

**A Facile Development of Chiral Alkenylboranes from Chiral Diynes for Asymmetric
Hydrogenation of Silyl Enol Ethers**

Xiaoyu Ren, Gen Li, Simin Wei, and Haifeng Du*

Beijing National Laboratory for Molecular Sciences, CAS Key laboratory of Molecular Recognition and Function, Institute of Chemistry, Chinese Academy of Sciences, Beijing 100190, China

Supporting Information

General consideration: All air-sensitive compounds were handled under an atmosphere of argon or in a nitrogen-filled glovebox. ^1H NMR and ^{13}C NMR spectra were recorded on Bruker AV 400 at ambient temperature with CDCl_3 as solvent and TMS as internal standard. Chemical shifts (δ) were given in ppm, referenced to the residual proton resonance of TMS (0), to the carbon resonance of the CDCl_3 (77.23). Coupling constants (J) were given in Hertz (Hz). IR spectrums were recorded on Perkin-Elmer-983 spectrometer. Optical rotations were measured with PerkinElmer 341 polarimeter. Flash column chromatography was performed on silica gel (200-300 mesh). All solvents were purified by conventional methods, distilled before use. Commercially available reagents were used without further purification. $\text{H}(\text{C}_6\text{F}_5)_2$ was prepared according to the reported method (D. J. Parks, W. E. Piers, G. P. A. Yap, *Organometallics* **1998**, *17*, 5492-5503).

Representative procedure for the synthesis of chiral diyne **5a:**

Synthesis of compound **8a:** A solution of PPh_3 (2.6229 g, 10 mmol) in CH_2Cl_2 (5 mL) was added dropwise to a solution of CBr_4 (1.6581 g, 5 mmol) in CH_2Cl_2 (3 mL) at 0 °C, stirred for 1 h under argon atmosphere. A solution of **7a** (0.6870 g, 1.0 mmol) in CH_2Cl_2 (2 mL) was added dropwise and the mixture was stirred for an additional 1 h. The solution was filtered through a short of silica gel column and washed with CH_2Cl_2 . Then the solution of filtration was evaporated and petroleum ether was added. The resulting solution was filtered again through a short of silica gel column and washed with petroleum ether to remove triphenylphosphine oxide. The solvent was then removed under reduced pressure to yield compound **8a** (0.9587 g, 96% yield). Compound **8a** was used for the next step directly without isolation.

Synthesis of compound **5a:** Compound **8a** (0.9587 g, 0.96 mmol) was dissolved in THF (10 mL) and cooled to -78 °C under argon atmosphere. *n*-BuLi (2.5 M in pentane, 3.8 mL, 9.6 mmol) was added dropwise over 15 min and the solution was stirred at -78 °C for 1 h, then the addition of saturated NH_4Cl solution (10 mL) and warm to room temperature for 30 min. The solution was extracted with Et_2O (3×10 mL). The combined organic phase was washed with brine, dried over Na_2SO_4 , filtered. After removal of

solvents, the residue was purified by column chromatography on silica gel (petroleum ether) to afford compound **5a** as a light yellow foam (0.3520 g, 54% yield).

O. Vechorkin, A. Godinat, R. Scopelliti, and X. Hu, *Angew. Chem.* **2011**, *123*, 11981–11985; *Angew. Chem. Int. Ed.* **2011**, *50*, 11777–11781.

Procedure for the synthesis of chiral diyne **5f:** A solution of compound **5a** (0.2765 g, 0.4 mmol), in THF (4 mL) was cooled to -40 °C under argon atmosphere. *n*-BuLi (2.5 M in pentane, 1.0 mL, 2.5 mmol) was added dropwise over 10 min. After stirring at -40 °C for 2 h, a solution of iodomethane (0.3548 g, 2.5 mmol) in THF (1 mL) was added and the mixture was stirred for 2 h at room temperature. Saturated NH₄Cl solution (5 mL) and water (10 mL) was added, and the mixture was extracted with Et₂O (3 × 10 mL). The combined organic layers was dried over Na₂SO₄, filtered, and concentrated. The resulting residue was purified by column chromatography on silica gel (petroleum ether) to afford compound **5f** as a light yellow foam (0.1158 g, 74% yield).

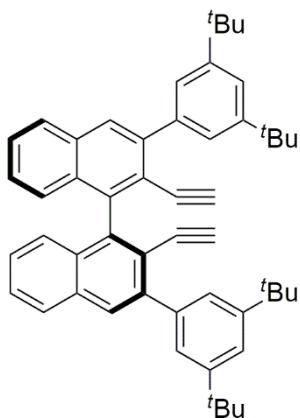
Q. Zhou, H. D. Srinivas, S. Dasgupta, and M. P. Watson, *J. Am. Chem. Soc.* **2013**, *135*, 3307 –3310.

Representative procedure for the metal-free catalytic asymmetric hydrogenation of asymmetric hydrogenation of silyl enol ethers (Table 2, entry 1): To a glass test tube (10 mL), HB(C₆F₅)₂ (0.0138 g, 0.04 mmol), chiral diyne **5a** (0.0136 g, 0.02 mmol), and dry mesitylene (0.2 mL) were added in a nitrogen atmosphere glovebox. The resulting mixture was stirred for 5 min at room temperature followed by addition of P'Bu₃ (10% w in *n*-pentane, 0.0810 g, 0.04 mmol), enol silyl ether **9a** (0.0769 g, 0.4 mmol). Then the tube was moved to a stainless-steel autoclave. After being sealed, the autoclave was purged three times with H₂ and the final pressure of hydrogen was adjusted to 40 bar. The reaction mixture was stirred at 50 °C for 24 h. The reaction mixture was cooled to room temperature and TBAF (0.4 mmol, 0.4 mL, 1.0 M in THF) was added and stirred at room temperature for 1 h. The resulting solution was added water (15 mL) and extracted with CH₂Cl₂ (3 × 10 mL). The combined organic layers was dried over Na₂SO₄, filtered, and concentrated. The resulting residue was purified by column chromatography on

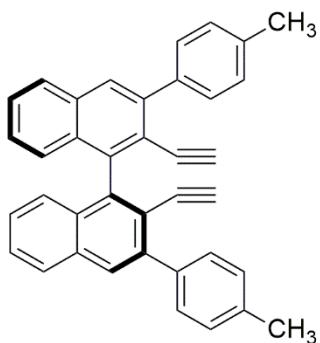
silica gel (petroleum ether/ethyl acetate = 20/1) to afford the desired (*R*)-**10a** as a colorless oil (0.0440 g, 90% yield, 95% ee).

Procedure for NMR study on chiral alkenylborane **6a:** To a glass test tube (10 mL) equipped with a magnetic stir bar was added HB(C₆F₅)₂ (0.0276 g, 0.04 mmol), chiral diyne **5a** (0.0272 g, 0.02 mmol) and C₆D₆ (0.5 mL) in a nitrogen atmosphere glovebox. After stirred at room temperature for 5 min, the resulting mixture was transferred to an NMR tube for NMR studies.

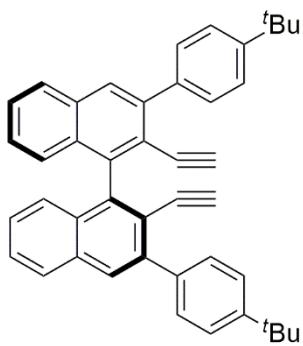
Characterization of chiral diynes:



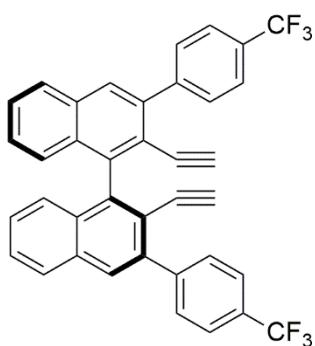
5a: Light yellow foam; m.p. 156-160 °C; $[\alpha]_D^{25} = +103.6$ (*c* 0.50, CHCl₃); IR (film): 3301, 2103, 1594, 1362 cm⁻¹; ¹H NMR (400 MHz, CDCl₃, ppm) δ 8.00 (s, 2H), 7.95 (d, *J* = 8.4 Hz, 2H), 7.64 (s, 2H), 7.63 (s, 2H), 7.50 (dd, *J* = 7.2, 7.2 Hz, 2H), 7.44 (dd, *J* = 1.6, 1.6 Hz, 2H), 7.30 (dd, *J* = 7.2, 7.2 Hz, 2H), 7.26-7.22 (m, 2H), 2.75 (s, 2H), 1.38 (s, 36H); ¹³C NMR (100 MHz, CDCl₃, ppm) δ 150.2, 143.0, 142.2, 139.5, 133.3, 131.6, 128.6, 128.3, 127.2, 126.8, 126.6, 124.5, 121.5, 120.2, 84.6, 82.5, 35.2, 31.8; HRMS (ESI) calcd for C₅₂H₅₅ (M+H): 679.4298; Found: 679.4288.



5b: Light yellow foam; m.p. 108-112 °C; $[\alpha]_D^{25} = +122.8$ (*c* 0.50, CHCl₃); IR (film): 3289, 2102, 1514, 1264 cm⁻¹; ¹H NMR (400 MHz, CDCl₃, ppm) δ 7.93 (s, 2H), 7.91 (d, *J* = 8.0 Hz, 2H), 7.66 (d, *J* = 8.0 Hz, 4H), 7.50 (dd, *J* = 8.0, 8.0 Hz, 2H), 7.34-7.21 (m, 6H), 7.20 (d, *J* = 8.0 Hz, 2H), 2.75 (s, 2H), 2.43 (s, 6H); ¹³C NMR (100 MHz, CDCl₃, ppm) δ 142.8, 141.2, 137.9, 137.4, 133.3, 131.6, 129.8, 128.8, 128.5, 128.2, 127.2, 126.8, 126.5, 120.1, 84.6, 82.1, 21.5; HRMS (ESI) calcd. for C₃₈H₂₇(M+H): 483.2107; Found: 483.2104.

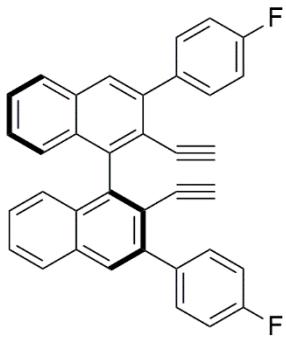


5c: Light yellow foam; m.p. 140-144 °C; $[\alpha]_D^{25} = +114.6$ (*c* 0.50, CHCl₃); IR (film): 3292, 2100, 1516, 1363, 1266 cm⁻¹; ¹H NMR (400 MHz, CDCl₃, ppm) δ 7.96 (s, 2H), 7.91 (d, *J* = 8.4 Hz, 2H), 7.72 (d, *J* = 8.4 Hz, 4H), 7.51-7.45 (m, 6H), 7.31-7.24 (m, 2H), 7.22-7.18 (m, 2H), 2.74 (s, 2H), 1.38 (s, 18H); ¹³C NMR (100 MHz, CDCl₃, ppm) δ 150.6, 142.9, 141.0, 137.7, 133.3, 131.6, 129.6, 128.6, 128.3, 127.2, 126.9, 126.6, 125.0, 120.0, 84.8, 82.1, 34.8, 31.6; HRMS (ESI) calcd. for C₄₄H₃₉(M+H): 567.3046; Found: 567.3040.

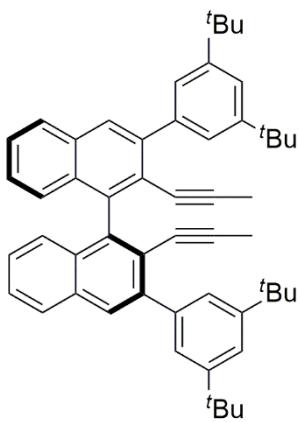


5d: Light yellow foam; m.p. 180-185 °C; $[\alpha]_D^{25} = +77.8$ (*c* 0.50, CHCl₃); IR (film): 3291, 2102, 1616, 1324 cm⁻¹; ¹H NMR (400 MHz, CDCl₃, ppm) δ 7.97 (s, 2H), 7.96 (d, *J* = 8.0 Hz, 2H), 7.87 (d, *J* = 8.0 Hz, 4H), 7.72 (d, *J* = 8.4 Hz, 4H), 7.55 (dd, *J* = 8.0, 8.0 Hz, 2H), 7.36 (dd, *J* = 8.0, 8.0 Hz, 2H), 7.23 (d, *J* =

8.4 Hz, 2H), 2.77 (s, 2H); ^{13}C NMR (100 MHz, CDCl_3 , ppm) δ 144.2, 142.7, 139.8, 133.1, 131.9, 130.3, 129.8 (q, $J_{\text{C}-\text{F}} = 32.2$ Hz), 129.0, 128.5, 127.7, 127.6, 126.5, 125.1, 124.5 (q, $J_{\text{C}-\text{F}} = 270.0$ Hz), 119.6, 85.3, 81.4; ^{19}F NMR (470 MHz, CDCl_3 , ppm) δ -62.4 (s, 6F); HRMS (ESI) calcd. for $\text{C}_{38}\text{H}_{21}\text{F}_6$ ($\text{M}+\text{H}$): 591.1542; Found: 591.1537.



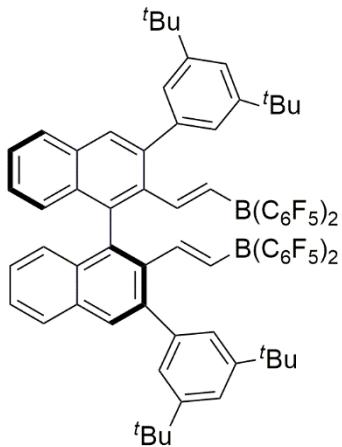
5e: Light yellow foam; m.p. 107-112 °C; $[\alpha]_D^{25} = +87.2$ (c 0.50, CHCl_3); IR (film): 3292, 2102, 1605, 1510, 1265 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 , ppm) δ 7.94 (s, 2H), 7.93 (d, $J = 8.0$ Hz, 2H), 7.71 (m, 4H), 7.52 (dd, $J = 7.2, 7.2$ Hz, 2H), 7.34 (dd, $J = 7.2, 7.2$ Hz, 2H), 7.21 (d, $J = 8.0$ Hz, 2H), 7.14 (dd, $J = 8.4, 8.4$ Hz, 4H), 2.75 (s, 2H); ^{13}C NMR (100 MHz, CDCl_3 , ppm) δ 162.7 (d, $J_{\text{C}-\text{F}} = 245.0$ Hz), 142.7, 140.2, 136.7 (d, $J_{\text{C}-\text{F}} = 3.0$ Hz), 133.2, 131.6, 131.59 (d, $J_{\text{C}-\text{F}} = 8.3$ Hz), 128.7, 128.3, 127.5, 127.1, 126.5, 120.0, 115.0 (d, $J_{\text{C}-\text{F}} = 21.3$ Hz), 84.9, 81.8; ^{19}F NMR (470 MHz, CDCl_3 , ppm) δ -115.0 (s, 2F); HRMS (ESI) calcd. for $\text{C}_{36}\text{H}_{21}\text{F}_2$ ($\text{M}+\text{H}$): 491.1606; Found: 491.1603.



5f: Light yellow foam; m.p. 140-143 °C; $[\alpha]_D^{22} = +159.4$ (c 0.62, CHCl_3); IR (film): 2220, 1594, 1362, 1246, 1218 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 , ppm) δ 7.96 (s, 2H), 7.93 (d, $J = 8.0$ Hz, 2H), 7.67 (s, 2H), 7.66 (s, 2H), 7.58-7.40 (m, 4H), 7.31-7.20 (m, 4H), 1.48 (s, 6H), 1.40 (s, 36H); ^{13}C NMR (100 MHz,

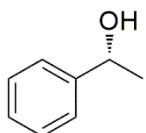
CDCl_3 , ppm) δ 150.1, 142.4, 141.6, 140.2, 132.8, 131.8, 128.13, 128.07, 126.7, 126.4, 126.3, 124.4, 122.0, 121.3, 93.3, 79.1, 35.2, 31.8, 4.7; HRMS (ESI) calcd. for $\text{C}_{54}\text{H}_{59}$ ($\text{M}+\text{H}$): 707.4611; Found: 707.4601.

Characterization of chiral alkenylborane 6a:



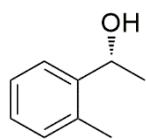
^1H NMR (400 MHz, C_6D_6 , ppm) δ 7.96 (s, 2H), 7.64 (m, 8H), 7.29 (d, $J = 8.4$ Hz, 2H), 7.23-7.18 (m, 4H), 6.88 (dd, $J = 7.6, 7.6$ Hz, 2H), 6.80 (d, $J = 18.0$ Hz, 2H), 1.43 (s, 36H); ^{13}C NMR (100 MHz, C_6D_6 , ppm) δ 163.6, 151.6, 141.7, 141.0, 139.8, 139.6, 134.4, 134.1, 131.5, 131.0, 128.6, 126.4, 124.7, 121.9, 35.2, 31.6 (Quaternary carbons of C_6F_5 ring were not located); ^{19}F NMR (470 MHz, C_6D_6 , ppm): δ -129.5 (d, $J = 18.8$ Hz, 8F), -148.6 (m, 4F), -161.7 (m, 8F); ^{11}B NMR (160 MHz, C_6D_6 , ppm): δ -3.48 (s, 2B); HRMS (ESI) calcd. for $\text{C}_{76}\text{H}_{57}\text{B}_2\text{F}_{20}\text{O}$ ($\text{M}+\text{H}_2\text{O}-\text{H}$): 1387.4282; Found: 1387.4289.

Characterization of products:



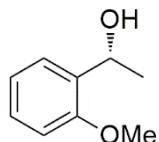
(R)-1-phenylethanol (10a): Colorless oil, 0.0440 g, 90% yield, 95% ee, $[\alpha]_D^{26} = +96.7$ (c 0.28, CHCl_3), [lit.: $[\alpha]_D^{20} = +42.92$ (c 1.04, CHCl_3) (96% ee for *R*-isomer)]; ^1H NMR (400 MHz, CDCl_3 , ppm) δ 7.39-7.32 (m, 4H), 7.30-7.24 (m, 1H), 4.88 (q, $J = 6.4$ Hz, 1H), 1.93 (s, 1H), 1.49 (d, $J = 6.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm) δ 146.0, 128.7, 127.7, 125.6, 70.6, 25.3.

Y. S. Sokeirik, H. Mori, M. Omote, K. Sato, A. Tarui, I. Kumadaki, A. Ando, *Org. Lett.* **2007**, *9*, 1927-1929.



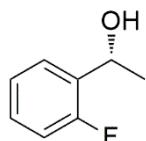
(R)-1-(o-tolyl)ethanol (10b): Colorless oil, 0.0507 g, 93% yield, 98% ee, $[\alpha]_D^{27} = +56.1$ (c 0.96, CHCl_3), [lit.: $[\alpha]_D^{20} = -73.8$ (c 0.90, CHCl_3) (99% ee for *S*-isomer)]; ^1H NMR (400 MHz, CDCl_3 , ppm) δ 7.52 (d, J = 7.6 Hz, 1H), 7.28-7.21 (m, 1H), 7.20-7.10 (m, 2H), 5.18-5.10 (m, 1H), 2.35 (s, 3H), 1.69 (s, 1H), 1.47 (d, J = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm) δ 144.0, 134.4, 130.6, 127.4, 126.6, 124.7, 67.0, 24.1, 19.1.

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(R)-1-(2-methoxyphenyl)ethanol (10c): Colorless oil, 0.0560 g, 92% yield, 99% ee, $[\alpha]_D^{26} = +23.8$ (c 0.98, CHCl_3), [lit.: $[\alpha]_D^{24} = +20.535$ (c 1.14, CHCl_3) (83% ee for *R*-isomer)]; ^1H NMR (400 MHz, CDCl_3 , ppm) δ 7.34 (dd, J = 8.0 Hz, J = 1.6 Hz, 1H), 7.27-7.21 (m, 1H), 6.96 (dd, J = 8.0, 8.0 Hz, 1H), 6.88 (d, J = 8.4 Hz, 1H), 5.09 (q, J = 6.4 Hz, 1H), 3.86 (s, 3H), 2.64 (s, 1H), 1.51 (d, J = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm) δ 156.6, 133.7, 128.4, 126.2, 120.9, 110.5, 66.5, 55.4, 23.1.

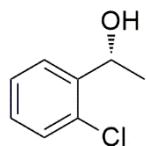
Z. Zhang, P. Jain, J. C. Antilla, *Angew. Chem.* **2011**, *123*, 11153-11156; *Angew. Chem. Int. Ed.* **2011**, *50*, 10961-10964.



(R)-1-(2-fluorophenyl)ethanol (10d): Colorless oil, 0.0477 g, 85% yield, 99% ee, $[\alpha]_D^{26} = +41.6$ (c 0.51, CHCl_3), [lit.: $[\alpha]_D^{20} = +36.61$ (c 1.23, CHCl_3) (89% ee for *R*-isomer)]; ^1H NMR (400 MHz, CDCl_3 , ppm) δ 7.49 (ddd, J = 7.6, 7.6, 1.6 Hz, 1H), 7.29-7.20 (m, 1H), 7.18-7.12 (m, 1H), 7.06-6.97 (m, 1H), 5.20 (q, J = 6.4 Hz, 1H), 1.94 (s, 1H), 1.52 (d, J = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm) δ 160.0 (d, J_{C-F} =

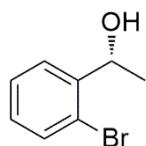
244.0 Hz), 132.8 (d, $J_{C-F} = 14.0$ Hz), 129.0 (d, $J_{C-F} = 8.0$ Hz), 126.8 (d, $J_{C-F} = 5.0$ Hz), 124.5 (d, $J_{C-F} = 3.0$ Hz), 115.5 (d, $J_{C-F} = 2.0$ Hz), 64.7 (d, $J_{C-F} = 3.0$ Hz), 24.2.

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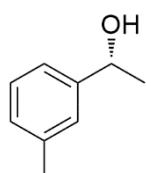
(R)-1-(2-chlorophenyl)ethanol (10e): Colorless oil, 0.0564 g, 90% yield, 99% ee, $[\alpha]_D^{26} = +55.3$ (c 1.91, CHCl₃), [lit.: $[\alpha]_D^{20} = +47.19$ (c 1.53, CHCl₃) (91% ee for *R*-isomer)]; ¹H NMR (400 MHz, CDCl₃, ppm) δ 7.60 (dd, $J = 7.6$ Hz, $J = 1.6$ Hz, 1H), 7.34-7.27 (m, 2H), 7.20 (ddd, $J = 7.6$, 7.6, 1.6 Hz, 1H), 5.30 (qd, $J = 6.4$ Hz, $J = 3.6$ Hz, 1H), 1.98 (d, $J = 3.6$ Hz, 1H), 1.50 (d, $J = 6.4$ Hz, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm) δ 143.3, 131.9, 129.6, 128.6, 127.4, 126.6, 67.2, 23.7.

Y. S. Sokeirik, H. Mori, M. Omote, K. Sato, A. Tarui, I. Kumadaki, A. Ando, *Org. Lett.* **2007**, 9, 1927-1929.



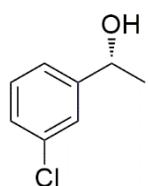
(R)-1-(2-bromophenyl)ethanol (10f): Colorless oil, 0.0708 g, 88% yield, 99% ee, $[\alpha]_D^{26} = +45.2$ (c 1.72, CHCl₃), [lit.: $[\alpha]_D^{20} = -54.5$ (c 1.65, CHCl₃) (99% ee for *S*-isomer)]; ¹H NMR (400 MHz, CDCl₃, ppm) δ 7.59 (dd, $J = 7.6$, $J = 1.6$ Hz, 1H), 7.51 (dd, $J = 8.0$, $J = 0.8$ Hz, 1H), 7.34 (dd, $J = 8.0$, 8.0 Hz, 1H), 7.12 (ddd, $J = 8.0$, 8.0, 1.6 Hz, 1H), 5.24 (q, $J = 6.4$ Hz, 1H), 2.02 (s, 1H), 1.49 (d, $J = 6.4$ Hz, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm) δ 144.8, 132.9, 129.0, 128.1, 126.9, 122.0, 69.4, 23.8.

Y. Li, S. Yu, X. Wu, J. Xiao, W. Shen, Z. Dong, J. Gao, *J. Am. Chem. Soc.* **2014**, 136, 4031-4039.



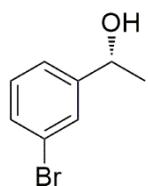
(R)-1-(m-tolyl)ethanol (10g): Colorless oil, 0.0507 g, 93% yield, 89% ee, $[\alpha]_D^{26} = +12.3$ (*c* 0.65, CHCl₃), [lit.: $[\alpha]_D^{20} = -47.2$ (*c* 1.09, CHCl₃) (95% ee for *S*-isomer)]; ¹H NMR (400 MHz, CDCl₃, ppm) δ 7.26-7.20 (m, 1H), 7.19-7.13 (m, 2H), 7.08 (d, *J* = 7.2 Hz, 1H), 4.85 (q, *J* = 6.4 Hz, 1H), 2.36 (s, 3H), 1.91 (s, 1H), 1.48 (d, *J* = 6.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm) δ 146.0, 138.4, 128.6, 128.4, 126.3, 122.6, 70.6, 25.3, 21.6.

Y. Li, S. Yu, X. Wu, J. Xiao, W. Shen, Z. Dong, J. Gao, *J. Am. Chem. Soc.* **2014**, *136*, 4031-4039.



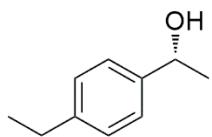
(R)-1-(3-chlorophenyl)ethanol (10h): Colorless oil, 0.0570 g, 91% yield, 97% ee, $[\alpha]_D^{26} = +33.5$ (*c* 1.14, CHCl₃), [lit.: $[\alpha]_D^{20} = +42.4$ (*c* 1.0, CHCl₃) (97% ee for *R*-isomer)]; ¹H NMR (400 MHz, CDCl₃, ppm) δ 7.38 (s, 1H), 7.32-7.20 (m, 3H), 4.88 (q, *J* = 6.4 Hz, 1H), 1.89 (s, 1H), 1.48 (d, *J* = 6.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm) δ 148.1, 134.6, 123.0, 127.8, 125.8, 123.7, 70.0, 25.4.

M. N. Cheemala, M. Gayral, J. M. Brown, K. Rossen, P. Knochel, *Synthesis* **2007**, 3877-3885.



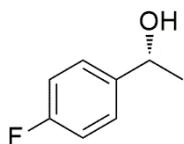
(R)-1-(3-bromophenyl)ethanol (10i): Colorless oil, 0.0740 g, 92% yield, 98% ee, $[\alpha]_D^{26} = +25.8$ (*c* 1.41, CHCl₃), [lit.: $[\alpha]_D^{20} = -32.6$ (*c* 1.57, CHCl₃) (93% ee for *S*-isomer)]; ¹H NMR (400 MHz, CDCl₃, ppm) δ 7.55-7.52 (m, 1H), 7.39 (d, *J* = 7.6 Hz, 1H), 7.28 (d, *J* = 7.6 Hz, 1H), 7.22 (dd, *J* = 7.6, 7.6 Hz, 1H), 4.88 (qd, *J* = 6.4 Hz, *J* = 3.2 Hz, 1H), 1.81 (d, *J* = 3.2 Hz, 1H), 1.49 (d, *J* = 6.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm) δ 148.3, 130.7, 130.3, 128.8, 124.2, 122.8, 69.9, 25.5.

Y. Li, S. Yu, X. Wu, J. Xiao, W. Shen, Z. Dong, J. Gao, *J. Am. Chem. Soc.* **2014**, *136*, 4031-4039.



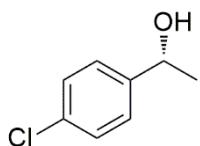
(R)-1-(4-ethylphenyl)ethanol (10j): Colorless oil, 0.0505 g, 84% yield, 92% ee, $[\alpha]_D^{25} = +40.3$ (c 0.78, CHCl₃), [lit.: $[\alpha]_D^{20} = -45.3$ (c 1.13, CHCl₃) (97% ee for *S*-isomer)]; ¹H NMR (400 MHz, CDCl₃, ppm) δ 7.29 (d, J = 8.0 Hz, 2H), 7.18 (d, J = 8.0 Hz, 2H), 4.87 (q, J = 6.4 Hz, 1H), 2.64 (q, J = 7.6 Hz, 2H), 1.77 (s, 1H), 1.49 (d, J = 6.4 Hz, 3H), 1.23 (t, J = 7.6 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm) δ 143.8, 143.3, 128.2, 125.6, 70.5, 28.7, 25.2, 15.8.

Y. Li, S. Yu, X. Wu, J. Xiao, W. Shen, Z. Dong, J. Gao, *J. Am. Chem. Soc.* **2014**, *136*, 4031-4039.



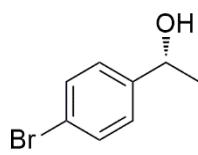
(R)-1-(4-fluorophenyl)ethanol (10k): Colorless oil, 0.0448 g, 80% yield, 98% ee, $[\alpha]_D^{26} = +29.2$ (c 0.32, CHCl₃), [lit.: $[\alpha]_D^{20} = -81.5$ (c 1.58, CHCl₃) (96% ee for *S*-isomer)]; ¹H NMR (400 MHz, CDCl₃, ppm) δ 7.38-7.31 (m, 2H), 7.03 (dd, J = 8.8 Hz, J = 8.4 Hz, 2H), 4.90 (q, J = 6.4 Hz, 1H), 1.77 (s, 1H), 1.49 (d, J = 6.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm) δ 162.3 (d, J_{C-F} = 243.6 Hz), 141.7 (d, J_{C-F} = 3.1 Hz), 127.2 (d, J_{C-F} = 8.0 Hz), 115.4 (d, J_{C-F} = 21.3 Hz), 69.9, 25.4.

Y. Li, S. Yu, X. Wu, J. Xiao, W. Shen, Z. Dong, J. Gao, *J. Am. Chem. Soc.* **2014**, *136*, 4031-4039.



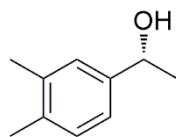
(R)-1-(4-chlorophenyl)ethanol (10l): Colorless oil, 0.0558 g, 89% yield, 93% ee, $[\alpha]_D^{26} = +38.7$ (c 1.12, CHCl₃), [lit.: $[\alpha]_D^{20} = +26.8$ (c 1.0, CHCl₃) (96% ee for *R*-isomer)]; ¹H NMR (400 MHz, CDCl₃, ppm) δ 7.33-7.27 (m, 4H), 4.89 (q, J = 6.4 Hz, 1H), 1.81 (s, 1H), 1.48 (d, J = 6.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm) δ 144.4, 133.3, 128.8, 70.0, 25.5.

M. N. Cheemala, M. Gayral, J. M. Brown, K. Rossen, P. Knochel, *Synthesis* **2007**, 3877-3885.



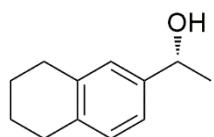
(R)-1-(4-bromophenyl)ethanol (10m): Colorless oil, 0.0740 g, 92% yield, 94% ee, $[\alpha]_D^{27} = +34.6$ (*c* 1.27, CHCl₃), [lit.: $[\alpha]_D^{24} = +30.856$ (*c* 0.915, CHCl₃) (95% ee for *R*-isomer)]; ¹H NMR (400 MHz, CDCl₃, ppm) δ 7.47 (d, *J* = 8.4 Hz, 2H), 7.25 (d, *J* = 8.0 Hz, 2H), 4.87 (q, *J* = 6.4 Hz, 1H), 1.84 (s, 1H), 1.47 (d, *J* = 6.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm) δ 145.0, 131.8, 127.4, 121.4, 70.0, 25.5.

Z. Zhang, P. Jain, J. C. Antilla, *Angew. Chem.* **2011**, *123*, 11153-11156; *Angew. Chem. Int. Ed.* **2011**, *50*, 10961-10964.

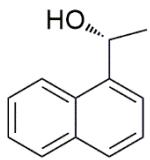


(R)-1-(3,4-dimethylphenyl)ethanol (10n): Colorless oil, 0.0535 g, 89% yield, 91% ee, $[\alpha]_D^{26} = +37.6$ (*c* 1.05, CHCl₃), [lit.: $[\alpha]_D^{20} = +50.0$ (*c* 1.0, CHCl₃) (96% ee for *R*-isomer)]; ¹H NMR (400 MHz, CDCl₃, ppm) δ 7.15 (s, 1H), 7.13-7.07 (m, 2H), 4.84 (q, *J* = 6.4, 1H), 2.27 (s, 3H), 2.25 (s, 3H), 1.74 (s, 1H), 1.48 (d, *J* = 6.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm) δ 143.6, 136.9, 136.0, 123.0, 126.9, 123.0, 70.5, 25.2, 20.0, 19.6.

M. N. Cheemala, M. Gayral, J. M. Brown, K. Rossen, P. Knochel, *Synthesis* **2007**, 3877-3885.

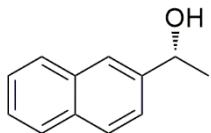


(R)-1-(5,6,7,8-tetrahydronaphthalen-2-yl)ethanol (10o): Colorless oil, 0.0684 g, 97% yield, 96% ee, $[\alpha]_D^{25} = +33.3$ (*c* 2.92, CHCl₃), [lit.: $[\alpha]_D^{20} = -48.3$ (*c* 1.8, CHCl₃) (>99% ee for *S*-isomer)]; ¹H NMR (400 MHz, CDCl₃, ppm) δ 7.11-7.03 (m, 3H), 4.83 (qd, *J* = 6.4 Hz, *J* = 2.8 Hz, 1H), 2.78-2.74 (m, 4H), 1.83-1.76 (m, 4H), 1.70 (d, *J* = 2.8 Hz, 1H), 1.48 (d, *J* = 6.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm) δ 143.2, 137.5, 136.7, 129.5, 126.3, 122.8, 70.5, 29.7, 29.3, 25.2, 23.4.



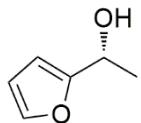
(R)-1-(naphthalen-1-yl)ethanol (10p): Colorless oil, 0.0675 g, 98% yield, 98% ee, $[\alpha]_D^{27} = +53.3$ (c 1.16, CHCl₃), [lit.: $[\alpha]_D^{20} = +68.8$ (c 1.0, CHCl₃) (96% ee for *R*-isomer)]; ¹H NMR (400 MHz, CDCl₃, ppm) δ 8.12 (d, J = 8.0 Hz, 1H), 7.87 (d, J = 7.2 Hz, 1H), 7.78 (d, J = 8.0 Hz, 1H), 7.68 (d, J = 7.2 Hz, 1H), 7.55-7.45 (m, 3H), 5.69 (q, J = 6.4 Hz, 1H), 1.93 (s, 1H), 1.67 (d, J = 6.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm) δ 141.5, 134.0, 130.4, 129.1, 128.1, 126.2, 125.7, 123.3, 122.2, 67.3, 24.5.

M. N. Cheemala, M. Gayral, J. M. Brown, K. Rossen, P. Knochel, *Synthesis* **2007**, 3877-3885.



(R)-1-(naphthalen-2-yl)ethanol (10q): White solid, 0.0668 g, 97% yield, 87% ee, $[\alpha]_D^{28} = +30.4$ (c 0.97, CHCl₃), [lit.: $[\alpha]_D^{20} = +37.77$ (c 1.04, CHCl₃) (95% ee for *R*-isomer)]; ¹H NMR (400 MHz, CDCl₃, ppm) δ 7.87-7.80 (m, 4H), 7.53-7.43 (m, 3H), 5.08 (qd, J = 6.4 Hz, J = 3.2 Hz, 1H), 1.85 (d, J = 3.2 Hz, 1H), 1.59 (d, J = 6.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm) δ 143.4, 133.6, 133.1, 128.5, 128.1, 127.9, 126.4, 126.0, 124.0, 70.7, 25.3.

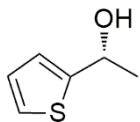
Y. S. Sokeirik, H. Mori, M. Omote, K. Sato, A. Tarui, I. Kumadaki, A. Ando, *Org. Lett.* **2007**, 9, 1927-1929.



(R)-1-(furan-2-yl)ethanol (10r): Colorless oil, 0.0381 g, 85% yield, 94% ee, $[\alpha]_D^{25} = +14.5$ (c 0.47, CHCl₃), [lit.: $[\alpha]_D^{20} = +7.86$ (c 0.9, CHCl₃) (97% ee for *R*-isomer)]; ¹H NMR (400 MHz, CDCl₃, ppm) δ

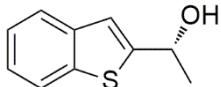
7.37 (s, 1H), 6.32 (s, 1H), 6.22 (d, $J = 2.8$ Hz, 1H), 4.95-4.85 (m, 1H), 1.95 (d, $J = 4.0$ Hz, 1H), 1.54 (d, $J = 6.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm) δ 157.8, 142.1, 110.3, 105.3, 63.8, 21.4.

Y. S. Sokeirik, H. Mori, M. Omote, K. Sato, A. Tarui, I. Kumadaki, A. Ando, *Org. Lett.* **2007**, 9, 1927-1929.



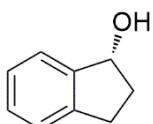
(R)-1-(thiophen-2-yl)ethanol (10s): Colorless oil, 0.0487 g, 95% yield, 96% ee, $[\alpha]_D^{25} = +19.9$ (c 1.04, CHCl_3), [lit.: $[\alpha]_D^{20} = +16.4$ (c 1.0, CHCl_3) (92% ee for *R*-isomer)]; ^1H NMR (400 MHz, CDCl_3 , ppm) δ 7.26-7.22 (m, 1H), 6.98-6.94 (m, 2H), 5.18-5.09 (m, 1H), 2.00 (s, 1H), 1.60 (d, $J = 6.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm) δ 150.1, 126.8, 124.6, 123.4, 66.4, 25.4.

M. N. Cheemala, M. Gayral, J. M. Brown, K. Rossen, P. Knochel, *Synthesis* **2007**, 3877-3885.



(R)-1-(benzo[b]thiophen-2-yl)ethanol (10t): White solid, 0.0706 g, 99% yield, 93% ee, $[\alpha]_D^{24} = +22.9$ (c 2.89, CHCl_3), [lit.: $[\alpha]_D^{24} = -21.2$ (c 1.00, CHCl_3) (>99% ee for *S*-isomer)]; ^1H NMR (400 MHz, CDCl_3 , ppm) δ 7.81 (d, $J = 8.0$ Hz, 1H), 7.72 (d, $J = 8.0$ Hz, 1H), 7.40-7.24 (m, 2H), 7.19 (s, 1H), 5.24-5.17 (m, 1H), 2.07 (d, $J = 4.0$ Hz, 1H), 1.66 (d, $J = 6.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm) δ 150.7, 139.7, 139.5, 124.5, 124.3, 123.6, 122.7, 119.7, 67.0, 25.3.

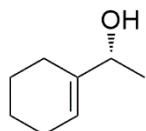
M. I. Tosa, P. V. Podea, C. Paizs, F. D. Irimie, *Tetrahedron: Asymmetry* **2008**, 19, 2068-2071.



(R)-2,3-dihydro-1H-inden-1-ol (10u): Colorless oil, 0.0494 g, 92% yield, 98% ee, $[\alpha]_D^{25} = -24.6$ (c 0.88, CHCl_3), [lit.: $[\alpha]_D^{23} = -23.88$ (c 2.68, CHCl_3) (86% ee for *R*-isomer)]; ^1H NMR (400 MHz, CDCl_3 , ppm) δ 7.43-7.40 (m, 1H), 7.26-7.20 (m, 3H), 5.25 (q, $J = 6.4$ Hz, 1H), 3.11-3.01 (m, 1H), 2.87-2.77 (m, 1H),

2.54-2.44 (m, 1H), 2.02-1.90 (m, 1H), 1.70 (d, $J = 6.4$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3 , ppm) δ 145.2, 143.5, 128.5, 126.9, 125.1, 124.4, 76.7, 36.1, 30.0.

P. He, X. Liu, H. Zheng, W. Li, L. Lin, X. Feng, *Org. Lett.* **2012**, *14*, 5134-5137.

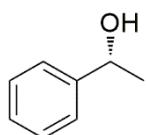


(R)-1-(cyclohex-1-en-1-yl)ethanol (10v): Colorless oil, 0.0414 g, 82% yield, 97% ee, $[\alpha]_D^{23} = +6.9$ (c 0.17, CHCl_3), [lit.: $[\alpha]_D^{20} = -9.5$ (c 1.2, CHCl_3) (97% ee for *S*-isomer)]; ^1H NMR (400 MHz, CDCl_3 , ppm) δ 5.67 (m, 2H), 4.16 (q, $J = 6.4$ Hz, 1H), 2.10-1.90 (m, 4H), 1.66-1.57 (m, 5H), 1.25 (d, $J = 6.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm) δ 141.5, 121.7, 72.4, 25.1, 23.9, 22.9, 22.8, 21.7.

D. R. Li, A. He, J. R. Falck, *Org. Lett.* **2010**, *12*, 1756-1759.

The chromatography for the determination of the enantiomeric excess

Table 2, entry 1



HPLC Conditions: Column: Chiralcel OD-H, Daicel Chemical Industries, Ltd., Eluent: Hexanes/IPA(95/5); Flow rate: 1.0 mL/min; Detection: UV 210 nm

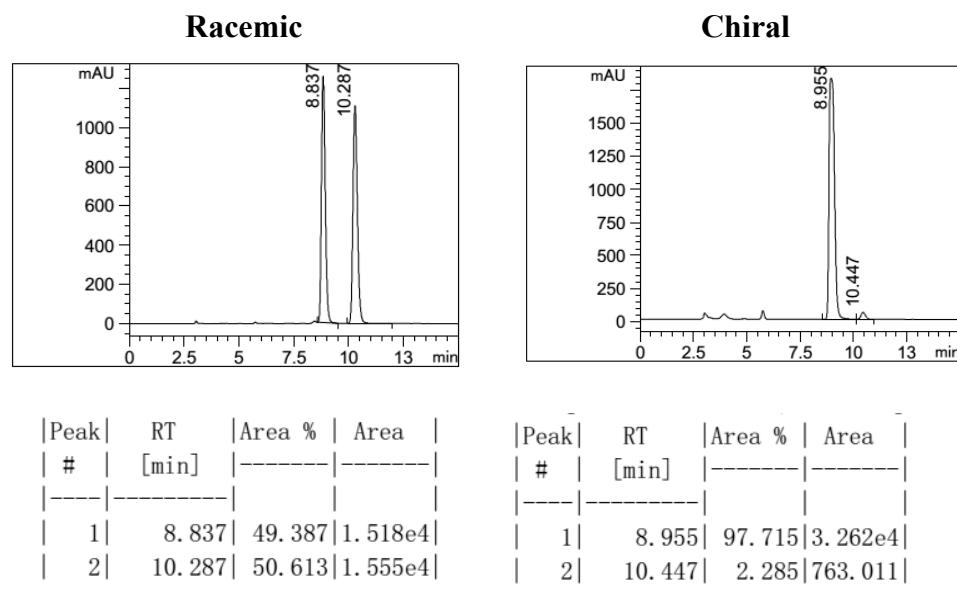
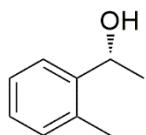


Table 2, entry 2



HPLC Conditions: Column: Chiralcel OD-H, Daicel Chemical Industries, Ltd., Eluent: Hexanes/IPA(99/1); Flow rate: 1.0 mL/min; Detection: UV 210 nm

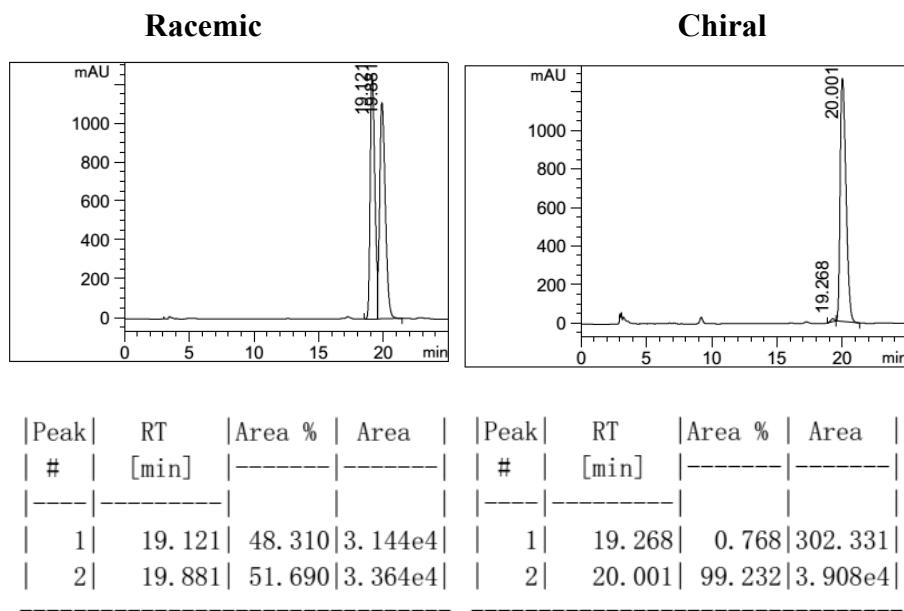
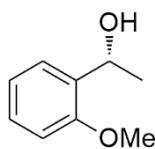
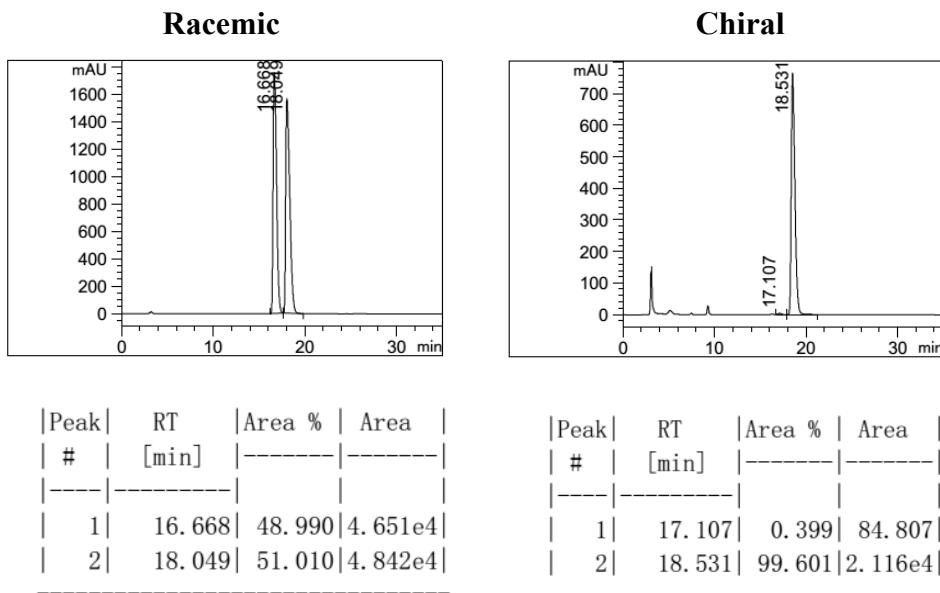
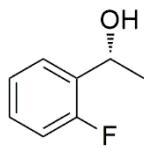


Table 2, entry 3

HPLC Conditions: Column: Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent:** Hexanes/IPA(98/2); **Flow rate:** 1.0 mL/min; **Detection:** UV 210 nm

**Table 2, entry 4**

HPLC Conditions: Column: Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent:** Hexanes/IPA(200/1); **Flow rate:** 1.0 mL/min; **Detection:** UV 210 nm

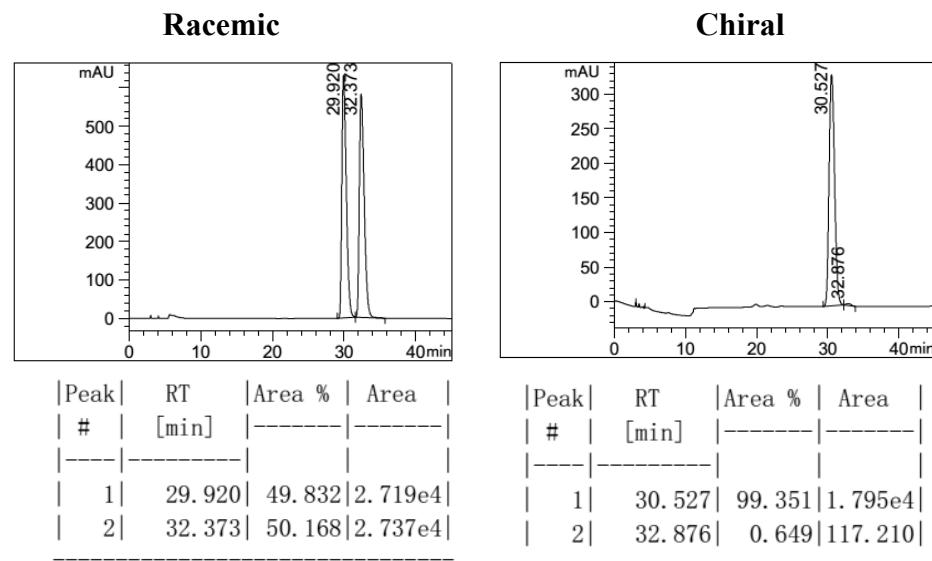
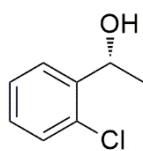
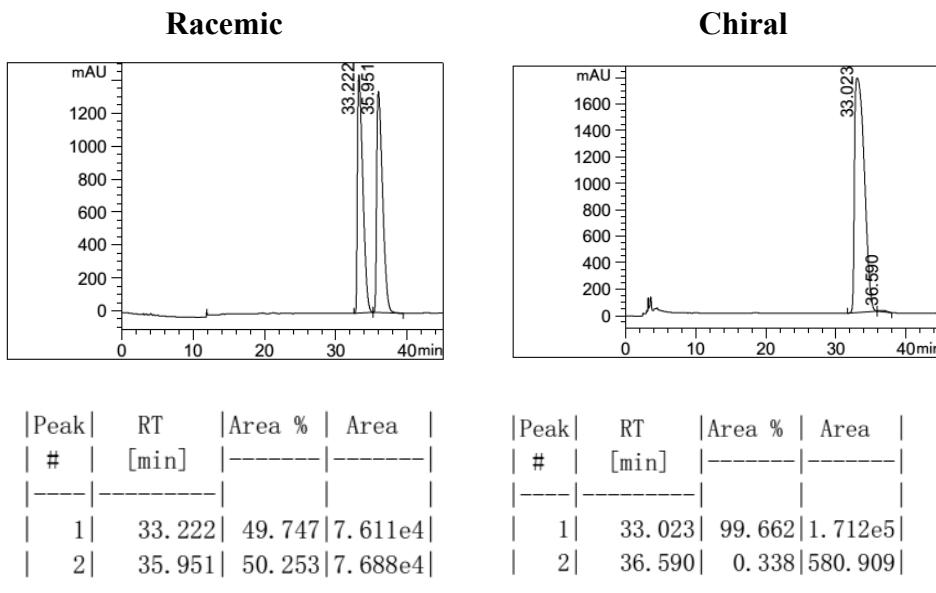
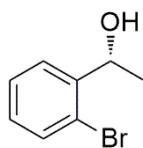


Table 2, entry 5

HPLC Conditions: Column: Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent:** Hexanes/IPA(200/1); **Flow rate:** 1.0 mL/min; **Detection:** UV 210 nm

**Table 2, entry 6**

HPLC Conditions: Column: Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent:** Hexanes/IPA(99/1); **Flow rate:** 1.0 mL/min; **Detection:** UV 210 nm

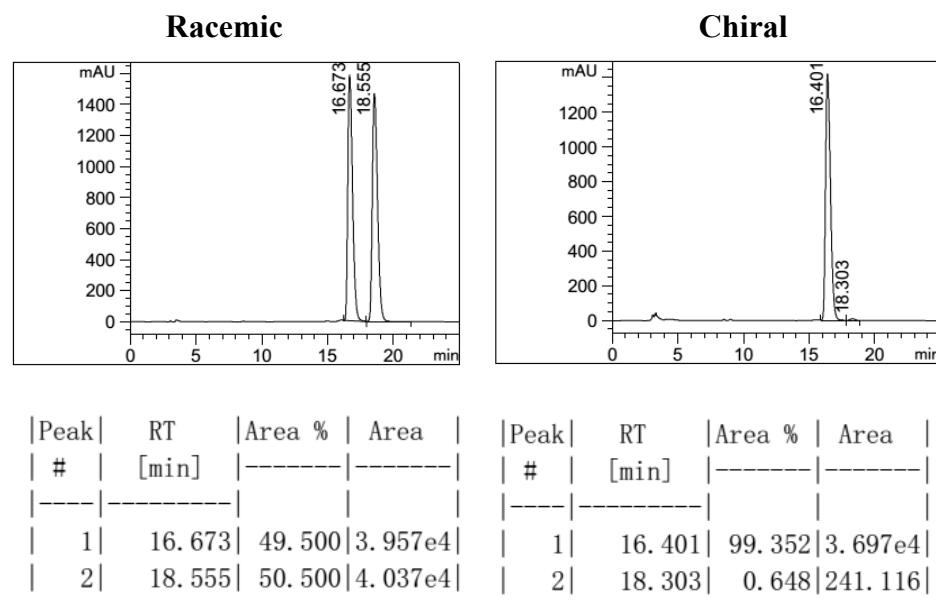
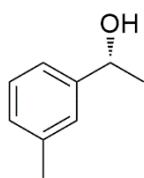
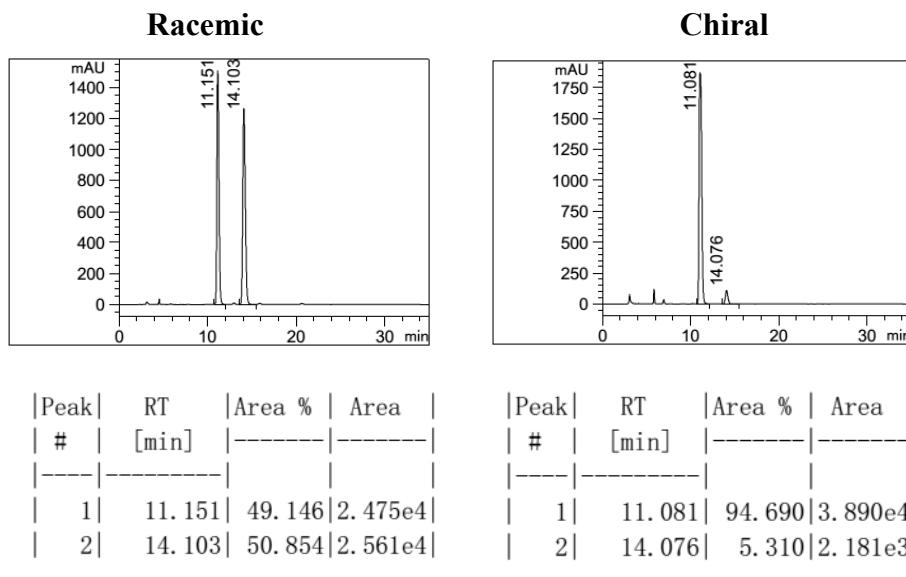
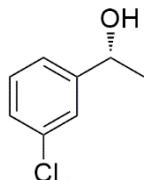


Table 2, entry 7

HPLC Conditions: Column: Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent:** Hexanes/IPA(97/3); **Flow rate:** 1.0 mL/min; **Detection:** UV 210 nm

**Table 2, entry 8**

HPLC Conditions: Column: Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent:** Hexanes/IPA(99/1); **Flow rate:** 1.0 mL/min; **Detection:** UV 210 nm

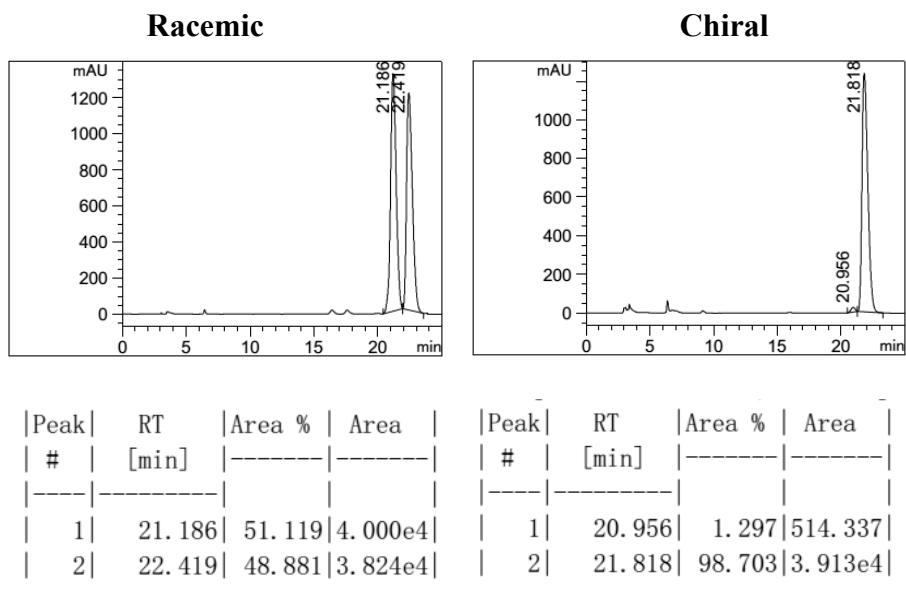
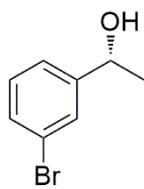
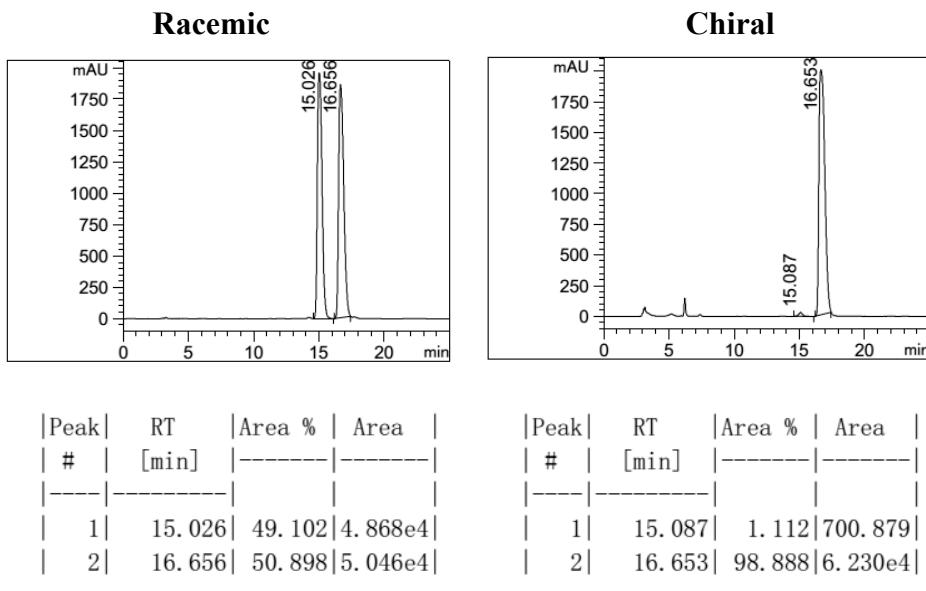
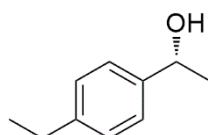


Table 2, entry 9

HPLC Conditions: Column: Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent:** Hexanes/IPA(98/2); **Flow rate:** 1.0 mL/min; **Detection:** UV 210 nm

**Table 2, entry 10**

HPLC Conditions: Column: Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent:** Hexanes/IPA(99/1); **Flow rate:** 1.0 mL/min; **Detection:** UV 210 nm

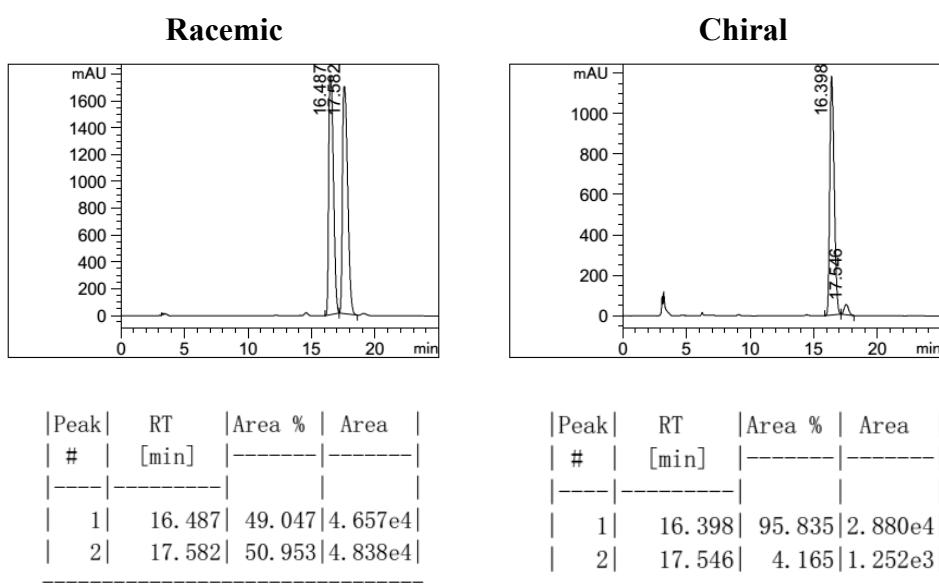
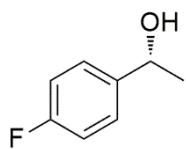
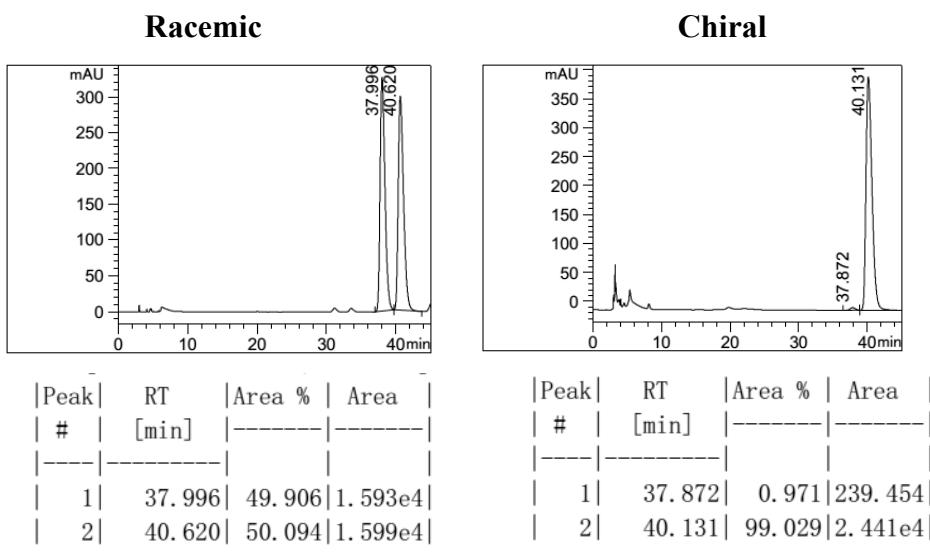
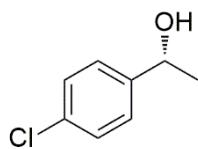


Table 2, entry 11

HPLC Conditions: Column: Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent:** Hexanes/IPA(200/1); **Flow rate:** 1.0 mL/min; **Detection:** UV 210 nm

**Table 2, entry 12**

HPLC Conditions: Column: Chiraldpak IC, Daicel Chemical Industries, Ltd., **Eluent:** Hexanes/IPA(99/1); **Flow rate:** 1.0 mL/min; **Detection:** UV 210 nm

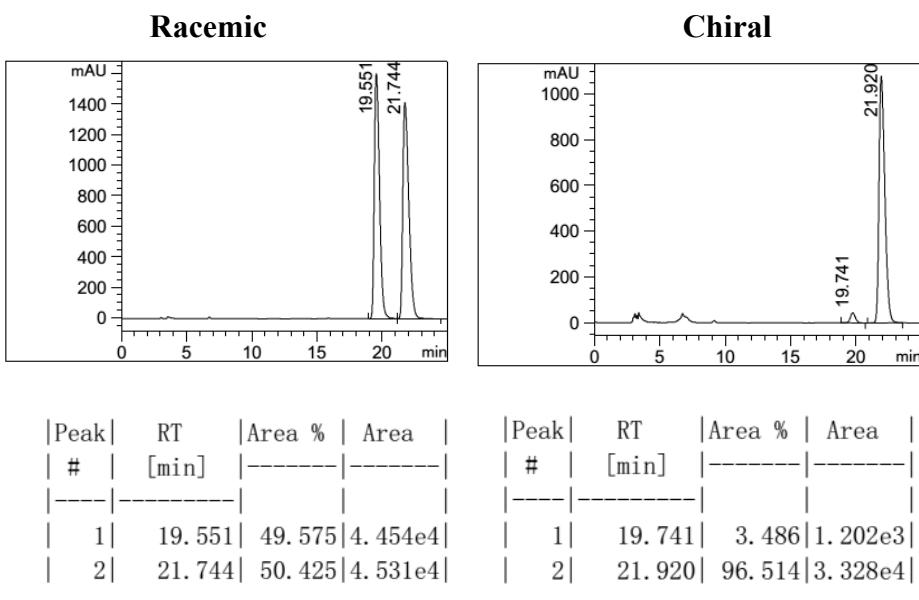
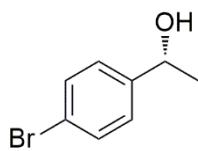
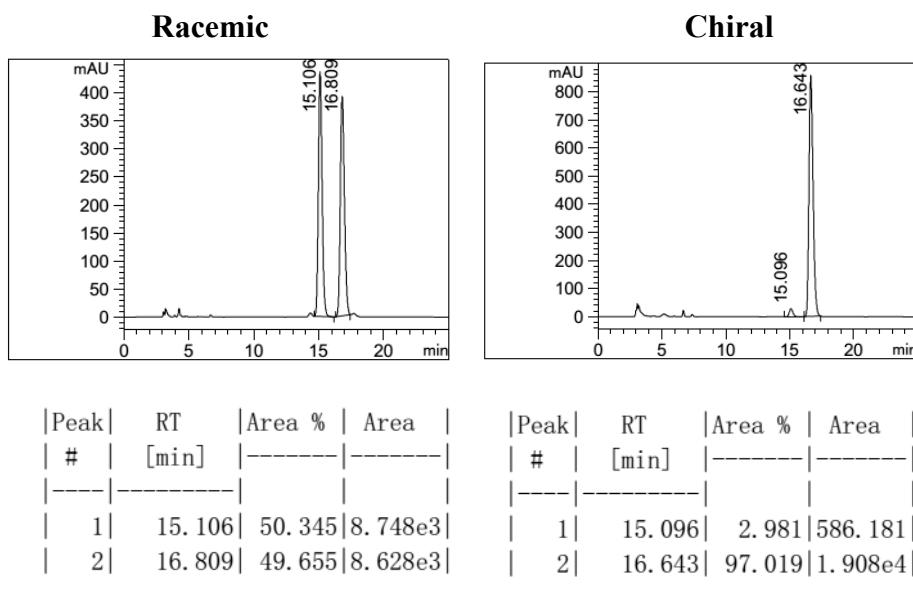
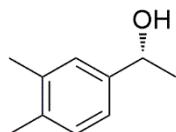


Table 2, entry 13

HPLC Conditions: Column: Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent:** Hexanes/IPA(98/2); **Flow rate:** 1.0 mL/min; **Detection:** UV 210 nm

**Table 2, entry 14**

HPLC Conditions: Column: Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent:** Hexanes/IPA(200/1); **Flow rate:** 1.0 mL/min; **Detection:** UV 210 nm

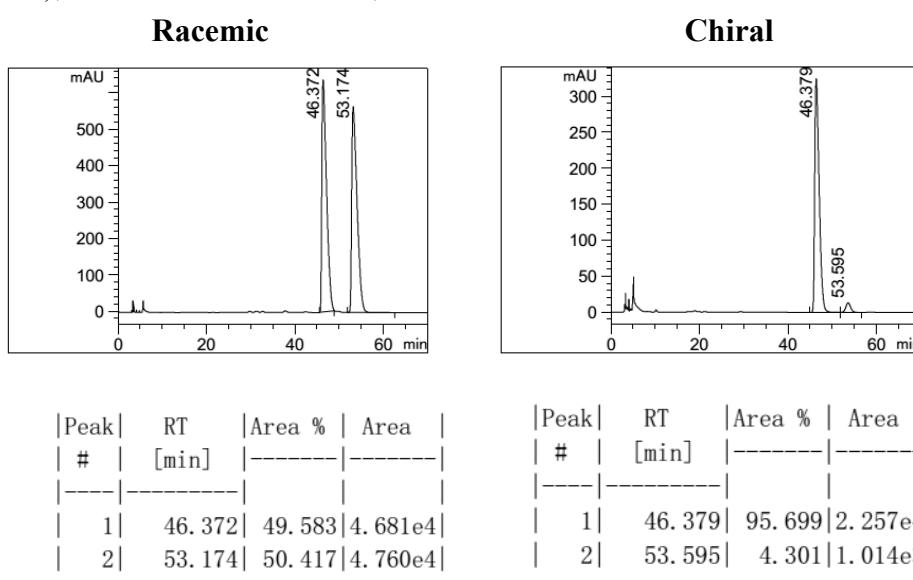
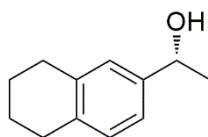
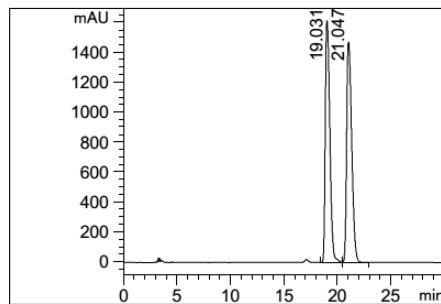
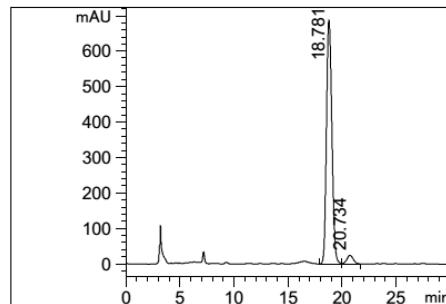


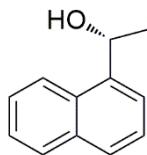
Table 2, entry 15

HPLC Conditions: Column: Chiralcel OD-H, Daicel Chemical Industries, Ltd., **Eluent:** Hexanes/IPA(99/1); **Flow rate:** 1.0 mL/min; **Detection:** UV 210 nm

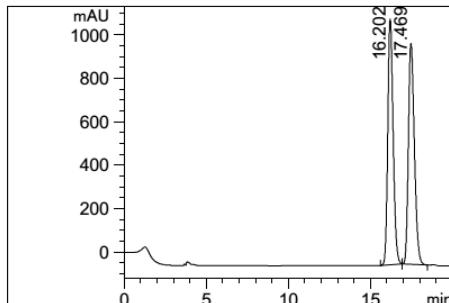
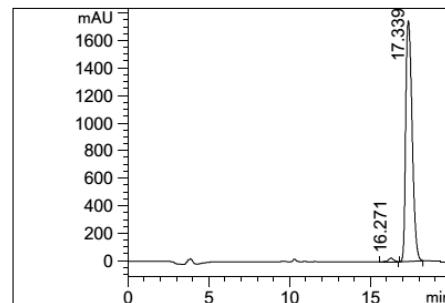
Racemic**Chiral**

Peak #	RT [min]	Area %	Area
1	19.031	49.571	4.775e4
2	21.047	50.429	4.858e4

Peak #	RT [min]	Area %	Area
1	18.781	96.112	2.413e4
2	20.734	3.888	975.952

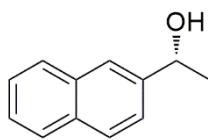
Table 2, entry 16

HPLC Conditions: Column: Chiralcel AS-H, Daicel Chemical Industries, Ltd., **Eluent:** Hexanes/IPA(97/3); **Flow rate:** 1.0 mL/min; **Detection:** UV 210 nm

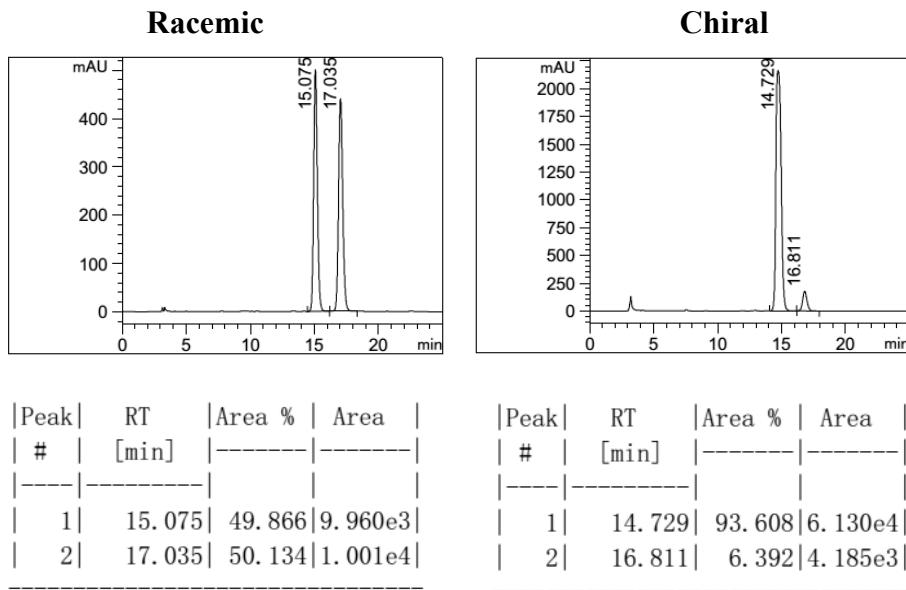
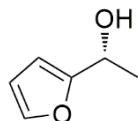
Racemic**Chiral**

Peak #	RT [min]	Area %	Area
1	16.202	49.841	2.565e4
2	17.469	50.159	2.581e4

Peak #	RT [min]	Area %	Area
1	16.271	1.101	534.679
2	17.339	98.899	4.801e4

Table 2, entry 17

HPLC Conditions: Column: Chiralcel AS-H, Daicel Chemical Industries, Ltd., Eluent: Hexanes/IPA(97/3); Flow rate: 1.0 mL/min; Detection: UV 210 nm

**Table 2, entry 18**

HPLC Conditions: Column: Chiralcel OJ-H, Daicel Chemical Industries, Ltd., Eluent: Hexanes/IPA(97/3); Flow rate: 1.0 mL/min; Detection: UV 210 nm

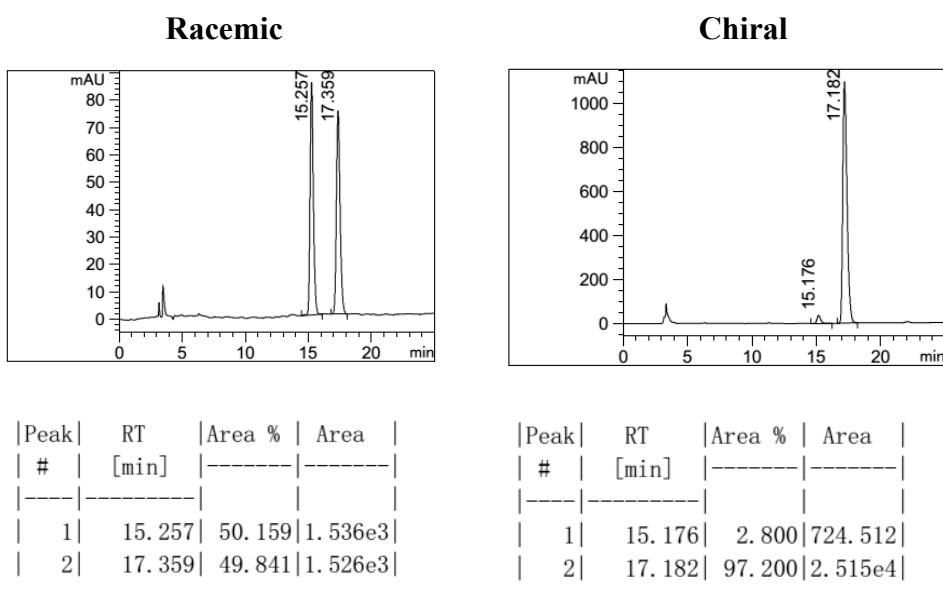
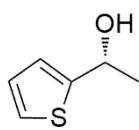
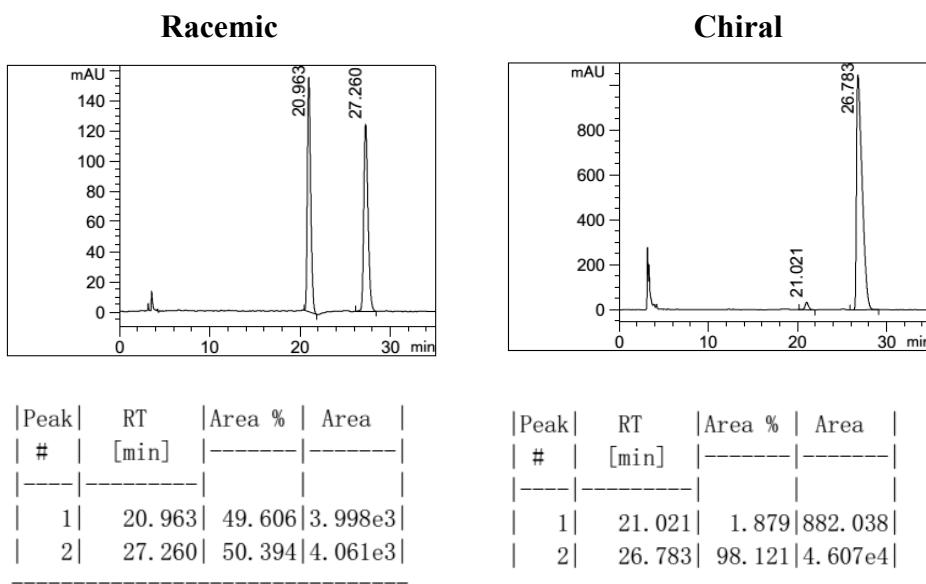
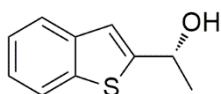


Table 2, entry 19

HPLC Conditions: Column: Chiralcel OJ-H, Daicel Chemical Industries, Ltd., Eluent: Hexanes/IPA(98/2); Flow rate: 1.0 mL/min; Detection: UV 210 nm

**Table 2, entry 20**

HPLC Conditions: Column: Chiralcel OB-H, Daicel Chemical Industries, Ltd., Eluent: Hexanes/IPA(90/10); Flow rate: 1.0 mL/min; Detection: UV 210 nm

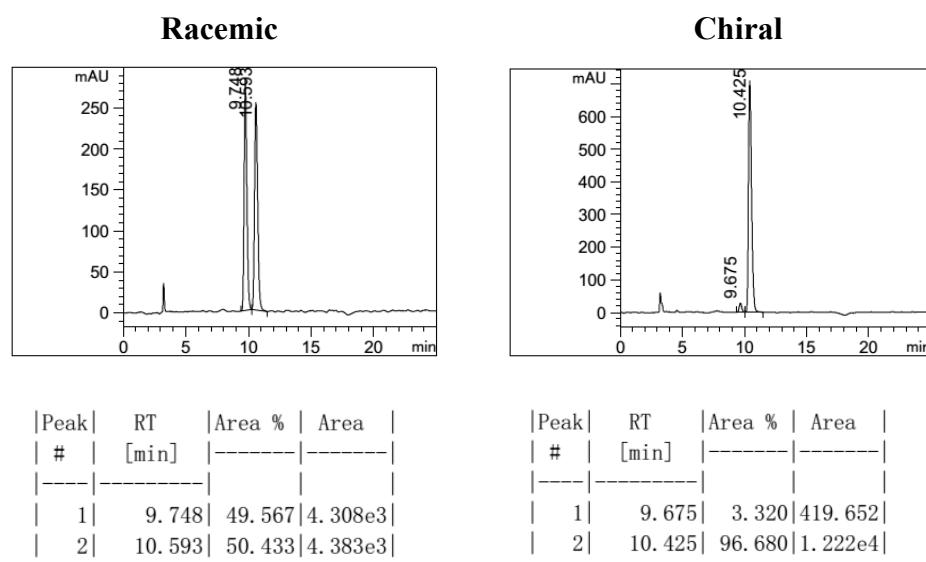
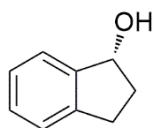
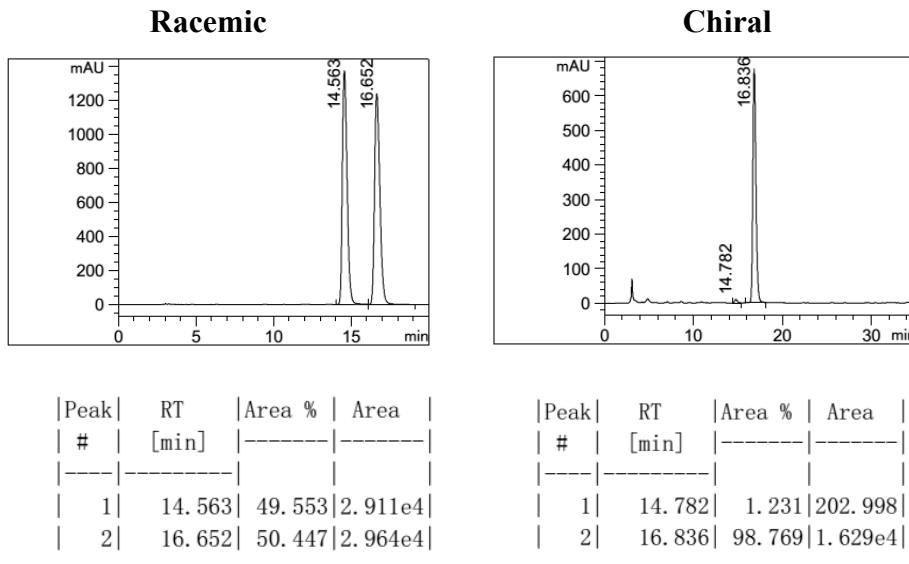
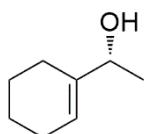
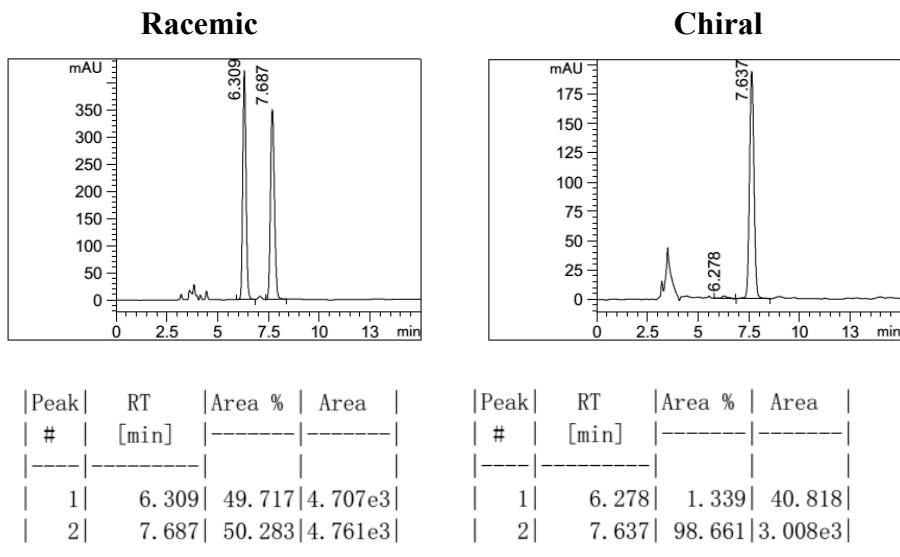


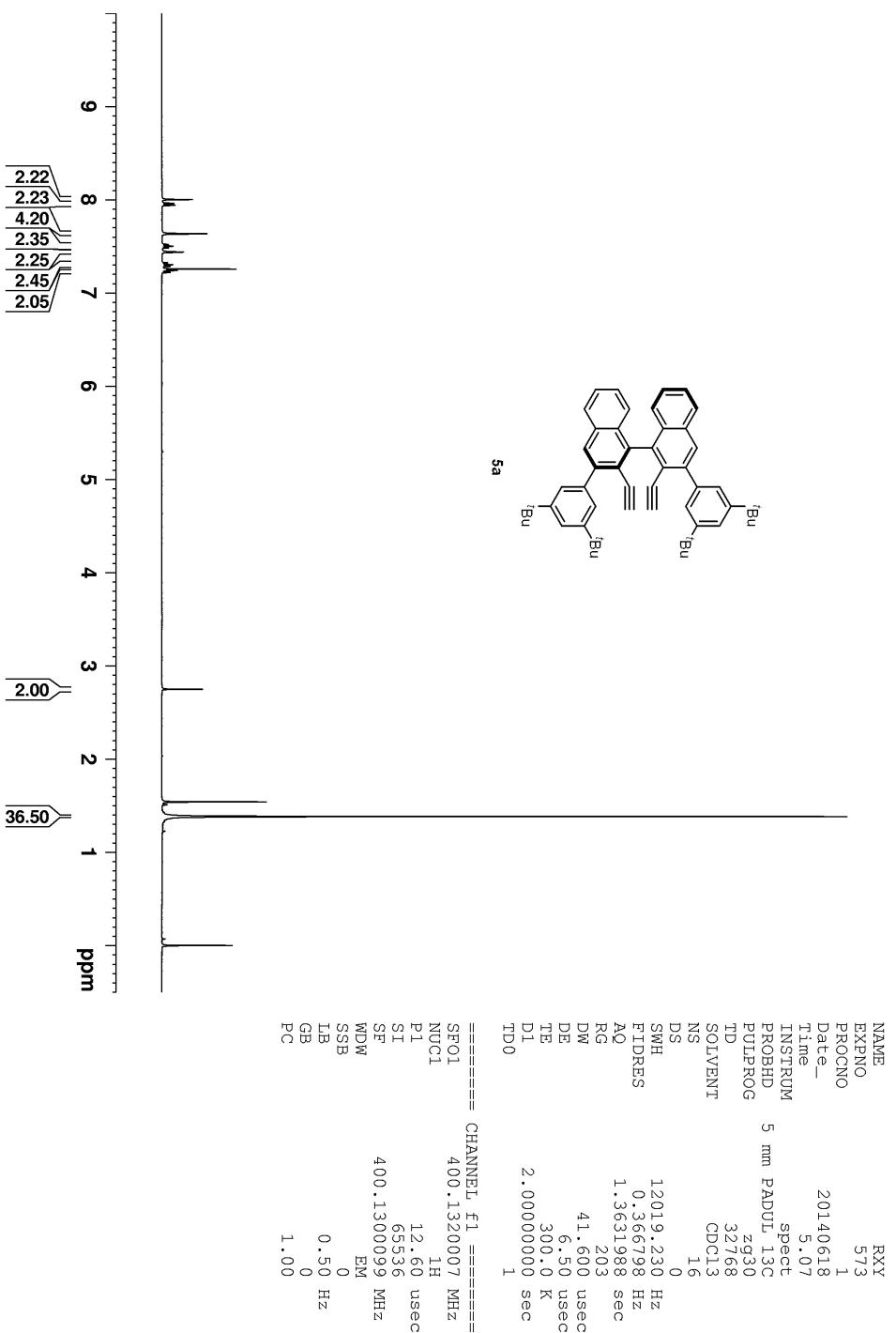
Table 2, entry 21

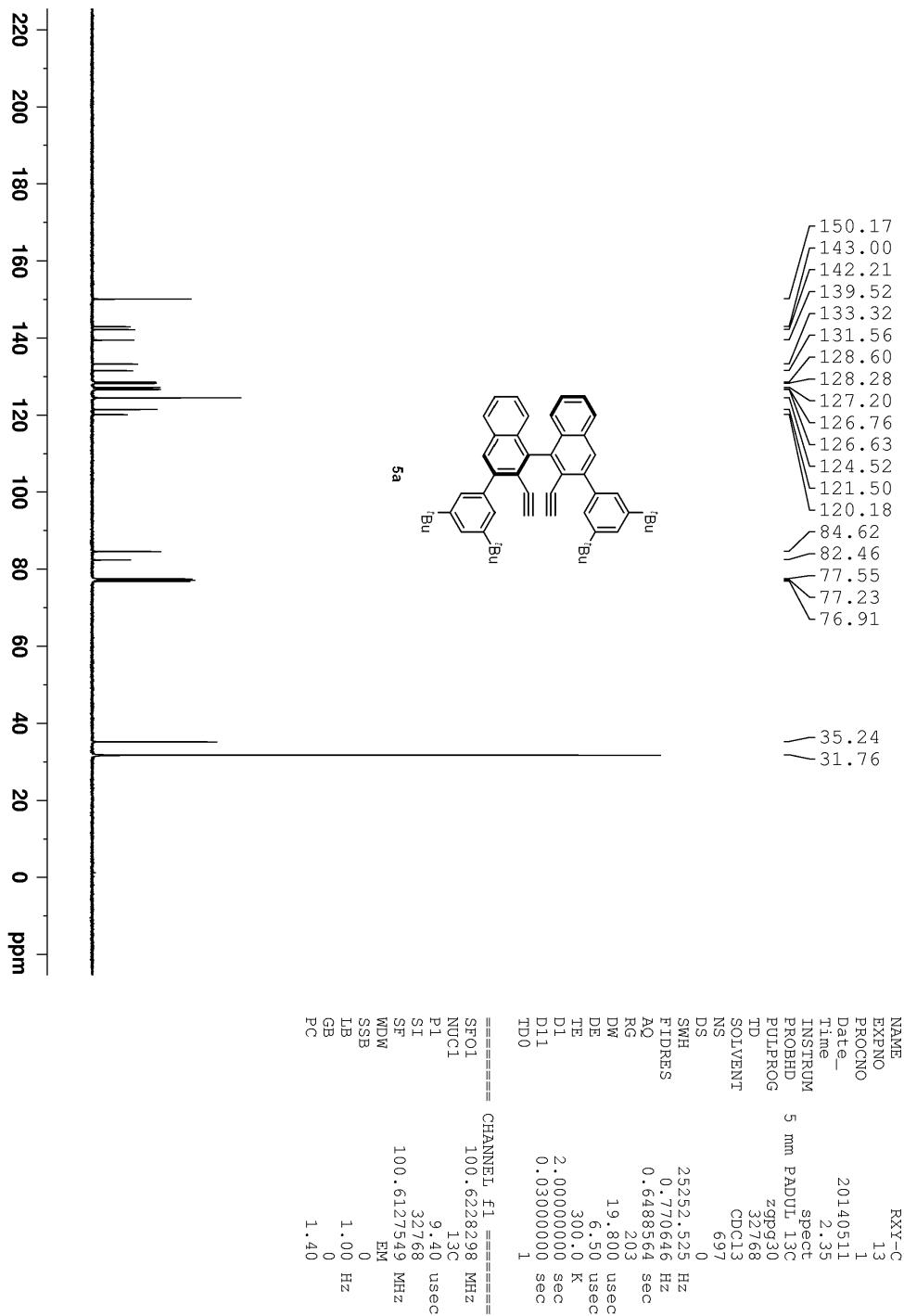
HPLC Conditions: Column: Chiralcel OD-H, Daicel Chemical Industries, Ltd., Eluent: Hexanes/IPA(97/3); Flow rate: 1.0 mL/min; Detection: UV 210 nm

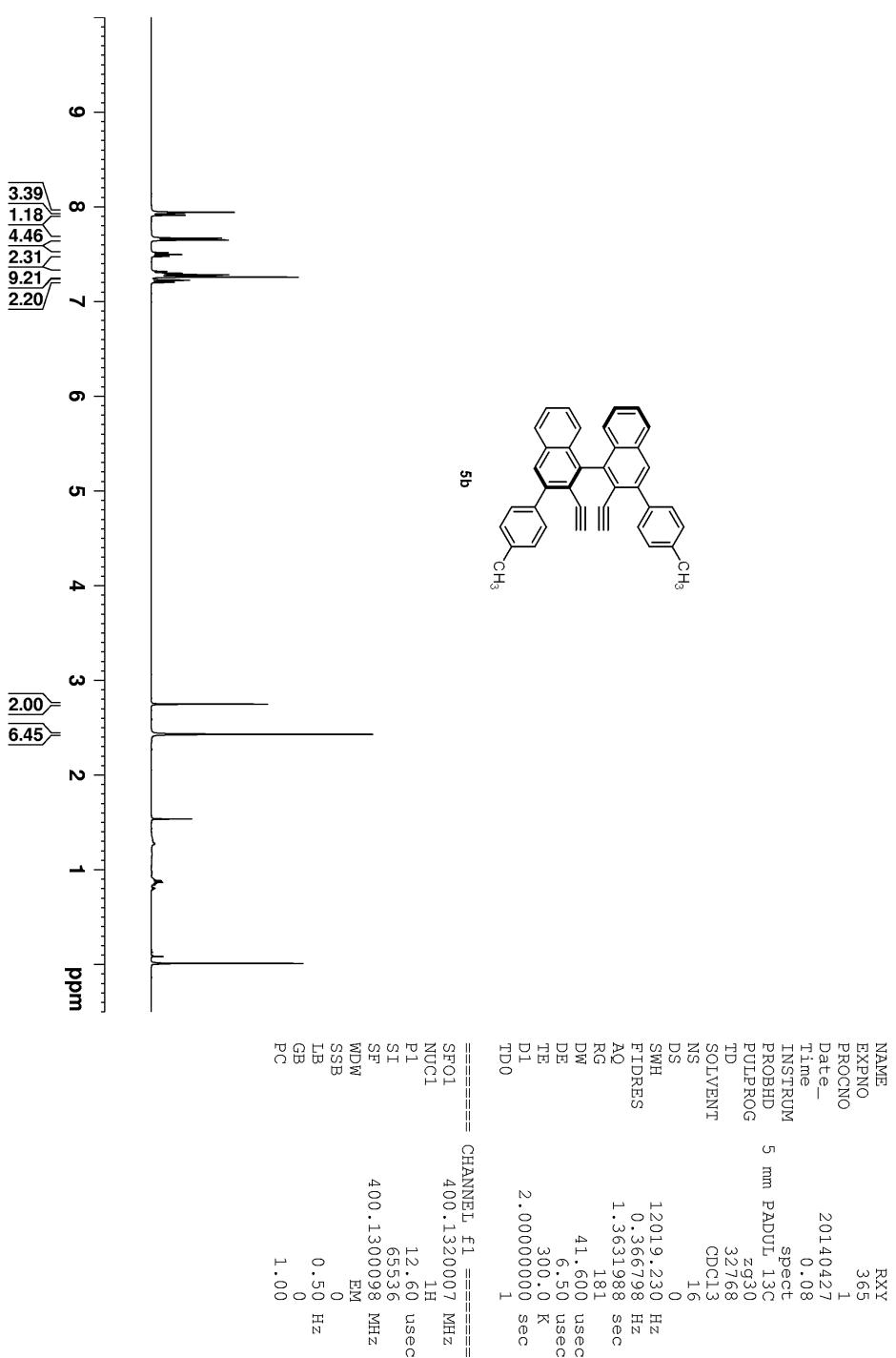
**Table 2, entry 22**

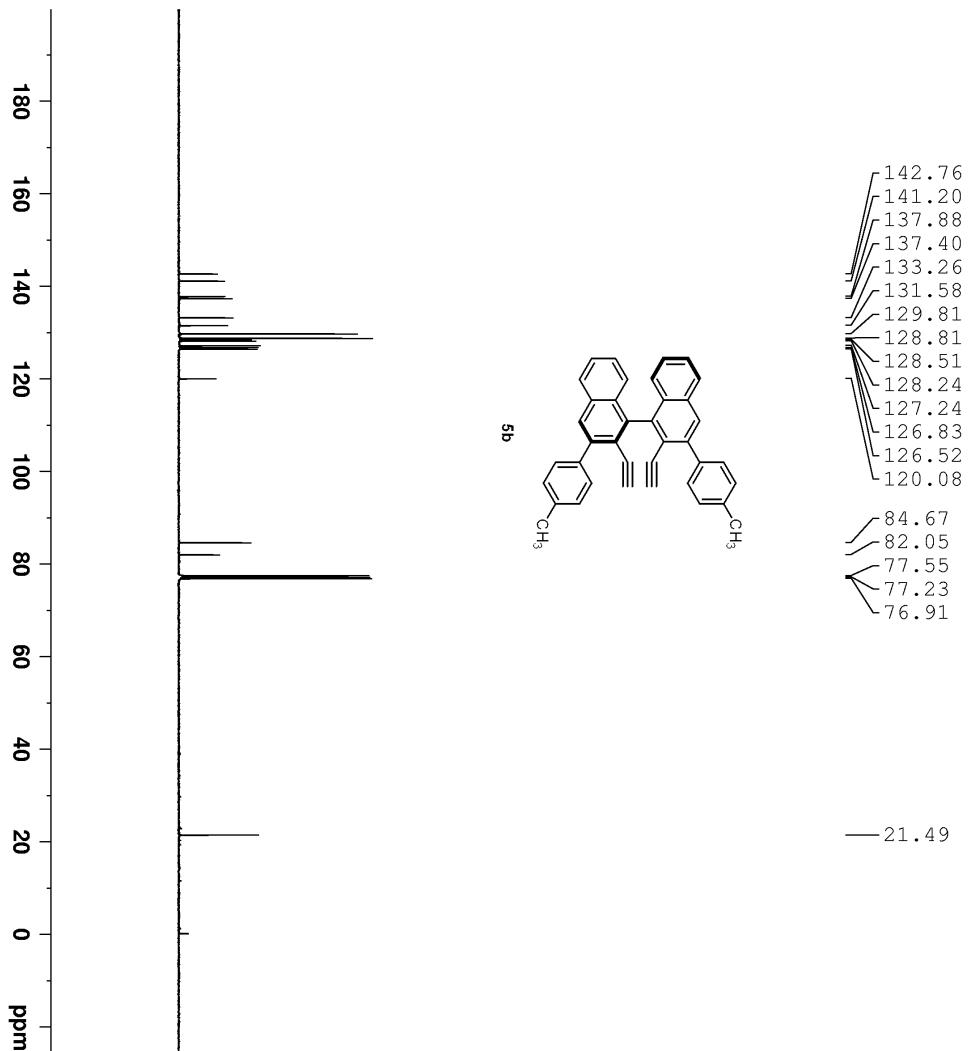
HPLC Conditions: Column: Chiralcel OB-H, Daicel Chemical Industries, Ltd., Eluent: Hexanes/IPA(99/1); Flow rate: 1.0 mL/min; Detection: UV 210 nm







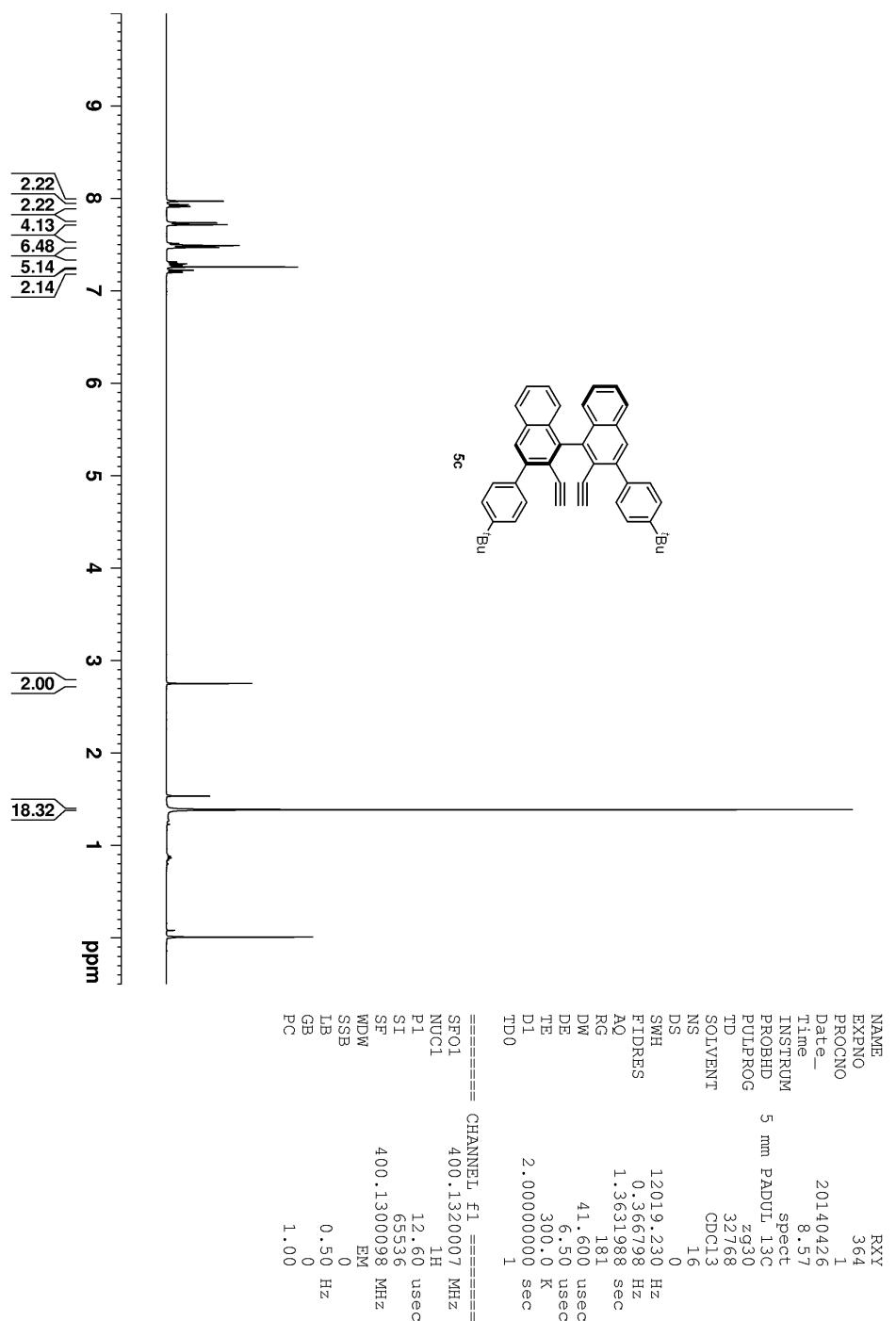


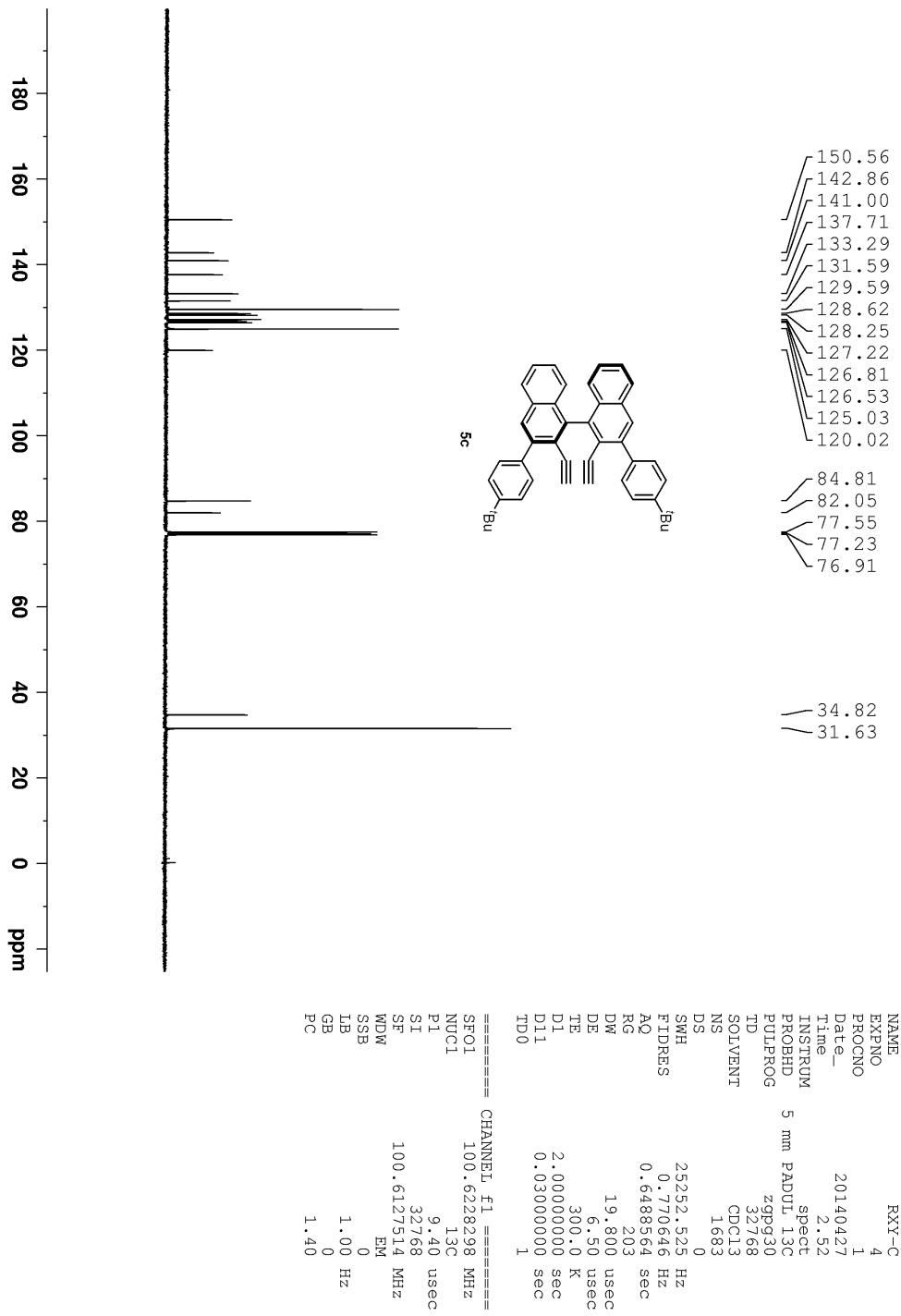


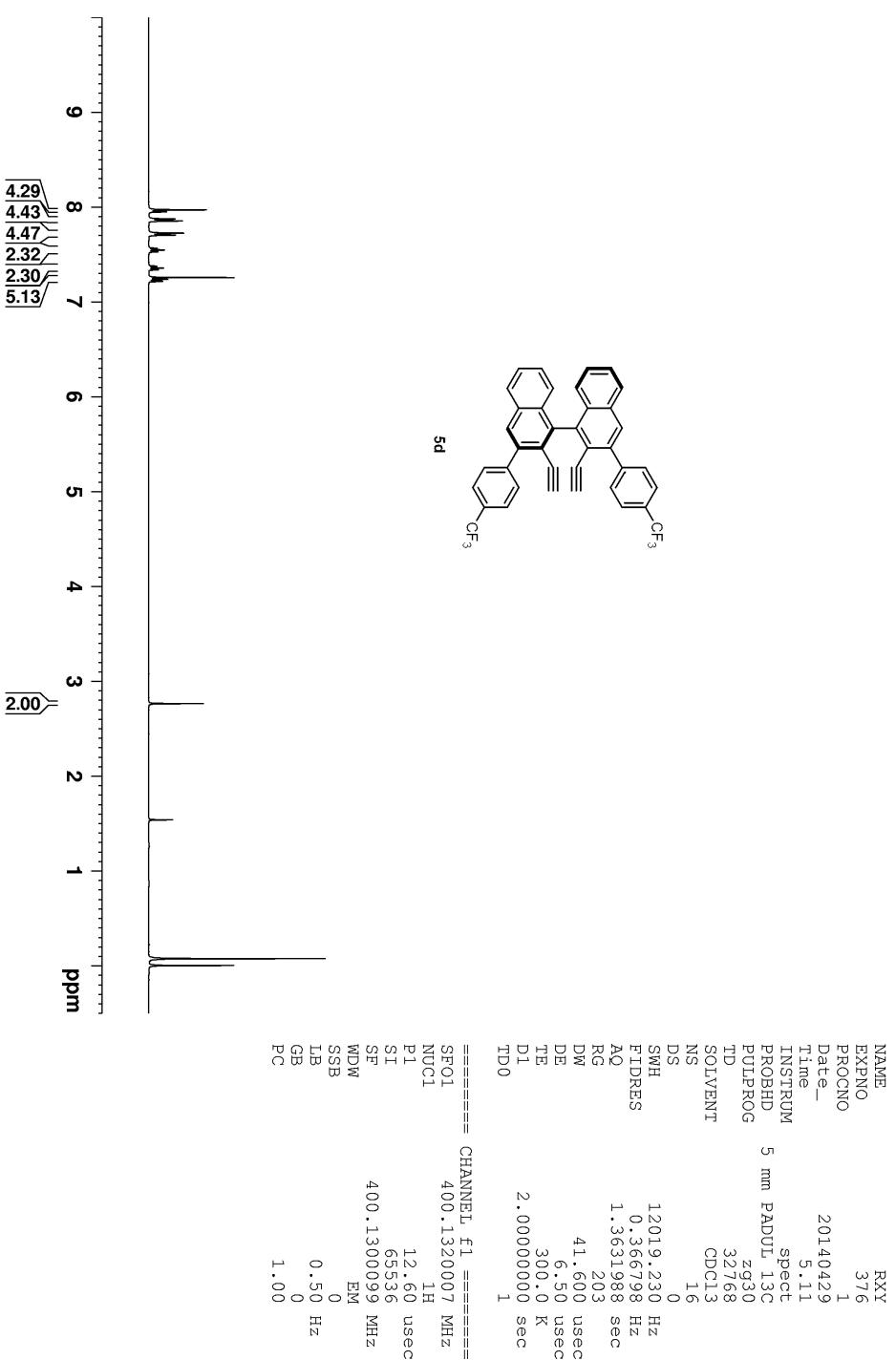
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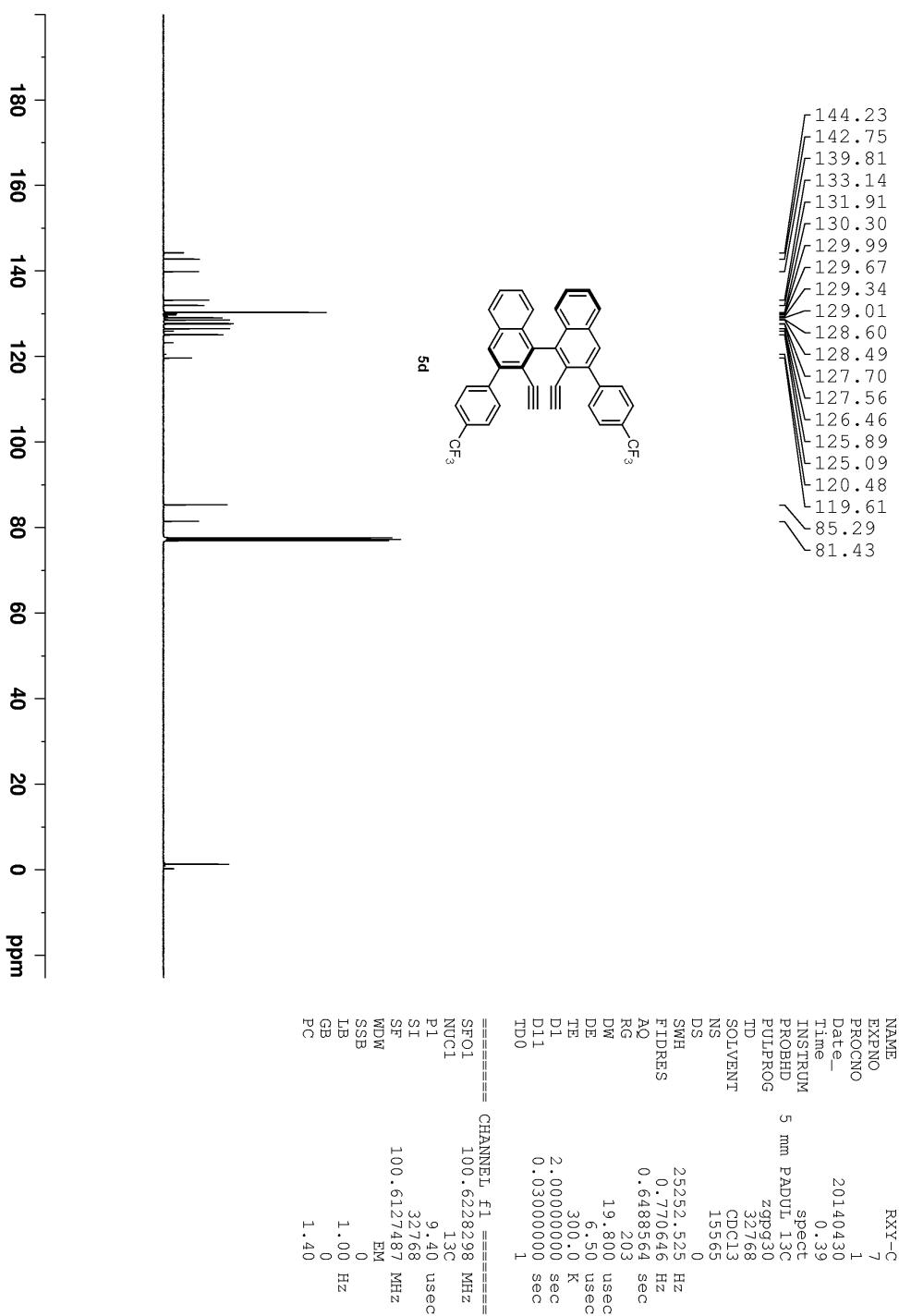
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NUC1 13C
P1 9.40 usec
SI 32768
SF 100.6127514 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

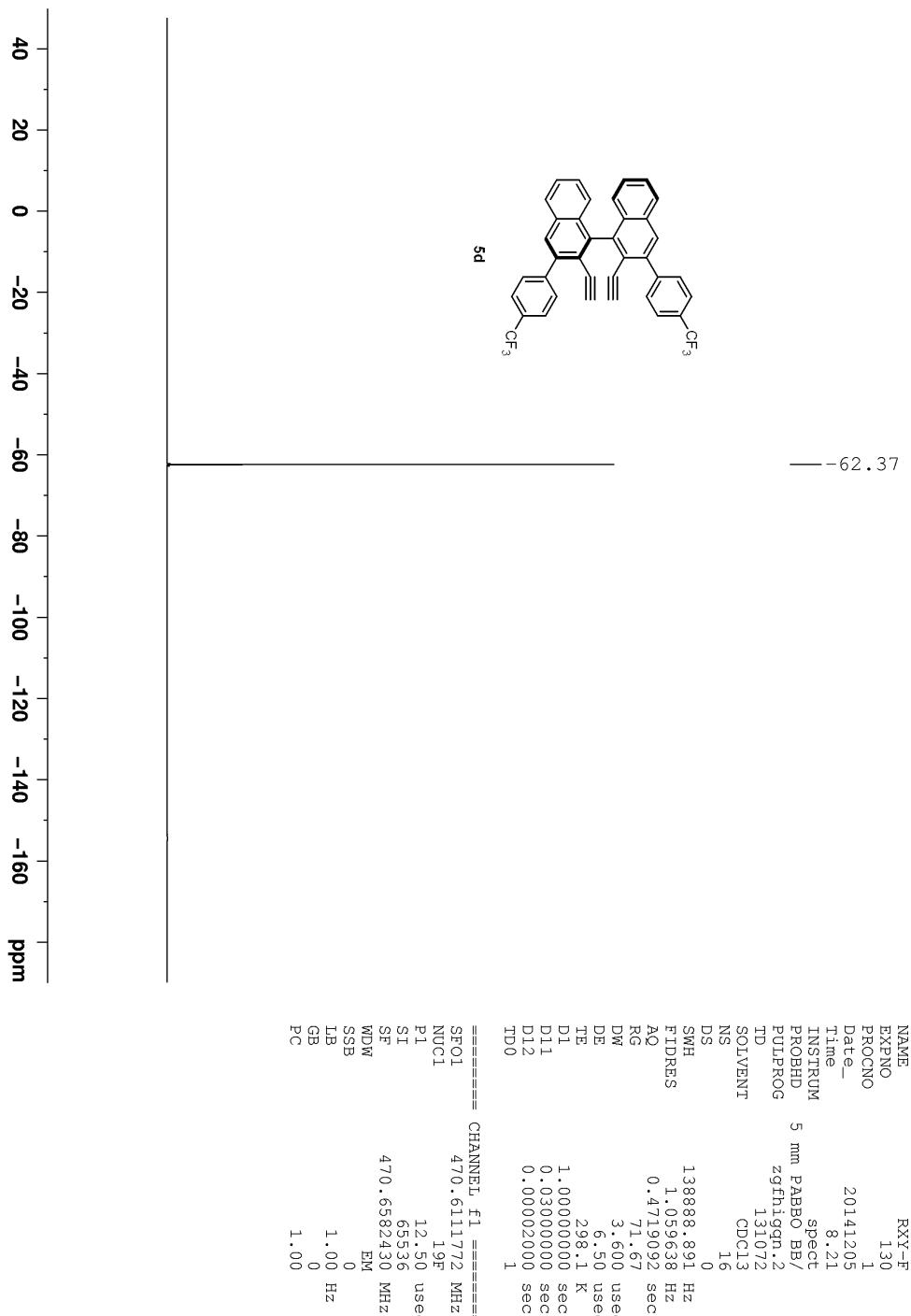
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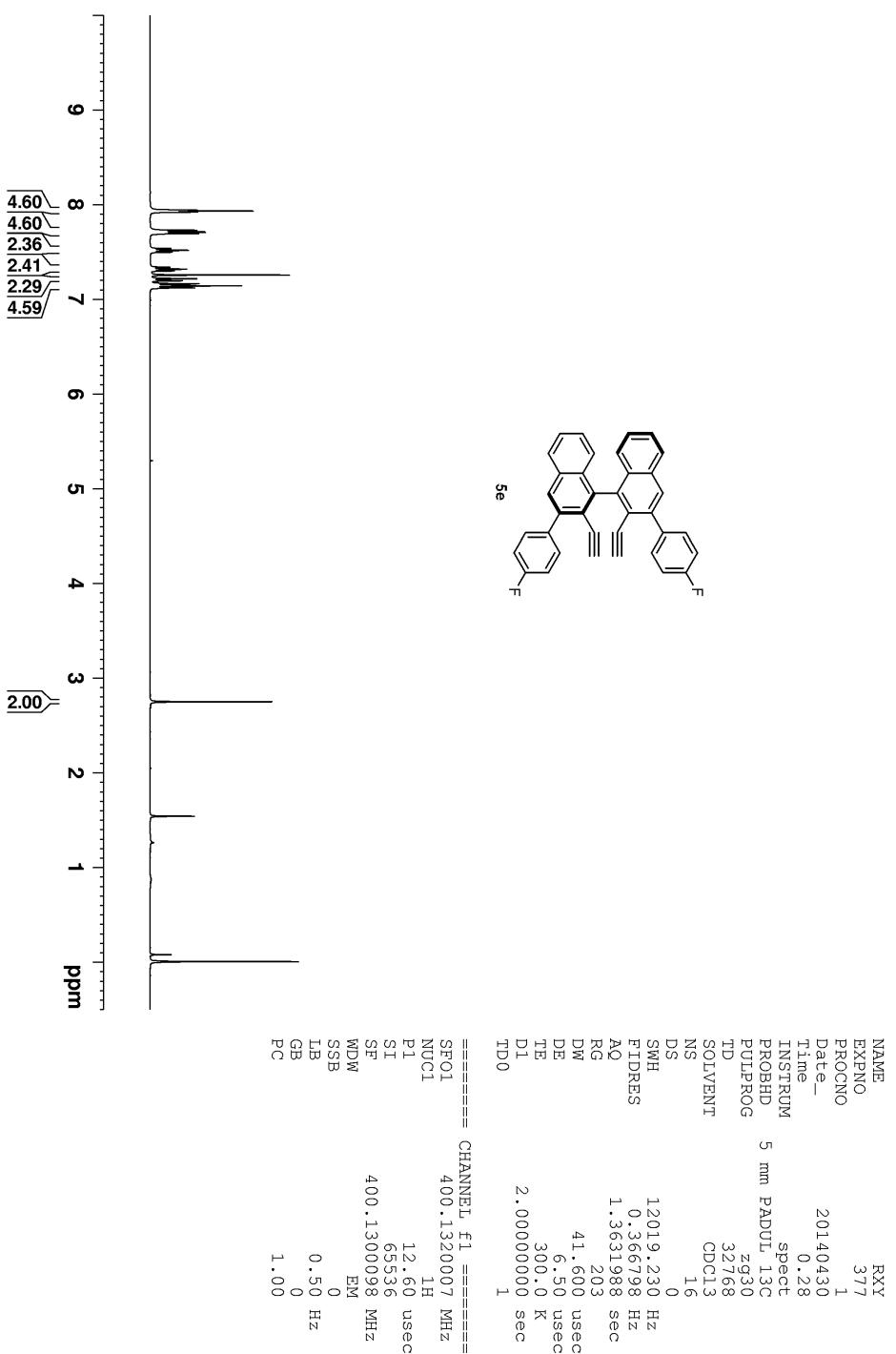


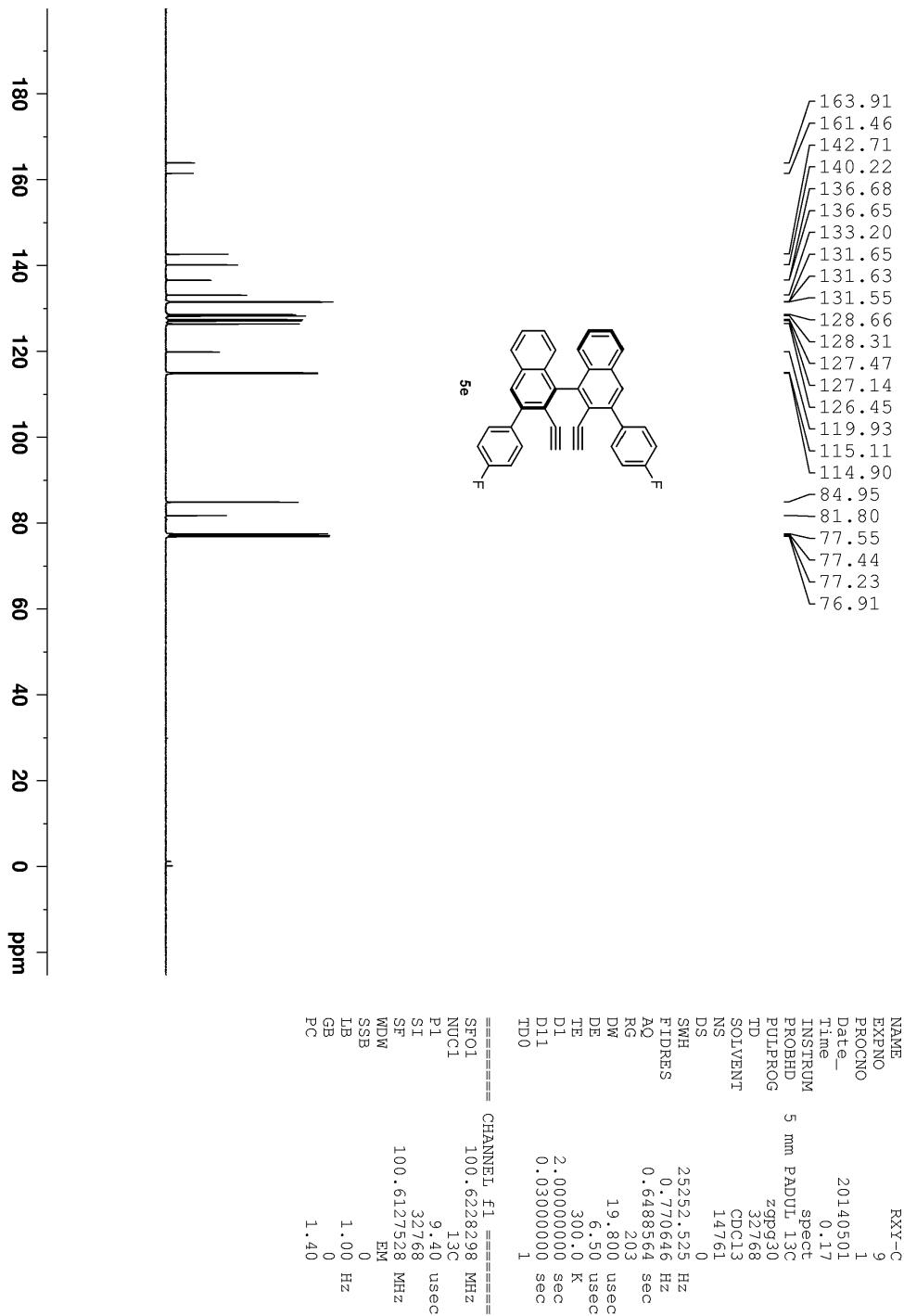


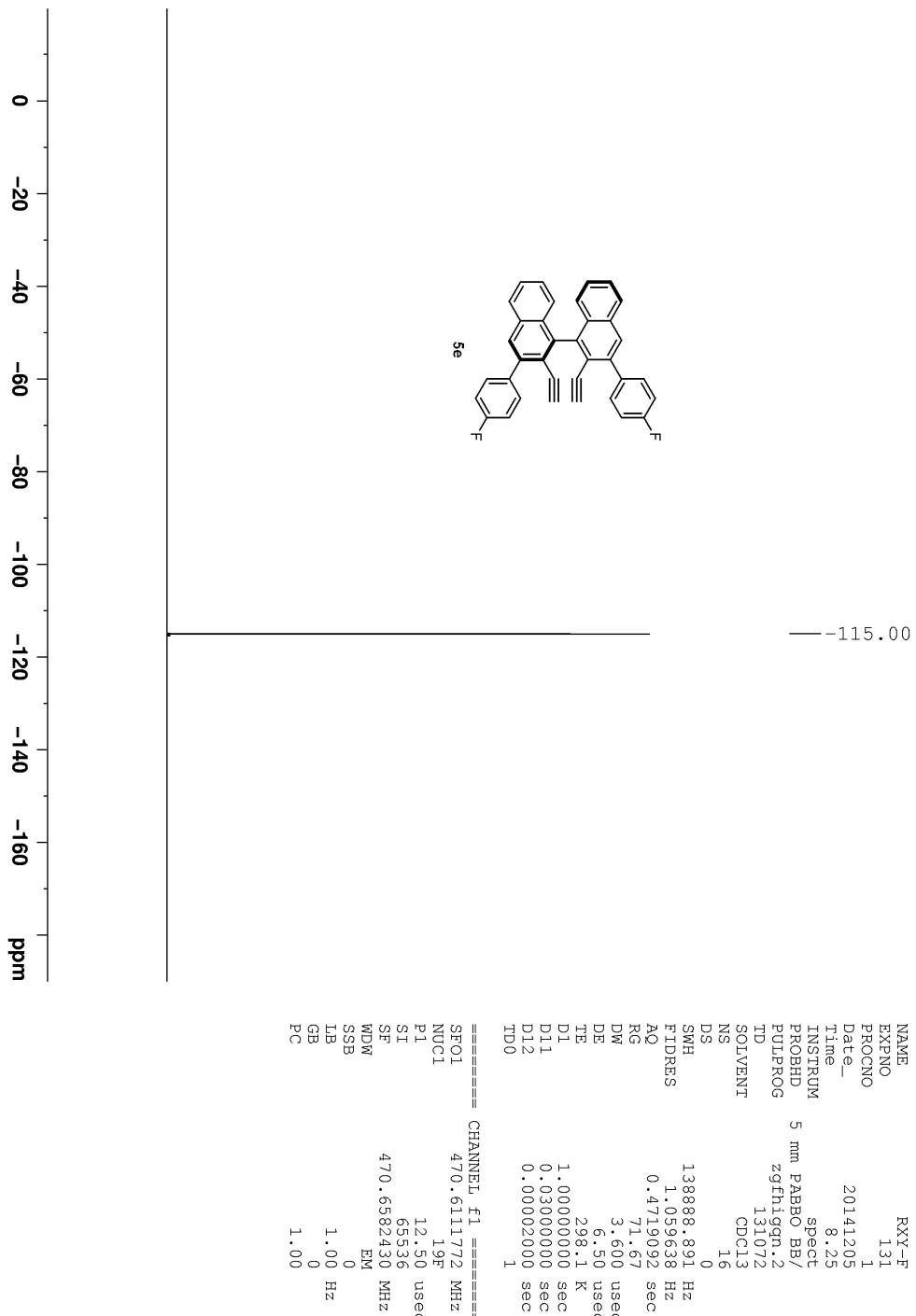


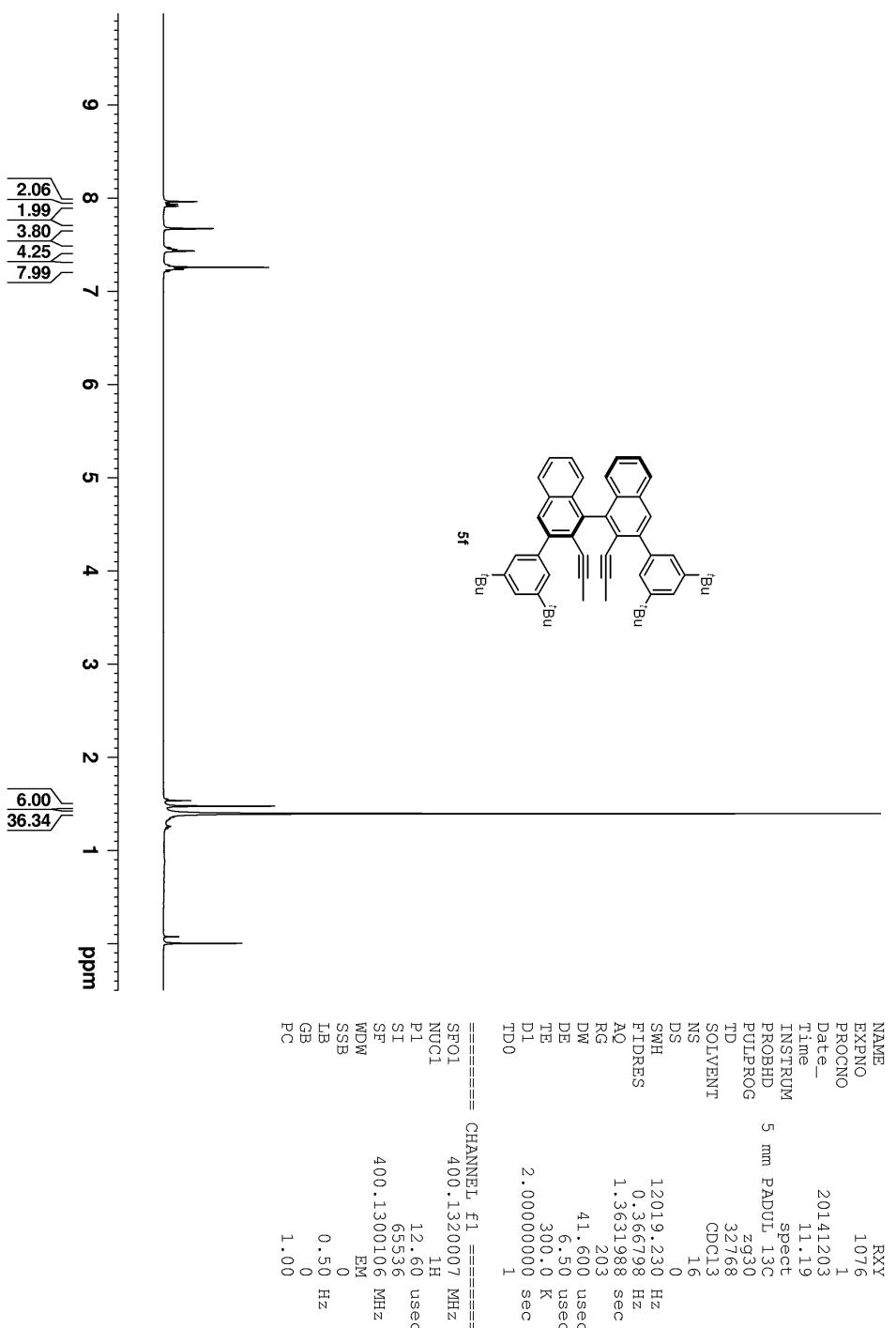


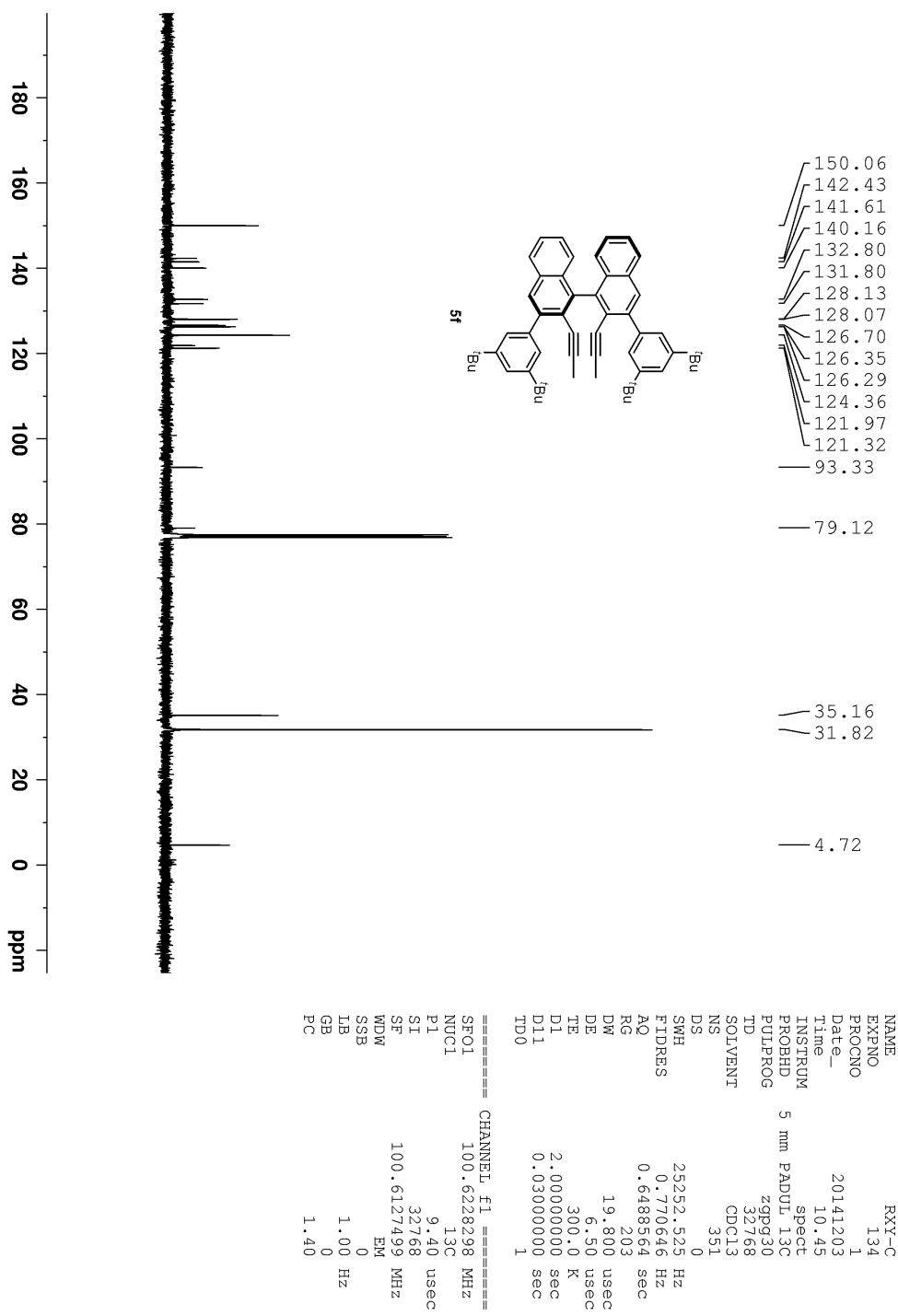




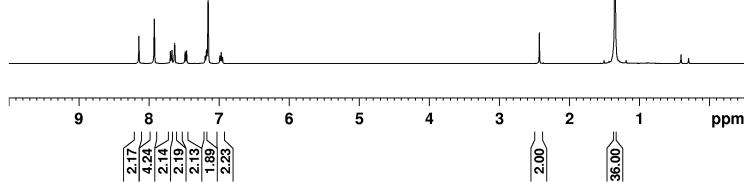
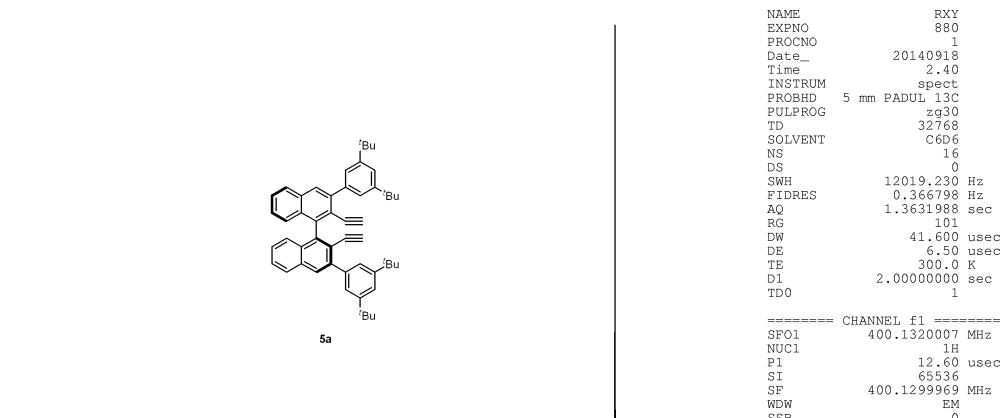






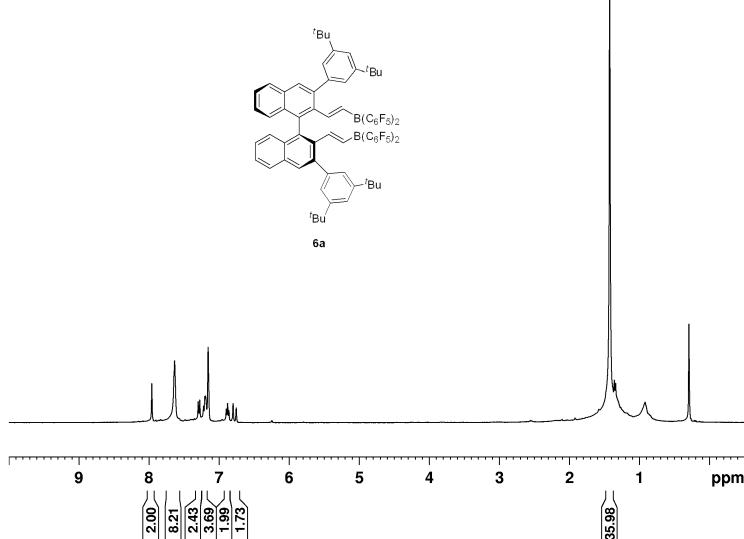


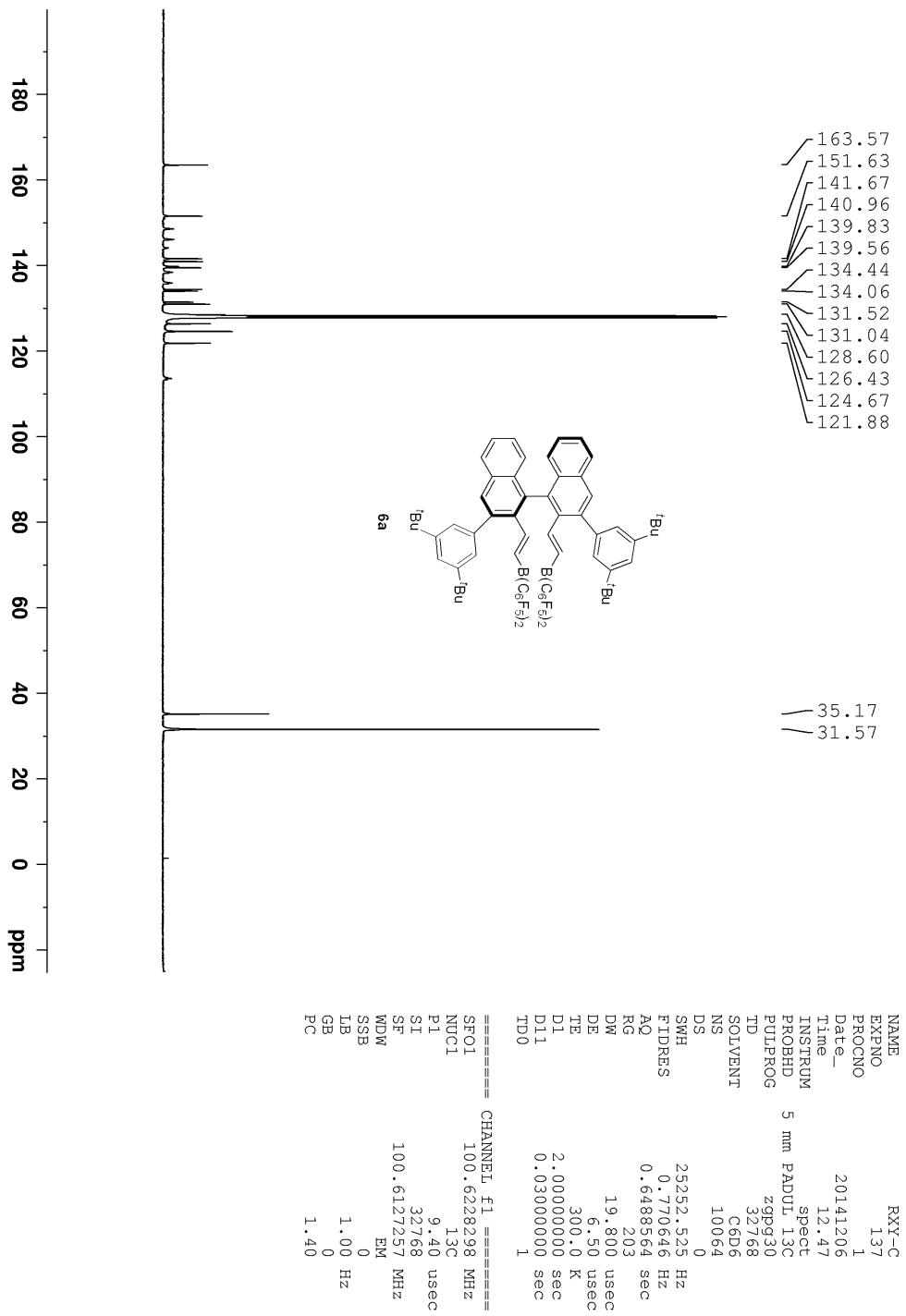
NMR study for chiral alkenylborane 6a

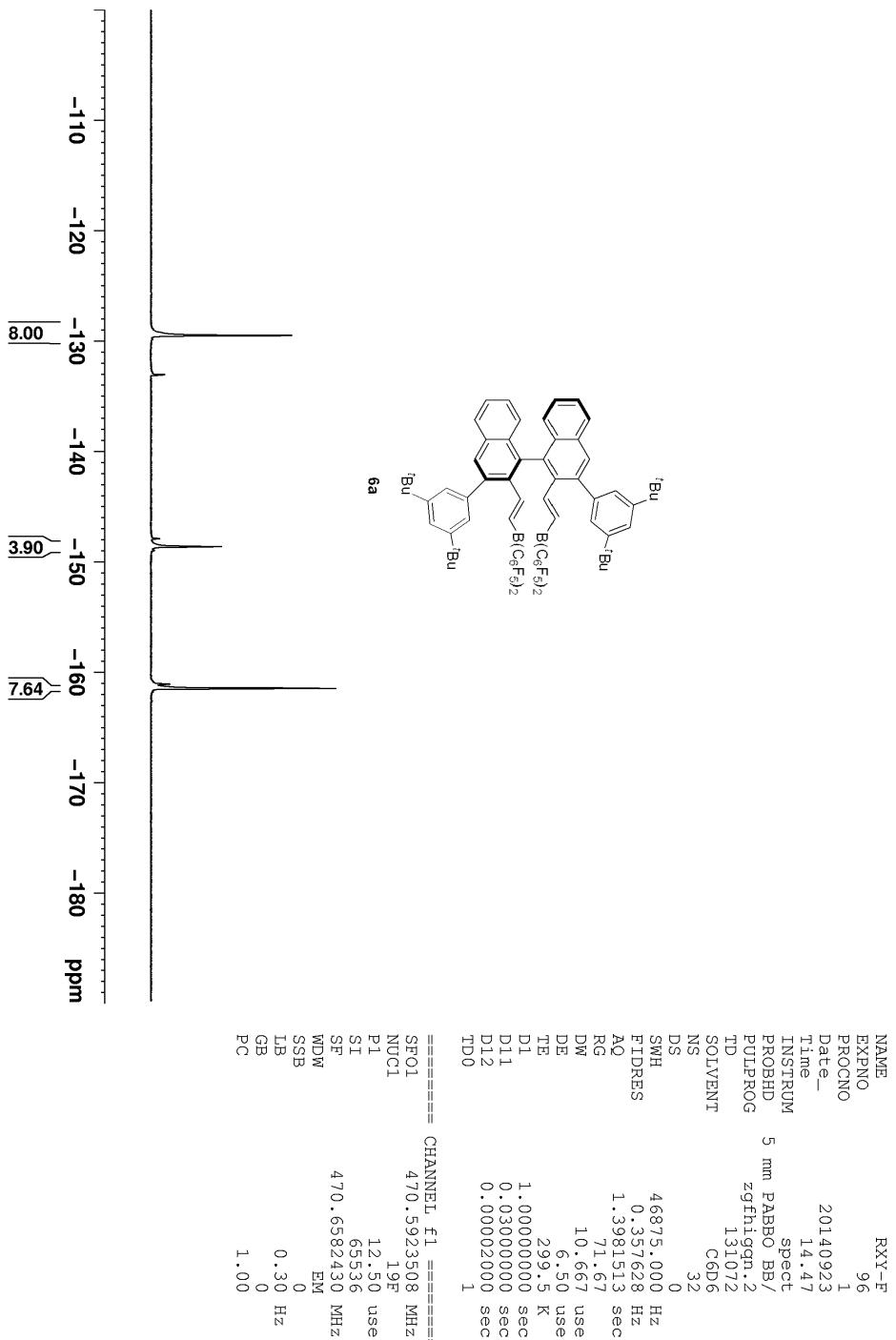


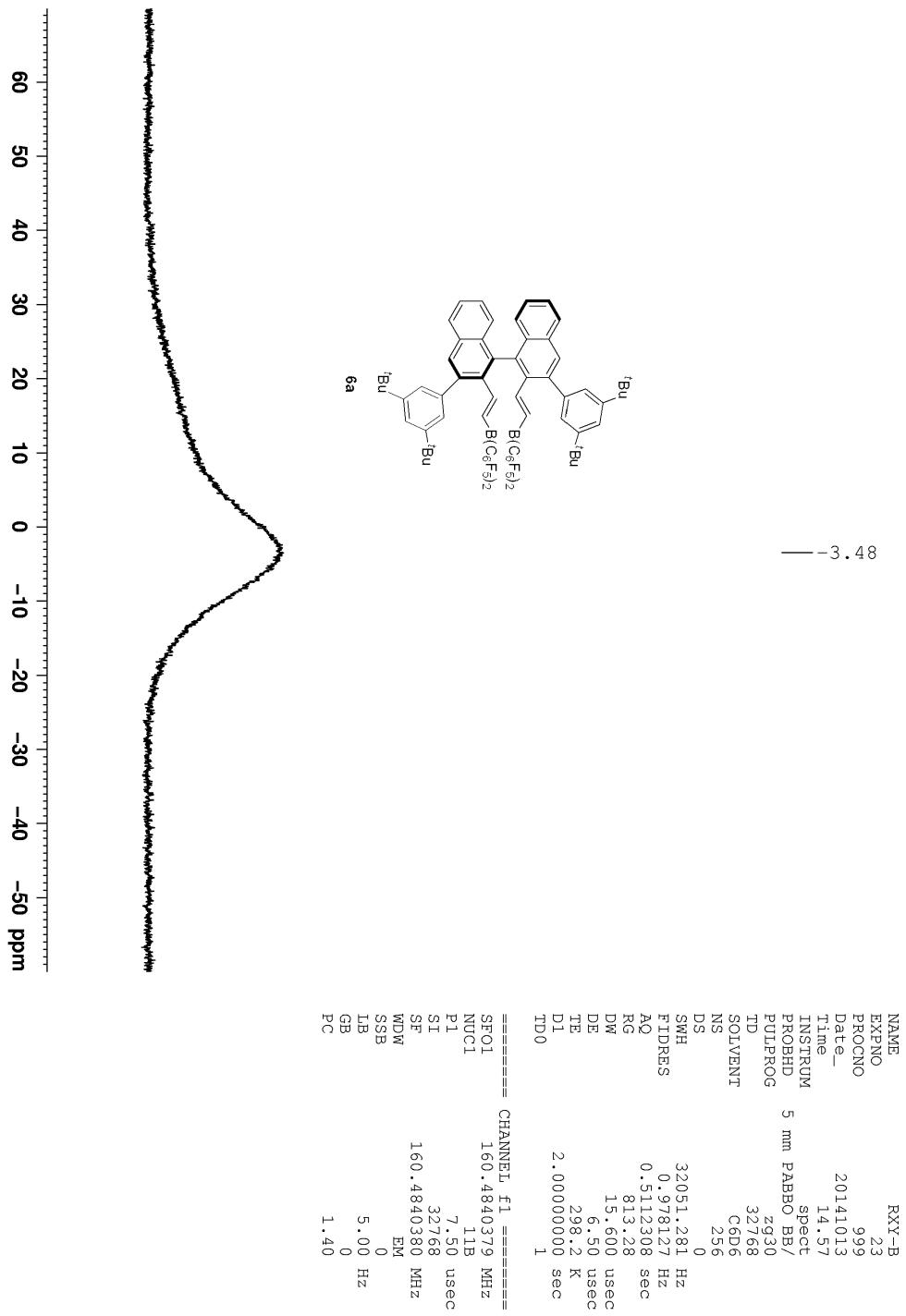
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 EXPNO 888
 PROCNO 1
 Date_ 20140918
 Time 9.40
 INSTRUM spect
 PROBHD 5 mm PADUL 13C
 PULPROG zg30
 TD 32768
 SOLVENT C6D6
 NS 16
 DS 0
 SWH 12019.230 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 203
 DW 41.600 usec
 DE 6.50 usec
 TE 300.0 K
 D1 2.0000000 sec
 TDO 1

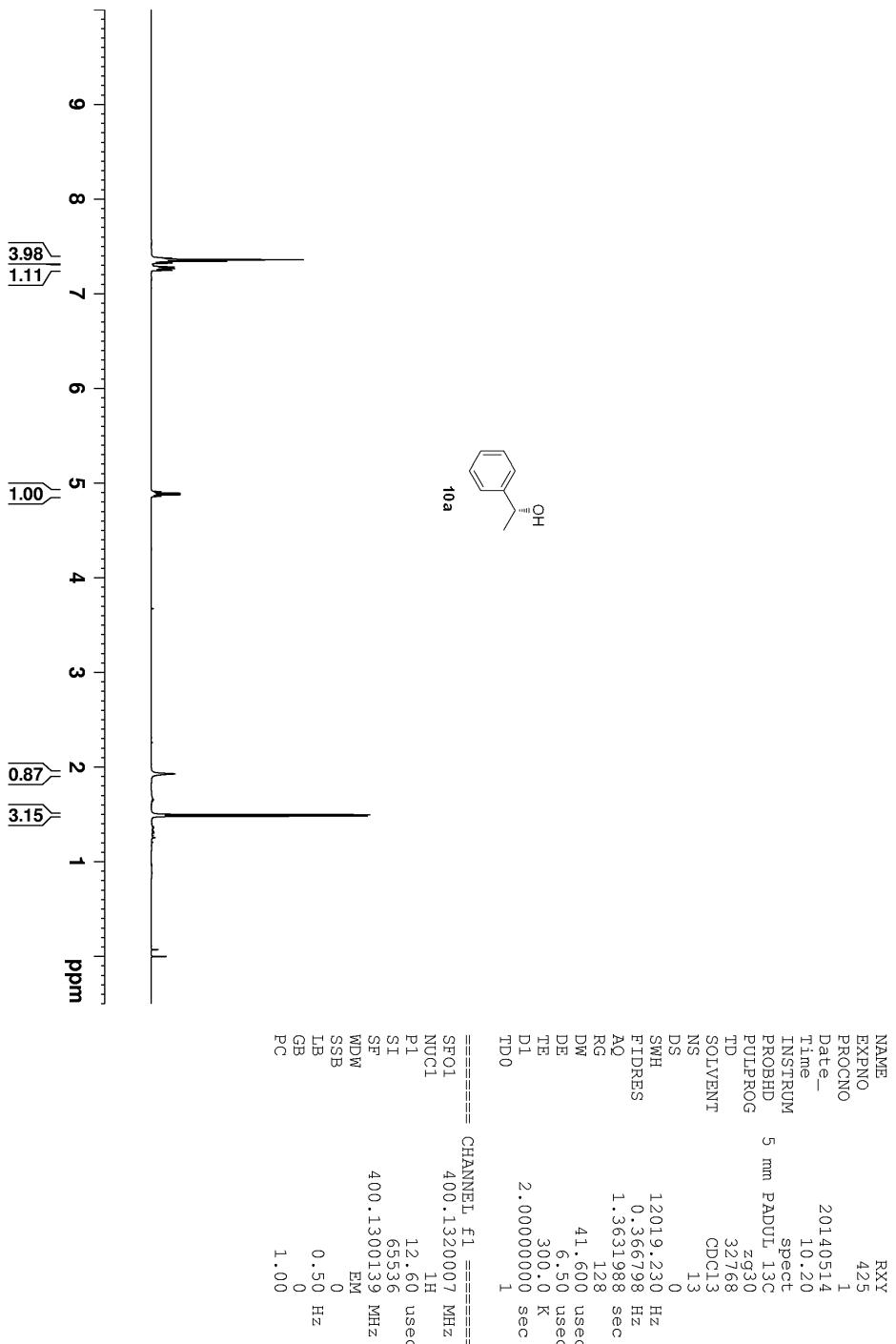
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 NUC1 1H
 P1 12.60 usec
 SI 65536
 SF 400.1299966 MHz
 WDW EM
 SSB 0
 LB 0.50 Hz
 GB 0
 PC 1.00

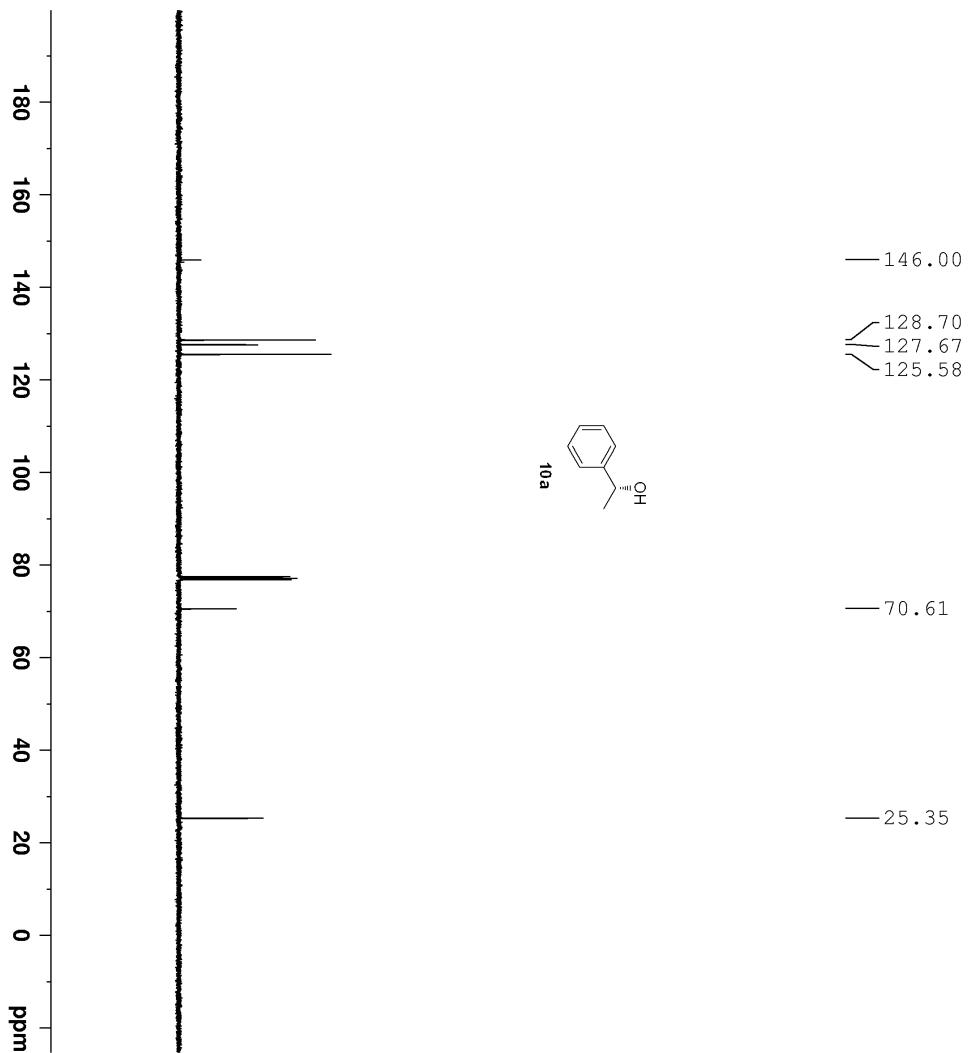










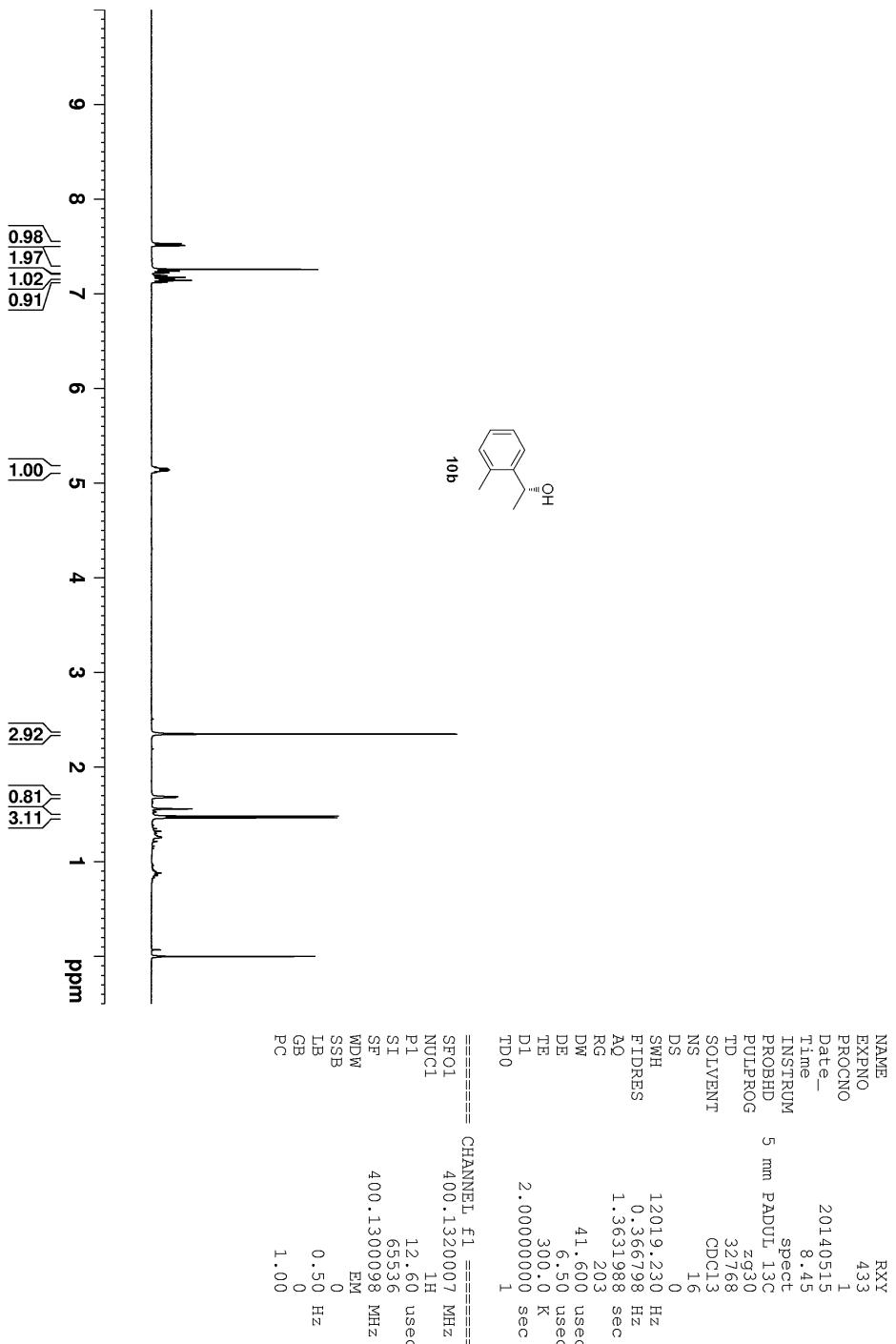


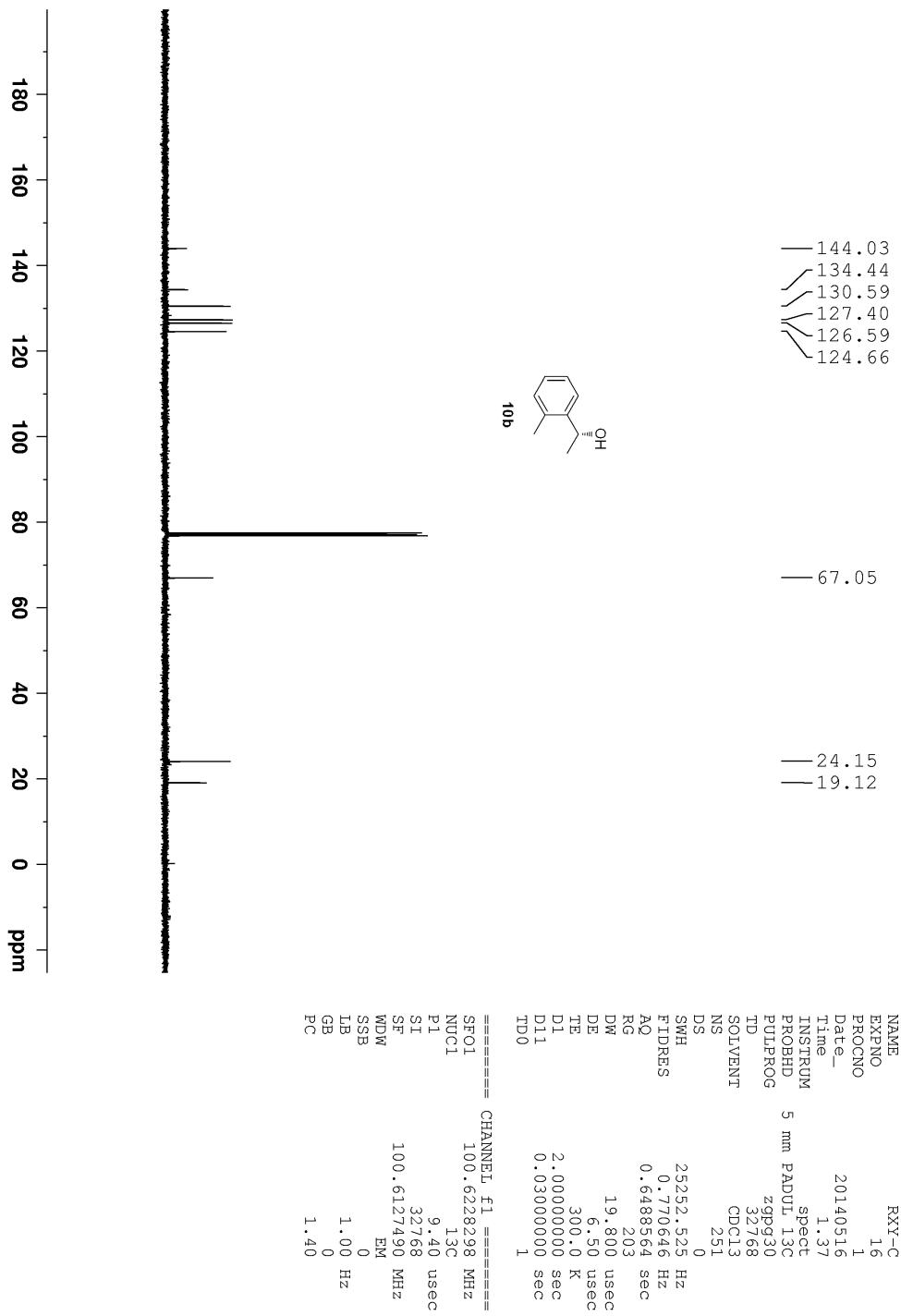
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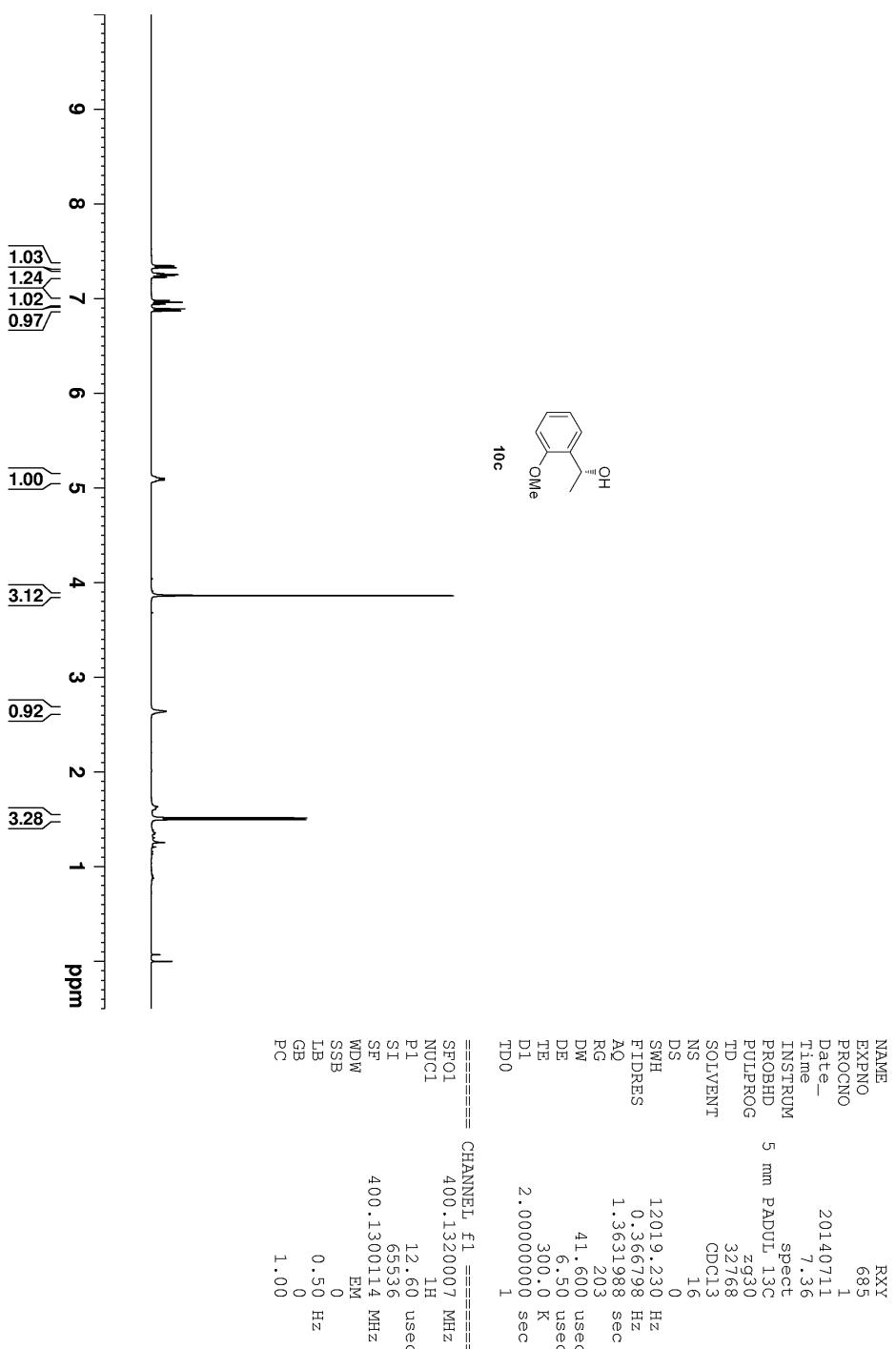
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EXPNO    14
PROCNO   1
Date_    20140514
Time    10:30
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD      32768
SOLVENT CDCl3
NS       123
DS        0
SWH     25252.525 Hz
FIDRES  0.770646 Hz
AQ      0.6488554 sec
RG      203
DW      19.800 usec
DE      6.50 usec
TE      300.0 K
D1      2.0000000 sec
D11     0.03000000 sec
TD0      1

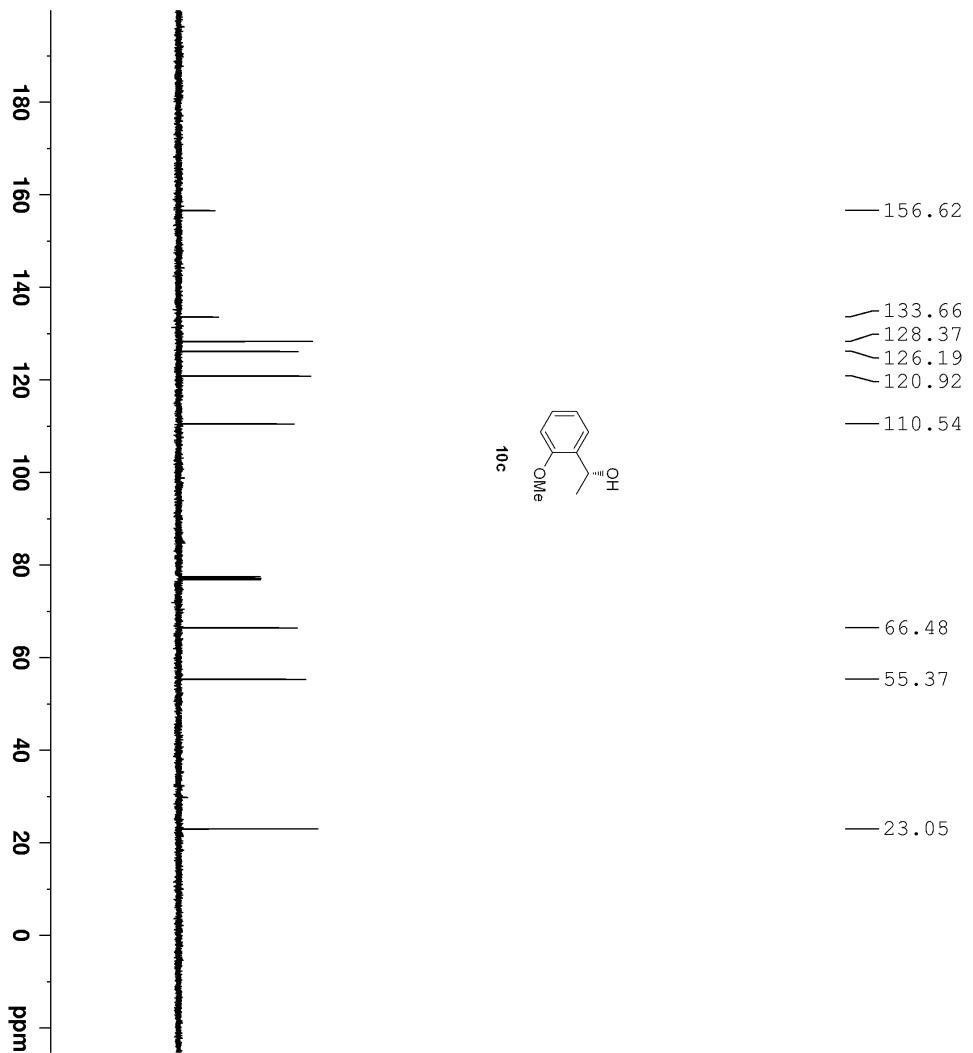
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CHANNEL f1 =====
SF01    100.622828 MHz
NUC1    13C
P1      9.40 usec
SI      32768
SF      100.6127510 MHz
WDW
SSB
LB      1.00 Hz
GB      0
PC      1.40

```







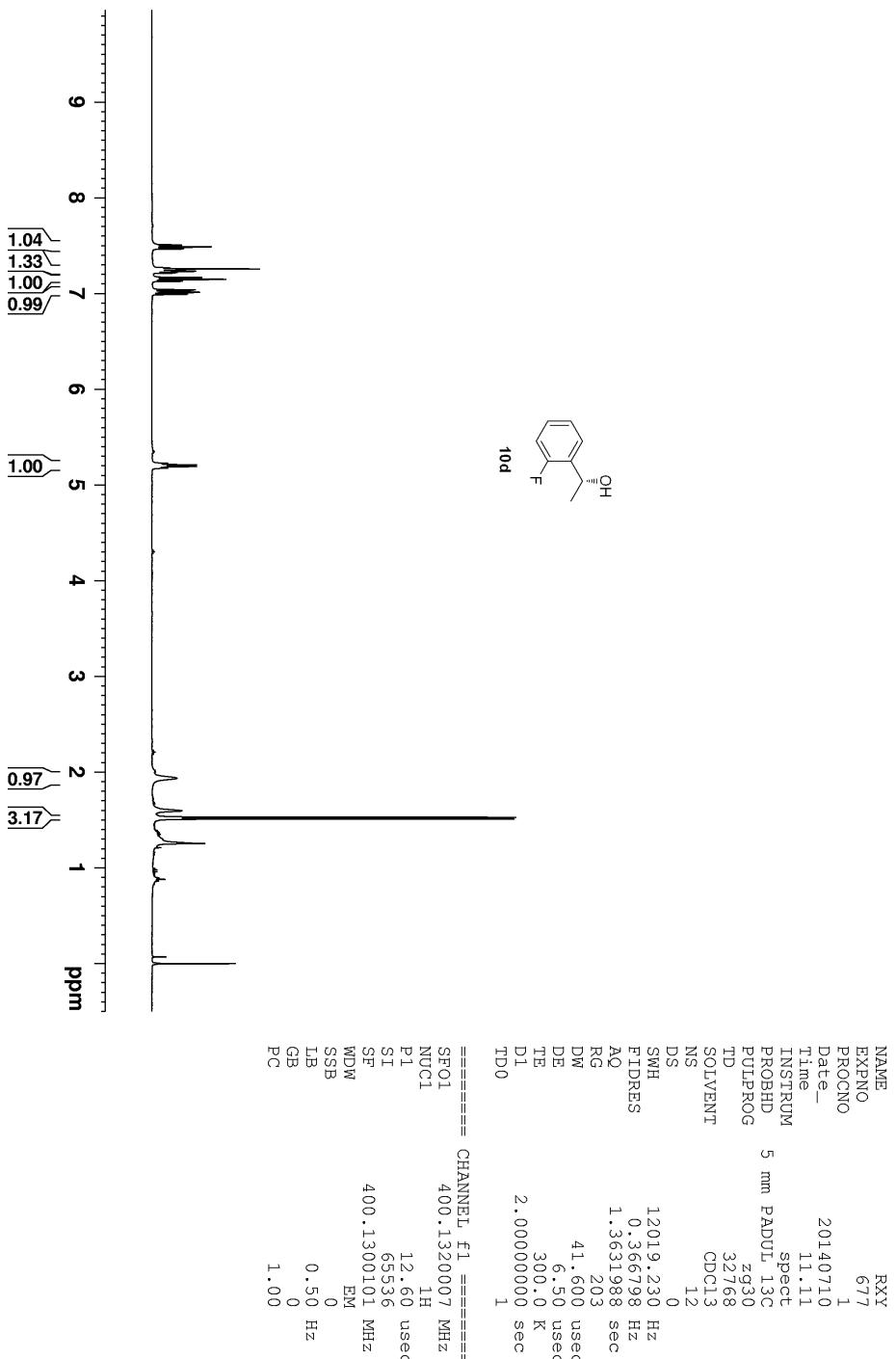


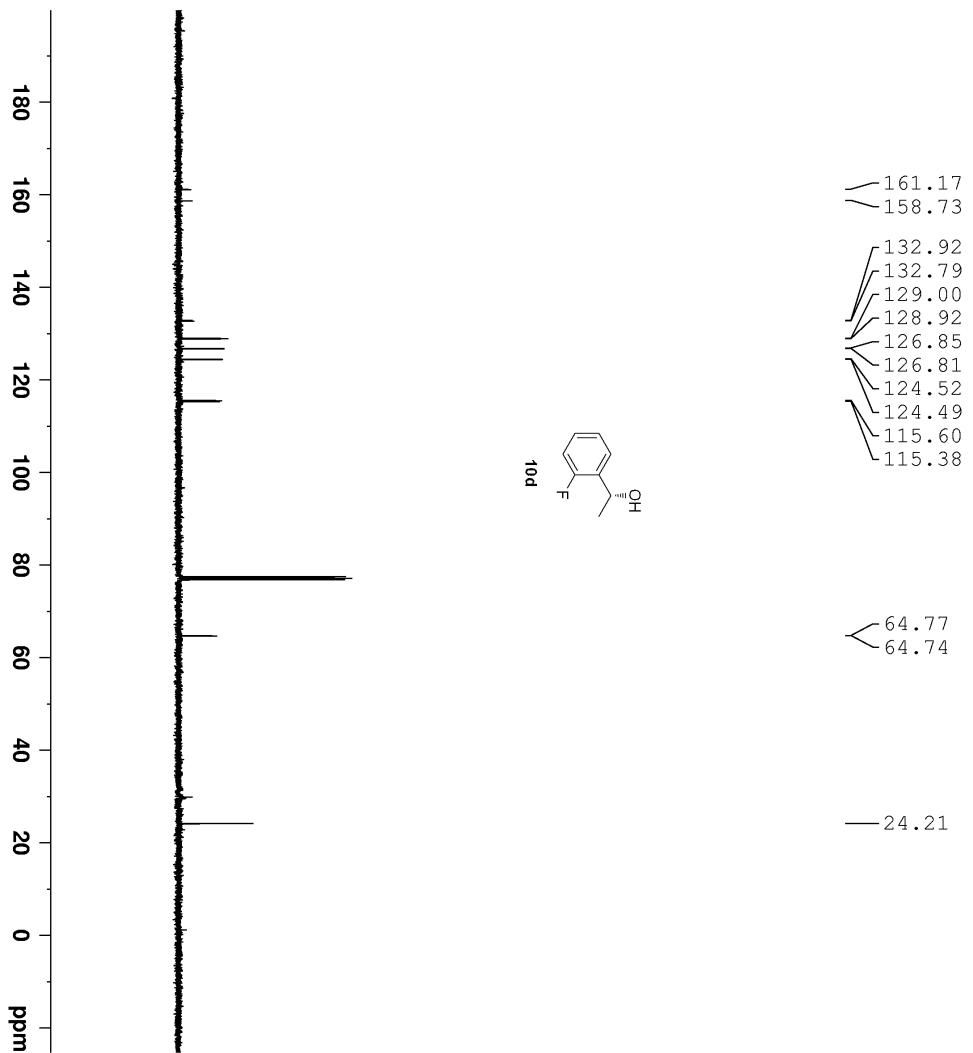
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EXPNO    66
PROCNO   1
Date_    20140711
Time     7.47
INSTRUM spect
PROBHD  5 mm PADUL 13C
PULPROG zgpg30
TD        32768
SOLVENT  CDCl3
NS       13
DS        0
SWH      25252.525 Hz
FIDRES   0.6488554 sec
AQ        203
RG        19.800 usec
DW        6.500 usec
DE        300.0 K
TE        2.0000000 sec
D1       0.03000000 sec
T1       1
TD0      1

===== CHANNEL f1 =====
SF01    100.622828 MHz
NUC1    13C
P1      9.40 usec
SI       32768
SF      100.6127579 MHz
WDW
SSB
LB      1.00 Hz
GB      0
PC      1.40

```

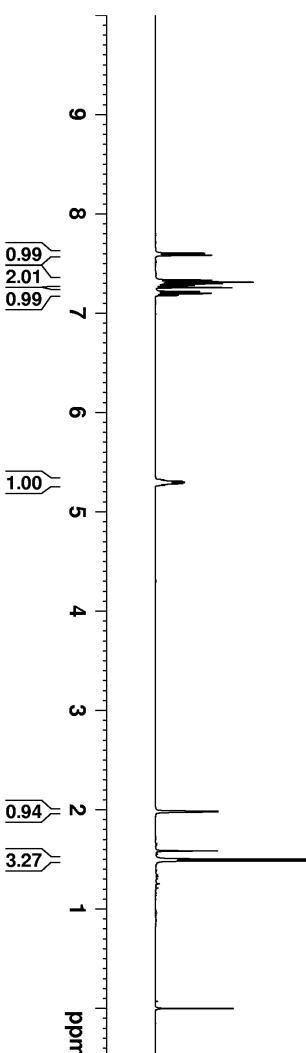




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=====
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PROCNO   1
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Time_    11:33
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PULPROG zgpg30
TD      32768
SOLVENT CDCl3
NS      72
DS      0
SWH    25252.525 Hz
FIDRES 0.770646 Hz
AQ     0.6488554 sec
RG      203
DW     19.800 usec
DE     6.500 usec
TE     300.0 K
D1     2.0000000 sec
D11    0.03000000 sec
TD0     1
===== CHANNEL f1 =====
SF01    100.622828 MHz
NUC1    13C
P1      9.40 usec
SI      32768
SF      100.6127436 MHz
WDW
SSB
LB     1.00 Hz
GB     0
PC     1.40

```

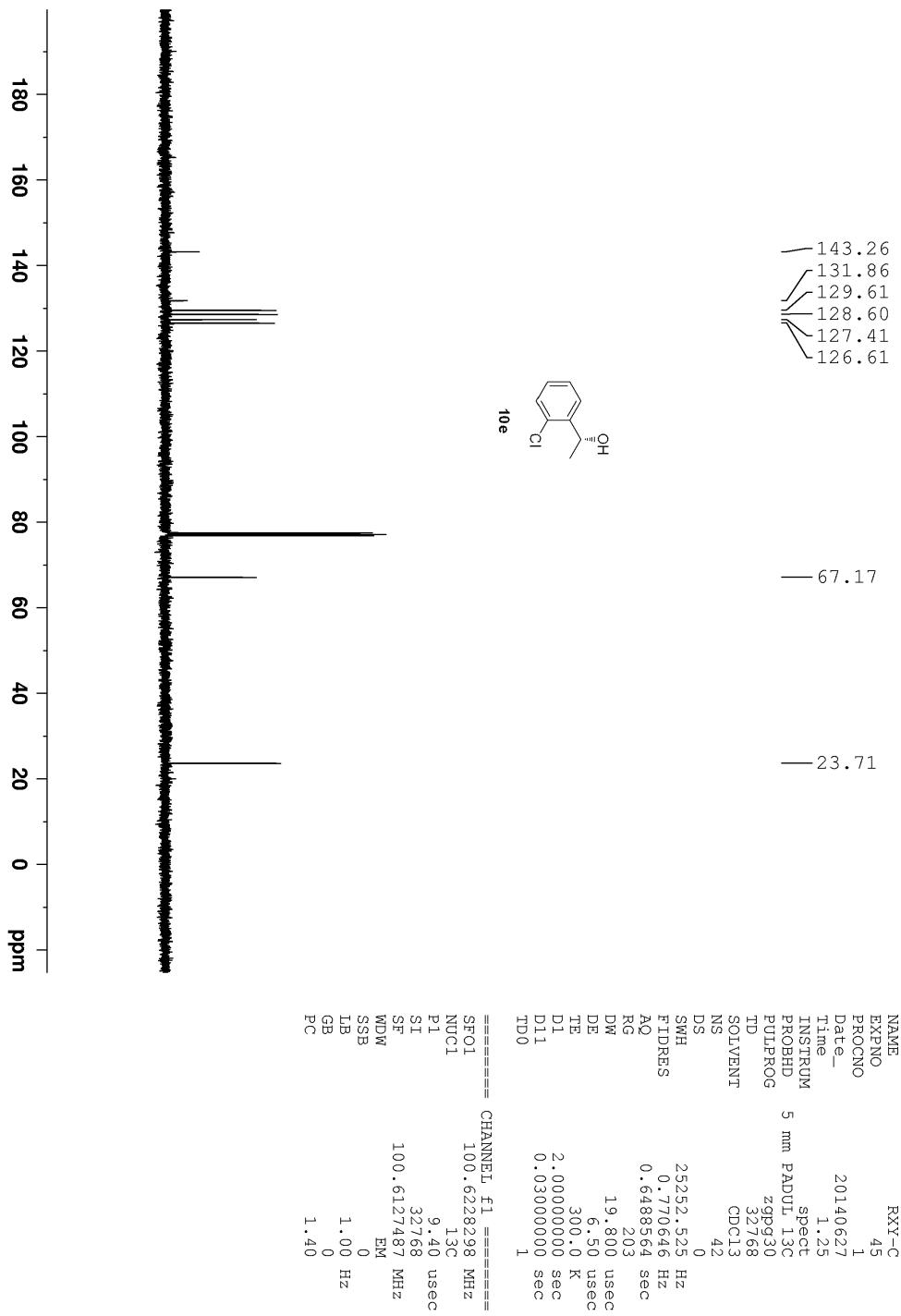


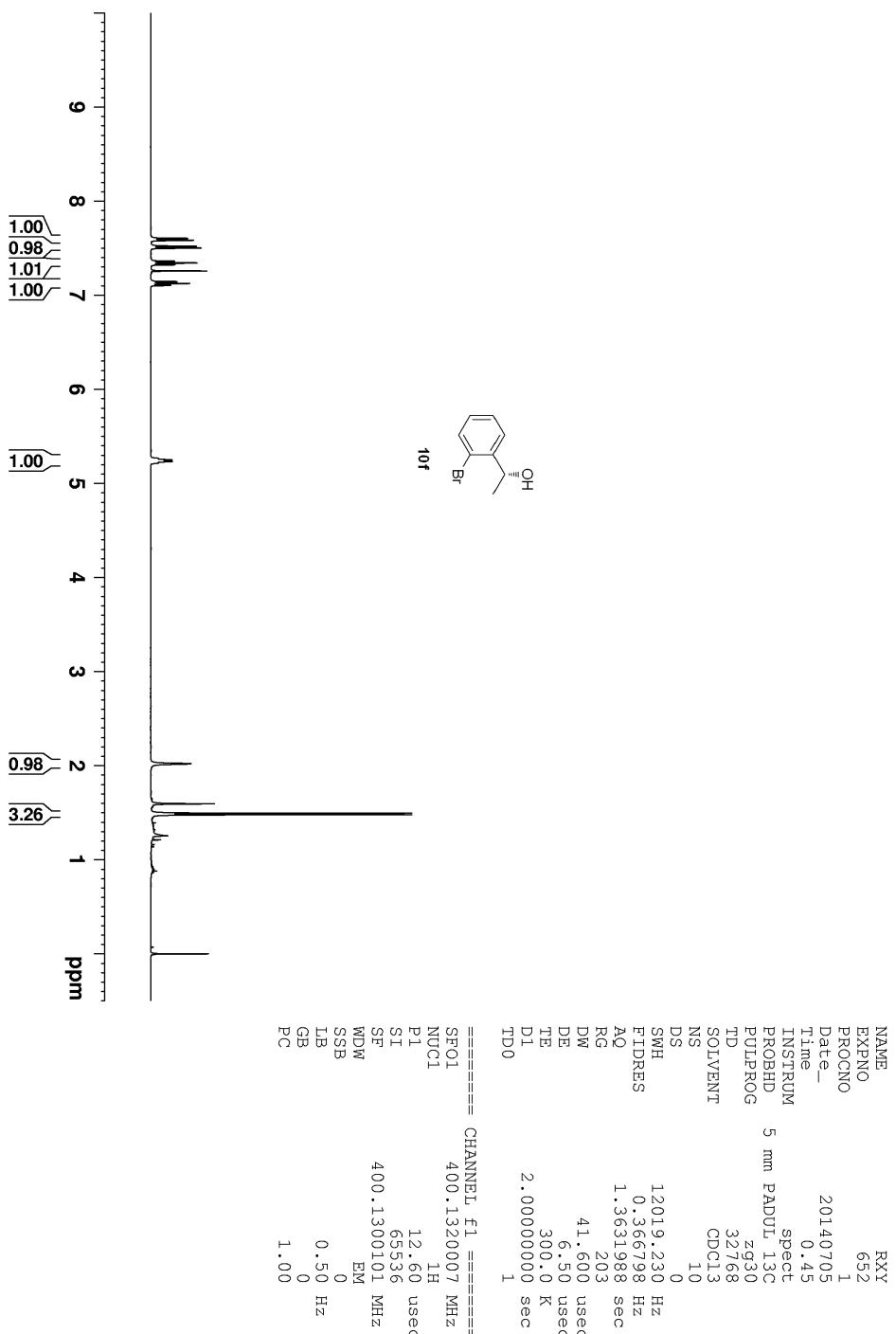
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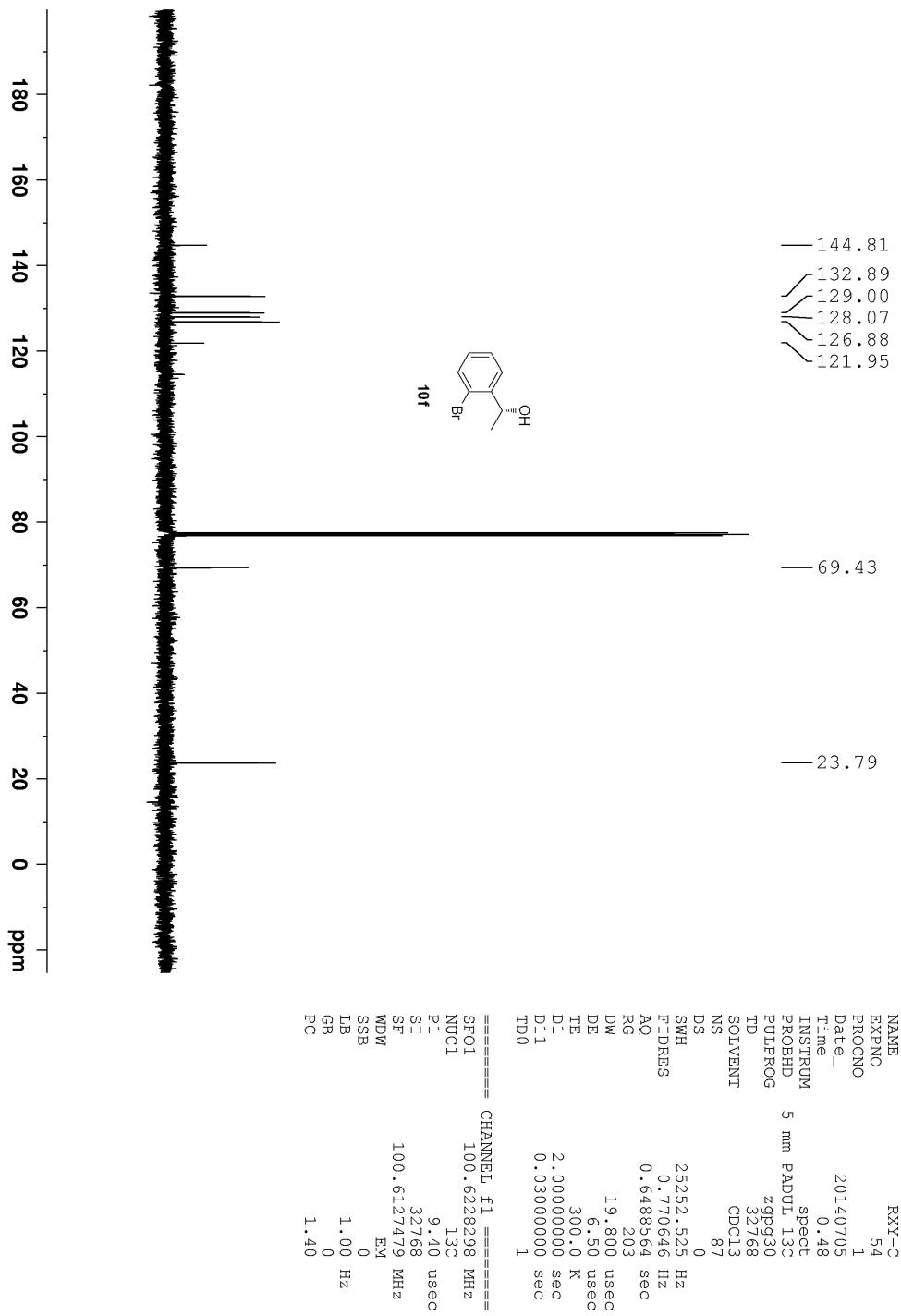
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EXPNO        450
PROCNO       1
Date_        20140519
Time         1.58
INSTRUM     spect
PROBHD      5 mm PADUL 13C
PULPROG    zg30
TD          32768
SOLVENT      CDCl3
NS           16
DS            0
SWH         12019.230 Hz
FIDRES     0.366798 Hz
AQ          1.3631988 sec
RG           203
DW           41.600 usec
DE           6.500 usec
TE           300.0 K
T1           2.0000000 sec
D1            1
TDO

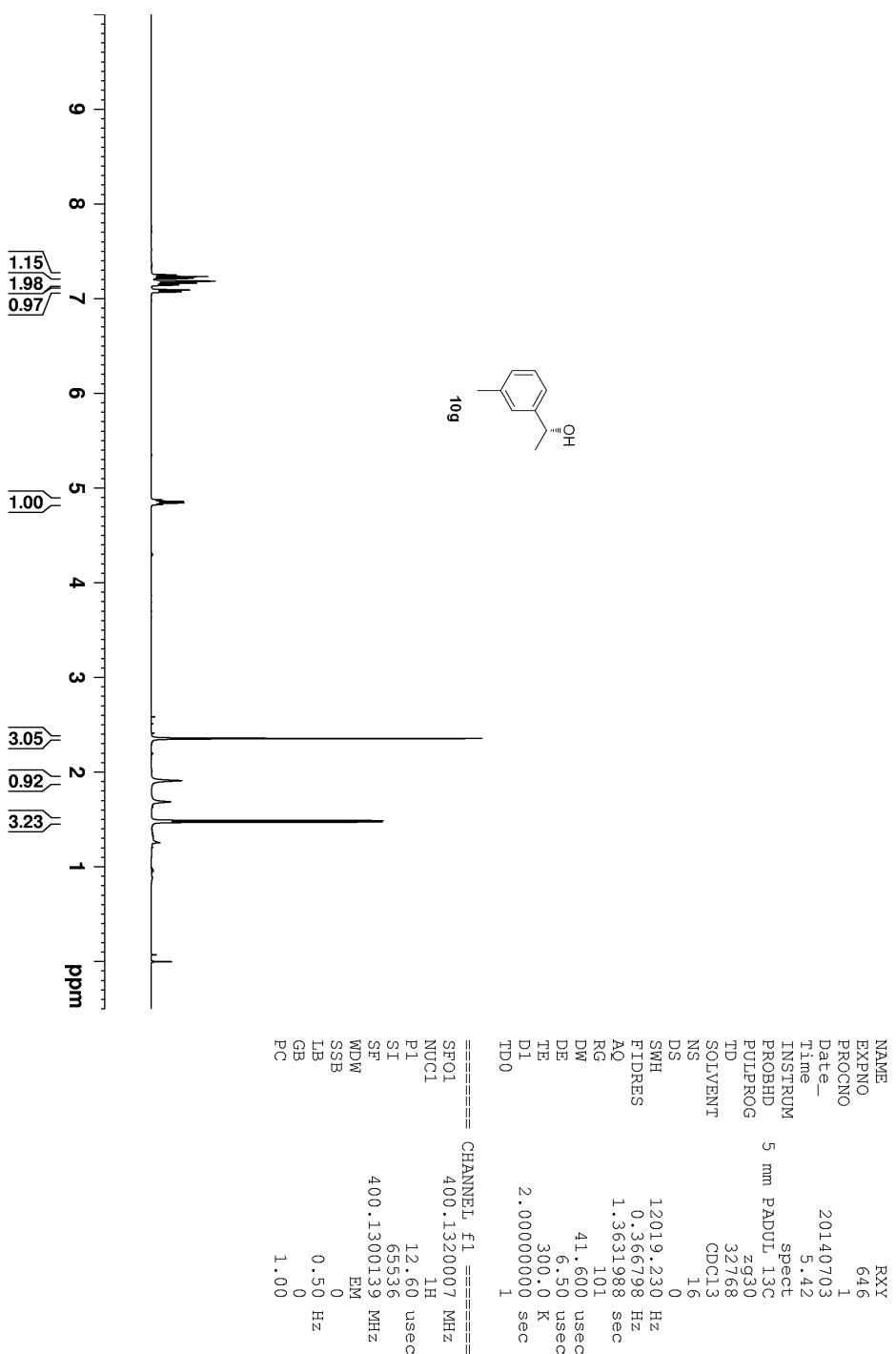
===== CHANNEL f1 =====
SF01        400.1320007 MHz
NUCL1        1H
P1           12.60 usec
SI            65536
SF          400.1300100 MHz
WDW         EM
SSB          0
LB           0.50 Hz
GB           0
PC           1.00

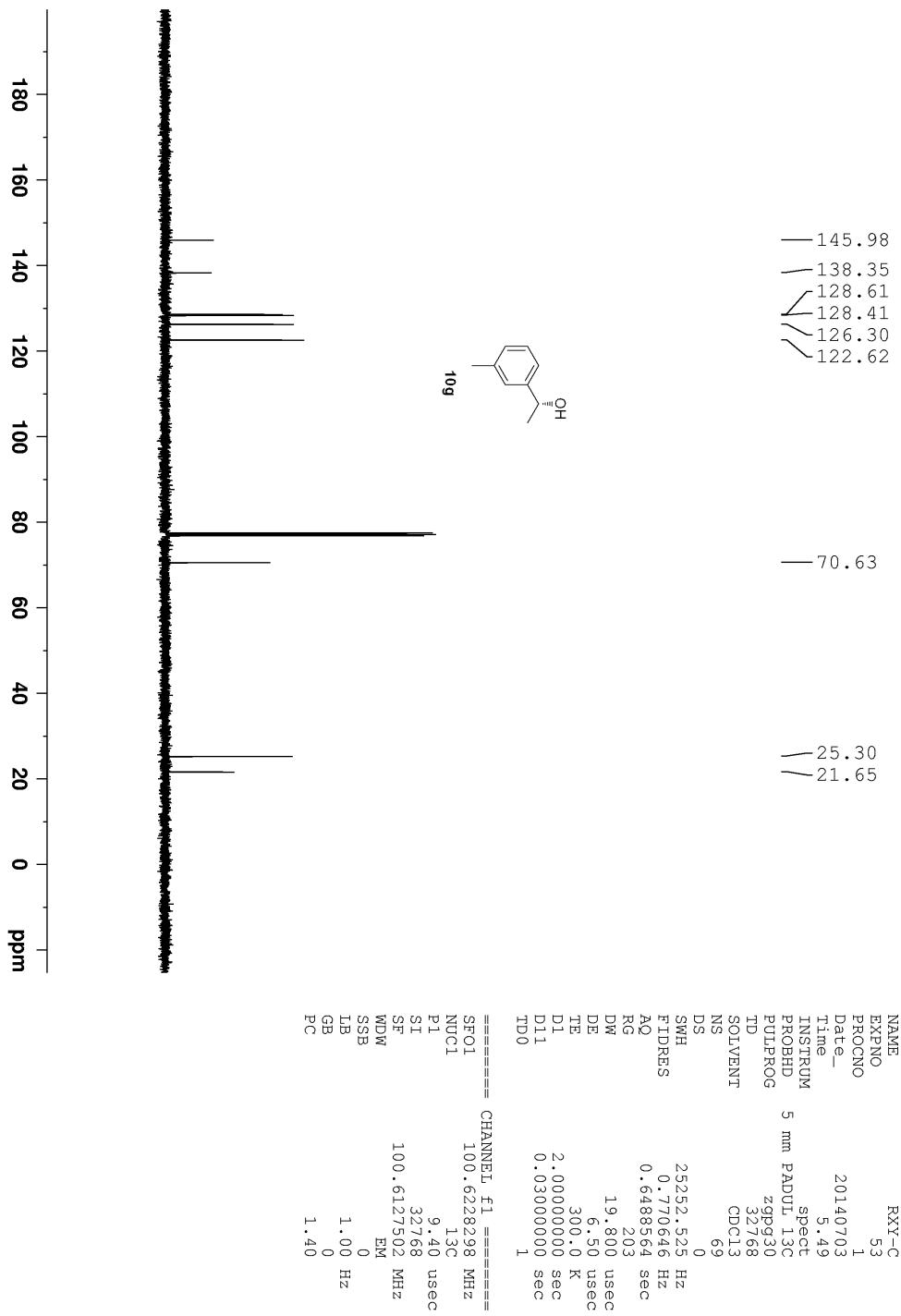
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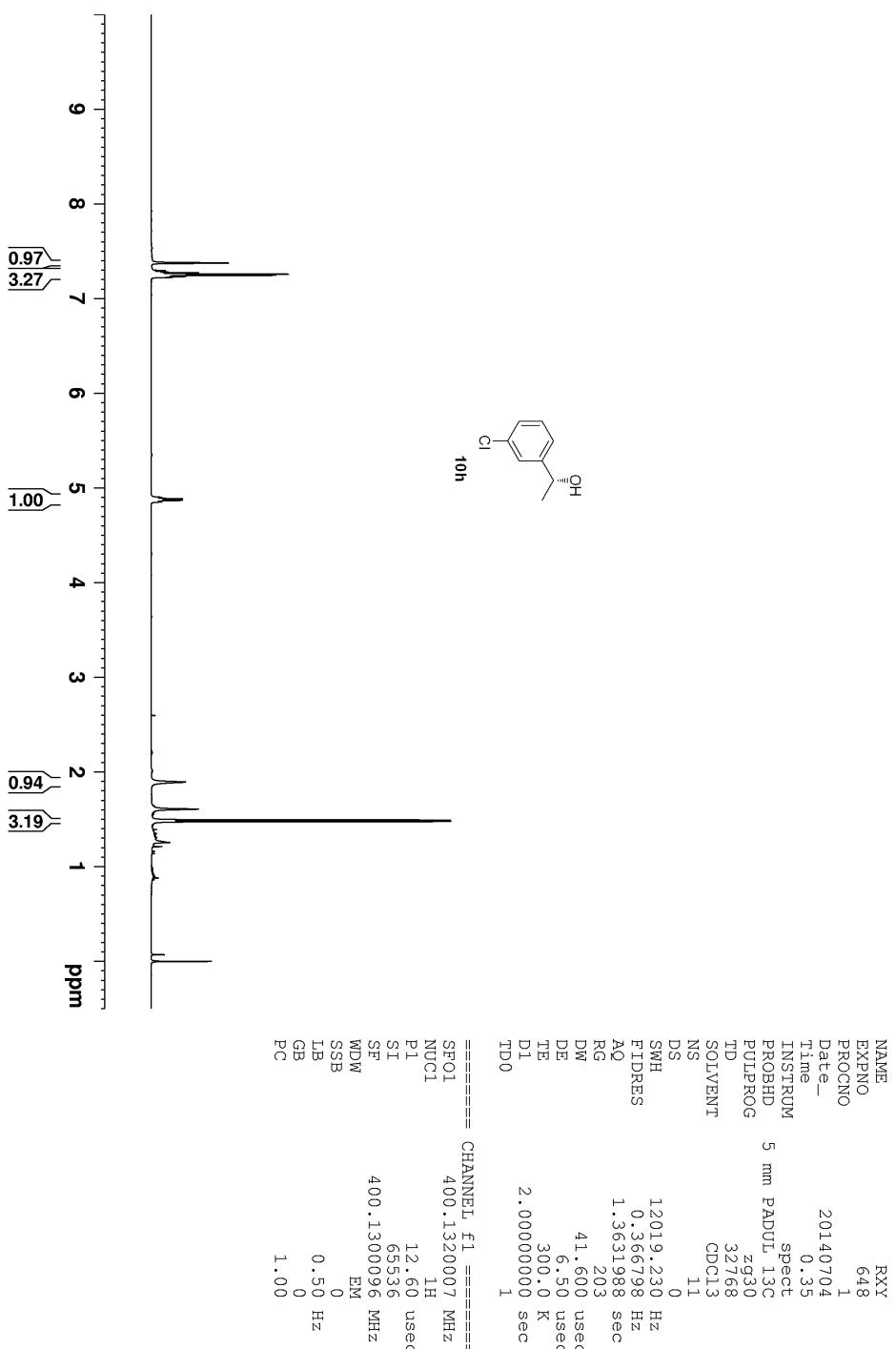


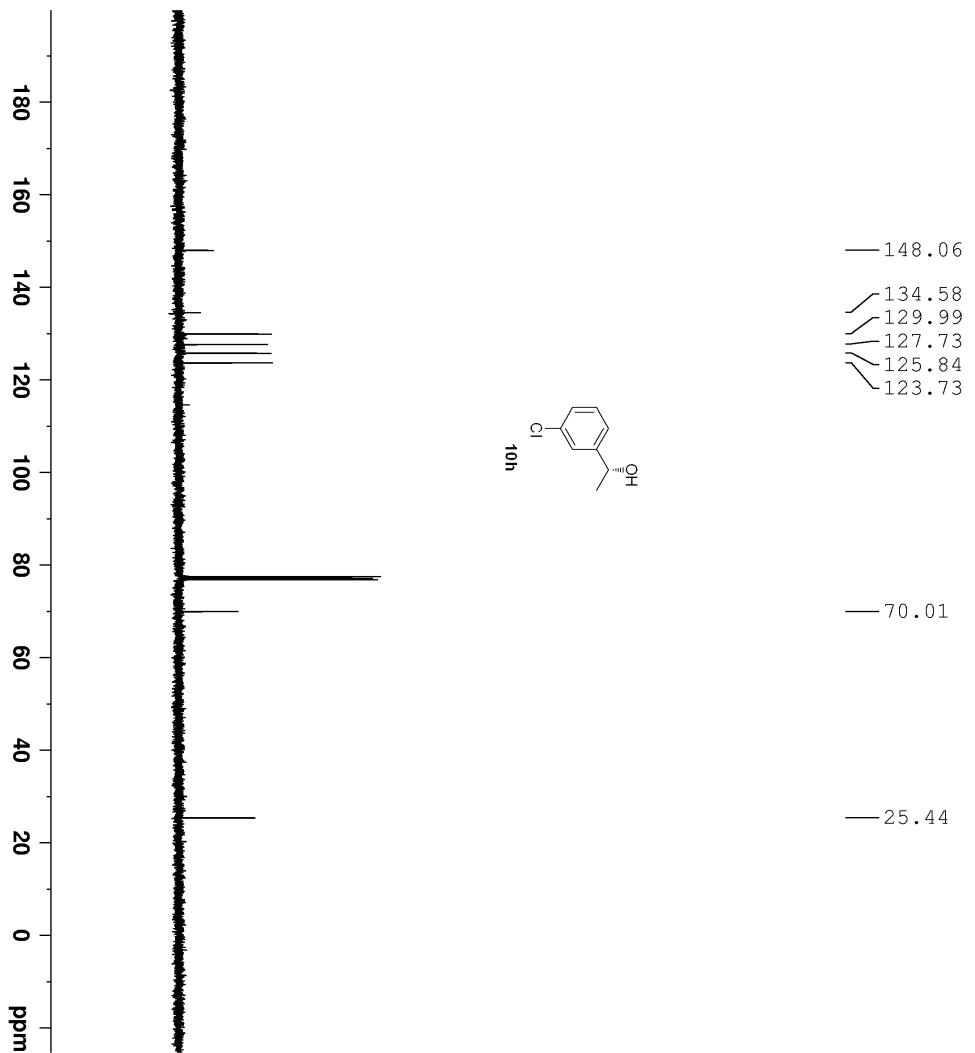








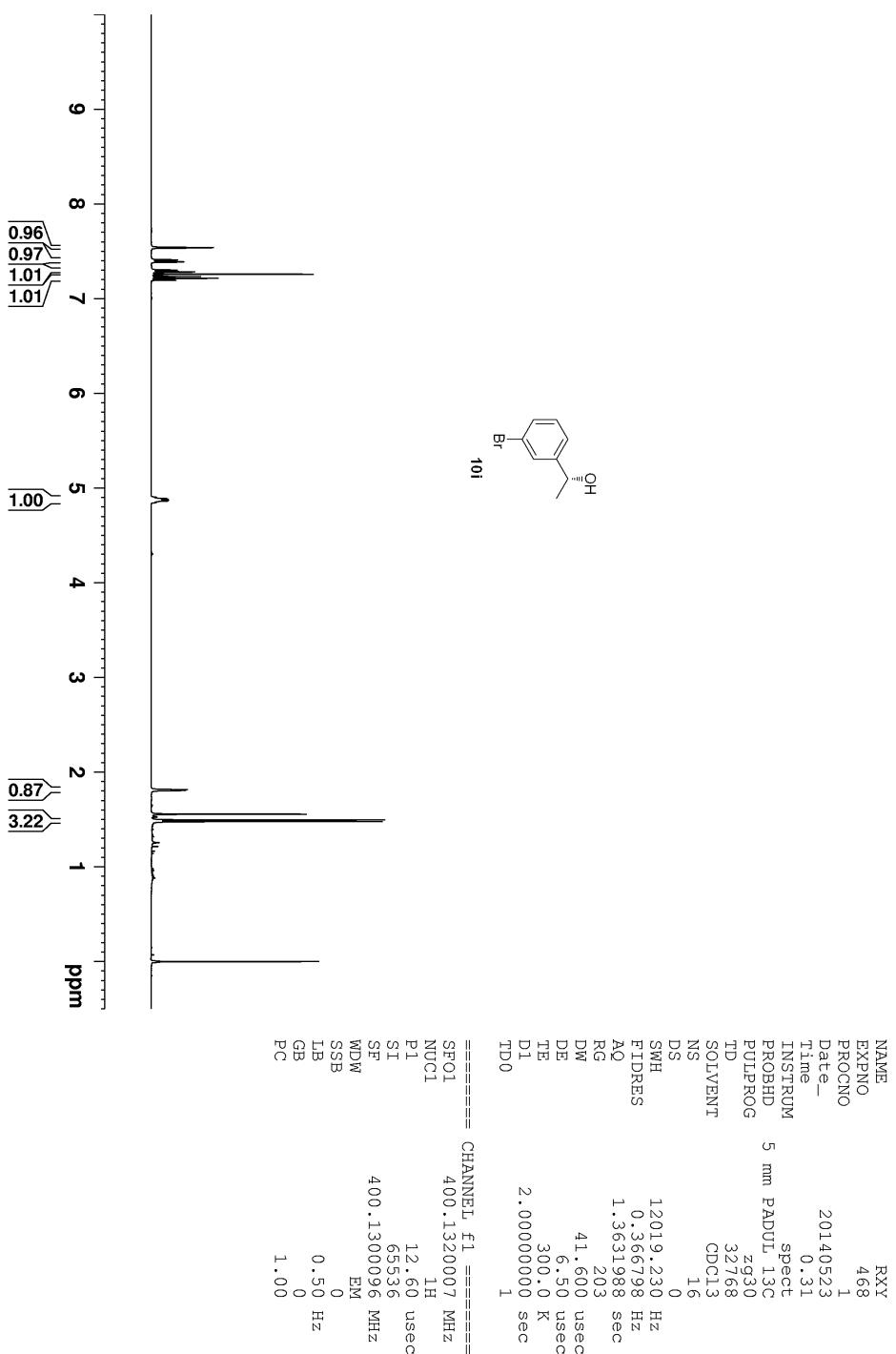


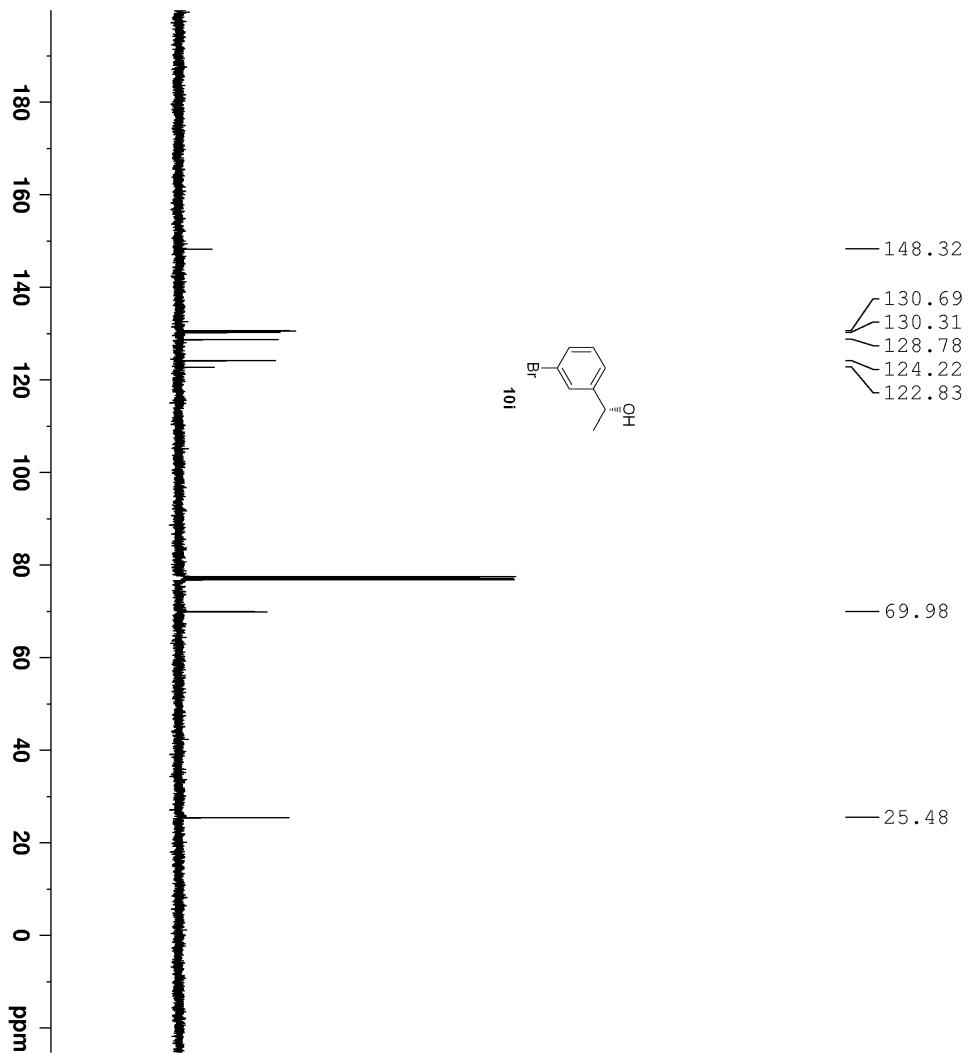


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PROCNO   1
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Time     4.24
INSTRUM spect
PROBHD  5 mm PADUL 13C
PULPROG zqpg30
TD        32768
SOLVENT  CDCl3
NS       42
DS        0
SWH      25252.525 Hz
FIDRES  0.6488554 sec
AQ        203
RG        19.800 usec
DW        6.500 usec
DE        300.0 K
TE        2.0000000 sec
D1       0.03000000 sec
T1       1
TD0

===== CHANNEL f1 =====
SF01    100.622828 MHz
NUC1    13C
P1      9.40 usec
SI      32768
SF      100.6127491 MHz
WDW
SSB
LB      1.00 Hz
GB      0
PC      1.40
  
```



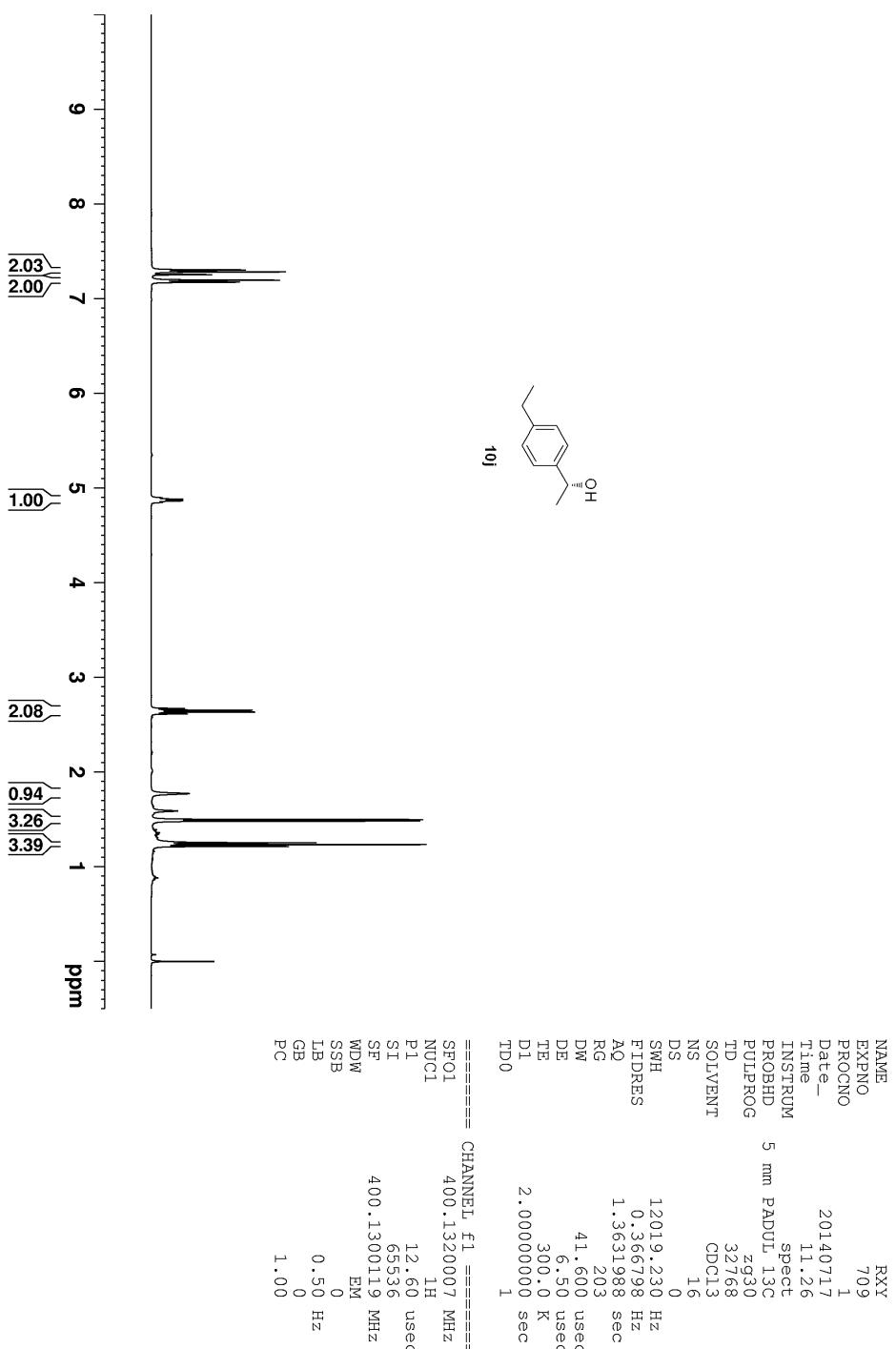


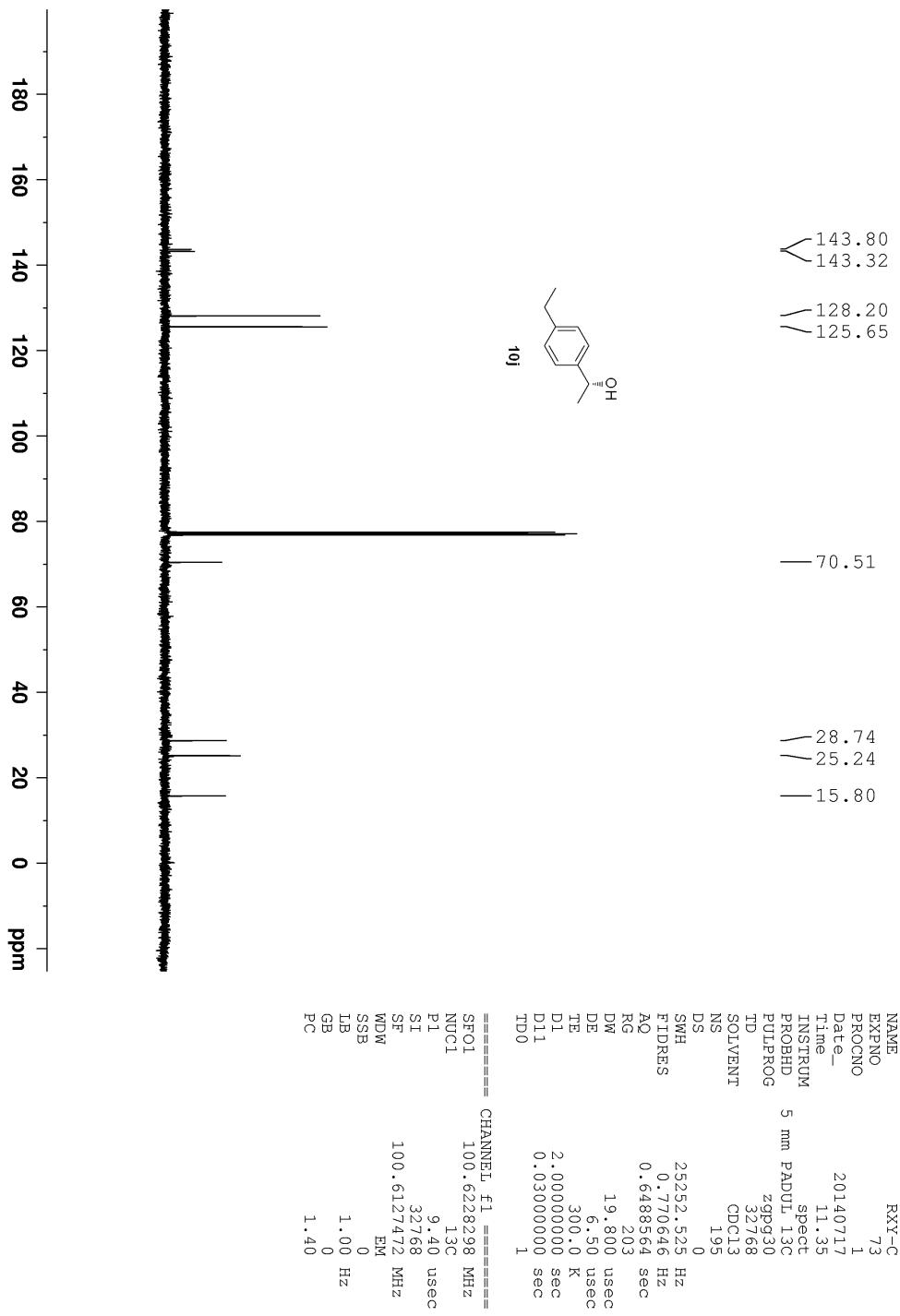
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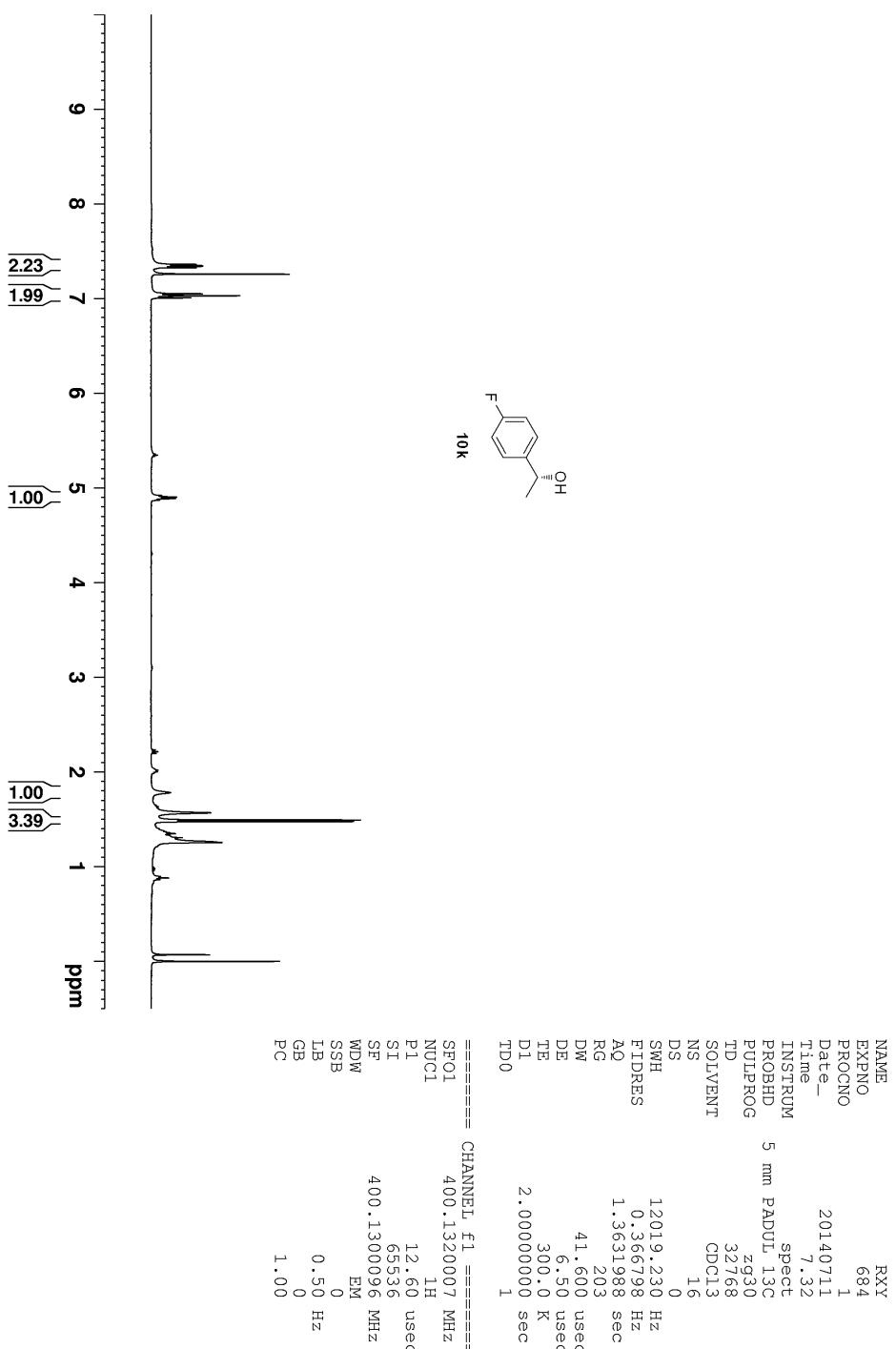
=====
NAME      RXY-C
EXPNO    24
PROCNO   1
Date_    20140523
Time     1.57
INSTRUM spect
PROBHD  5 mm PADUL 13C
PULPROG zqpg30
TD        32768
SOLVENT  CDCl3
NS       167
DS        0
SWH      25252.525 Hz
FIDRES   0.6488554 sec
AQ        203
RG        19.800 usec
DW        6.500 usec
DE        300.0 K
TE        2.0000000 sec
D1        0.03000000 sec
T1        1
TD0

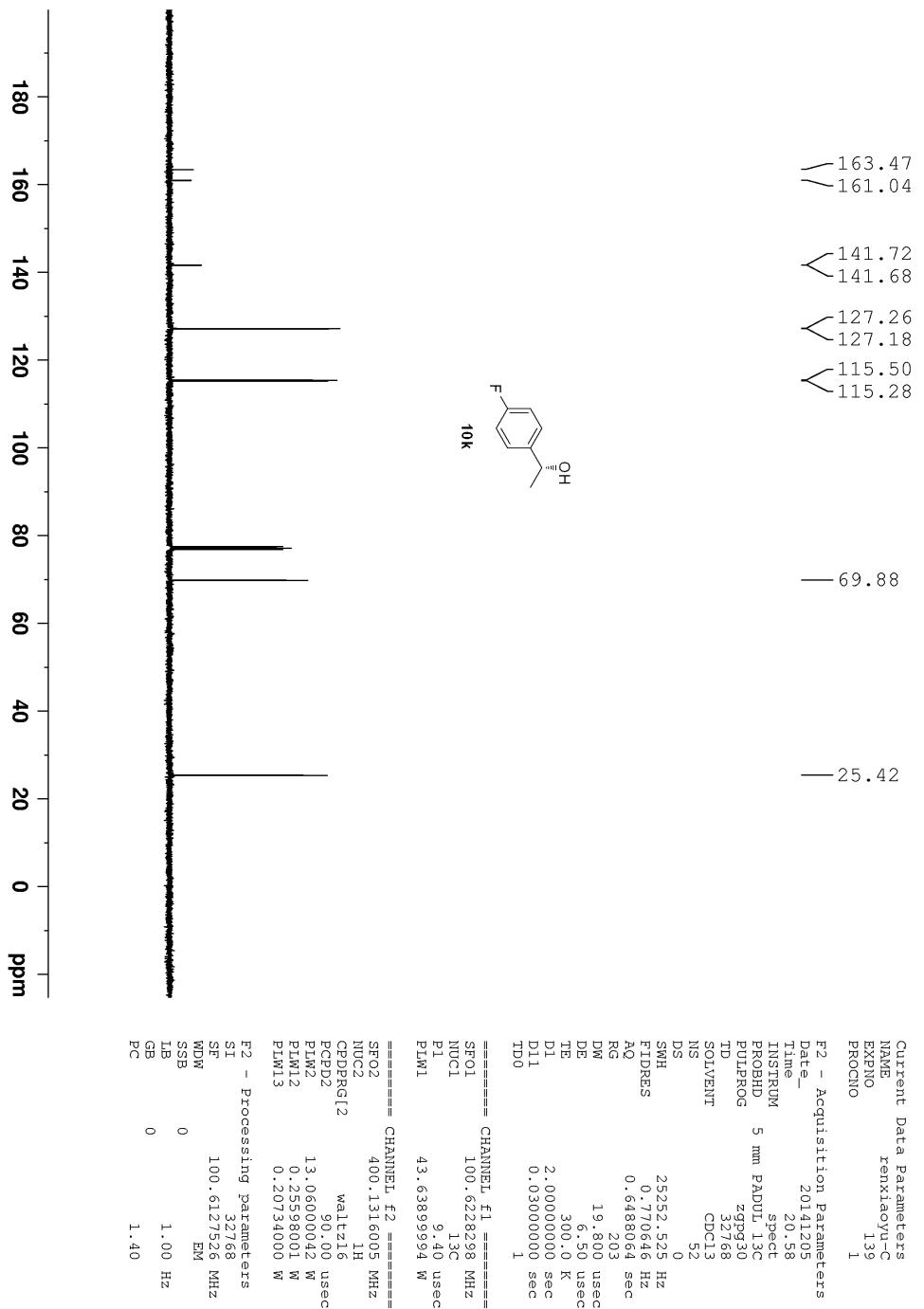
===== CHANNEL f1 =====
SF01    100.622828 MHz
NUC1    13C
P1      9.40 usec
SI      32768
SF      100.6127483 MHz
WDW
SSB
LB      1.00 Hz
GB      0
PC      1.40

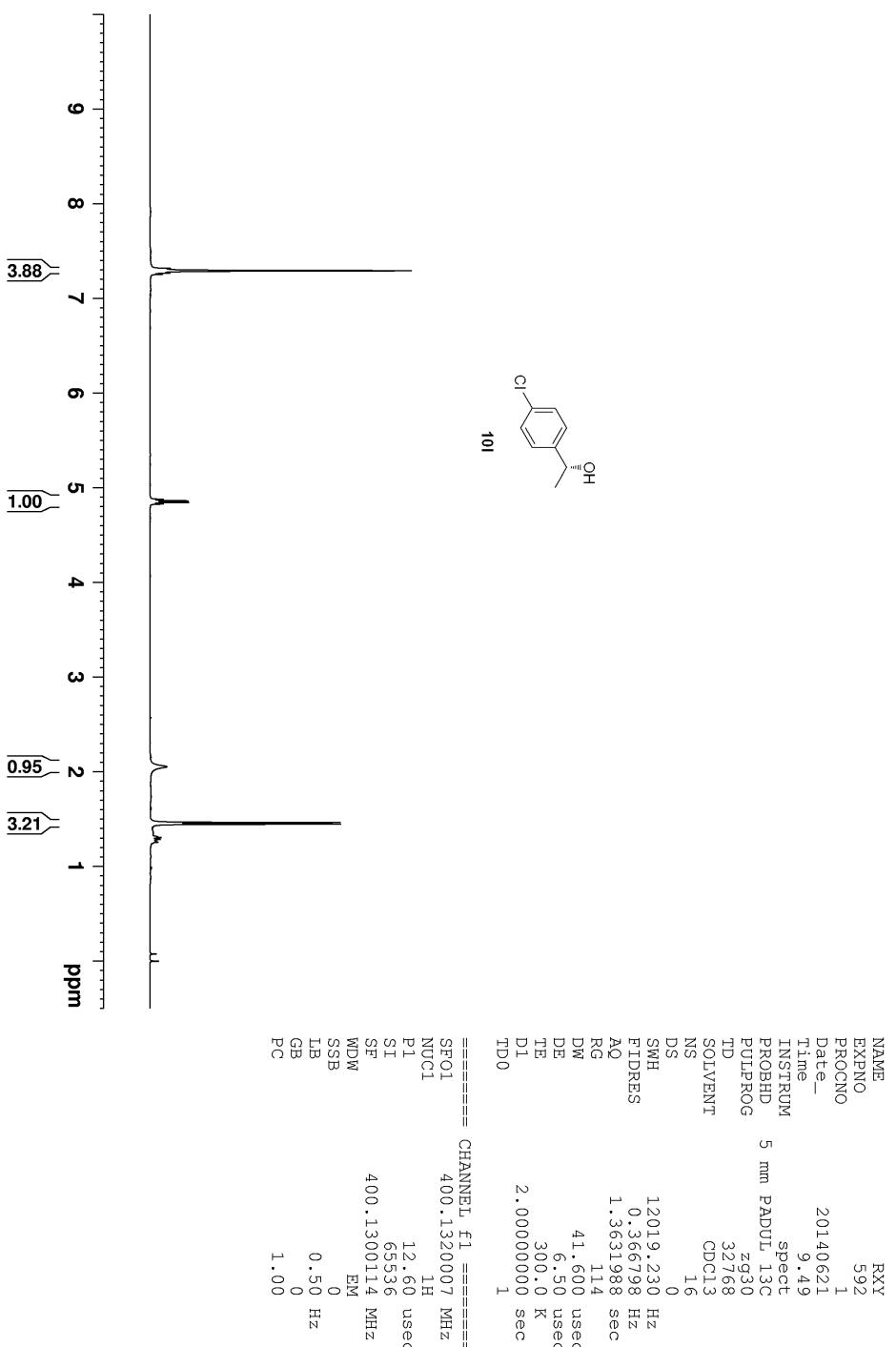
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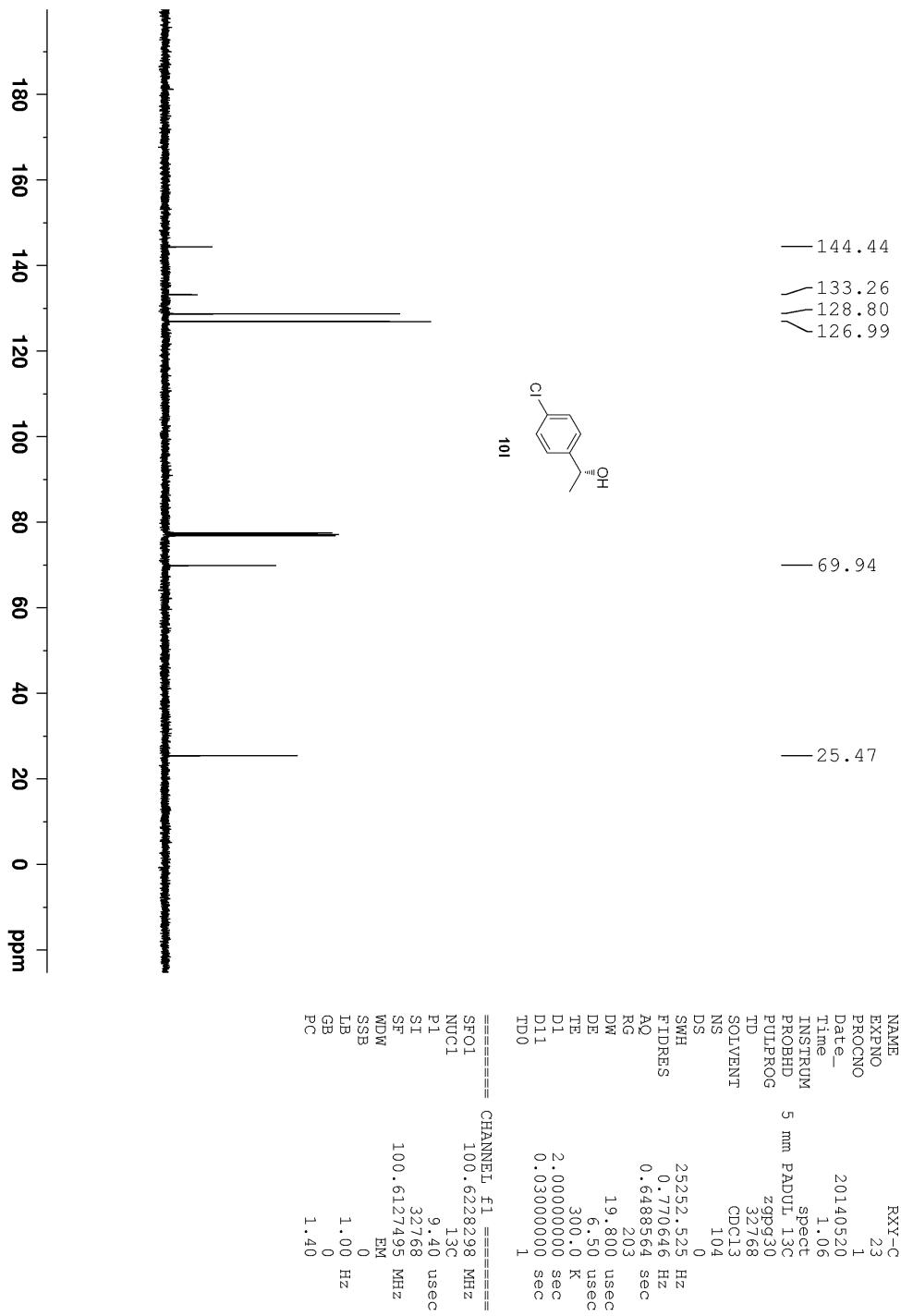


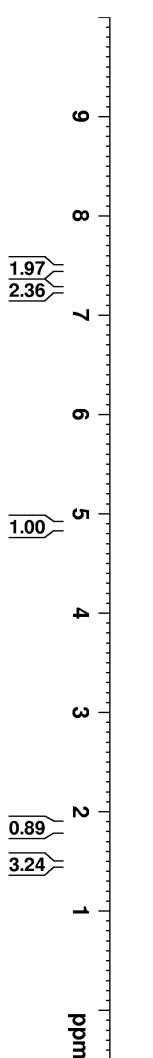
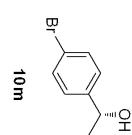










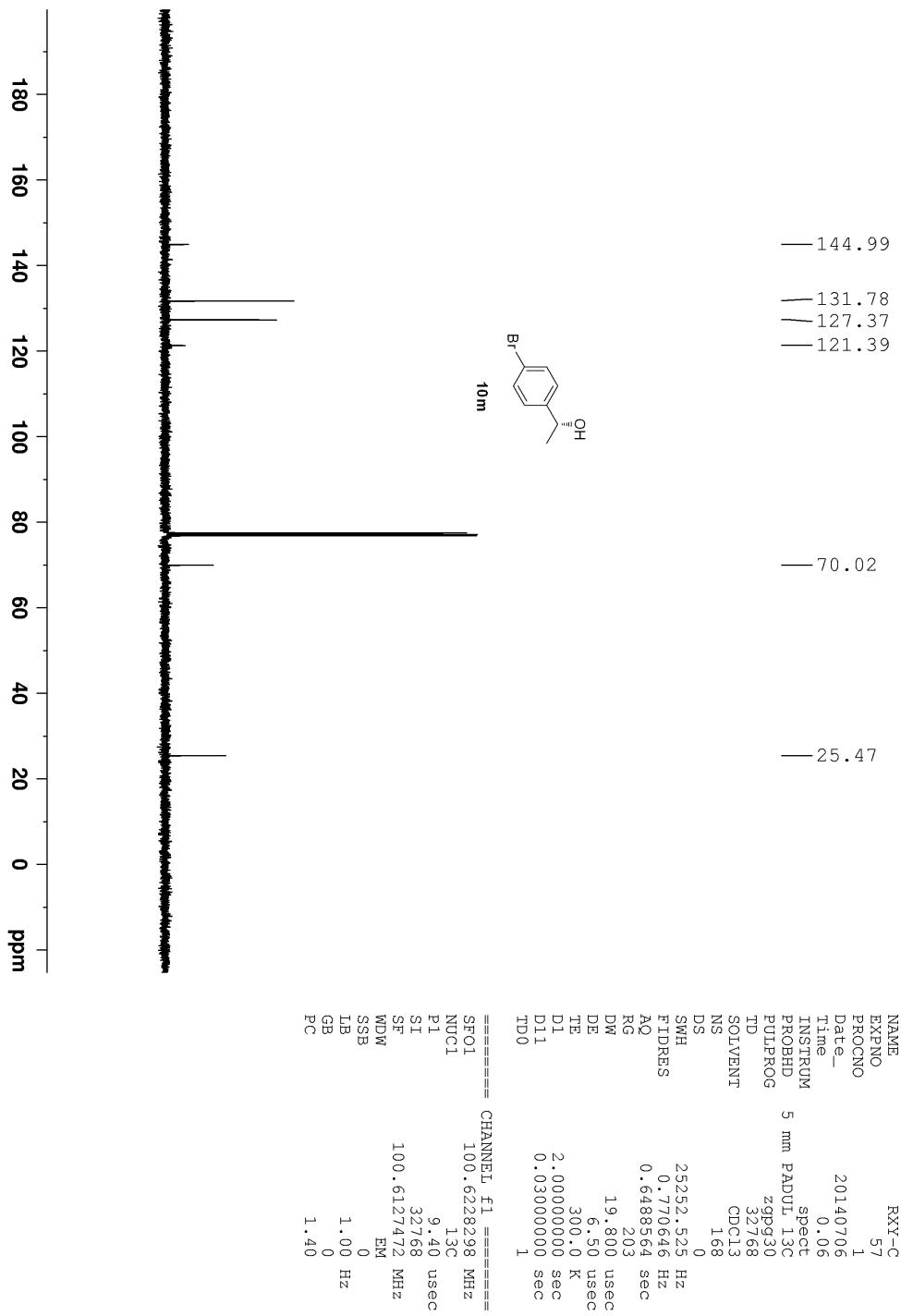


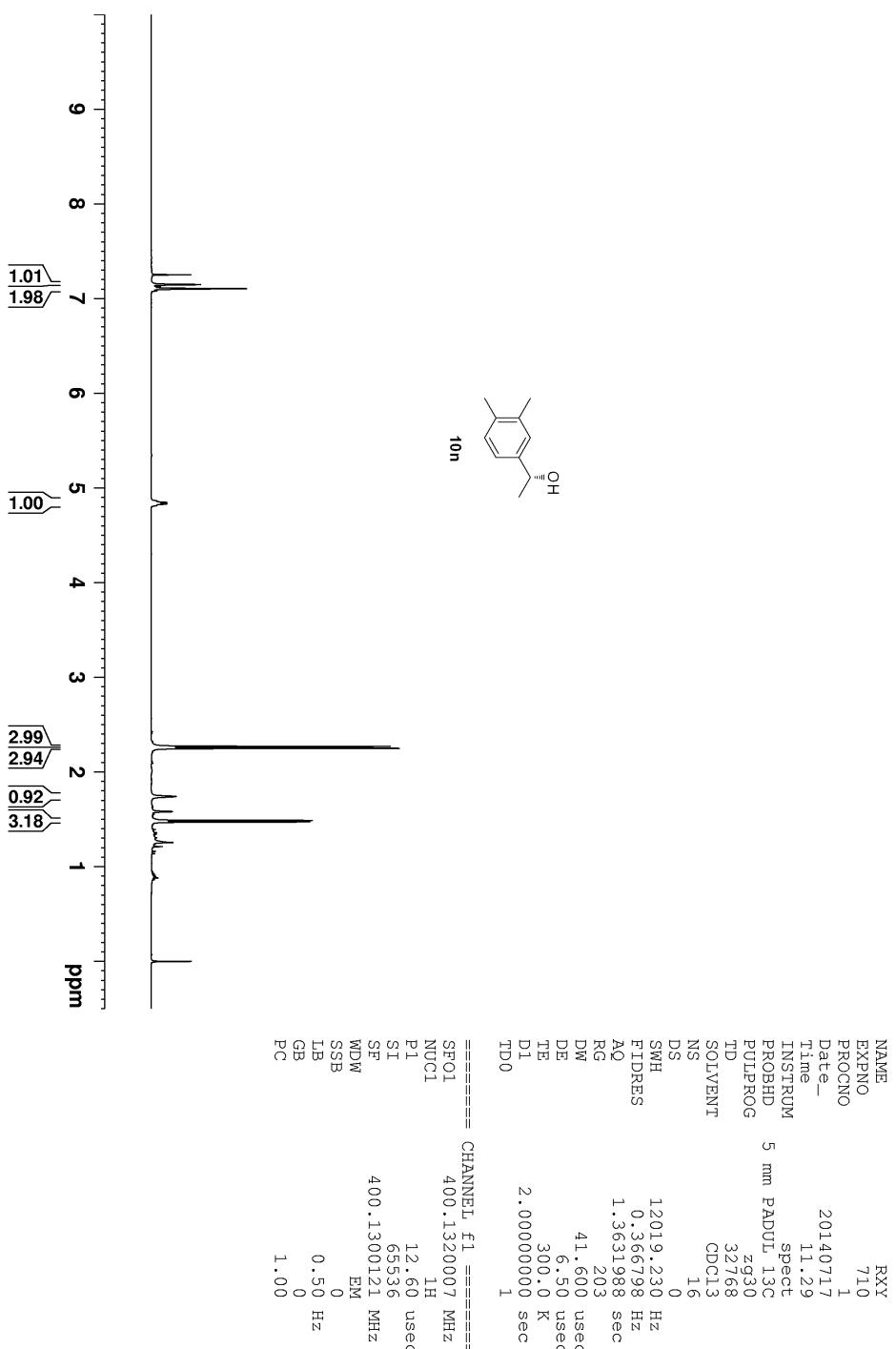
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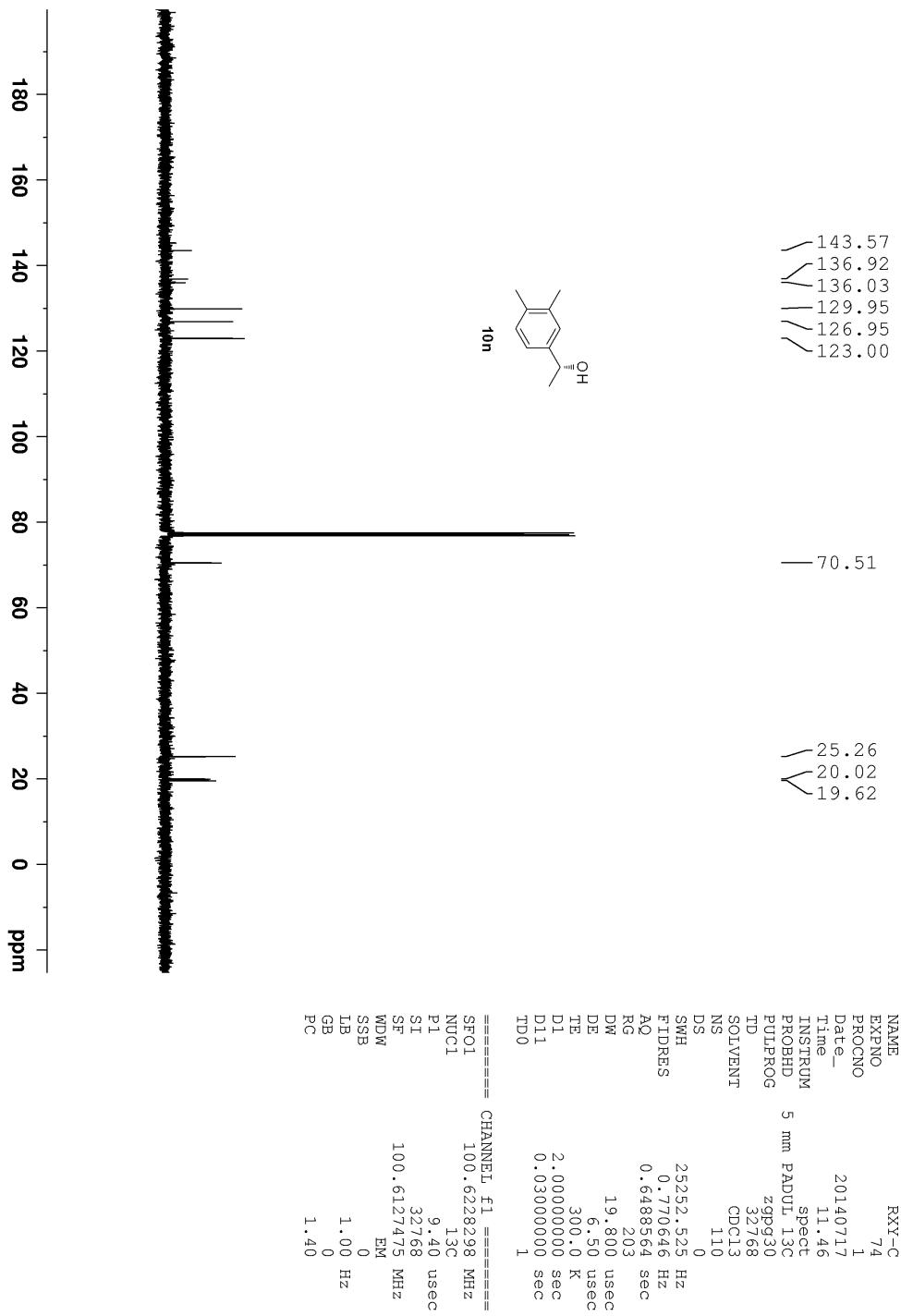
=====
NAME          RXY
EXPNO        664
PROCNO       1
Date_        20140705
Time         23.51
INSTRUM     spect
PROBHD      5 mm PADUL 13C
PULPROG     zg30
TD          32768
SOLVENT      CDCl3
NS           13
DS            0
SWH         12019.230 Hz
FIDRES     0.366798 Hz
AQ          1.3631988 sec
RG           203
DW           41.600 usec
DE           6.500 usec
TE           300.0 K
D1          2.0000000 sec
TDO          1

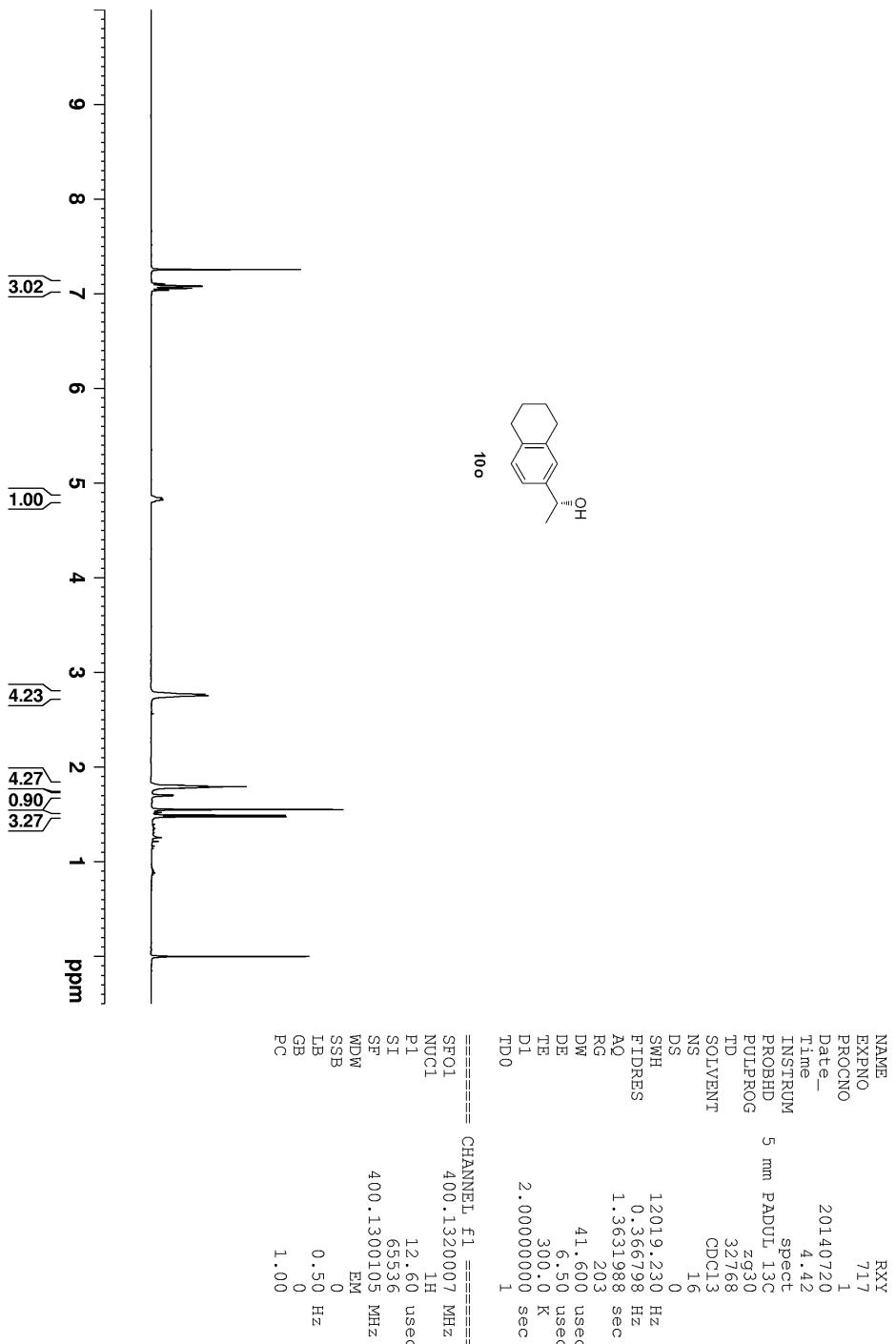
===== CHANNEL f1 =====
SFO1        400.1320007 MHz
NUC1         1H
P1          12.60 usec
SI           65536
SF          400.1300096 MHz
WDW         EM
SSB          0
LB          0.50 Hz
GB          1.00
PC

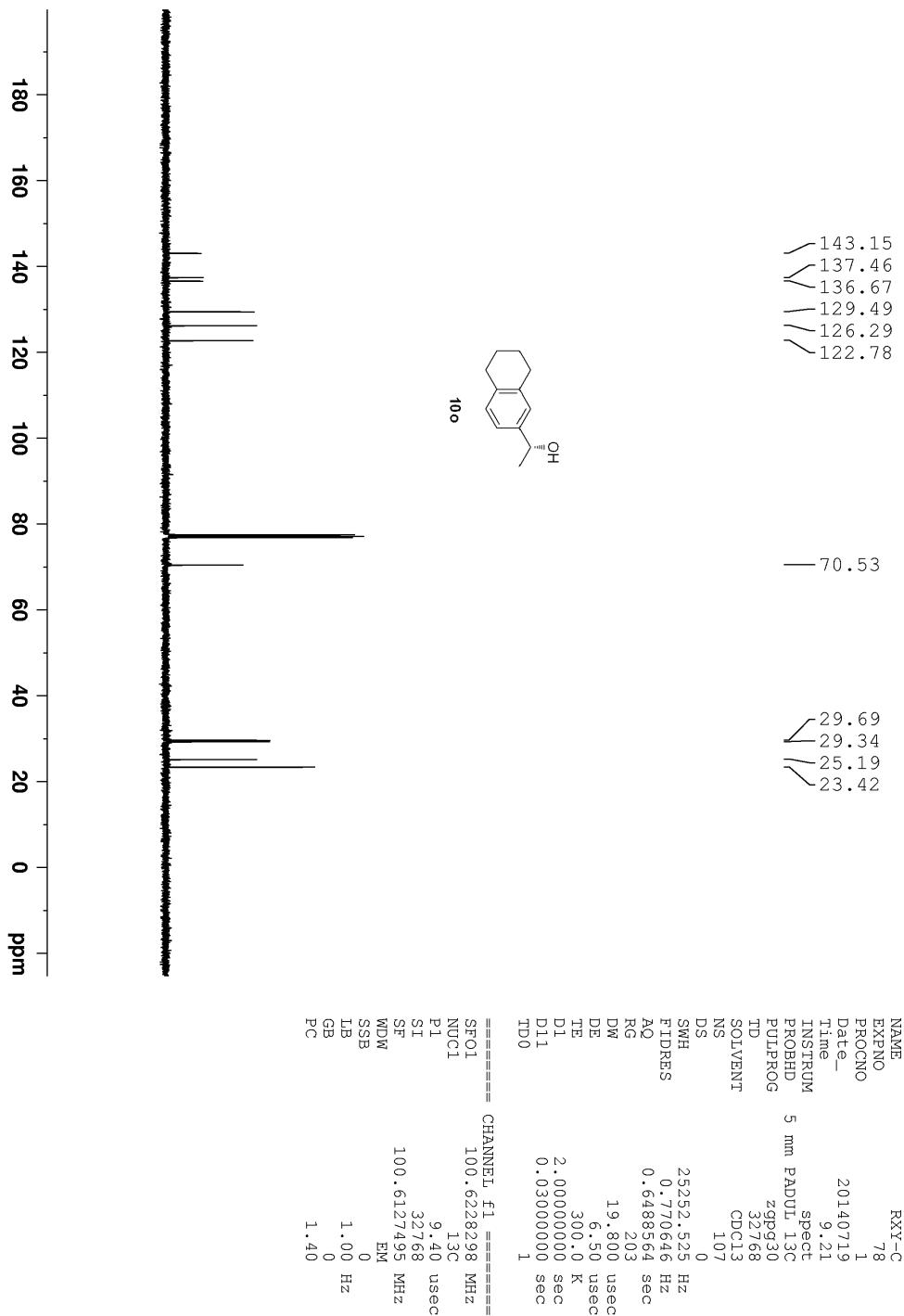
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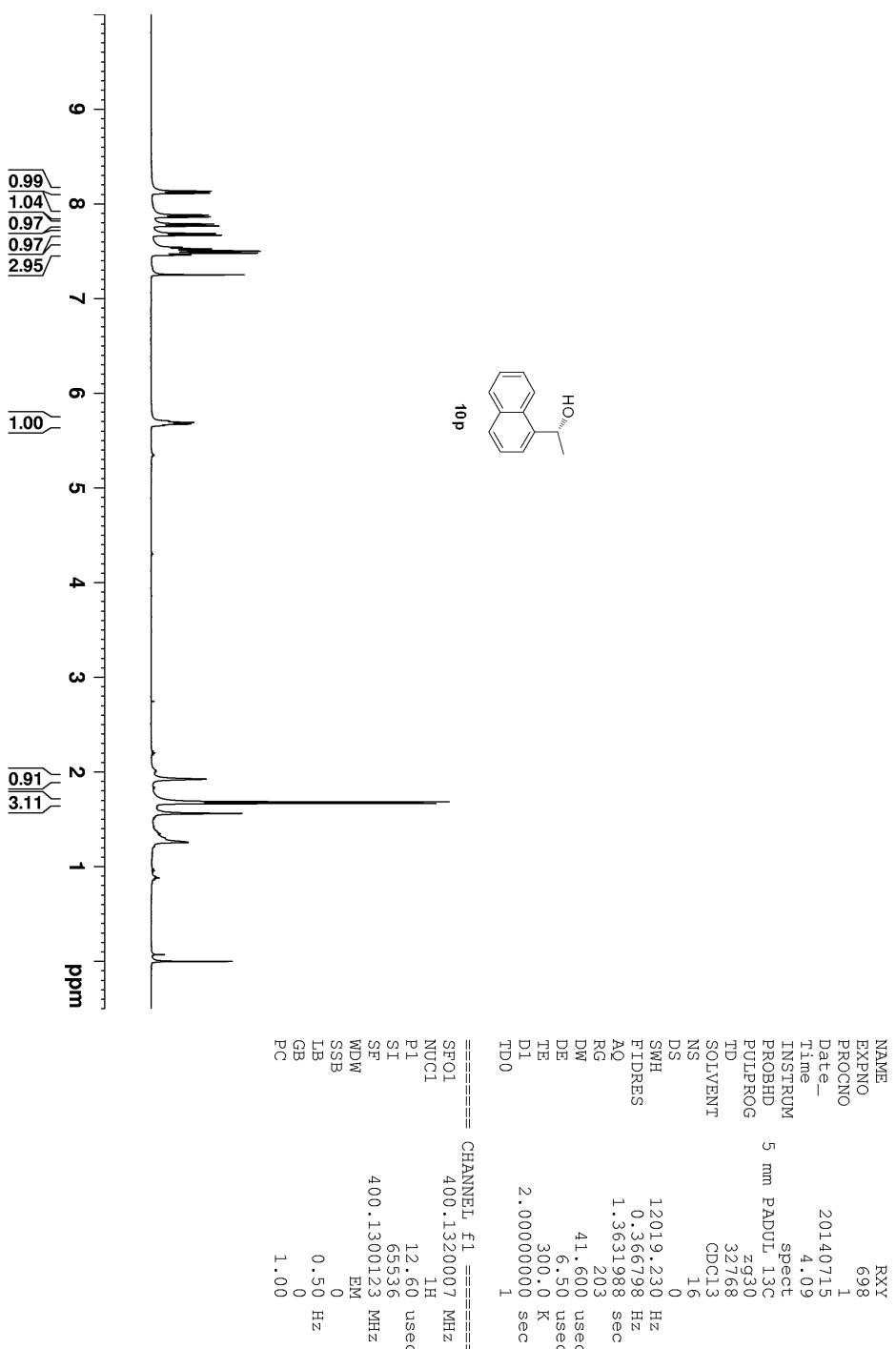


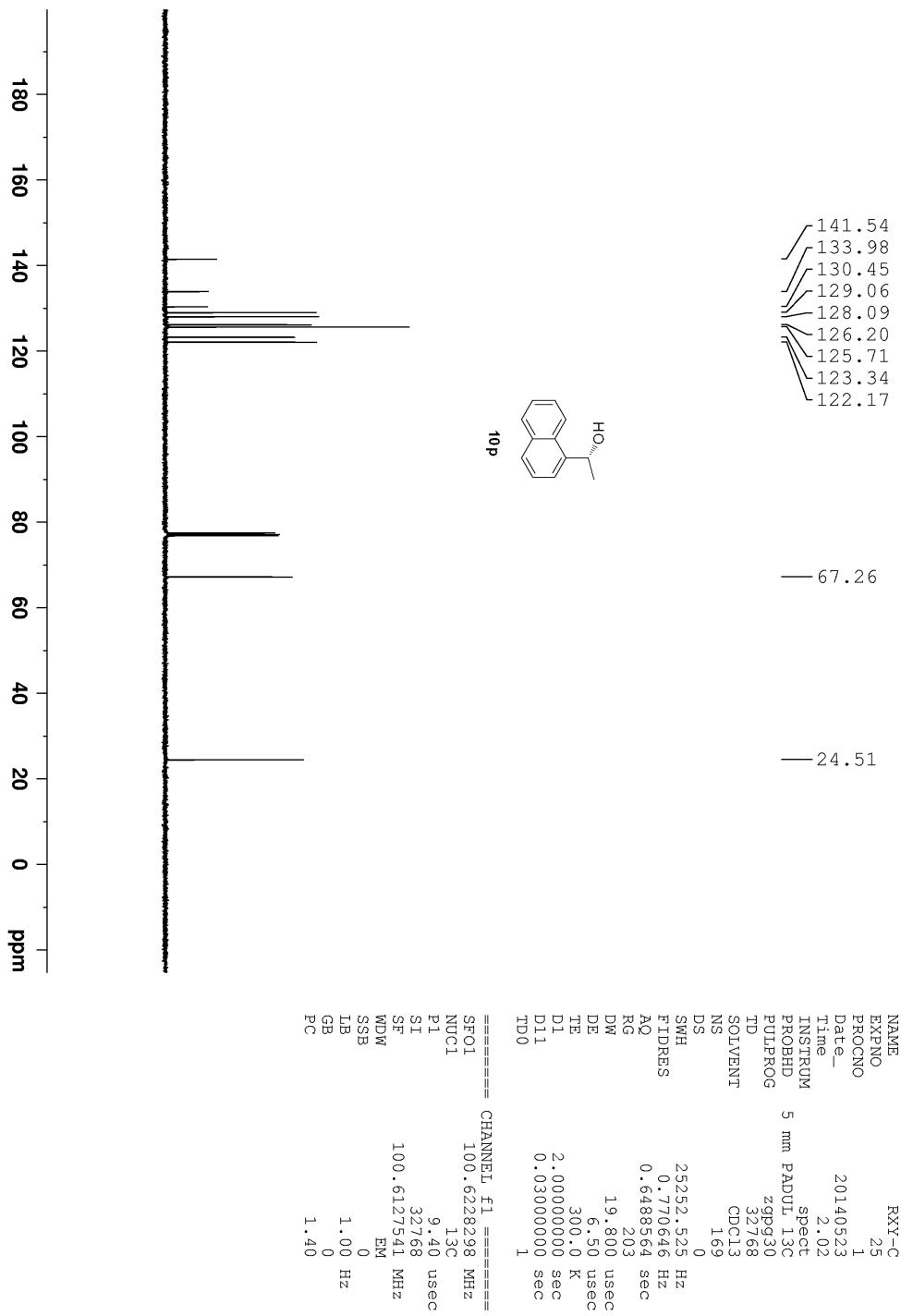


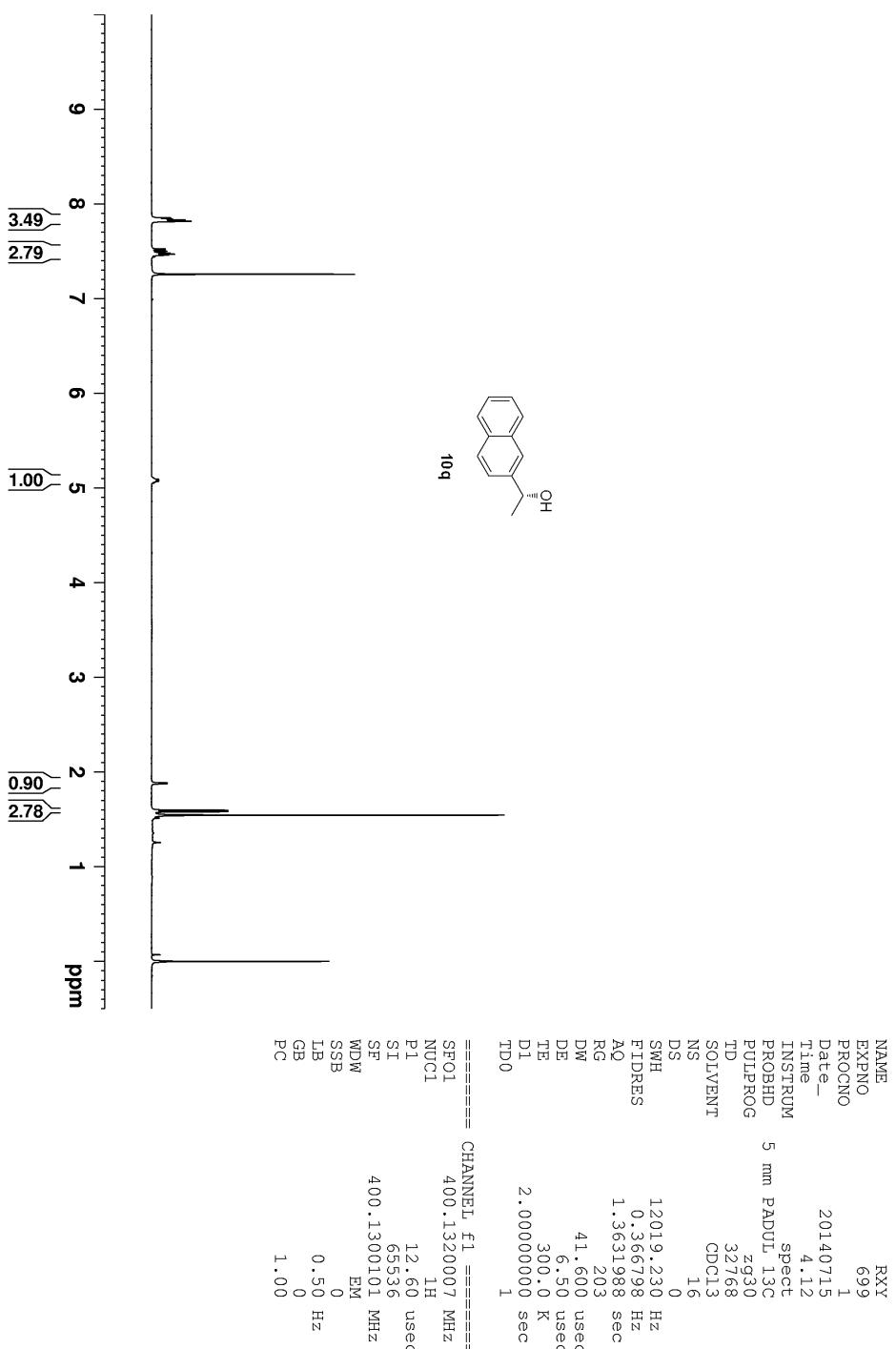


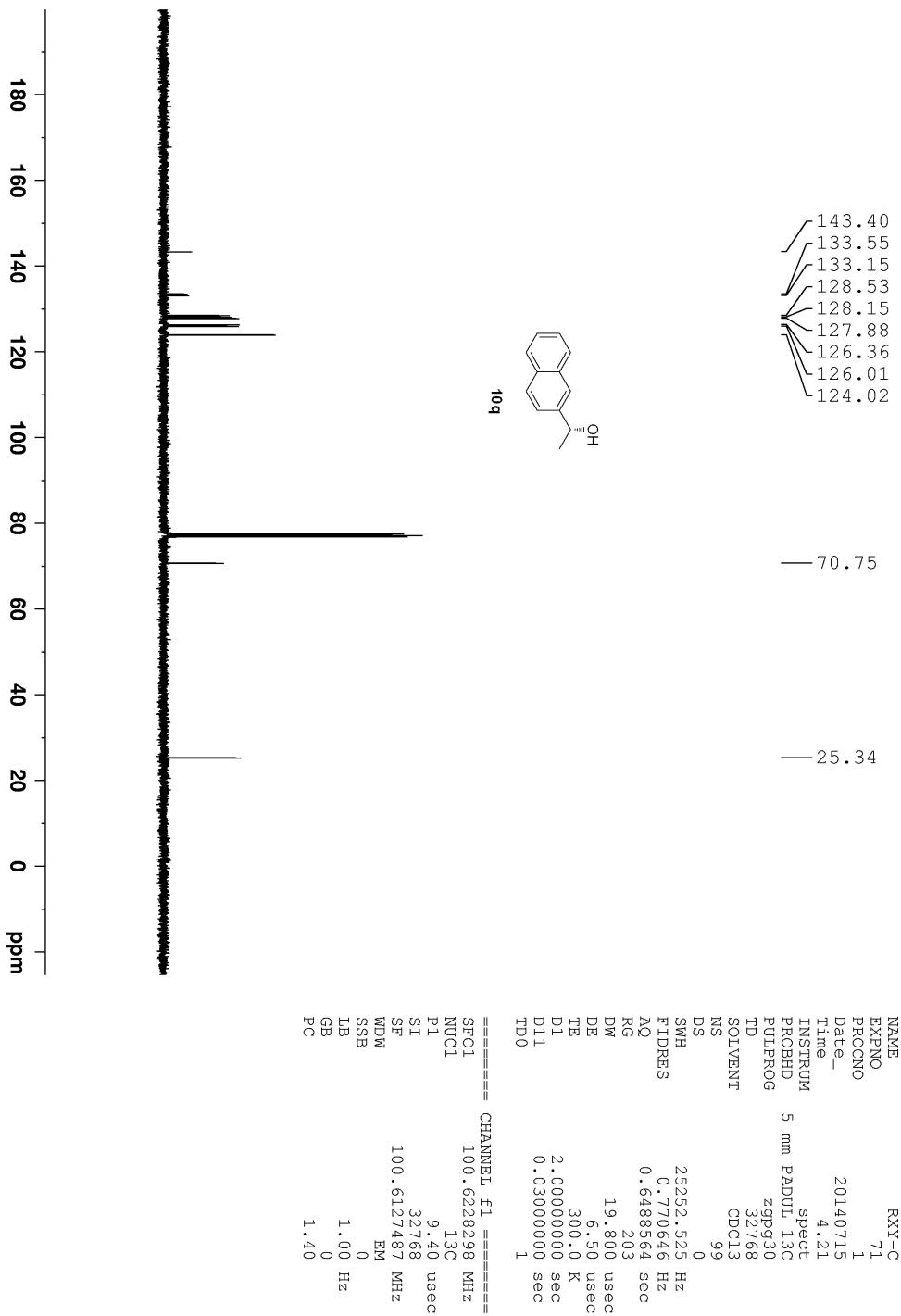


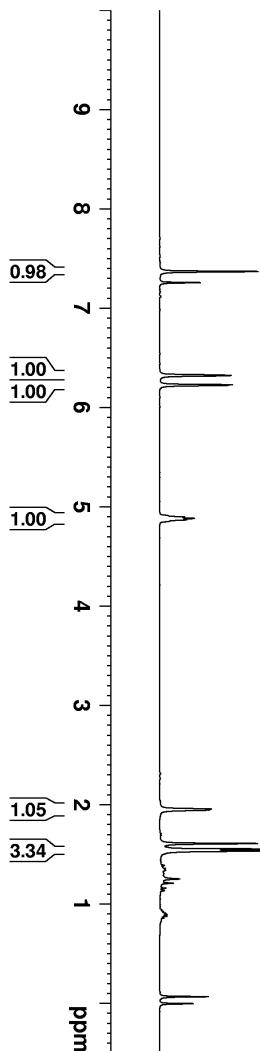










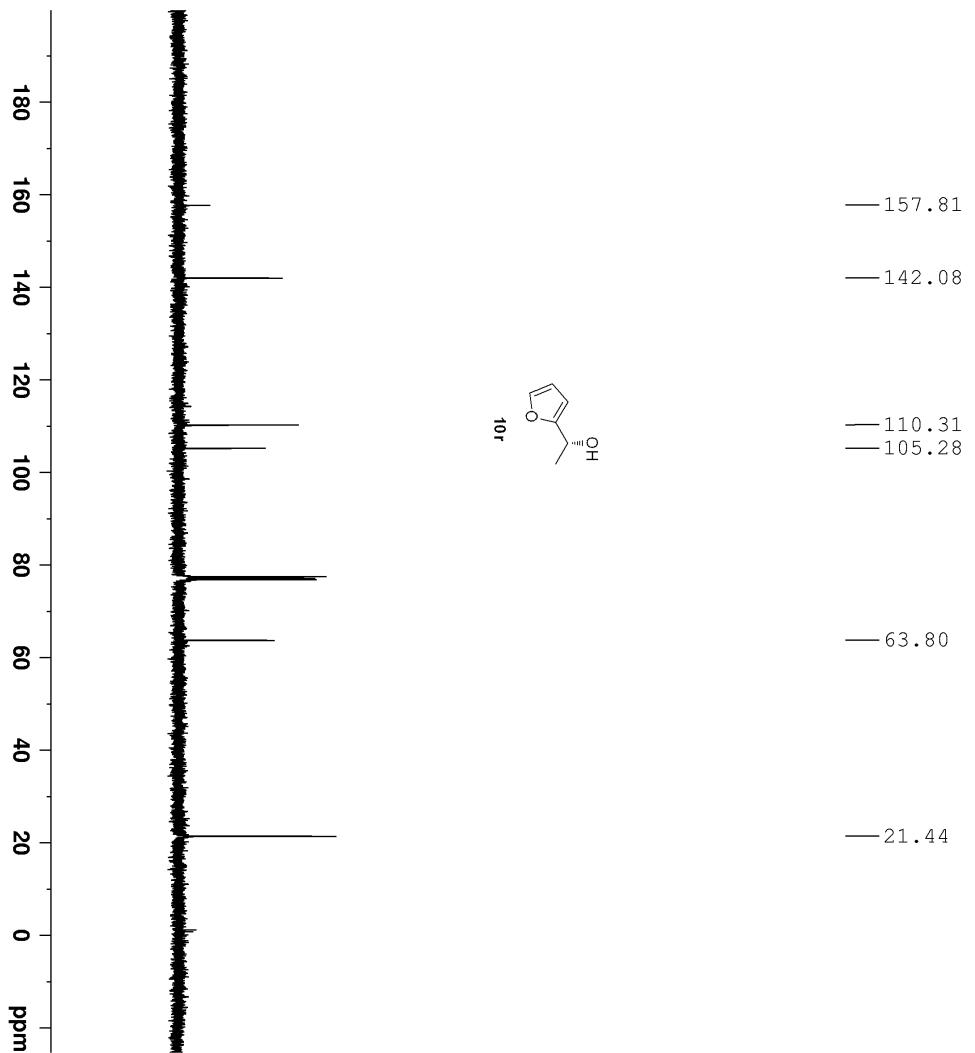


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=====
NAME          RXY
EXPNO        920
PROCNO       1
Date_        20140922
Time         8.17
INSTRUM     spect
PROBHD      5 mm PADUL 13C
PULPROG     zg30
TD          32768
SOLVENT      CDCl3
NS           16
DS            0
SWH         12019.230 Hz
FIDRES     0.366798 Hz
AQ          1.3631988 sec
RG           203
DW           41.600 usec
DE           6.500 usec
TE           300.0 K
D1          2.0000000 sec
TDO          1

===== CHANNEL f1 =====
SF01        400.1320007 MHz
NUC1         1H
P1          12.60 usec
SI           65536
SF          400.1300097 MHz
WDW         EM
SSB          0
LB          0.50 Hz
GB          1.00
PC

```

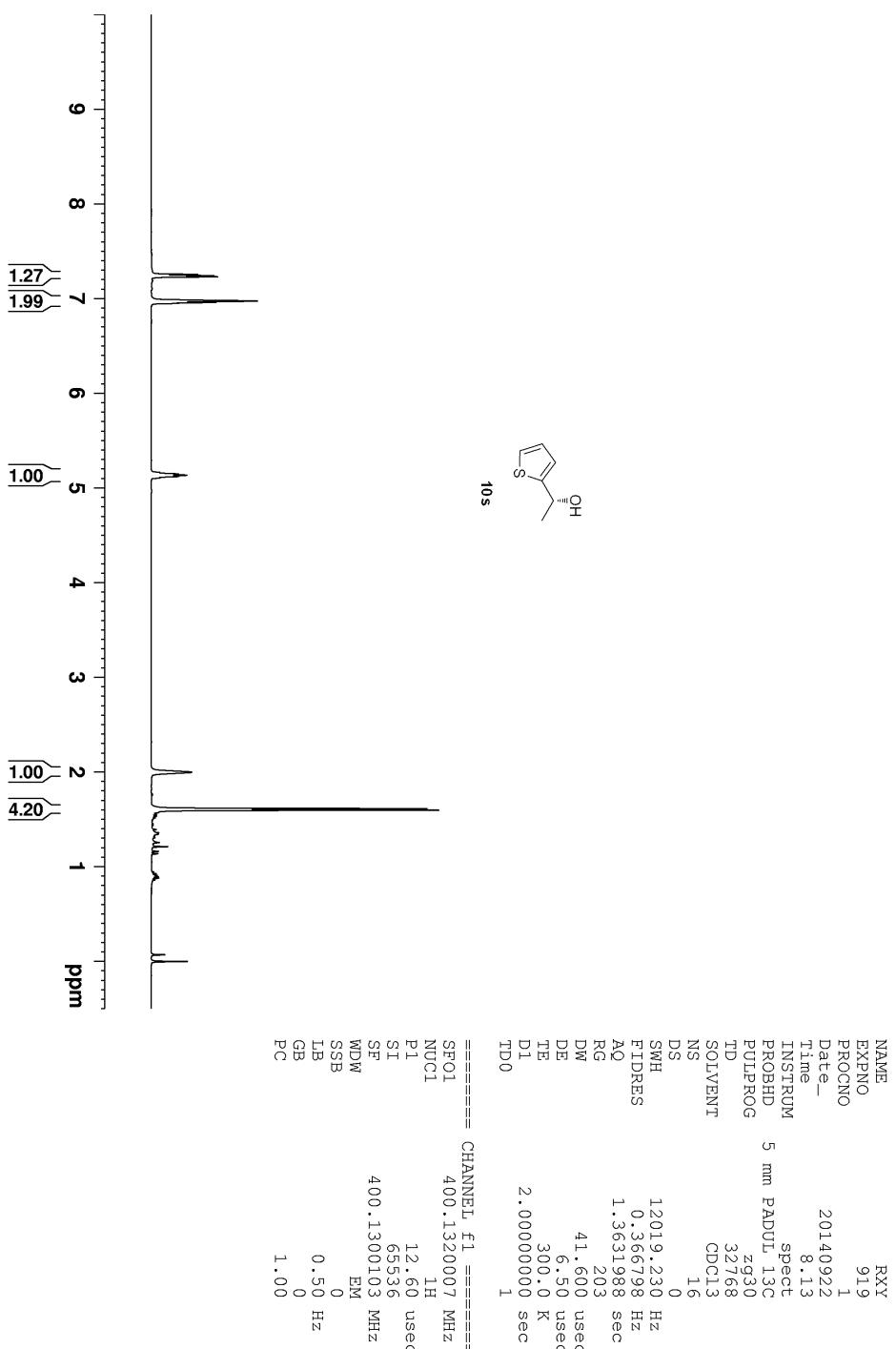


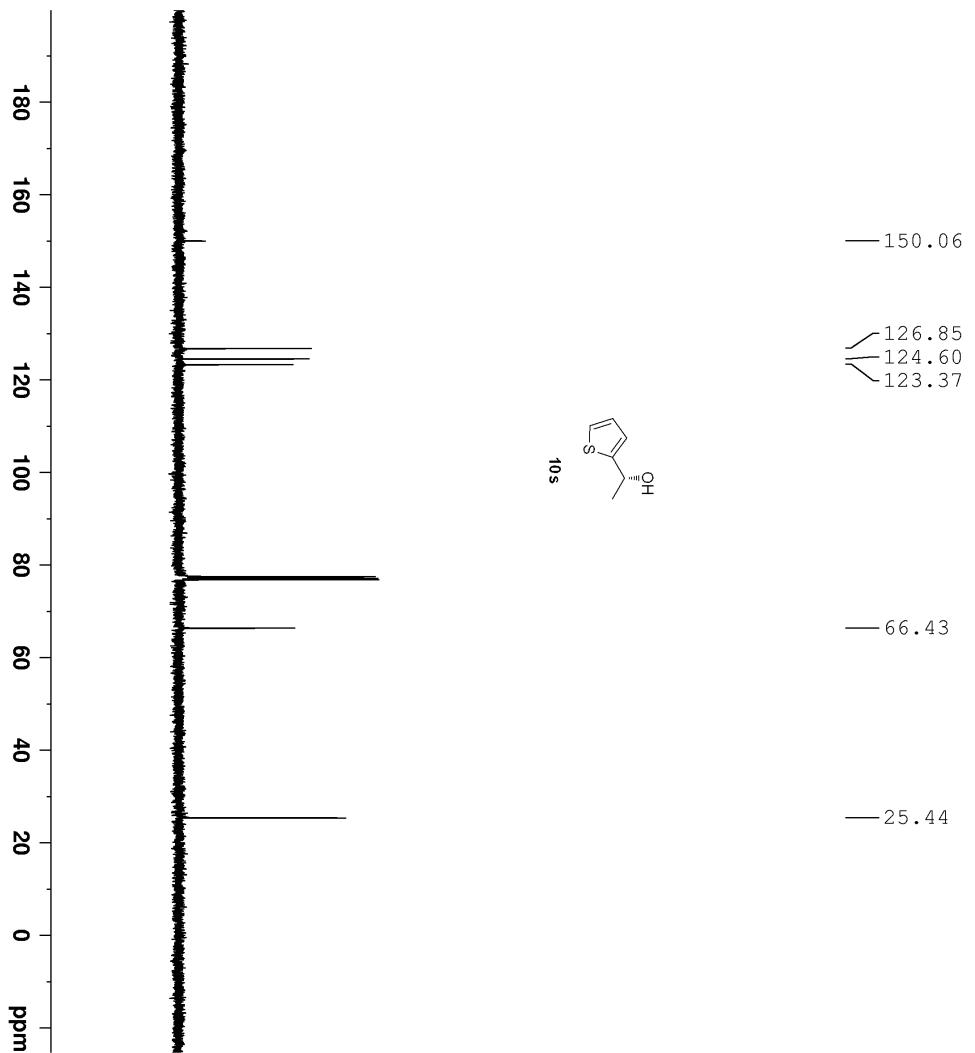
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=====
NAME      RXY-C
EXPNO    100
PROCNO   1
Date_    20140922
Time     7.54
INSTRUM spect
PROBHD  5 mm PADUL 13C
PULPROG zgpg30
TD        32768
SOLVENT  CDCl3
NS       87
DS        0
SWH      25252.525 Hz
FIDRES   0.6488554 sec
AQ        203
RG        19.800 usec
DW        6.50 usec
DE        300.0 K
TE        2.0000000 sec
D1       0.03000000 sec
T1       1
TD0

===== CHANNEL f1 =====
SF01      100.622828 MHz
NUC1      13C
P1        9.40 usec
SI        32768
SF        100.6127498 MHz
WDW
SSB      0
LB        1.00 Hz
GB        0
PC        1.40

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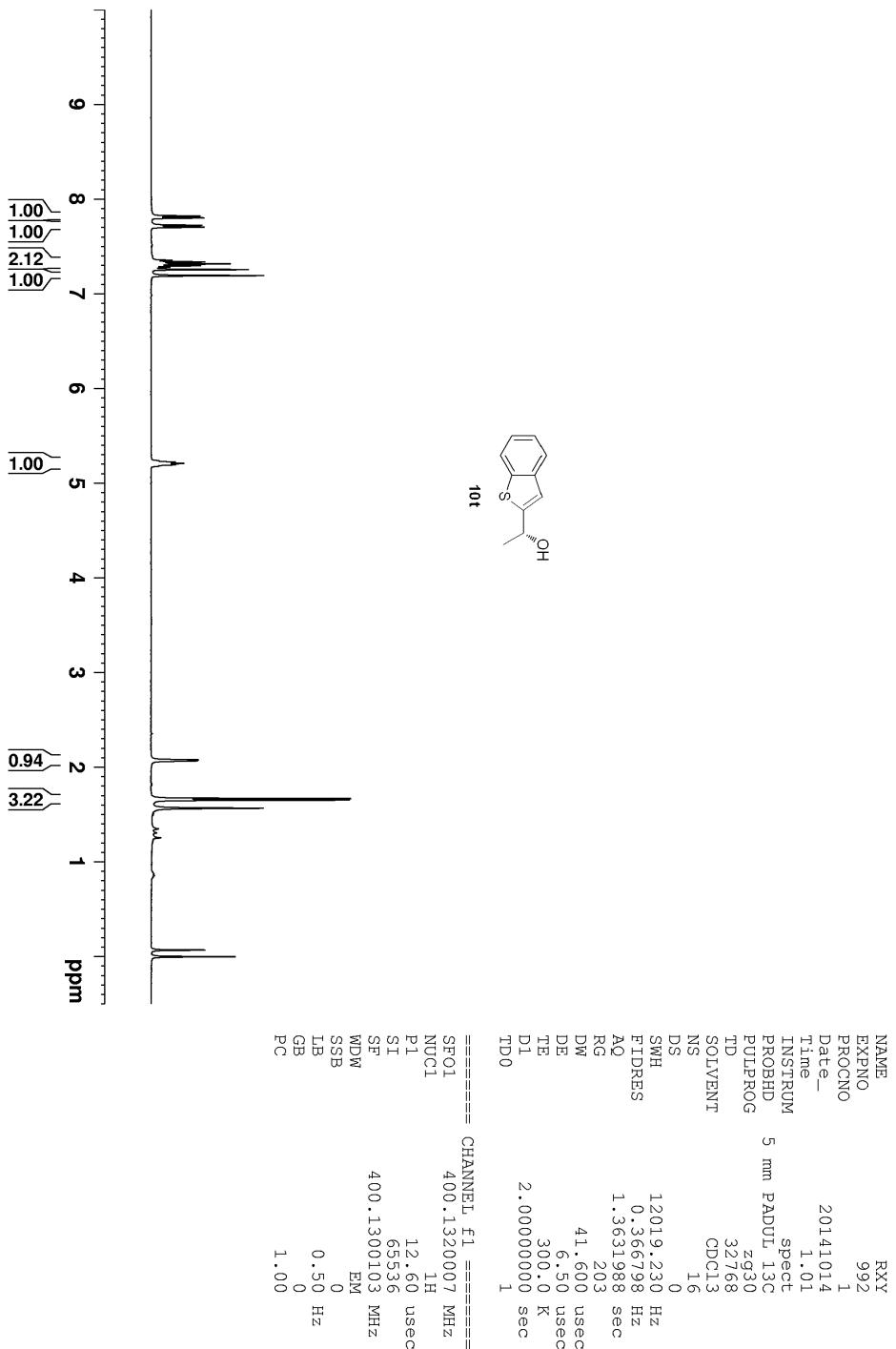


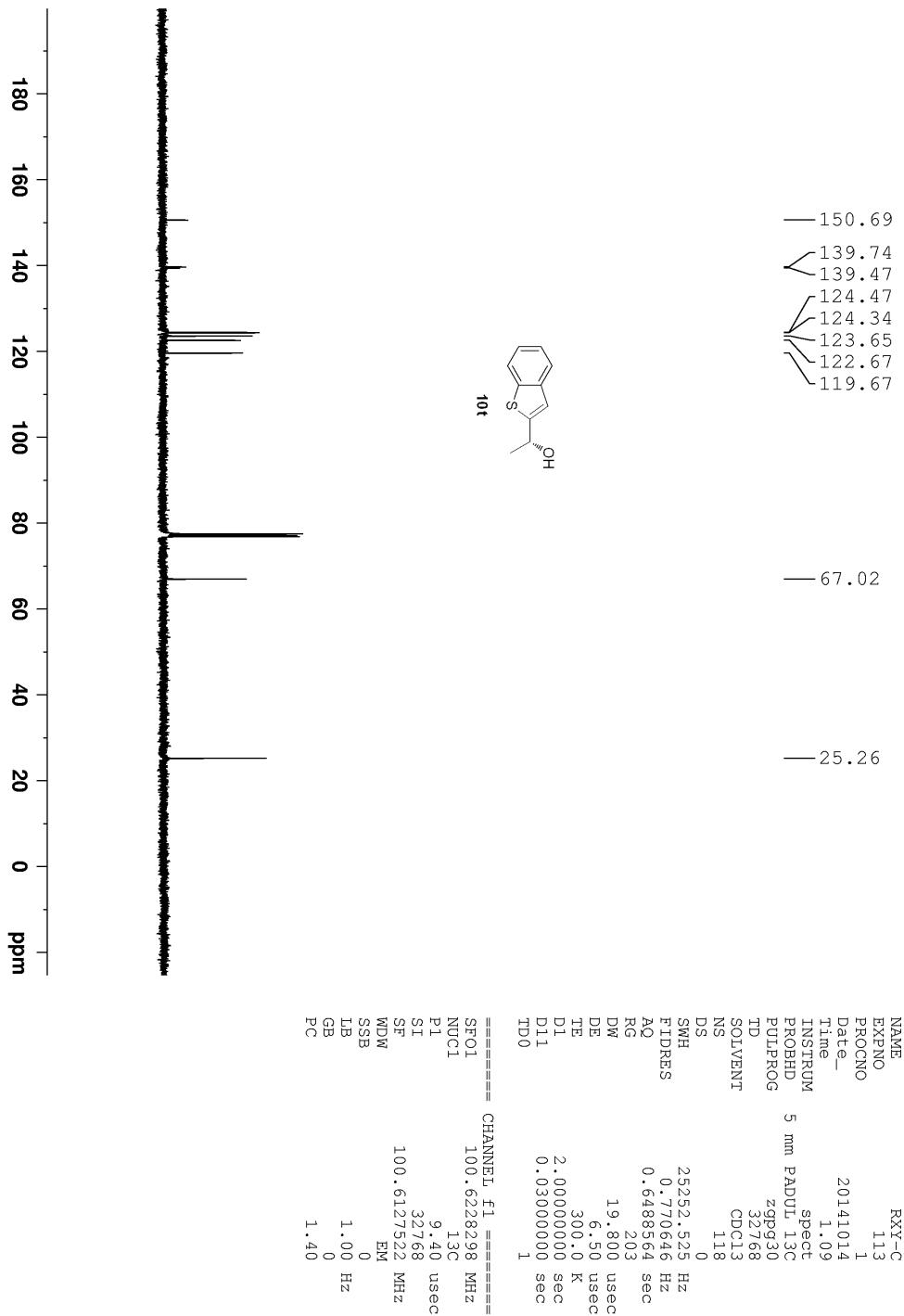
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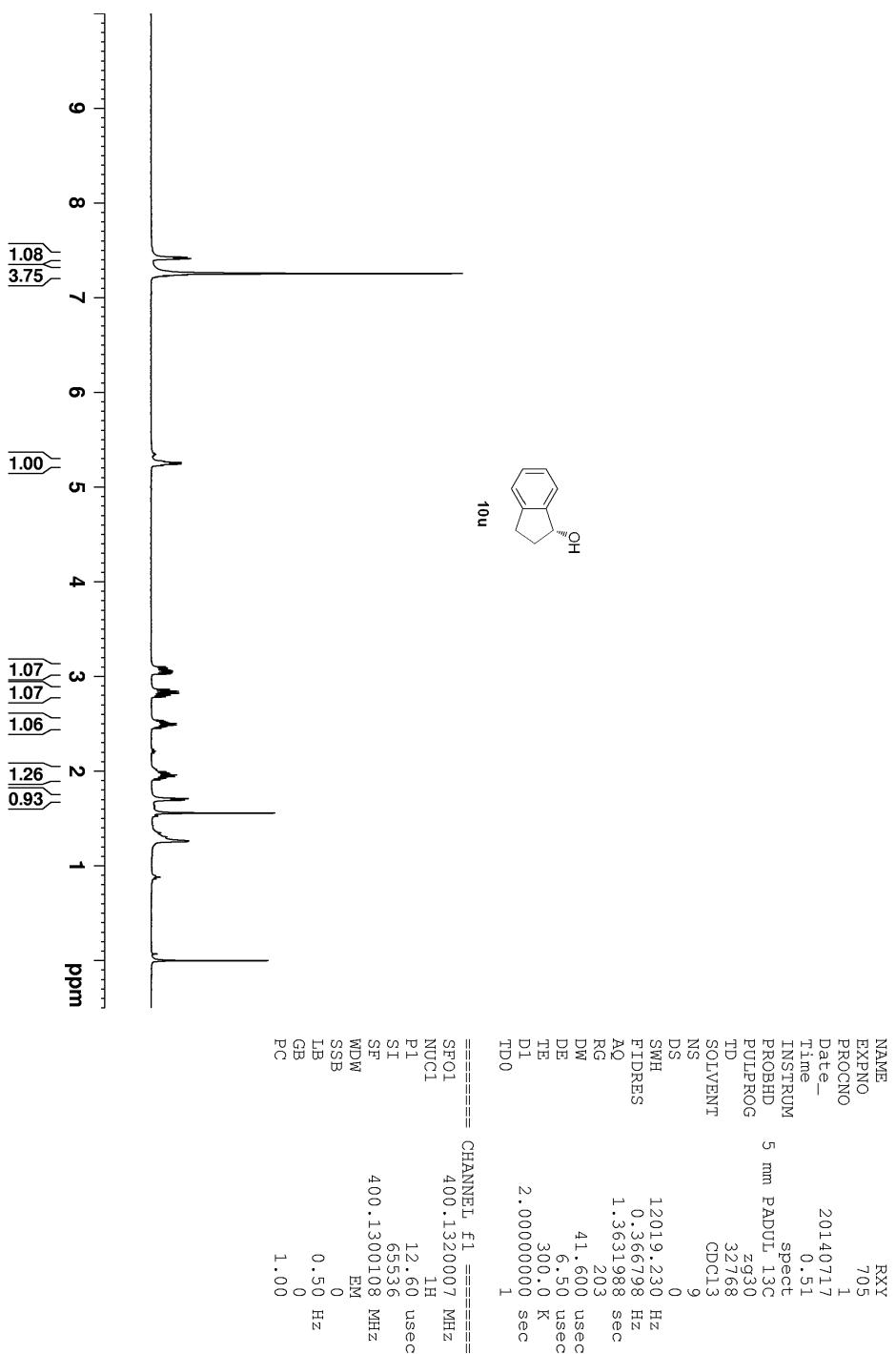
=====
NAME      RXY-C
EXPNO    101
PROCNO   1
Date_    20140922
Time     8.02
INSTRUM spect
PROBHD  5 mm PADUL 13C
PULPROG zgppg30
TD        32768
SOLVENT  CDCl3
NS       149
DS        0
SWH      25252.525 Hz
FIDRES  0.6488554 sec
AQ        203
RG        19.800 usec
DW        6.500 usec
DE        300.0 K
TE        2.0000000 sec
D1       0.03000000 sec
T1       1
TD0

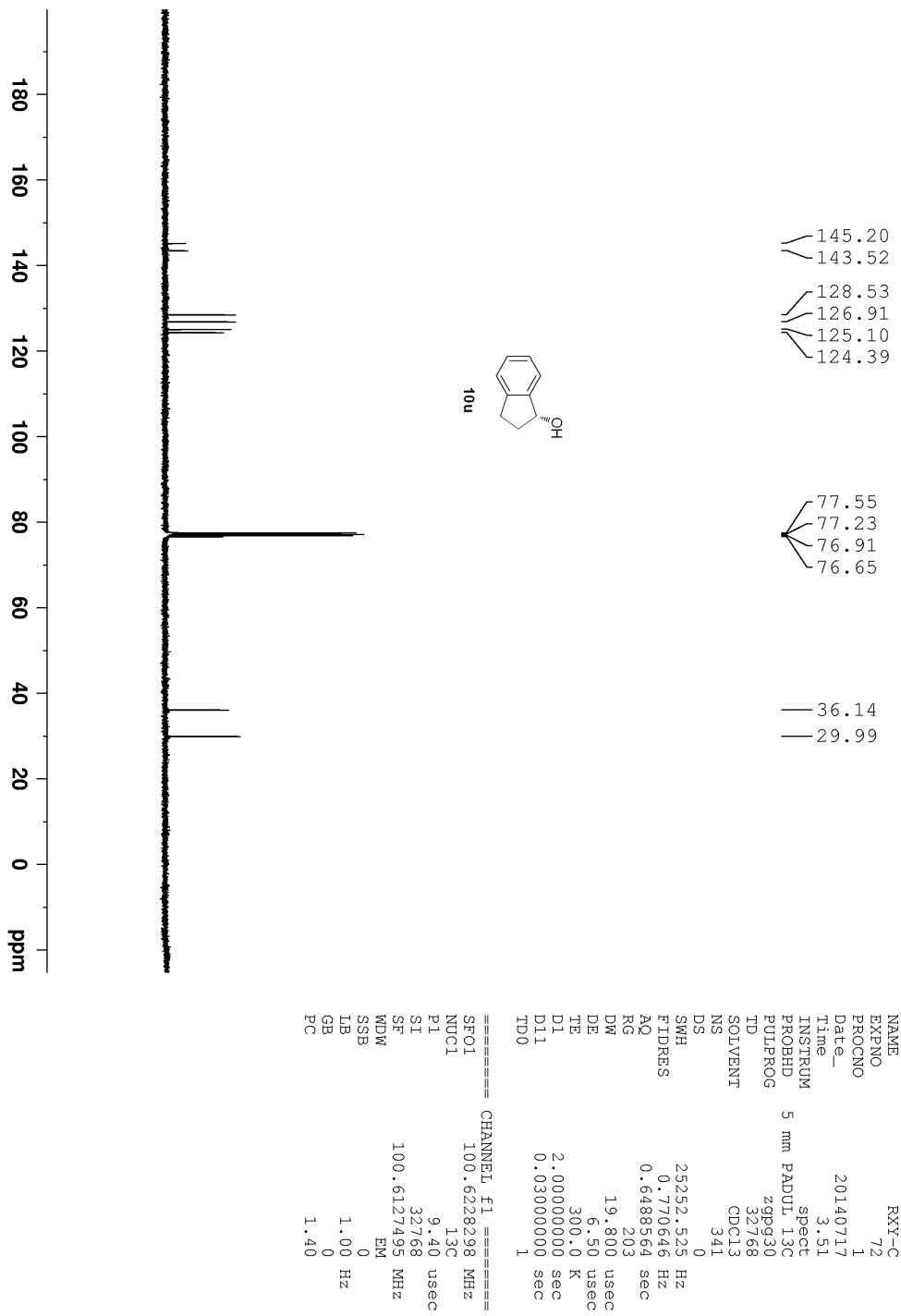
===== CHANNEL f1 =====
SF01    100.622828 MHz
NUC1    13C
P1      9.40 usec
SI      32768
SF      100.6127505 MHz
WDW
SSB
LB      1.00 Hz
GB      0
PC      1.40

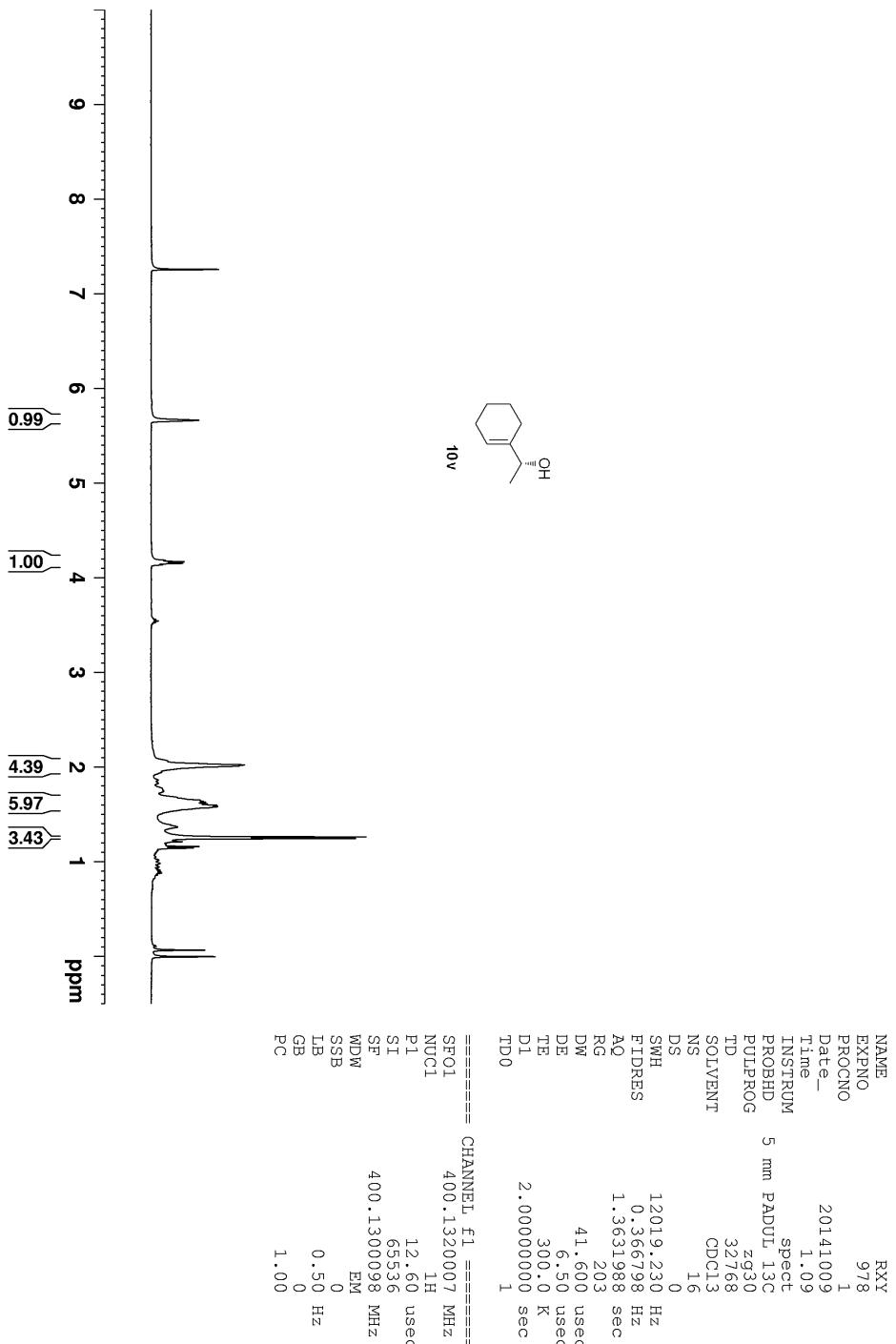
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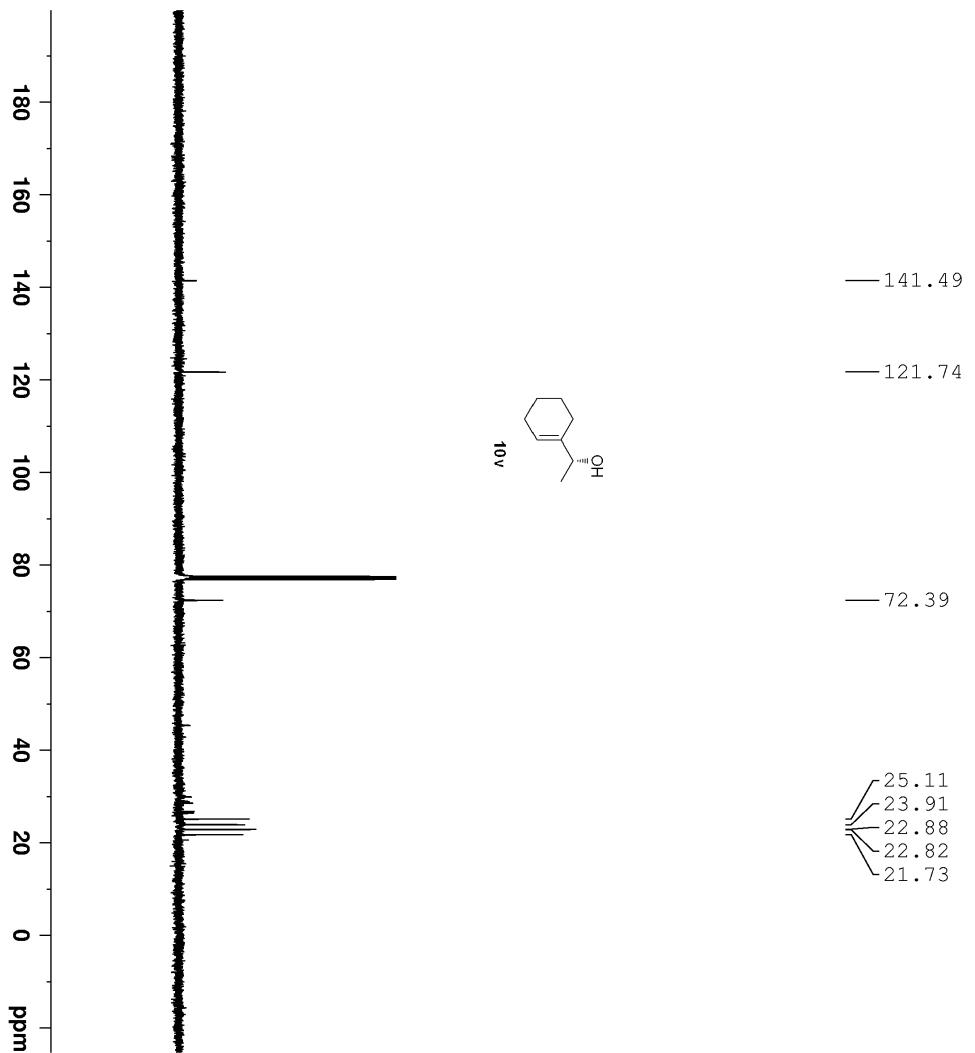












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=====
NAME          RXY-C
EXPNO        110
PROCNO       1
Date_        20141009
Time         1.20
INSTRUM     spect
PROBHD      5 mm PADUL 13C
PULPROG     zgpg30
TD           32768
SOLVENT      CDCl3
NS            277
DS             0
SWH          25252.525 Hz
FIDRES      0.6488554 sec
AQ            203
RG            19.800 usec
DE            6.500 usec
TE            300.0 K
TEUNIF       2.0000000 sec
D1           0.03000000 sec
T1           1
TD0          1

===== CHANNEL f1 =====
SF01        100.622828 MHz
NUC1         13C
P1           9.40 usec
SI            32768
SF          100.6127456 MHz
WDW
SSB
LB            1.00 Hz
GB            0
PC           1.40
  
```