Negishi Coupling of Secondary Alkylzinc Halides with Aryl Bromide and Chloride

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Supporting Information

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I. General Information.

General Analytical Information. Nuclear Magnetic Resonance spectra were recorded on a Varian 300 MHz instrument at ambient temperature. All ^{1}H NMR spectra were measured in parts per million (ppm) relative to the signals for residue chloroform in the deuterated solvent, unless otherwise stated. Data for ^{1}H NMR are reported as follows: chemical shift, multiplicity (app = apparent, br = broad, ovrlp = overlapping, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants, and integration. All ^{13}C NMR spectra are reported in ppm relative to deuterochloroform (77.23 ppm), unless otherwise stated, and were obtained with complete ^{1}H decoupling. All ^{31}P NMR spectra are reported in ppm relative to H_3PO_4 (0 ppm – external standard). All GC analyses were performed on an Agilent 6890 gas chromatograph with an FID detector using a J & W DB-1 column (10 m, 0.1 mm I.D.). Tetradecane was used as an internal standard to determine the GC yields if necessary. IR spectra were recorded on a Perkin-Elmer System 2000 FT-IR instrument (KBr disc). Melting points (uncorrected) were obtained on a Mel-Temp II capillary melting point apparatus. Elemental analyses were performed by Atlantic Microlabs, Inc., Norcross, GA.

General Reagent Information. THF, Et_2O, CH_2Cl_2 and toluene were purchased from J.T. Baker in CYCLE-TAINER® solvent-delivery kegs and vigorously purged with argon for 1 h. The solvents were further purified by passing them under argon pressure through two packed columns of neutral alumina (for THF and Et_2O) or through neutral alumina and copper (II) oxide (for toluene and CH_2Cl_2). $Pd(OAc)_2$ was received as a gift from BASF and aryl halides were purchased from Aldrich, Alfa Aesar, Acros, or TCI America. SPhos and XPhos were received as gifts from Shasun. RuPhos and DavePhos were purchased from Strem. Dimethylamine (2.0 M solution in THF), LiHMDS (1.0 M solution in THF), *n*-butyllithium (2.5 M solution in hexanes), and 2-bromopropane-d_7 were purchased from Aldrich. 2-bromopropane-1,1,1-d_3 was purchased from CDN isotope. $ClPCy_2$ was received as a gift from Nippon Chemical. Zinc powder (325 mesh) and LiCl were purchased from Strem. LiCl was stored and weighed out in a nitrogen-filled glovebox. Cyclopropyl zinc bromide solution in THF was purchased from Rieke Metals. Commercially obtained materials were used as received unless otherwise noted. *t*-BuXPhos precatalyst,[1] $PdCl_2$(dppf),[2] *tert*-butyl 4-iodopiperidine-1-carboxylate,[3] and 6-iodo-2-methylhept-2-ene[4] were prepared according to literature procedures.

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[1] Biscoe, M. R.; Fors, B. P.; Buchwald, S. L. *J. Am. Chem. Soc.* **2008**, *130*, 6686.

[2] Hayashi, T.; Konishi, M.; Kobori, Y.; Kumada, M.; Higuchi, T.; Hirotsu, K. *J. Am. Chem. Soc.* **1984**, *106*, 158.

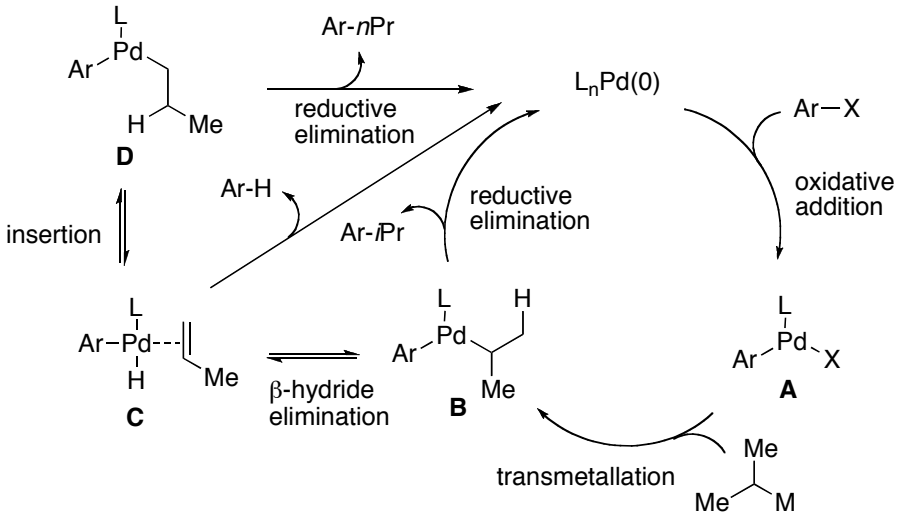
[3] Corley, E. G.; Conrad, K.; Murry, J. A.; Savarin, C.; Holko, J.; Boice, G. *J. Org. Chem.* **2004**, *69*, 5120.

[4] Vyvyan, J. R.; Loitz, C.; Looper, R. E.; Mattingly, C. S.; Peterson, E. A.; Staben, S. T. *J. Org. Chem.* **2004**, *69*, 2461.

General Considerations. All reactions were carried out in resealable test tubes with Teflon septa under an argon or nitrogen atmosphere. Flash column chromatography was performed was performed using a) Silicycle silica gel (ultra pure grade) or b) a Biotage SP4 instrument with pre-packed silica cartridges. Yields reported in the publication are of isolated material and represent an average of at least two independent runs. Compounds previously described in the literature were characterized by comparing their ^{1}H NMR and ^{13}C NMR spectra, and melting points (m.p.) to the previously reported data. All new compounds were characterized by ^{1}H NMR, ^{13}C NMR, and IR spectroscopy; their purity was confirmed by gas chromatography (GC) or elemental analysis. Copies of the ^{1}H, ^{13}C, and ^{31}P NMR spectra can be found at the end of the Supporting Information.

II. Experimental Procedures and Characterization Data.

A) Deuterium-Labeling Experiments.



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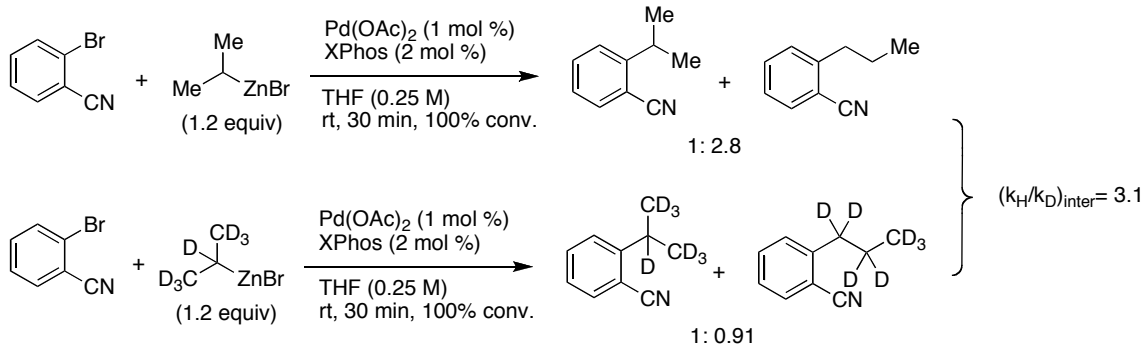

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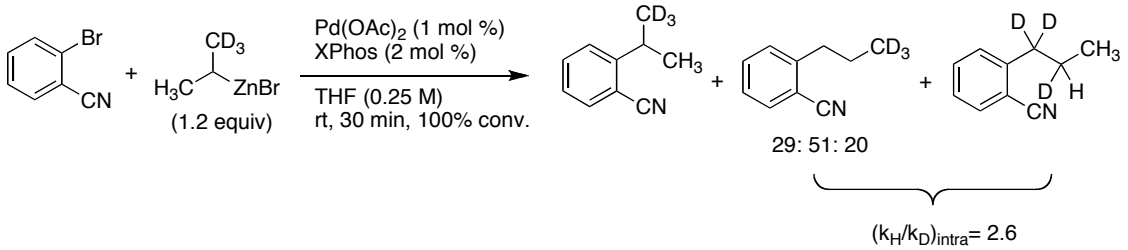
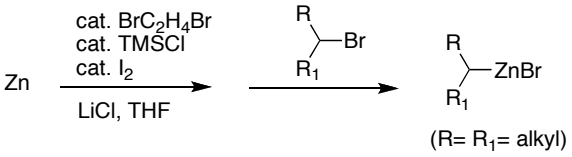
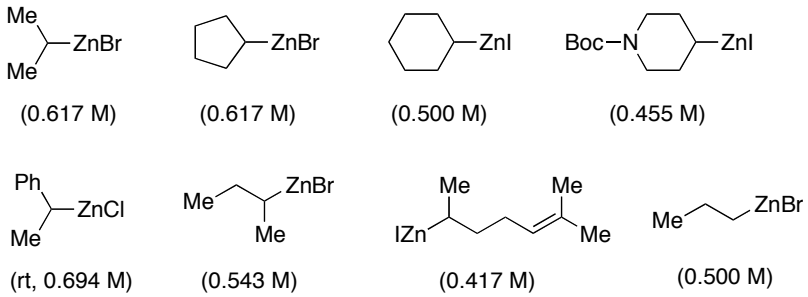


B) Preparation of Secondary Alkylzinc Halides Using Knochel's Protocol.[5]



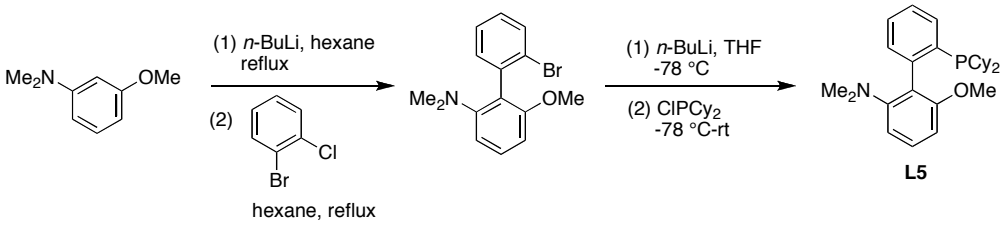
General Procedure: An oven-dried 250 mL round-bottom flask equipped with a magnetic stir bar and a rubber septum was charged with LiCl (8.48 g, 200 mmol). The vessel was heated with a heat gun for 10 min under high vacuum and backfilled with argon after cooling to room temperature. Zinc dust (13.1 g, 200 mmol) was added. The vessel was again heated with a heat gun for 10 min under high vacuum and backfilled with argon after cooling to room temperature. THF (100 mL) and 1,2-dibromoethane (431 μL, 5.00 mmol) were added via syringe and the reaction mixture was heated at 60 °C until bubbling occurred. After cooling to room temperature, trimethylsilyl chloride (126 μL, 1.00 mmol) and a solution of iodine (127 mg, 0.500 mmol) in THF (1 mL) were added via syringe. The reaction mixture was heated at 60 °C for 20 min and then cooled to room temperature. Alkyl halides (100 mmol) was added and the reaction was stirred at 50 °C for 18 h. The reaction mixture was allowed to stand at room temperature for 1 h and the supernatant solution was carefully transferred to a dry vessel via cannula. The concentration of the organozinc solution was determined by iodometric titration using Knochel's procedure.[6]



[5] Krasovskiy, A.; Malakhov, V.; Gavryushin, A.; Knochel, P. *Angew. Chem., Int. Ed.* **2006**, *45*, 6040.

[6] Krasovskiy, A.; Knochel, P. *Synthesis* **2006**, 890.

C) Preparation of Biarylphosphine Ligands L5 and L6 (CPhos).

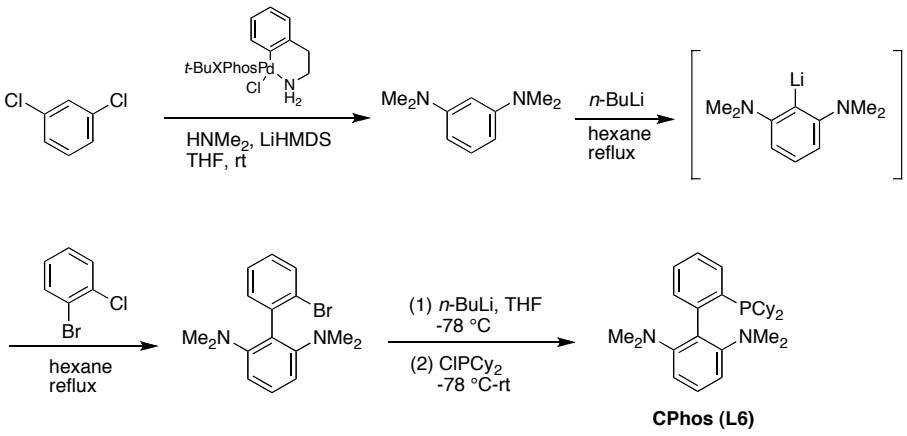


2'-(Dicyclohexylphosphino)-6-methoxy-*N*,*N*-dimethylbiphenyl-2-amine (L5). An oven-dried 500 mL three-neck round-bottom flask equipped with a magnetic stir bar and a reflux condenser was capped with rubber septa, fitted with an argon inlet adapter. The flask was then evacuated and backfilled with argon (this sequence was repeated three times). The reaction vessel was charged with 3-methoxy-*N*,*N*-dimethylaniline (8.07 mL, 55.0 mmol), anhydrous hexane (125 mL), and *n*-butyllithium (24.0 mL, 60.0 mmol, 2.5 M solution in hexanes). The reaction mixture was heated under reflux for 1 h (oil bath temperature at 80 °C) and cooled to room temperature. 2-bromochlorobenzene (5.81 mL, 50.0 mmol) was then added dropwise over 15 min with vigorous stirring (CAUTION: EXOTHERMIC!). The reaction mixture was heated at reflux for another 2 h and then cooled to room temperature (GC analysis of a reaction aliquot which was quenched by addition of ethanol indicated that complete consumption of the 2-bromochlorobenzene and clean conversion to 2'-bromo-6-methoxy-*N*,*N*-dimethylbiphenyl-2-amine had occurred). The reaction mixture was quenched by slow addition of water. The aqueous layer was extracted with ether (3×). The combined organic layer was dried over Na_2SO_4, filtered, and concentrated in vacuo. The resulting crude was purified by recrystallization from methanol/diethyl ether to afford 2'-bromo-6-methoxy-*N*,*N*-dimethylbiphenyl-2-amine as a brown solid (7.72 g, 50% yield), which was used directly for the next step.

An oven-dried 250 mL round-bottom flask, which was equipped with a magnetic stir bar and fitted with a Teflon septum, was charged with 2'-bromo-6-methoxy-*N*,*N*-dimethylbiphenyl-2-amine (6.12 g, 20.0 mmol). The vessel was evacuated and backfilled with argon (this process was repeated a total of 3 times). THF (80.0 mL) was added via syringe and the solution was cooled to -78 °C. *n*-Butyllithium (8.40 mL, 21.0 mmol, 2.5 M solution in hexanes) was added dropwise via syringe over 15 min. The resulting mixture was stirred at -78 °C for 30 min (periodic swirling of the reaction flask by hand was required as magnetic stirring became difficult). Chlorodicyclohexylphosphine (4.64 mL, 21.0 mmol) was then added dropwise via syringe over 15 min. The reaction mixture was stirred at -78 °C for 1 h and then allowed to slowly warm to room temperature. The reaction was quenched by addition of methanol and filtered through a pad of silica gel topped with a layer of Celite, eluting with ethyl acetate (400 mL). The filtrate was concentrated in vacuo to afford a yellow solid. Recrystallization from

dichloromethane and methanol provided the title compound as a pale-yellow solid (first crop: 7.24 g; second crop: 0.95 g; 96% combined yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.20 (d, *J* = 8.7 Hz, 2H), 6.90 (d, *J* = 8.7 Hz, 2H), 3.83 (s, 3H), 2.92 (septet, *J* = 6.9 Hz, 1H), 1.28 (d, *J* = 6.9 Hz, 6H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ: 158.0, 153.2, 145.3, 144.9, 136.2, 135.9, 133.0, 133.0, 132.9, 132.8, 128.8, 128.1, 125.9, 124.4, 124.3, 110.8, 104.2, 55.4, 43.8 (2C), 36.0, 35.8, 33.2, 33.0, 30.7, 30.4, 30.1, 30.0, 29.9, 29.8, 28.6, 28.5, 28.2, 28.0, 27.9, 27.8, 27.8, 27.7, 27.6, 27.5, 27.0, 26.7 ppm (observed complexity due to P-C splitting; definitive assignments have not been made). ^{31}P NMR (121 MHz, $CDCl_3$) δ: -8.75 ppm. IR (neat, cm^{-1}): 2920, 2850, 1592, 1576, 1468, 1448, 1427, 1252, 1080, 1003, 979, 910, 850, 791, 761, 729.



2'-Bromo-*N*^{2},*N*^{2},*N*^{6},*N*^{6}-tetramethylbiphenyl-2,6-diamine. An oven-dried flask was charged with *t*-BuXPhos precatalyst[1] (275 mg, 0.400 mmol). The flask was then evacuated and backfilled with argon (this process was repeated a total of 3 times). 1,3-dichlorobenzene (2.28 mL, 20.0 mmol) and dimethylamine (30.0 mL, 60.0 mmol, 2.0 M solution in THF) were added via syringe. The solution was cooled to 0 °C in an ice bath and LiHMDS (60.0 mL, 60.0 mmol, 1.0 M solution in THF) was added slowly via syringe. The ice bath was removed after the addition and the reaction was stirred at room temperature for 16 h. The reaction mixture was then quenched by the addition of water and extracted with ethyl acetate (3×). The combined organic layer was dried over Na_2SO_4, filtered, and concentrated in vacuo. The resulting residue was purified via flash chromatography on silica gel (hexanes: ethyl acetate = 6: 1) to afford *N*^{1},*N*^{1},*N*^{3},*N*^{3}-tetramethylbenzene-1,3-diamine as a pale-yellow oil (3.10 g, 94% yield).

An oven-dried 250 mL three-neck round-bottom flask equipped with a magnetic stir bar and a reflux condenser was capped with rubber septa, fitted with an argon inlet adapter. The flask was then evacuated and backfilled with argon (this sequence was repeated three times). The reaction vessel was charged with *N*^{1},*N*^{1},*N*^{3},*N*^{3}-tetramethylbenzene-1,3-diamine (3.29 g, 20.0 mmol), anhydrous hexane (50.0 mL), and *n*-butyllithium (8.80 mL, 22.0 mmol, 2.5 M solution in hexanes). The reaction mixture was heated at reflux for 1 h (oil bath temperature at 80 °C) and

cooled to room temperature. 2-bromochlorobenzene (2.55 mL, 22.0 mmol) was then added dropwise over 15 min with vigorous stirring (CAUTION: EXOTHERMIC!). The reaction mixture was heated at reflux for another 2 h and then cooled to room temperature (GC analysis of a reaction aliquot which was quenched by addition of ethanol indicated that complete consumption of the 2-bromochlorobenzene and clean conversion to 2'-bromo-N^2,N^2,N^6,N^6-tetramethylbiphenyl-2,6-diamine had occurred). The reaction mixture was quenched by slow addition of water. The aqueous layer was extracted with ether (3×). The combined organic layer was dried over Na_2SO_4, filtered, and concentrated in vacuo. The resulting crude was washed with cold methanol to afford the title compound as a pale-yellow solid (3.20 g). The filtrate was concentrated and purified using column chromatography on silica gel (hexanes: ethyl acetate = 20: 1) to provide the title compound as a yellow solid (2.76 g, 93% combined yield). m.p. 68-71 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.71 (d, J = 8.1 Hz, 1H), 7.40-7.34 (m, 3H), 7.22-7.16 (m, 1H), 6.95 (d, J = 8.1 Hz, 2H), 2.50 (s, 12H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ: 153.4, 140.3, 133.4, 132.8, 130.4, 129.1, 127.8, 127.0, 126.4, 114.0, 44.1 ppm. IR (neat, cm^{-1}): 2941, 2827, 2783, 1576, 1473, 1427, 1296, 1161, 1048, 1009, 945, 879, 805, 759, 744, 668. Anal. Calcd. for $C_{16}H_{19}BrN_2$: C, 60.20; H, 6.00. Found: C, 60.32; H, 5.85.

2'-(Dicyclohexylphosphino)-N^2,N^2,N^6,N^6-tetramethylbiphenyl-2,6-diamine (L6). An oven-dried 250 mL round-bottom flask, which was equipped with a magnetic stir bar and fitted with a Teflon septum, was charged with 2'-bromo-N^2,N^2,N^6,N^6-tetramethylbiphenyl-2,6-diamine (3.20 g, 10.0 mmol). The vessel was evacuated and backfilled with argon (this process was repeated a total of 3 times). THF (40.0 mL) was added via syringe and the solution was cooled to -78 °C. *n*-Butyllithium (4.20 mL, 10.5 mmol, 2.5 M solution in hexanes) was added dropwise via syringe over 15 min. The resulting mixture was stirred at -78 °C for 30 min (periodic swirling of the reaction flask by hand was required as magnetic stirring became difficult). Chlorodicyclohexylphosphine (2.32 mL, 10.5 mmol) was then added dropwise via syringe over 15 min. The reaction mixture was stirred at -78 °C for 1 h and then allowed to slowly warm to room temperature. The reaction was quenched by addition of methanol and filtered through a pad of silica gel topped with a layer of Celite, eluting with ethyl acetate (200 mL). The filtrate was concentrated in vacuo. Recrystallization from dichloromethane and methanol provided the title compound as a white solid (first crop: 3.62 g; second crop: 0.18 g; 87% combined yield). m.p. 111-113 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.69 (d, J = 7.2 Hz, 1H), 7.41-7.31 (m, 4H), 6.91 (d, J = 8.1 Hz, 2H), 3.49 (s, 12H), 2.02-1.58 (m, 12H), 1.38-0.60 (m, 10H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ: 153.5, 146.6, 146.1, 137.0, 136.8, 133.2, 133.1, 132.7, 132.7, 132.2, 132.1, 128.6, 127.6, 125.8, 114.0, 44.9, 44.8, 35.3, 35.1, 31.4, 31.2, 29.7, 29.6, 28.1, 27.9, 27.7, 27.6, 27.0 ppm (observed complexity due to P-C splitting; definitive assignments have not been made). ^{31}P NMR (121 MHz, $CDCl_3$) δ: -8.63 ppm. IR (neat, cm^{-1}): 2922, 2849, 2778, 1576, 1448, 1299, 1164, 1050, 1011, 910, 851, 802, 738. Anal. Calcd. for $C_{28}H_{41}N_2P$: C, 77.02; H, 9.47. Found: C, 77.24; H, 9.52.

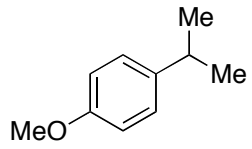
D) Experimental Procedures and Characterization Data for Examples Described in Table 1 and 2.

General Procedure A: Pd-Catalyzed Negishi Coupling of Aryl Halides with Secondary Alkylzinc Halides.

An oven-dried screw-cap test tube, which was equipped with a magnetic stir bar and fitted with a Teflon septum, was charged with $Pd(OAc)_2$ (2.25 mg, 0.0100 mmol) and biarylphosphine ligand **L6** (8.73 mg, 0.0200 mmol). The vessel was evacuated and backfilled with argon (this process was repeated a total of 3 times) and then the aryl bromide or chloride (1.00 mmol, if liquid) and THF or toluene (1.50-2.50 mL which makes the total volume of the reaction 4.00 mL) were added via syringe (aryl chlorides or amines that were solids at room temperature were added with the palladium catalyst and ligand). The solution was cooled to 0 °C in an ice bath and a THF solution of alkyl zinc halide (1.20 mmol, 1.50-2.50 mL) was added slowly via syringe. The ice bath was removed after the addition and the reaction was stirred at room temperature for indicated time until the starting material was completely consumed as monitored by GC. The reaction mixture was then quenched by addition of hydrochloric acid (1N) and extracted with ethyl acetate (3×).The combined organic layer was dried over Na_2SO_4, filtered, and concentrated in vacuo. The resulting residue was purified by flash chromatography using the Biotage SP4 (SNAP 10g or 25 g cartridges) to afford the desired product.

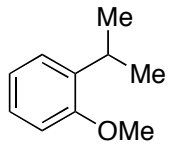
General Procedure B: Pd-Catalyzed Negishi Coupling of Aryl Halides with Secondary Alkylzinc Halides.

Procedure A was used with the following changes: A THF solution of alkyl zinc halide (1.20 mmol, 1.50-2.50 mL) was slowly added via syringe at room temperature instead of at 0 °C.

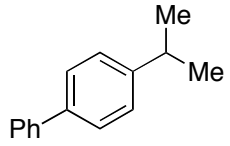


4-Isopropylanisole [CAS # 2944-47-0].[7] (Table 1, Entry 1) Following general procedure **A** with 4-bromoanisole (125 μL, 1.00 mmol), isopropyl zinc bromide solution (1.94 mL, 1.20 mmol, 0.617 M in THF), and THF (2.06 mL) at room temperature for 30 min, the crude product was purified via the Biotage SP4 (silica-packed SNAP 10 g; 0-6% ethyl acetate/hexanes) to provide a mixture of products as a light brown oil (138 mg, 92% yield). The ratio of the branched to the linear cross-coupled product was determined to be 37:1 by GC and ^{1}H NMR analysis. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.20 (d, *J* = 8.7 Hz, 2H), 6.90 (d, *J* = 8.7 Hz, 2H), 3.83 (s, 3H), 2.92 (septet, *J* = 6.9 Hz, 1H), 1.28 (d, *J* = 6.9 Hz, 6H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ: 157.8, 141.2, 127.4 (2C), 113.8 (2C), 55.4, 33.5, 24.4 (2C) ppm.

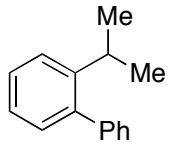
[7] Dreher, S. D.; Dormer, P. G.; Sandrock, D. L.; Molander, G. A. *J. Am. Chem. Soc.* **2008**, *130*, 9257.



2-Isopropylanisole [CAS # 2944-47-0].[7] (Table 1, Entry 7) Following general procedure **A** with 2-bromoanisole (125 μL, 1.00 mmol), isopropyl zinc bromide solution (1.94 mL, 1.20 mmol, 0.617 M in THF), and THF (2.06 mL) at room temperature for 30 min, the crude product was purified via the Biotage SP4 (silica-packed SNAP 10 g; 0-6% ethyl acetate/hexanes) to provide a mixture of products as a colorless oil (146 mg, 97% yield). The ratio of the branched to the linear cross-coupled product was determined to be 27:1 by GC and ^{1}H NMR analysis. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.24 (d, *J* = 7.5 Hz, 1H), 7.19 (dd, *J* = 8.1, 7.5 Hz, 1H), 6.95 (t, *J* = 7.5 Hz, 1H), 6.87 (d, *J* = 8.1 Hz, 1H), 3.85 (s, 3H), 3.34 (septet, *J* = 6.9 Hz, 1H), 1.23 (d, *J* = 6.9 Hz, 6H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ: 156.9, 137.1, 126.7, 126.2, 120.7, 110.4, 55.5, 26.8, 22.9 (2C) ppm.



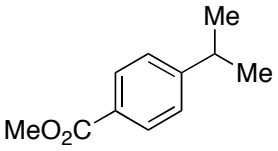
4-Isopropylbiphenyl [CAS # 7116-95-2].[8] (Table 1, Entry 2) Following general procedure **A** with 4-bromobiphenyl (233 mg, 1.00 mmol), isopropyl zinc bromide solution (1.94 mL, 1.20 mmol, 0.617 M in THF), and THF (2.06 mL) at room temperature for 30 min, the crude product was purified via the Biotage SP4 (silica-packed SNAP 10 g; 0-2% ethyl acetate/hexanes) to provide a mixture of products as a colorless oil (187 mg, 95% yield). The ratio of the branched to the linear cross-coupled product was determined to be 39:1 by GC and ^{1}H NMR analysis. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.76-7.45 (9H, m), 3.11 (septet, *J* = 6.9 Hz, 1H), 1.45 (d, *J* = 6.9 Hz, 6H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ: 148.1, 141.4, 138.9, 128.9 (2C), 127.3 (2C), 127.2 (2C), 127.1 (2C), 127.0, 34.0, 24.2 (2C) ppm.



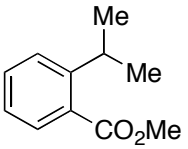
2-Isopropylbiphenyl [CAS # 19486-60-3].[9] (Table 1, Entry 8) Following general procedure **A** with 2-bromobiphenyl (172 μL, 1.00 mmol), isopropyl zinc bromide solution (1.94 mL, 1.20 mmol, 0.617 M in THF), and THF (2.06 mL) at room temperature for 3 h, the crude product was purified via the Biotage SP4 (silica-packed SNAP 10 g; 0-2% ethyl acetate/hexanes) to provide a mixture of products as a colorless oil (190 mg, 97% yield). The ratio of the branched to the linear cross-coupled product was determined to be 22:1 by GC and ^{1}H NMR analysis. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.45-7.18 (9H, m), 3.07 (septet, *J* = 6.9 Hz, 1H), 1.18 (d, *J* = 6.9 Hz, 6H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ: 146.5, 142.3, 141.3, 130.1, 129.5 (2C), 128.2 (2C), 127.9, 126.9, 125.7, 125.5, 29.5, 24.5 (2C) ppm.

[8] Smith, R. C.; Bodner, C. R.; Earl, M. J.; Sears, N. C.; Hill, N. E.; Bishop, L. M.; Sizemore, N.; Hehemann, D. T.; Bohn, J. J.; Protasiewicz, J. D. *J. Organomet. Chem.* **2005**, *690*, 477.

[9] Autio, J.; Vuoti, S.; Haukka, M.; Pursiainen, J. *Inorg. Chim. Acta* **2008**, *361*, 1372.

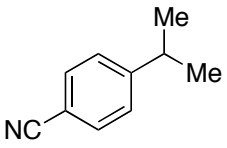


Methyl 4-isopropylbenzoate [CAS # 20185-55-1].[10] (Table 1, Entry 3) Following general procedure **A** with methyl 4-bromobenzoate (215 mg, 1.00 mmol), isopropyl zinc bromide solution (2.00 mL, 1.20 mmol, 0.600 M in THF), and toluene (2.00 mL) at room temperature for 30 min, the crude product was purified via the Biotage SP4 (silica-packed SNAP 10 g; 0-6% ethyl acetate/hexanes) to provide a mixture of products as a light yellow oil (167 mg, 94% yield). The ratio of the branched to the linear cross-coupled product was determined to be 46:1 by GC and ^{1}H NMR analysis. Or following general procedure **B** with methyl 4-chlorobenzoate (171 mg, 1.00 mmol) and isopropyl zinc bromide solution (2.40 mL, 1.20 mmol, 0.500 M in THF), and toluene (1.60 mL) at room temperature for 3 h, the crude product was purified via the Biotage SP4 (silica-packed SNAP 10 g; 0-6% ethyl acetate/hexanes) to provide a mixture of products as a light yellow oil (174 mg, 98% yield). The ratio of the branched to the linear cross-coupled product was determined to be 45:1 by GC and ^{1}H NMR analysis. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.97 (d, *J* = 8.4 Hz, 2H), 7.30 (d, *J* = 8.4 Hz, 2H), 3.91 (3H, s), 2.97 (septet, *J* = 6.9 Hz, 1H), 1.27 (d, *J* = 6.9 Hz, 6H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ: 167.3, 154.4, 129.9 (2C), 127.9, 126.6 (2C), 52.1, 34.4, 23.8 (2C) ppm. IR (neat, cm^{-1}): 2962, 1725, 1611, 1465, 1437, 1418, 1311, 1278, 1181, 1113, 1019, 775, 708.

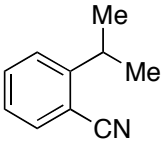


Methyl 2-isopropylbenzoate [CAS # 6623-98-9]. (Table 1, Entry 9) Following general procedure **A** with methyl 2-bromobenzoate (140 μL, 1.00 mmol), isopropyl zinc bromide solution (2.40 mL, 1.20 mmol, 0.500 M in THF), and toluene (1.60 mL) at room temperature for 30 min, the crude product was purified via the Biotage SP4 (silica-packed SNAP 10 g; 0-6% ethyl acetate/hexanes) to provide a mixture of products as a colorless oil (163 mg, 91% yield). The ratio of the branched to the linear cross-coupled product was determined to be 37:1 by GC and ^{1}H NMR analysis. Or following general procedure **B** with methyl 2-chlorobenzoate (143 μL, 1.00 mmol) and isopropyl zinc bromide solution (2.40 mL, 1.20 mmol, 0.500 M in THF), and toluene (1.60 mL) at room temperature for 6 h, the crude product was purified via the Biotage SP4 (silica-packed SNAP 10 g; 0-6% ethyl acetate/hexanes) to provide a mixture of products as a colorless oil (172 mg, 97% yield). The ratio of the branched to the linear cross-coupled product was determined to be 30:1 by GC and ^{1}H NMR analysis. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.74 (d, *J* = 8.1 Hz, 1H), 7.47 (t, *J* = 8.1 Hz, 1H), 7.43 (d, *J* = 8.1 Hz, 1H), 7.23 (t, *J* = 8.1 Hz, 1H), 3.90 (3H, s), 3.75 (septet, *J* = 6.9 Hz, 1H), 1.28 (d, *J* = 6.9 Hz, 6H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ: 168.9, 149.8, 131.9, 130.0, 129.9, 126.3, 125.6, 52.1, 29.7, 24.1 (2C) ppm. IR (neat, cm^{-1}): 2966, 1718, 1457, 1437, 1288, 1251, 1125, 1074, 761, 716. Anal. Calcd. for $C_{11}H_{14}O_2$: C, 74.13; H, 7.92. Found: C, 73.91; H, 8.09.

[10] Arnold, D. R.; de P. Nicholas, A. M.; Snow, M. S. *Can. J. Chem.* **1985**, *63*, 1150.

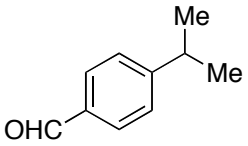


4-Isopropylbenzonitrile [CAS # 13816-33-6].[10] (Table 1, Entry 4) Following general procedure **A** with 4-bromobenzonitrile (182 mg, 1.00 mmol), isopropyl zinc bromide solution (2.40 mL, 1.20 mmol, 0.500 M in THF), and toluene (1.60 mL) at room temperature for 30 min, the crude product was purified via the Biotage SP4 (silica-packed SNAP 10 g; 0-6% ethyl acetate/hexanes) to provide a mixture of products as a colorless oil (126 mg, 87% yield). The ratio of the branched to the linear cross-coupled product was determined to be 59:1 by GC and ^{1}H NMR analysis. Or following general procedure **B** with 4-chlorobenzonitrile (138 mg, 1.00 mmol) and isopropyl zinc bromide solution (2.40 mL, 1.20 mmol, 0.500 M in THF), and toluene (1.60 mL) at room temperature for 30 min, the crude product was purified via the Biotage SP4 (silica-packed SNAP 10 g; 0-6% ethyl acetate/hexanes) to provide a mixture of products as a colorless oil (137 mg, 94% yield). The ratio of the branched to the linear cross-coupled product was determined to be 43:1 by GC and ^{1}H NMR analysis. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.57 (d, J = 8.4 Hz, 2H), 7.32 (d, J = 8.4 Hz, 2H), 2.96 (septet, J = 6.9 Hz, 1H), 1.25 (d, J = 6.9 Hz, 6H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ: 154.5, 132.3 (2C), 127.4 (2C), 119.3, 109.7, 34.5, 23.6 (2C) ppm. IR (neat, cm^{-1}): 2965, 2228, 1609, 1506, 1457, 1419, 1055, 1019, 836.

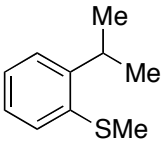


2-Isopropylbenzonitrile [CAS # 40751-52-8].[11] (Table 1, Entry 10) Following general procedure **A** with 2-bromobenzonitrile (182 mg, 1.00 mmol), isopropyl zinc bromide solution (2.40 mL, 1.20 mmol, 0.500 M in THF), and toluene (1.60 mL) at room temperature for 30 min, the crude product was purified via the Biotage SP4 (silica-packed SNAP 10 g; 0-6% ethyl acetate/hexanes) to provide a mixture of products as a colorless oil (189 mg, 89% yield). The ratio of the branched to the linear cross-coupled product was determined to be 20:1 by GC and ^{1}H NMR analysis. Or following general procedure **B** with 2-chlorobenzonitrile (138 mg, 1.00 mmol) and isopropyl zinc bromide solution (2.40 mL, 1.20 mmol, 0.500 M in THF), and toluene (1.60 mL) at room temperature for 3 h, the crude product was purified via the Biotage SP4 (silica-packed SNAP 10 g; 0-6% ethyl acetate/hexanes) to provide a mixture of products as a colorless oil (136 mg, 94% yield). The ratio of the branched to the linear cross-coupled product was determined to be 22:1 by GC and ^{1}H NMR analysis. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.61 (d, J = 7.5 Hz, 1H), 7.56 (t, J = 7.5 Hz, 1H), 7.40 (d, J = 7.5 Hz, 1H), 7.28 (t, J = 7.5 Hz, 1H), 3.39 (septet, J = 6.9 Hz, 1H), 1.32 (d, J = 6.9 Hz, 6H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ: 152.4, 133.1, 132.9, 126.4, 126.0, 118.3, 111.7, 32.5, 23.3 (2C) ppm. IR (neat, cm^{-1}): 2967, 2874, 2223, 1600, 1484, 1465, 1448, 1388, 1367, 1082, 1032, 762, 526.

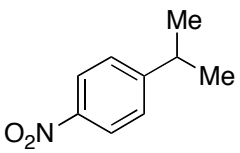
[11] Schareina, T.; Zapf, A.; Mägerlein, W.; Müller, N.; Beller, M. *Chem. Eur. J.* **2007**, *13*, 6249.



4-Isopropylbenzaldehyde [CAS # 122-03-2]. (Table 1, Entry 5) Following general procedure **A** with 4-bromobenzaldehyde (185 mg, 1.00 mmol), isopropyl zinc bromide solution (2.40 mL, 1.20 mmol, 0.500 M in THF), and toluene (1.60 mL) at room temperature for 30 min, the reaction was quenched by saturated ammonium chloride solution instead of hydrochloric acid (1N). The crude product was purified via the Biotage SP4 (silica-packed SNAP 10 g; 0-6% ethyl acetate/hexanes) to provide a mixture of products as a colorless oil (132 mg, 89% yield). The ratio of the branched to the linear cross-coupled product was determined to be 43:1 by GC and ^{1}H NMR analysis. Or following general procedure **B** with 4-chlorobenzaldehyde (141 mg, 1.00 mmol) and isopropyl zinc bromide solution (2.40 mL, 1.20 mmol, 0.500 M in THF), and toluene (1.60 mL) at room temperature for 30 min, the reaction was quenched by saturated ammonium chloride solution instead of hydrochloric acid (1N). The crude product was purified via the Biotage SP4 (silica-packed SNAP 10 g; 0-6% ethyl acetate/hexanes) to provide a mixture of products as a colorless oil (138 mg, 93% yield). The ratio of the branched to the linear cross-coupled product was determined to be 47:1 by GC and ^{1}H NMR analysis. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 9.98 (s, 1H), 7.83 (d, *J* = 8.4 Hz, 2H), 7.39 (d, *J* = 8.4 Hz, 2H), 3.00 (septet, *J* = 6.9 Hz, 1H), 1.29 (d, *J* = 6.9 Hz, 6H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ: 192.0, 156.2, 134.6, 130.0 (2C), 127.2 (2C), 34.5, 23.7 (2C) ppm. IR (neat, cm^{-1}): 2964, 2872, 2826, 2733, 1701, 1608, 1576, 1306, 1213, 1170, 1055, 840, 829, 730, 541.



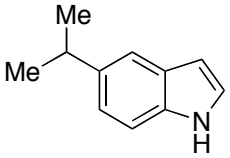
2-Isopropylthioanisole [CAS # 2976-66-1].[12] (Table 1, Entry 11) Following general procedure **A** with 2-bromothioanisole (133 μL, 1.00 mmol), isopropyl zinc bromide solution (1.94 mL, 1.20 mmol, 0.617 M in THF), and THF (2.06 mL) at room temperature for 1 h, the crude product was purified via the Biotage SP4 (silica-packed SNAP 10 g; 0-2% ethyl acetate/hexanes) to provide a mixture of products as a colorless oil (158 mg, 95% yield). The ratio of the branched to the linear cross-coupled product was determined to be 30:1 by GC and ^{1}H NMR analysis. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.26-7.15 (m, 4H), 3.39 (septet, *J* = 6.9 Hz, 1H), 2.47 (3H, s), 1.26 (d, *J* = 6.9 Hz, 6H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ: 146.6, 136.6, 126.5, 125.7, 125.4, 125.2, 30.1, 23.3 (2C), 16.2 ppm.



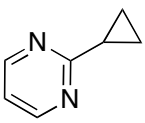
1-Isopropyl-4-nitrobenzene [CAS # 1817-47-6].[13] (Table 1, Entry 6) Following general procedure **A** with 1-bromo-4-nitrobenzene (185 mg, 1.00 mmol), isopropyl zinc bromide solution (2.40 mL, 1.20 mmol, 0.500 M in THF), and toluene (1.60 mL) at 0 °C for 30 min, the reaction was quenched by water instead of hydrochloric acid (1N).

[12] Ruff, F.; Kucsman, A. *J. Chem. Soc., Perkin Trans. II* **1988**, 1123.
[13] Shi, M.; Cui, S.-C. *Adv. Synth. Catal.* **2003**, *345*, 1329.

The crude product was purified via the Biotage SP4 (silica-packed SNAP 10 g; 0-2% ethyl acetate/hexanes) to provide a mixture of products as a yellow oil (83 mg, 50% yield). The ratio of the branched to the linear cross-coupled product was determined to be 28:1 by ^{1}H NMR analysis. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 8.14 (d, *J* = 8.7 Hz, 2H), 7.37 (d, *J* = 8.7 Hz, 2H), 3.02 (septet, *J* = 6.9 Hz, 1H), 1.28 (d, *J* = 6.9 Hz, 6H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ: 156.7, 127.4 (2C), 124.4, 123.8 (2C), 34.4, 23.7 (2C) ppm. IR (neat, cm^{-1}): 3078, 2966, 2932, 2873, 1606, 1521, 1347, 1112, 1055, 854, 757, 700.



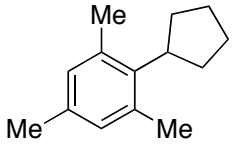
5-Isopropyl-1*H*-indole [CAS # 97820-51-4].[14] (Table 1, Entry 12) Following general procedure **A** with 5-bromoindole (196 mg, 1.00 mmol), isopropyl zinc bromide solution (2.09 mL, 1.20 mmol, 0.575 M in THF), and THF (1.91 mL) at room temperature for 30 min, the reaction was quenched by saturated ammonium chloride solution instead of hydrochloric acid (1N). The crude product was purified via the Biotage SP4 (silica-packed SNAP 10 g; 0-32% ethyl acetate/hexanes) to provide a mixture of products as a brown oil (153 mg, 96% yield). The ratio of the branched to the linear cross-coupled product was determined to be 58:1 by GC and ^{1}H NMR analysis. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 8.05 (br s, 1H), 7.52 (m, 1 H), 7.34 (d, *J* = 8.4 Hz, 1H), 7.19 (dd, *J* = 3.0, 2.7 Hz, 1H), 7.13 (dd, *J* = 8.4, 1.8 Hz, 1H), 6.54-6.52 (m, 1H), 3.04 (septet, *J* = 6.9 Hz, 1H), 1.34 (d, *J* = 6.9 Hz, 6H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ: 140.7, 134.5, 128.1, 124.5, 121.4, 117.7, 111.0, 102.4, 34.4, 24.9 (2C) ppm. IR (neat, cm^{-1}): 3586, 2958, 2868, 1576, 1476, 1457, 1415, 1351, 1308, 1092, 878, 806, 765, 724, 607.



1a

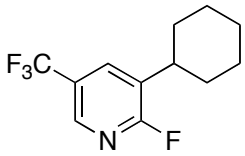
2-Cyclopropylpyrimidine [CAS # 58173-74-3]. (Scheme 2, **1a**) Following general procedure **A** with 2-bromopyrimidine (196 mg, 1.00 mmol), cyclopropyl zinc bromide solution (3.00 mL, 1.20 mmol, 0.400 M in THF), and THF (1.00 mL) at room temperature for 30 min, the reaction was quenched by saturated ammonium chloride solution instead of hydrochloric acid (1N). The crude product was purified via the Biotage SP4 (silica-packed SNAP 10 g; 0-60% ethyl acetate/hexanes) to provide the title compound as a light yellow oil (90 mg, 75% yield). ^{1}H NMR (300 MHz, $CDCl_3$) δ: 8.50 (d, *J* = 4.8 Hz, 2H), 6.98 (t, *J* = 4.8 Hz, 1H), 2.19 (tt, *J* = 8.1, 4.8 Hz, 1H), 1.07 (dt, *J* = 4.8, 2.7 Hz, 2H), 1.02 (dt, *J* = 8.1, 2.7 Hz, 2H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ: 172.3, 156.9, 156.8, 118.0, 18.3, 11.0 (2C) ppm. IR (neat, cm^{-1}): 3042, 3008, 1559, 1443, 1371, 1230, 1104, 1047, 1027, 910, 794, 635, 586, 537. Anal. Calcd. for $C_7H_8N_2$: C, 69.97; H, 6.71. Found: C, 69.16; H, 7.04.

[14] Bromidge, S. M. et al. *J. Med. Chem.* **1998**, *41*, 1598.



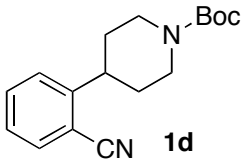
1b

2-Cyclopentyl-1,3,5-trimethylbenzene [CAS # 30628-31-0]. (Scheme 2, **1b**) Following general procedure **A** with 2-bromo-1,3,5-trimethylbenzene (151 μL, 1.00 mmol), cyclopentyl zinc bromide solution (1.94 mL, 1.20 mmol, 0.617 M in THF), and THF (2.06 mL) at room temperature for 2 h, the crude product was purified via the Biotage SP4 (silica-packed SNAP 10 g; 0-1% ethyl acetate/hexanes) to provide the title compound as a colorless oil (179 mg, 95% yield). ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.04 (s, 2H), 3.69 (quintet, J = 9.6 Hz, 1H), 2.56 (s, 6H), 2.45 (s, 3H), 2.19-2.02 (m, 6H), 1.96-1.86 (m, 2H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ: 138.6, 136.6 (2C), 134.9, 130.2 (2C), 40.4, 31.2 (2C), 27.3 (2C), 21.5 (2C), 20.8 ppm. IR (neat, cm^{-1}): 2946, 2868, 1617, 1457, 1375, 1025, 850, 571. Anal. Calcd. for $C_{14}H_{20}$: C, 89.29; H, 10.71. Found: C, 89.53; H, 10.95.



1c

3-Cyclohexyl-2-fluoro-5-(trifluoromethyl)pyridine. (Scheme 2, **1c**) Following general procedure **B** with 3-chloro-2-fluoro-5-(trifluoromethyl)pyridine (131 μL, 1.00 mmol), cyclohexyl zinc iodide solution (2.40 mL, 1.20 mmol, 0.500 M in THF), $Pd(OAc)_2$ (4.50 mg, 0.0200 mmol), CPhos **L6** (17.5 mg, 0.0400 mmol), and toluene (1.60 mL) at 60 °C for 1 h, the reaction was quenched by saturated ammonium chloride solution instead of hydrochloric acid (1N). The crude product was purified via the Biotage SP4 (silica-packed SNAP 25 g; 0-6% ethyl acetate/hexanes) to provide the title compound as a yellow oil (208 mg, 84% yield). ^{1}H NMR (300 MHz, $CDCl_3$) δ: 8.33 (s, 1H), 7.84 (d, J = 5.7 Hz, 2H), 2.88-2.78 (m, 1H), 1.98-1.20 (m, 10H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ: 165.0, 161.7, 142.6, 142.5, 142.3, 142.3, 135.6, 135.6, 135.5, 135.5, 135.4, 135.4, 130.4, 130.0, 128.9, 125.9, 125.8, 125.5, 125.4, 125.2, 125.0, 124.9, 124.6, 124.5, 121.6, 118.0, 37.3, 32.5, 26.5, 25.9 ppm (observed complexity due to F-C splitting; definitive assignments have not been made). IR (neat, cm^{-1}): 2933, 2857, 1599, 1452, 1416, 1338, 1264, 1133, 1009, 927, 772, 631, 619. Anal. Calcd. for $C_{12}H_{13}F_4N$: C, 58.30; H, 5.30. Found: C, 58.50; H, 5.43.

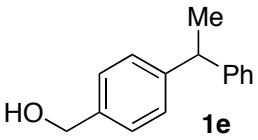


1d

***tert*-Butyl 4-(2-cyanophenyl)piperidine-1-carboxylate [CAS # 256951-72-1].**[15] (Scheme 2, **1d**) Following general procedure **B** with 2-chloro-benzonitrile (138 mg, 1.00 mmol), (1-(*tert*-butoxycarbonyl)piperidin-4-yl)zinc iodide solution (2.64 mL, 1.20 mmol, 0.455 M in THF), and toluene (1.36 mL) at 60 °C for 30 min, the reaction was quenched by saturated ammonium chloride solution instead of hydrochloric acid (1N). The crude product was purified via the Biotage SP4 (silica-packed SNAP 25 g; 0-50% ethyl acetate/hexanes) to provide the title compound as a light yellow solid (260 mg, 91% yield). The isomeric products were not observed by GC and ^{1}H NMR analysis. m.p. 83-85 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.62 (d, J = 7.5 Hz, 1H), 7.56 (t, J = 7.5 Hz, 1H), 7.33 (d, J =

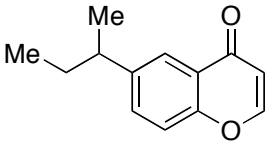
[15] Barrow, J. C. et al. *J. Med. Chem.* **2000**, *43*, 2703.

7.5 Hz, 1H), 7.30 (t, *J* = 7.5 Hz, 1H), 4.26 (br s, 2H), 3.12 (tt, *J* = 12.0, 3.6 Hz, 1H), 2.86 (br t, *J* = 12.0 Hz, 2H), 1.85 (br d, *J* = 12.0 Hz, 2H), 1.70-1.56 (m, 2H), 1.48 (s, 9H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ: 154.4, 148.9, 133.0, 132.9, 126.7, 126.3, 117.7, 111.7, 79.3, 43.8, 40.7 (2C), 32.2 (2C), 28.3 (3C) ppm. IR (neat, cm^{-1}): 2976, 2934, 2854, 2223, 1695, 1600, 1426, 1366, 1326, 1281, 1235, 1166, 1126, 1064, 1013, 986, 940, 888, 862, 764, 736, 520.



1e

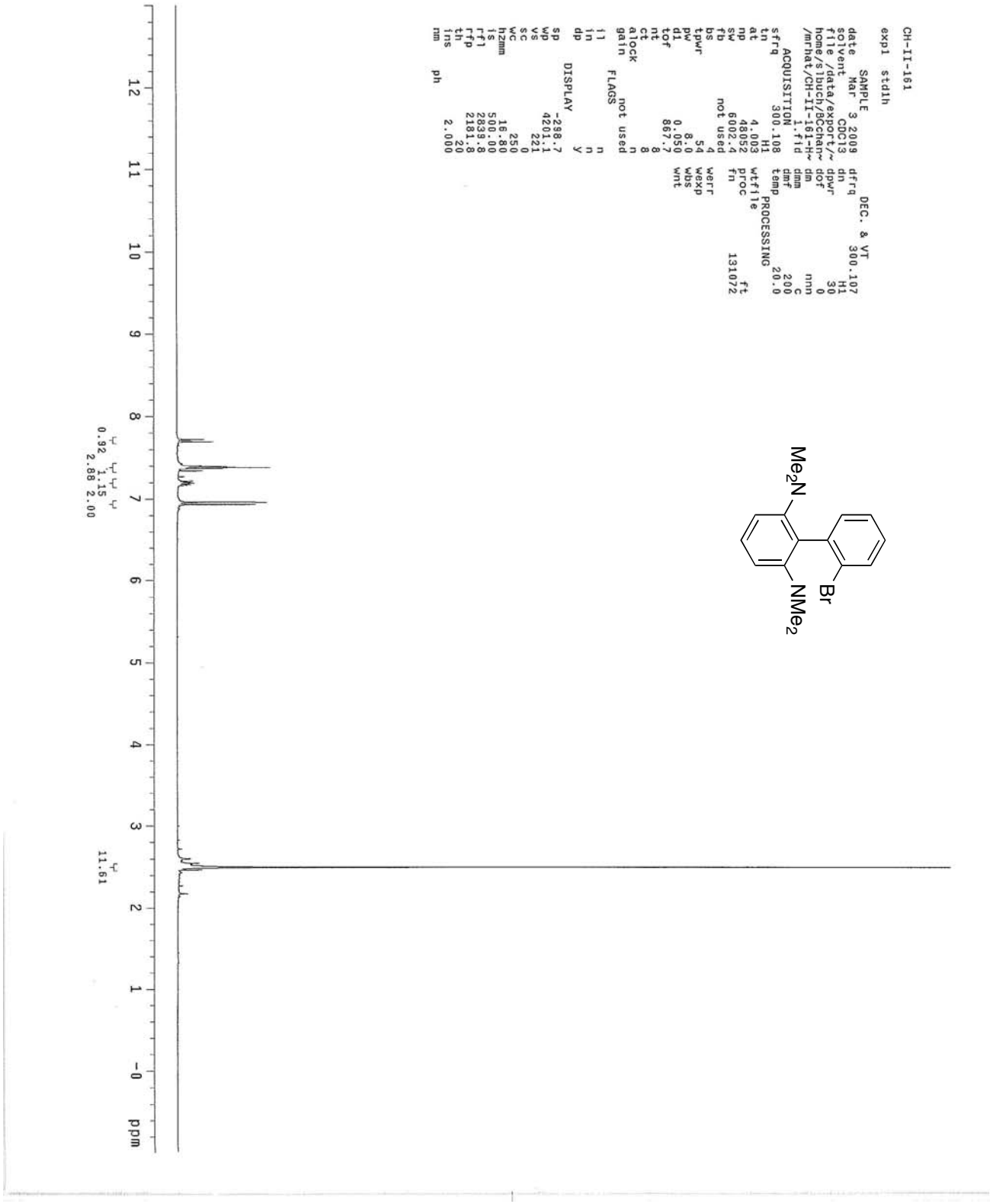
(4-(1-Phenylethyl)phenyl)methanol [CAS # 117455-21-7]. (Scheme 2, **1e**) Following general procedure **B** with 4-bromobenzyl alcohol (187 mg, 1.00 mmol), (1-phenylethyl)zinc chloride solution (2.16 mL, 1.50 mmol, 0.694 M in THF), and toluene (1.84 mL) at room temperature for 1 h, except that the alkylzinc solution was added dropwise over 30 min. The crude product was purified via the Biotage SP4 (silica-packed SNAP 10 g; 0-50% ethyl acetate/hexanes) to provide the title compound as a colorless oil (183 mg, 86% yield). The isomeric linear product was not observed by GC and ^{1}H NMR analysis. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.39-7.22 (m, 9H), 4.62 (s, 2H), 4.22 (q, *J* = 7.2 Hz, 1H), 2.42 (br s, 1H), 1.71 (d, *J* = 7.2 Hz, 3H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ: 146.4, 145.8, 138.7, 128.5 (2C), 127.8 (2C), 127.6 (2C), 127.3 (2C), 126.1, 64.8, 44.5, 21.9 ppm. IR (neat, cm^{-1}): 3324, 3024, 2966, 2872, 1560, 1487, 1457, 1419, 1209, 1010, 794, 700.

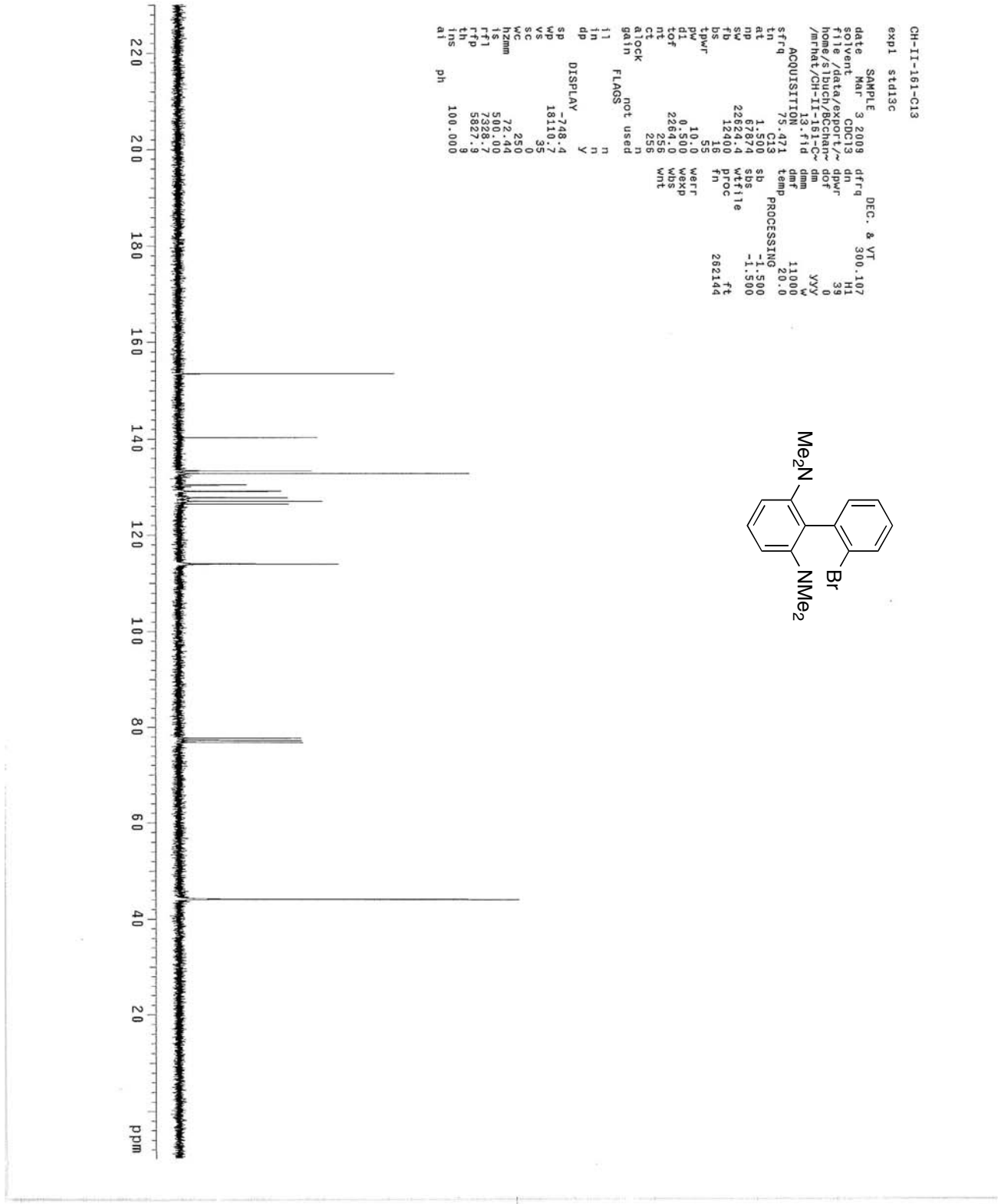


1f

6-*sec*-Butyl-4*H*-chromen-4-one. (Scheme 2, **1f**) Following general procedure **A** with 6-bromochromone (225 mg, 1.00 mmol), *sec*-butylzinc bromide solution (2.21 mL, 1.20 mmol, 0.543 M in THF), and toluene (1.80 mL) at room temperature for 1 h, the crude product was purified via the Biotage SP4 (silica-packed SNAP 25 g; 0-60% ethyl acetate/hexanes) to provide a mixture of products as a yellow oil (180 mg, 89% yield). The ratio of the branched to the linear cross-coupled product was determined to be 23:1 by GC and ^{1}H NMR analysis. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 8.00 (d, *J* = 2.1 Hz, 1H), 7.84 (d, *J* = 6.3 Hz, 1H), 7.50 (dd, *J* = 8.7, 2.1 Hz, 1H), 7.39 (d, *J* = 8.7 Hz, 1H), 6.33 (d, *J* = 6.3 Hz, 1H), 2.72 (sextet, *J* = 7.2 Hz, 1H), 1.63 (dq, *J* = 7.5, 7.2 Hz, 2H), 1.27 (d, *J* = 7.2 Hz, 3H), 0.80 (d, *J* = 7.5 Hz, 3H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ: 178.1, 155.4, 145.2, 133.3, 124.8, 123.5, 118.2, 113.0, 112.9, 41.5, 31.2, 22.0, 12.3 ppm. IR (neat, cm^{-1}): 3073, 2962, 2930, 2874, 1653, 1617, 1482, 1448, 1395, 1358, 1313, 1256, 1202, 1135, 1028, 866, 833, 585, 536. Anal. Calcd. for $C_{13}H_{14}O_2$: C, 77.20; H, 6.98. Found: C, 76.37; H, 6.88.

III. Attached NMR Spectra.




CH-II-161-C13
exp1 std13c
SAMPLE
date Mar 3 2009
solvent CDCl3
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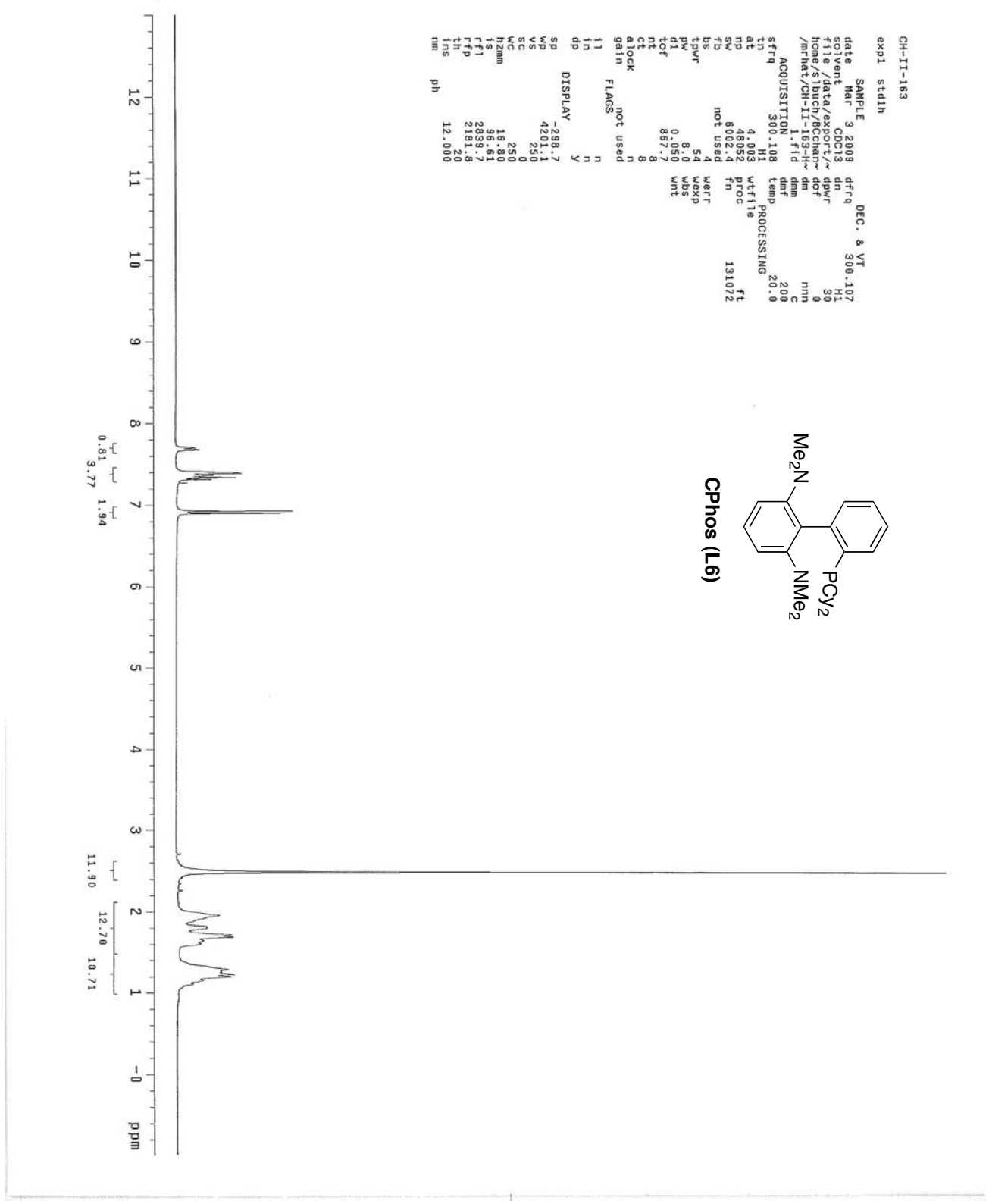
home/sibuch/BCchan~
/mrhat/CH-II-161-C~
ACQUISITION
sfrq 75.471 dfrq 300.107
tn C13 dn H1
at 1.500 dpwr 39
np 67874 dof 0
sw 22624.4 dm yyy
fb 12400 dmm w
bs 16 dmf 11000
tpwr 55 temp 20.0
pw 10.0 PROCESSING
d1 0.500 wexp -1.500
tof 2264.0 wbs sbs -1.500
nt 256 wnt
ct 256
alock n
gain FLAGS not used
il n
in n
dp y
DISPLAY
sp -748.4
wp 18110.7
vs 35
sc 0
wc 250
hzmm 72.44
is 500.00
rfl 7328.7
rfp 5827.9
th
ins 100.000
at ph
DEC. & VT
dfrq 300.107
dn H1
dpwr 39
dof 0
dm yyy
dmm w
dmf 11000
temp 20.0
PROCESSING
sb -1.500
sbs -1.500
wtfile
proc ft
fn 262144
werr
wexp
wbs
wnt
Me2N
NMe2
Br
220
200
180
160
140
120
100
80
60
40
20
ppm


CH-II-163
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solvent CDCl3 dn H1
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home/sibuch/BCchan~ dof 0
/mrhat/CH-II-163-H~ dm nnn
1.fid dmm c
ACQUISITION dmf 200
sfrq 300.108 temp 20.0
tn H1 PROCESSING
at 4.003 wtfile
np 48052 proc ft
sw 6002.4 fn 131072
fb not used
bs 4
tpwr 54 werr
pw 8.0 wexp
d1 0.050 wbs
tof 867.7 wnt
nt 8
ct 8
alock n
gain FLAGS not used
il n
in n
dp y
DISPLAY
sp -298.7
wp 4201.1
vs 250
sc 0
wc 250
hzmm 16.80
is 96.61
rfl 2839.7
rfp 2181.8
th 20
ins 12.000
nm ph
Me2N
PCy2
NMe2
CPhos (L6)
0.81
3.77
1.94
11.90
12.70
10.71
ppm

CH-II-163-C13

exp1 std13c

SAMPLE		DEC. & VT	
date	Mar 3 2009	dfrq	300.107
solvent	CDCl3	dn	H1
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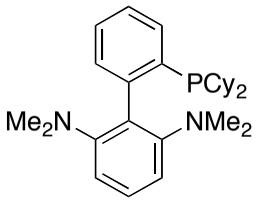
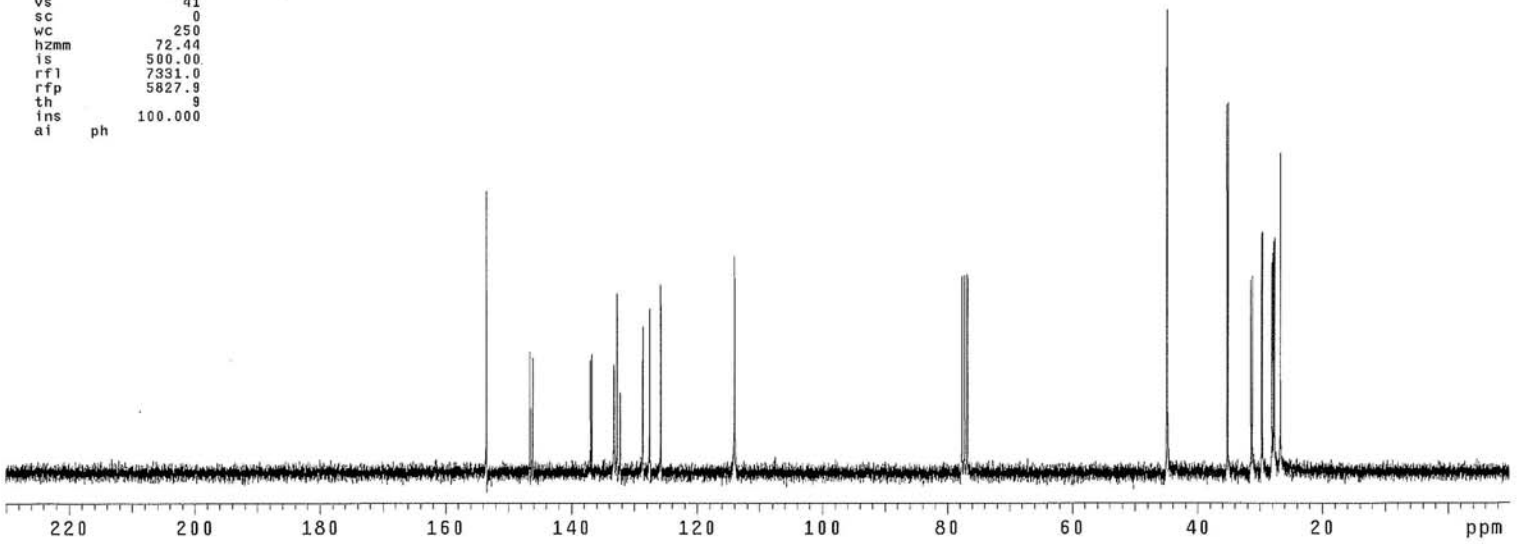
home/slbuch/BCchan~		dof	0
/mrhat/CH-II-163-C~		dm	yyy
	13.fid	dmm	w
ACQUISITION		dmf	11000
sfrq	75.471	temp	20.0
tn	C13	PROCESSING	
at	1.500	sb	-1.500
np	67874	sbs	-1.500
sw	22624.4	wtfile	
fb	12400	proc	ft
bs	16	fn	262144
tpwr	55		
pw	10.0	werr	
d1	0.500	wexp	
tof	2264.0	wbs	
nt	256	wnt	
ct	256		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-750.6		
wp	18110.7		
vs	41		
sc	0		
wc	250		
hzmm	72.44		
is	500.00		
rfl	7331.0		
rfp	5827.9		
th	9		
ins	100.000		
ai	ph		



CPhos (L6)



CH-II-163-P31

exp1 s2pul

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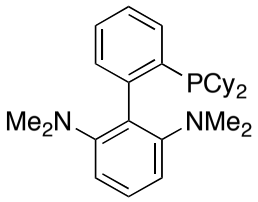
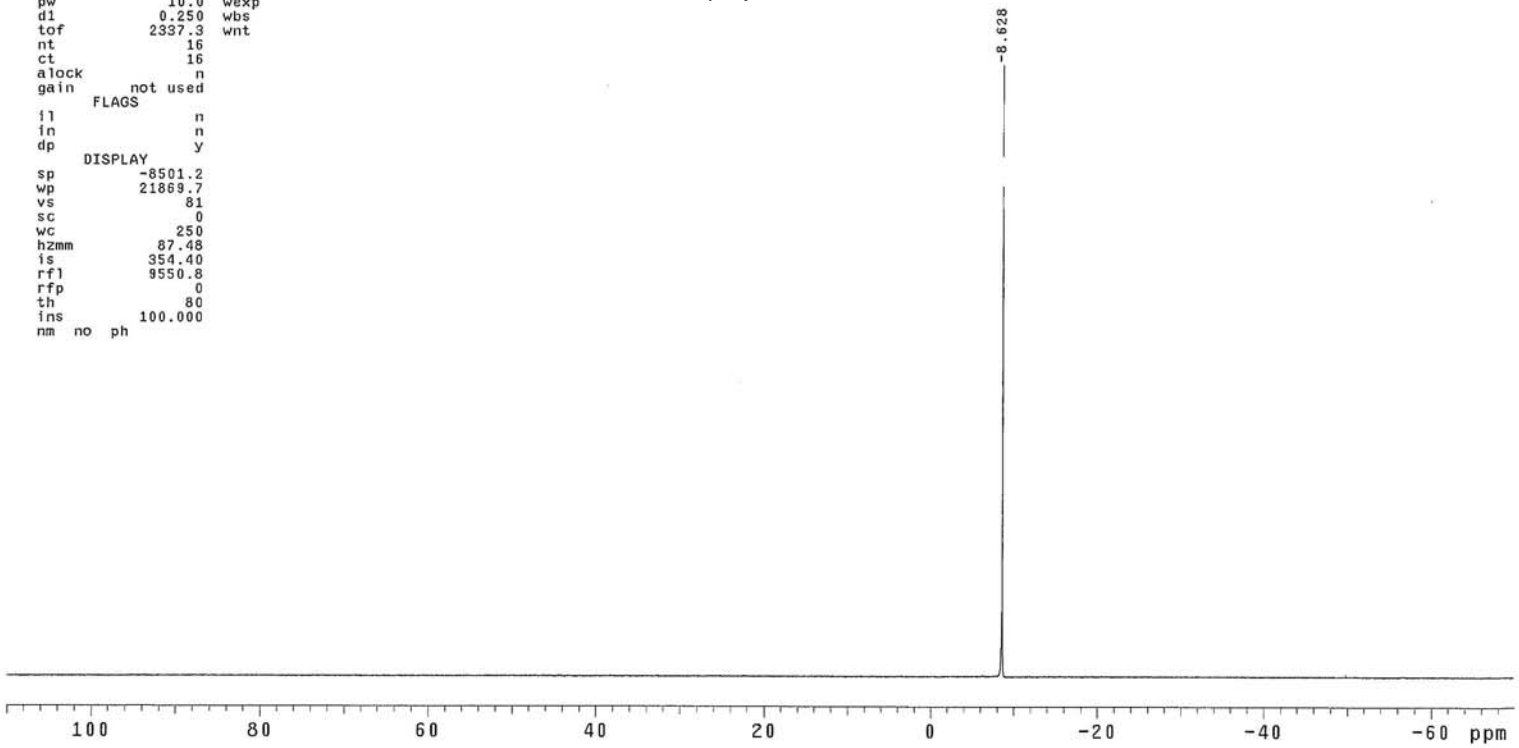
/mrhat/CH-II-163-P~		dm	yyy
	31.fid	dmm	w
ACQUISITION		dmf	11000
sfrq	121.487	temp	20.0
tn	P31	PROCESSING	
at	1.261	lb	1.00
np	61204	wtfile	
sw	24271.8	proc	ft
fb	13400	fn	not used
bs	16		
tpwr	62	werr	
pw	10.0	wexp	
d1	0.250	wbs	
tof	2337.3	wnt	
nt	16		
ct	16		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-8501.2		
wp	21869.7		
vs	81		
sc	0		
wc	250		
hzmm	87.48		
is	354.40		
rfl	9550.8		
rfp	0		
th	80		
ins	100.000		
nm no ph			



CPhos (L6)



CH-II-103

exp1 std1h

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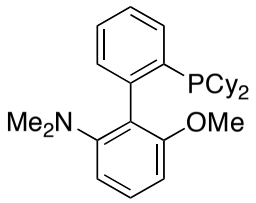
/mrhat/CH-II-103-H~		dm	nnn
	1.fid	dmm	c
ACQUISITION		dmf	200
sfrq	300.108	temp	20.0
tn	H1	PROCESSING	
at	4.003	wtfile	
np	48052	proc	ft
sw	6002.4	fn	131072
fb	not used		
bs	4	werr	
tpwr	54	wexp	
pw	8.0	wbs	
d1	0.050	wnt	
tof	867.7		
nt	8		
ct	8		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-299.1		
wp	4201.1		
vs	153		
sc	0		
wc	250		
hzmm	16.80		
is	146.11		
rfl	2840.2		
rfp	2181.8		
th	20		
ins	1.000		
nm	ph		



L5

CH-II-103-C13

exp1 std13c

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solvent	CDCl3	dn	H1
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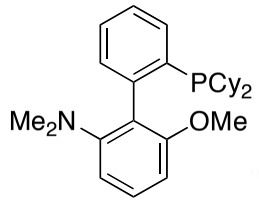
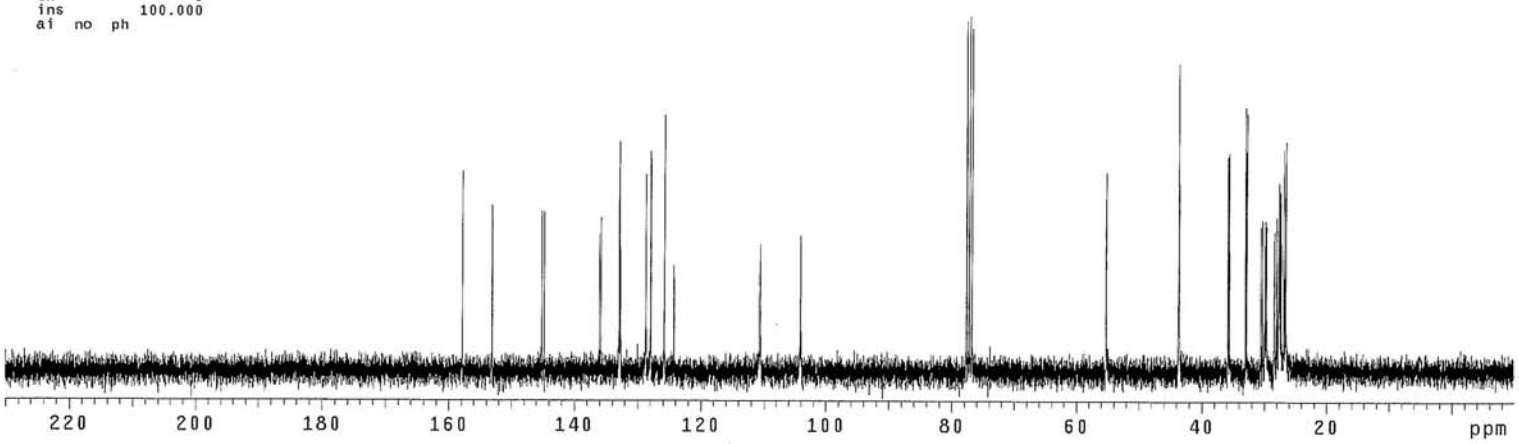
/mrhat/CH-II-103-C~		dm	yyy
	13.fid	dmm	w
ACQUISITION		dmf	11000
sfrq	75.471	temp	20.0
tn	C13	PROCESSING	
at	1.500	sb	-1.500
np	67874	sbs	-1.500
sw	22624.4	wtfile	
fb	12400	proc	ft
bs	16	fn	262144
tpwr	55		
pw	10.0	werr	
d1	0.500	wexp	
tof	2264.0	wbs	
nt	256	wnt	
ct	256		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-748.4		
wp	18110.7		
vs	71		
sc	0		
wc	250		
hzmm	72.44		
is	500.00		
rfl	7328.7		
rfp	5827.9		
th	9		
ins	100.000		
ai no ph			



L5



CH-II-103-P31

exp1 s2pul

SAMPLE		DEC. & VT	
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solvent	CDCl3	dn	H1
file	/data/export/~	dpwr	39
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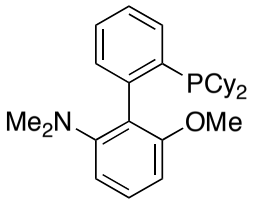
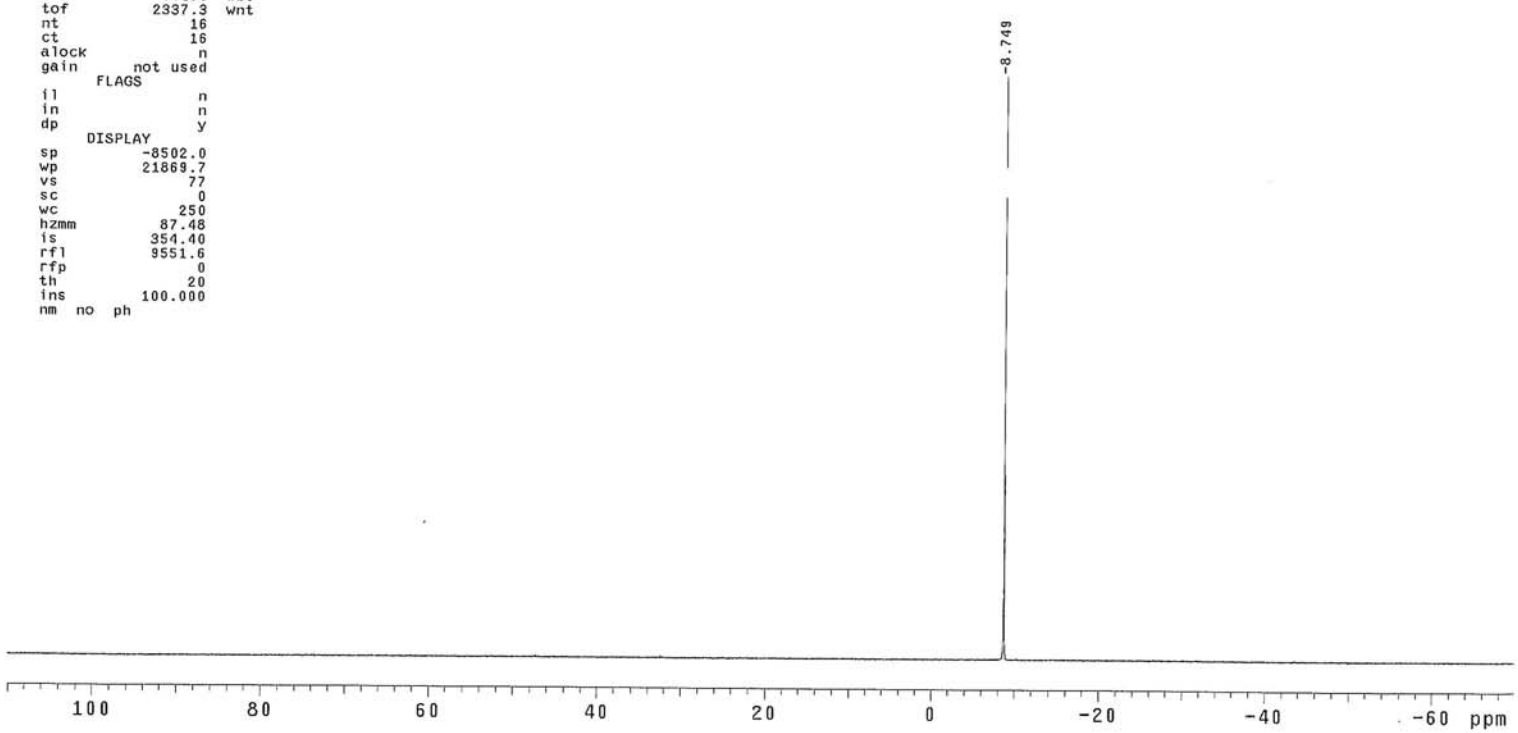
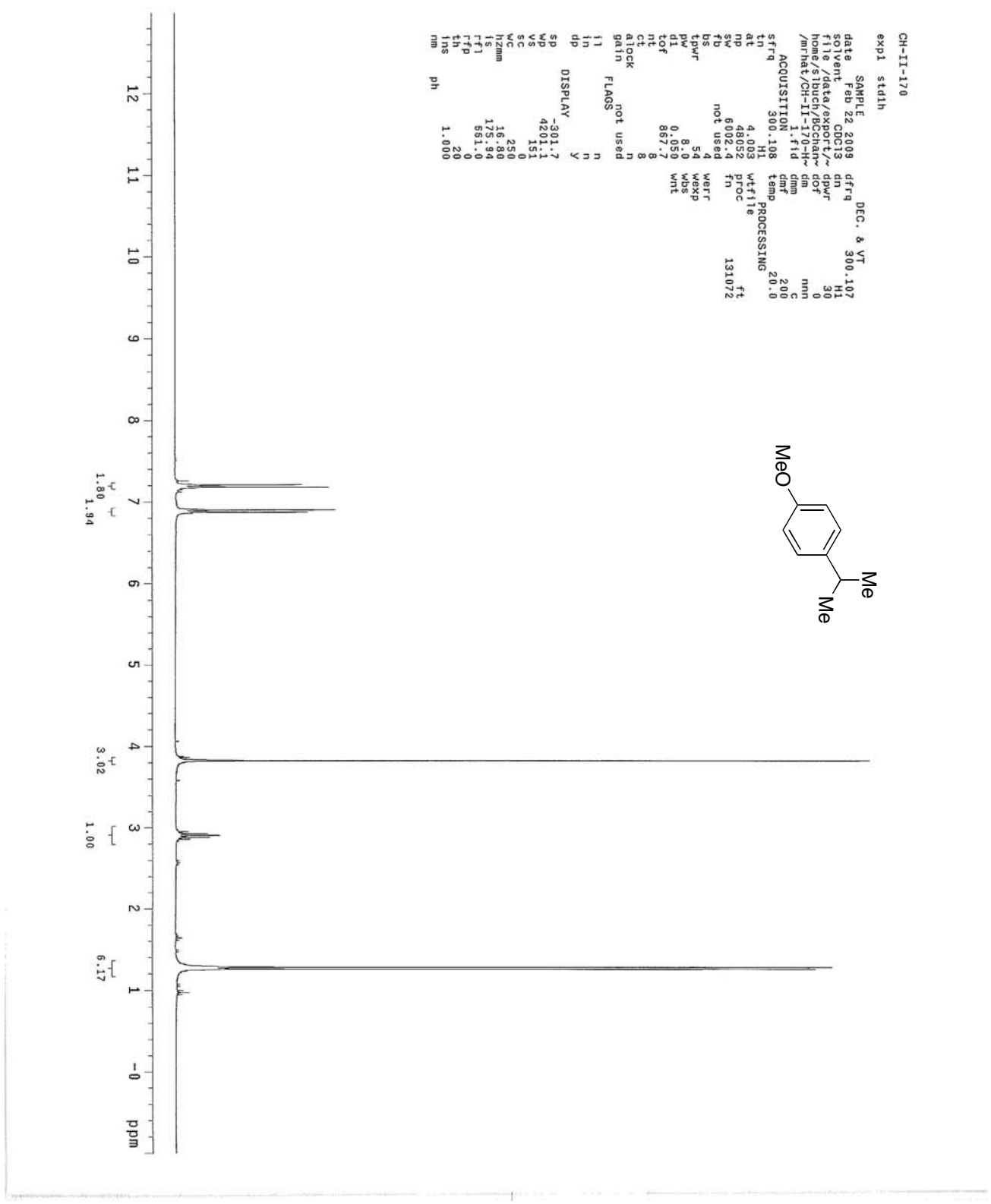
/mrhat/CH-II-103-P~		dm	yyy
	31.fid	dmm	w
ACQUISITION		dmf	11000
sfrq	121.487	temp	20.0
tn	P31	PROCESSING	
at	1.261	lb	1.00
np	61204	wtfile	
sw	24271.8	proc	ft
fb	13400	fn	not used
bs	16		
tpwr	62	werr	
pw	10.0	wexp	
d1	0.250	wbs	
tof	2337.3	wnt	
nt	16		
ct	16		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-8502.0		
wp	21869.7		
vs	77		
sc	0		
wc	250		
hzmm	87.48		
is	354.40		
rfl	9551.6		
rfp	0		
th	20		
ins	100.000		
nm no ph			



L5




CH-II-170
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date Feb 22 2009
solvent CDCl3
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home/slbuch/BCchan~
/mrhat/CH-II-170-H~
1.fid
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sfrq 300.108
tn H1
at 4.003
np 48052
sw 6002.4
fb not used
bs 4
tpwr 54
pw 8.0
d1 0.050
tof 867.7
nt 8
ct 8
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -301.7
wp 4201.1
vs 151
sc 0
wc 250
hzmm 16.80
ls 125.94
rfl 661.0
rfp 0
th 20
ins 1.000
nm
ph
DEC. & VT
dfrq 300.107
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
temp 20.0
PROCESSING
wtfile
proc ft
fn 131072
werr
wexp
wbs
wnt
MeO
Me
Me
12
11
10
9
8
7
6
5
4
3
2
1
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ppm
1.80
1.94
3.02
1.00
6.17

CH-II-170-C13

exp1 std13c

SAMPLE		DEC. & VT	
date	Feb 22 2009	dfrq	300.107
solvent	CDCl3	dn	H1
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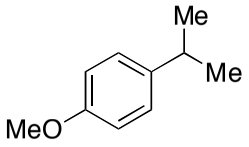
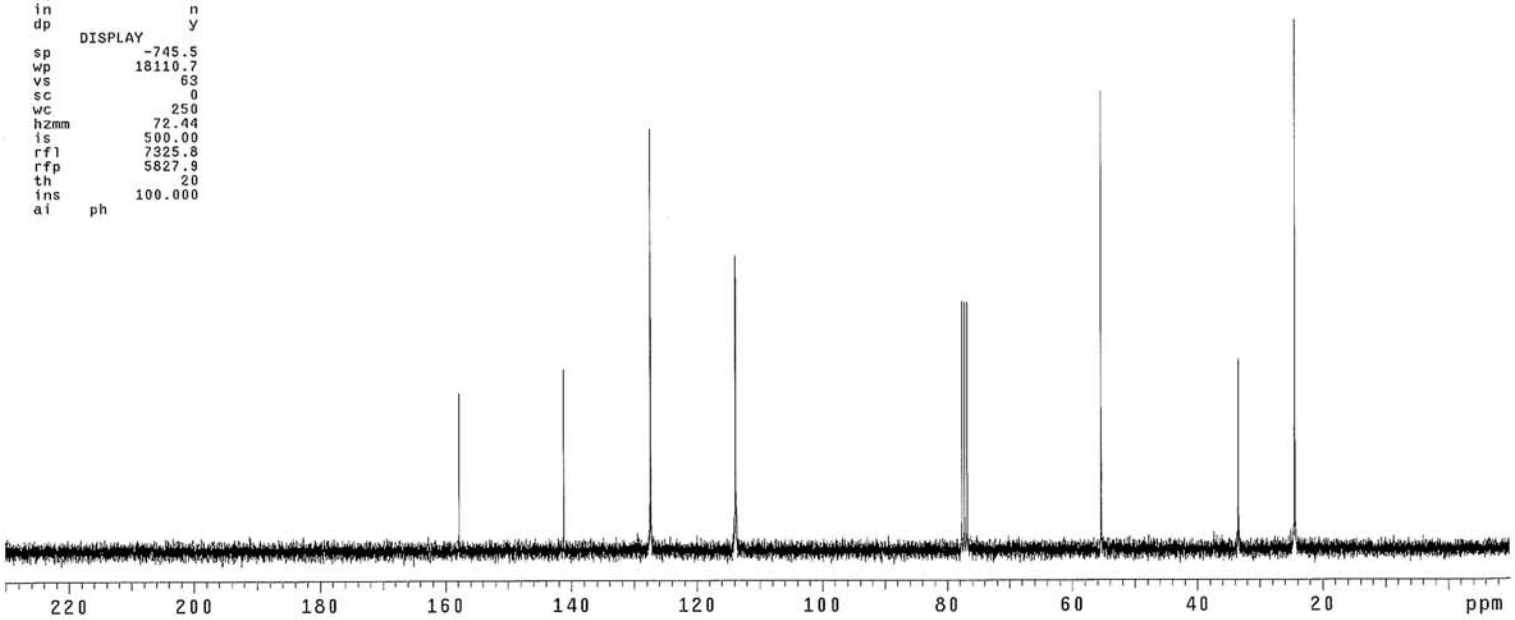
/mrhat/CH-II-170-C~		dm	yyy
	13.fid	dmm	w
ACQUISITION		dmf	11000
sfrq	75.471	temp	20.0
tn	C13	PROCESSING	
at	1.500	sb	-1.500
np	67874	sbs	-1.500
sw	22624.4	wtfile	
fb	12400	proc	ft
bs	8	fn	262144
tpwr	55		
pw	10.0	werr	
d1	0.500	wexp	
tof	2264.0	wbs	
nt	512	wnt	
ct	512		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-745.5		
wp	18110.7		
vs	63		
sc	0		
wc	250		
hzmm	72.44		
is	500.00		
rfl	7325.8		
rfp	5827.9		
th	20		
ins	100.000		
ai ph			





CH-II-165

exp1 std1h

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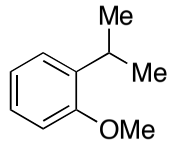
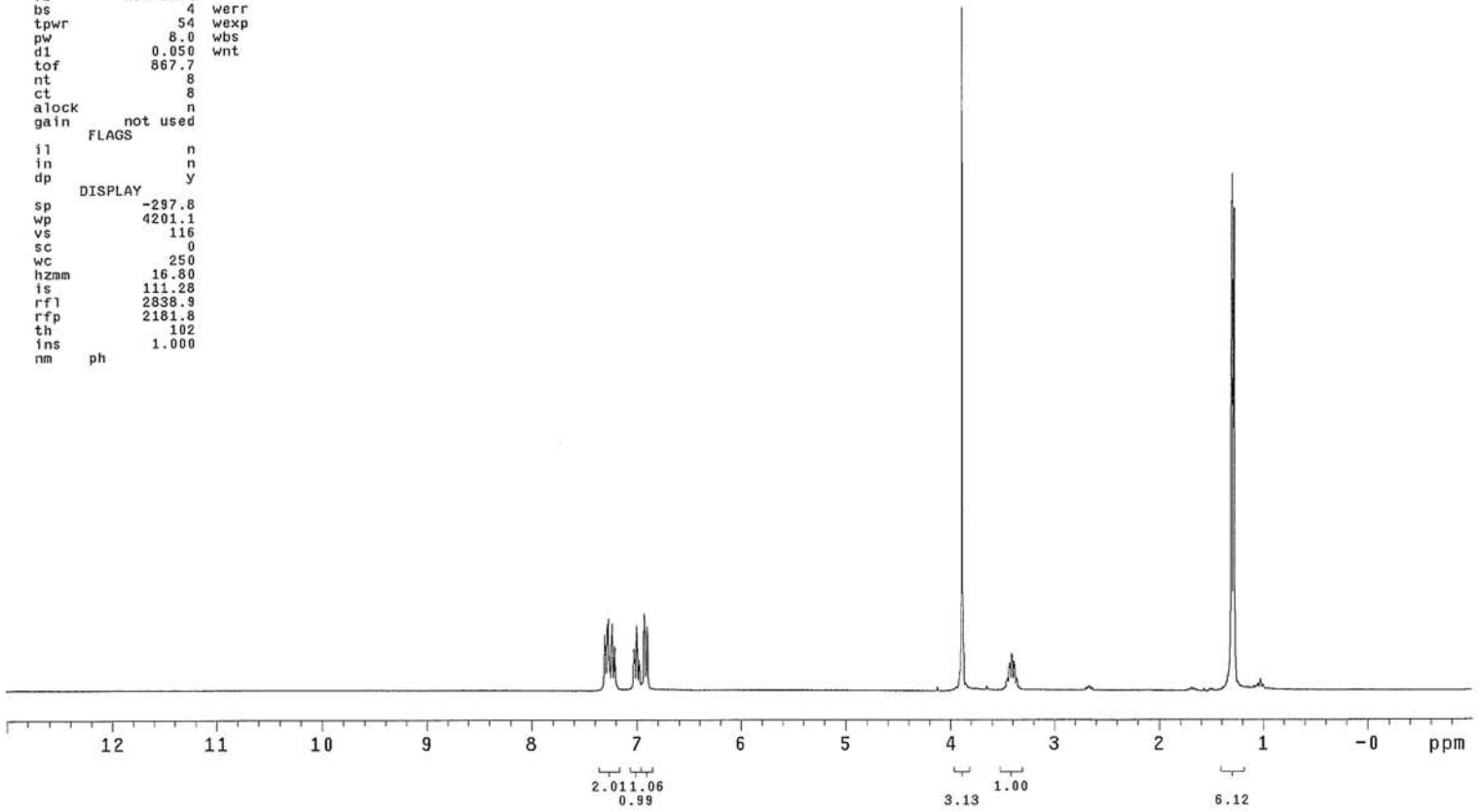
	1.fid	dmm	c
ACQUISITION		dmf	200
sfrq	300.108	temp	20.0
tn	H1	PROCESSING	
at	4.003	wtfile	
np	48052	proc	ft
sw	6002.4	fn	131072
fb	not used		
bs	4	werr	
tpwr	54	wexp	
pw	8.0	wbs	
d1	0.050	wnt	
tof	867.7		
nt	8		
ct	8		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-297.8		
wp	4201.1		
vs	116		
sc	0		
wc	250		
hzmm	16.80		
is	111.28		
rfl	2838.9		
rfp	2181.8		
th	102		
ins	1.000		
nm	ph		





CH-II-170-C13

exp1 std13c

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solvent	CDCl3	dn	H1
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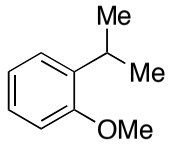
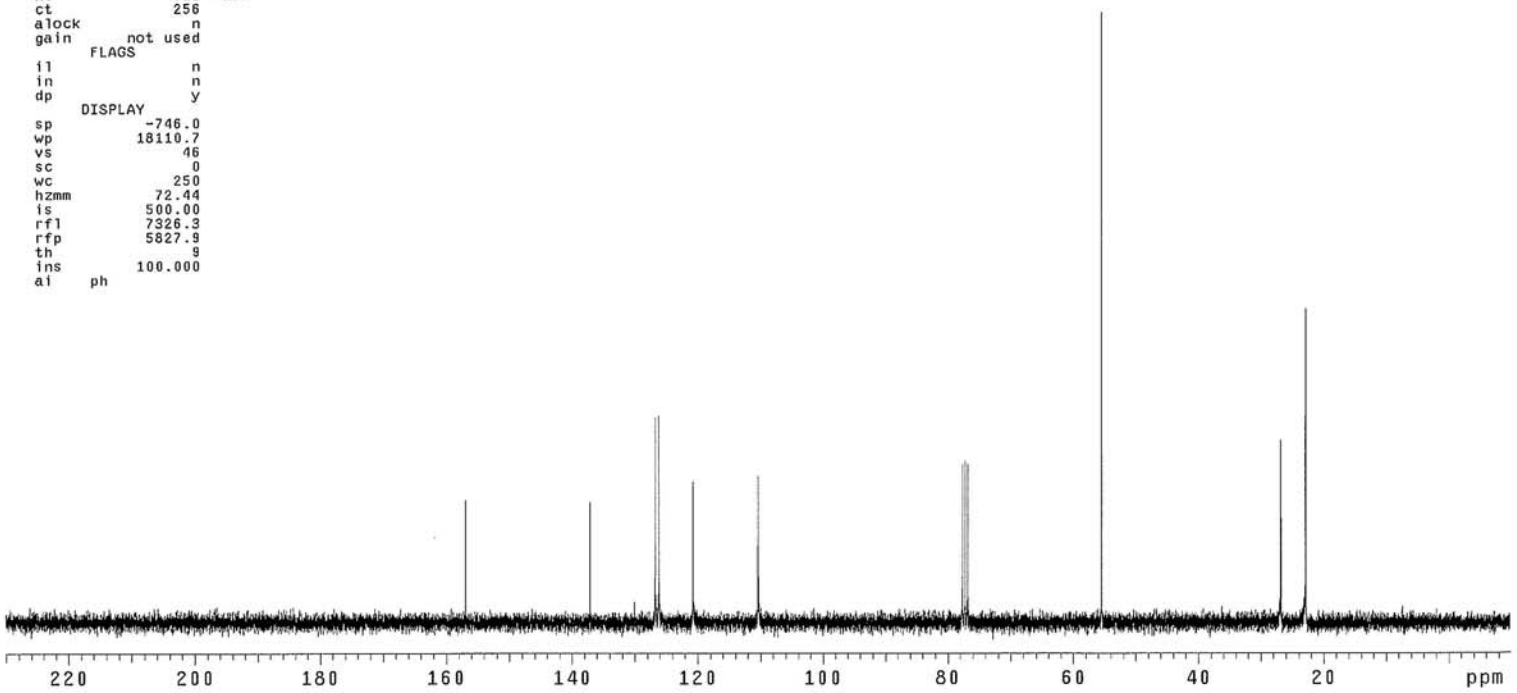
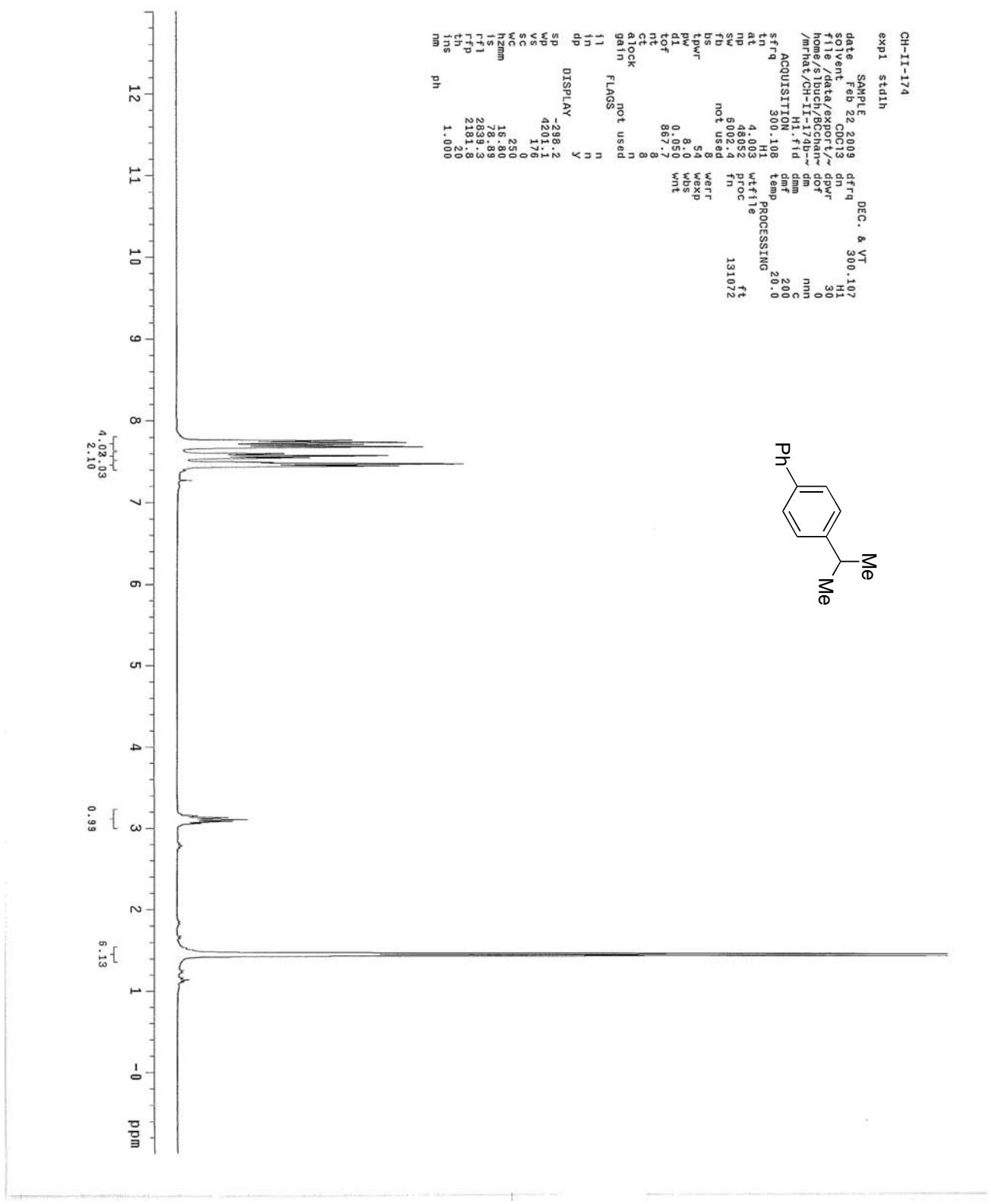
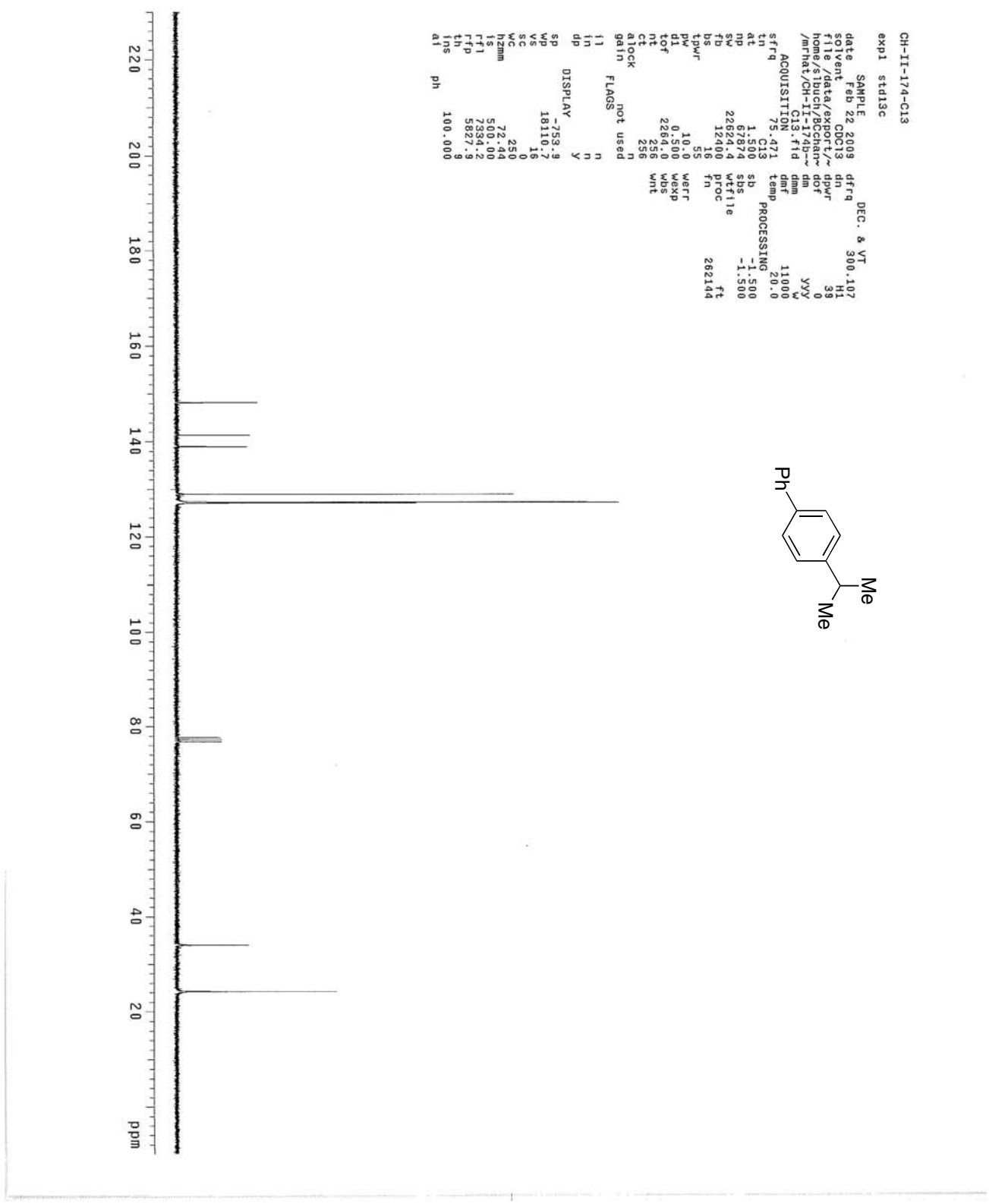
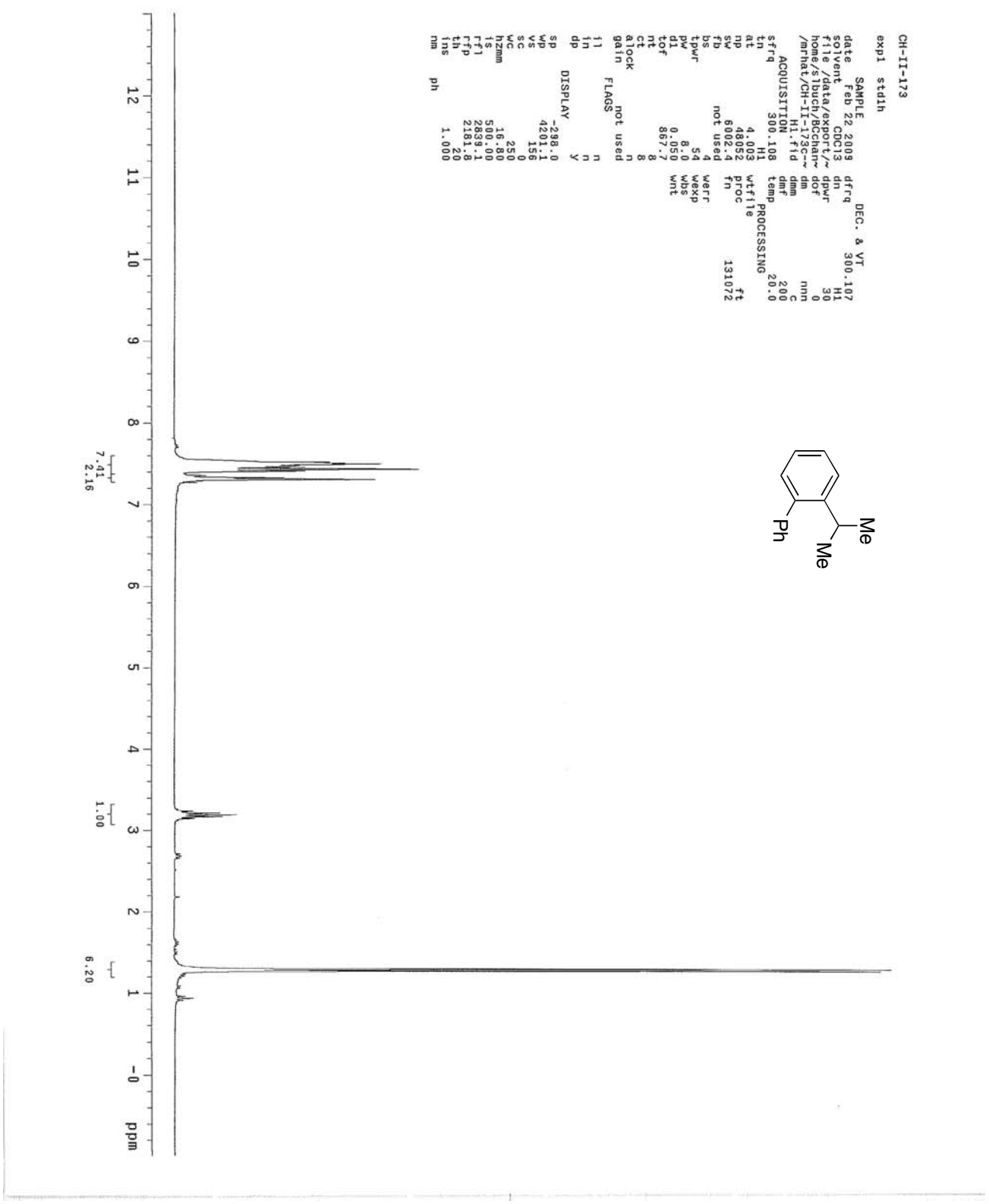
/mrhat/CH-II-165-C~		dm	yyy
	13.fid	dmm	w
ACQUISITION		dmf	11000
sfrq	75.471	temp	20.0
tn	C13	PROCESSING	
at	1.500	sb	-1.500
np	67874	sbs	-1.500
sw	22624.4	wtfile	
fb	12400	proc	ft
bs	32	fn	262144
tpwr	55		
pw	10.0	werr	
d1	0.500	wexp	
tof	2264.0	wbs	
nt	512	wnt	
ct	256		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-746.0		
wp	18110.7		
vs	46		
sc	0		
wc	250		
hzmm	72.44		
is	500.00		
rfl	7326.3		
rfp	5827.9		
th	9		
ins	100.000		
ai ph			






CH-II-174
exp1 std1h
SAMPLE
date Feb 22 2009
solvent CDCl3
file /data/export/~
home/slbuch/BCchan~
/mrhat/CH-II-174b~
ACQUISITION
sfrq 300.108
tn H1
at 4.003
np 48052
sw 6002.4
fb 8
bs 54
tpwr 8.0
pw 0.050
d1 867.7
tof 8
nt 8
ct
alock n
gain FLAGS not used
il n
in n
dp y
DISPLAY
sp -298.2
wp 4201.1
vs 176
sc 0
wc 250
hzmm 16.80
is 78.89
rfl 2839.3
rfp 2181.8
th 20
ins 1.000
nm ph
DEC. & VT
dfrq 300.107
dn H1
dpwr 30
dcf 0
dm nnn
dmm c
dmf 200
temp 20.0
PROCESSING
wtfile
proc ft
fn 131072
werr
wexp
wbs
wnt
Ph
Me
Me
4.02
2.03
2.10
0.99
6.13
12
11
10
9
8
7
6
5
4
3
2
1
-0
ppm

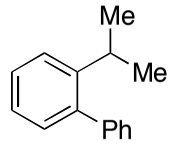

CH-II-174-C13
exp1 std13c
SAMPLE
date Feb 22 2009
solvent CDCl3
file /data/export/~
home/sbuch/BCchan~
/mrhat/CH-II-174b~
C13.fid
ACQUISITION
sfrq 75.471
tn C13
at 1.500
np 67874
sw 22624.4
fb 12400
bs 16
tpwr 55
pw 10.0
d1 0.500
tof 2264.0
nt 256
ct 256
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -753.9
wp 18110.7
vs 16
sc 0
wc 250
hzmm 72.44
is 500.00
rfl 7334.2
rfp 5827.9
th
ins 100.000
al ph
DEC. & VT
dfrq 300.107
dn H1
dpwr 39
dof 0
dm yyy
dmm w
dmf 11000
temp 20.0
PROCESSING
sb -1.500
sbs -1.500
wtfile
proc ft
fn 262144
werr
wexp
wbs
wnt
Ph
Me
Me
220
200
180
160
140
120
100
80
60
40
20
ppm

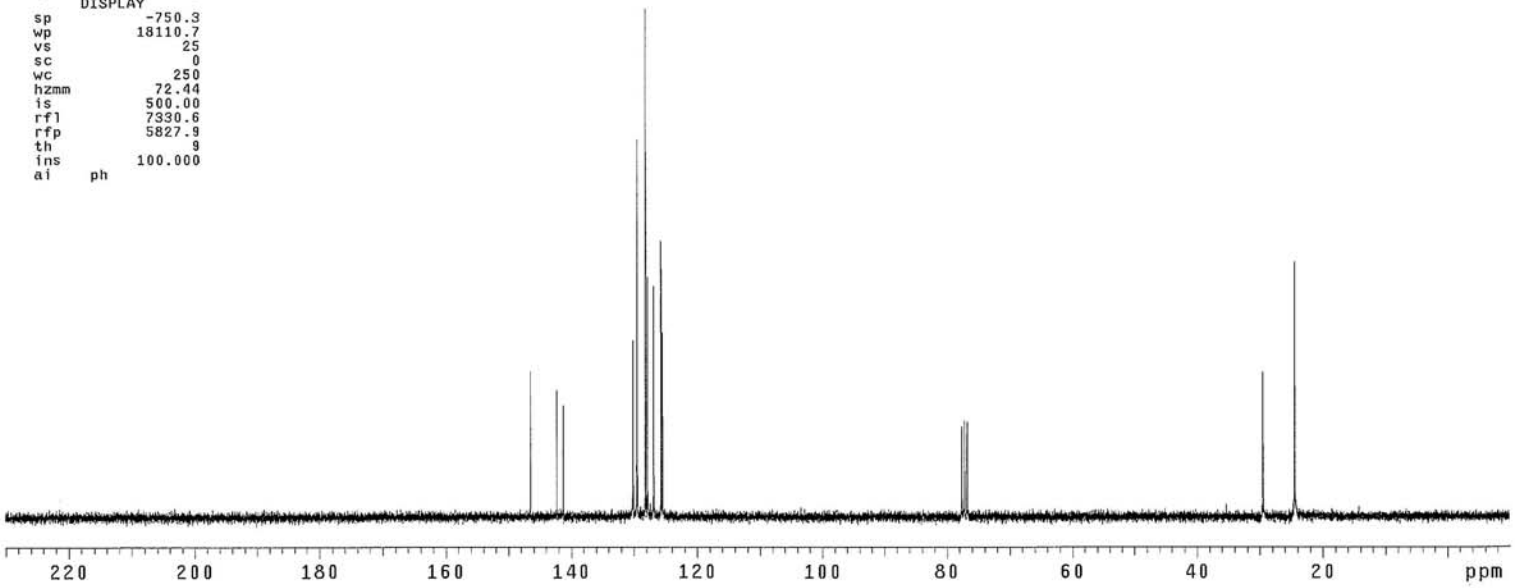

CH-II-173
exp1 stdih
SAMPLE
date Feb 22 2009
solvent CDCl3
file /data/export/~
home/sibuch/BCchan~
/mrhat/CH-II-173c~~
ACQUISITION
sfrq 300.108
tn H1
at 4.003
np 48052
sw 6002.4
fb not used
bs 4
tpwr 54
pw 8.0
d1 0.050
tof 867.7
nt 8
ct 8
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -298.0
wp 4201.1
vs 156
sc 0
wc 250
hzmm 16.80
is 500.00
rfl 2839.1
rfp 2181.8
th 20
ins 1.000
nm
ph
DEC. & VT
dfrq 300.107
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
temp 20.0
PROCESSING
wtfile
proc ft
fn 131072
werr
wexp
wbs
wnt
Me
Me
Ph
7.41
2.16
1.00
6.20
12
11
10
9
8
7
6
5
4
3
2
1
-0
ppm

CH-II-173-C13

exp1 std13c

SAMPLE		DEC. & VT	
date	Feb 22 2009	dfrq	300.107
solvent	CDCl3	dn	H1
file	/data/export/~	dpwr	39
home/slbuch/BCchan~		dof	0
/mrhat/CH-II-173c~		dm	yyy
	C13.fid	dmm	w
ACQUISITION		dmf	11000
sfrq	75.471	temp	20.0
tn	C13	PROCESSING	
at	1.500	sb	-1.500
np	67874	sbs	-1.500
sw	22624.4	wtfile	
fb	12400	proc	ft
bs	16	fn	262144
tpwr	55		
pw	10.0	werr	
d1	0.500	wexp	
tof	2264.0	wbs	
nt	256	wnt	
ct	256		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-750.3		
wp	18110.7		
vs	25		
sc	0		
wc	250		
hzmm	72.44		
is	500.00		
rfl	7330.6		
rfp	5827.9		
th	9		
ins	100.000		
ai	ph		

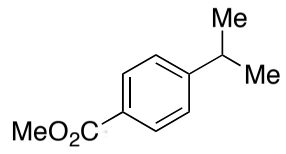


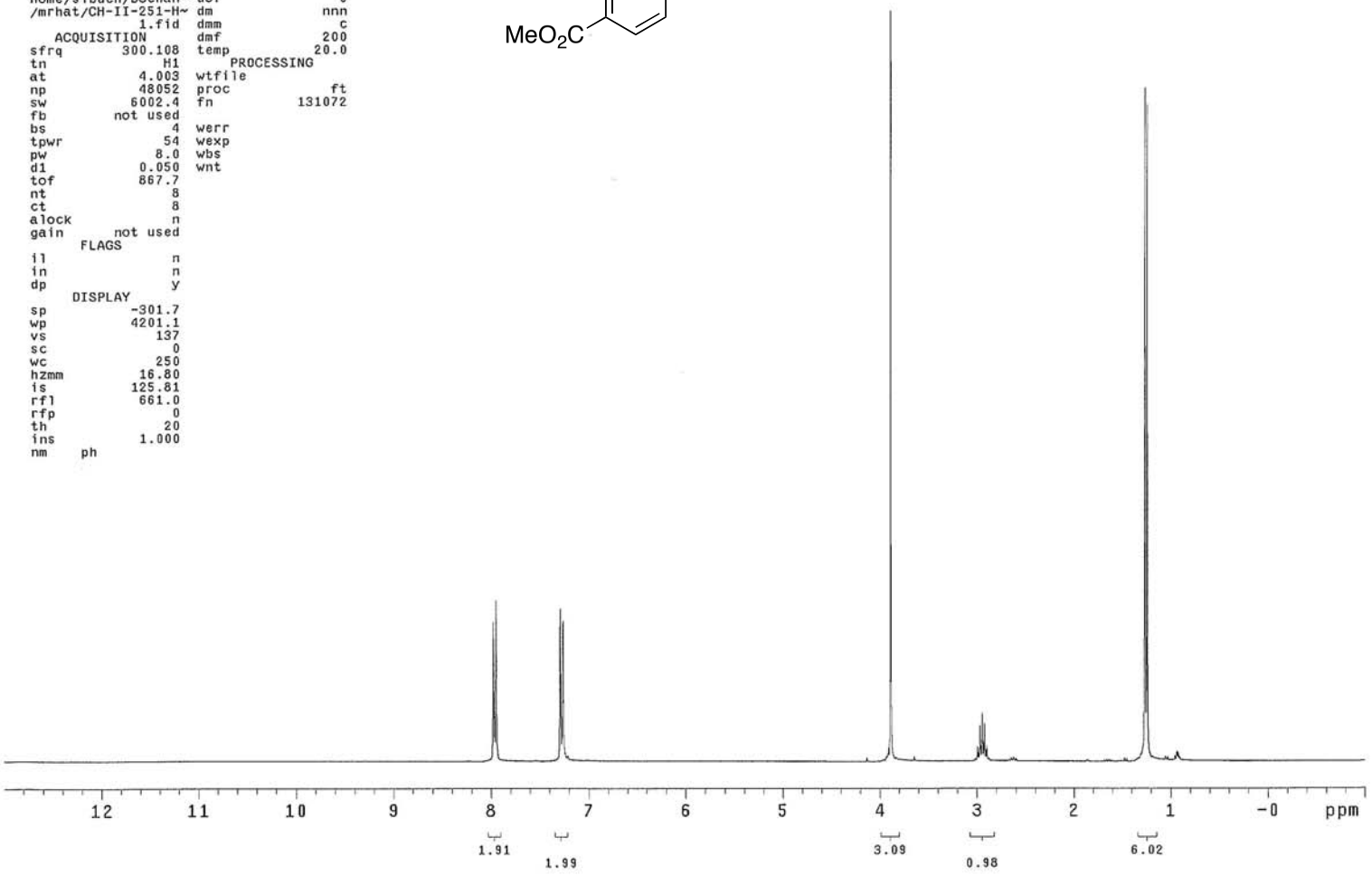


CH-II-251

exp1 std1h

SAMPLE		DEC. & VT	
date	Feb 22 2009	dfrq	300.107
solvent	CDCl3	dn	H1
file	/data/export/~	dpwr	30
home/slbuch/BCchan~		dof	0
/mrhat/CH-II-251-H~		dm	nnn
	1.fid	dmm	c
ACQUISITION		dmf	200
sfrq	300.108	temp	20.0
tn	H1	PROCESSING	
at	4.003	wtfile	
np	48052	proc	ft
sw	6002.4	fn	131072
fb	not used		
bs	4	werr	
tpwr	54	wexp	
pw	8.0	wbs	
d1	0.050	wnt	
tof	867.7		
nt	8		
ct	8		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-301.7		
wp	4201.1		
vs	137		
sc	0		
wc	250		
hzmm	16.80		
is	125.81		
rfl	661.0		
rfp	0		
th	20		
ins	1.000		
nm	ph		

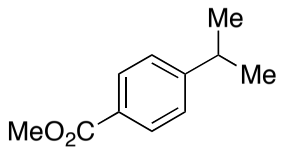


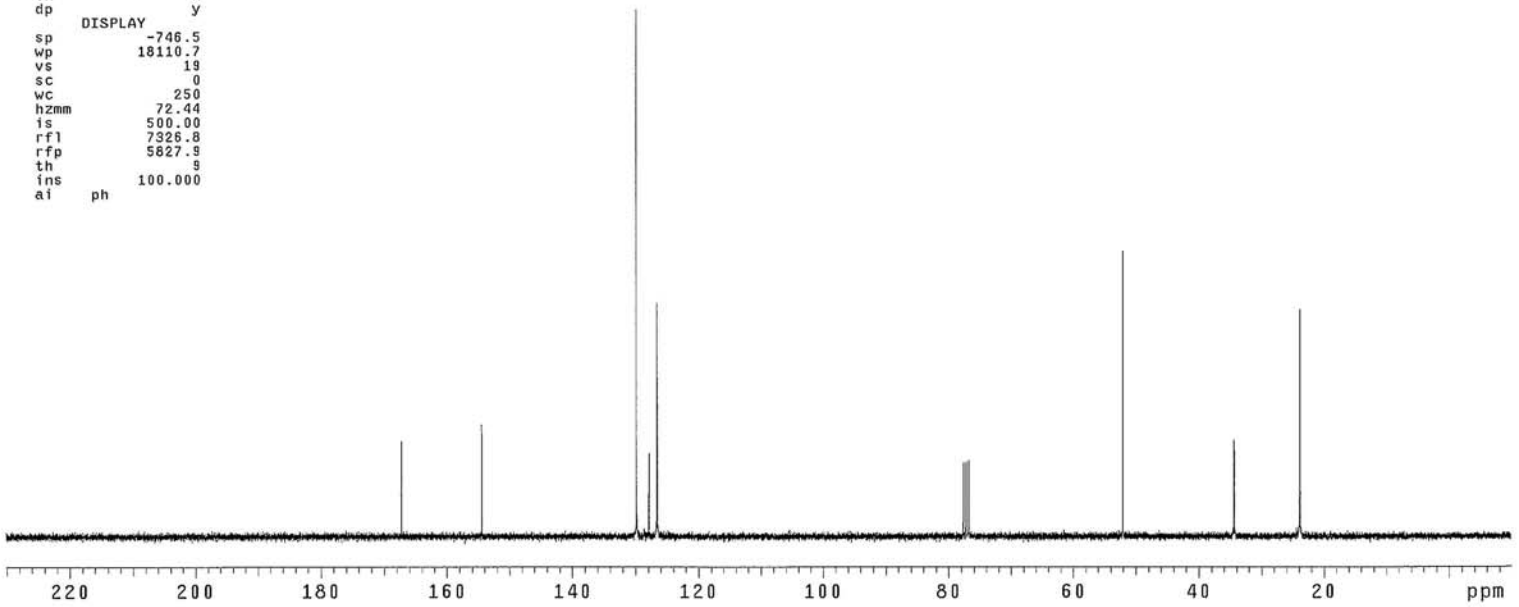


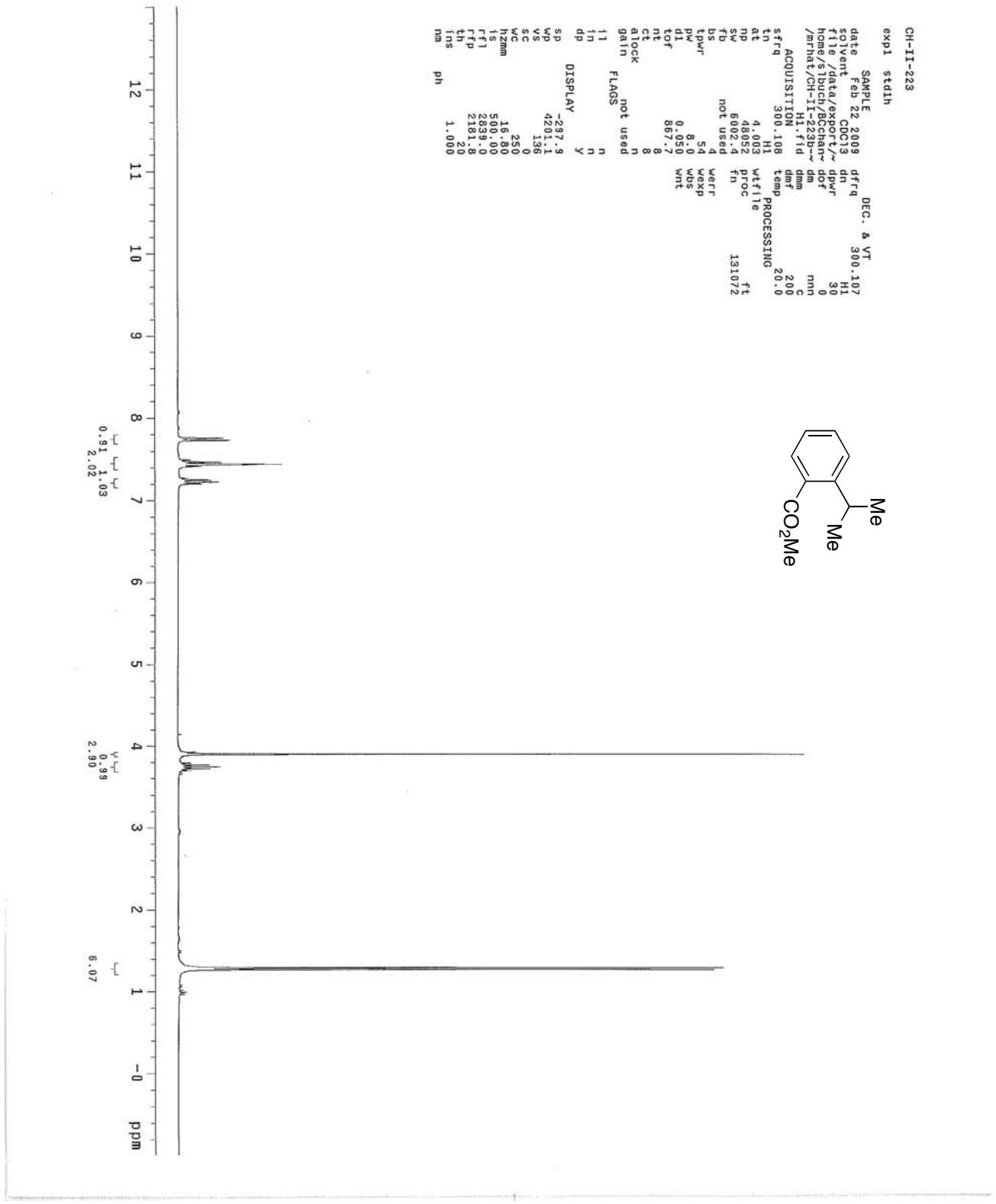
CH-II-251-C13

exp1 std13c

SAMPLE		DEC. & VT	
date	Feb 22 2009	dfrq	300.107
solvent	CDCl3	dn	H1
file	/data/export/~	dpwr	39
home/slbuch/BCchan~		dof	0
/mrhat/CH-II-251-C~		dm	yyy
	13.fid	dmm	w
ACQUISITION		dmf	11000
sfrq	75.471	temp	20.0
tn	C13	PROCESSING	
at	1.500	sb	-1.500
np	67874	sbs	-1.500
sw	22624.4	wtfile	
fb	12400	proc	ft
bs	16	fn	262144
tpwr	55		
pw	10.0	werr	
d1	0.500	wexp	
tof	2264.0	wbs	
nt	256	wnt	
ct	256		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-746.5		
wp	18110.7		
vs	19		
sc	0		
wc	250		
hzmm	72.44		
is	500.00		
rfl	7326.8		
rfp	5827.9		
th	9		
ins	100.000		
ai	ph		



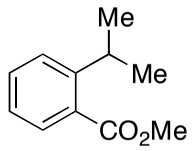


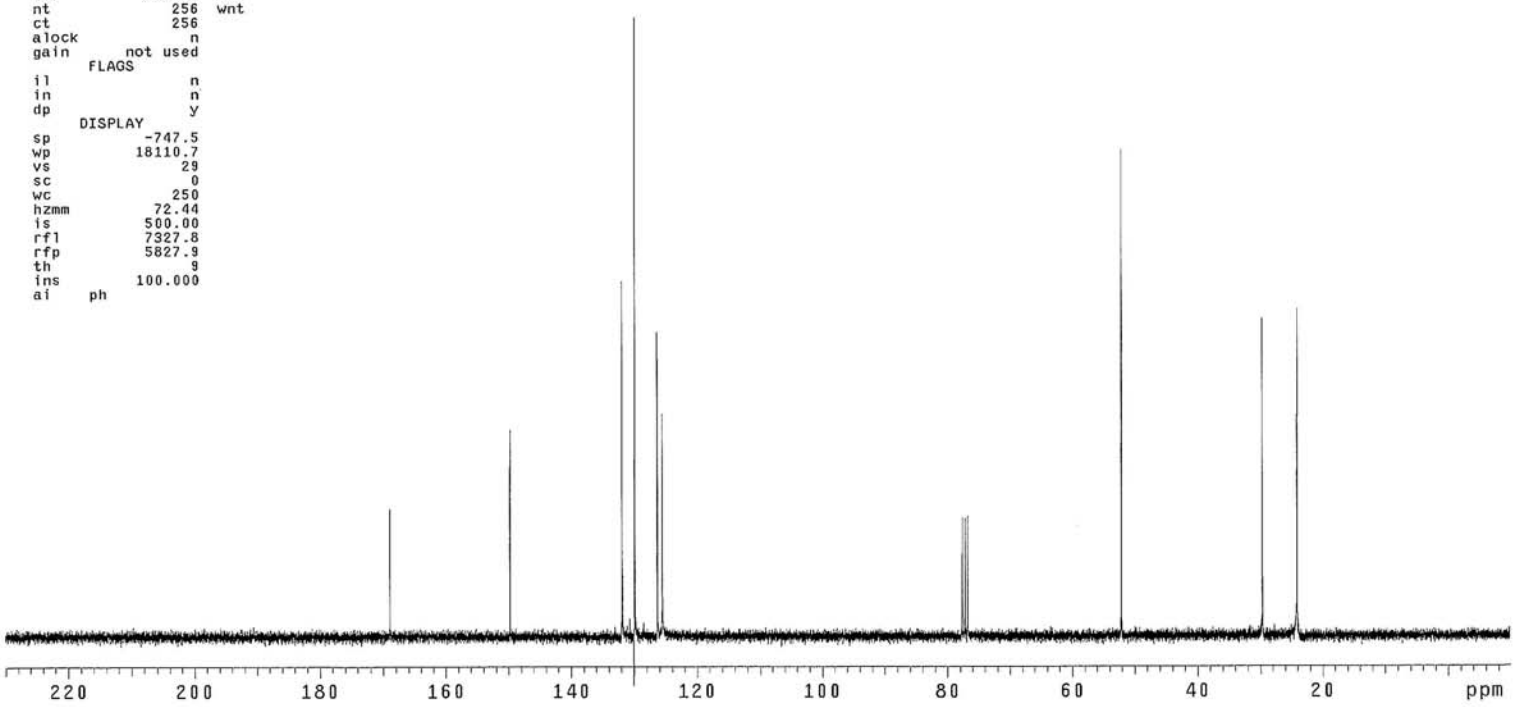

CH-II-223
exp1 stdih
SAMPLE
date Feb 22 2009
solvent CDCl3
file /data/export/~
home/slbuch/BCchan~
/mrhat/CH-II-223b~
ACQUISITION
sfrq 300.108
tn H1
at 4.003
np 48052
sw 6002.4
fb 131072
bs not used
tpwr 54
pw 8.0
d1 0.050
tof 867.7
nt 8
ct 8
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -297.9
wp 4201.1
vs 136
sc 0
wc 250
hzmm 16.80
is 500.00
rfl 2839.0
rfp 2181.8
th 20
ins 1.000
nm
ph
DEC. & VT
dfrq 300.107
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
temp 20.0
PROCESSING
wtfile
proc ft
fn
werr
wexp
wbs
wnt
Me
Me
CO2Me
0.91 1.03
2.02
0.99
2.90
6.07
12
11
10
9
8
7
6
5
4
3
2
1
-0
ppm

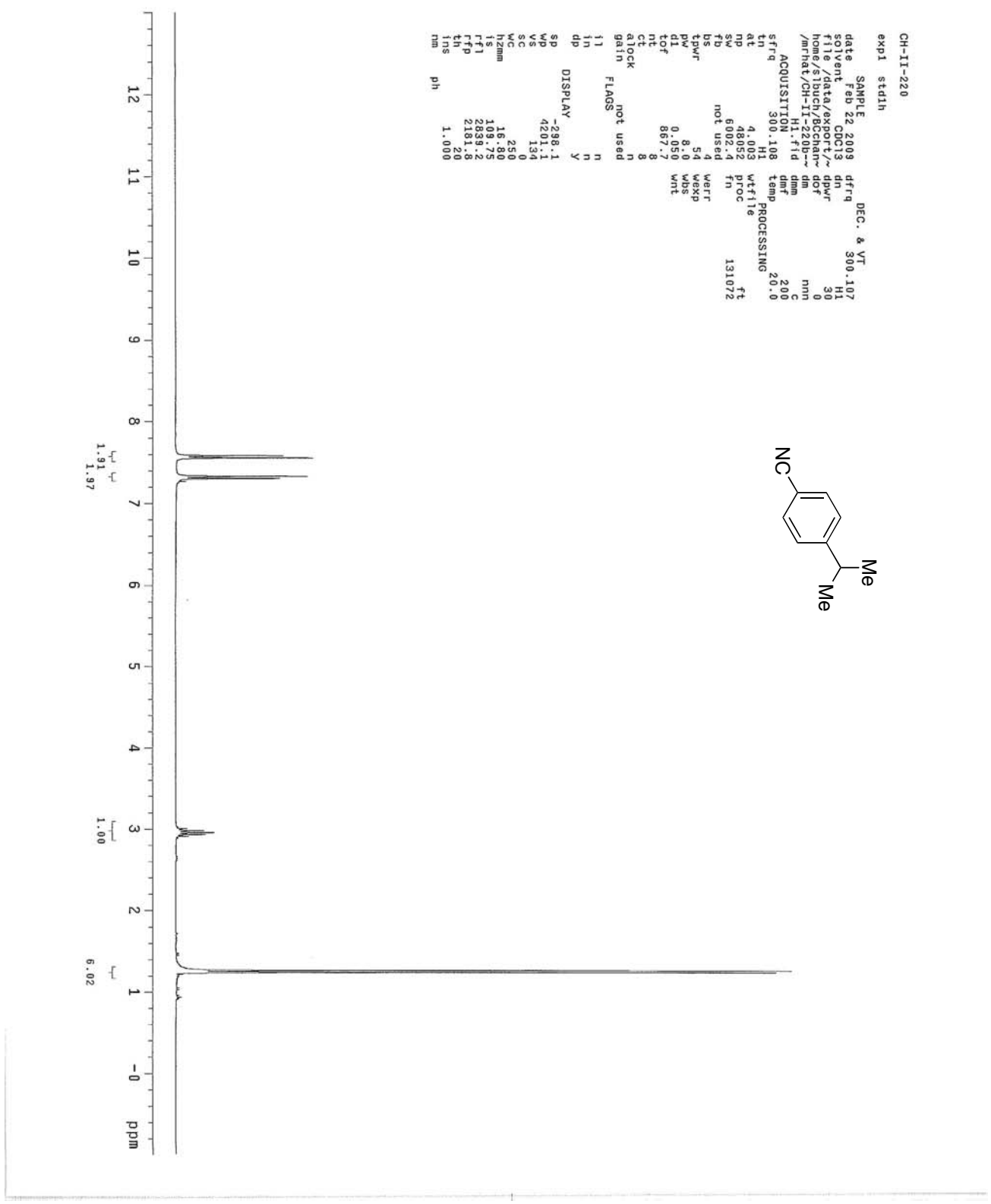
CH-II-223-C13

exp1 std13c

SAMPLE		DEC. & VT	
date	Feb 22 2009	dfrq	300.107
solvent	CDCl3	dn	H1
file	/data/export/~	dpwr	39
home/slbuch/BCchan~		dof	0
/mrhat/CH-II-223b~~		dm	yyy
	C13.fid	dmm	w
ACQUISITION		dmf	11000
sfrq	75.471	temp	20.0
tn	C13	PROCESSING	
at	1.500	sb	-1.500
np	67874	sbs	-1.500
sw	22624.4	wtfile	
fb	12400	proc	ft
bs	16	fn	262144
tpwr	55		
pw	10.0	werr	
d1	0.500	wexp	
tof	2264.0	wbs	
nt	256	wnt	
ct	256		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-747.5		
wp	18110.7		
vs	29		
sc	0		
wc	250		
hzmm	72.44		
is	500.00		
rfl	7327.8		
rfp	5827.9		
th	9		
ins	100.000		
ai ph			



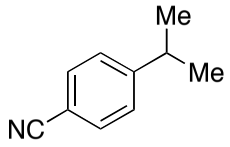


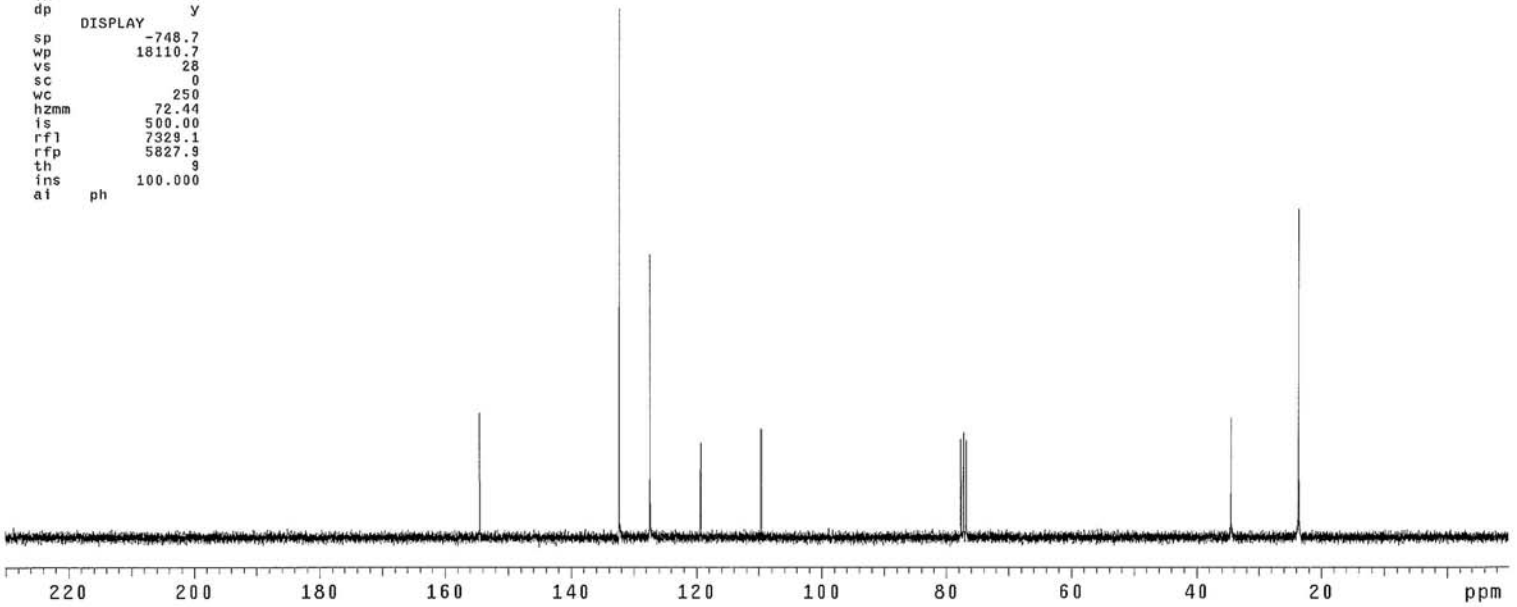

CH-II-220
exp1 stdih
SAMPLE
date Feb 22 2009
solvent CDCl3
file /data/export/~
home/stbuch/BCchan~
/mrhat/CH-II-220b~~
H1.fid
ACQUISITION
sfrq 300.108
tn H1
at 4.003
np 48052
sw 6002.4
fb not used
bs 4
tpwr 54
pw 8.0
d1 0.050
tof 867.7
nt 8
ct 8
alock n
gain n
FLAGS
il n
in n
dp y
DISPLAY
sp -298.1
wp 4201.1
vs 134
sc 0
wc 250
hzmm 16.80
is 109.75
rfl 2839.2
rfp 2181.8
th 20
ins 1.000
nm
ph
DEC. & VT
dfrq 300.107
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
temp 20.0
PROCESSING
wtfile
proc ft
fn 131072
werr
wexp
wbs
wnt
NC
Me
Me
12
11
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.91
1.97
1.00
6.02

CH-II-220-C13

exp1 std13c

SAMPLE		DEC. & VT	
date	Feb 22 2009	dfrq	300.107
solvent	CDCl3	dn	H1
file	/data/export/~	dpwr	39
home/slbuch/BCchan~		dof	0
/mrhat/CH-II-220b-~		dm	yyy
	C13.fid	dmm	w
ACQUISITION		dmf	11000
sfrq	75.471	temp	20.0
tn	C13	PROCESSING	
at	1.500	sb	-1.500
np	67874	sbs	-1.500
sw	22624.4	wtfile	
fb	12400	proc	ft
bs	16	fn	262144
tpwr	55		
pw	10.0	werr	
d1	0.500	wexp	
tof	2264.0	wbs	
nt	256	wnt	
ct	256		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-748.7		
wp	18110.7		
vs	28		
sc	0		
wc	250		
hzmm	72.44		
is	500.00		
rfl	7329.1		
rfp	5827.9		
th	9		
ins	100.000		
ai	ph		

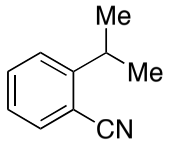


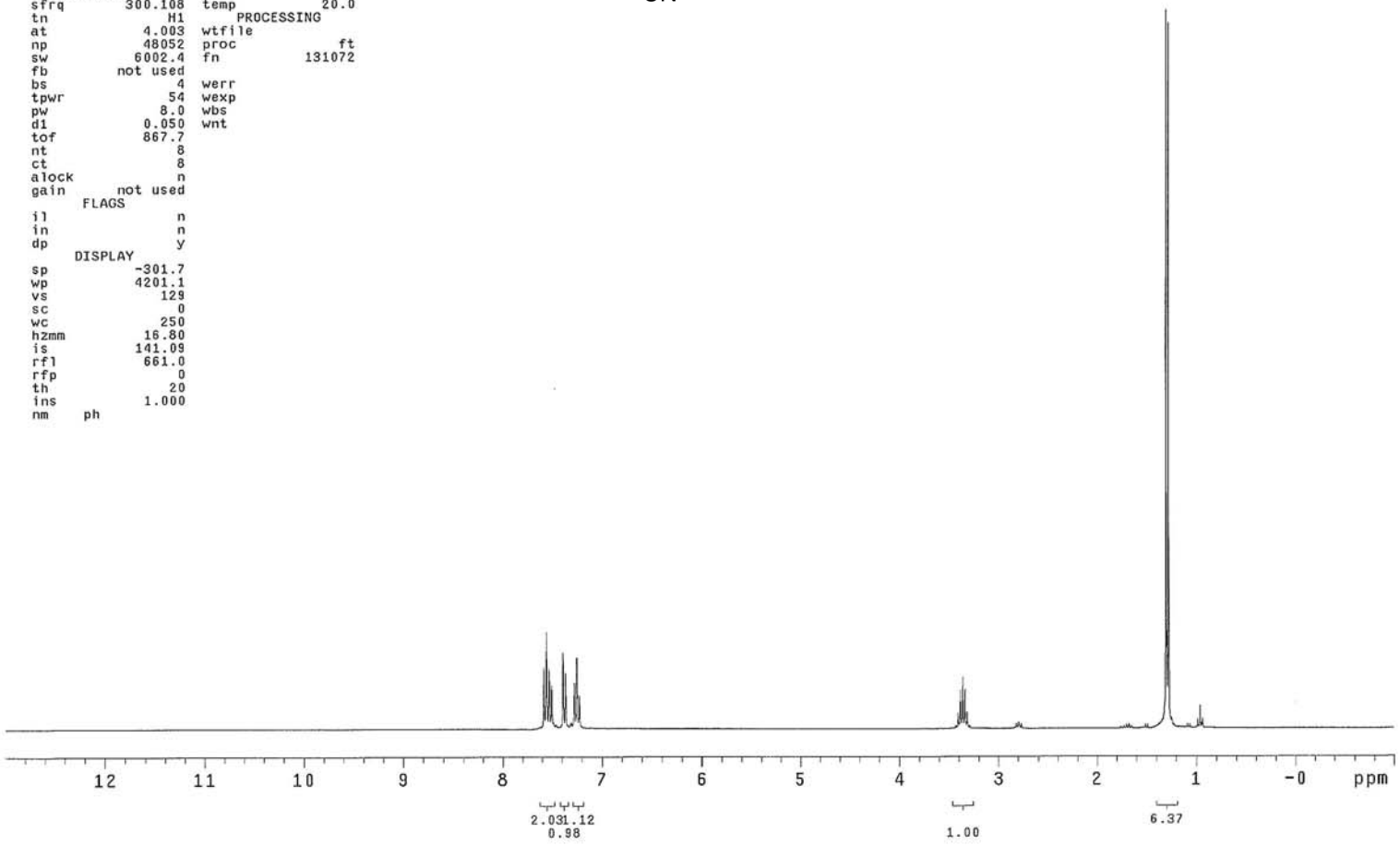


CH-II-238

exp1 std1h

SAMPLE		DEC. & VT	
date	Feb 22 2009	dfrq	300.107
solvent	CDCl3	dn	H1
file	/data/export/~	dpwr	30
home/slbuch/BCchan~		dof	0
/mrhat/CH-II-238c~~		dm	nnn
	H1.fid	dmm	c
ACQUISITION		dmf	200
sfrq	300.108	temp	20.0
tn	H1	PROCESSING	
at	4.003	wtfile	
np	48052	proc	ft
sw	6002.4	fn	131072
fb	not used		
bs	4	werr	
tpwr	54	wexp	
pw	8.0	wbs	
d1	0.050	wnt	
tof	867.7		
nt	8		
ct	8		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-301.7		
wp	4201.1		
vs	129		
sc	0		
wc	250		
hzmm	16.80		
is	141.09		
rfl	661.0		
rfp	0		
th	20		
ins	1.000		
nm	ph		

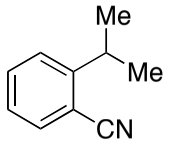


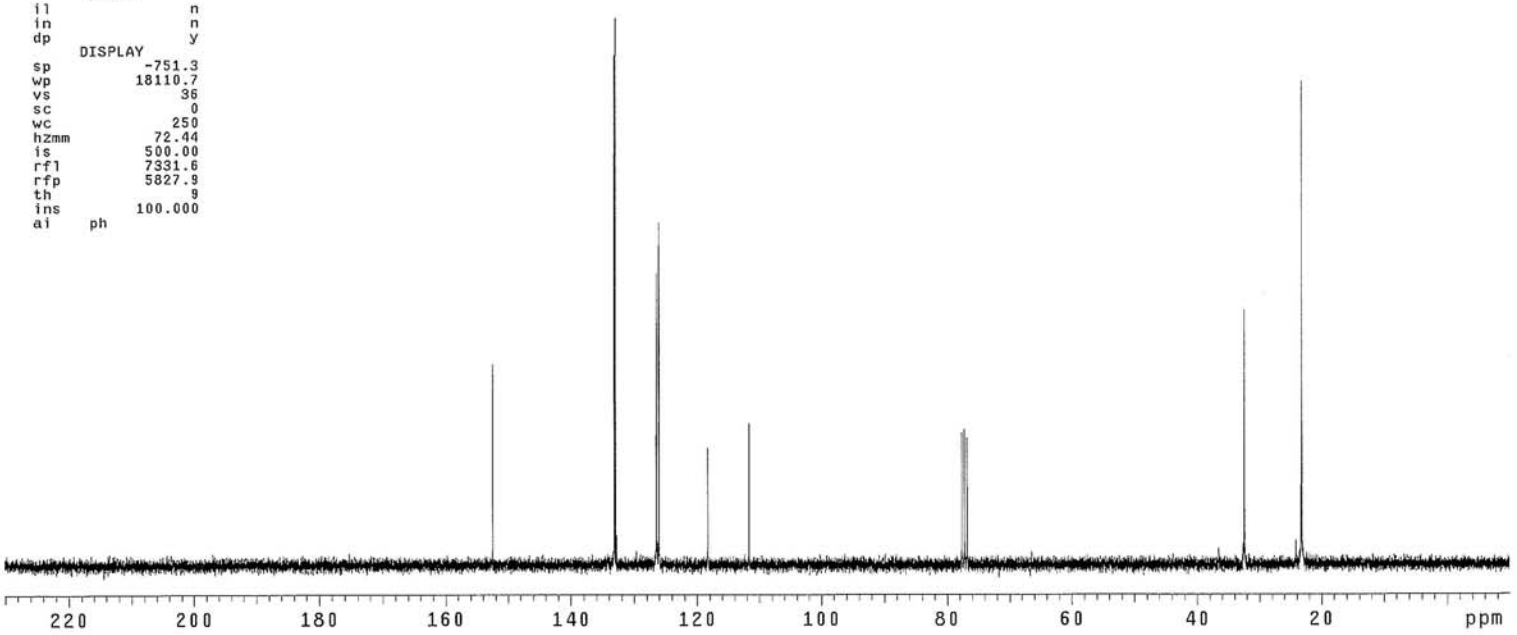


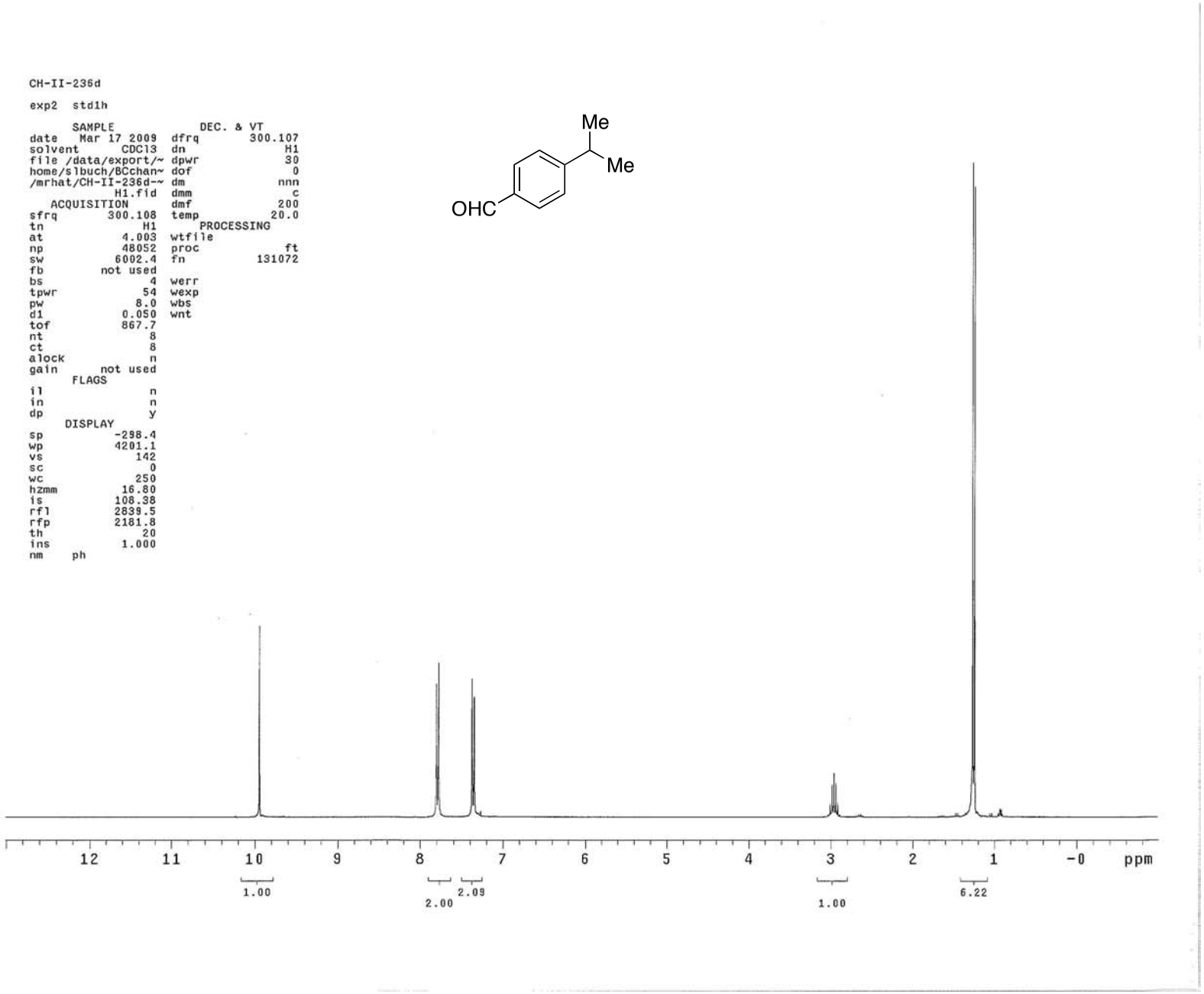
CH-II-238-C13

exp1 std13c

SAMPLE		DEC. & VT	
date	Feb 22 2009	dfrq	300.107
solvent	CDCl3	dn	H1
file	/data/export/~	dpwr	39
home/slbuch/BCchan~		dof	0
/mrhat/CH-II-238c~~		dm	yyy
	C13.fid	dmm	w
ACQUISITION		dmf	11000
sfrq	75.471	temp	20.0
tn	C13	PROCESSING	
at	1.500	sb	-1.500
np	67874	sbs	-1.500
sw	22624.4	wtfile	
fb	12400	proc	ft
bs	16	fn	262144
tpwr	55		
pw	10.0	werr	
d1	0.500	wexp	
tof	2264.0	wbs	
nt	256	wnt	
ct	256		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-751.3		
wp	18110.7		
vs	36		
sc	0		
wc	250		
hzmm	72.44		
is	500.00		
rfl	7331.6		
rfp	5827.9		
th	9		
ins	100.000		
ai	ph		



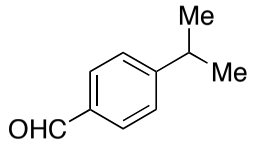


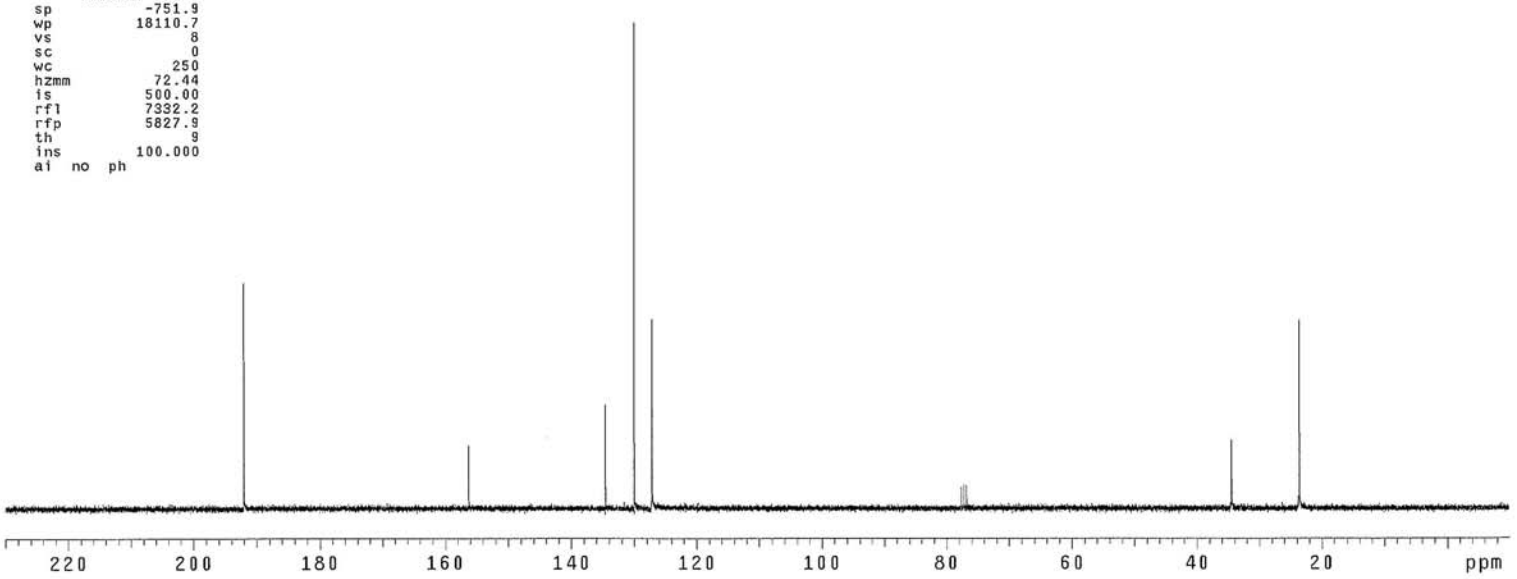

CH-II-236d
exp2 std1h
SAMPLE
date Mar 17 2009
solvent CDCl3
file /data/export/~
home/slbuch/BCchan~
/mrhat/CH-II-236d--~
H1.fid
ACQUISITION
sfrq 300.108
tn H1
at 4.003
np 48052
sw 6002.4
fb not used
bs 4
tpwr 54
pw 8.0
d1 0.050
tof 867.7
nt 8
ct 8
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -298.4
wp 4201.1
vs 142
sc 0
wc 250
hzmm 16.80
is 108.38
rfl 2839.5
rfp 2181.8
th 20
ins 1.000
nm ph
DEC. & VT
dfrq 300.107
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
temp 20.0
PROCESSING
wtfile
proc ft
fn 131072
werr
wexp
wbs
wnt
Me
Me
OHC
12
11
10
9
8
7
6
5
4
3
2
1
-0
ppm
1.00
2.00
2.09
1.00
6.22

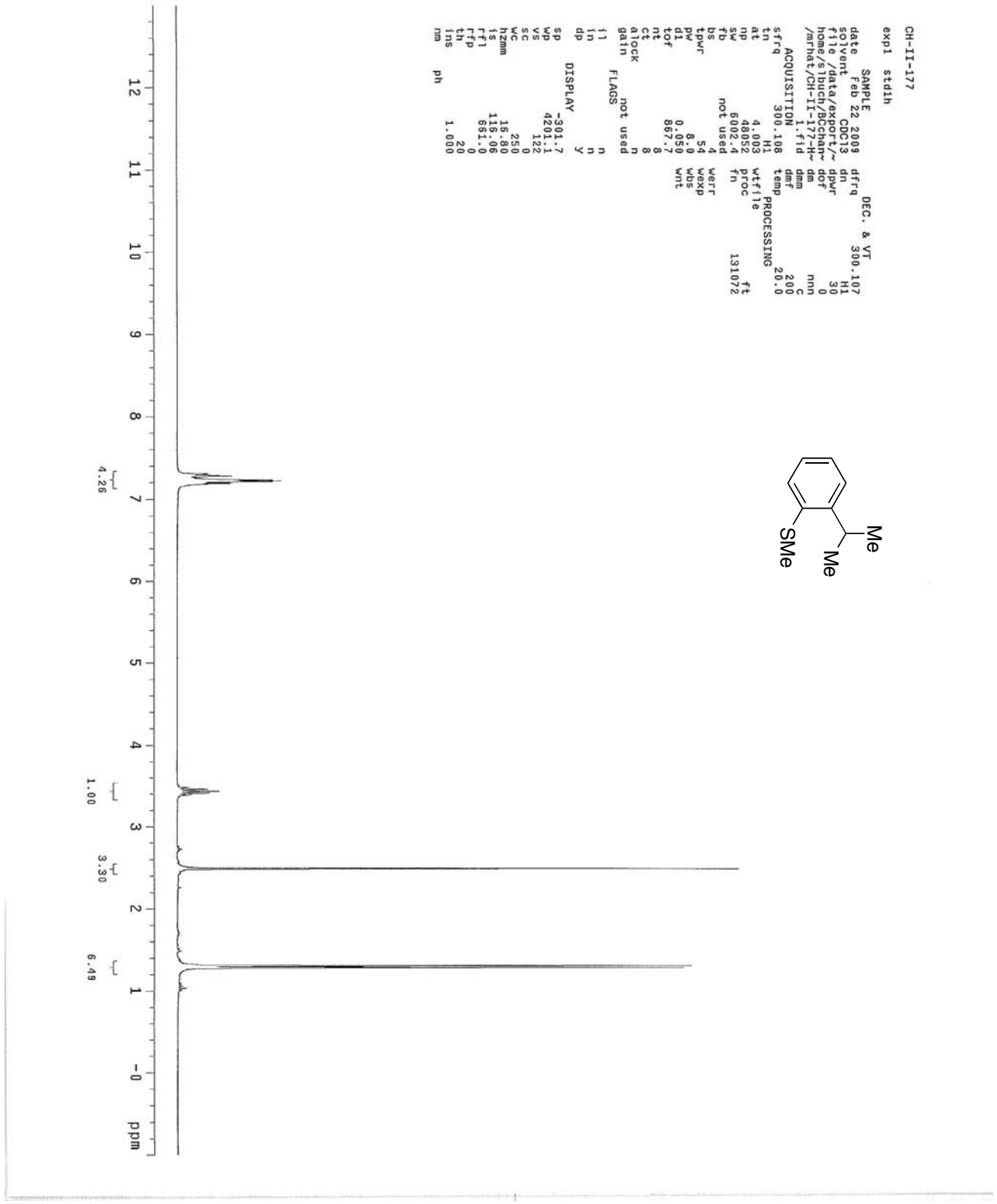
CH-II-236-C13

exp1 std13c

SAMPLE		DEC. & VT	
date	Mar 2 2009	dfrq	300.107
solvent	CDCl3	dn	H1
file	/data/export/~	dpwr	39
home/slbuch/BCchan~		dof	0
/mrhat/CH-II-236c-~		dm	yyy
	C13.fid	dmm	w
ACQUISITION		dmf	11000
sfrq	75.471	temp	20.0
tn	C13	PROCESSING	
at	1.500	sb	-1.500
np	67874	sbs	-1.500
sw	22624.4	wtfile	
fb	12400	proc	ft
bs	16	fn	262144
tpwr	55		
pw	10.0	werr	
d1	0.500	wexp	
tof	2264.0	wbs	
nt	256	wnt	
ct	64		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-751.9		
wp	18110.7		
vs	8		
sc	0		
wc	250		
hzmm	72.44		
is	500.00		
rfl	7332.2		
rfp	5827.9		
th	9		
ins	100.000		
ai no ph			



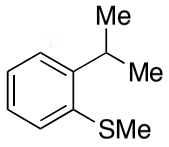


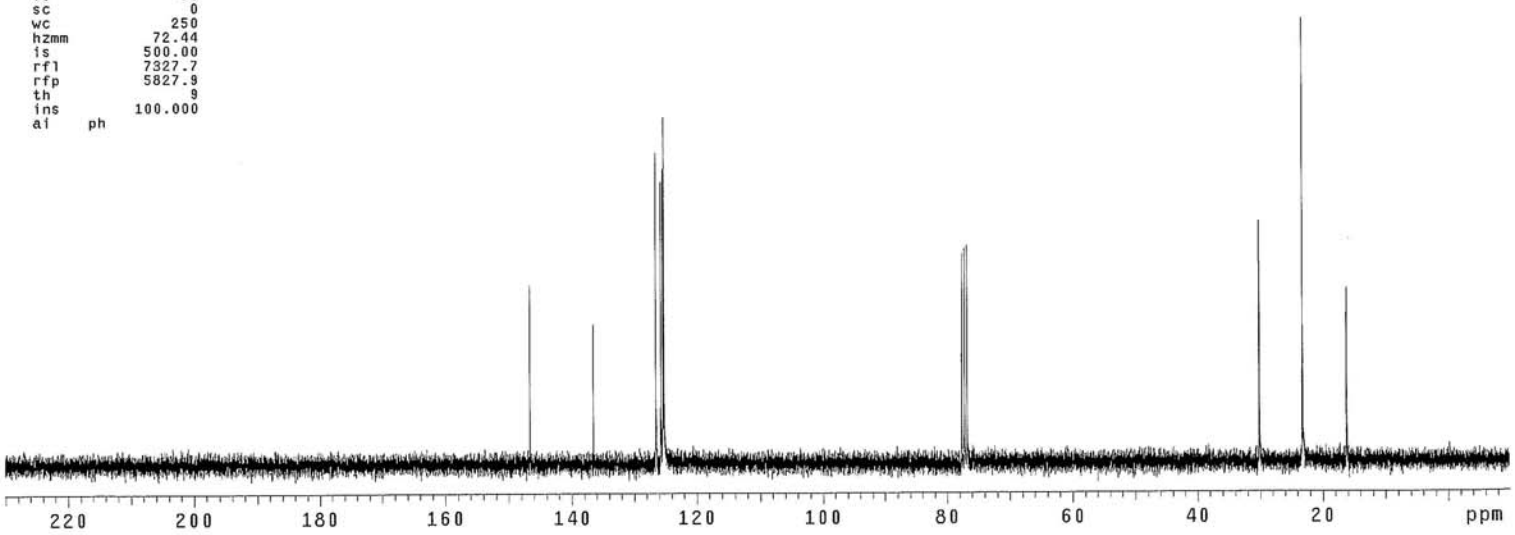

CH-II-177
exp1 stdih
SAMPLE
date Feb 22 2009
solvent CDCl3
file /data/export/~
home/stbuch/BCchan~
/mrhat/CH-II-177-H~
ACQUISITION
sfrq 300.108
tn H1
at 4.003
np 48052
sw 6002.4
fb 6002.4
bs not used
tpwr 54
pw 8.0
d1 0.050
tof 867.7
nt 8
ct 8
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -301.7
wp 4201.1
vs 122
sc 0
wc 250
hzmm 16.80
is 116.06
rfl 661.0
rfp 0
th 20
ins 1.000
nm
ph
DEC. & VT
dfrq 300.107
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
temp 20.0
PROCESSING
wtfile
proc ft
fn 131072
werr
wexp
wbs
wnt
Me
Me
SMe
4.26
1.00
3.30
6.49
12
11
10
9
8
7
6
5
4
3
2
1
-0
ppm

CH-II-177-C13

exp1 std13c

SAMPLE		DEC. & VT	
date	Feb 22 2009	dfrq	300.107
solvent	CDCl3	dn	H1
file	/data/export/~	dpwr	39
home/slbuch/BCchan~		dof	0
/mrhat/CH-II-177-C~		dm	yyy
	13.fid	dmm	w
ACQUISITION		dmf	11000
sfrq	75.471	temp	20.0
tn	C13	PROCESSING	
at	1.500	sb	-1.500
np	67874	sbs	-1.500
sw	22624.4	wtfile	
fb	12400	proc	ft
bs	16	fn	262144
tpwr	55		
pw	10.0	werr	
d1	0.500	wexp	
tof	2264.0	wbs	
nt	256	wnt	
ct	256		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-747.4		
wp	18110.7		
vs	53		
sc	0		
wc	250		
hzmm	72.44		
is	500.00		
rfl	7327.7		
rfp	5827.9		
th	9		
ins	100.000		
ai ph			

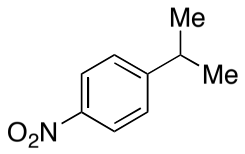


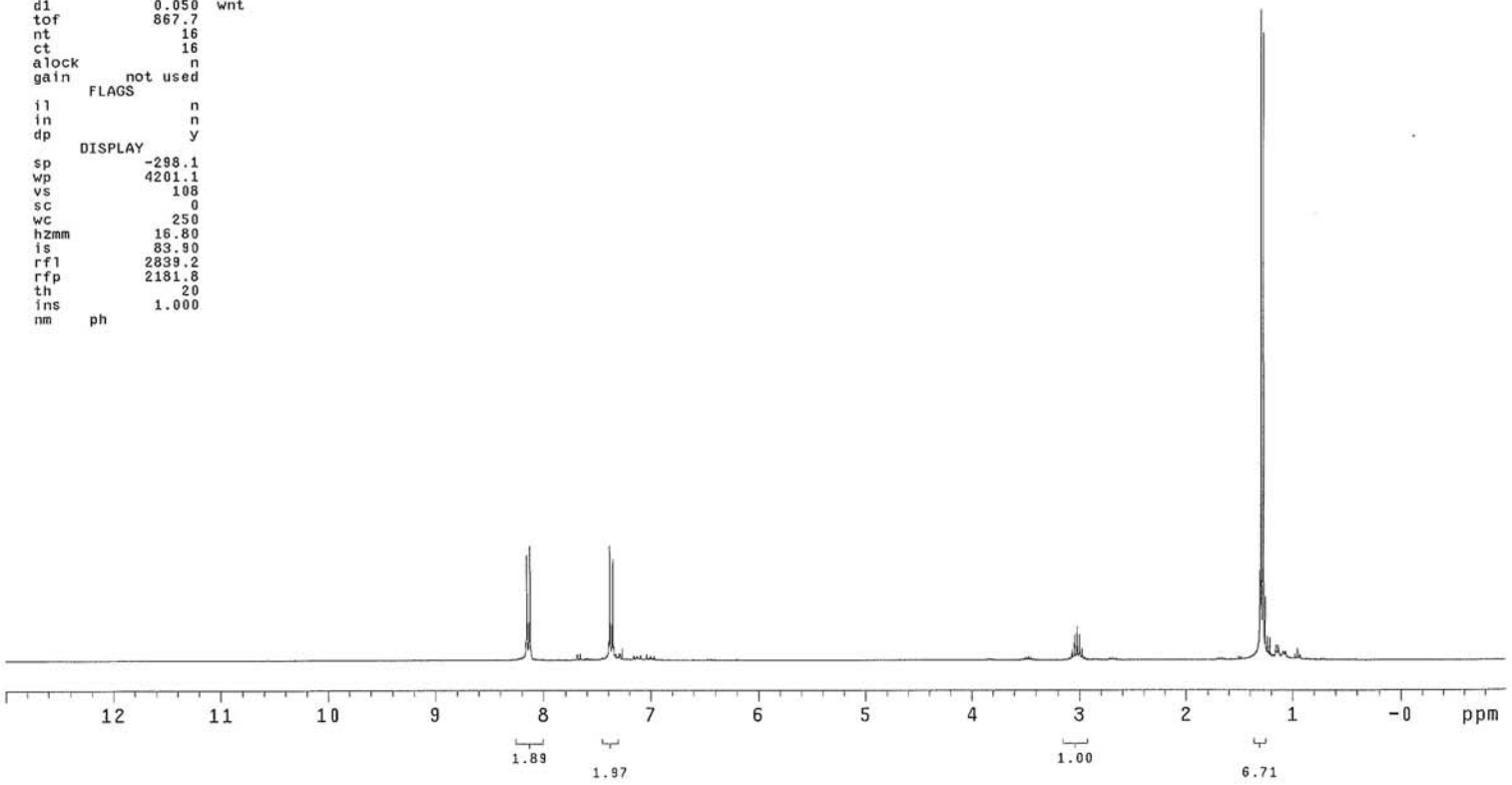


CH-II-221b

expl std1h

SAMPLE		DEC. & VT	
date	Mar 2 2009	dfrq	300.107
solvent	CDCl3	dn	H1
file	/data/export/~	dpwr	30
home/slbuch/BCchan~		dof	0
/mrhat/CH-II-221b~		dm	nnn
	H1.fid	dmm	c
ACQUISITION		dmf	200
sfrq	300.108	temp	20.0
tn	H1	PROCESSING	
at	4.003	wtfile	
np	48052	proc	ft
sw	6002.4	fn	131072
fb	not used		
bs	8	werr	
tpwr	54	wexp	
pw	8.0	wbs	
d1	0.050	wnt	
tof	867.7		
nt	16		
ct	16		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-298.1		
wp	4201.1		
vs	108		
sc	0		
wc	250		
hzmm	16.80		
is	83.90		
rfl	2839.2		
rfp	2181.8		
th	20		
ins	1.000		
nm	ph		

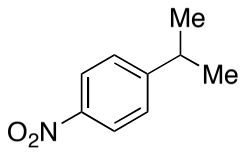


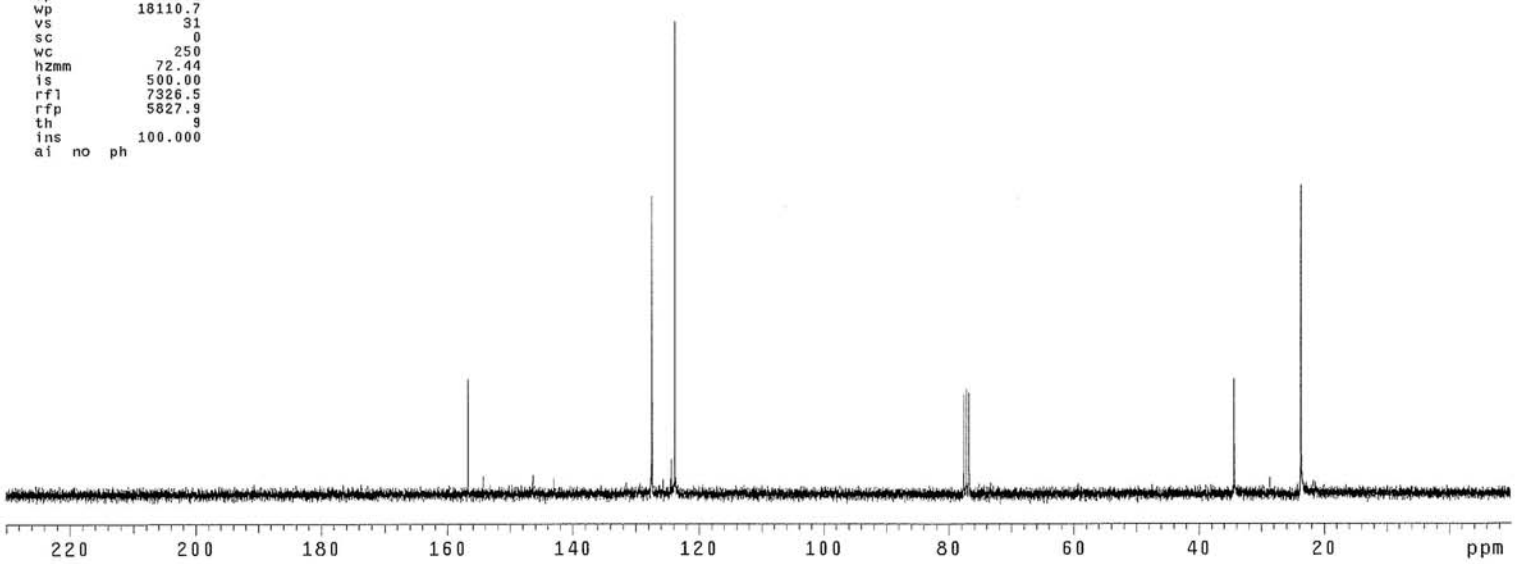


CH-II-221b-C13

exp1 std13c

SAMPLE		DEC. & VT	
date	Mar 2 2009	dfrq	300.107
solvent	CDCl3	dn	H1
file	/data/export/~	dpwr	39
home/slbuch/BCchan~		dof	0
/mrhat/CH-II-221b~~		dm	yyy
	C13.fid	dmm	w
ACQUISITION		dmf	11000
sfrq	75.471	temp	20.0
tn	C13	PROCESSING	
at	1.500	sb	-1.500
np	67874	sbs	-1.500
sw	22624.4	wtfile	
fb	12400	proc	ft
bs	16	fn	262144
tpwr	55		
pw	10.0	werr	
d1	0.500	wexp	
tof	2264.0	wbs	
nt	256	wnt	
ct	256		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-746.2		
wp	18110.7		
vs	31		
sc	0		
wc	250		
hzmm	72.44		
is	500.00		
rfl	7326.5		
rfp	5827.9		
th	9		
ins	100.000		
ai no ph			

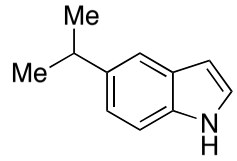


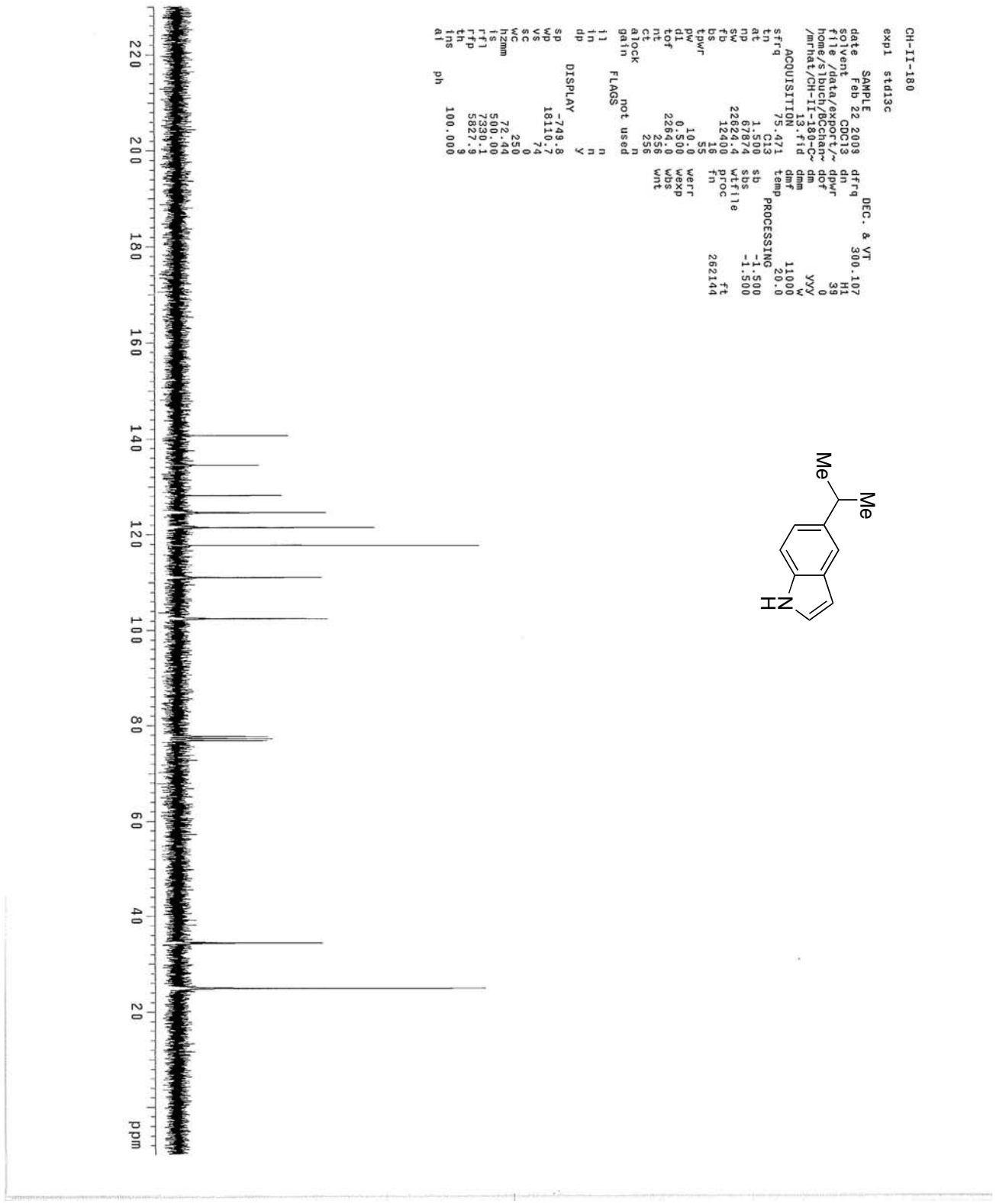


CH-II-180

exp1 std1h

SAMPLE		DEC. & VT	
date	Mar 2 2009	dfrq	300.107
solvent	CDCl3	dn	H1
file	/data/export/~	dpwr	30
home/slbuch/BCchan~		dof	0
/mrhat/CH-II-180-H~		dm	nnn
	1.fid	dmm	c
ACQUISITION		dmf	200
sfrq	300.108	temp	20.0
tn	H1	PROCESSING	
at	4.003	wtfile	
np	48052	proc	ft
sw	6002.4	fn	131072
fb	not used		
bs	8	werr	
tpwr	54	wexp	
pw	8.0	wbs	
d1	0.050	wnt	
tof	867.7		
nt	16		
ct	16		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-302.9		
wp	4204.0		
vs	160		
sc	0		
wc	250		
hzmm	16.82		
is	58.68		
rfl	2839.6		
rfp	2181.8		
th	20		
ins	1.000		
nm	ph		

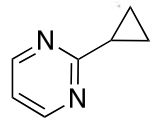


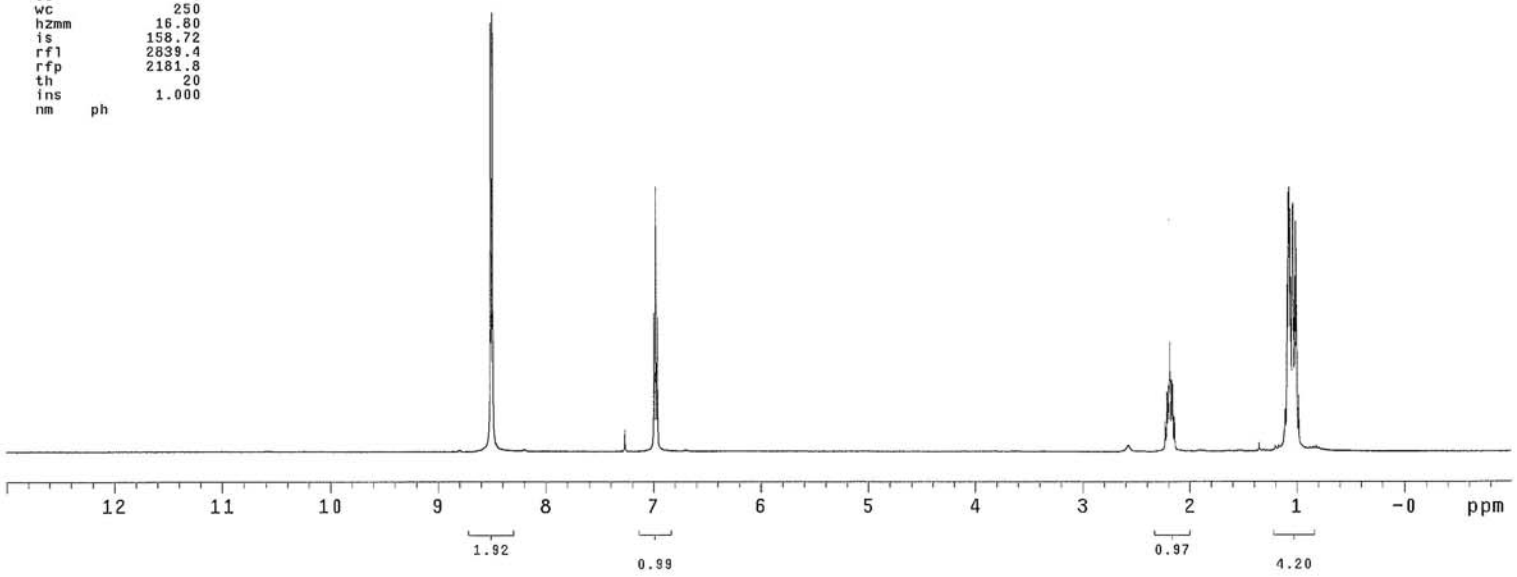

CH-II-180
exp1 std13c
SAMPLE
date Feb 22 2009
solvent CDCl3
file /data/export/~
home/slbuch/BCchan~
/mrhat/CH-II-180-C~
ACQUISITION
sfrq 75.471
tn C13
at 1.500
np 67874
sw 22624.4
fb 12400
bs 16
tpwr 55
pw 10.0
d1 0.500
tof 2264.0
nt 256
ct 256
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -749.8
wp 18110.7
vs 74
sc 0
wc 250
hzmm 72.44
is 500.00
rfl 7330.1
rfp 5827.9
th
ins 100.000
al ph
DEC. & VT
dfrq 300.107
dn H1
dpwr 39
dof 0
dm yyy
dmm w
dmf 11000
temp 20.0
PROCESSING
sb -1.500
sbs -1.500
wtfile
proc ft
fn 262144
werr
wexp
wbs
wnt
Me
Me
N
H
220
200
180
160
140
120
100
80
60
40
20
ppm

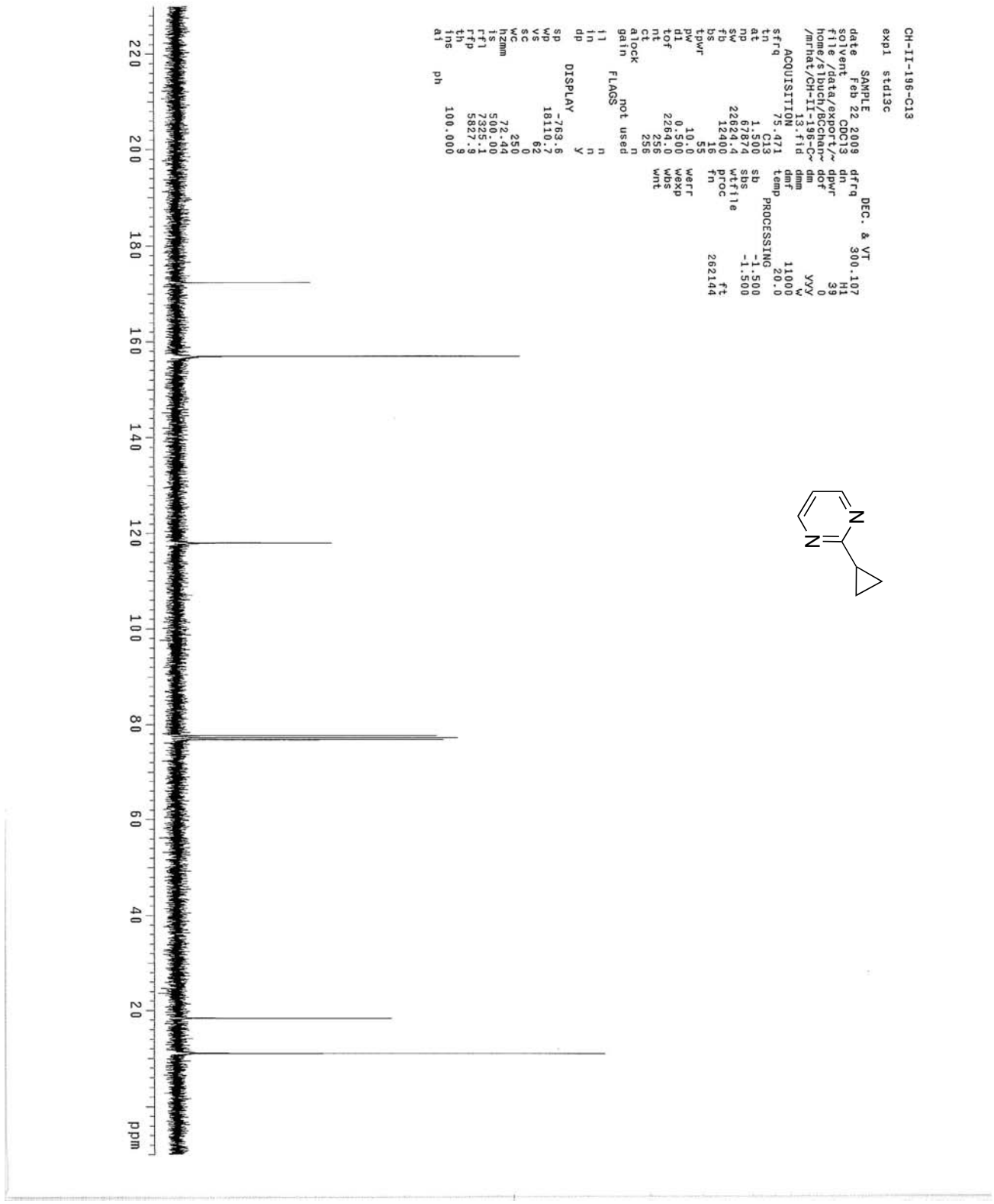
CH-II-196

expl std1h

SAMPLE		DEC. & VT	
date	Feb 22 2009	dfrq	300.107
solvent	CDCl3	dn	H1
file	/data/export/~	dpwr	30
home/slbuch/BCchan~		dof	0
/mrhat/CH-II-196-H~		dm	nnn
	1.fid	dmm	c
ACQUISITION		dmf	200
sfrq	300.108	temp	20.0
tn	H1	PROCESSING	
at	4.003	wtfile	
np	48052	proc	ft
sw	6002.4	fn	131072
fb	not used		
bs	4	werr	
tpwr	54	wexp	
pw	8.0	wbs	
d1	0.050	wnt	
tof	867.7		
nt	8		
ct	8		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-298.3		
wp	4201.1		
vs	73		
sc	0		
wc	250		
hzmm	16.80		
is	158.72		
rfl	2839.4		
rfp	2181.8		
th	20		
ins	1.000		
nm	ph		



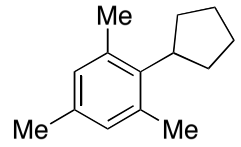


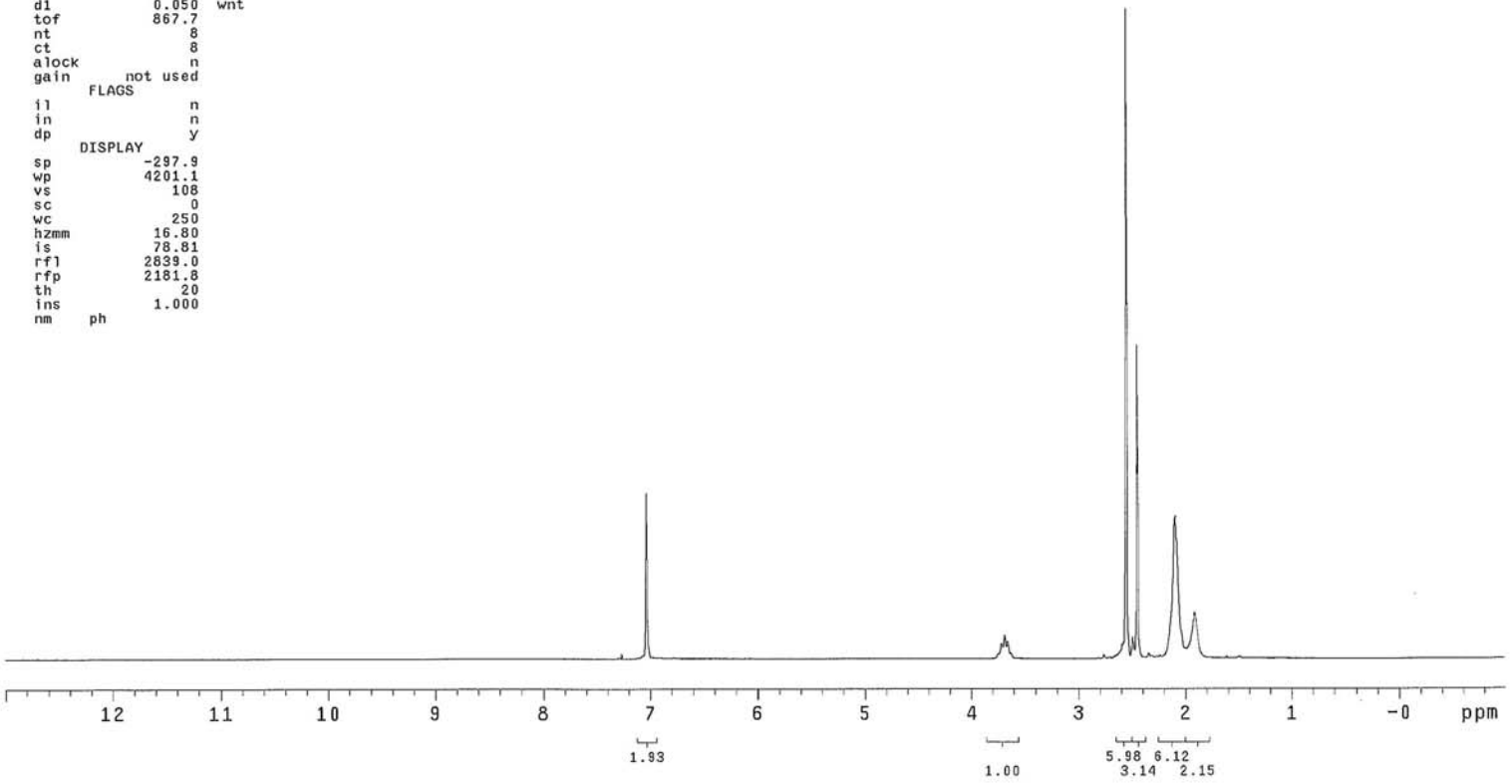

CH-II-196-C13
exp1 std13c
SAMPLE
date Feb 22 2009
solvent CDCl3
file /data/export/~
home/slbuch/BCchan~
/mrhat/CH-II-196-C~
13.fid
ACQUISITION
sfrq 75.471
tn C13
at 1.500
np 67874
sw 22624.4
fb 12400
bs 16
tpwr 55
pw 10.0
d1 0.500
tof 2264.0
nt 256
ct 256
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -763.6
wp 18110.7
vs 62
sc 0
wc 250
hzmm 72.44
is 500.00
rfl 7325.1
rfp 5827.9
th 0
ins 100.000
al ph
DEC. & VT
dfrq 300.107
dn H1
dpwr 39
dof 0
dm yyy
dmm w
dmf 11000
temp 20.0
PROCESSING
sb -1.500
sbs -1.500
wtfile
proc ft
fn 262144
wexp
wbs
wnt
N
N
220
200
180
160
140
120
100
80
60
40
20
ppm

CH-II-182

exp1 std1h

SAMPLE		DEC. & VT	
date	Feb 23 2009	dfrq	300.107
solvent	CDCl3	dn	H1
file	/data/export/~	dpwr	30
home/slbuch/BCchan~		dof	0
/mrhat/CH-II-182-H~		dm	nnn
	1.fid	dmm	c
ACQUISITION		dmf	200
sfrq	300.108	temp	20.0
tn	H1	PROCESSING	
at	4.003	wtfile	
np	48052	proc	ft
sw	6002.4	fn	131072
fb	not used		
bs	4	werr	
tpwr	54	wexp	
pw	8.0	wbs	
d1	0.050	wnt	
tof	867.7		
nt	8		
ct	8		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-297.9		
wp	4201.1		
vs	108		
sc	0		
wc	250		
hzmm	16.80		
is	78.81		
rfl	2839.0		
rfp	2181.8		
th	20		
ins	1.000		
nm	ph		

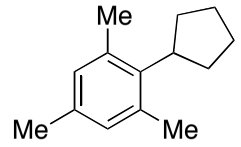


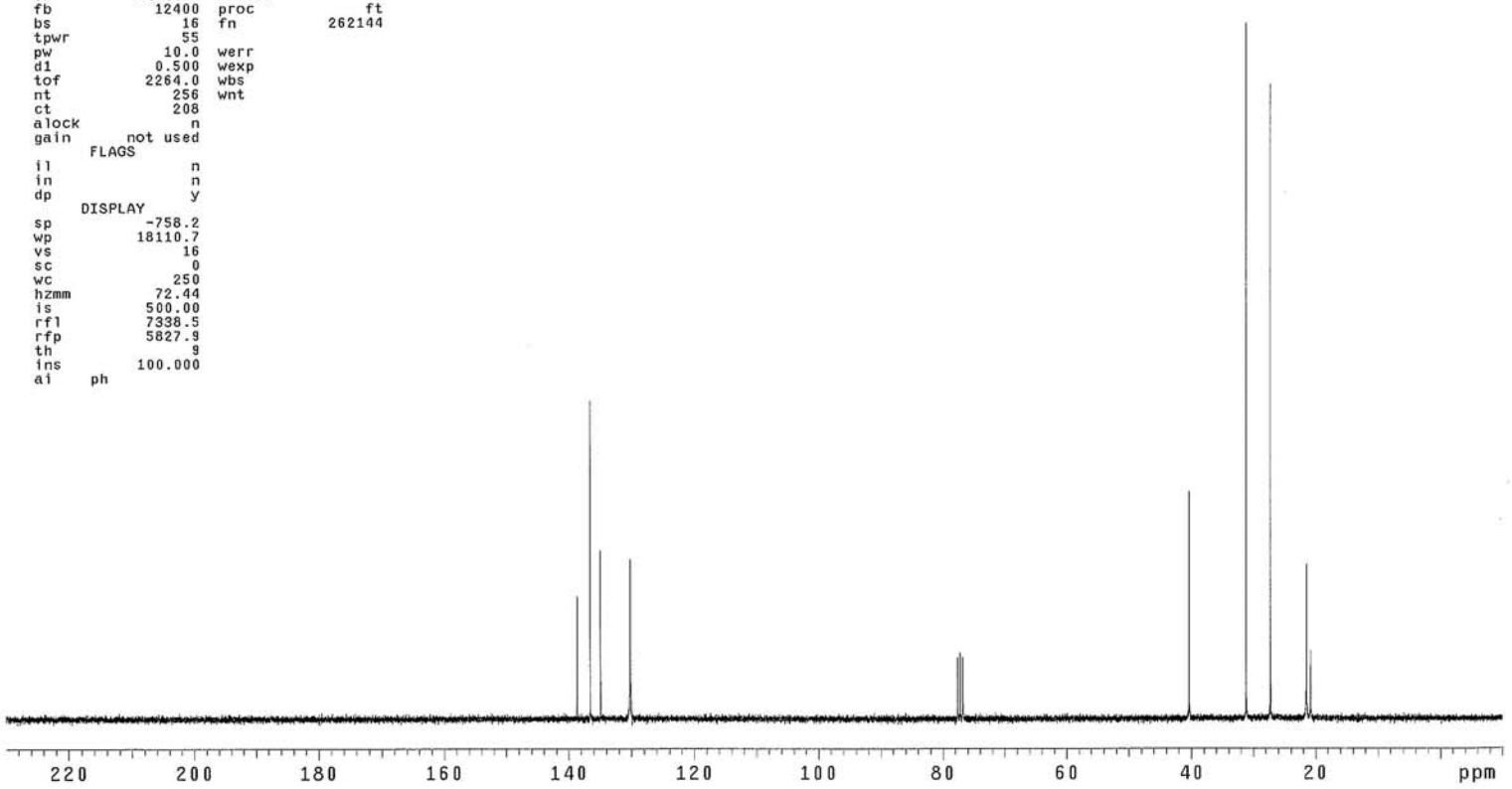


CH-II-182-C13

exp1 std13c

SAMPLE		DEC. & VT	
date	Feb 23 2009	dfrq	300.107
solvent	CDCl3	dn	H1
file	/data/export/~	dpwr	39
home/slbuch/BCchan~		dof	0
/mrhat/CH-II-182-C~		dm	yyy
	13.fid	dmm	w
ACQUISITION		dmf	11000
sfrq	75.471	temp	20.0
tn	C13	PROCESSING	
at	1.500	sb	-1.500
np	67874	sbs	-1.500
sw	22624.4	wtfile	
fb	12400	proc	ft
bs	16	fn	262144
tpwr	55		
pw	10.0	werr	
d1	0.500	wexp	
tof	2264.0	wbs	
nt	256	wnt	
ct	208		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-758.2		
wp	18110.7		
vs	16		
sc	0		
wc	250		
hzmm	72.44		
is	500.00		
rfl	7338.5		
rfp	5827.9		
th	9		
ins	100.000		
ai ph			

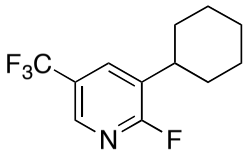


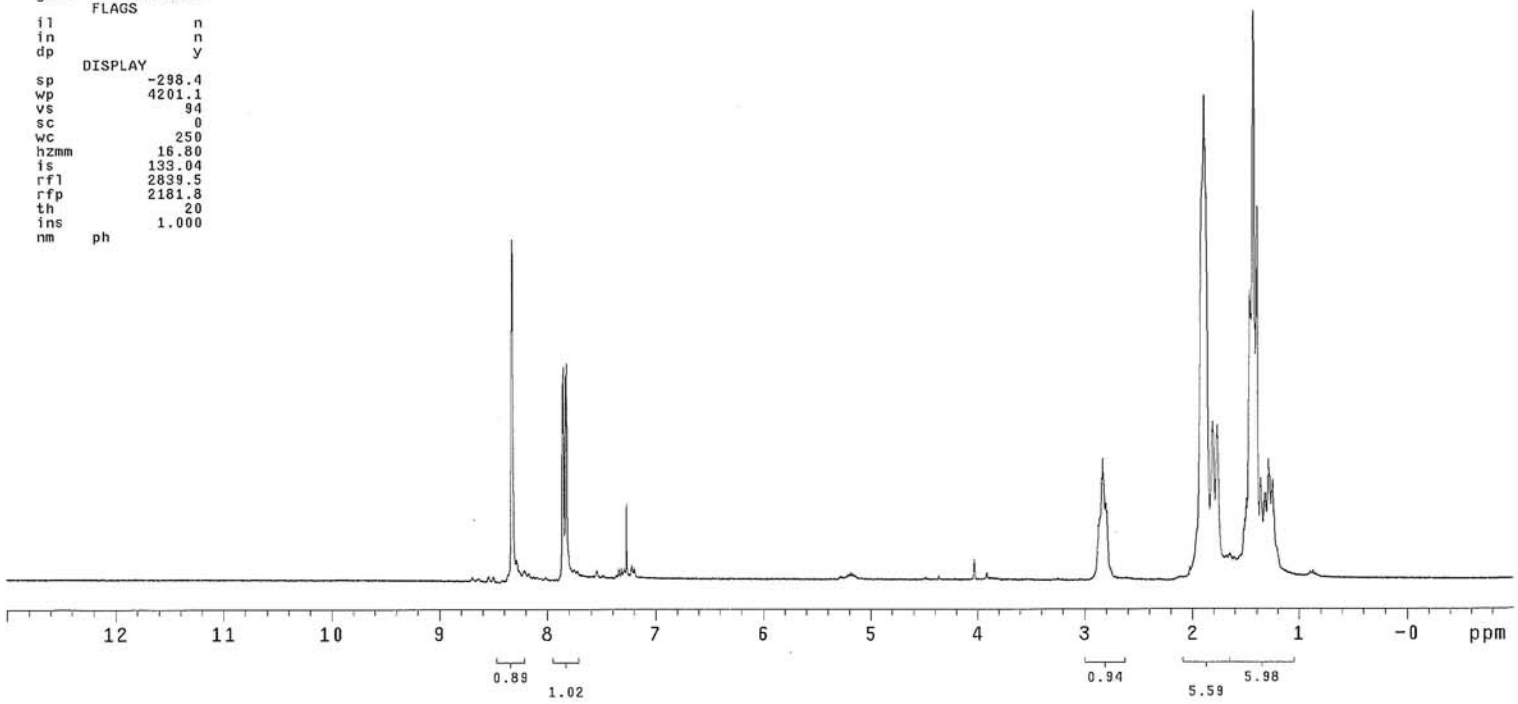


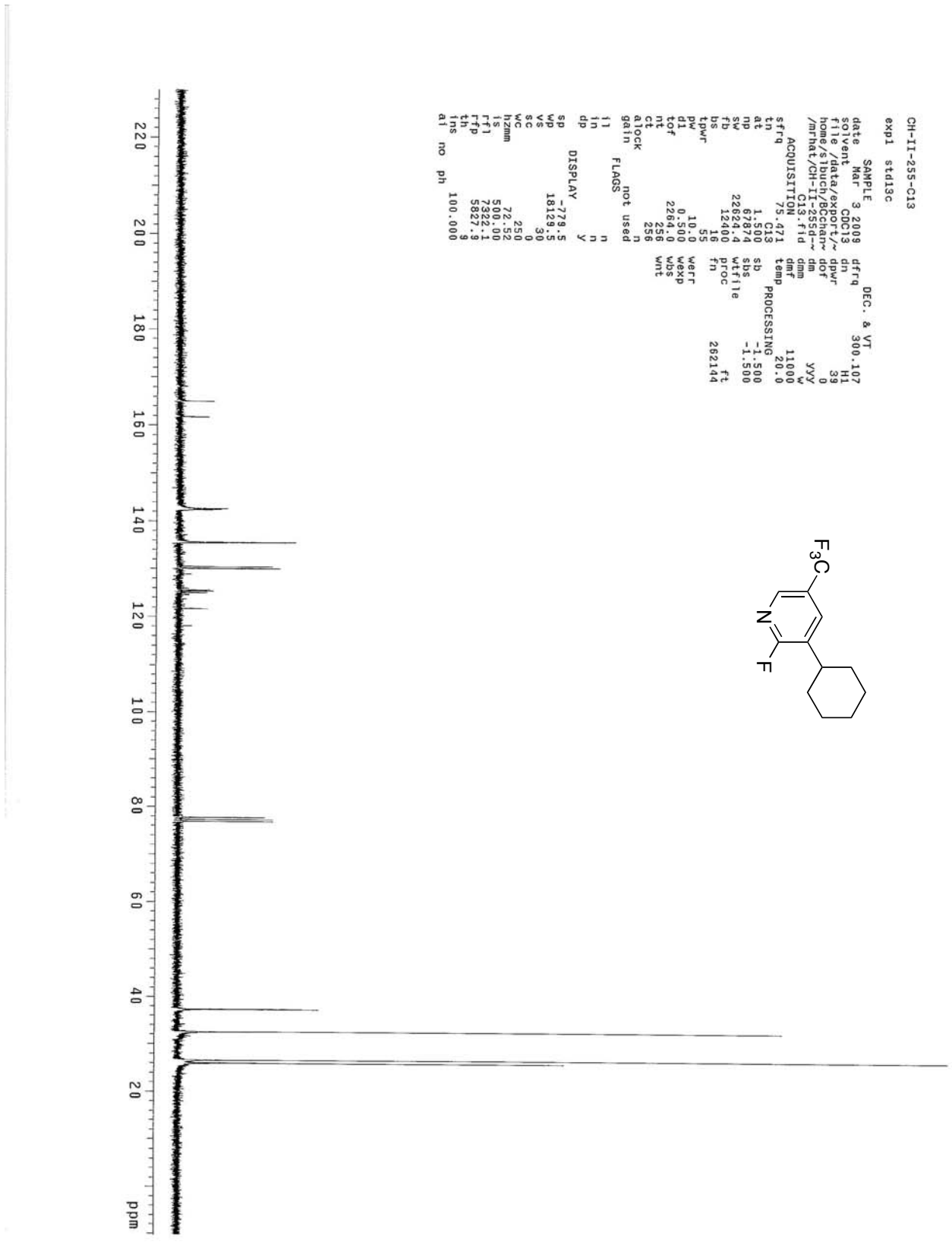
CH-II-255

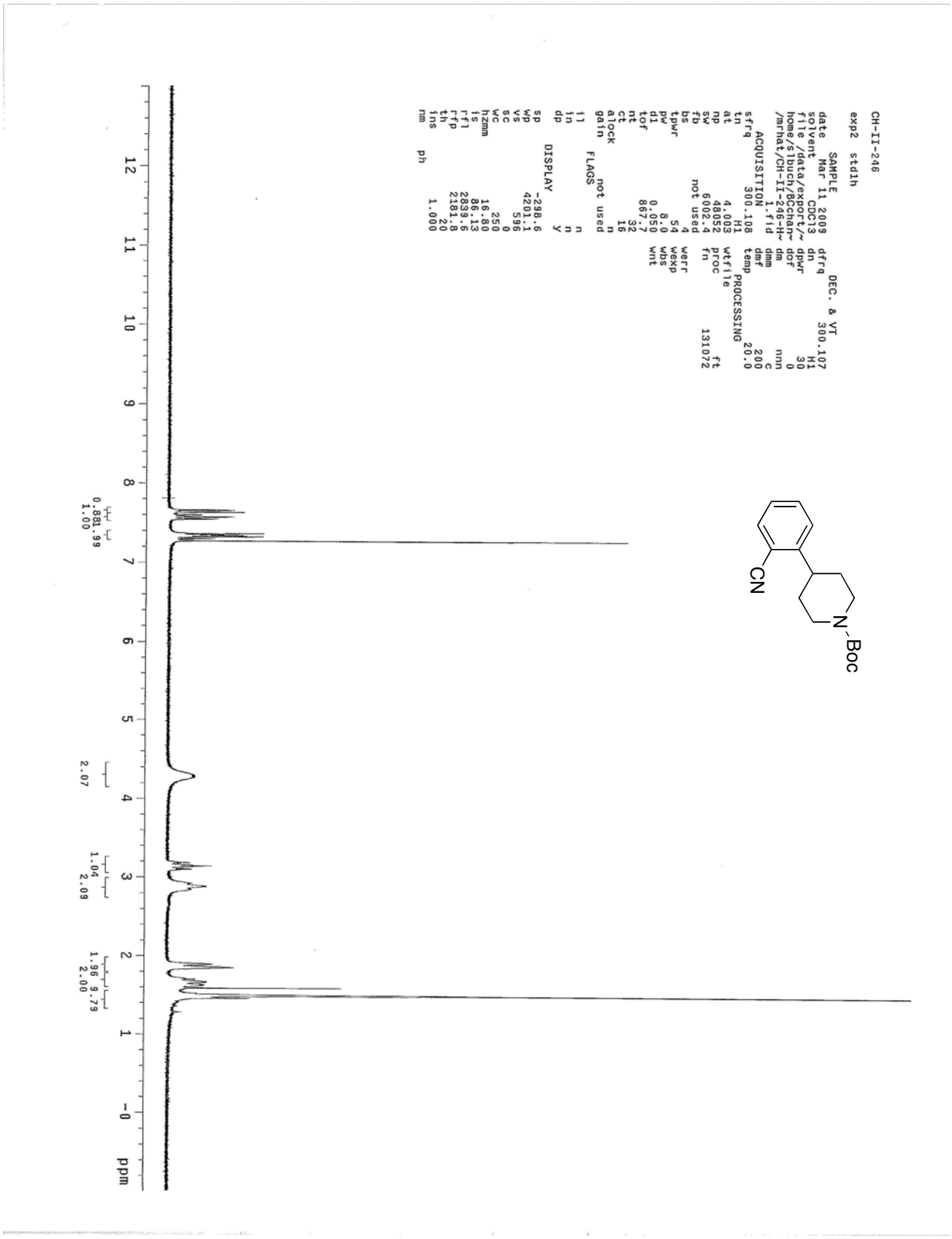
exp1 std1h

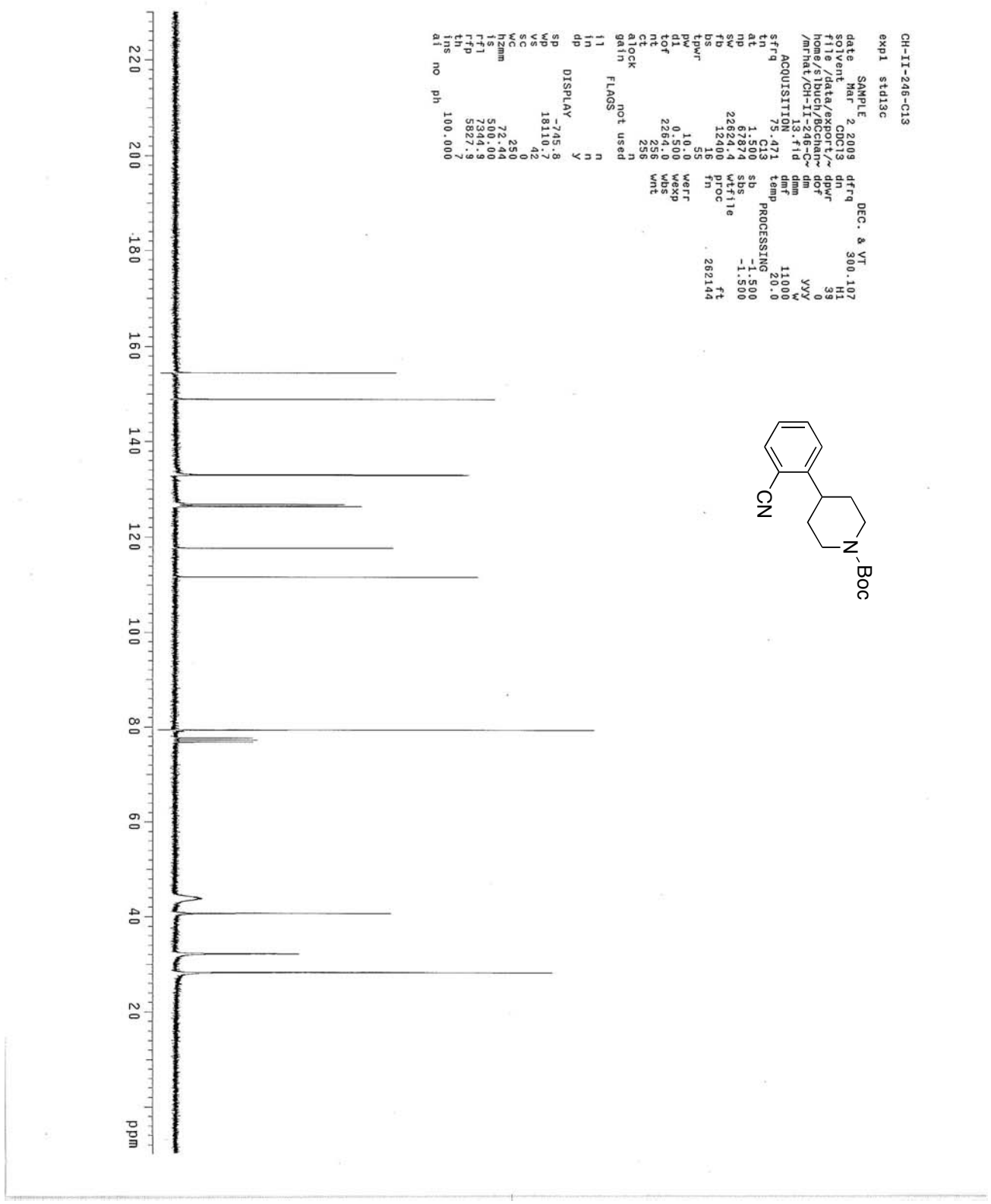
SAMPLE		DEC. & VT	
date	Feb 23 2009	dfrq	300.107
solvent	CDCl3	dn	H1
file	/data/export/~	dpwr	30
home/slbuch/BCchan~		dof	0
/mrhat/CH-II-255-H~		dm	nnn
	1.fid	dmm	c
ACQUISITION		dmf	200
sfrq	300.108	temp	20.0
tn	H1	PROCESSING	
at	4.003	wtfile	
np	48052	proc	ft
sw	6002.4	fn	131072
fb	not used		
bs	4	werr	
tpwr	54	wexp	
pw	8.0	wbs	
d1	0.050	wnt	
tof	867.7		
nt	8		
ct	8		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-298.4		
wp	4201.1		
vs	94		
sc	0		
wc	250		
hzmm	16.80		
is	133.04		
rfl	2839.5		
rfp	2181.8		
th	20		
ins	1.000		
nm	ph		






CH-II-255-C13
exp1 std13c
SAMPLE
date Mar 3 2009
solvent CDCl3
file /data/export/~
home/s1buch/BCchan~
/mrhat/CH-II-255d~
C13.fid
ACQUISITION
sfrq 75.471
tn C13
at 1.500
np 67874
sw 22624.4
fb 12400
bs 16
tpwr 55
pw 10.0
d1 0.500
tof 226.0
nt 256
ct 256
alock n
gain 50
FLAGS
il n
in n
dp y
DISPLAY
sp -779.5
wp 18129.5
vs 30
sc 0
wc 250
hzmm 72.52
is 500.00
rfl 7322.1
rfp 5827.9
th 9
ins 9
al no ph 100.000
DEC. & VT
dfrq 300.107
dn H1
dpwr 39
dof 0
dm yyy
dmm w
dmf 11000
temp 20.0
PROCESSING
sb -1.500
sbs -1.500
wtfile
proc ft
fn 262144
werr
wexp
wbs
wnt
F3C
N
F
220
200
180
160
140
120
100
80
60
40
20
ppm

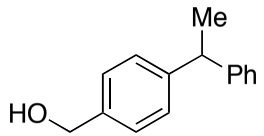

CH-II-246
exp2 std1h
SAMPLE
date Mar 11 2009
solvent CDCl3
file /data/export/~
home/slbuch/BCchan~
/mrhat/CH-II-246-H~
1.fid
ACQUISITION
sfrq 300.108
tn H1
at 4.003
np 48052
sw 6002.4
fb
bs
tpwr 54
pw 8.50
dl 0.050
tof 867.7
nt 32
ct 16
alock n
gain
FLAGS
il n
in n
dp y
DISPLAY
sp -298.6
wp 4201.1
vs 596
sc 0
wc 250
hzmm 16.80
is 86.13
rfl 2839.6
rfp 2181.8
th 20
ins 1.000
nm ph
DEC. & VT
dfrq 300.107
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
temp 20.0
PROCESSING
wtfile
proc ft
fn 131072
not used
not used
not used
werr
wexp
wbs
wnt
CN
N
Boc
0.88
1.00
1.99
2.07
1.04
2.09
1.96
2.00
9.79
12
11
10
9
8
7
6
5
4
3
2
1
-0
ppm

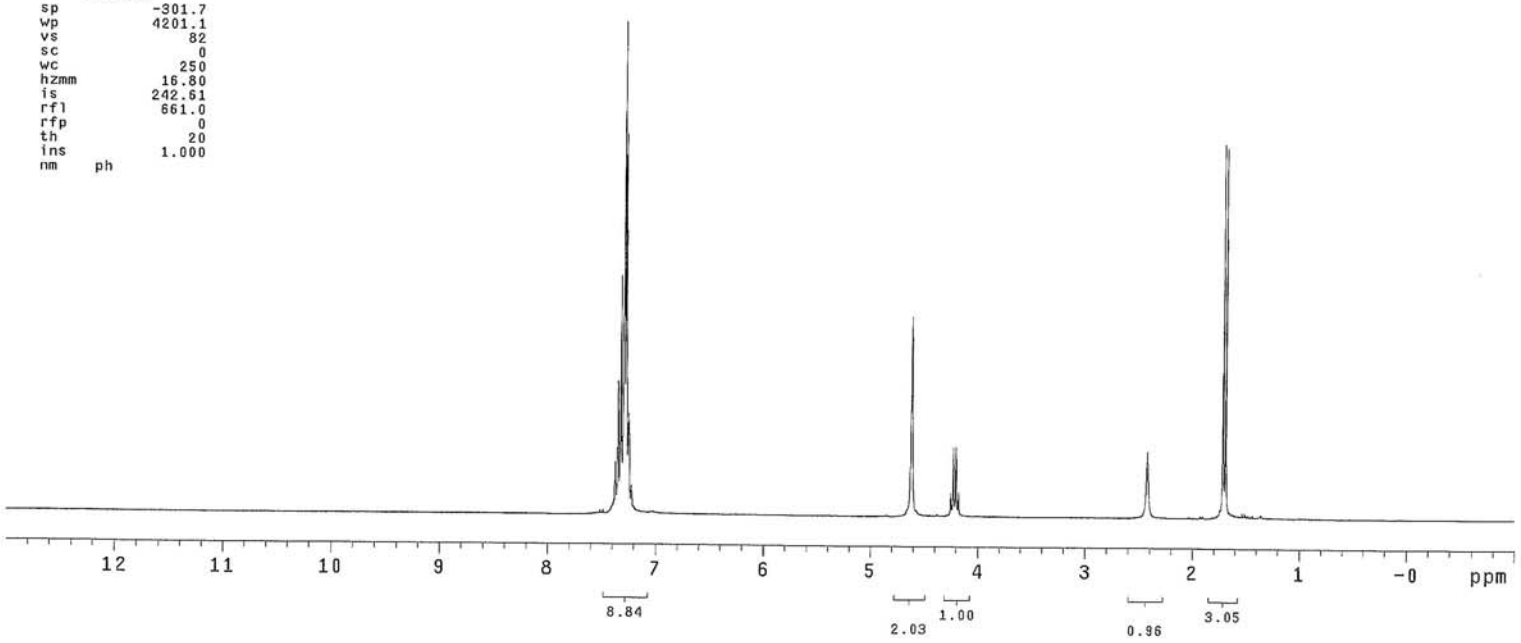

CH-II-246-C13
exp1 std13c
SAMPLE
date Mar 2 2009
solvent CDCl3
file /data/export/~
home/slbuch/BCchan~
/mrhat/CH-II-246-C~
ACQUISITION
sfrq 75.471
tn C13
at 1.500
np 67874
sw 22624.4
fb 12400
bs 16
tpwr 55
pw 10.0
d1 0.500
tof 2264.0
nt 256
ct 256
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -745.8
wp 1810.7
vs 42
sc 0
wc 250
hzmm 72.44
is 500.00
rfl 7344.9
rfp 5827.7
th 7
ins
al no ph 100.000
DEC. & VT
dfrq 300.107
dn H1
dpwr 39
dof 0
dm yyy
dmm w
dmf 11000
temp 20.0
PROCESSING
sb -1.500
sbs -1.500
wtfile
proc ft
fn 262144
werr
wexp
wbs
wnt
CN
N
Boc
220
200
180
160
140
120
100
80
60
40
20
ppm

CH-III-8

exp2 std1h

SAMPLE		DEC. & VT	
date	Mar 13 2009	dfrq	300.107
solvent	CDCl3	dn	H1
file	/data/export/~	dpwr	30
home/slbuch/BCchan~		dof	0
/mrhat/CH-III-8-H1~		dm	nnn
	.fid	dmm	c
ACQUISITION		dmf	200
sfrq	300.108	temp	20.0
tn	H1	PROCESSING	
at	4.003	wtfile	
np	48052	proc	ft
sw	6002.4	fn	131072
fb	not used		
bs	4	werr	
tpwr	54	wexp	
pw	8.0	wbs	
d1	0.050	wnt	
tof	867.7		
nt	32		
ct	8		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-301.7		
wp	4201.1		
vs	82		
sc	0		
wc	250		
hzmm	16.80		
is	242.61		
rfl	661.0		
rfp	0		
th	20		
ins	1.000		
nm	ph		

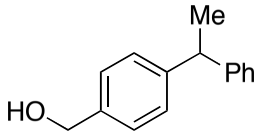


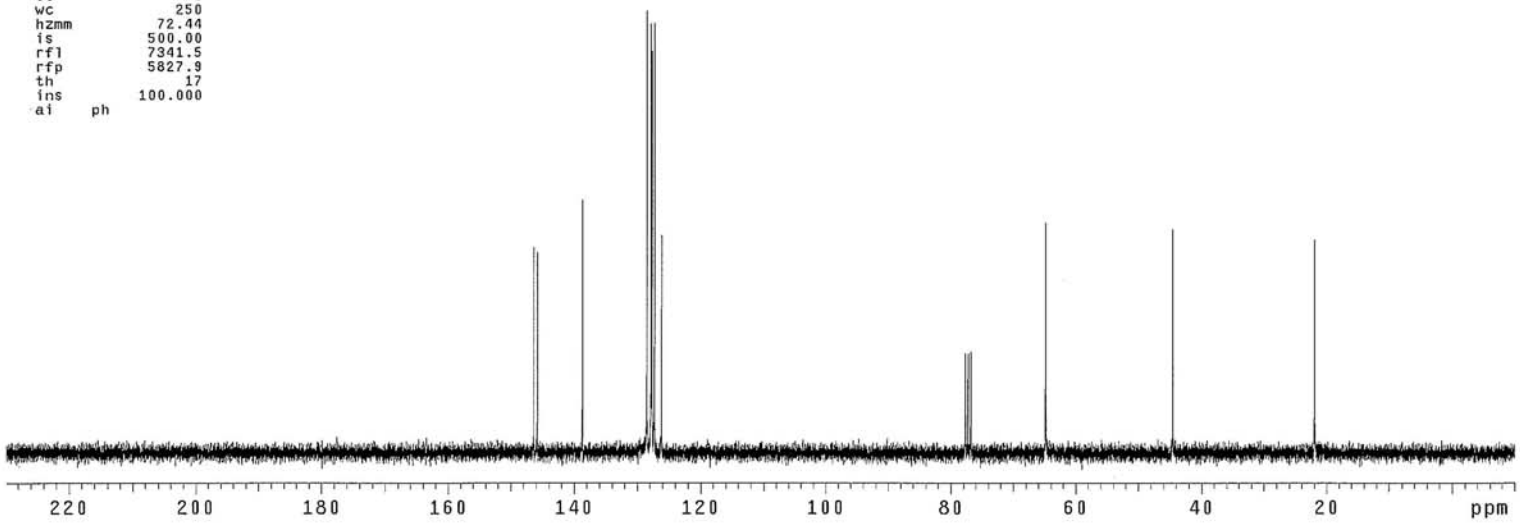


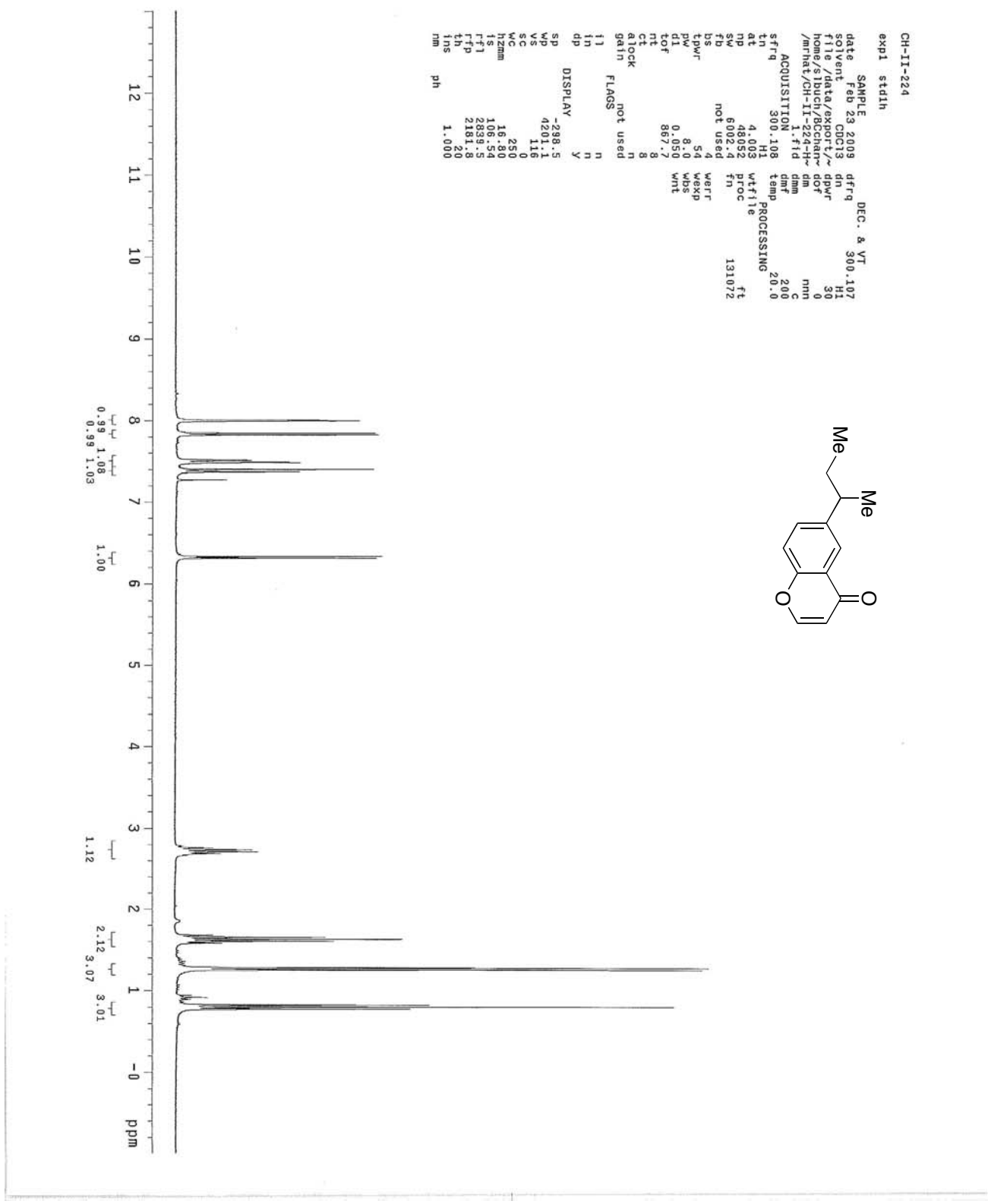
CH-III-8-C13

exp2 std13c

SAMPLE		DEC. & VT	
date	Mar 13 2009	dfrq	300.107
solvent	CDCl3	dn	H1
file	/data/export/~	dpwr	39
home/slbuch/BCchan~		dof	0
/mrhat/CH-III-8-C1~		dm	yyy
	3.fid	dmm	w
ACQUISITION		dmf	11000
sfrq	75.471	temp	20.0
tn	C13	PROCESSING	
at	1.500	sb	-1.500
np	67874	sbs	-1.500
sw	22624.4	wtfile	
fb	12400	proc	ft
bs	16	fn	262144
tpwr	55		
pw	10.0	werr	
d1	0.500	wexp	
tof	2264.0	wbs	
nt	256	wnt	
ct	64		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-761.2		
wp	18110.7		
vs	22		
sc	0		
wc	250		
hzmm	72.44		
is	500.00		
rfl	7341.5		
rfp	5827.9		
th	17		
ins	100.000		
ai ph			






CH-II-224
expl stdih
SAMPLE
date Feb 23 2009
solvent CDCl3
file /data/export/~
home/sibuch/BCchan~
/mrhat/CH-II-224-H~
1.fid
ACQUISITION
sfrq 300.108
tn H1
at 4.003
np 48052
sw 6002.4
fb
bs 4
tpwr 54
pw 8.0
d1 0.050
tof 867.7
nt 8
ct 8
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -298.5
wp 4201.1
vs 116
sc 0
wc 250
hzmm 16.80
fs 106.54
rfl 2839.5
rfp 2181.8
th 20
ins 1.000
nm
ph
DEC. & VT
dfrq 300.107
dn H1
dpwr 30
dcf 0
dm nnn
dmm c
dmf 200
temp 20.0
PROCESSING
wtfile
proc ft
fn 131072
werr
wexp
wbs
wnt
Me
Me
O
O
12
11
10
9
8
7
6
5
4
3
2
1
-0
ppm
0.99
1.08
0.99
1.03
1.00
1.12
2.12
3.07
3.01

CH-II-224-C13

exp1 std13c

SAMPLE		DEC. & VT	
date	Feb 23 2009	dfrq	300.107
solvent	CDCl3	dn	H1
file	/data/export/~	dpwr	39
home/slbuch/BCchan~		dof	0
/mrhat/CH-II-224-C~		dm	yyy
	13.fid	dmm	w
ACQUISITION		dmf	11000
sfrq	75.471	temp	20.0
tn	C13	PROCESSING	
at	1.500	sb	-1.500
np	67874	sbs	-1.500
sw	22624.4	wtfile	
fb	12400	proc	ft
bs	16	fn	262144
tpwr	55		
pw	10.0	werr	
d1	0.500	wexp	
tof	2264.0	wbs	
nt	256	wnt	
ct	256		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-762.2		
wp	18110.7		
vs	76		
sc	0		
wc	250		
hzmm	72.44		
is	500.00		
rfl	7323.7		
rfp	5827.9		
th	9		
ins	1.000		
ai ph