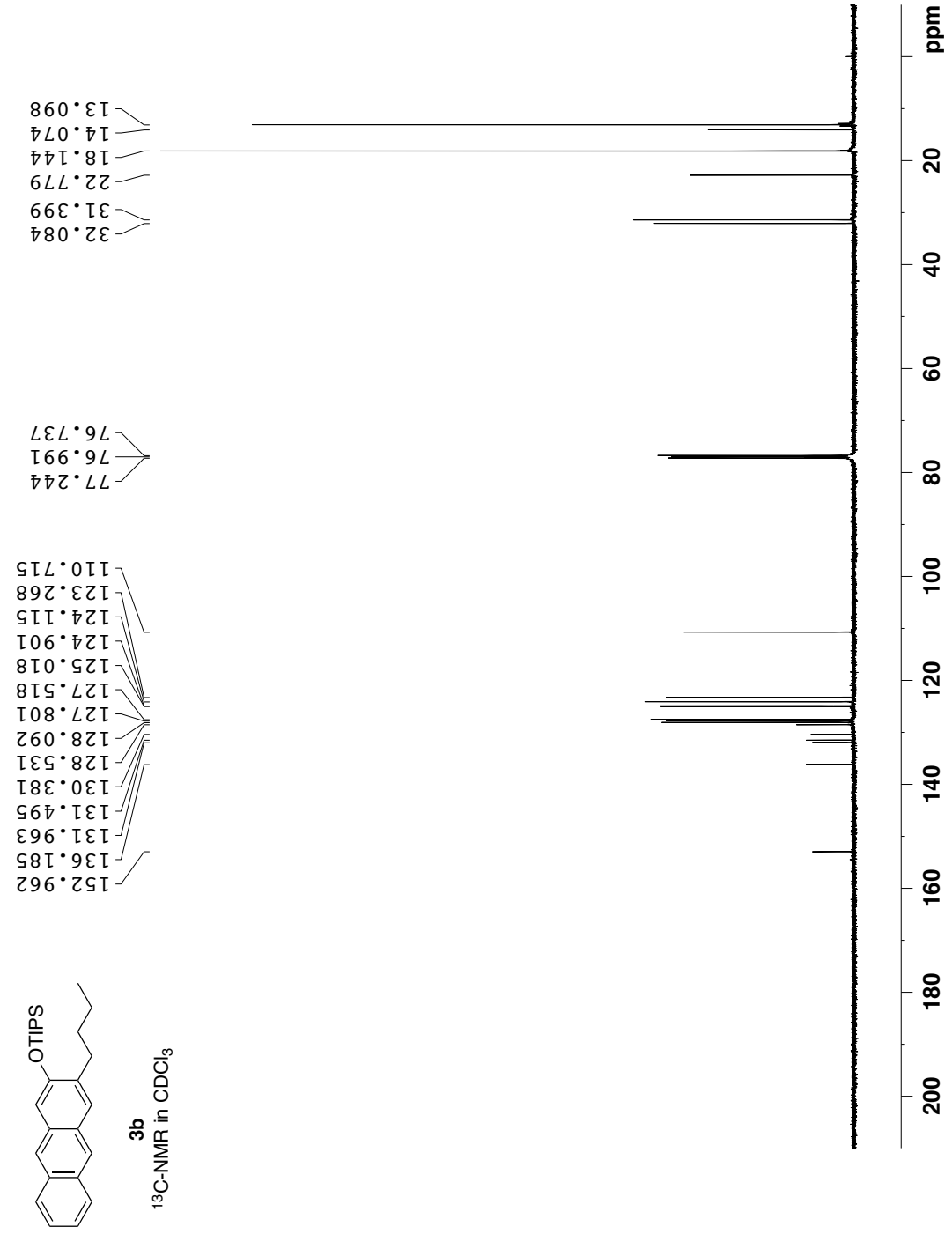
SSB                  1.00 Hz
LB                      0
GB                   1.40
PC
```

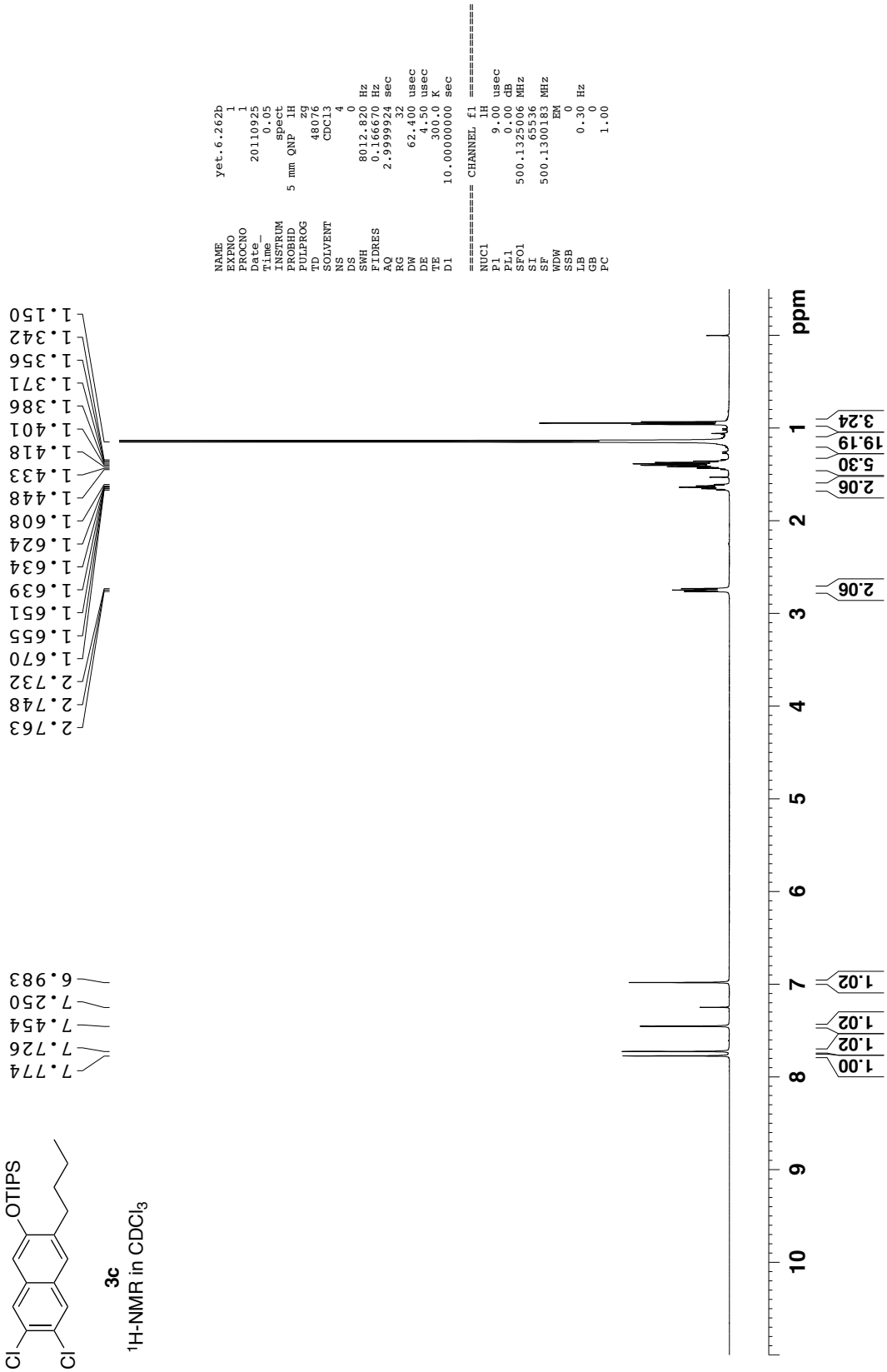

OTIPS
3b
^{1}H-NMR in $CDCl_3$
NAME yet.6.260b
EXPNO 1
PROCNO 1
Date_ 20110923
Time_ 22.11
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg
TD 48076
SOLVENT CDCl3
NS 4
DS 0
SWH 8012.820 Hz
FIDRES 0.166670 Hz
AQ 2.9999924 sec
RG 64
DW 62.400 usec
DE 4.50 usec
TE 300.0 K
D1 10.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 0.00 dB
SFO1 500.1325006 MHz
SI 65536
SF 500.1300195 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
3.16
18.88
5.32
2.07
2.05
1.03
2.06
1.01
2.06
1.00
1.00

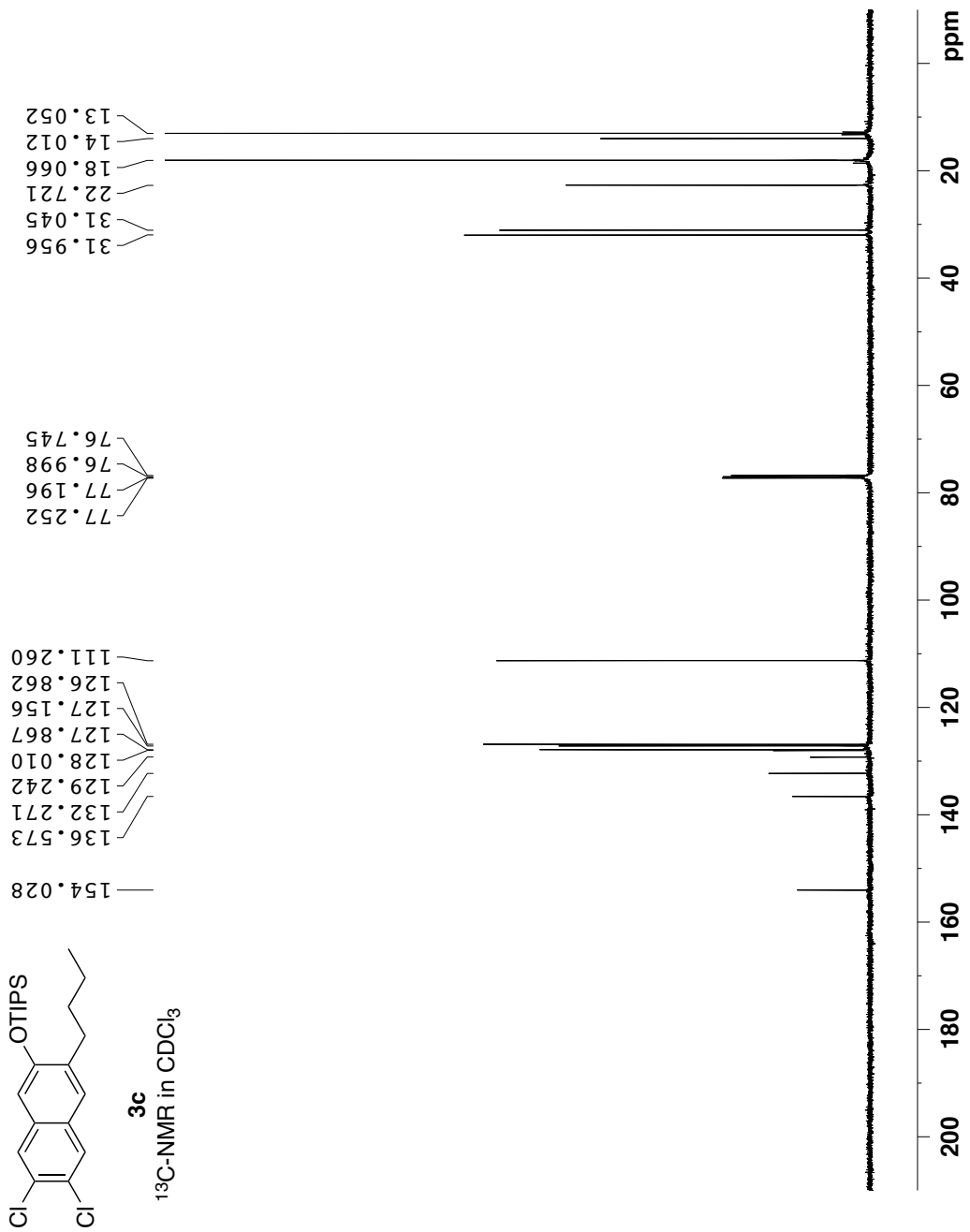



```
NAME                yet.6.260b
EXPNO                        2
PROCNO                       1
Date_                20110923
Time_                   22.27
INSTRUM                  spect
PROBHD     5 mm QNP 1H
PULPROG                 zgdc
TD                      75184
SOLVENT                 C6D6
NS                       2996
DS                          0
SWH                37593.984 Hz
FIDRES                 0.500026 Hz
AQ                     0.999997 sec
RG                       8192
DW                     13.300 usec
DE                      7.50 usec
TE                     300.0 K
D1                     0.10000000 sec
d11                    0.03000000 sec

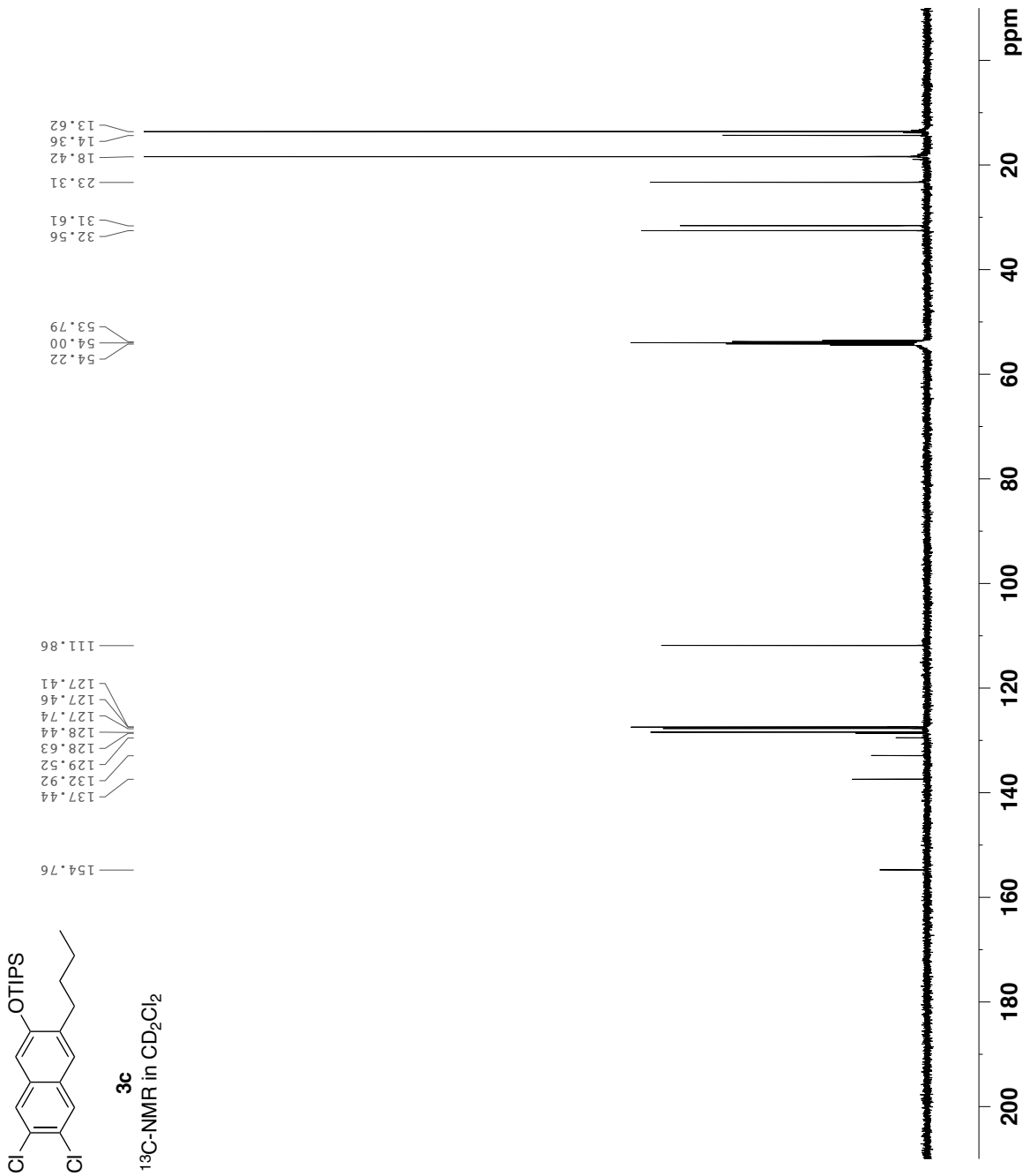
========== CHANNEL f1 ============
NUC1                        13C
P1                        4.60 usec
PL1                       0.00 dB
SFO1               125.7690572 MHz

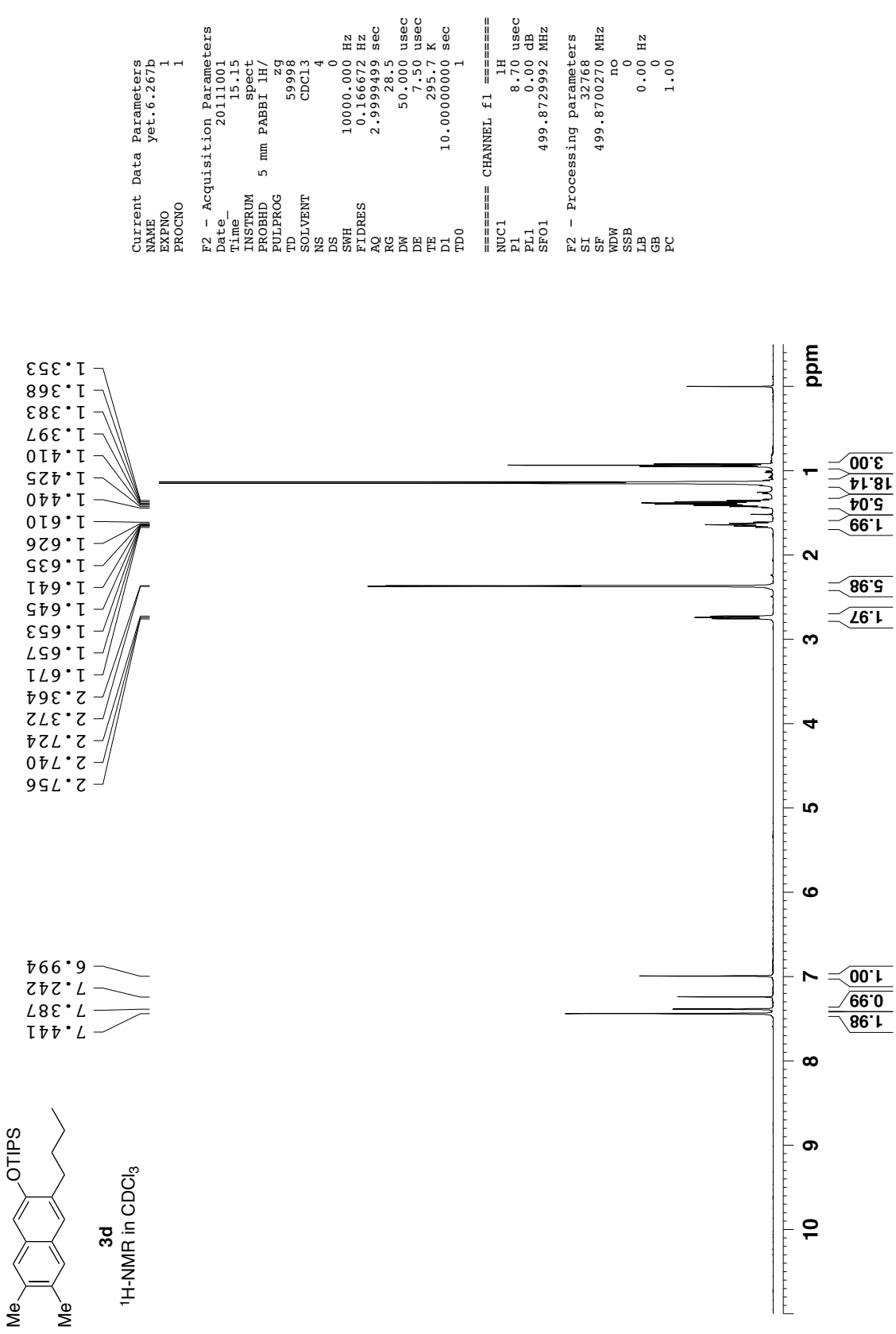
========== CHANNEL f2 ============
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                     90.00 usec
PL2                      120.00 dB
PL12                      19.00 dB
SFO2               500.1320005 MHz
SI                       32768
SF                 125.7577929 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```

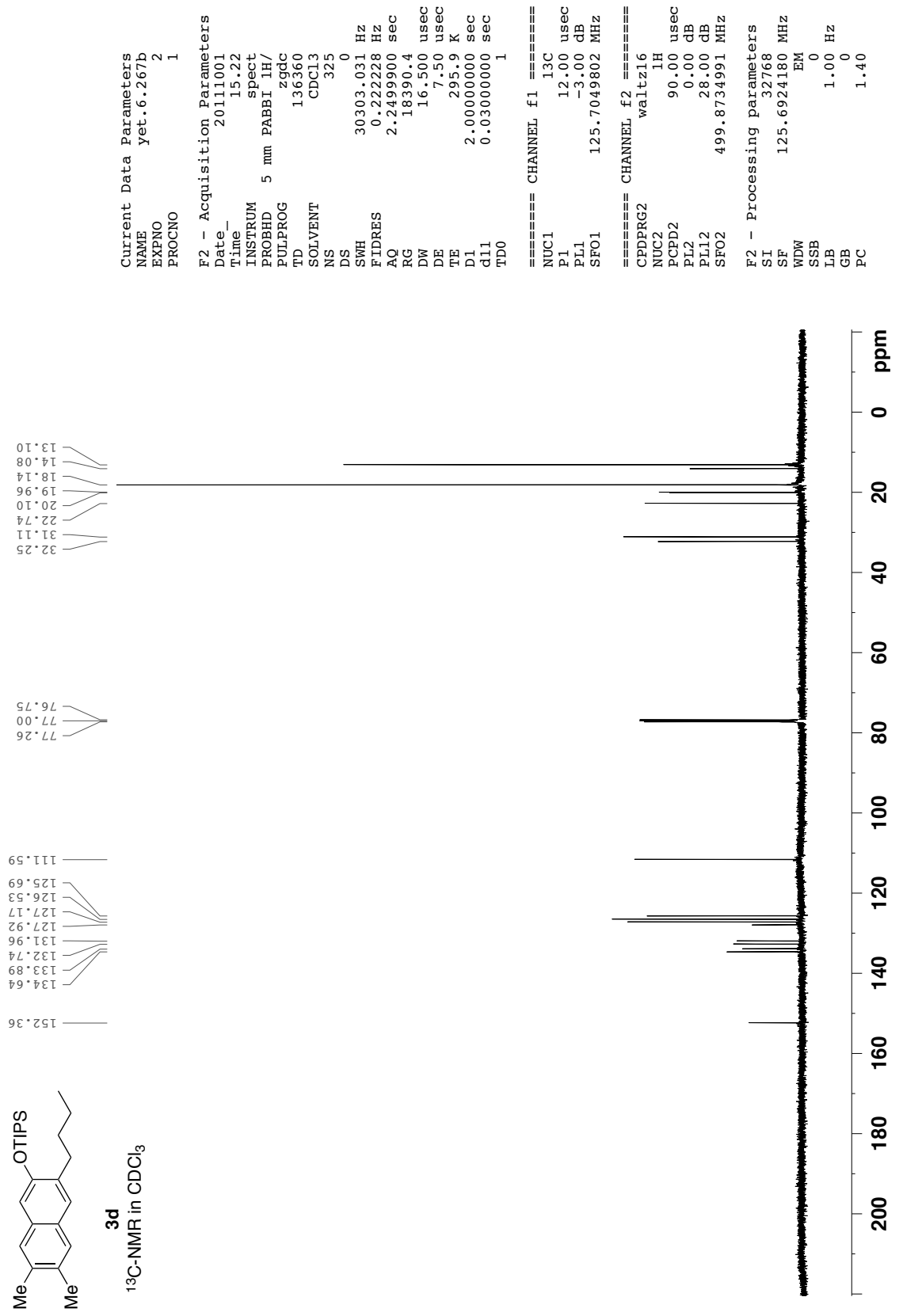

OTIPS
Cl
Cl
3c
^{1}H-NMR in $CDCl_3$
NAME yet.6.262b
EXPNO 1
PROCNO 1
Date_ 20110925
Time_ 0.05
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg
TD 48076
SOLVENT CDCl3
NS 4
DS 0
SWH 8012.820 Hz
FIDRES 0.166670 Hz
AQ 2.9999924 sec
RG 32
DW 62.400 usec
DE 4.50 usec
TE 300.0 K
D1 10.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 0.00 dB
SFO1 500.1325006 MHz
SI 65536
SF 500.1300183 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.774
7.726
7.454
7.250
6.983
2.763
2.748
2.732
1.670
1.655
1.651
1.639
1.634
1.624
1.608
1.448
1.433
1.418
1.401
1.386
1.371
1.356
1.342
1.150
ppm
1 2 3 4 5 6 7 8 9 10
3.24
19.19
5.30
2.06
2.06
1.02
1.02
1.02
1.00

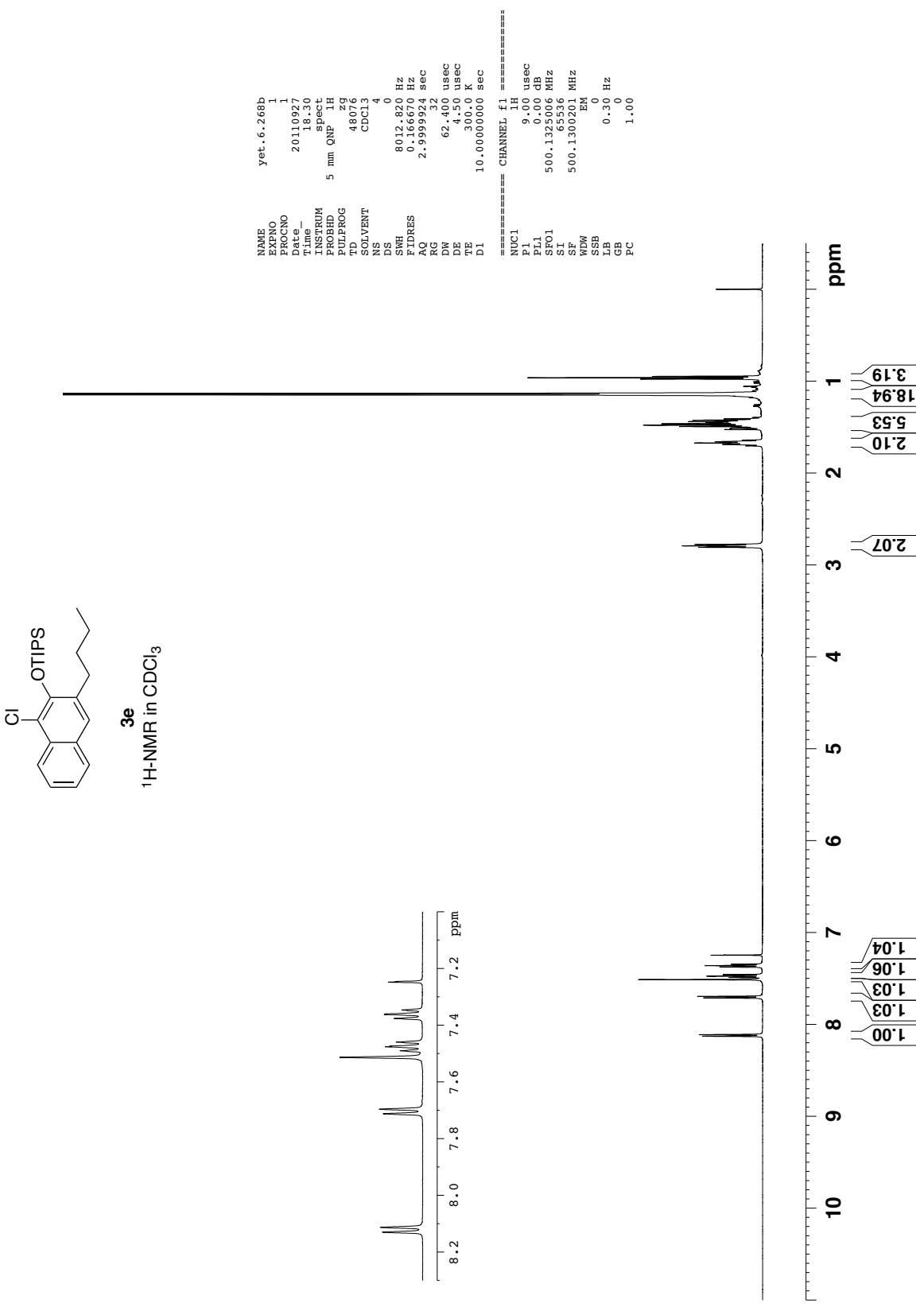


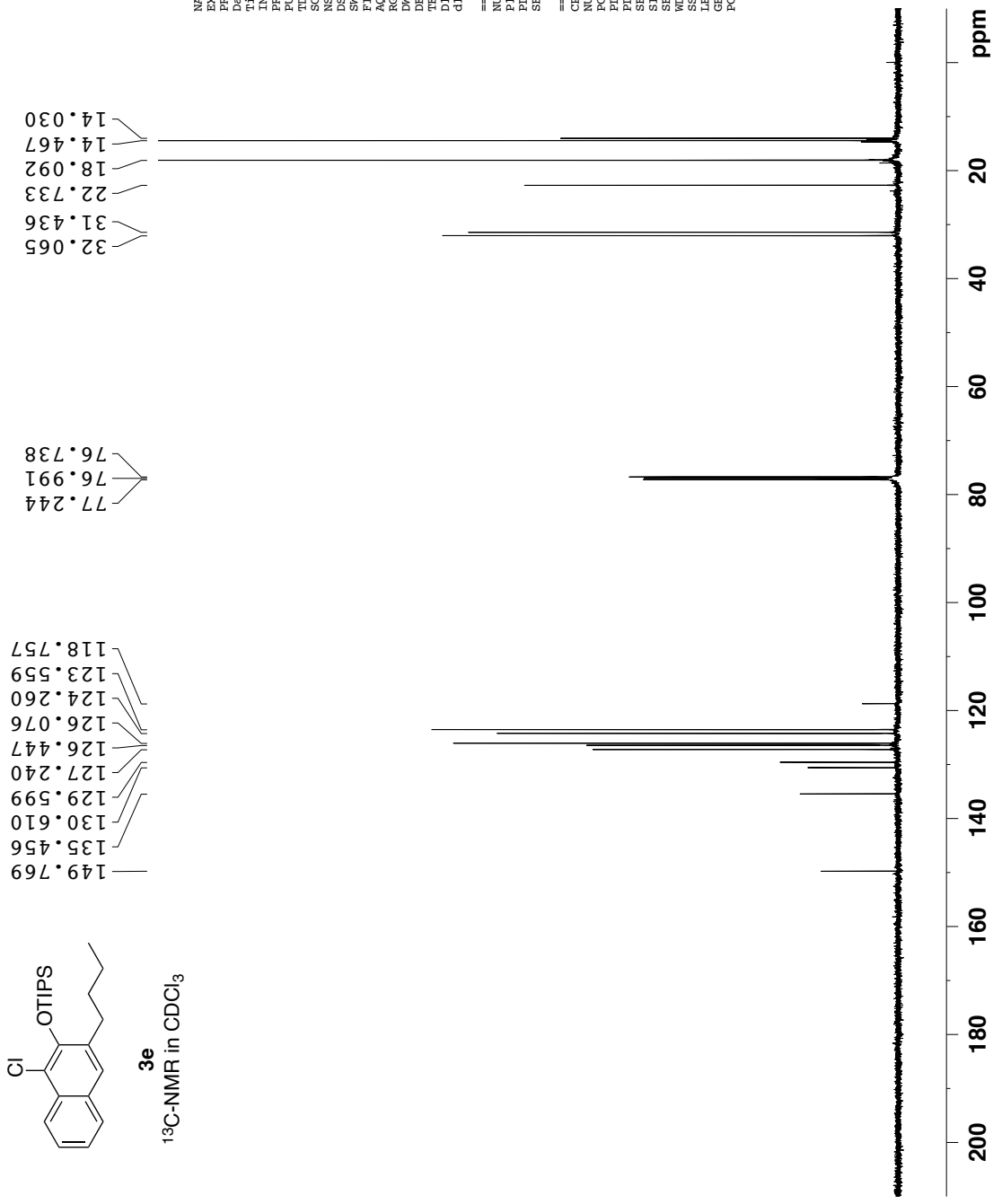

```
NAME                yet.6.262b
EXPNO                        2
PROCNO                       1
Date_                20110925
Time                     0.09
INSTRUM                  spect
PROBHD     5 mm QNP  1H
PULPROG                  zgdc
TD                      75184
SOLVENT                 C6D6
NS                       996
DS                         0
SWH                37593.984 Hz
FIDRES             0.500026 Hz
AQ                 0.9999972 sec
RG                        8192
DW                      13.300 usec
DE                        7.50 usec
TE                       300.0 K
D1                 0.10000000 sec
d11                0.03000000 sec
============== CHANNEL f1 ==============
NUC1                       13C
P1                        4.60 usec
PL1                       0.00 dB
SFO1              125.7690572 MHz
============== CHANNEL f2 ==============
CPDPRG2                 waltz16
NUC2                       1H
PCPD2                    90.00 usec
PL2                      120.00 dB
PL12                      19.00 dB
SFO2              500.1320005 MHz
SI                        32768
SF                125.7577918 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```

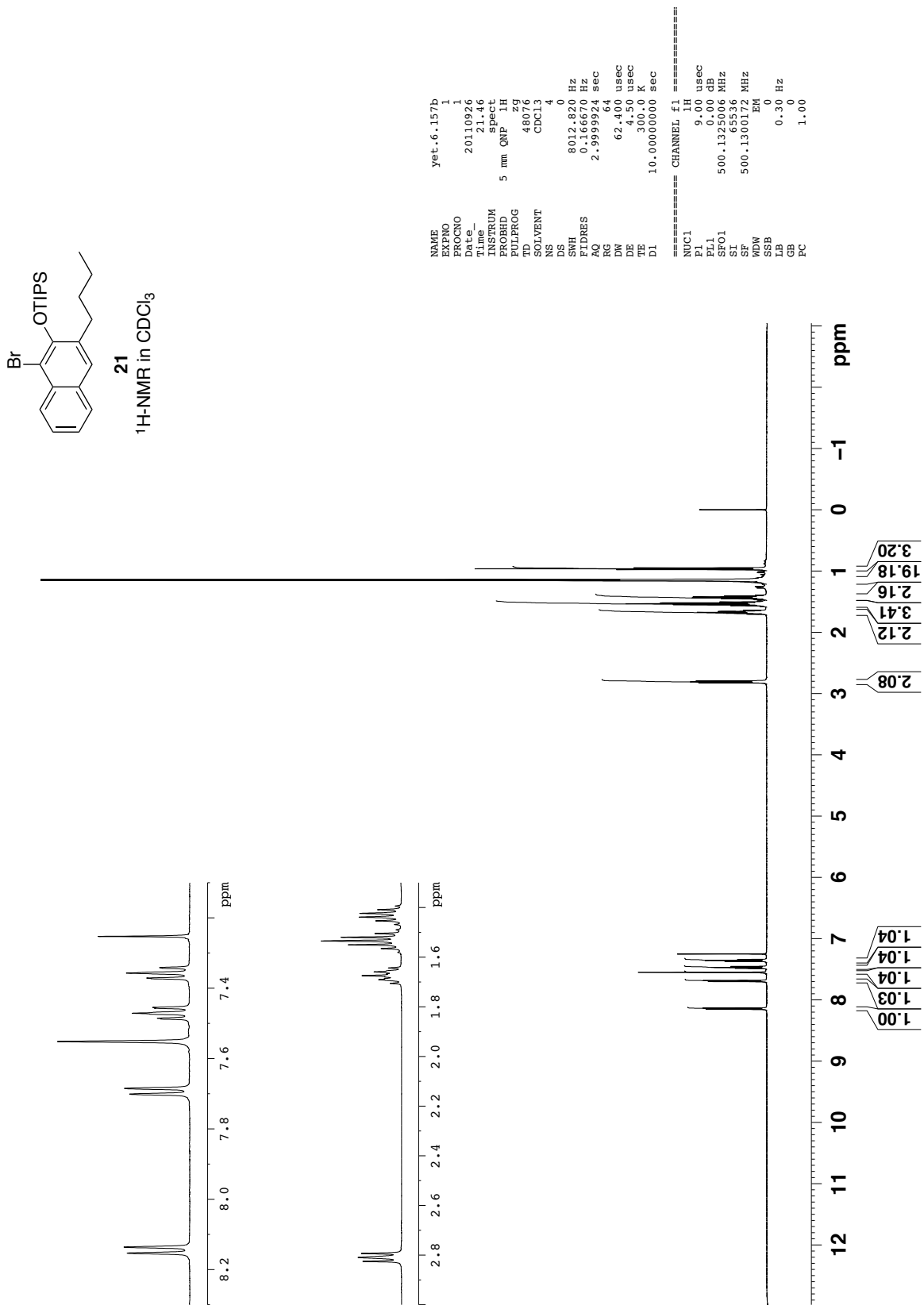

OTIPS
Cl
Cl
3c
^{13}C-NMR in CD_2Cl_2
13.62
14.36
18.42
23.31
31.61
32.56
53.79
54.00
54.22
111.86
127.41
127.46
127.74
128.44
128.63
129.52
132.92
137.44
154.76
ppm
NAME yet.6.264a
EXPNO 2
PROCNO 1
Date_ 20120128
Time_ 15.09
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgdc
TD 142854
SOLVENT CD2Cl2
NS 457
DS 0
SWH 30303.031 Hz
FIDRES 0.212126 Hz
AQ 2.3571410 se
RG 18390.4
DW 16.500 us
DE 7.50 us
TE 295.5 K
D1 2.00000000 se
d11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
NUC1 13C
P1 8.50 us
PL1 0.00 dB
SFO1 125.7049802 MH
======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 us
PL2 1.00 dB
PL12 21.00 dB
SFO2 499.8734991 MH
SI 32768
SF 125.6923404 MH
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

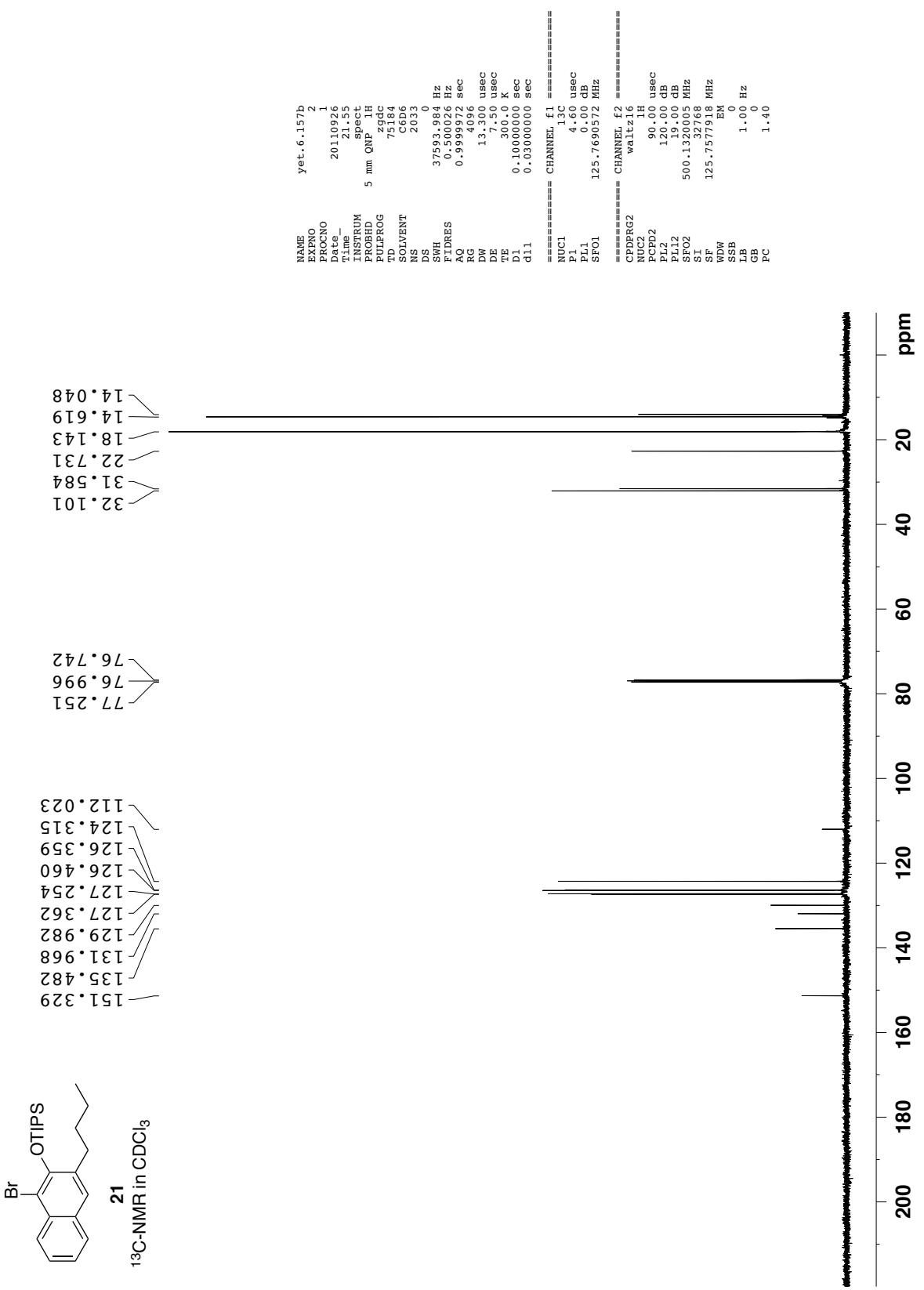

Current Data Parameters
NAME yet.6.267b
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111001
Time_ 15.15
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 4
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 sec
RG 28.5
DW 50.000 usec
DE 7.50 usec
TE 295.7 K
D1 10.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.70 usec
PL1 0.00 dB
SFO1 499.8729992 MHz
F2 - Processing parameters
SI 32768
SF 499.8700270 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7.441
7.387
7.242
6.994
2.756
2.740
2.724
2.372
2.364
1.671
1.657
1.653
1.645
1.641
1.635
1.626
1.610
1.440
1.425
1.410
1.397
1.383
1.368
1.353
1.98
0.99
1.00
1.97
5.98
1.99
5.04
18.14
3.00
ppm
OTIPS
Me
Me
3d
^{1}H-NMR in $CDCl_3$


Current Data Parameters
NAME yet.6.267b
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111001
Time 15.22
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgdc
TD 136360
SOLVENT CDCl3
NS 325
DS 0
SWH 30303.031 Hz
FIDRES 0.222228 Hz
AQ 2.2499900 sec
RG 18390.4
DW 16.500 usec
DE 7.50 usec
TE 295.9 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 -3.00 dB
SFO1 125.7049802 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 28.00 dB
SFO2 499.8734991 MHz
F2 - Processing parameters
SI 32768
SF 125.6924180 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
152.36
134.64
133.89
132.74
131.96
127.92
127.17
126.53
125.69
111.59
77.26
77.00
76.75
32.25
31.11
22.74
20.10
19.96
18.14
14.08
13.10
200
180
160
140
120
100
80
60
40
20
0
ppm
OTIPS
Me
Me
3d
^{13}C-NMR in $CDCl_3$


Cl
OTIPS
3e
^{1}H-NMR in $CDCl_3$
NAME yet.6.268b
EXPNO 1
PROCNO 1
Date_ 20110927
Time 18.30
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg
TD 48076
SOLVENT CDCl3
NS 4
DS 0
SWH 8012.820 Hz
FIDRES 0.166670 Hz
AQ 2.9999924 sec
RG 32
DW 62.400 usec
DE 4.50 usec
TE 300.0 K
D1 10.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 0.00 dB
SFO1 500.1325006 MHz
SI 65536
SF 500.1300201 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
1.00
1.03
1.03
1.06
1.04
2.07
2.10
5.53
18.94
3.19


14.030
14.467
18.092
22.733
31.436
32.065
76.738
76.991
77.244
118.757
123.559
124.260
126.076
126.447
127.240
129.599
130.610
135.456
149.769
ppm
20
40
60
80
100
120
140
160
180
200
Cl
OTIPS
3e
13C-NMR in CDCl3

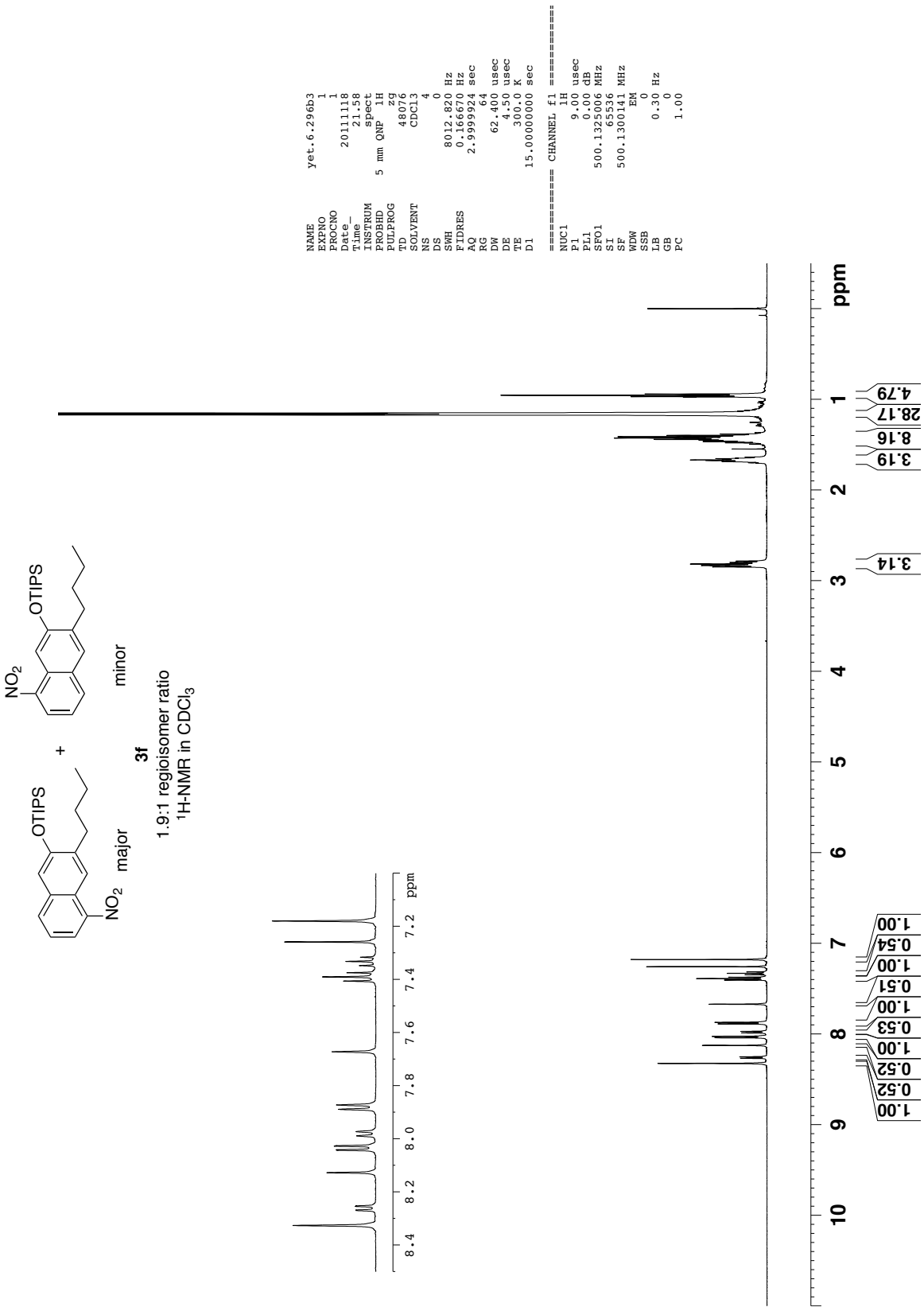

Br
OTIPS
21
^{1}H-NMR in $CDCl_3$
NAME yet.6.157b
EXPNO 1
PROCNO 1
Date_ 20110926
Time_ 21.46
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg
TD 48076
SOLVENT CDCl3
NS 4
DS 0
SWH 8012.820 Hz
FIDRES 0.166670 Hz
AQ 2.9999924 sec
RG 64
DW 62.400 usec
DE 4.50 usec
TE 300.0 K
D1 10.00000000 sec
CHANNEL f1
NUC1 1H
P1 9.00 usec
PL1 0.00 dB
SFO1 500.1325006 MHz
SI 65536
SF 500.1300172 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
3.20
19.18
2.16
3.41
2.12
2.08
1.00
1.03
1.04
1.04
1.04

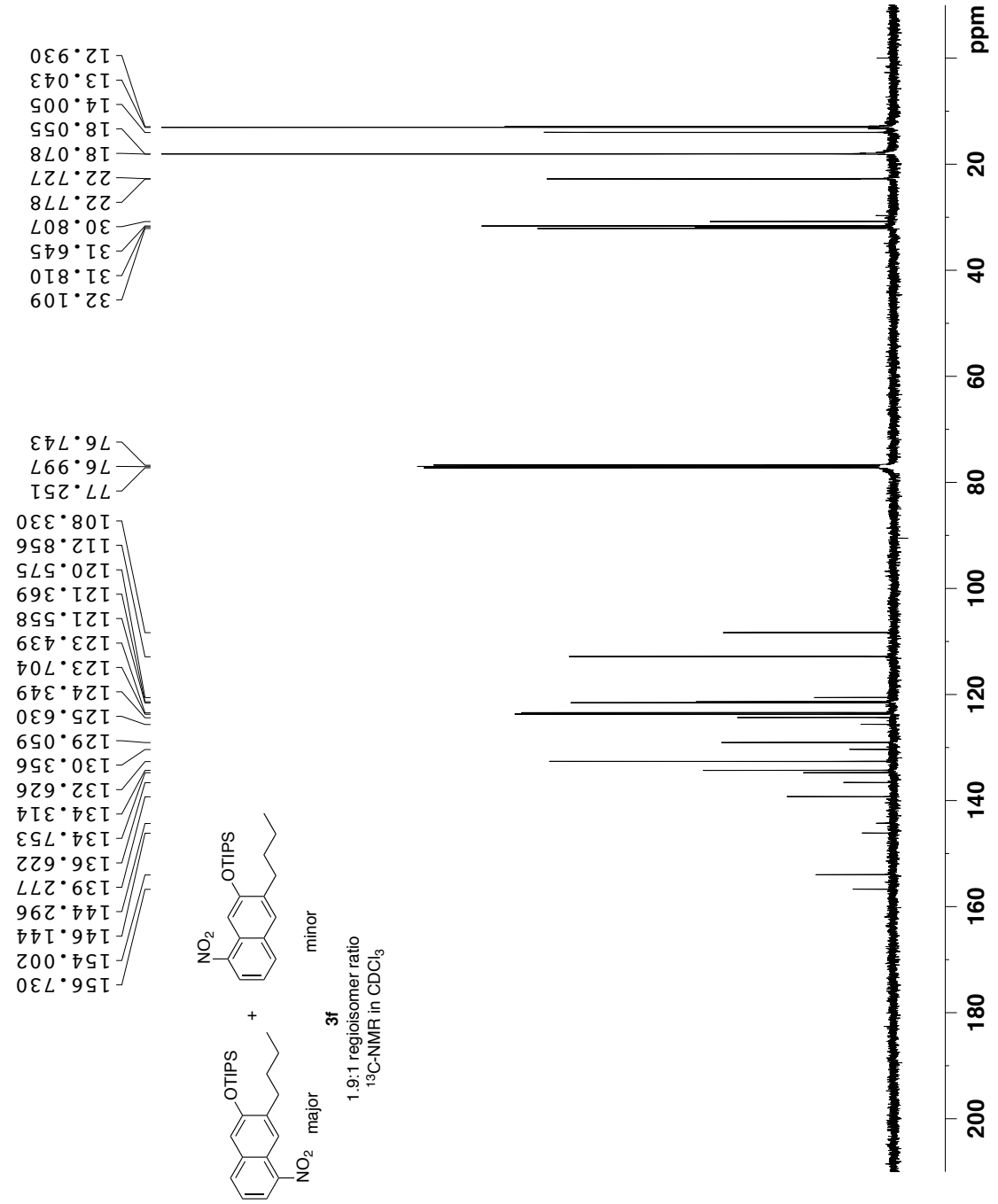



```
NAME                yet.6.157b
EXPNO                        2
PROCNO                       1
Date_                20110926
Time                    21.55
INSTRUM                 spect
PROBHD   5 mm QNP  1H
PULPROG                  zgdc
TD                      75184
SOLVENT                 C6D6
NS                       2033
DS                          0
SWH                37593.984 Hz
FIDRES                 0.500026 Hz
AQ                     0.999997 sec
RG                        4096
DW                      13.300 usec
DE                       7.50 usec
TE                      300.0 K
D1                  0.10000000 sec
d11                 0.03000000 sec

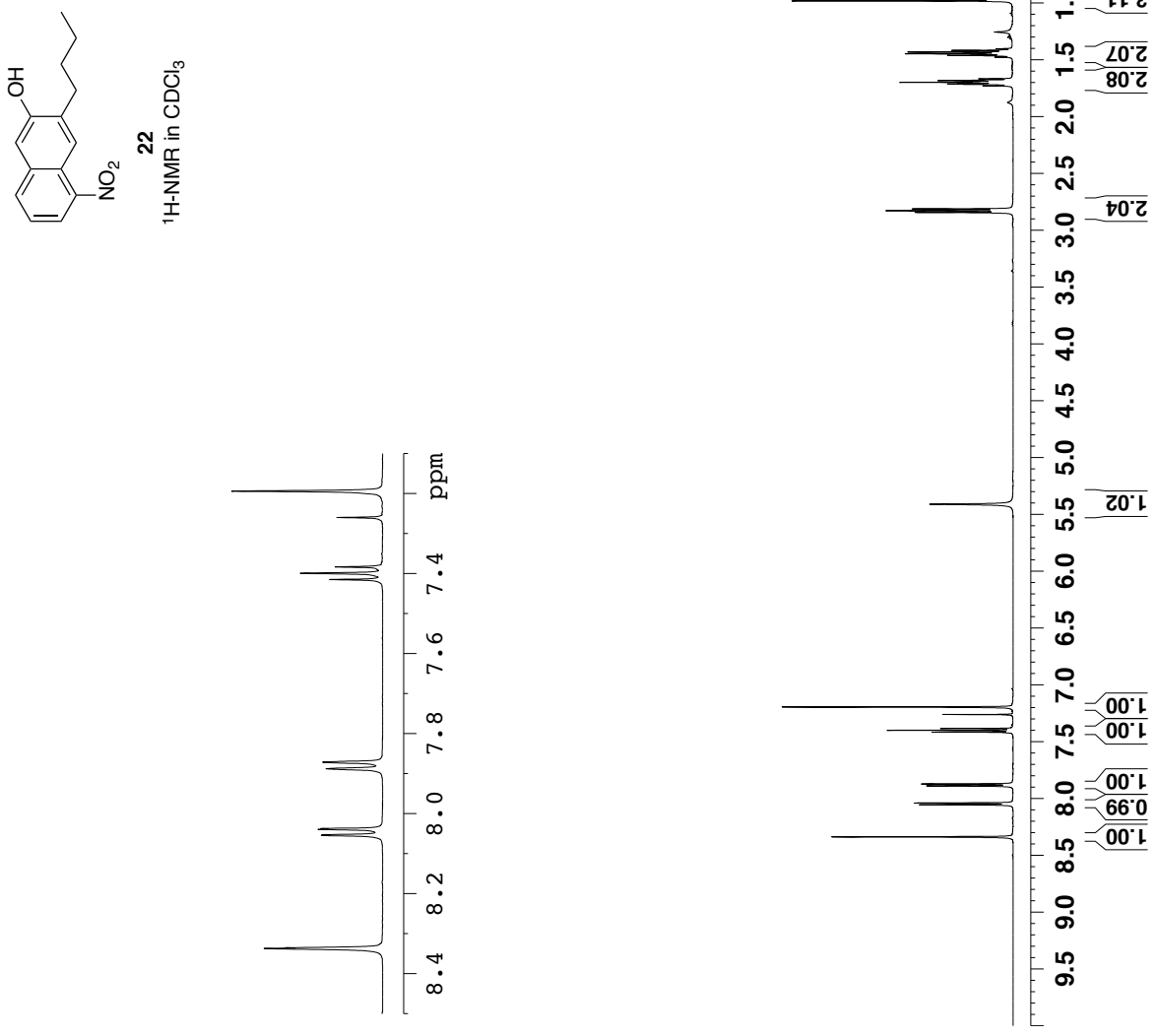
======== CHANNEL f1 ========
NUC1                       13C
P1                        4.60 usec
PL1                       0.00 dB
SFO1               125.7690572 MHz

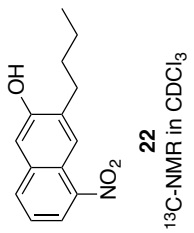
======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                         1H
PCPD2                     90.00 usec
PL2                      120.00 dB
PL12                      19.00 dB
SFO2               500.1320005 MHz
SI                        32768
SF                 125.7577918 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```


OTIPS
NO2
major
+
OTIPS
NO2
minor
3f
1.9:1 regioisomer ratio
1H-NMR in CDCl3
NAME yet.6.296b3
EXPNO 1
PROCNO 1
Date_ 20111118
Time_ 21.58
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg
TD 48076
SOLVENT CDCl3
NS 4
DS 0
SWH 8012.820 Hz
FIDRES 0.166670 Hz
AQ 2.9999924 sec
RG 64
DW 62.400 usec
DE 4.50 usec
TE 300.0 K
D1 15.00000000 sec
CHANNEL f1
NUC1 1H
P1 9.00 usec
PL1 0.00 dB
SFO1 500.1325006 MHz
SI 65536
SF 500.1300141 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
4.79
28.17
8.16
3.19
3.14
1.00
0.54
1.00
0.51
1.00
0.53
1.00
0.52
0.52
1.00




```
NAME                yet.6.296b3
EXPNO                         2
PROCNO                        1
Date_              20111118
Time_                    22.18
INSTRUM                  spect
PROBHD     5 mm QNP  1H
PULPROG                  zgdc
TD                      75184
SOLVENT                 CDCl3
NS                       2981
DS                          0
SWH                37593.984 Hz
FIDRES                 0.500026 Hz
AQ                     0.999997 sec
RG                       8192
DW                     13.300 usec
DE                      7.50 usec
TE                     300.0 K
D1                     0.10000000 sec
d11                    0.03000000 sec
========== CHANNEL f1 ==========
NUC1                       13C
P1                        4.60 usec
PL1                       0.00 dB
SFO1               125.7690572 MHz
========== CHANNEL f2 ==========
CPDPRG2                 waltz16
NUC2                         1H
PCPD2                    90.00 usec
PL2                     120.00 dB
PL12                     19.00 dB
SFO2           500.1320005 MHz
SI                       32768
SF             125.7577918 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```


OH
NO2
22
1H-NMR in CDCl3
NAME yet.7.284b1
EXPNO 1
PROCNO 1
Date_ 20120421
Time_ 14.21
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 4
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 sec
RG 31.72
DW 50.000 usec
DE 10.00 usec
TE 294.9 K
D1 15.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.00 usec
SI 65536
SF 500.1300131 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
ppm
3.11
2.07
2.08
2.04
1.02
1.00
1.00
0.99
1.00

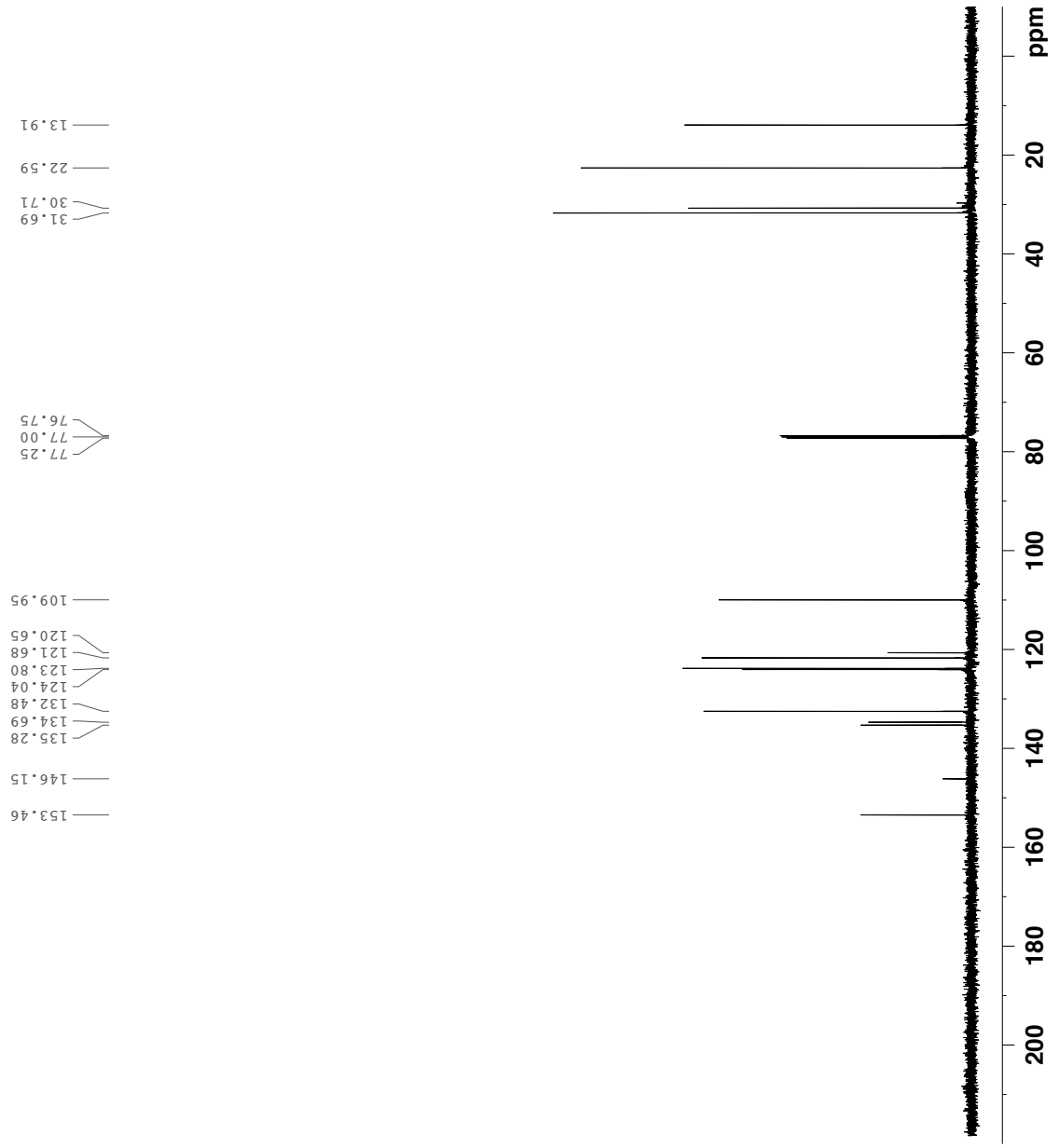


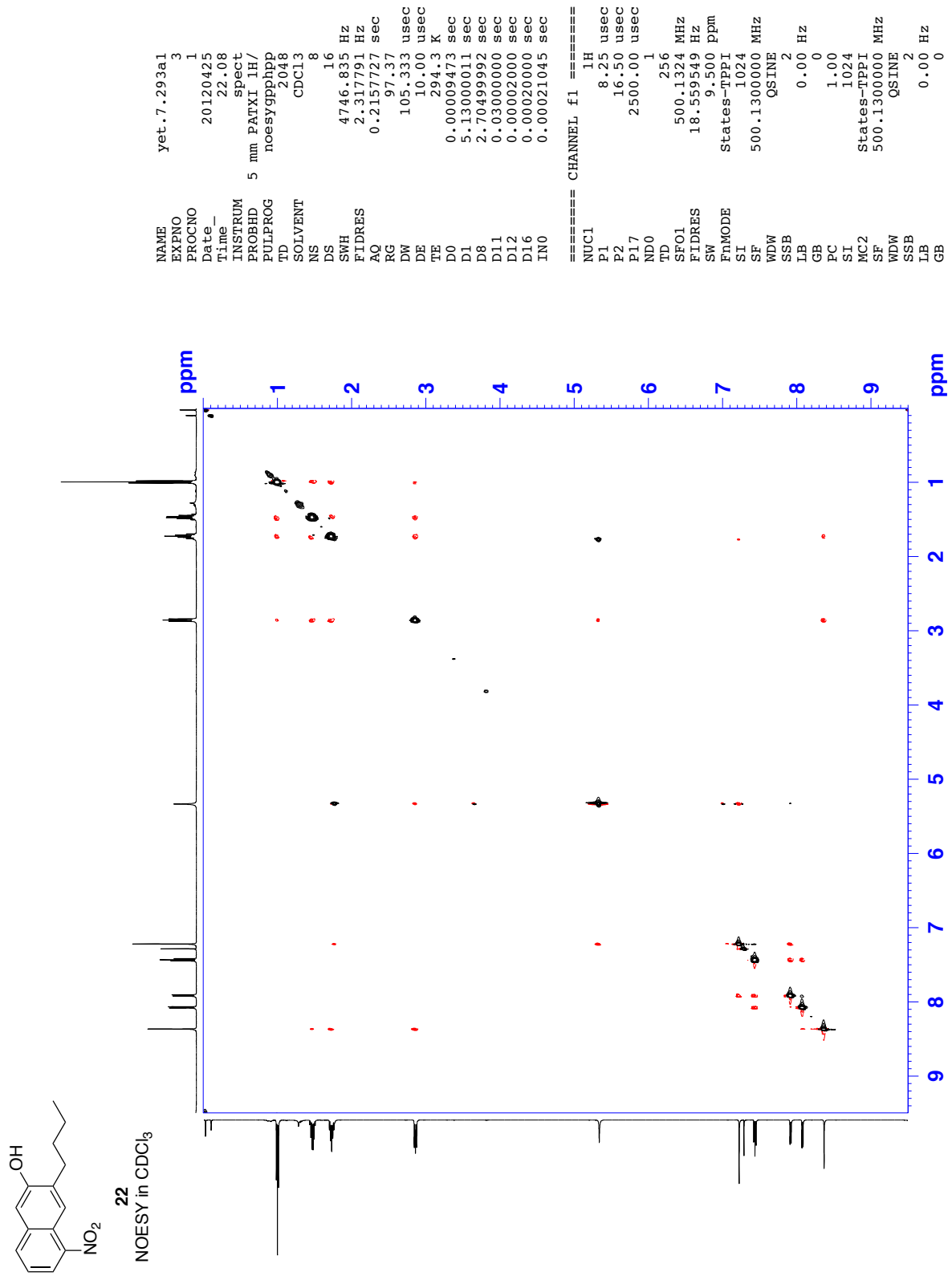
22

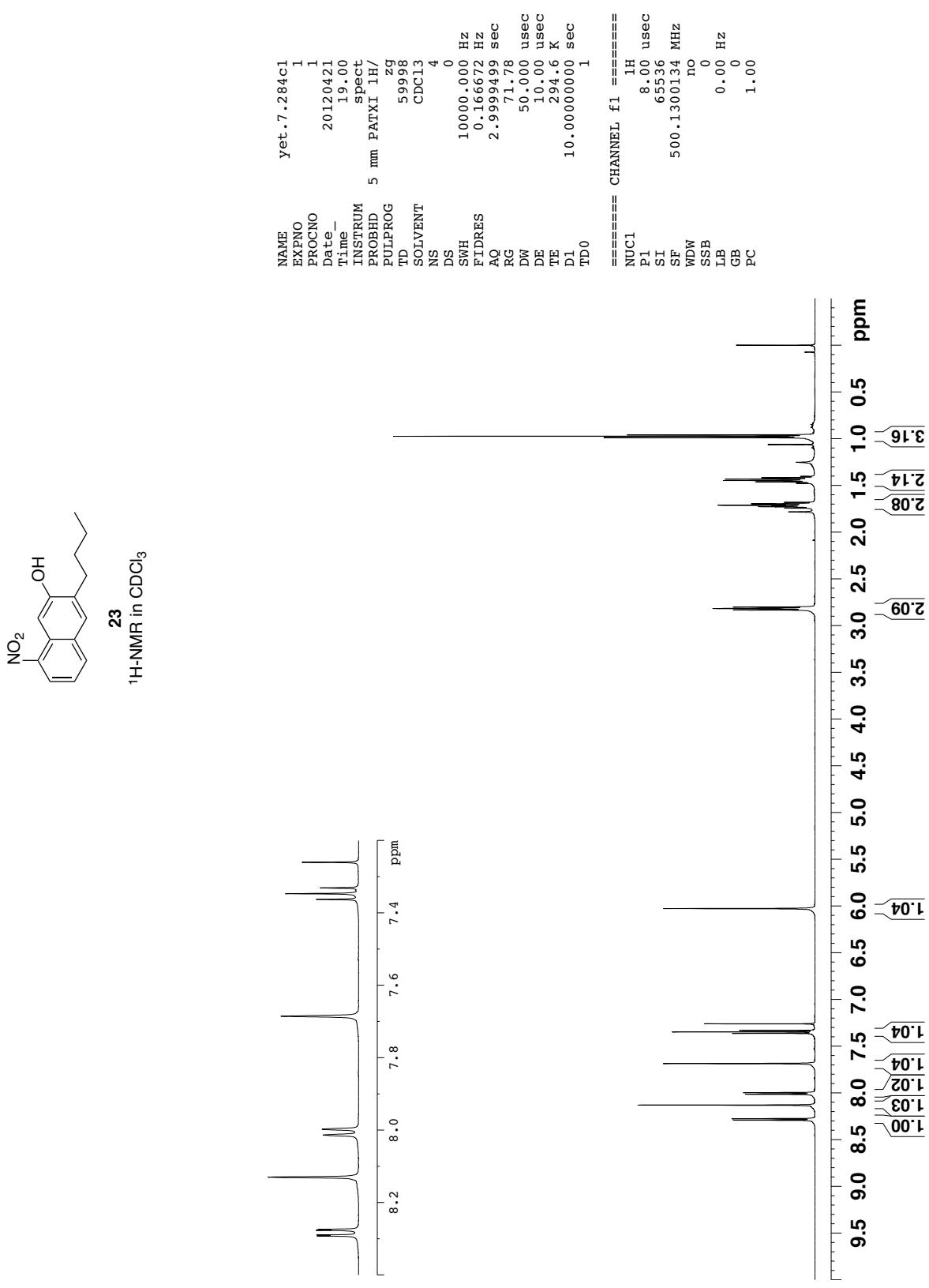
^{13}C-NMR in $CDCl_3$

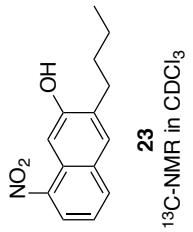
```
NAME                yet.7.284b
EXPNO                        2
PROCNO                       1
Date_                 20120420
Time_                    19.27
INSTRUM                  spect
PROBHD   5 mm PATXI 1H/
PULPROG                   zgdc
TD                      178568
SOLVENT                 CDCl3
NS                         395
DS                           0
SWH                  29761.904 Hz
FIDRES                0.166670 Hz
AQ                    2.9999924 sec
RG                       196.79
DW                       16.800 usec
DE                       10.00 usec
TE                       296.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        13C
P1                        14.00 usec
SI                        131072
SF                   125.7577942 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```




NAME yet.7.293a1
EXPNO 3
PROCNO 1
Date_ 20120425
Time_ 22.08
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG noesygpphpp
TD 2048
SOLVENT CDCl3
NS 8
DS 16
SWH 4746.835 Hz
FIDRES 2.317791 Hz
AQ 0.2157727 sec
RG 97.37
DW 105.333 usec
DE 10.00 usec
TE 294.3 K
D0 0.00009473 sec
D1 5.13000011 sec
D8 2.70499992 sec
D11 0.03000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00021045 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.25 usec
P2 16.50 usec
P17 2500.00 usec
ND0 1
TD 256
SFO1 500.1324 MHz
FIDRES 18.559549 Hz
SW 9.500 ppm
FnMODE States-TPPI
SI 1024
SF 500.1300000 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.00
SI 1024
MC2 States-TPPI
SF 500.1300000 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
ppm
ppm
OH
NO2
22
NOESY in CDCl3


OH
NO2
23
1H-NMR in CDCl3
NAME yet.7.284c1
EXPNO 1
PROCNO 1
Date_ 20120421
Time_ 19.00
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg
TD 59998
SOLVENT CDCl3
NS 4
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 sec
RG 71.78
DW 50.000 usec
DE 10.00 usec
TE 294.6 K
D1 10.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.00 usec
SI 65536
SF 500.1300134 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
ppm
3.16
2.14
2.08
2.09
1.04
1.04
1.04
1.02
1.03
1.00

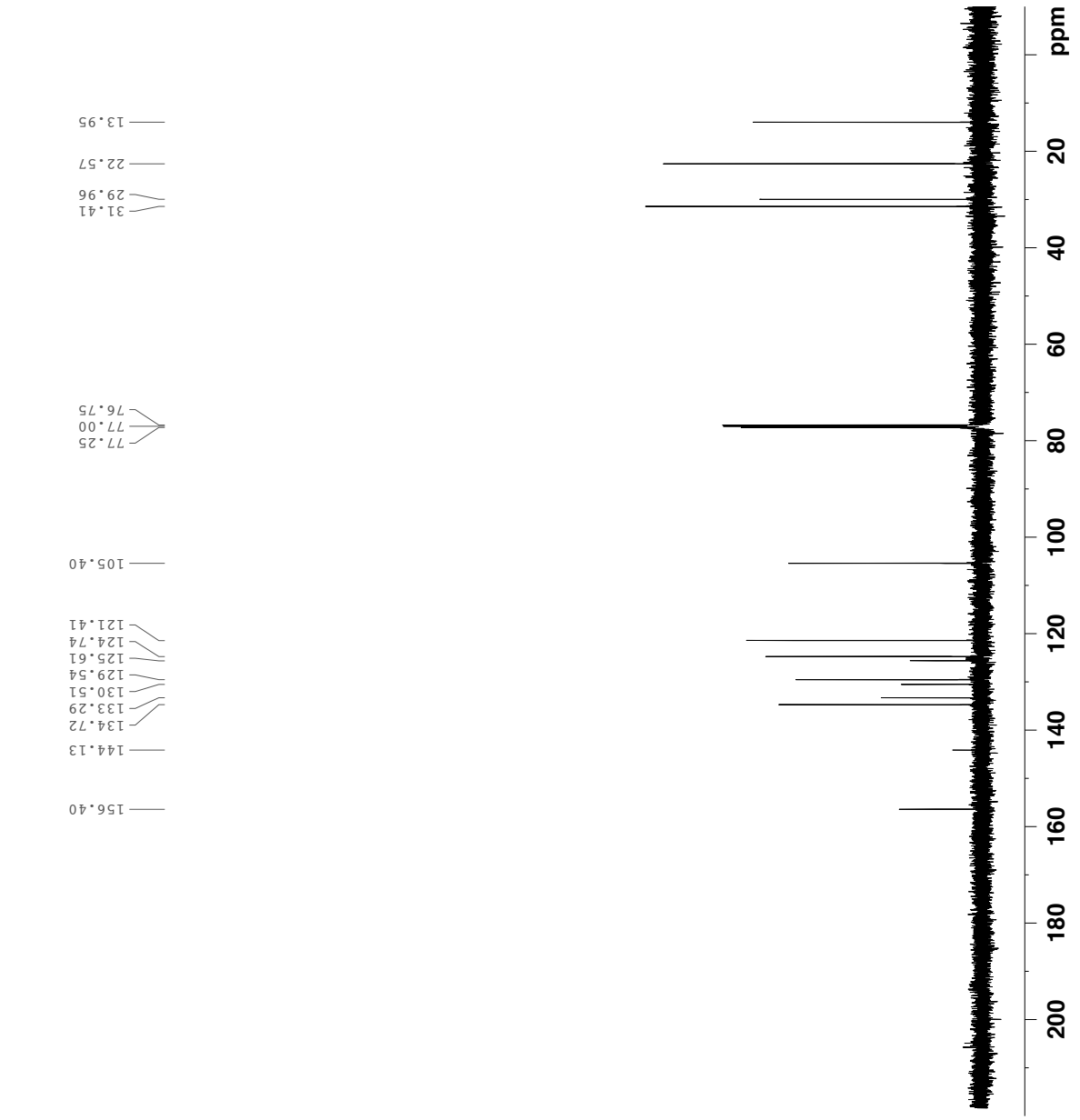


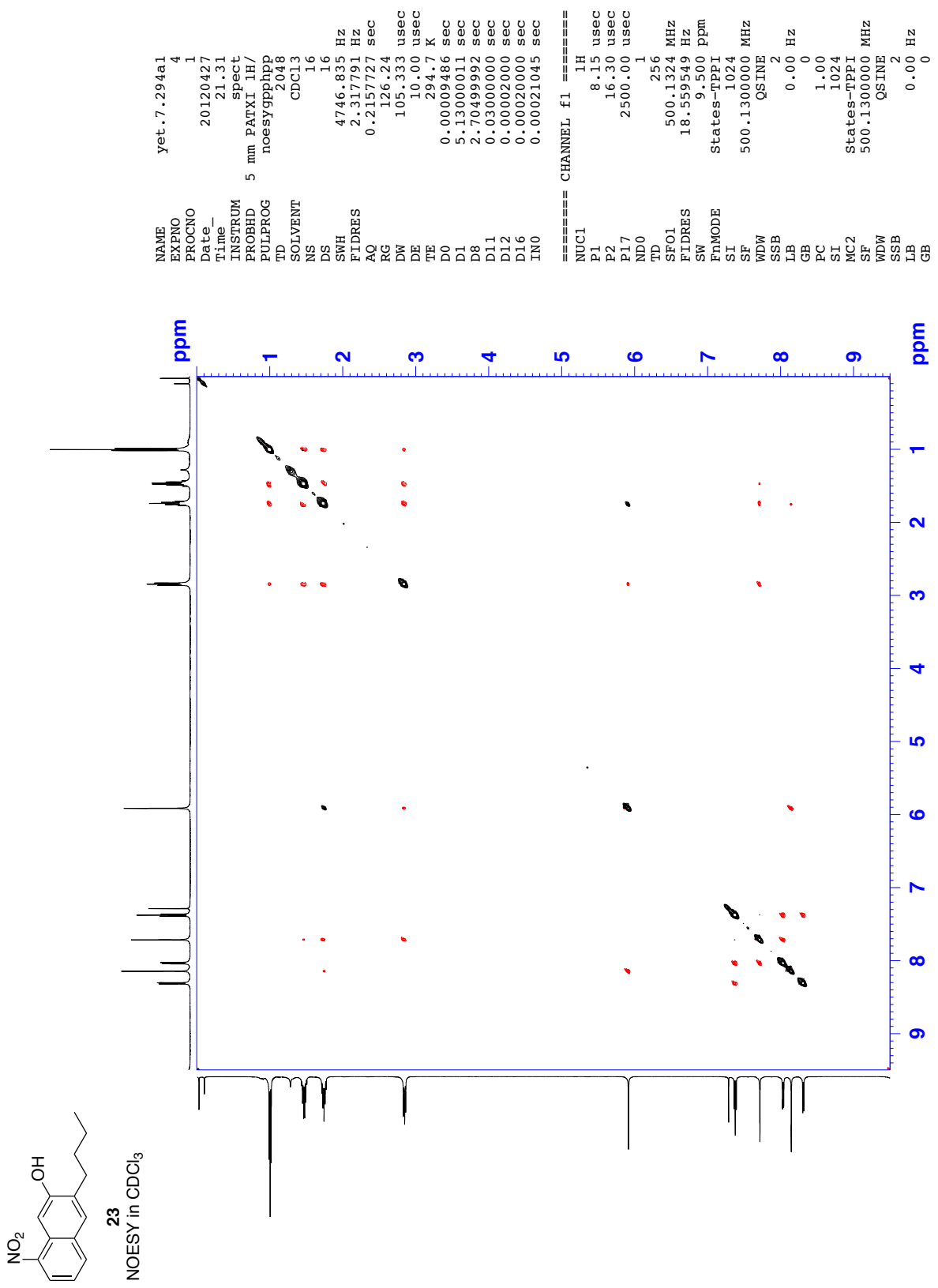
23

^{13}C-NMR in $CDCl_3$

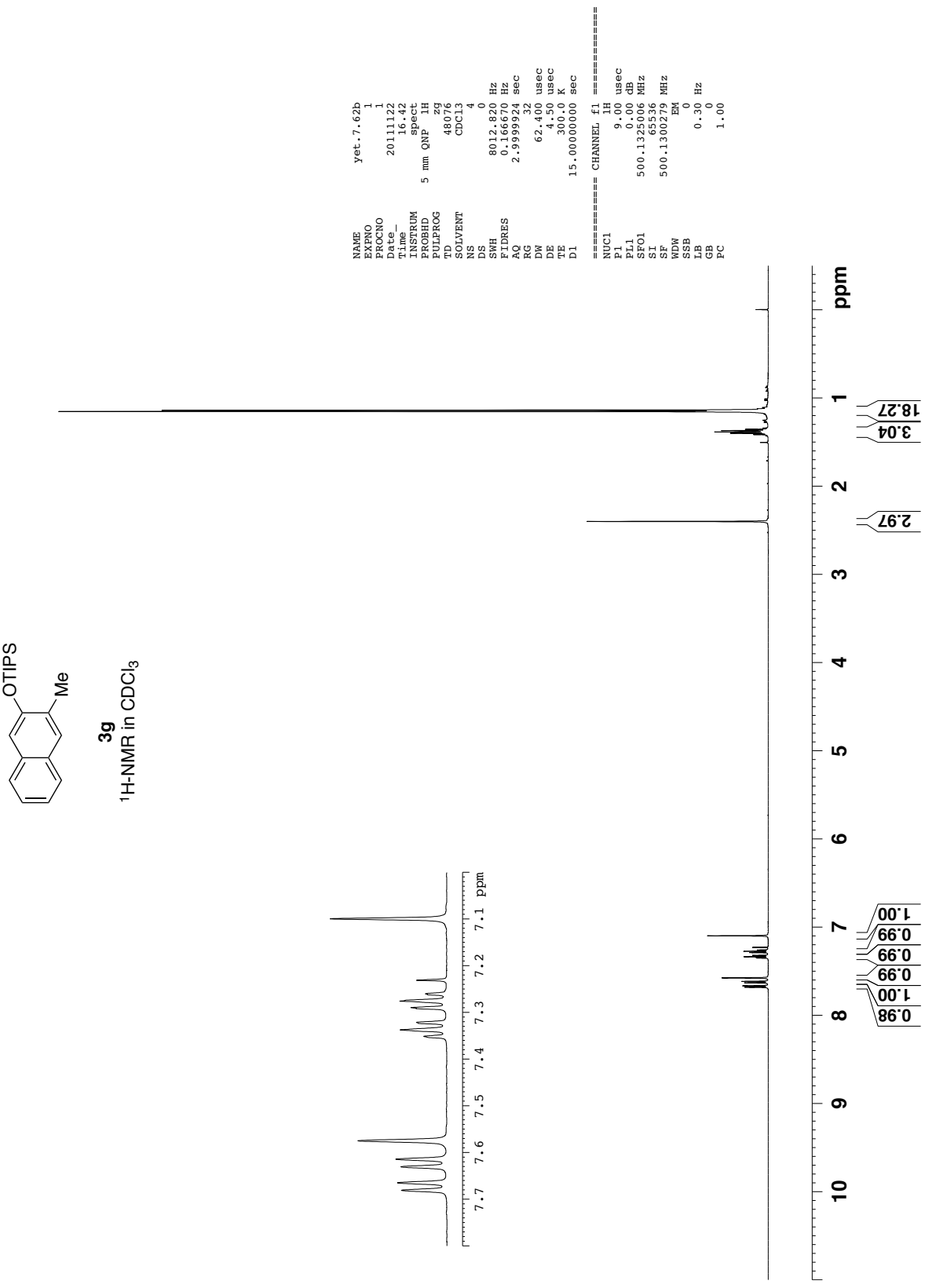
```
NAME          yet.7.284c1
EXPNO                   2
PROCNO                  1
Date_            20120421
Time_               19.14
INSTRUM             spect
PROBHD   5 mm PATXI 1H/
PULPROG               zgdc
TD                  178568
SOLVENT             CDCl3
NS                     401
DS                       0
SWH             29761.904 Hz
FIDRES          0.166670 Hz
AQ              2.9999924 sec
RG                   196.79
DW                   16.800 usec
DE                   10.00 usec
TE                   296.3 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

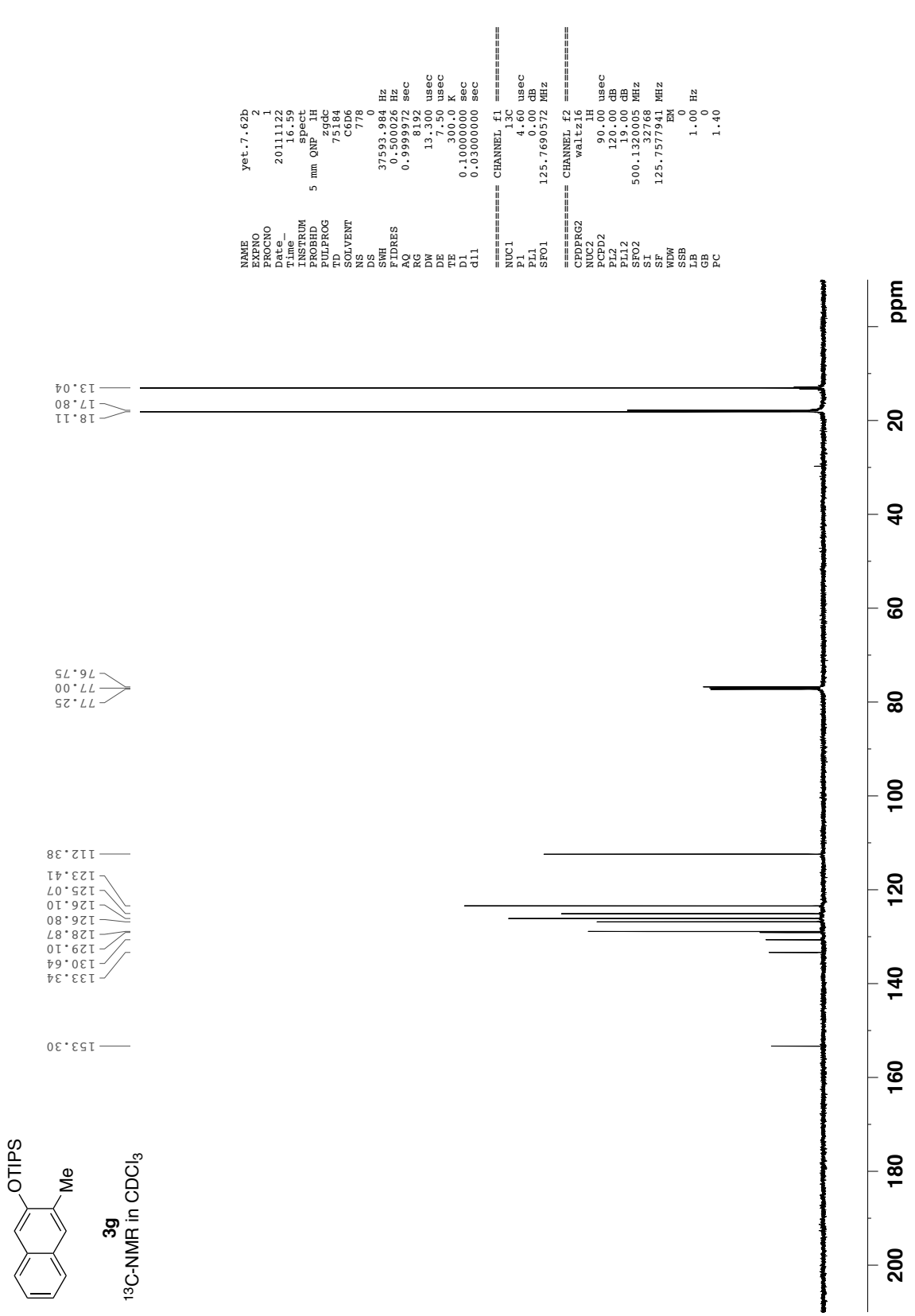
======== CHANNEL f1 ========
NUC1                   13C
P1                   14.00 usec
SI                   131072
SF              125.7577925 MHz
WDW                     EM
SSB                      0
LB                   1.00 Hz
GB                       0
PC                    1.40
```

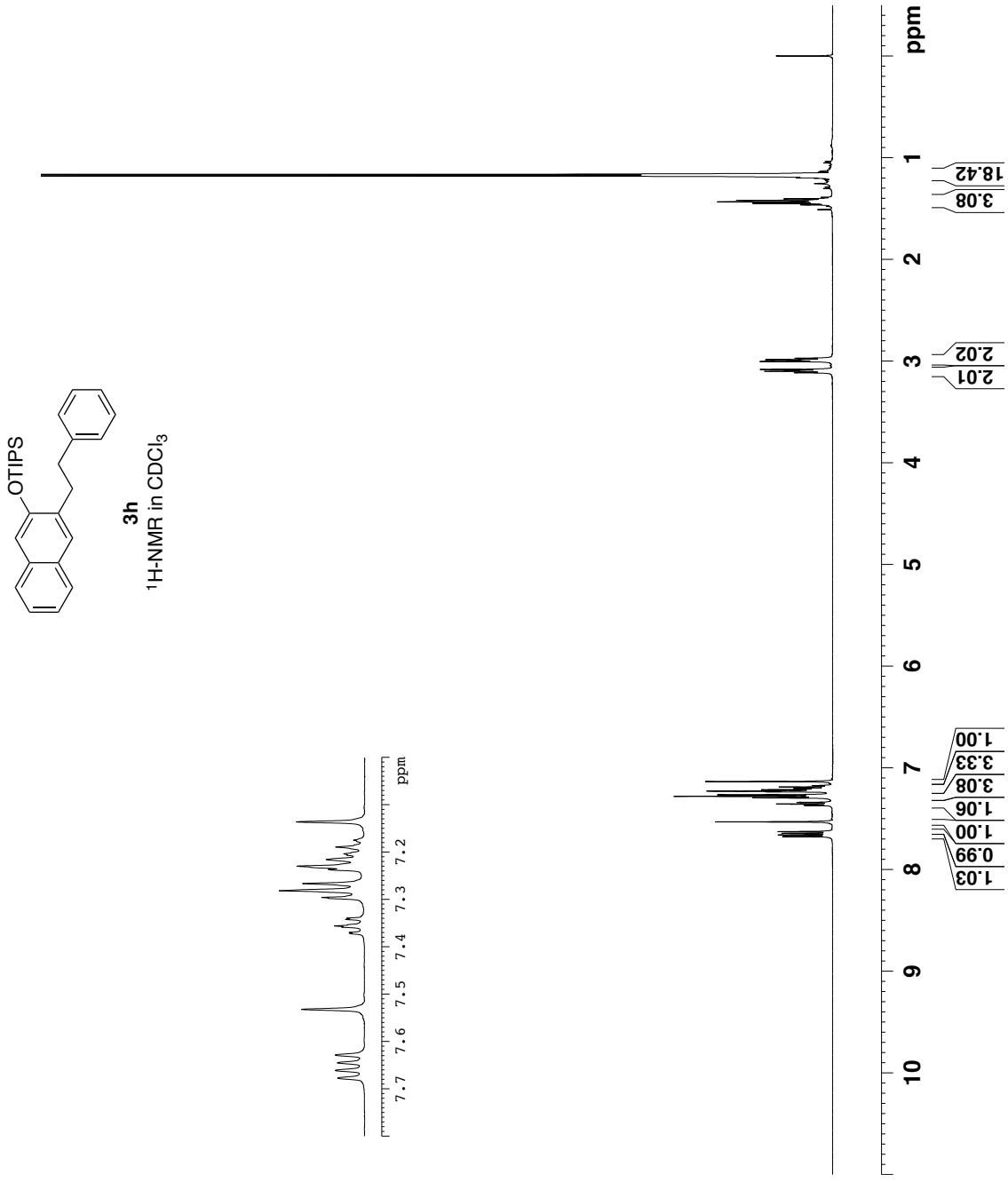


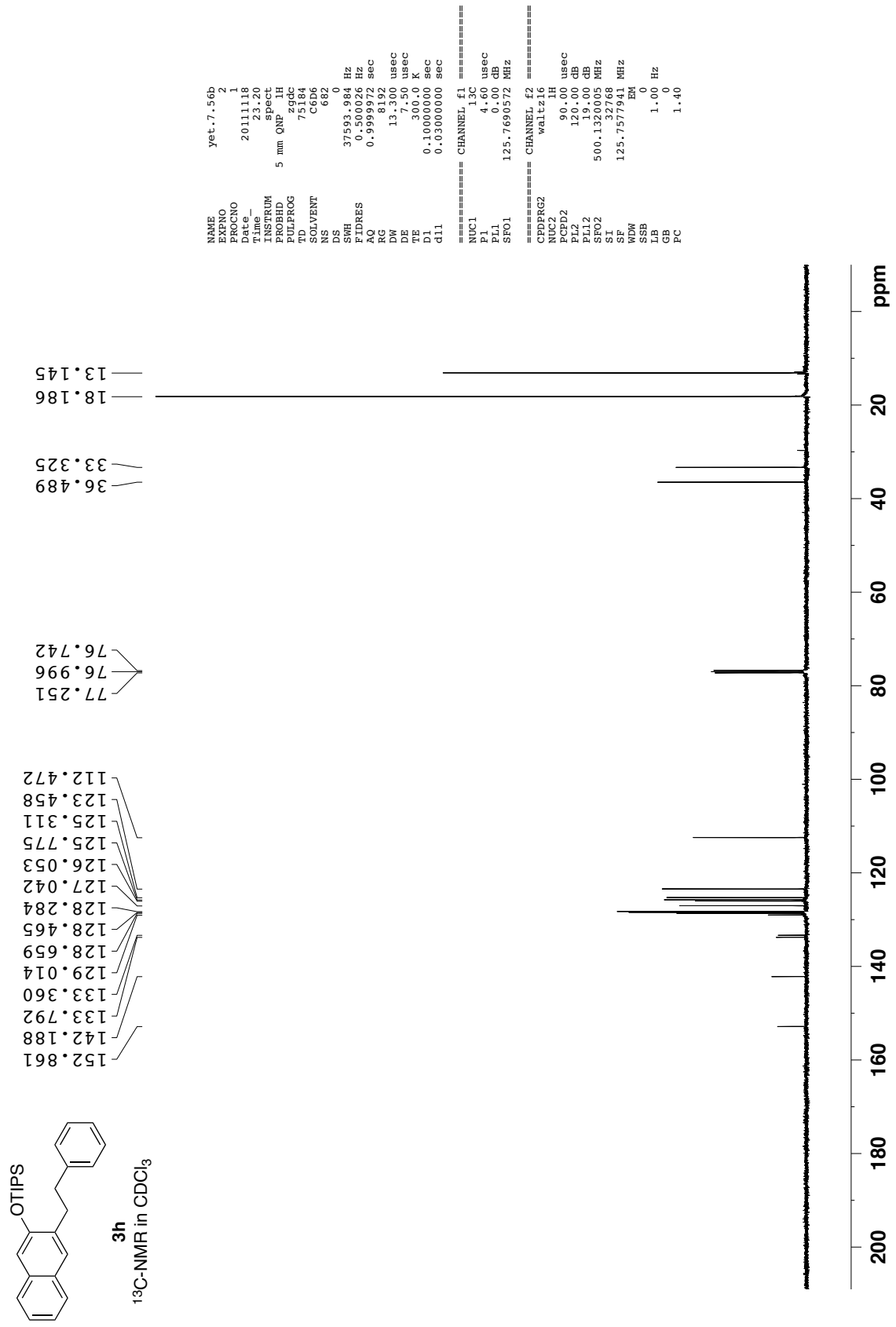


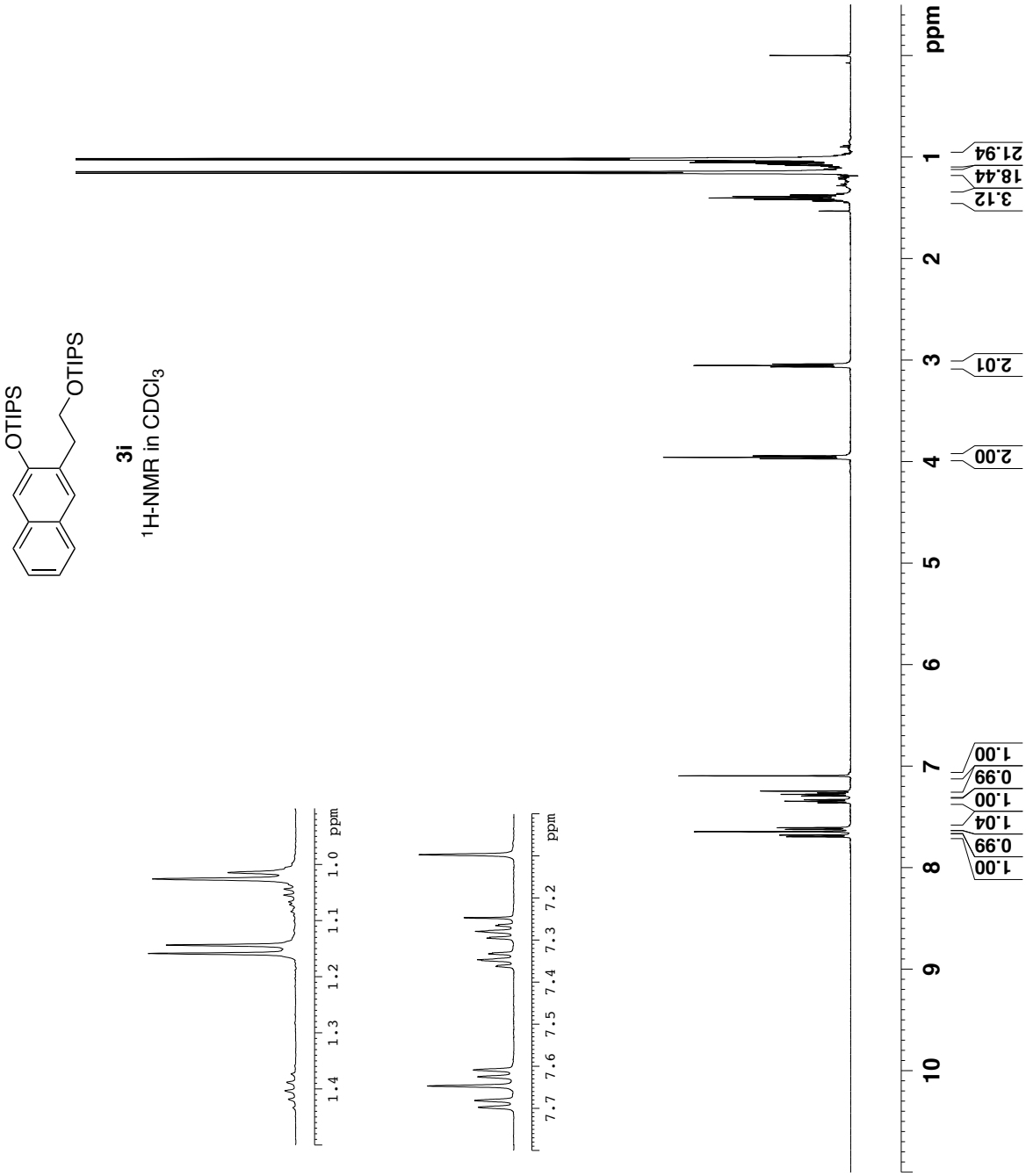
23
NOESY in $CDCl_3$

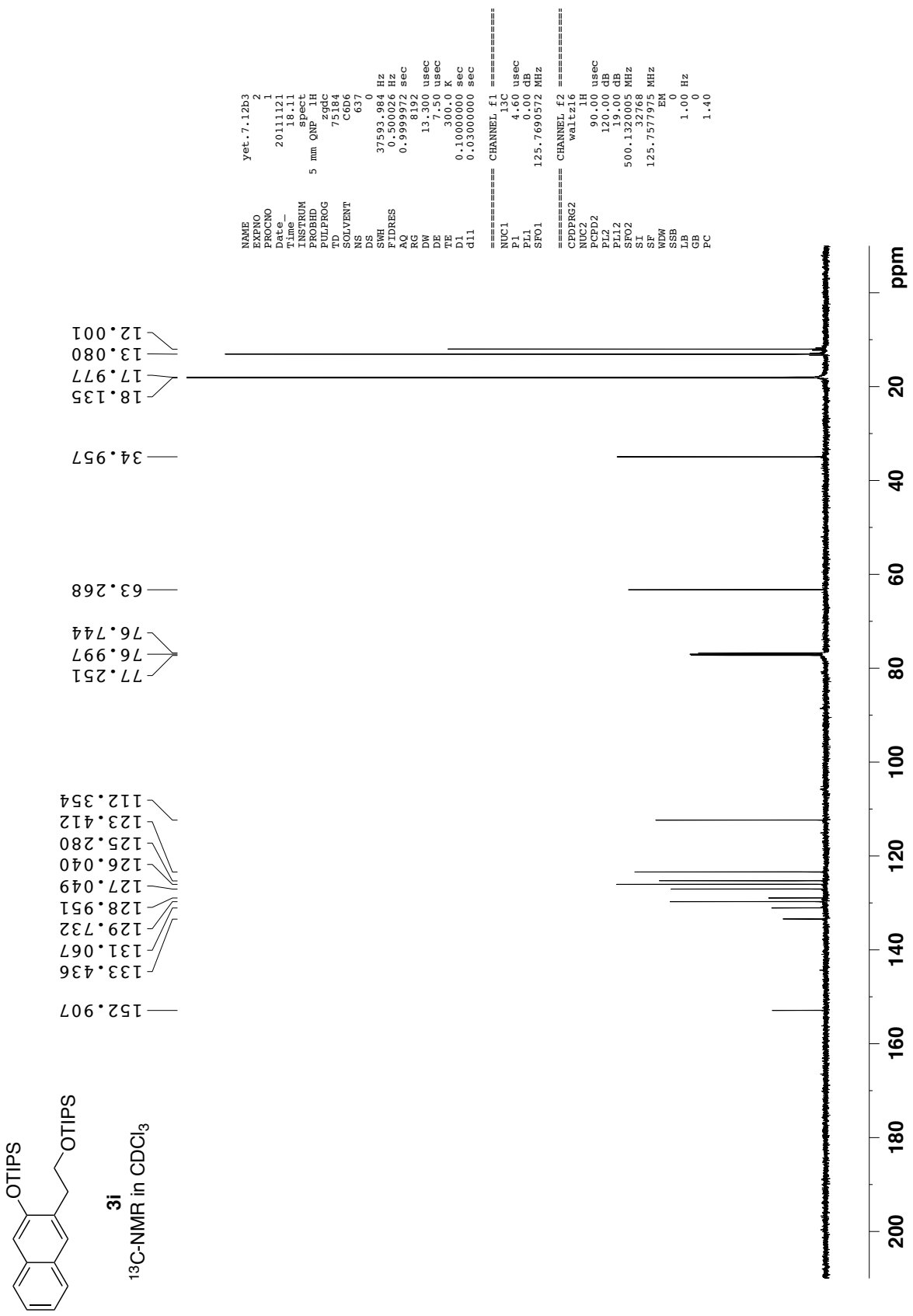

OTIPS
Me
3g
^{1}H-NMR in $CDCl_3$
NAME yet.7.62b
EXPNO 1
PROCNO 1
Date_ 20111122
Time_ 16.42
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg
TD 48076
SOLVENT CDCl3
NS 4
DS 0
SWH 8012.820 Hz
FIDRES 0.166670 Hz
AQ 2.9999924 sec
RG 32
DW 62.400 usec
DE 4.50 usec
TE 300.0 K
D1 15.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 0.00 dB
SFO1 500.1325006 MHz
SI 65536
SF 500.1300279 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
18.27
3.04
2.97
0.98
1.00
0.99
0.99
0.99
1.00
ppm
7.7 7.6 7.5 7.4 7.3 7.2 7.1 ppm

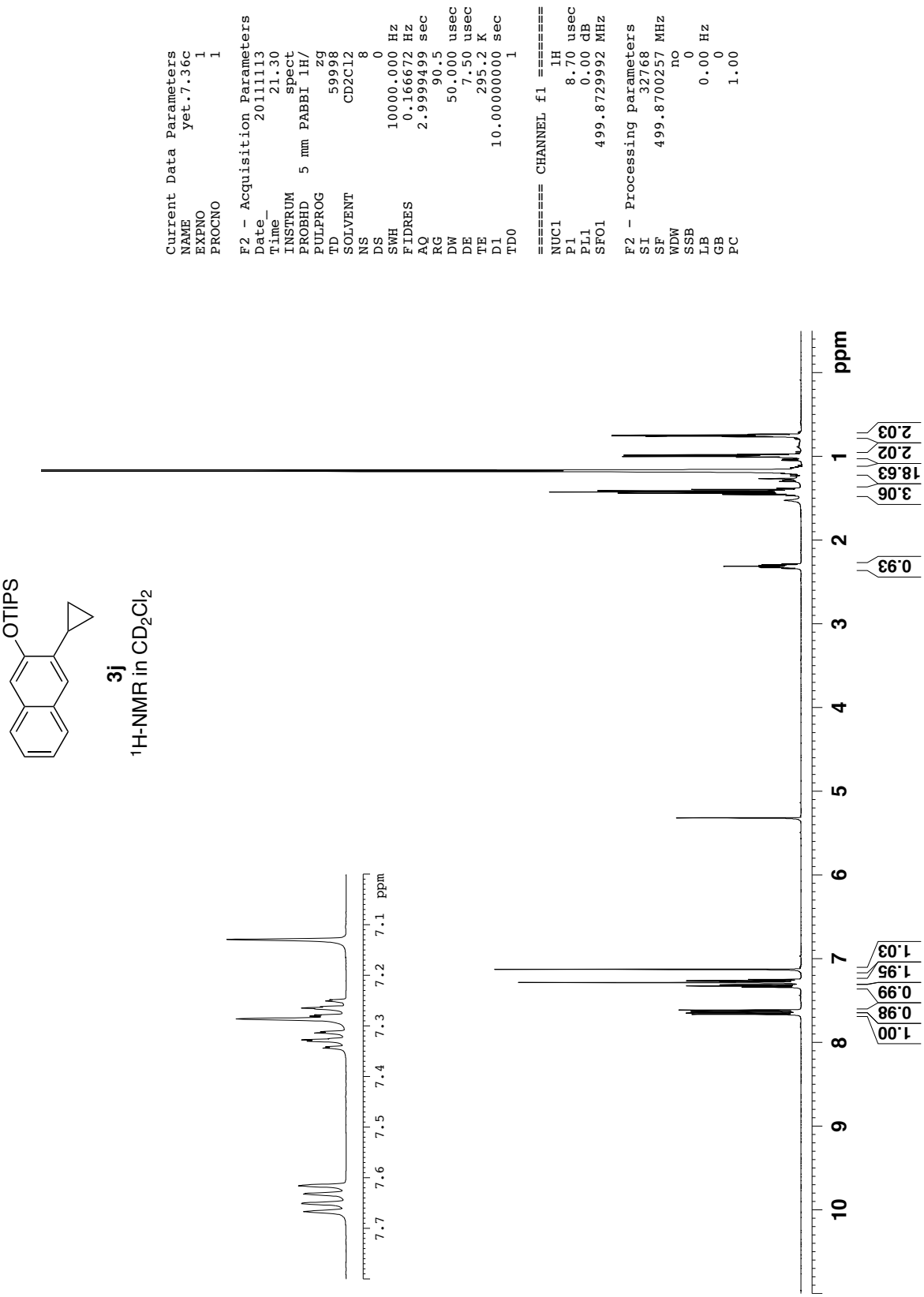

OTIPS
Me
3g
13C-NMR in CDCl3
153.30
133.34
130.64
129.10
128.87
126.80
126.10
125.07
123.41
112.38
77.25
77.00
76.75
18.11
17.80
13.04
ppm
200
180
160
140
120
100
80
60
40
20

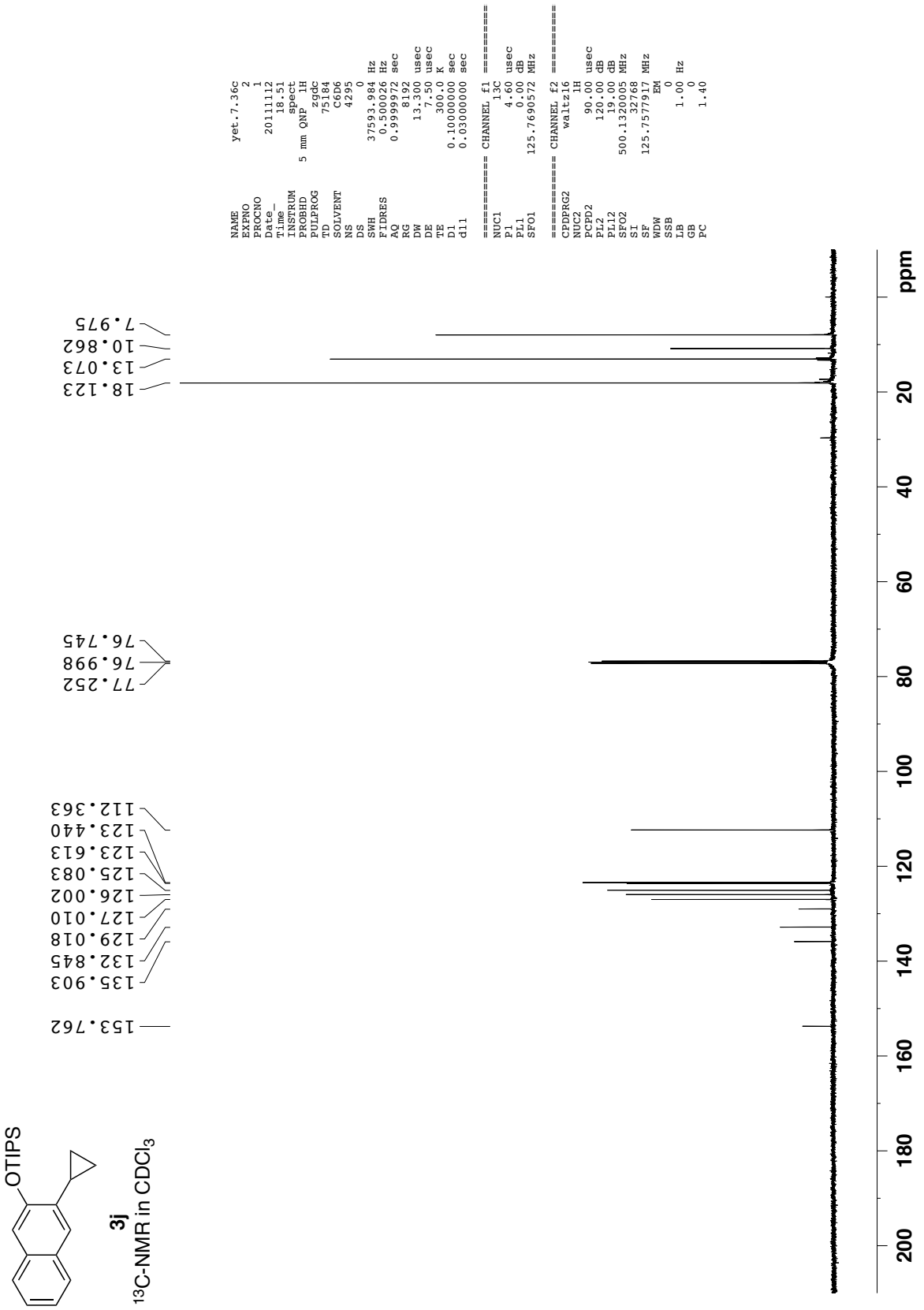

OTIPS
3h
^{1}H-NMR in $CDCl_3$
NAME yet.7.56b
EXPNO 1
PROCNO 1
Date_ 20111118
Time_ 23.10
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg
TD 48076
SOLVENT CDCl3
NS 4
DS 0
SWH 8012.820 Hz
FIDRES 0.166670 Hz
AQ 2.9999924 sec
RG 64
DW 62.400 usec
DE 4.50 usec
TE 300.0 K
D1 15.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 0.00 dB
SFO1 500.1325006 MHz
SI 65536
SF 500.1300248 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
18.42
3.08
2.02
2.01
1.03
0.99
1.00
1.06
3.08
3.33
1.00
7.7 7.6 7.5 7.4 7.3 7.2 ppm

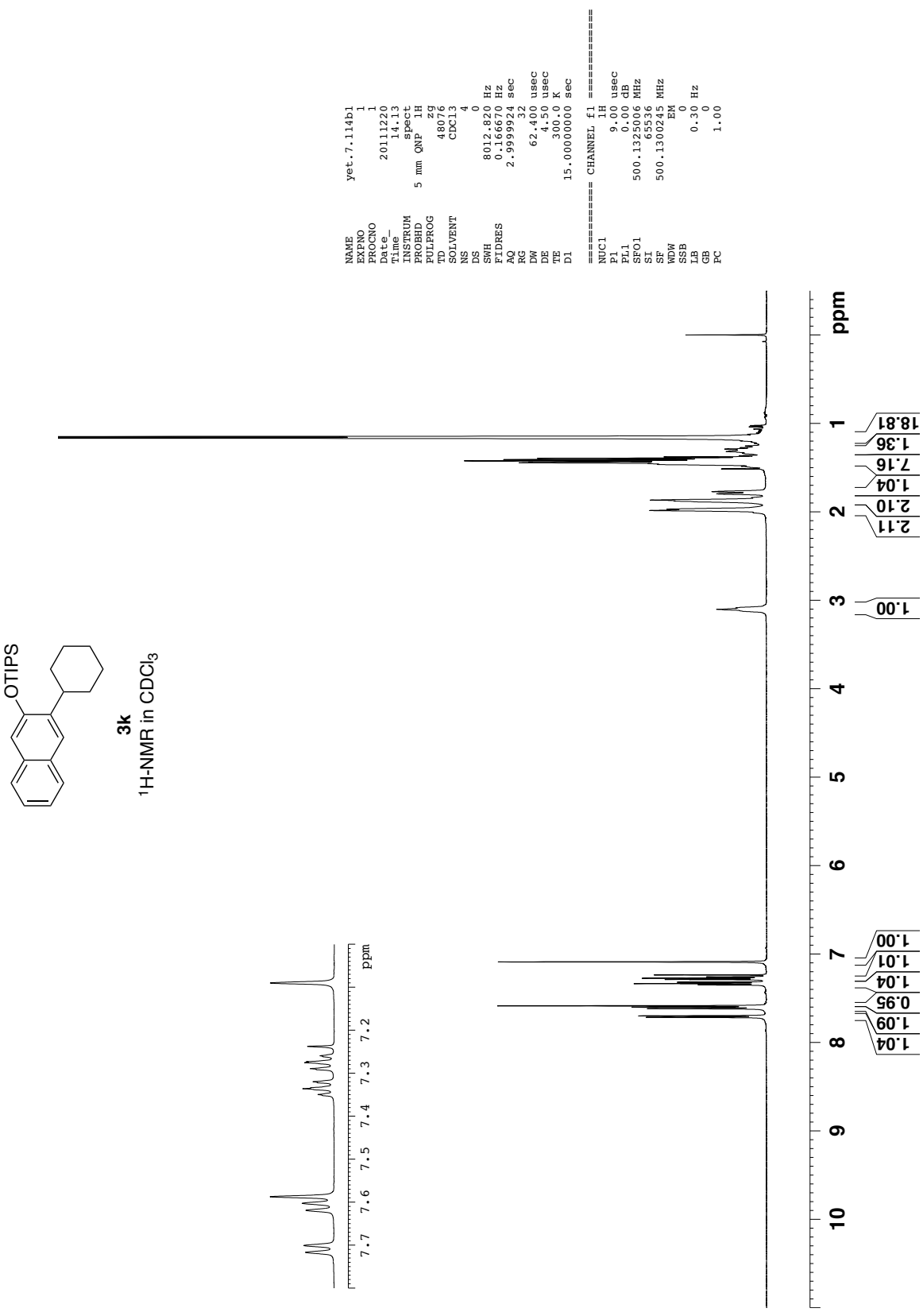

OTIPS
3h
13C-NMR in CDCl3
152.861
142.188
133.792
133.360
129.014
128.659
128.465
128.284
127.042
126.053
125.775
125.311
123.458
112.472
77.251
76.996
76.742
36.489
33.325
18.186
13.145
ppm
200
180
160
140
120
100
80
60
40
20
NAME yet.7.56b
EXPNO 2
PROCNO 1
Date_ 20111118
TIme_ 23.20
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgdc
TD 75184
SOLVENT C6D6
NS 682
DS 0
SWH 37593.984 Hz
FIDRES 0.500026 Hz
AQ 0.9999972 sec
RG 8192
DW 13.300 usec
DE 7.50 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec
========== CHANNEL f1 ==========
NUC1 13C
P1 4.60 usec
PL1 0.00 dB
SFO1 125.7690572 MHz
========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 120.00 dB
PL12 19.00 dB
SFO2 500.1320005 MHz
SI 32768
SF 125.7577941 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

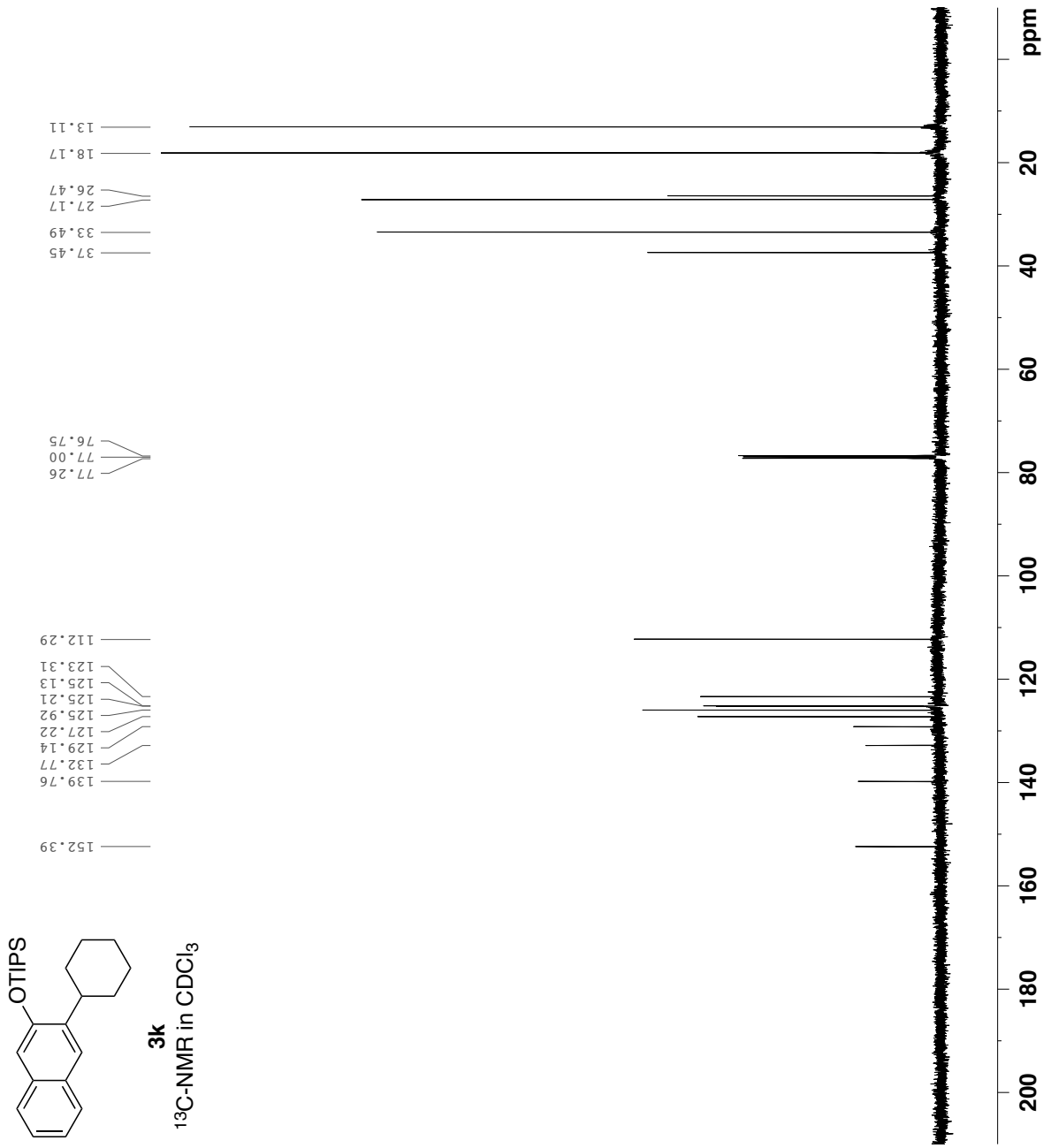

OTIPS
OTIPS
3i
^{1}H-NMR in $CDCl_3$
NAME yet.7.12b3
EXPNO 1
PROCNO 1
Date_ 20111121
Time_ 17.57
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg
TD 48076
SOLVENT C6D6
NS 4
DS 0
SWH 8012.820 Hz
FIDRES 0.166670 Hz
AQ 2.9999924 sec
RG 32
DW 62.400 usec
DE 4.50 usec
TE 300.0 K
D1 15.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 0.00 dB
SFO1 500.1325006 MHz
SI 65536
SF 500.1300399 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
21.94
18.44
3.12
2.01
2.00
1.00
0.99
1.04
1.00
0.99
1.00
1.4 1.3 1.2 1.1 1.0 ppm
7.7 7.6 7.5 7.4 7.3 7.2 ppm

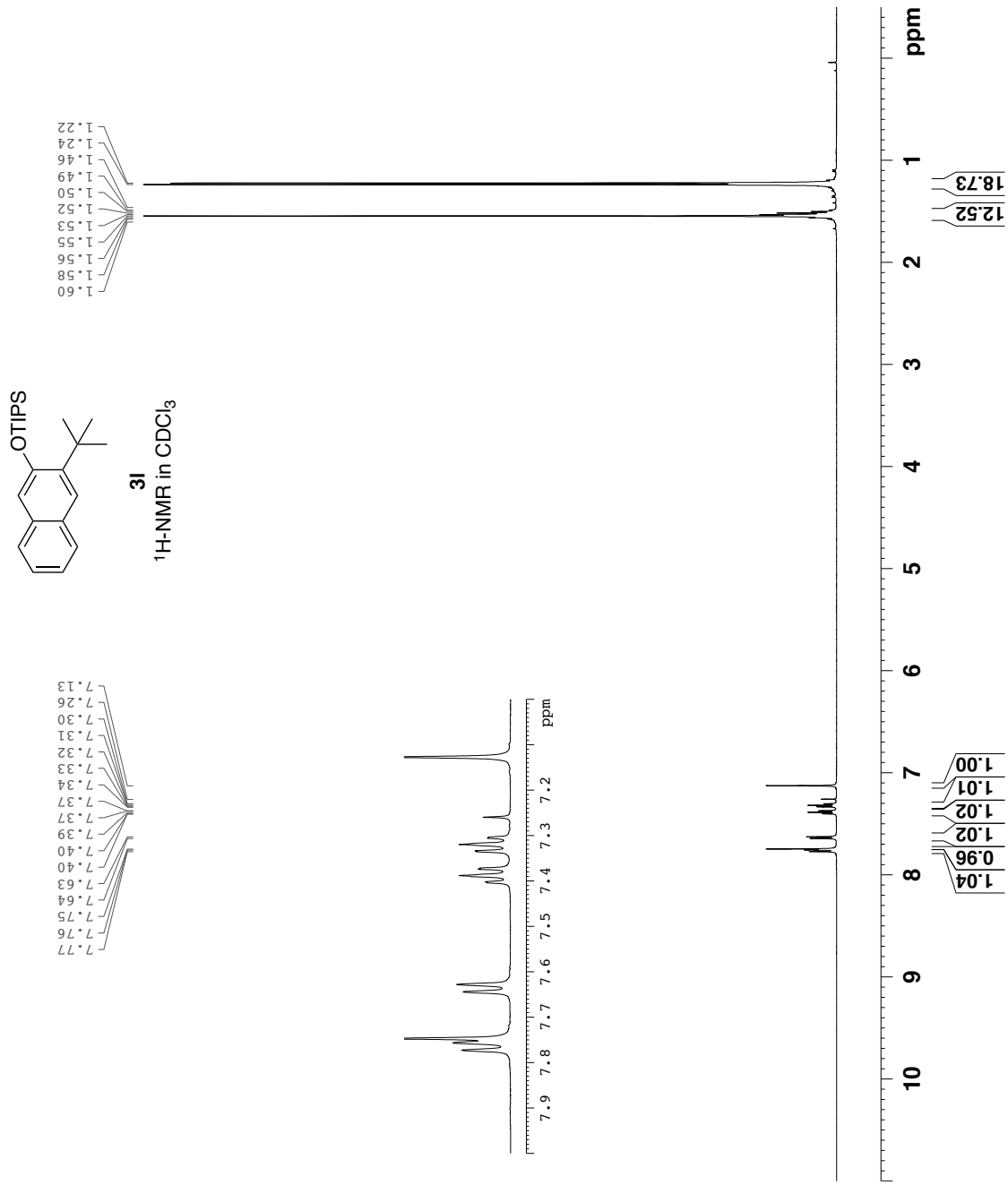

OTIPS
OTIPS
3i
13C-NMR in CDCl3
152.907
133.436
131.067
129.732
128.951
127.049
126.040
125.280
123.412
112.354
77.251
76.997
76.744
63.268
34.957
18.135
17.977
13.080
12.001
ppm
200
180
160
140
120
100
80
60
40
20

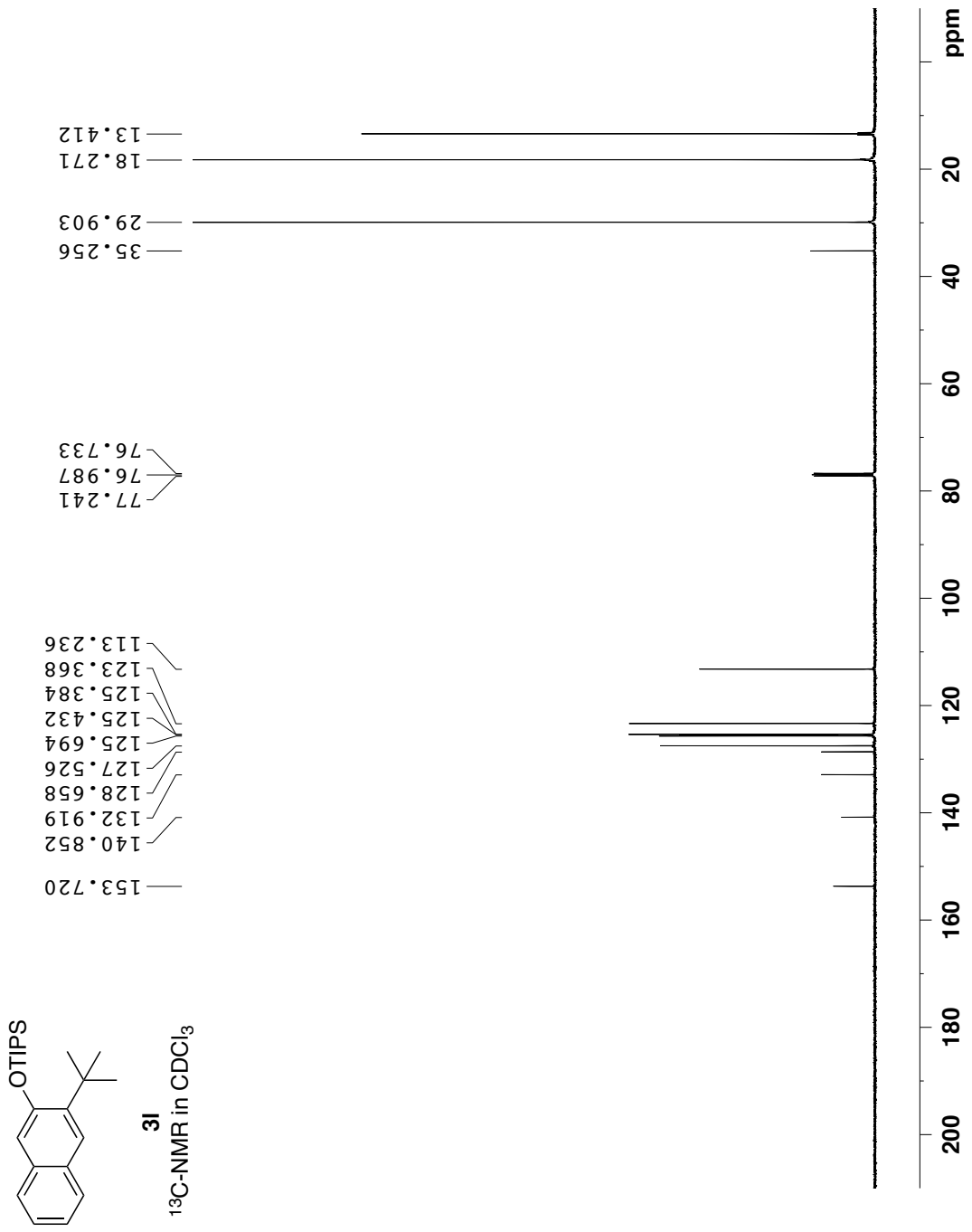

Current Data Parameters
NAME yet.7.36c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111113
Time_ 21.30
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CD2Cl2
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 sec
RG 90.5
DW 50.000 usec
DE 7.50 usec
TE 295.2 K
D1 10.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.70 usec
PL1 0.00 dB
SFO1 499.8729992 MHz
F2 - Processing parameters
SI 32768
SF 499.8700257 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
OTIPS
3j
^{1}H-NMR in CD_2Cl_2
ppm
0.93
3.06
18.63
2.02
2.03
1.00
0.98
0.99
1.95
1.03


OTIPS
3j
^{13}C-NMR in $CDCl_3$
153.762
135.903
132.845
129.018
127.010
126.002
125.083
123.613
123.440
112.363
77.252
76.998
76.745
18.123
13.073
10.862
7.975
ppm
200
180
160
140
120
100
80
60
40
20

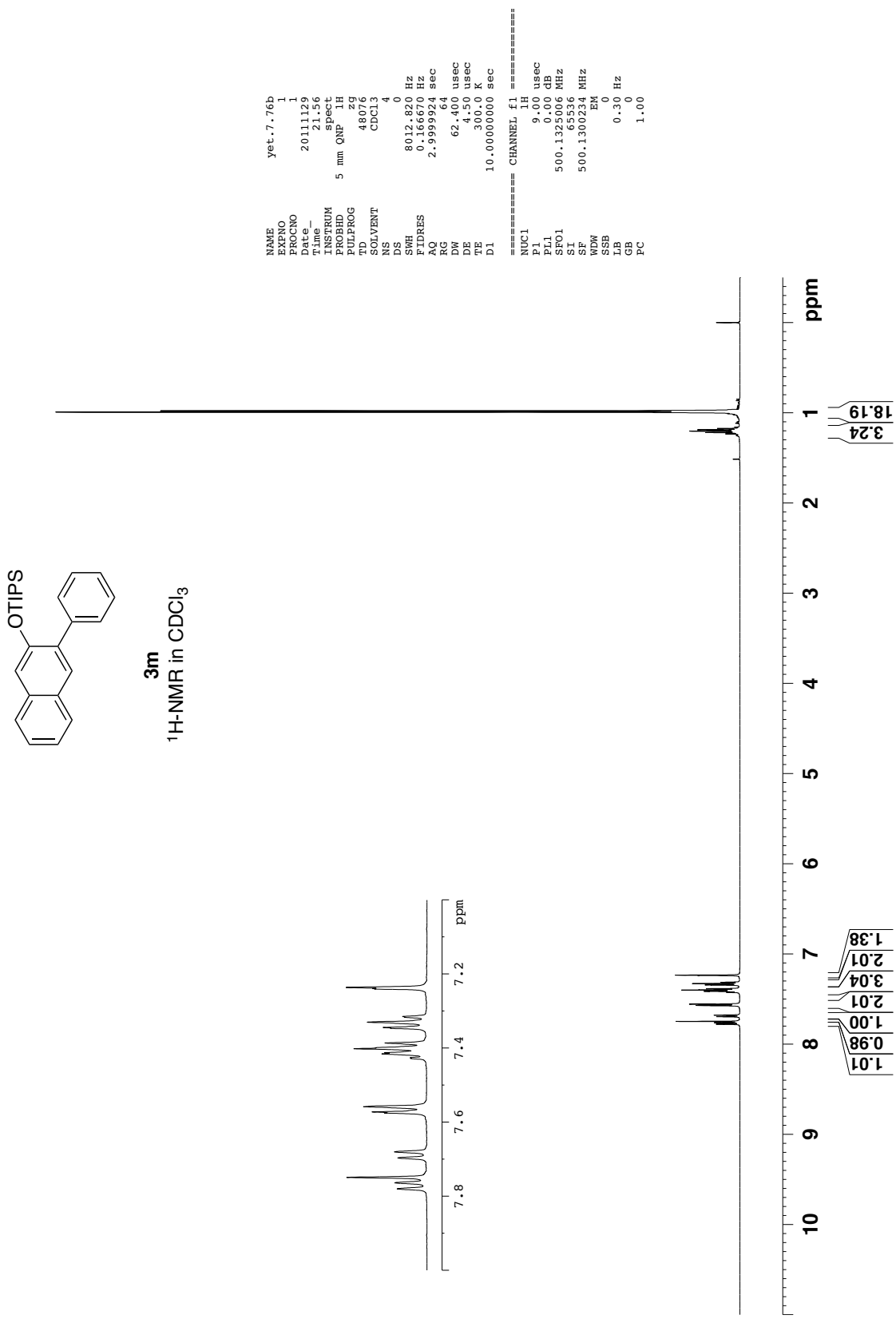

OTIPS
3k
^{1}H-NMR in $CDCl_3$
NAME yet.7.114b1
EXPNO 1
PROCNO 1
Date_ 20111220
Time_ 14.13
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg
TD 48076
SOLVENT CDCl3
NS 4
DS 0
SWH 8012.820 Hz
FIDRES 0.166670 Hz
AQ 2.9999924 sec
RG 32
DW 62.400 usec
DE 4.50 usec
TE 300.0 K
D1 15.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 0.00 dB
SFO1 500.1325006 MHz
SI 65536
SF 500.1300245 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
18.81
1.36
7.16
1.04
2.10
2.11
1.00
1.00
1.01
1.04
0.95
1.09
1.04
7.7 7.6 7.5 7.4 7.3 7.2 ppm

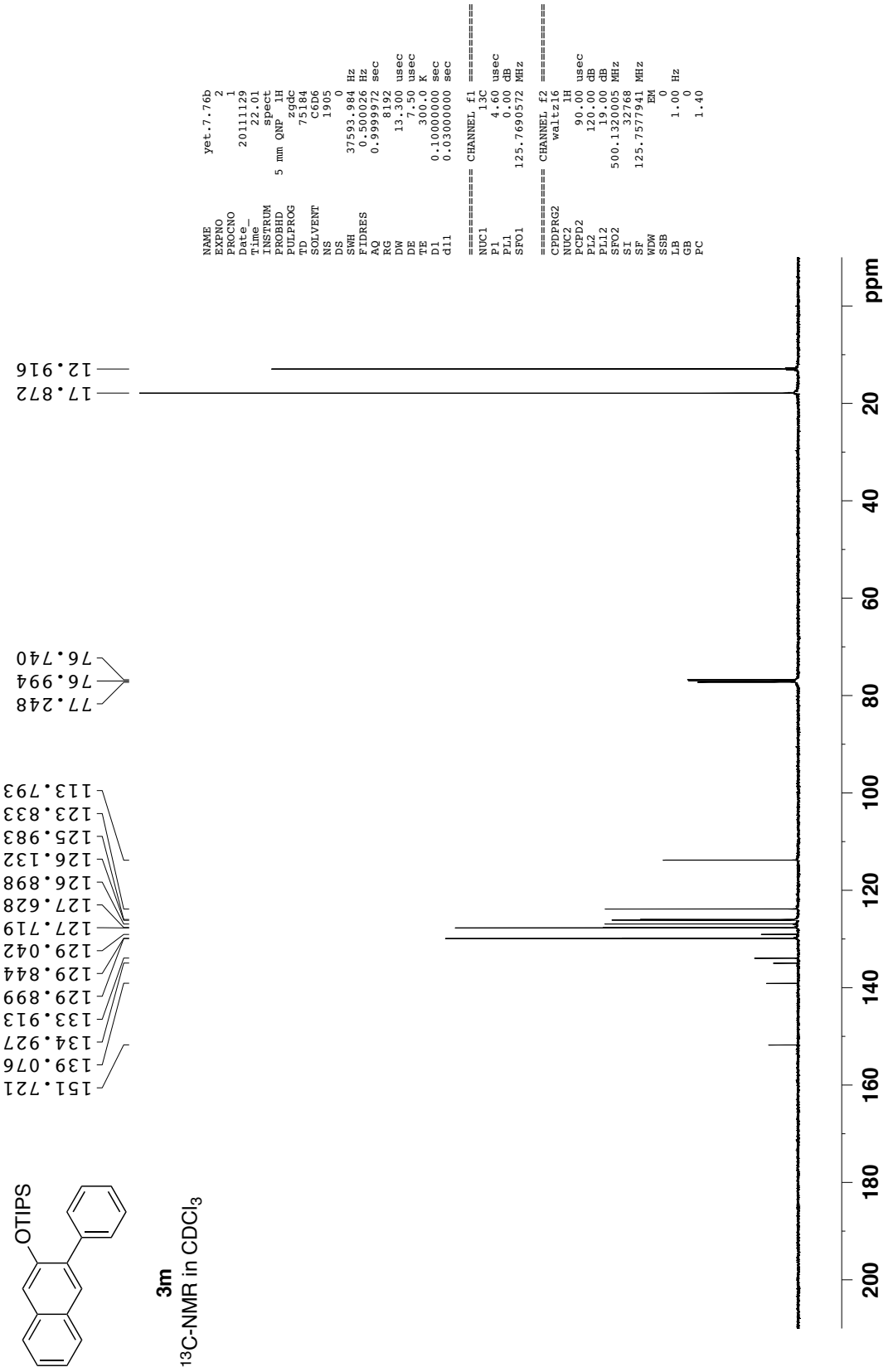

OTIPS
3k
13C-NMR in CDCl3
Current Data Parameters
NAME yet.7.1114b
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111220
Time 18.03
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgdc
TD 136360
SOLVENT CDCl3
NS 147
DS 0
SWH 30303.031 Hz
FIDRES 0.222228 Hz
AQ 2.2499900 sec
RG 16384
DW 16.500 usec
DE 7.50 usec
TE 295.1 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.00 usec
PL1 -3.00 dB
SFO1 125.7049802 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 28.00 dB
SFO2 499.8734991 MHz
F2 - Processing parameters
SI 32768
SF 125.6924188 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
13.11
18.17
26.47
27.17
33.49
37.45
76.75
77.00
77.26
112.29
123.31
125.13
125.21
125.92
127.22
129.14
132.77
139.76
152.39
ppm
200
180
160
140
120
100
80
60
40
20


OTIPS
3l
^{1}H-NMR in $CDCl_3$
NAME yet.7.126b1
EXPNO 1
PROCNO 1
Date_ 20120102
Time 14.25
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg
TD 48076
SOLVENT CDCl3
NS 4
DS 0
SWH 8012.820 Hz
FIDRES 0.166670 Hz
AQ 2.99999924 sec
RG 16
DW 62.400 usec
DE 4.50 usec
TE 300.0 K
D1 15.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 0.00 dB
SFO1 500.1325006 MHz
SI 65536
SF 500.1300134 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.60
1.58
1.56
1.55
1.53
1.52
1.50
1.49
1.46
1.24
1.22
7.77
7.76
7.75
7.64
7.63
7.40
7.40
7.39
7.37
7.37
7.34
7.33
7.32
7.31
7.30
7.26
7.13
7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 ppm
ppm
18.73
12.52
1.04
0.96
1.02
1.02
1.01
1.00

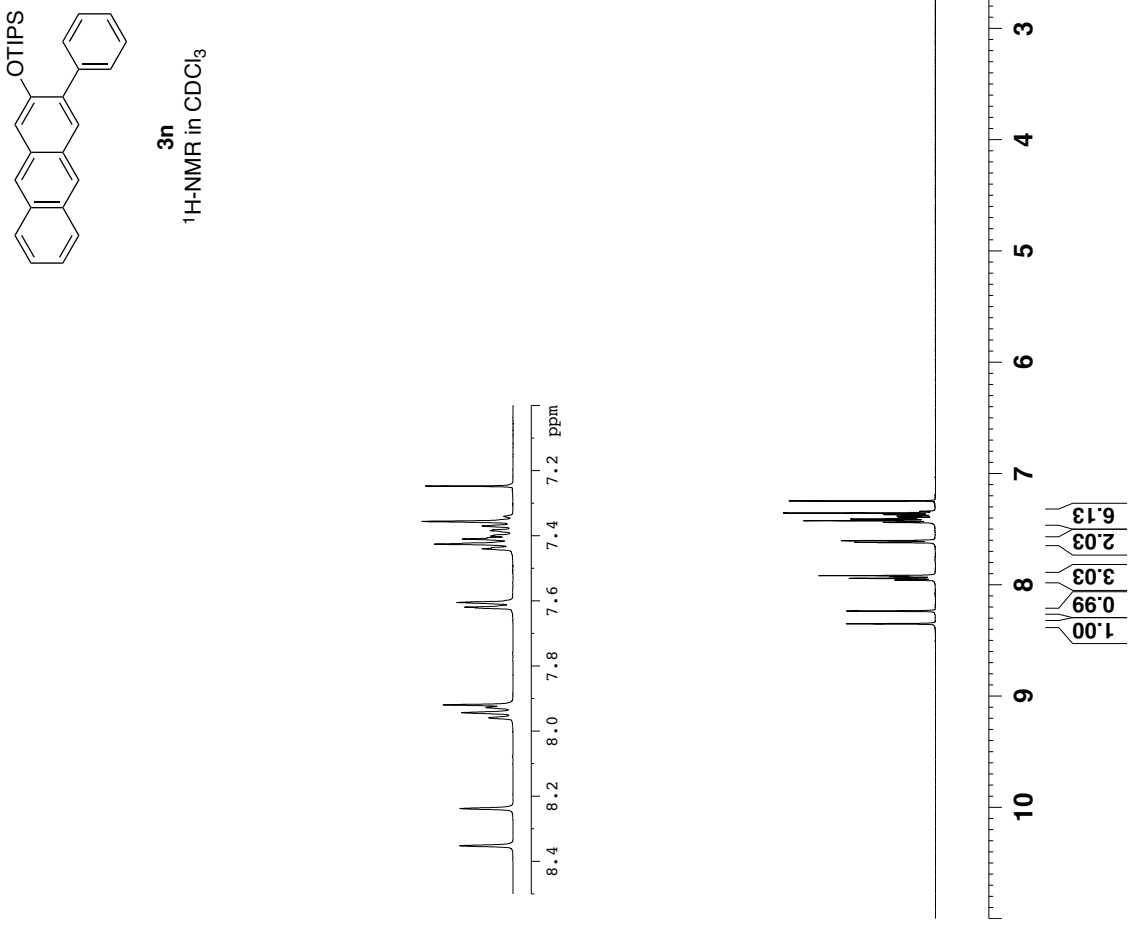


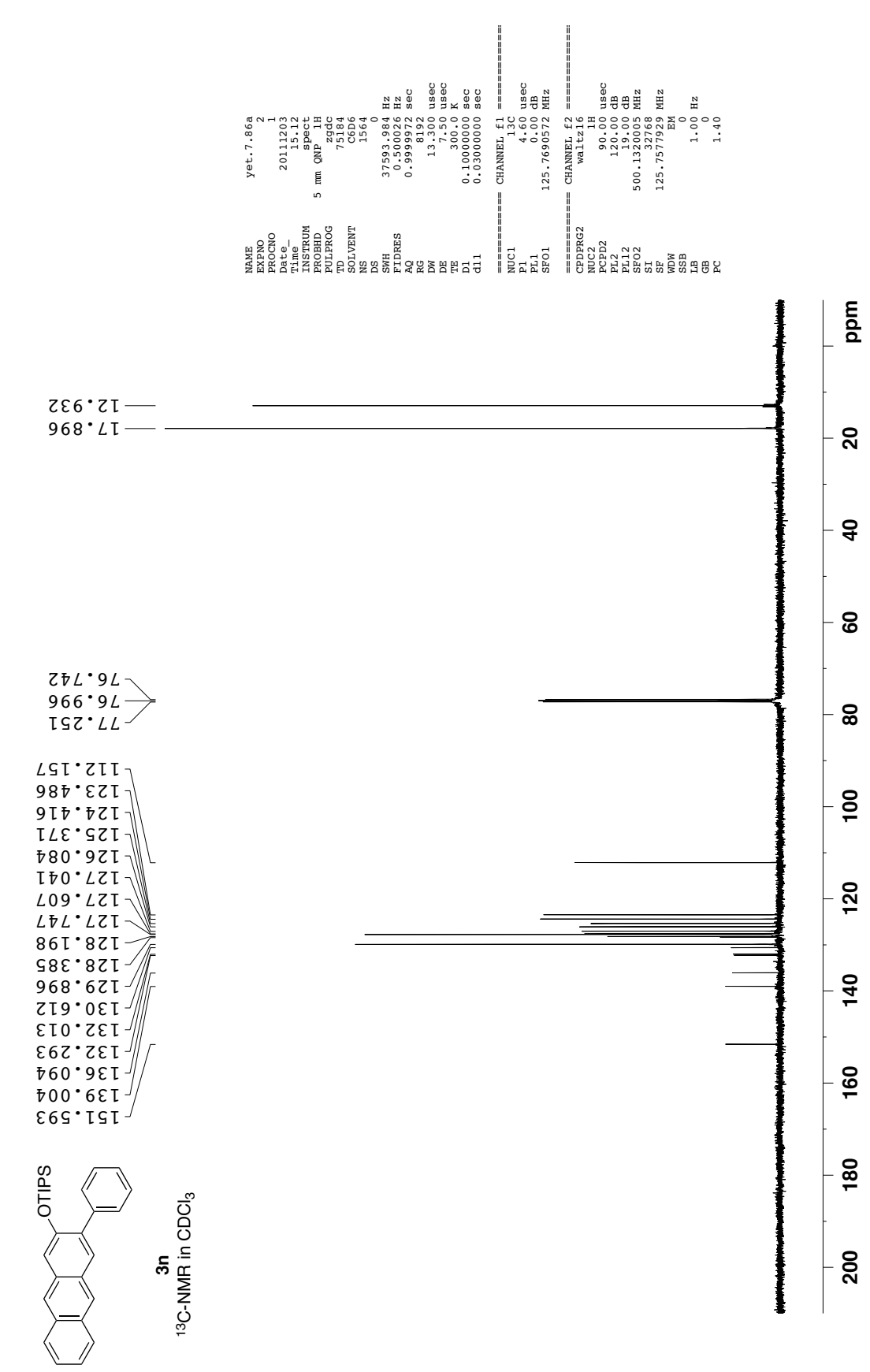

```
NAME                yet.7.126b1
EXPNO                         2
PROCNO                        1
Date_               20120102
Time                    14.34
INSTRUM                  spect
PROBHD     5 mm QNP  1H
PULPROG                  zgdc
TD                      75184
SOLVENT                 C6D6
NS                        900
DS                          0
SWH                 37593.984 Hz
FIDRES              0.500026 Hz
AQ                  0.9999972 sec
RG                       8192
DW                     13.300 usec
DE                      7.50 usec
TE                     300.0 K
D1                  0.10000000 sec
d11                 0.03000000 sec
============== CHANNEL f1 ============
NUC1                      13C
P1                      4.60 usec
PL1                     0.00 dB
SFO1               125.7690572 MHz
============== CHANNEL f2 ============
CPDPRG2                 waltz16
NUC2                         1H
PCPD2                   90.00 usec
PL2                     120.00 dB
PL12                     19.00 dB
SFO2               500.1320005 MHz
SI                        32768
SF                 125.7577963 MHz
WDW                          EM
SSB                           0
LB                     1.00 Hz
GB                            0
PC                     1.40
```

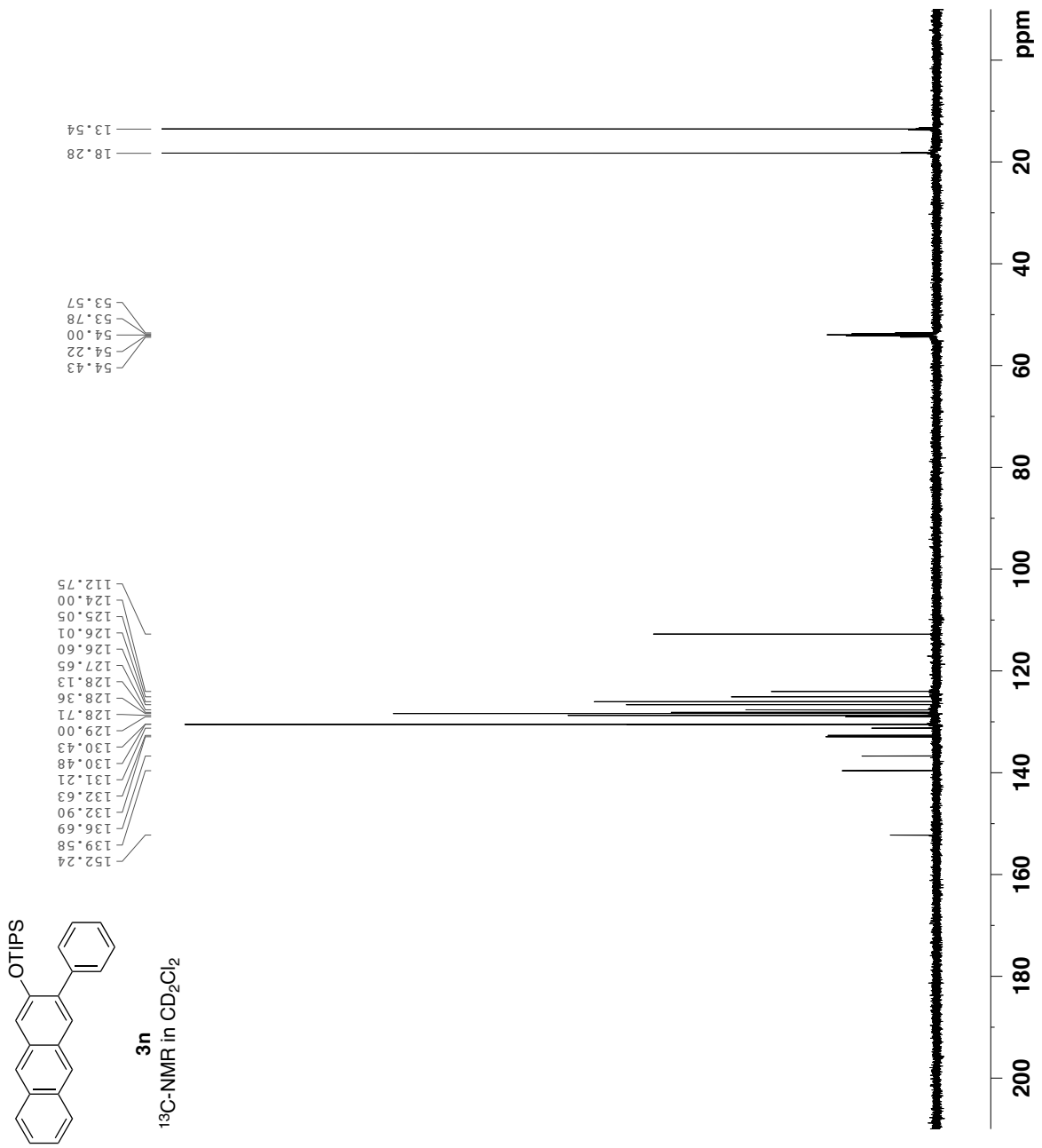

OTIPS
3m
^{1}H-NMR in $CDCl_3$
NAME yet.7.76b
EXPNO 1
PROCNO 1
Date_ 20111129
Time_ 21.56
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg
TD 48076
SOLVENT CDCl3
NS 4
DS 0
SWH 8012.820 Hz
FIDRES 0.166670 Hz
AQ 2.9999924 sec
RG 64
DW 62.400 usec
DE 4.50 usec
TE 300.0 K
D1 10.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 0.00 dB
SFO1 500.1325006 MHz
SI 65536
SF 500.1300234 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
18.19
3.24
1.01
0.98
1.00
2.01
3.04
2.01
1.38




```
NAME                yet.7.76b
EXPNO                       2
PROCNO                      1
Date_                20111129
Time_                   22.01
INSTRUM                 spect
PROBHD     5 mm QNP  1H
PULPROG                 zgdc
TD                     75184
SOLVENT                 C606
NS                      1905
DS                         0
SWH                37593.984 Hz
FIDRES             0.500026 Hz
AQ                 0.9999972 sec
RG                       8192
DW                     13.300 usec
DE                       7.50 usec
TE                      300.0 K
D1                 0.10000000 sec
d11                0.03000000 sec
============== CHANNEL f1 ==========
NUC1                      13C
P1                       4.60 usec
PL1                      0.00 dB
SFO1             125.7690572 MHz
============== CHANNEL f2 ==========
CPDPRG2                waltz16
NUC2                        1H
PCPD2                   90.00 usec
PL2                     120.00 dB
PL12                     19.00 dB
SFO2             500.1320005 MHz
SI                       32768
SF               125.7577941 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```


OTIPS
3n
^{1}H-NMR in $CDCl_3$
NAME yet.7.86a
EXPNO 1
PROCNO 1
Date_ 20111203
Time_ 14.59
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg
TD 48076
SOLVENT CDCl3
NS 6
DS 0
SWH 8012.820 Hz
FIDRES 0.166670 Hz
AQ 2.9999924 sec
RG 128
DW 62.400 usec
DE 4.50 usec
TE 300.0 K
D1 15.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 0.00 dB
SFO1 500.1325006 MHz
SI 65536
SF 500.1300196 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.4 8.2 8.0 7.8 7.6 7.4 7.2 ppm
10 9 8 7 6 5 4 3 2 1 ppm
1.00
0.99
3.03
2.03
6.13
3.31
18.45

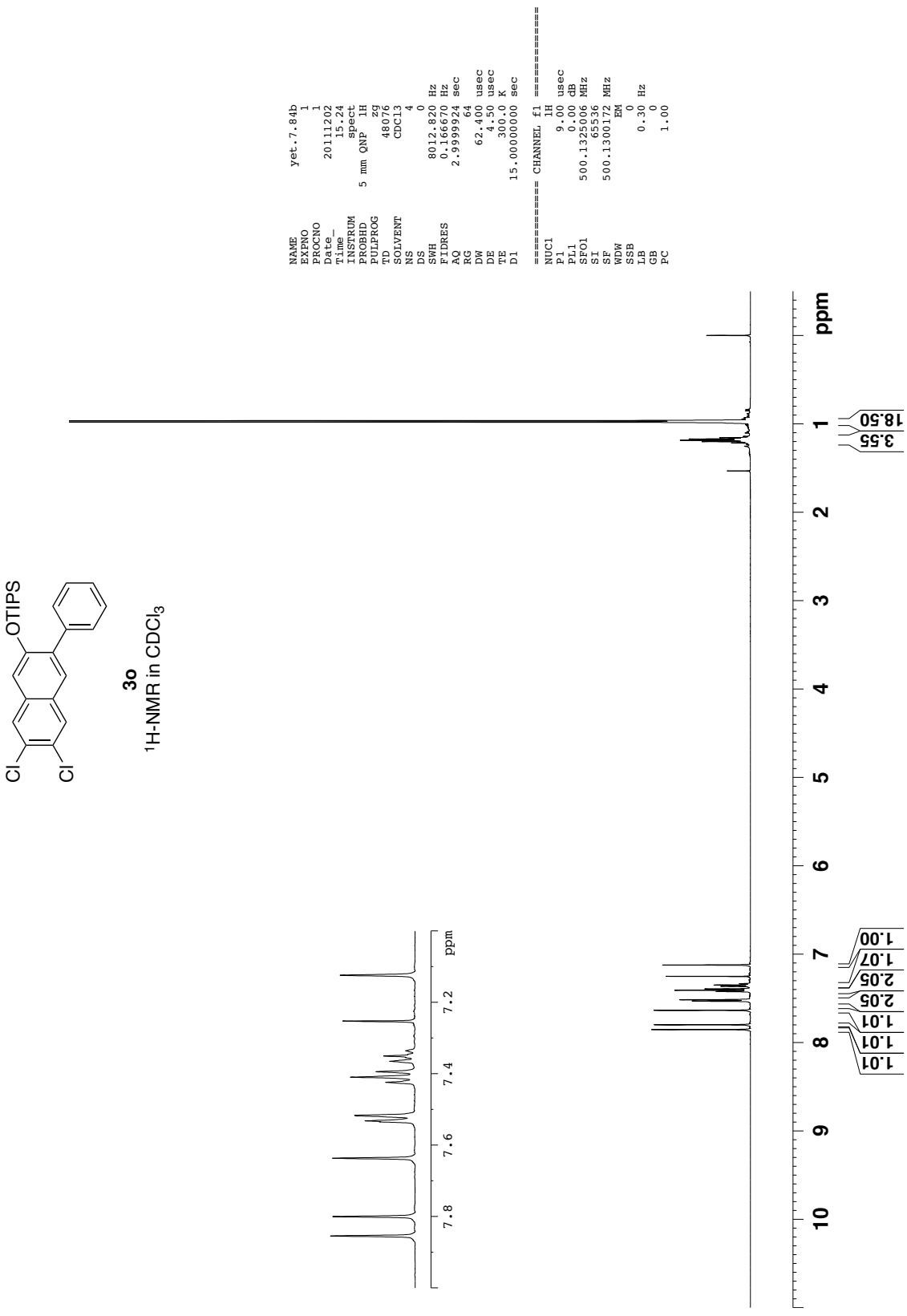

OTIPS
3n
^{13}C-NMR in $CDCl_3$
151.593
139.004
136.094
132.293
132.013
130.612
129.896
128.385
128.198
127.747
127.607
127.041
126.084
125.371
124.416
123.486
112.157
77.251
76.996
76.742
17.896
12.932
ppm
200
180
160
140
120
100
80
60
40
20

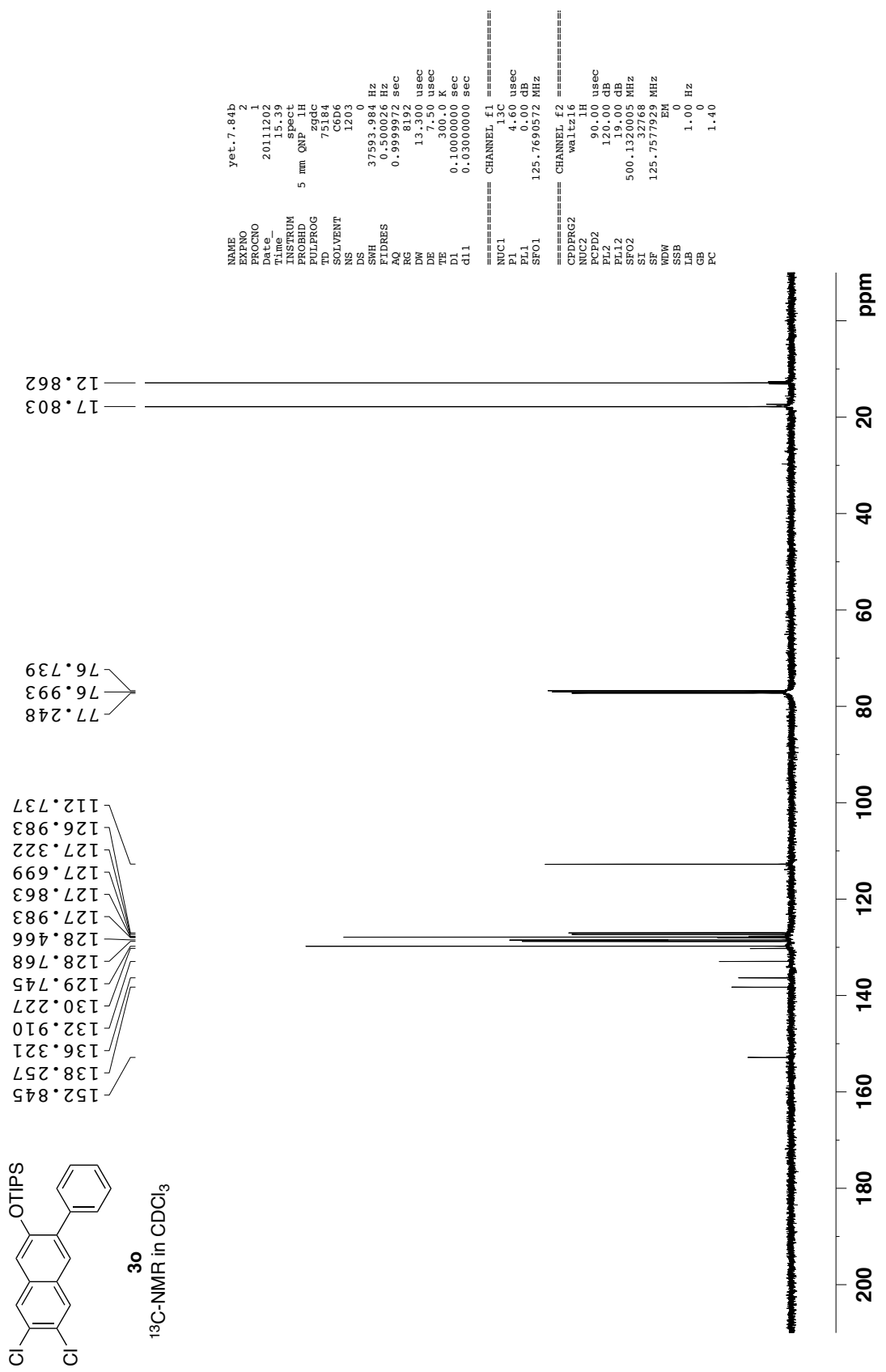



```
NAME          yet.7.86a2
EXPNO                  1
PROCNO                 1
Date_           20120129
Time_               5.57
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG              zgdc
TD                 142854
SOLVENT           CD2Cl2
NS                    188
DS                      0
SWH            30303.031 Hz
FIDRES          0.212126 Hz
AQ              2.3571410 sec
RG                18390.4
DW                16.500 usec
DE                 7.50 usec
TE                 295.5 K
D1             2.00000000 sec
d11            0.03000000 sec
TD0                     1

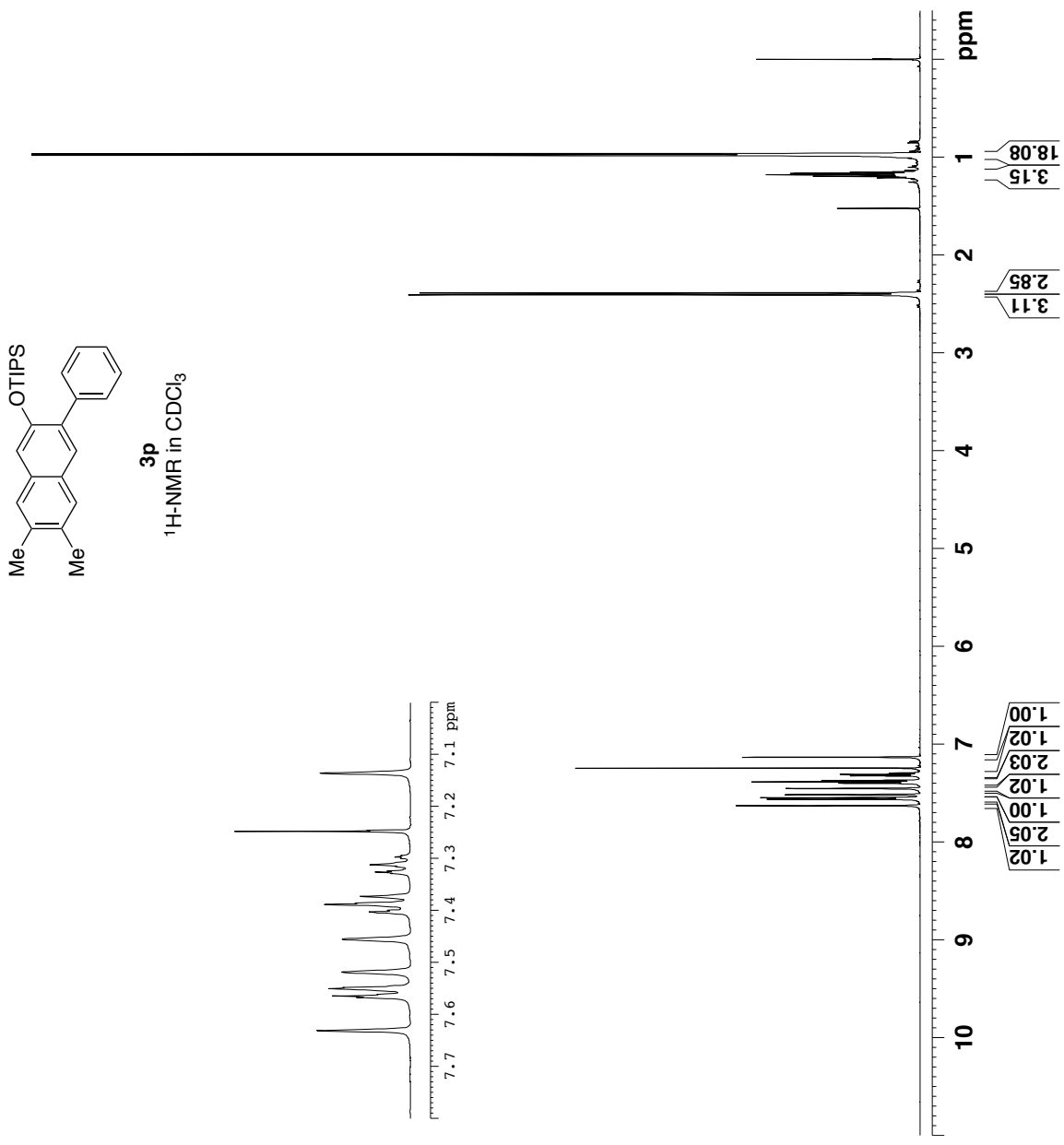
======== CHANNEL f1 ========
NUC1                  13C
P1                 8.50 usec
PL1                0.00 dB
SFO1         125.7049802 MHz

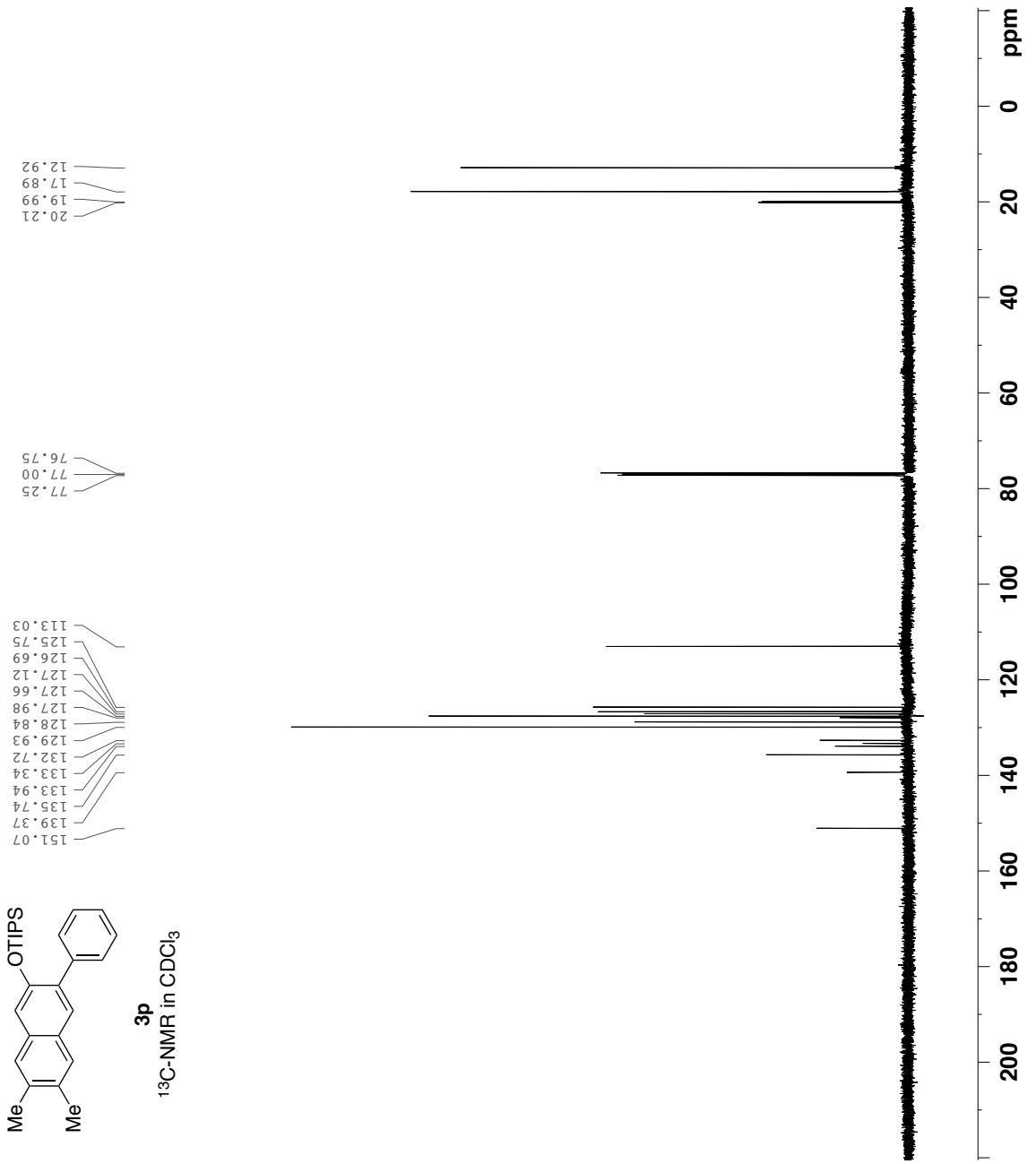
======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2              90.00 usec
PL2                 1.00 dB
PL12               21.00 dB
SFO2         499.8734991 MHz
SI                  32768
SF           125.6923423 MHz
WDW                    no
SSB                     0
LB                 0.00 Hz
GB                      0
PC                   1.40
```


OTIPS
Cl
Cl
3o
^{1}H-NMR in $CDCl_3$
NAME yet.7.84b
EXPNO 1
PROCNO 1
Date_ 20111202
Time_ 15.24
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg
TD 48076
SOLVENT CDCl3
NS 4
DS 0
SWH 8012.820 Hz
FIDRES 0.166670 Hz
AQ 2.9999924 sec
RG 64
DW 62.400 usec
DE 4.50 usec
TE 300.0 K
D1 15.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 0.00 dB
SFO1 500.1325006 MHz
SI 65536
SF 500.1300172 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
18.50
3.55
1.00
1.07
1.07
2.05
2.05
1.01
1.01
1.01
ppm




```
NAME            yet.7.84b
EXPNO                   2
PROCNO                  1
Date_            20111202
Time_               15.39
INSTRUM              spect
PROBHD   5 mm QNP  1H
PULPROG              zgdc
TD                  75184
SOLVENT              C6D6
NS                    1203
DS                       0
SWH             37593.984 Hz
FIDRES              0.500026 Hz
AQ                  0.9999972 sec
RG                       8192
DW                     13.300 usec
DE                      7.50 usec
TE                     300.0 K
D1              0.10000000 sec
d11             0.03000000 sec
================ CHANNEL f1 ============
NUC1                    13C
P1                     4.60 usec
PL1                    0.00 dB
SFO1            125.7690572 MHz
================ CHANNEL f2 ============
CPDPRG2              waltz16
NUC2                    1H
PCPD2                 90.00 usec
PL2                  120.00 dB
PL12                  19.00 dB
SFO2            500.1320005 MHz
SI                     32768
SF              125.7577929 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```


OTIPS
Me
Me
3p
^{1}H-NMR in $CDCl_3$
Current Data Parameters
NAME yet.7.88a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameter
Date_ 20111204
Time_ 20.13
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg
TD 59998
SOLVENT CDC13
NS 4
DS 0
SWH 10000.000 Hz
FIDRES 0.166672 Hz
AQ 2.9999499 se
RG 80.6
DW 50.000 us
DE 7.50 us
TE 295.4 K
D1 15.00000000 se
TD0 1
======== CHANNEL f1 ======
NUC1 1H
P1 8.70 us
PL1 0.00 dB
SFO1 499.8729992 MH
F2 - Processing parameters
SI 32768
SF 499.8700248 MH
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
ppm
18.08
3.15
2.85
3.11
1.00
1.02
1.03
2.03
1.02
1.00
2.05
1.02
7.1 ppm




```
Current Data Parameters
NAME         yet.7.88a
EXPNO                2
PROCNO               1

F2 - Acquisition Parameters
Date_         20111204
Time               20.21
INSTRUM            spect
PROBHD   5 mm PABBI 1H/
PULPROG             zgdc
TD                136360
SOLVENT           CDCl3
NS                  1534
DS                     0
SWH            30303.031 Hz
FIDRES             0.222228 Hz
AQ                 2.2499900 sec
RG                 18390.4
DW                 16.500 usec
DE                 7.50 usec
TE                 295.3 K
D1                 2.00000000 sec
d11                0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 12.00 usec
PL1                -3.00 dB
SFO1         125.7049802 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              90.00 usec
PL2                 0.00 dB
PL12               28.00 dB
SFO2         499.8734991 MHz

F2 - Processing parameters
SI                 32768
SF           125.6924180 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.40
```