Copper-Catalyzed Suzuki-Miyaura Coupling of Alkylboron Reagents: Disproportionation of Anionic (Alkyl)(Alkoxy)borates to Anionic Dialkylborates Prior to Transmetalation

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Table of Contents

1. General Information

Reactions were set up in a nitrogen-filled glovebox unless stated otherwise. All glassware were properly dried in an oven before use. Bulk solvents were obtained from EMD. Anhydrous solvents (DMF, DMSO, NMP, toluene, dioxane) were obtained from Sigma-Aldrich and were used directly without further purification. HMPA was dried over CaH_2 followed by distillation and stored under N_2 in 4 Å molecular sieves. Deuterated solvents were purchased from Cambridge Isotope. 9-BBN (0.50 M solution in THF), B-methoxy-9-BBN (1.0 M solution in hexanes) and B-Br-9-BBN (1.0 M Solution in CH_2Cl_2) were obtained in SureSeal bottles from Sigma-Aldrich. Aryl halides and olefins were purchased from Acros, Sigma-Aldrich, Oakwood, TCI-America, Matrix and Alfa-Aesar. CuI (99.999%) was procured from Sigma-Aldrich. *o*-Allyloxyiodobenzene was synthesized following a literature procedure.[1] ^{1}H, ^{13}C, ^{19}F and ^{11}B NMR spectra were recorded on a Bruker instrument (300, 75, 282, and 96 MHz, respectively) and internally referenced to the residual solvent signals of $CDCl_3$ for ^{1}H and ^{13}C NMR at 7.26 and 77.16 ppm, respectively, C_6F_6 for ^{19}F NMR at −164.9 ppm, and boric acid for ^{11}B NMR at 36.0 ppm. NMR chemical shifts and the coupling constants (*J*) for ^{1}H, ^{13}C, ^{19}F and ^{11}B NMR are reported in parts per million (ppm) and in Hertz, respectively. The following conventions are used for multiplicities: s, singlet; d, doublet; t, triplet; m, multiplet; dd, doublet of doublet. High resolution mass and NMR spectra of new compounds were recorded at the Mass Spectrometry and NMR Facilities, Department of Chemistry and Chemical Biology, University of New Mexico (UNM). X-ray diffraction was performed on Bruker Kappa APEX II CCD diffractometer at the Department of Chemistry and Chemical Biology, UNM.

2. Experimental Section

2.1. Generation of B-alkyl-9-BBN Reagents

In an oven-dried 15 mL pressure tube, olefin (1.5 mmol) was added to a solution of 9-BBN in THF (3 mL, 0.5 M in THF). The pressure tube was tightly capped and heated at 60 °C for 2 hours. The reaction mixture was then cooled to room temperature and the

[1] Molander, G. A.; Harring, L. S. *J. Org. Chem.* **1990**, *55*, 6171

solvent was removed under vacuum. The B-alkyl-9-BBN generated *in situ* was directly used for subsequent reactions without further purification.

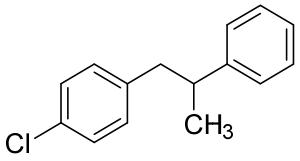
2.2. Procedure for Screening Reaction Conditions

B-(2-phenylpropyl)-9-BBN (36.0 mg, 0.15 mmol), 1-chloro-4-iodobenzene (23.8 mg, 0.10 mmol), Li*O*t*Bu (12 mg, 0.15 mmol) or other bases (0.15 mmol), and CuI (1.9 mg, 0.010 mmol) were weighed in a 1-dram vial and dissolved in HMPA or other solvents (0.5 mL). The vial was then tightly capped and placed in a hotplate pre-heated to 80 °C with vigorous stirring. After 48 h, the reaction mixture was cooled to room temperature, 20 μL of pyrene (0.010 mmol, 0.5 M stock solution) as an internal standard was added, diluted with EtOAc (1 mL) and filtered through a short pad of silica gel in a pipette. The reaction mixture was then analyzed by GC.

2.3. General Procedure for Tables 2 and 3

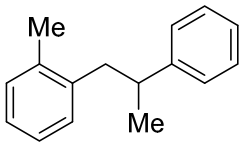
In an oven-dried 15 mL pressure tube, B-alkyl-9-BBN reagent (1.5 – 2.0 mmol), aryl iodide (1.0 mmol), Li*O*t*Bu (120.0 mg, 1.5 mmol) or K_3PO_4 (636 mg, 3 mmol) with 20 μL water and CuI (19 mg, 0.10 mmol) were weighed and dissolved in HMPA or DMF (5 mL). The pressure vessel was then tightly capped and placed in an oil bath pre-heated to 60 – 120 °C with vigorous stirring. After 12-48 h, the reaction mixture was cooled to room temperature, diluted with ethyl acetate (15 mL) and washed with H_2O (5 mL × 3). The aqueous fraction was extracted back with ethyl acetate (5 mL × 3) and combined with the first ethyl acetate fraction. The combined ethyl acetate fraction was dried over Na_2SO_4 and the solvent was removed in a rotary evaporator. The non-heterocyclic products were purified by silica gel column chromatography using hexanes as an eluting solvent. Heterocyclic products were purified by silica gel column chromatography using 10–20% ethyl acetate/hexanes.

2.4. Characterization data for compounds



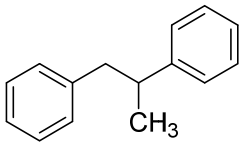
3

*1-Chloro-4-(2-phenylpropyl)benzene (**3**)*:[2] Reaction was conducted in 10.0 mmol scale in HMPA at 120 °C for 48 h with 1.5 equiv of alkylboron reagent using LiO*t*Bu as a base. The title compound **3** was obtained as yellow oil (1684 mg, 73%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.28 (d, *J* = 6.0 Hz, 3H), 2.76-2.83 (m, 1H), 2.89-3.06 (m, 2H), 7.01 (dd, *J*= 6.0 Hz, 3.0 Hz, 2H), 7.17-7.24 (m, 5H), 7.29-7.34 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.3, 41.9, 44.5, 126.3, 127.2, 128.3, 128.5, 130.6, 131.7, 139.3, 146.5; GCMS (m/z) 230.1.



5

*1-Methyl-2-(2-phenylpropyl)benzene (**5**)*: Reaction was conducted in 5.0 mmol scale in HMPA at 120 °C for 48 h with 1.5 equiv of alkylboron reagent using LiO*t*Bu as a base. The title compound **5** was obtained as colorless oil (747 mg, 71%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.33 (d, *J* = 6.0 Hz, 3H), 2.32 (s, 3H), 2.80-2.87 (m, 1H), 2.95-3.08 (m, 2H), 7.05-7.18 (m, 4H), 7.22-7.26 (m, 3H), 7.31-7.35 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 19.6, 21.2, 40.8, 42.5, 125.7, 126.1, 126.2, 127.1, 128.4, 130.2, 130.3, 136.3, 139.2, 147.3; IR (neat) cm^{-1} 2958, 1510, 1243, 1035; HRMS (TOF) Calcd for $C_{16}H_{18}$ (M^+) 210.1409, found 210.1416.

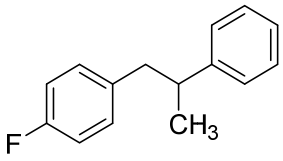


6

*Propane-1,2-diyldibenzene (**6**)*:[2] Reaction was conducted in 5.0 mmol scale in HMPA at 120 °C for 24 h with 1.5 equiv of alkylboron reagent using LiO*t*Bu as a base. The title compound **6** was obtained as yellow oil (716 mg, 73%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.29 (d, *J* = 6.0 Hz, 3H), 2.78-2.86 (m, 1H), 2.97-3.09 (m, 2H), 7.12-7.15 (m, 2H), 7.21-7.35 (m, 8H); ^{13}C NMR (75

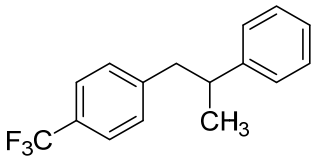
[2] Kita, Y.; Hida, S.; Higashihara, K.; Jena, H. S.; Higashida, K.; Mashima, K. *Angew. Chem. Int. Ed.* **2016**, *55*, 8299.

MHz, $CDCl_3$) δ 21.3, 42.0, 45.2, 126.0, 126.1, 127.2, 128.2, 128.4, 129.3, 140.9, 147.1; GCMS (m/z) 196.1.



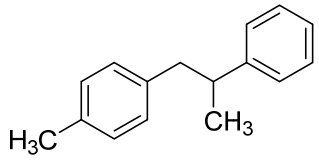
7

*1-Fluoro-4-(2-phenylpropyl)benzene (**7**)*: Reaction was conducted in 5.0 mmol scale in HMPA at 120 °C for 48 h with 1.5 equiv of alkylboron reagent using LiO*t*Bu as a base. The title compound **7** was obtained as yellow oil (621 mg, 58%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.26 (d, J = 6.0 Hz, 3H), 2.74-2.81 (m, 1H), 2.87-3.01 (m, 2H), 6.89-6.94 (m, 2H), 6.99-7.03 (m, 2H), 7.15-7.22 (m, 3H), 7.26-7.31 (m, 2H); ^{13}C NMR (75 MHz,$CDCl_3$) δ 21.3, 42.1, 44.3, 114.9 (d, J_{CF} = 79.0 Hz), 126.2, 127.2, 128.5, 130.6 (d, J_{CF} = 28.2 Hz), 136.5 (d, J_{CF} = 11.3 Hz), 146.7, 159.8, 163.1; ^{19}F NMR (282 MHz, $CDCl_3$) δ -116.1; IR (neat) cm^{-1} 2930, 1602, 1508, 1452, 1219, 1157, 1014; HRMS (TOF) Calcd for $C_{15}H_{15}F$ (M^+) 214.1158, found 214.1148.



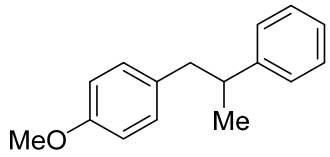
8

*1-(2-Phenylpropyl)-4-(trifluoromethyl)benzene (**8**)*: Reaction was conducted in HMPA at 120 °C for 48 h with 1.5 equiv of alkylboron reagent using LiO*t*Bu as a base. The title compound **8** was obtained as colorless oil (214 mg, 81%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.27 (d, J = 6.0 Hz, 3H), 2.81-2.91 (m, 1H), 2.95-3.06 (m, 2H), 7.15-7.23 (m, 5H), 7.26-7.32 (m, 2H), 7.48 (d, J = 6.0 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.4, 41.8, 44.9, 125.1 (q, J_{CF} = 16.9 Hz), 126.4, 127.1, 128.6, 129.5, 145.0, 146.3; ^{19}F NMR (282 MHz, $CDCl_3$) δ -60.7; IR (neat) cm^{-1} 2948, 1322, 1112, 1066; HRMS (TOF) Calcd for $C_{16}H_{15}F_3$ (M^+) 264.1126, found 264.1139.



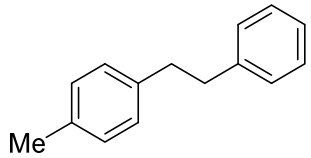
9

*1-Methyl-4-(2-phenylpropyl)benzene (**9**)*: Reaction was conducted in HMPA at 120 °C for 48 h with 1.5 equiv of alkylboron reagent using LiO*t*Bu as a base. The title compound **9** was obtained as yellow oil (164 mg, 78%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.26 (d, *J* = 6.0 Hz, 3H), 2.33 (s, 3H), 2.71-2.81 (m, 1H), 2.92-3.07 (m, 2H), 7.00-7.09 (m, 4H), 7.18-7.24 (m, 3H), 7.28-7.34 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.2, 21.3, 42.0, 44.7, 126.1, 127.2, 128.4, 128.9, 129.2, 135.4, 137.9, 147.3; IR (neat) cm^{-1} 2921, 1515, 1451; HRMS (TOF) Calcd for $C_{16}H_{18}$ (M^+) 210.1409, found 210.1408.



10

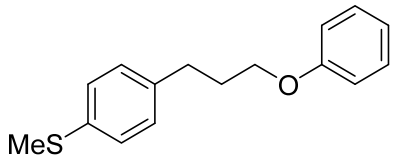
*1-Methoxy-4-(2-phenylpropyl)benzene (**10**)*:[3] Reaction was conducted in HMPA at 120 °C for 48 h with 2.0 equiv of alkylboron reagent using LiO*t*Bu as a base. The title compound **10** was obtained as yellow oil (179 mg, 79%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.26 (d, *J* = 6.0 Hz, 3H), 2.70-2.77 (m, 1H), 2.88-3.04 (m, 2H), 3.79(s, 3H), 6.81 (d, *J* = 9.0 Hz, 2H), 7.1 (d, *J* = 6.0 Hz, 2H), 7.18-7.22 (m, 3H), 7.27-7.33 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.2, 42.2, 44.3, 55.3, 113.6, 126.1, 127.2, 128.4, 130.2, 133.0, 147.2, 157.9; HRMS (APCI) Calcd for $C_{16}H_{19}O$ $(MH)^+$ 227.1436, found 227.1431.



11

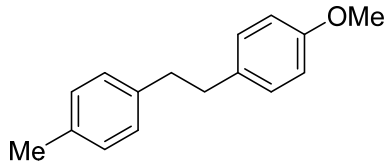
[3] Too, P. C.; Chan, G. H.; Tnay, Y. L.; Hirao, H.; Chiba, S. *Angew. Chem. Int. Ed.* **2016**, *55*, 3719.

*1-Methyl-4-phenethylbenzene (**11**)*:[4] Reaction was conducted in HMPA at 120 °C for 48 h with 2.0 equiv of alkylboron reagent using LiO*t*Bu as a base. The title compound **11** was obtained as yellow oil (100 mg, 51%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.36 (s, 3H), 2.93, (s, 4H), 7.13 (s, 4H), 7.21-7.25 (m, 3H), 7.30-7.35 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.2, 37.7, 38.2, 126.0, 128.4, 128.6, 129.2, 135.5, 138.9, 142.1; GCMS (m/z) 196.1.



12

Methyl(4-(3-phenoxypropyl)phenyl)sulfane ***(12)***: Reaction was conducted in HMPA at 120 °C for 48 h with 1.5 equiv of alkylboron reagent using K_3PO_4 as a base. The title compound **12** was obtained as white solid (173 mg, 67%) after purification by silica gel column chromatography.^{1}H NMR (300 MHz, $CDCl_3$) δ 2.05-2.14 (m, 2H), 2.48 (s, 3H), 2.80 (t, $J = 7.5$ Hz, 2H), 3.97 (t, $J = 6.0$ Hz , 2H), 6.90-6.98 (m, 3H), 7.14-7.33 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 16.4, 31.0, 31.7, 66.7, 114.6, 120.7, 127.3, 129.2, 129.6, 135.6, 138.8, 159.1; IR (neat) cm^{-1} 2918, 1489, 1238, 1174, 1042; HRMS (ESI) Calcd for $C_{16}H_{18}NaOS$ $(MNa)^+$ 281.0976, found 281.0974.

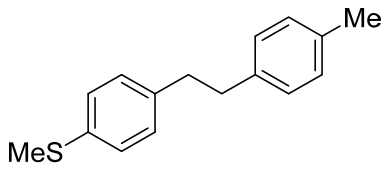


13

1-Methoxy-4-(4-methylphenethyl)benzene ***(13)***: Reaction was conducted in HMPA at 120°C for 48 h with 1.5 equiv of alkylboron reagent using LiO*t*Bu as a base. The title compound **13** was obtained as yellow oil (104 mg, 46%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.32 (s, 3H), 2.85 (s, 4H), 3.79 (s, 3H), 6.80-6.85 (m, 2H), 7.08 (s, 5H), 7.12 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.2, 37.3, 37.9, 55.4, 113.9, 128.5, 129.1, 129.5, 134.2, 135.4, 138.9, 157.9; IR (neat) cm^{-1}

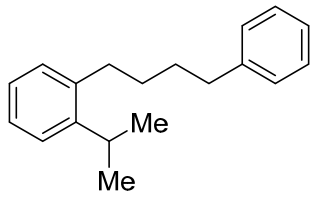
[4] Kantam, M. L.; Chakravarti, R.; Chintareddy, V. R.; Sreedhar, B.; Bhargava, S. *Adv. Syn. Catal.* **2008**, *350*, 2544.

2918, 1509, 1241, 1030; HRMS (APCI) Calcd for $C_{16}H_{19}O$ $(MH)^+$ 227.1436, found 227.1438.



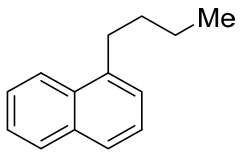
14

*Methyl(4-(4-methylphenethyl)phenyl)sulfane (**14**)*: Reaction was conducted in HMPA at 120 °C for 48 h with 2.0 equiv of alkylboron reagent using LiO*t*Bu as a base. The title compound **14** was obtained as yellow oil (172 mg, 71%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.38 (s, 3H), 2.52 (s, 3H), 2.91 (s, 4H), 7.13 (s, 4H), 7.17 (s, 2H), 7.23 (s, 1H), 7.26 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 16.4, 21.2, 37.5, 37.6, 127.2, 128.4, 129.1, 135.5, 138.6, 139.1; IR (neat) cm^{-1} 2916, 2850, 1419, 1091; HRMS (APCI) Calcd for $C_{16}H_{19}S$ $(MH)^+$ 243.1207, found 243.1212.



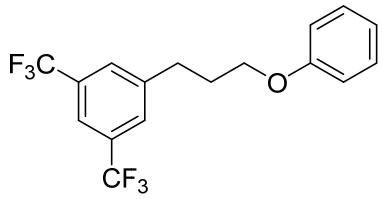
15

*1-Isopropyl-2-(4-phenylbutyl)benzene (**15**)*: Reaction was conducted in HMPA at 120 °C for 48 h with 1.5 equiv of alkylboron reagent using LiO*t*Bu as a base. The title compound **15** was obtained as yellow oil (103 mg, 41%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.22 (dd, J = 6.0 Hz, 3.0 Hz, 6H), 1.58-1.76 (m, 4H), 2.67 (t, J = 7.5 Hz, 4H), 3.09-3.19 (m, 1H), 7.11-7.30 (m, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 24.2, 28.7, 31.5, 31.6, 33.0, 36.0, 125.4, 125.6, 125.8, 126.3, 128.4, 128.6, 129.5, 139.4, 142.7, 146.6; IR (neat) cm^{-1} 2929, 1489, 1453, 1032; HRMS (TOF) Calcd for $C_{19}H_{24}$ (M^+) 252.1878, found 252.1898.



16

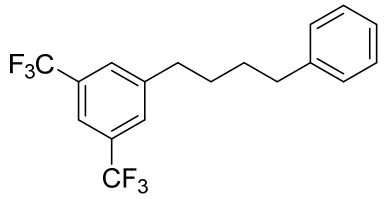
*1-Butylnaphthalene (**16**)*:[5] Reaction was conducted in HMPA at 120 °C for 48 h with 1.5 equiv of alkylboron reagent using K_3PO_4 as a base. The title compound **16** was obtained as yellow oil (108 mg, 59%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.99 (t, *J* = 7.5 Hz, 3H), 1.42-1.54 (m, 2H), 1.71-1.81 (m, 2H), 3.09 (t, *J* = 7.5 Hz, 2H), 7.33-7.43 (m, 2H), 7.47-7.53 (m, 2H), 7.72 (d, *J* = 9.0 Hz, 1H), 7.85-7.88 (m, 1H), 8.05-8.09 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 14.2, 23.0, 33.0, 33.2, 124.1, 125.5, 125.7, 125.8, 126.0, 126.5, 128.9, 132.1, 134.0, 139.1; HRMS (APPI) Calcd for $C_{14}H_{16}$ $(M)^+$ 184.1252, found 184.1259.



17

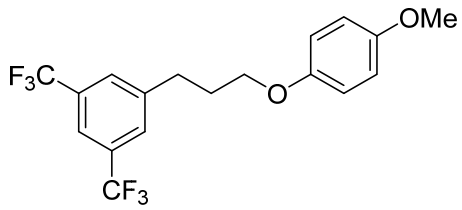
*1-(3-Phenoxypropyl)-3,5-bis(trifluoromethyl)benzene (**17**)*: Reaction was conducted in HMPA at 80 °C for 36 h with 1.5 equiv of alkylboron reagent using K_3PO_4 as a base. The title compound **17** was obtained as yellow oil (281 mg, 81%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.11-2.20 (m, 2H), 2.98 (t, *J* = 7.5 Hz, 2H), 4.01 (t, *J* = 6.0 Hz, 2H), 6.88-6.99 (m, 3H), 7.27-7.33 (m, 2H), 7.68 (s, 2H), 7.73 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 30.7, 32.3, 66.5, 114.6, 120.2 (t, J_{CF} = 14.1 Hz), 121.1, 121.8, 125.4, 128.8, 129.7, 131.6 (q, J_{CF} = 124.1 Hz), 144.2, 158.9; ^{19}F NMR (282 MHz, $CDCl_3$) δ -61.3; IR (neat) cm^{-1} 2925, 1601, 1275, 1124; HRMS (APPI) Calcd for $C_{17}H_{14}F_6O$ $(M)^+$ 348.0949, found 348.0948.

[5] Agrawal, T.; Cook, S. P. *Org. Lett.* **2013**, *15*, 96.



18

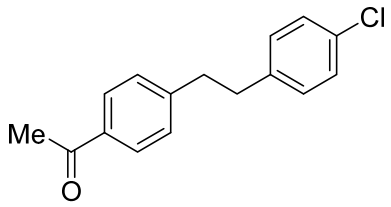
*1-(4-Phenylbutyl)-3,5-bis(trifluoromethyl)benzene (**18**)*:[6] Reaction was conducted in HMPA at 80 °C for 48 h with 1.5 equiv of alkylboron reagent using K_3PO_4 as a base. The title compound **18** was obtained as yellow oil (273 mg, 79%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.70-1.75 (m, 4H), 2.69 (t, *J* = 6.0 Hz, 2H), 2.78 (t, *J* = 7.5 Hz, 2H), 7.19-7.24 (m, 3H), 7.29-7.35 (m, 2H), 7.63 (s, 2H), 7.73 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 30.7, 31.0, 35.6, 35.8, 120.0 (t, J_{CF} = 14.1 Hz), 121.8, 125.4, 126.0, 128.5, 128.6, 131.5 (q, J_{CF} = 124.1 Hz), 142.2, 145.0; ^{19}F NMR (282 MHz, $CDCl_3$) δ -61.3; HRMS (APCI) Calcd for $C_{18}H_{16}F_6$ $(M)^+$ 346.1156, found 346.1157.



19

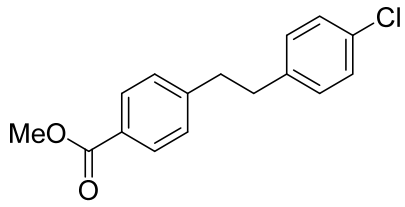
*1-(3-(4-Methoxyphenoxy)propyl)-3,5-bis(trifluoromethyl)benzene (**19**)*: Reaction was conducted in HMPA at 80 °C for 48 h with 1.5 equiv of alkylboron reagent using K_3PO_4 as a base. The title compound **19** was obtained as colorless oil (310 mg, 82%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.08-2.17 (m, 2H), 2.96 (t, *J* = 7.5 Hz, 2H), 3.78 (s, 3H), 3.95 (t, *J* = 6.0 Hz, 2H), 6.84 (s, 4H), 7.67 (s, 2H), 7.73 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 30.8, 32.3, 55.9, 67.3, 114.9, 115.6, 120.2 (br d, J_{CF} = 16.9 Hz), 125.4, 128.8, 131.8 (q, J_{CF} = 126.9 Hz), 144.3, 153.0, 154.1; ^{19}F NMR (282 MHz, $CDCl_3$) δ −61.2; IR (neat) cm^{-1} 2927, 1508, 1276, 1126; HRMS (ESI) Calcd for $C_{18}H_{17}F_6O_2$ $(MH)^+$ 379.1133, found 379.1141.

[6] Bair, J. S.; Schramm, Y.; Sergeev, A. G.; Clot, E.; Eisenstein, O.; Hartwig, J. F. *J. Am. Chem. Soc.* **2014**, *136*, 13098.



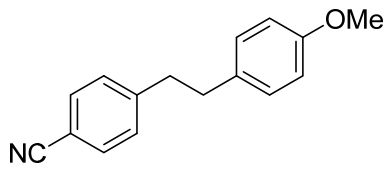
20

*1-(4-(4-Chlorophenethyl)phenyl)ethan-1-one (**20**)*: Reaction was conducted in HMPA at 100°C for 24 h with 1.5 equiv of alkylboron reagent using K_3PO_4 as a base. The title compound **20** was obtained as light yellow solid (111 mg, 43%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.57 (s, 3H), 2.86-2.98 (m, 4H), 7.05 (d, J = 9.0 Hz, 2H), 7.20-7.24 (m, 4H), 7.87 (d, J= 6.0 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 26.6, 36.7, 37.7, 128.5, 128.6, 128.8, 129.9, 131.9, 135.3, 139.5, 147.0, 197.8; IR (neat) cm^{-1} 2916, 1674, 1360, 1264, 1090; HRMS (ESI) Calcd for $C_{16}H_{16}{}^{35}ClO$ $(MH)^+$ 259.0890, found 259.0881.



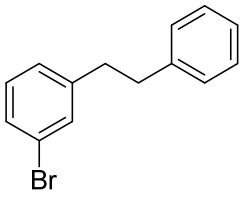
21

*Methyl 4-(4-chlorophenethyl)benzoate (**21**)*: Reaction was conducted in HMPA at 100 °C for 48 h with 1.5 equiv of alkylboron reagent using K_3PO_4 as a base. The title compound **21** was obtained as white solid (140 mg, 51%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.88-2.97 (m, 4H), 3.90 (s, 3H), 7.05 (d, J = 9.0 Hz, 2H), 7.18-7.24 (m, 4H), 7.95 (d, J = 9.0 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 36.9, 37.9, 52.2, 128.2, 128.6, 128.7, 129.9, 130.0, 132.0, 139.6, 146.8, 167.2; IR (neat) cm^{-1} 2922, 1711, 1507, 1279, 1096; HRMS (ESI) Calcd for $C_{16}H_{16}{}^{35}ClO_2$ $(MH)^+$ 275.0839, found 275.0834.



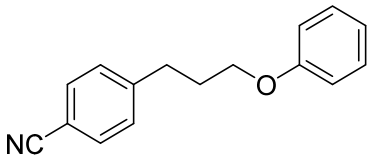
22

*4-(4-Methoxyphenethyl)benzonitrile (**22**)*:[7] Reaction was conducted in HMPA at 100 °C for 48 h with 1.5 equiv of alkylboron reagent using K_3PO_4 as a base. The title compound **22** was obtained as yellow oil (182 mg, 77%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.83-2.98 (m, 4H), 3.79 (s, 3H), 6.79-6.84 (m, 2H), 7.01-7.06 (m, 2H), 7.21 (s, 1H), 7.24 (s, 1H), 7.53-7.56 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 36.5, 38.3, 55.4, 109.9, 113.9, 119.2, 129.5, 132.2, 132.8, 147.4, 158.1; HRMS (ESI) Calcd for $C_{16}H_{16}NO$ $(MH)^+$ 238.1232, found 238.1227.



23

*1-Bromo-3-phenethylbenzene (**23**)*:[8] Reaction was conducted in HMPA at 100 °C for 48 h with 1.5 equiv of alkylboron reagent using K_3PO_4 as a base. The title compound **23** was obtained as yellow oil (184 mg, 71%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.92 (s, 4H), 7.09-7.26 (m, 5H), 7.29-7.37 (m, 4H); ^{13}C NMR (75 MHz, CDCl3) δ 37.7, 37.8, 122.5, 126.2, 127.3, 128.5, 129.2, 130.0, 131.6, 141.3, 144.2; GCMS (m/z) 260.0.

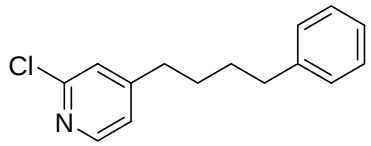


24

*4-(3-Phenoxypropyl)benzonitrile (**24**)*: Reaction was conducted in HMPA at 100 °C for 48 h with 1.5 equiv of alkylboron reagent using K_3PO_4 as a base. The title compound **24** was obtained as colorless oil (211 mg, 89%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.06-2.17 (m, 2H), 2.89 (t, J = 7.5 Hz, 2H), 3.96 (t, J = 6.0 Hz, 2H), 6.8 (d, J = 6.0 Hz, 2H), 6.95 (t, J = 7.5 Hz, 1H), 7.27-7.33

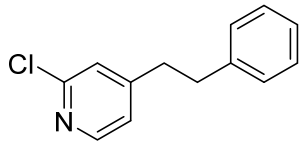
[7] Molander, G. A.; Sandrock, D. L. *Org. Lett.* **2009**, *11*, 2369.
[8] Gole, B.; Sanyal, U.; Banerjee, R.; Mukherjee, P. S. *Inorg. Chem.* **2016**, *55*, 2345.

(m, 4H), 7.58 (d, J = 9.0 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 30.5, 32.6, 66.4, 110.0, 114.6, 119.2, 120.9, 129.5, 129.6, 132.4, 147.4, 158.9; IR (neat) cm^{-1} 2928, 2227, 1733, 1600, 1496, 1241, 1042; HRMS (ESI) Calcd for $C_{16}H_{16}NO$ $(MH)^+$ 238.1232, found 238.1226.



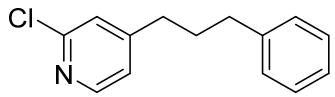
25

*2-Chloro-4-(4-phenylbutyl)pyridine (**25**)*: Reaction was conducted in HMPA at 80 °C for 24 h with 1.5 equiv of alkylboron reagent using K_3PO_4 as a base. The title compound **25** was obtained as yellow oil (174 mg, 71%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.64-1.69 (m, 4H), 2.58-2.68 (m, 4H), 7.00 (d, J = 6.0 Hz, 1H), 7.13-7.22 (m, 4H), 7.26-7.31 (m, 2H), 8.25 (d, J = 6.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 29.7, 30.9, 34.9, 35.7, 122.8, 124.2, 126.0, 128.5, 142.0 149.5, 151.7, 154.9; IR (neat) cm^{-1} 2932, 1591, 1545, 1385, 1085; HRMS (ESI) Calcd for $C_{15}H_{17}{}^{35}ClN$ $(MH)^+$ 246.1050, found 246.1046.



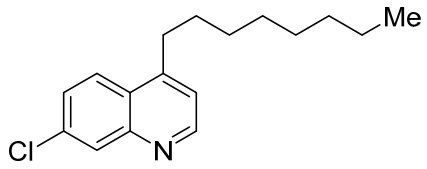
26

*2-Chloro-4-phenethylpyridine (**26**)*: Reaction was conducted in HMPA at 100 °C for 48 h with 1.5 equiv of alkylboron reagent using K_3PO_4 as a base. The title compound **26** was obtained as colorless solid (172 mg, 79%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.92 (s, 4H), 6.99 (dd, J = 6.0 Hz, 3.0 Hz, 1H), 7.12-7.15 (m, 3H), 7.19-7.32 (m, 3H), 8.25 (d, J = 6.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 36.5, 36.9, 122.9, 124.4, 126.6, 128.5, 128.7, 140.3, 149.6, 151.8, 154.0; IR (neat) cm^{-1} 2932, 1591, 1546, 1385, 1085; HRMS (ESI) Calcd for $C_{13}H_{13}{}^{35}ClN$ $(MH)^+$ 218.0737, found 218.0739.



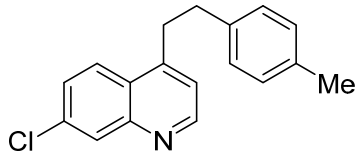
27

*2-Chloro-4-(3-phenylpropyl)pyridine (**27**)*: Reaction was conducted in HMPA at 80 °C for 48 h with 1.5 equiv of alkylboron reagent using K_3PO_4 as a base. The title compound **27** was obtained as yellow oil (169 mg, 73%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.91-2.02 (m, 2H), 2.60-2.69 (m, 4H), 7.03 (dd, *J* = 6.0 Hz, 3.0 Hz, 1H), 7.15-7.23 (m, 4H), 7.27-7.33 (m, 2H), 8.26 (d, *J* = 6.0 Hz, 1H); ^{13}C NMR (75 MHz, CDCl3) δ 31.6, 34.4, 35.3, 122.8, 124.3, 126.2, 128.5, 128.6, 141.4, 149.6, 151.7, 154.7; IR (neat) cm^{-1} 2931, 1591, 1545, 1385, 1085; HRMS (ESI) Calcd for $C_{14}H_{15}{}^{35}ClN$ $(MH)^+$ 232.0893, found 232.0895.



28

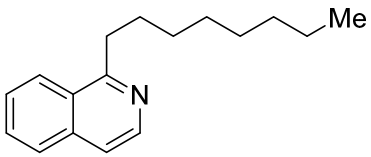
*7-Chloro-4-octylquinoline (**28**)*:[9] Reaction was conducted in HMPA at 80 °C for 24 h with 1.5 equiv of alkylboron reagent using K_3PO_4 as a base. The title compound **28** was obtained as yellow oil (242 mg, 88%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.88 (t, *J* = 7.5 Hz, 3H), 1.26-1.43 (m, 10H), 1.68-1.78 (m, 2H), 3.03 (t, *J* = 7.5 Hz, 2H), 7.22 (d, *J* = 3.0 Hz, 1H), 7.50 (dd, *J* = 9.0 Hz, 3.0 Hz, 1H), 7.97 (d, *J* = 9.0 Hz, 1H) 8.09 (s, 1H), 8.79 (d, *J* = 3.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 14.2, 22.8, 29.3, 29.5, 29.8, 30.2, 31.9, 32.2, 121.0, 125.1, 126.1, 127.2, 129.2, 134.9, 149.0, 151.3; HRMS (ESI) Calcd for $C_{17}H_{23}{}^{35}ClN$ $(MH)^+$ 276.1519, found 276.1515.



29

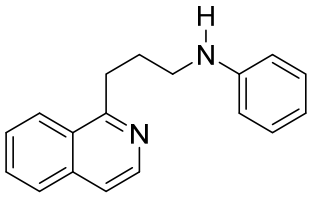
[9] Shrestha, B.; Thapa, S.; Gurung, S. K.; Pike, R. A. S.; Giri, R. *J. Org. Chem.* **2016**, *81*, 787.

*7-Chloro-4-(4-methylphenethyl)quinoline (**29**)*: Reaction was conducted in HMPA at 80 °C for 48 h with 1.5 equiv of alkylboron reagent using K_3PO_4 as a base. The title compound **29** was obtained as white solid (188 mg, 67%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.34 (s, 3H), 3.01 (t, *J* = 7.5 Hz, 2H), 3.34 (t, *J* = 7.5 Hz, 2H), 7.06-7.13 (m, 4H), 7.17 (d, *J* = 3.0 Hz, 1H), 7.51 (dd, *J* = 9.0 Hz, 3.0 Hz, 1H), 7.99 (d, *J* = 9.0 Hz, 1H), 8.13 (d, *J* = 3.0 Hz, 1H), 8.78 (d, *J* = 6.0 Hz, 1H); ^{13}C NMR (75 MHz,$CDCl_3$) δ 21.2, 34.2, 35.8, 121.2, 125.0, 126.0, 127.5, 128.3, 129.3, 129.4, 135.0, 136.1, 137.7, 147.8, 149.0, 151.4; IR (neat) cm^{-1} 2919, 1598, 1515, 1417, 1095; HRMS (ESI) Calcd for $C_{18}H_{17}{}^{35}ClN$ $(MH)^+$ 282.1050, found 282.1046.



30

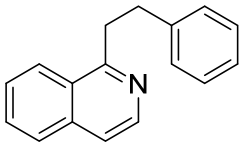
*1-Octylisoquinoline (**30**)*:[9] Reaction was conducted in HMPA at 80 °C for 24 h with 1.5 equiv of alkylboron reagent using K_3PO_4 as a base. The title compound **30** was obtained as yellow oil (205 mg, 85%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.87 (t, *J* = 6.0 Hz, 3H), 1.25-1.32 (m, 8H), 1.42-1.52 (m, 2H), 1.80-1.91 (m, 2H), 3.28 (t, *J* = 9.0 Hz, 2H), 7.47 (d, *J* = 6.0 Hz, 1H), 7.53-7.66 (m, 2H), 7.78 (d, *J* = 6.0 Hz, 1H), 8.15 (dd, *J* = 9.0 Hz, 3.0 Hz, 1H), 8.42 (d, *J* = 6.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 14.2, 22.8, 29.4, 29.6, 29.9, 30.0, 32.0, 35.7, 119.2, 125.5, 127.0, 127.5, 129.8, 136.4, 142.0, 162.6; HRMS (ESI) Calcd for $C_{17}H_{24}N$ $(MH)^+$ 242.1909, found 242.1907.



31

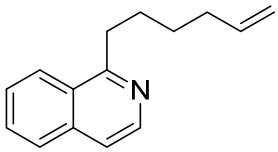
*N-(3-(isoquinolin-1-yl)propyl)aniline (**31**)*: Reaction was conducted in HMPA at 80 °C for 48 h with 1.5 equiv of alkylboron reagent using K_3PO_4 as a base. The title compound **31** was obtained as white solid (165 mg, 63%) after purification by silica gel column

chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.18-2.28 (m, 2H), 3.28 (t, *J* = 7.5 Hz, 2H), 3.43 (t, *J* = 7.5 Hz, 2H), 3.97 (s, 1H), 6.63 (d, *J* = 9.0 Hz, 2H), 6.69 (t, *J* = 7.5 Hz, 1H), 7.17 (t, *J* = 9.0 Hz, 2H), 7.52-7.60 (m, 2H), 7.64-7.70 (m, 1H), 7.83 (d, *J* = 9.0 Hz, 1H), 8.14 (d, *J* = 9.0 Hz, 1H), 8.45 (d, *J* = 6.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 28.7, 32.7, 43.7, 112.9, 117.2, 119.5, 125.2, 127.1, 127.2, 127.5, 129.3, 130.0, 136.3, 141.9, 148.5, 161.3; IR (neat) cm^{-1} 3735, 3628, 2924, 2308, 1457, 1010; HRMS (ESI) Calcd for $C_{18}H_{19}N_2$ $(MH)^+$ 263.1548, found 263.1547.



32

*1-Phenethylisoquinoline (**32**)*:[10] Reaction was conducted in HMPA at 80°C for 48 h with 1.5 equiv of alkylboron reagent using K_3PO_4 as a base. The title compound **32** was obtained as yellow oil (215 mg, 92%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.19-3.24 (m, 2H), 3.59-3.64 (m, 2H), 7.24 (t, *J* = 6.5 Hz, 1H), 7.33 (d, *J* = 6.0 Hz, 4H), 7.54 (d, *J* = 6.0 Hz, 1H), 7.60 (dd, *J*= 6.0 Hz, 3.0Hz, 1H), 7.65-7.70 (m, 1H), 7.83 (d, *J* = 6.0 Hz, 1H), 8.16 (d, *J* = 6.0 Hz, 1H), 8.49 (d, *J* = 6.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 35.6, 37.4, 119.5, 125.2, 126.2, 127.0, 127.2, 127.5, 128.5, 128.6,128.6, 129.9, 136.4, 142.0, 161.1; HRMS (ESI) Calcd for $C_{17}H_{16}N$ $(MH)^+$ 234.1283, found 234.1280.

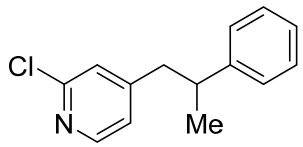


33

*1-(Hex-5-en-1-yl)isoquinoline (**33**)*:[11] Reaction was conducted in HMPA at 80 °C for 48 h with 1.5 equiv of alkylboron reagent using K_3PO_4 as a base. The title compound **33** was obtained as yellow oil (173 mg, 82%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.53-1.63 (m, 2H), 1.83-1.94 (m, 2H),

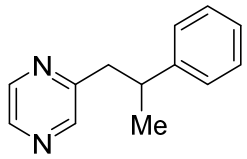
[10] Tang, R.-J.; Kang, L.; Yang, L. *Adv. Synth. Catal.* **2015**, *357*, 2055.
[11] Gisby, G. P.; Sammes, P. G.; Watt, R. A. *J. Chem. Soc., Perkin Trans. 1* **1982**, 249.

2.14 (q, *J* = 7.5 Hz, 2H), 3.30 (t, *J* = 7.5 Hz, 2H), 4.92-5.05 (m, 2H), 5.75-5.89 (m, 1H), 7.49 (d, *J* = 6.0 Hz, 1H), 7.55-7.67 (m, 2H), 7.80 (d, *J* = 9.0 Hz, 1H), 8.15 (d, *J* = 9.0 Hz, 1H), 8.43 (d, *J* = 6.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 29.2, 29.3, 33.8, 35.5, 114.6, 119.3, 125.4, 127.0, 127.5, 129.9, 136.4, 138.9, 142.0, 162.3; HRMS (ESI) Calcd for $C_{15}H_{18}N$ $(MH)^+$ 212.1439, found 212.1441.



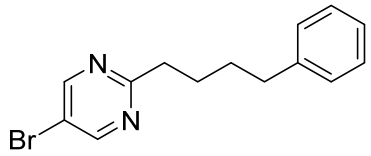
34

*2-Chloro-4-(2-phenylpropyl)pyridine (**34**)*: Reaction was conducted in HMPA at 100 °C for 24 h with 1.5 equiv of alkylboron reagent using K_3PO_4 as a base. The title compound **34** was obtained as colorless oil (164 mg, 71%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.29 (d, *J* = 6.0 Hz, 3H), 2.76-2.92 (m, 2H), 2.96-3.07 (m, 1H), 6.86 (dd, *J* = 6.0 Hz, 3.0 Hz, 1H), 7.02 (s, 1H), 7.10-7.14 (m, 2H), 7.17-7.23 (m, 1H), 7.25-7.31 (m, 2H), 8.19 (d, *J* = 6.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.6, 41.1, 44.0, 123.4, 124.9, 126.7, 127.0, 128.7, 145.3, 149.3, 151.5, 153.2; IR (neat) cm^{-1} 2963, 1591, 1385, 1086; HRMS (ESI) Calcd for $C_{14}H_{15}{}^{35}ClN$ $(MH)^+$ 232.0893, found 232.0888.



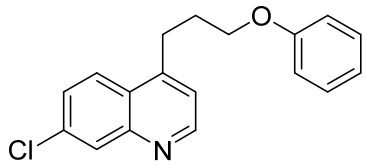
35

*2-(2-Phenylpropyl)pyrazine (**35**)*: Reaction was conducted in DMF at 80 °C for 24 h with 1.5 equiv of alkylboron reagent using K_3PO_4 as a base. The title compound **35** was obtained as yellow oil (137 mg, 69%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$)δ 1.31 (d, *J* = 6.0 Hz, 3H), 3.01-3.10 (m, 2H), 3.23-3.35 (m, 1H), 7.15-7.19 (m, 3H), 7.24-7.29 (m, 2H), 8.20 (s, 1H), 8.35 (d, *J* = 2.4 Hz, 1H), 8.49 (t, *J* = 3.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.6, 40.3, 44.3, 126.5, 127.0, 128.6, 142.3, 144.2, 145.3, 145.9, 156.4; IR (neat) cm^{-1} 2923, 2854, 1454, 1403, 1017; HRMS (ESI) Calcd for $C_{13}H_5N_2$ $(MH)^+$ 199.1235, found 199.1233.



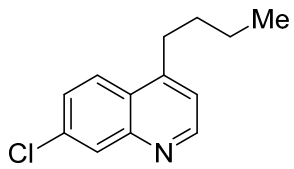
36

*5-Bromo-2-(4-phenylbutyl)pyrimidine (**36**)*: Reaction was conducted in DMF at 60 °C for 24 h with 1.5 equiv of alkylboron reagent using K_3PO_4 as a base. The title compound **36** was obtained as colorless oil (119 mg, 41%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.67-1.75 (m, 2H), 1.81-1.92 (m, 2H), 2.66 (t, *J* = 7.5 Hz, 2H), 2.95 (t, *J* = 7.5 Hz, 2H), 7.15-7.19 (m, 3H), 7.24-7.30 (m, 2H), 8.69 (s, 2H); ^{13}CNMR (75 MHz, $CDCl_3$) δ 28.3, 31.2, 35.8, 38.7, 117.7, 125.8, 128.4, 128.5, 142.4, 157.7, 169.7; IR (neat) cm^{-1} 2930, 1537, 1421, 1116, 1010; HRMS (ESI) Calcd for $C_{14}H_{16}BrN_2$ $(MH)^+$ 291.0497, found 291.0500.



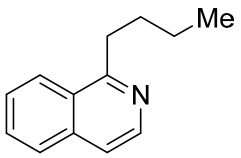
37

*7-Chloro-4-(3-phenoxypropyl)quinoline (**37**)*: Reaction was conducted in DMF at 100°C for 24 h with 1.5 equiv of alkylboron reagent using K_3PO_4 as a base. The title compound **37** was obtained as yellow oil (199 mg, 67%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.17-2.26 (m, 2H), 3.26 (t, *J* = 7.5 Hz, 2H), 4.02 (t, *J* = 6.0 Hz, 2H), 6.90-7.00 (m, 3H), 7.24-7.33 (m, 3H), 7.48 (dd, *J* = 9.0 Hz, 3.0 Hz, 1H), 8.00 (d, *J* = 9.0 Hz, 1H), 8.12 (d, *J* = 3.0 Hz, 1H), 8.79 (d, *J* = 6.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 28.6, 29.7, 66.5, 114.6, 121.0, 121.3, 125.1, 126.1, 127.5, 129.2, 129.6, 135.1, 147.9, 148.9, 151.3, 158.8; IR (neat) cm^{-1} 2930, 1584, 1496, 1238, 1055; HRMS (ESI) Calcd for $C_{18}H_{17}{}^{35}ClNO$ $(MH)^+$ 298.0999, found 298.0993.



38

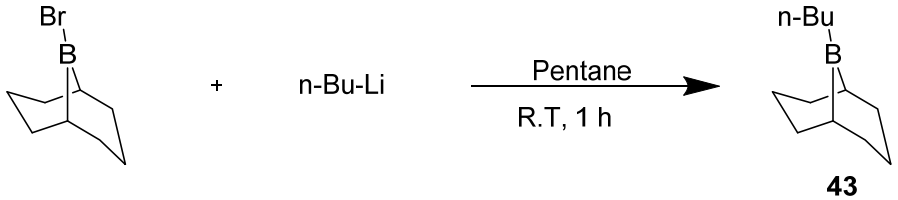
*4-Butyl-7-chloroquinoline (**38**)*: Reaction was conducted in DMF at 100 °C for 24 h with 1.5 equiv of alkylboron reagent using K_3PO_4 as a base. The title compound **38** was obtained as white solid (156 mg, 71%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.95 (t, *J* = 6.0 Hz, 3H), 1.36-1.48 (m, 2H), 1.64-1.74 (m, 2H), 2.99 (t, *J* = 6.0 Hz, 2H), 7.18 (d, *J* = 3.0 Hz, 1H), 7.45 (dd, *J* = 9.0 Hz, 3.0 Hz, 1H), 7.92 (d, *J* = 9.0 Hz, 1H), 8.07 (d, *J* = 3.0 Hz, 1H), 8.76 (d, *J* = 3.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 14.0, 22.8, 31.9, 32.2, 121.0, 125.1, 126.1, 127.2, 129.2, 134.8, 148.9, 151.3; IR (neat) cm^{-1} 2929; 1590, 1458, 1278, 1091; HRMS (ESI) Calcd for $C_{13}H_{15}{}^{35}ClN$ $(MH)^+$ 220.0893, found 220.0895.



39

*1-Butylisoquinoline (**39**)*:[9] Reaction was conducted in DMF at 80 °C for 12 h with 1.5 equiv of alkylboron reagent using K_3PO_4 as a base. The title compound **39** was obtained as yellow oil (167 mg, 90%) after purification by silica gel column chromatography. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.98 (t, *J* = 9.0 Hz, 3H), 1.43-1.56 (m, 2H), 1.79-1.90 (m, 2H), 3.29 (t, *J* = 9.0 Hz, 2H), 7.48 (d, *J* = 6.0 Hz, 1H), 7.54-7.65 (m, 2H), 7.66 (d, *J* = 3.0 Hz, 1H), 8.15 (d, *J* = 6.0 Hz, 1H), 8.43 (d, *J* = 6.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 14.1, 23.1, 32.0, 35.4, 119.2, 125.5, 126.9, 127.5, 129.8, 136.4, 142.1, 162.5; HRMS (ESI) Calcd for $C_{13}H_{16}N$ $(MH)^+$ 186.1283, found 186.1281.

2.5. Synthesis of B-*n*Butyl-9-BBN (43)[12]

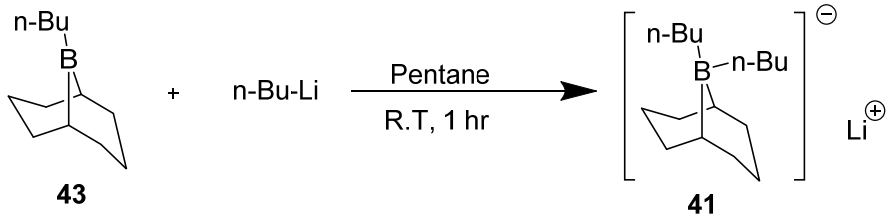


43

[12] Medina, J. R.; Cruz, G.; Cabrera, C. R.; Soderquist, J. A. *J. Org. Chem.* **2003**, *68*, 4631.

*n*Butyllithium (1.0 mmol, 0.625 mL from a 1.6 M solution in hexanes) was added dropwise to a solution of B-Br-9-BBN (1.0 mL from a 1.0 M solution in CH_2Cl_2) in pentane (1 mL) at room temperature. Immediately after the addition, lithium bromide precipitated as white solid. After stirring for 1 h, the reaction mixture was filtered through Celite. Solvent was removed under vacuum to obtain the title compound **43** as a colorless oil (170 mg, 96%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.91 (t, *J* = 7.5 Hz, 3H), 1.19-1.27 (m, 3H), 1.29-1.40 (m, 4H), 1.43-1.52 (m, 2H), 1.66-1.72 (m, 6H), 1.81-1.88 (m, 5H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 14.3, 23.4, 26.1, 26.9, 31.1, 33.3; ^{11}B NMR (96 MHz, $CDCl_3$) δ 103.6.

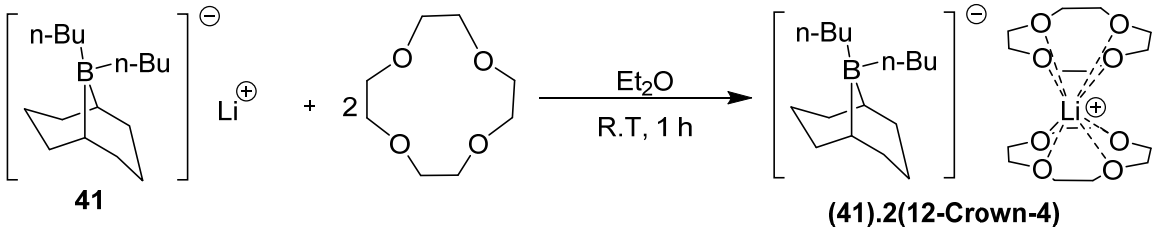
2.6. Synthesis of Lithium B-di-*n*butyl-9-BBN (41)[13]



To a solution of B-*n*butyl-9-BBN **43** (178 mg, 1.0 mmol) in pentane (3 mL), *n*BuLi (1.0 mmol, 0.625 mL from a 1.6 M solution in hexanes) was added dropwise at room temperature. Immediately after the addition, a white solid precipitated out of the solution. After stirring for 1 h, the suspension was filtered through a frit funnel and the residue was washed with pentane (2 mL × 3). The residue was then dried under vacuum to obtain the title compound **41** as a white solid (220 mg, 91%). ^{1}H NMR (300 MHz, DMSO) δ 0.04-0.10 (m, 6H),0.79 (t, *J* = 6.0 Hz, 6H), 0.89-1.00 (m, 4H), 1.08-1.19 (m, 4H), 1.23-1.41 (m, 6H), 1.57-1.75 (m, 2H), 1.80-1.92 (m, 4H); ^{13}C NMR (75 MHz, DMSO) δ 14.9, 23.9 (q, J_{BC} = 52.8 Hz), 26.8 (q, J_{BC} = 49.0 Hz), 27.0, 28.1 (apparent d, J_{BC} = 3.8 Hz), 29.2, 32.9; ^{11}B NMR (96 MHz, DMSO) δ -2.50.

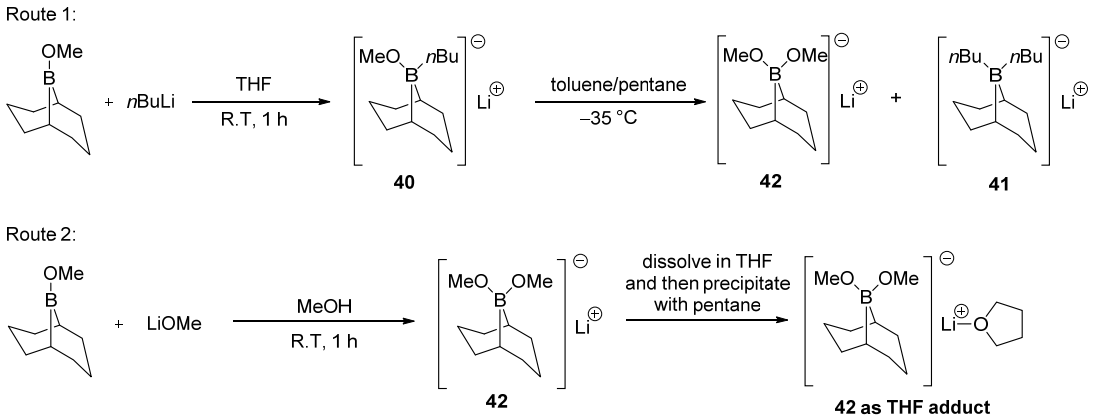
2.7. Synthesis of Lithium B-di-*n*butyl-9-BBN (41)•2(12-Crown-4) Complex

[13] Kramer, G. W.; Brown, H. C. *J. Am. Chem. Soc.* **1976**, *98*, 1964.



A solution of 12-Crown-4 (0.32 mL, 2.0 mmol) in diethyl ether (1 mL) was added dropwise to a solution of lithium B-di-*n*butyl-9-BBN **41** (242 mg, 1.0 mmol) in diethyl ether (2 mL) at room temperature. After stirring for 1 h, the solvent was removed under vacuum to obtain a white residue. The residue was washed with pentane (2 mL × 3) and dried under vacuum to obtain the title compound **(41)•2(12-Crown-4)** as a white solid (540 mg, 91%). ^{1}H NMR (300 MHz, DMSO) δ 0.04-0.09 (m, 6H), 0.79 (t, J = 6.0 Hz, 6H), 0.90-1.00 (m, 4H), 1.06-1.21 (m, 4H), 1.25-1.41 (m, 6H), 1.60-1.76 (m, 2H), 1.80-1.92 (m, 4H), 3.54 (s, 32H); ^{13}C NMR (75 MHz, DMSO) δ 14.8, 23.8 (q, J_{BC} = 52.8 Hz), 26.7 (q, J_{BC} = 49.0 Hz), 27.0, 28.1 (apparent d, J_{BC} = 4.8 Hz), 29.2, 32.9, 69.9; ^{11}B NMR (96 MHz, DMSO) δ -2.50. The title compound **(41)•2(12-Crown-4)** was crystallized by slow evaporation of a pentane/THF solution of **(41)•2(12-Crown-4)** under N_2 atmosphere.

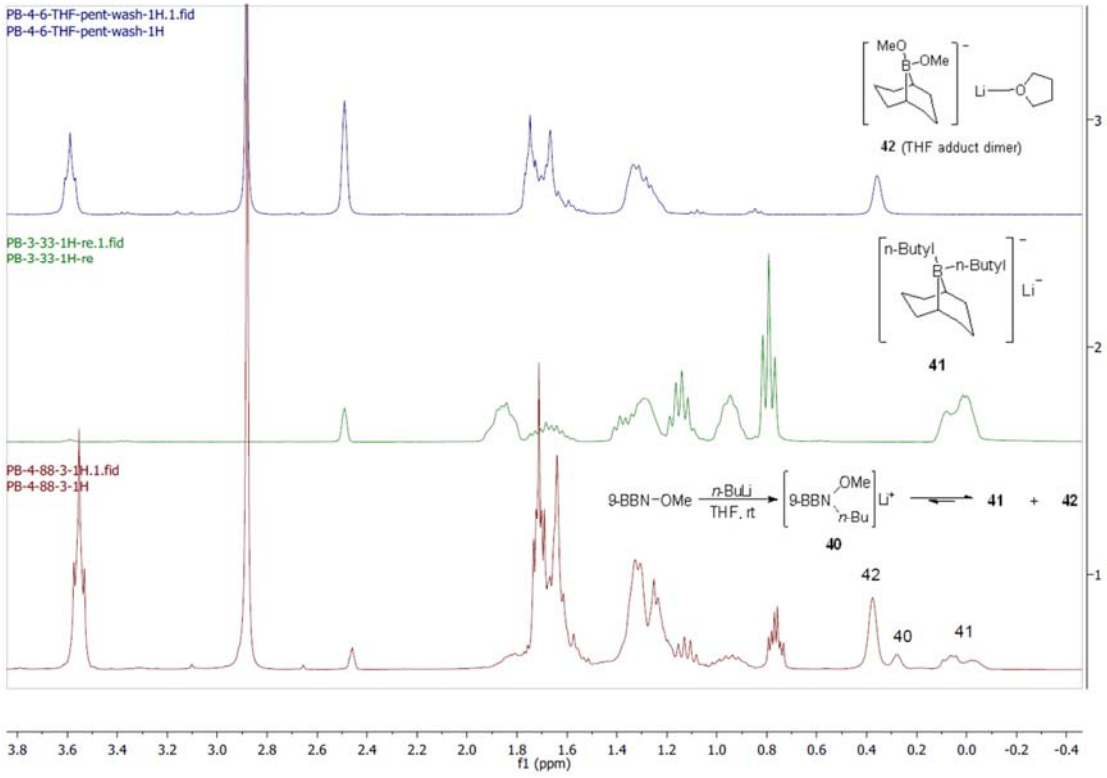
2.8. Synthesis of Lithium B-dimethoxy-9-BBN (42)



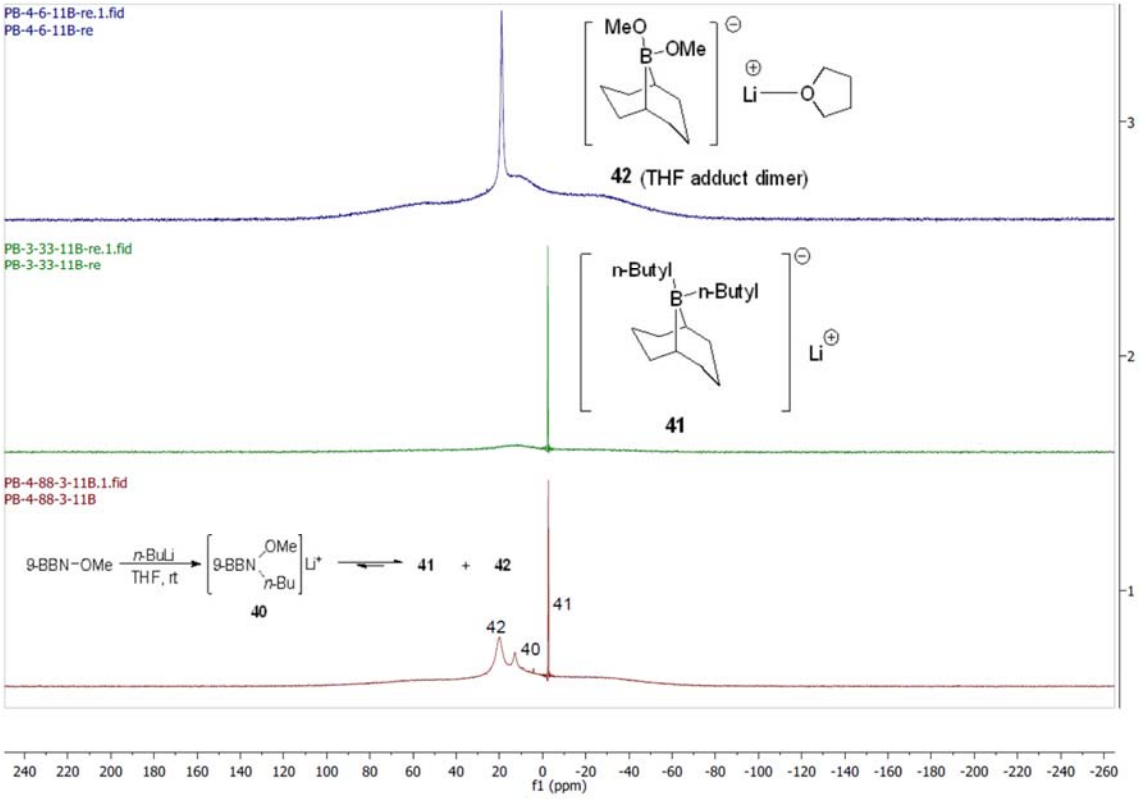
*n*BuLi (1.0 mmol, 0.625 mL from a 1.6 M solution in hexanes) was added dropwise to a solution of B-methoxy-9-BBN (1.0 mmol, 1.0 mL from a 1.0 M solution in hexanes) in THF (2 mL). After stirring for 1 h, the solvent was removed under vacuum to obtain a white residue. ^{1}H and ^{11}B NMR of the white solid in DMSO-d_6 reveals the formation of three compounds **40**-**42** (see the overlaid ^{1}H and ^{11}B NMR spectra below). The white

residue was then dissolved in minimum toluene, layered with pentane and placed in a freeze at -35 °C. Colorless crystals of the title compound **42** were formed in one week.

The title compound **42** was also synthesized independently as follows: B-methoxy-9-BBN (1.0 mmol, 1 mL from a 1.0 M solution in hexanes) was added dropwise to a solution of LiOMe (38 mg, 1.0 mmol) in methanol. After stirring the clear reaction solution for 1 h, the solvent was removed under vacuum to obtain a white residue. The residue was then dissolved in THF (2 mL) and precipitated with excess pentane. The precipitate was washed with pentane (2 mL × 5) and dried under vacuum to obtain the THF adduct of the title compound **42** as a white solid (177 mg, 93%). ^{1}H NMR (300 MHz, DMSO) δ 0.36 (s, 2H), 1.26–1.34 (m, 6H), 1.65-1.77 (m, 10H), 2.88 (s, 6H), 3.57-3.61 (m, 4H); ^{13}C NMR (75 MHz, DMSO) δ 25.1, 26.4, 32.7, 47.2, 67.0; ^{11}B NMR (96 MHz, DMSO) δ -19.0; IR (neat) cm^{-1} 2821, 1202, 1065, 1043.

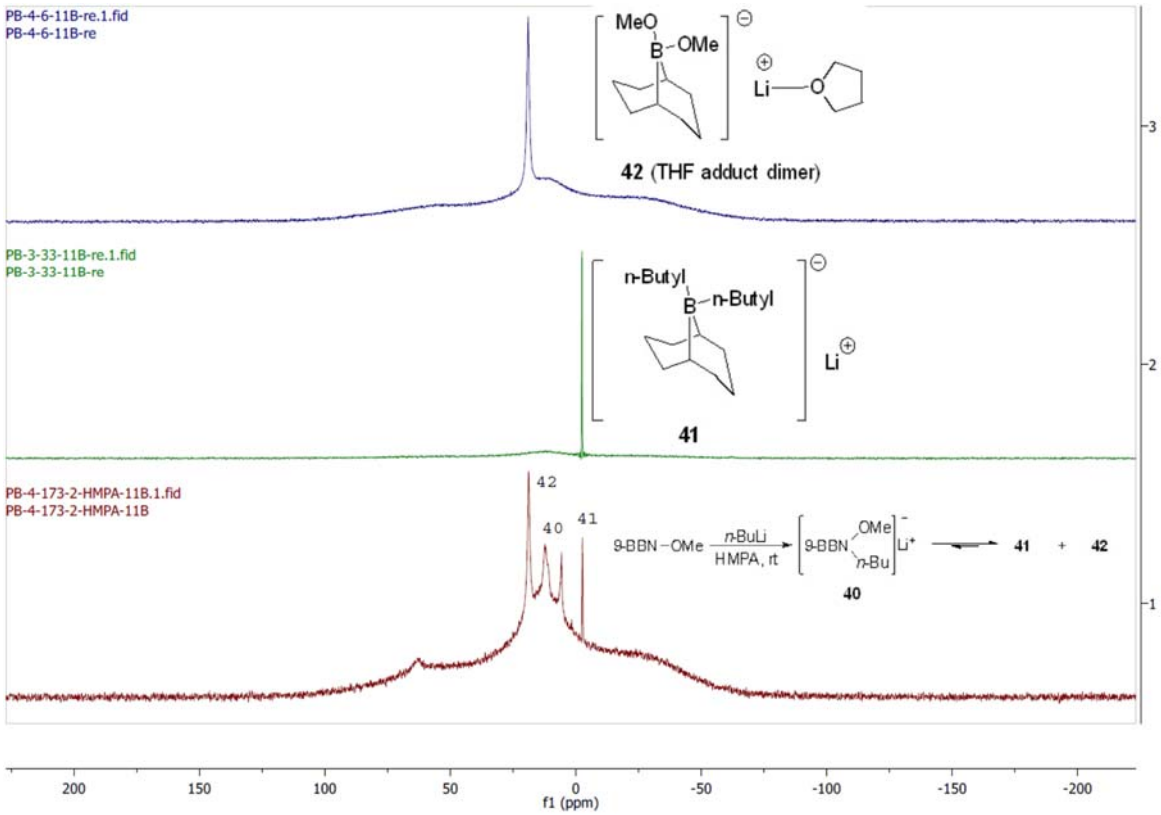


^{1}H NMR spectrum in DMSO-d_6 of the reaction mixture overlaid with the standard samples of the borate complexes **41** and **42**.



^{11}B NMR spectrum in DMSO-d_6 of the reaction mixture overlaid with the standard samples of the borate complexes **41** and **42**.

Reaction in HMPA and in situ formation of compounds 40-42: *n*BuLi (0.10 mmol, 0.062 mL from a 1.6 M solution in hexanes) was added dropwise to a solution of B-methoxy-9-BBN (0.10 mmol, 0.10 mL from a 1.0 M solution in hexanes) in HMPA (1 mL). After stirring for 0.5 h, ^{11}B NMR was acquired which revealed the formation of three compounds **40-42** (see the overlaid ^{11}B NMR spectra below).



^{11}B NMR spectrum in HMPA of the reaction mixture overlaid with the standard samples of the borate complexes **41** and **42**.

2.9. Reactivity of n-Butyl-9-BBN Complexes with 1-Iodoisoquinoline

*Reaction of n-butyl-9-BBN (**43**) with 1-iodoisoquinoline (Table 4, entry 1):* n-butyl-9-BBN (**43**) (17.8 mg, 0.10 mmol), 1-iodoisoquinoline (25.5 mg, 0.10 mmol), and CuI (1.9 mg, 0.010 mmol) were dissolved with DMF in a 1 dram vial and heated at 100 °C. After 3 h, the reaction mixture was cooled to room temperature, 20 μL of pyrene (0.010 mmol, 0.5 M stock solution) as an internal standard was added, diluted with EtOAc (1 mL) and filtered through a short pad of silica gel in a pipette. The reaction mixture was then analyzed by GC. The butylated product **39** was formed only in trace amounts.

*Reaction of n-butyl-9-BBN (**43**) with 1-iodoisoquinoline (Table 4, entry 2):* n-butyl-9-BBN (**43**) (17.8 mg, 0.10 mmol), 1-iodoisoquinoline (25.5 mg, 0.10 mmol), LiOMe (3.8 mg, 0.10 mmol), and CuI (1.9 mg, 0.010 mmol) were dissolved with DMF in a 1 dram vial and heated at 100 °C. After 3 h, the reaction mixture was cooled to room temperature, 20 μL of pyrene (0.010 mmol, 0.5 M stock solution) as an internal standard was added, diluted with EtOAc (1 mL) and filtered through a short pad of silica gel in a

pipette. The reaction mixture was then analyzed by GC. The butylated product **39** was formed in 94% yield.

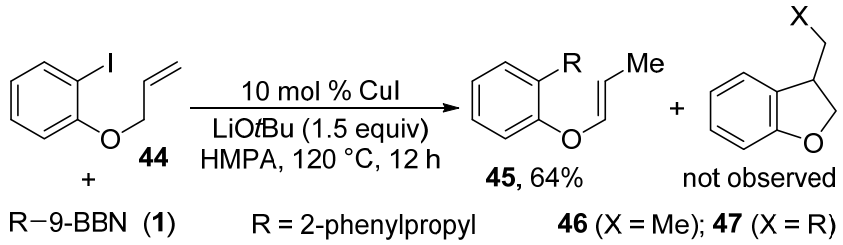
*Reaction of the complex **41** with 1-iodoisoquinoline (Table 4, entry 3):* complex **41** (12.1 mg, 0.050 mmol), 1-iodoisoquinoline (25.5 mg, 0.10 mmol), and CuI (1.9 mg, 0.010 mmol) were dissolved with DMF in a 1 dram vial and heated at 100 °C. After 3 h, the reaction mixture was cooled to room temperature, 20 μL of pyrene (0.010 mmol, 0.5 M stock solution) as an internal standard was added, diluted with EtOAc (1 mL) and filtered through a short pad of silica gel in a pipette. The reaction mixture was then analyzed by GC. The butylated product **39** was formed in 48% yield.

*Reaction of the complex **41** with 1-iodoisoquinoline (Table 4, entry 4):* complex **41** (24.2 mg, 0.10 mmol), 1-iodoisoquinoline (25.5 mg, 0.10 mmol), and CuI (1.9 mg, 0.010 mmol) were dissolved with DMF in a 1 dram vial and heated at 100 °C. After 3 h, the reaction mixture was cooled to room temperature, 20 μL of pyrene (0.010 mmol, 0.5 M stock solution) as an internal standard was added, diluted with EtOAc (1 mL) and filtered through a short pad of silica gel in a pipette. The reaction mixture was then analyzed by GC. The butylated product **39** was formed in 95% yield.

*Reaction of the complex **41** with 1-iodoisoquinoline (Table 4, entry 5):* complex **41** (12.1 mg, 0.050 mmol), 1-iodoisoquinoline (25.5 mg, 0.10 mmol), LiOMe (1.9 mg, 0.050 mmol), and CuI (1.9 mg, 0.010 mmol) were dissolved with DMF in a 1 dram vial and heated at 100 °C. After 3 h, the reaction mixture was cooled to room temperature, 20 μL of pyrene (0.010 mmol, 0.5 M stock solution) as an internal standard was added, diluted with EtOAc (1 mL) and filtered through a short pad of silica gel in a pipette. The reaction mixture was then analyzed by GC. The butylated product **39** was formed in 94% yield.

*Reaction of the complexes **41** and **42** with 1-iodoisoquinoline (Table 4, entry 6):* complex **41** (12.1 mg, 0.050 mmol), complex **42** (9.5 mg, 0.050 mmol), 1-iodoisoquinoline (25.5 mg, 0.10 mmol), and CuI (1.9 mg, 0.010 mmol) were dissolved with DMF in a 1 dram vial and heated at 100 °C. After 3 h, the reaction mixture was cooled to room temperature, 20 μL of pyrene (0.010 mmol, 0.5 M stock solution) as an internal standard was added, diluted with EtOAc (1 mL) and filtered through a short pad of silica gel in a pipette. The reaction mixture was then analyzed by GC. The butylated product **39** was formed in 97% yield.

2.10. Reaction of B-(2-Phenylpropyl)-9-BBN (1) with *o*-Allyloxyiodobenzene



o-Allyloxyiodobenzene (260.0 mg, 1.0 mmol), LiO*t*Bu (120 mg, 1.5 mmol), and CuI (19.0 mg, 0.10 mmol) were weighed in a 15 mL pressure tube and dissolved in HMPA (5 mL). B-(2-Phenylpropyl)-9-BBN (**1**) (360.0 mg, 1.5 mmol) was then added to the reaction mixture and tightly capped. The reaction mixture was placed in an oil bath pre-heated to 120 °C with vigorous stirring. After 24 h, reaction mixture was cooled to room temperature. An aliquot of the reaction mixture was analyzed by GC and GC-MS. Only the cross-coupled product **45** was formed. The cyclized product **46** and the cyclized-coupled product **47** were not detected.

The remainder of the reaction mixture was diluted with ethyl acetate (15 mL) and washed with H_2O (5 mL × 3). The aqueous fraction was extracted back with ethyl acetate (5 mL × 3) and combined with the first ethyl acetate fraction. The combined ethyl acetate fraction was dried over Na_2SO_4 and the solvent was removed in a rotary evaporator. The title compound (**45**) was obtained as yellow oil (161 mg, 64%) after purification by silica gel column chromatography using 5% ethyl acetate in hexanes. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.26 (d, *J* = 6.0Hz, 3H), 1.77 (dd, *J* = 6.0 Hz, 3.0Hz, 3H), 2.81-3.15 (m, 3H), 4.86 (p, *J* = 6.0 Hz, 1H), 6.34 (d, *J* = 6.0 Hz, 1H), 6.92 (q, *J* = 3.0 Hz, 2H), 7.03 (d, *J* = 6.0 Hz, 1H), 7.12-7.31 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 9.6, 21.1, 39.6, 40.5, 106.7, 114.8, 122.2, 126.0, 127.1, 127.3, 128.4, 130.4, 131.4, 141.6, 147.6, 155.8; GCMS (m/z) 252.2.

2.11. Hammett Plot

α-Methylstyrene (1.536 g, 13.0 mmol) and 9-BBN (10 mmol, 20 mL from a 0.5 M solution in THF) were mixed in a sealed tube, tightly capped and heated at 60 °C. After 4 h, the reaction mixture was transferred to a round-bottom flask, and subjected to high vacuum at room temperature until THF was removed and then at 40 °C for 2 h to remove

excess α-methylstyrene. The alkyl-9-BBN (**1**) thus obtained was directly used for the following kinetic experiment.

CuI (38.0 mg, 0.20 mmol) was weighed in a 1.0 mL volumetric flask and dissolved with HMPA to obtain a stock solution (0.20 M).

Alkyl-9-BBN (**1**) (480.4 mg, 2.0 mmol) was weighed in a 1.0 mL volumetric flask and dissolved with HMPA to obtain a stock solution (2.0 M).

LiOtBu (300.2 mg, 3.75 mmol) was weighed in a 5.0 mL volumetric flask and dissolved with HMPA by stirring with a magnetic stirrer for 8 h to obtain a stock solution (0.75 M).

ArI (5.0 mmol) was weighed in a 2.0 mL volumetric flask and dissolved with HMPA to obtain a stock solution (2.50 M).

CuI (50 μL, 0.010 mmol), LiOtBu (200 μL, 0.150 mmol), alkyl-9-BBN (**1**) (50 μL, 0.10 mmol) and *p*-XC_6H_4I (X = H, OMe, Me, F, Cl, CF_3) (200 μL, 0.50 mmol) were mixed in a 1 dram vial (total volume: 0.50 mL). The reaction mixture was then tightly capped and placed in a hotplate pre-heated to 100 °C. A total of 6 to 9 reactions were setup for each *p*-XC_6H_4I and were stopped at 6-9 time intervals. At least a duplicate reaction was setup for each of the data point to take an average. Product yields at different time points for the reaction of *p*-XC_6H_4I with alkyl-9-BBN (**1**) were determined by GC using pyrene as a standard. The product yields were then plotted against the corresponding reaction times and the slope of the linear portion of the curve (for less than 30% yield) was used to determine the initial rates of the reactions.

The initial rates of the reactions ($k_{X(initial)}$), $\log[k_{X(initial)}/k_{H(initial)}]$ and σ-values used to obtain the Hammett plot are given below:

Table SI-1. Values used to Obtain the Hammett Plot

iodoarenes	$k_{X(initial)}$ ($M\ s^{-1}$)	$\log[k_{X(initial)}/k_{H(initial)}]$	σ
X = H	1.34×10^{-5}	0.00	0.00
X = OMe	0.61×10^{-5}	-0.34	-0.27
X = Me	0.92×10^{-5}	-0.16	-0.17
X = F	1.68×10^{-5}	0.10	0.06
X = Cl	3.02×10^{-5}	0.35	0.23
X = CF_3	7.58×10^{-5}	0.75	0.54

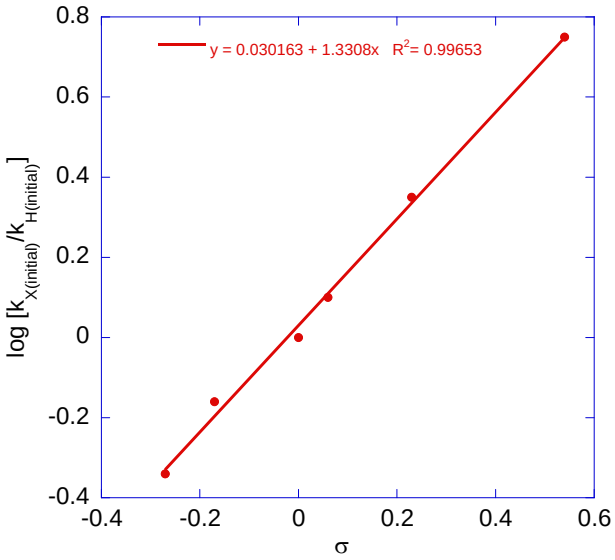


Figure SI-1. The Hammett plot for the reaction of alkyl-9-BBN (**1**) with 5.0 equivalents of *p*-XC_6H_4I (X = H, OMe, Me, F, Cl, CF_3). The curve depicts the result of an unweighted least-square fit to y = a*x + b (a = +1.33, b = 3.01×10^{-2}, R^2 = 0.99). Substituent constants (σ values) were adopted from C. Hansch, A. Leo, R. W. Taft, *Chem. Rev.* **1991**, *91*, 165-195.

3. X-ray Crystallographic Data for Complex 41

Table 1 Crystal data and structure refinement for complex **41.**

Empirical formula	$C_{32}H_{64}BLiO_8$
Formula weight	594.58
Temperature/K	99(2)
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	10.6111(3)
b/Å	22.4729(6)
c/Å	14.5760(4)
α/°	90
β/°	92.672(2)
γ/°	90
Volume/$Å^3$	3472.03(17)
Z	4
$\rho_{calc}g/cm^3$	1.138
μ/mm^{-1}	0.078
F(000)	1312.0

Crystal size/mm^3	0.841 × 0.315 × 0.216
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	3.334 to 55.016
Index ranges	-13 ≤ h ≤ 12, -29 ≤ k ≤ 29, -18 ≤ l ≤ 18
Reflections collected	33894
Independent reflections	7967 [R_{int} = 0.0277]
Data/restraints/parameters	7967/0/454
Goodness-of-fit on F^2	1.044
Final R indexes [I>=2σ (I)]	R_1 = 0.0476, wR_2 = 0.1224
Final R indexes [all data]	R_1 = 0.0599, wR_2 = 0.1304
Largest diff. peak/hole / e Å^{-3}	0.661/-0.313

Table 2. Atomic coordinates and equivalent isotropic atomic displacement parameters (Å^2) for **41**. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x/a	y/b	z/c	U(eq)
Li1	0.2285(2)	0.87839(11)	0.64336(17)	0.0213(5)
B1	0.77364(13)	0.63346(6)	0.65648(10)	0.0124(3)
C1	0.70466(12)	0.66246(6)	0.56294(9)	0.0152(3)
C2	0.56306(12)	0.64733(6)	0.55709(9)	0.0177(3)
C3	0.49108(12)	0.66703(6)	0.64118(9)	0.0168(3)
C4	0.56008(11)	0.65543(6)	0.73491(9)	0.0142(3)
C5	0.70279(11)	0.66834(6)	0.73955(8)	0.0120(2)
C6	0.73247(12)	0.73525(6)	0.73718(9)	0.0161(3)
C7	0.69778(13)	0.76581(6)	0.64465(10)	0.0194(3)
C8	0.72949(13)	0.72998(6)	0.55833(9)	0.0188(3)
C9	0.92681(12)	0.64689(6)	0.65863(9)	0.0142(3)
C10	0.00177(12)	0.63294(6)	0.74843(9)	0.0147(3)
C11	0.14354(12)	0.64338(6)	0.74390(9)	0.0150(3)
C12	0.21950(12)	0.62877(7)	0.83224(10)	0.0199(3)
C13	0.74887(12)	0.56106(6)	0.66290(9)	0.0148(3)
C14	0.79739(13)	0.52294(6)	0.58480(10)	0.0192(3)
C15	0.75079(14)	0.45881(6)	0.58324(10)	0.0228(3)
C16	0.80677(19)	0.42072(8)	0.50923(12)	0.0354(4)
C17	0.0721(4)	0.80070(15)	0.7721(3)	0.0231(8)
C18	0.0275(4)	0.86106(19)	0.7971(3)	0.0246(8)
C19	0.0335(3)	0.96052(14)	0.7402(3)	0.0295(8)
C20	0.1511(5)	0.9861(2)	0.7789(3)	0.0269(10)
C21	0.3749(5)	0.9776(2)	0.7579(4)	0.0270(10)
C22	0.4134(3)	0.92275(15)	0.8072(3)	0.0278(9)
C23	0.4134(3)	0.81784(18)	0.7832(3)	0.0214(7)
C24	0.2912(4)	0.79769(16)	0.8178(3)	0.0216(7)

	x/a	y/b	z/c	U(eq)
C17A	0.1195(3)	0.80968(13)	0.8123(2)	0.0183(6)
C18A	0.0103(3)	0.84106(17)	0.7634(3)	0.0202(7)
C19A	0.0621(3)	0.94353(14)	0.7923(2)	0.0191(7)
C20A	0.1304(5)	0.9940(2)	0.7449(3)	0.0235(10)
C21A	0.3462(5)	0.9758(2)	0.7906(3)	0.0231(9)
C22A	0.4550(3)	0.93862(13)	0.7577(2)	0.0207(7)
C23A	0.4088(3)	0.84251(16)	0.8169(2)	0.0182(6)
C24A	0.3359(3)	0.78768(14)	0.7866(2)	0.0181(7)
C25	0.24388(15)	0.75805(6)	0.54220(10)	0.0233(3)
C26	0.12662(15)	0.78226(7)	0.49555(10)	0.0251(3)
C27	0.98669(14)	0.86442(8)	0.51351(11)	0.0286(4)
C28	0.04871(15)	0.91367(7)	0.46428(11)	0.0272(3)
C29	0.21401(17)	0.98516(7)	0.49214(11)	0.0290(4)
C30	0.32984(15)	0.95318(7)	0.46384(10)	0.0259(3)
C31	0.47240(13)	0.87888(7)	0.52480(11)	0.0245(3)
C32	0.40837(14)	0.82226(7)	0.49535(10)	0.0241(3)
O1	0.20753(9)	0.80289(4)	0.74531(7)	0.0201(2)
O2	0.04792(10)	0.89766(4)	0.72320(7)	0.0217(2)
O3	0.25012(10)	0.97363(5)	0.71825(7)	0.0223(2)
O4	0.40996(10)	0.87748(4)	0.73950(7)	0.0213(2)
O5	0.33107(10)	0.80518(5)	0.56748(7)	0.0218(2)
O6	0.07881(10)	0.82480(5)	0.55665(7)	0.0235(2)
O7	0.12374(10)	0.94476(5)	0.53098(7)	0.0253(2)
O8	0.38170(10)	0.92487(5)	0.54373(7)	0.0257(2)

Table 3 Anisotropic Displacement Parameters ($Å^2$) for complex **41.** The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

	U11	U22	U33	U23	U13	U12
Li1	0.0238(12)	0.0229(12)	0.0173(12)	0.0002(9)	0.0023(9)	0.0012(9)
B1	0.0128(6)	0.0150(7)	0.0097(6)	0.0010(5)	0.0019(5)	0.0014(5)
C1	0.0158(6)	0.0206(7)	0.0093(6)	0.0009(5)	0.0021(5)	0.0038(5)
C2	0.0177(6)	0.0241(7)	0.0110(6)	-0.0003(5)	-0.0023(5)	0.0030(5)
C3	0.0118(6)	0.0243(7)	0.0141(6)	0.0001(5)	-0.0012(5)	0.0024(5)
C4	0.0127(6)	0.0190(6)	0.0110(6)	0.0002(5)	0.0024(5)	0.0017(5)
C5	0.0117(6)	0.0148(6)	0.0094(6)	0.0010(4)	0.0004(4)	0.0013(4)
C6	0.0170(6)	0.0165(6)	0.0149(6)	-0.0007(5)	0.0011(5)	0.0014(5)
C7	0.0217(7)	0.0159(6)	0.0205(7)	0.0044(5)	0.0012(5)	0.0019(5)
C8	0.0184(6)	0.0226(7)	0.0155(6)	0.0080(5)	0.0026(5)	0.0038(5)
C9	0.0140(6)	0.0167(6)	0.0123(6)	0.0020(5)	0.0030(5)	0.0018(5)
C10	0.0122(6)	0.0177(6)	0.0144(6)	0.0024(5)	0.0029(5)	0.0017(5)

C11	0.0130(6)	0.0160(6)	0.0161(6)	0.0002(5)	0.0025(5)	-0.0006(5)
C12	0.0138(6)	0.0247(7)	0.0209(7)	0.0017(6)	-0.0007(5)	0.0002(5)
C13	0.0161(6)	0.0166(6)	0.0117(6)	-0.0004(5)	0.0017(5)	0.0013(5)
C14	0.0239(7)	0.0180(7)	0.0159(7)	-0.0017(5)	0.0041(5)	0.0025(5)
C15	0.0263(7)	0.0199(7)	0.0225(7)	-0.0057(6)	0.0022(6)	-0.0001(6)
C16	0.0568(11)	0.0235(8)	0.0264(9)	-0.0090(7)	0.0074(8)	0.0034(7)
C17	0.0248(18)	0.0206(17)	0.025(2)	0.0032(14)	0.0121(15)	-0.0040(14)
C18	0.0307(19)	0.021(2)	0.023(2)	0.0035(15)	0.0139(16)	0.0029(15)
C19	0.0333(17)	0.0246(16)	0.031(2)	-0.0025(14)	0.0092(14)	0.0103(13)
C20	0.038(2)	0.018(2)	0.026(3)	-0.0038(19)	0.010(2)	0.0047(16)
C21	0.029(2)	0.0211(19)	0.031(3)	-0.007(2)	-0.002(2)	-0.0046(16)
C22	0.0296(17)	0.0255(17)	0.027(2)	-0.0056(14)	-0.0086(15)	-0.0032(13)
C23	0.0248(17)	0.0217(18)	0.0173(18)	0.0017(14)	-0.0044(13)	0.0058(15)
C24	0.030(2)	0.0192(17)	0.0151(17)	0.0038(13)	-0.0009(15)	0.0011(14)
C17A	0.0236(15)	0.0217(14)	0.0097(14)	0.0011(11)	0.0032(12)	-0.0047(11)
C18A	0.0212(15)	0.0245(19)	0.0156(17)	-0.0014(13)	0.0072(12)	-0.0062(14)
C19A	0.0251(15)	0.0219(15)	0.0108(15)	-0.0001(12)	0.0050(12)	0.0097(12)
C20A	0.035(2)	0.0170(19)	0.019(2)	0.0032(18)	0.005(2)	0.0107(15)
C21A	0.032(2)	0.0199(17)	0.017(2)	-0.0070(17)	-0.0015(16)	-0.0020(15)
C22A	0.0218(14)	0.0225(14)	0.0176(15)	0.0012(11)	-0.0005(11)	-0.0097(11)
C23A	0.0226(14)	0.0192(16)	0.0126(15)	0.0008(12)	-0.0018(11)	0.0026(12)
C24A	0.0218(17)	0.0192(15)	0.0135(16)	0.0026(12)	0.0020(12)	0.0088(13)
C25	0.0391(8)	0.0154(7)	0.0154(7)	-0.0012(5)	0.0022(6)	-0.0009(6)
C26	0.0311(8)	0.0250(8)	0.0189(7)	-0.0038(6)	-0.0003(6)	-0.0038(6)
C27	0.0175(7)	0.0487(10)	0.0191(7)	-0.0011(7)	-0.0038(5)	0.0055(6)
C28	0.0288(8)	0.0333(8)	0.0189(7)	-0.0017(6)	-0.0056(6)	0.0093(6)
C29	0.0492(10)	0.0169(7)	0.0207(7)	0.0021(6)	0.0009(7)	0.0065(7)
C30	0.0344(8)	0.0244(8)	0.0189(7)	0.0043(6)	0.0023(6)	-0.0012(6)
C31	0.0167(6)	0.0371(9)	0.0202(7)	0.0016(6)	0.0045(5)	0.0019(6)
C32	0.0252(7)	0.0304(8)	0.0171(7)	0.0016(6)	0.0051(6)	0.0087(6)
O1	0.0270(5)	0.0218(5)	0.0117(5)	-0.0002(4)	0.0045(4)	0.0019(4)
O2	0.0288(5)	0.0202(5)	0.0165(5)	-0.0003(4)	0.0058(4)	0.0005(4)
O3	0.0257(5)	0.0244(5)	0.0168(5)	0.0005(4)	0.0016(4)	0.0028(4)
O4	0.0271(5)	0.0207(5)	0.0159(5)	-0.0016(4)	-0.0012(4)	0.0017(4)
O5	0.0254(5)	0.0273(5)	0.0129(5)	-0.0002(4)	0.0014(4)	0.0051(4)
O6	0.0272(5)	0.0304(6)	0.0128(5)	-0.0011(4)	-0.0013(4)	0.0010(4)
O7	0.0312(6)	0.0300(6)	0.0147(5)	-0.0033(4)	0.0012(4)	0.0064(4)
O8	0.0291(5)	0.0319(6)	0.0162(5)	0.0005(4)	0.0011(4)	0.0007(4)

Table 4 Bond Lengths (Å) for complex **41.**

Li1-O1	2.273(3)	Li1-O5	2.286(3)
Li1-O6	2.320(3)	Li1-O4	2.328(3)

Li1-O2	2.329(3)	Li1-O3	2.409(3)
Li1-O7	2.444(3)	Li1-O8	2.462(3)
B1-C1	1.6512(18)	B1-C13	1.6516(19)
B1-C9	1.6520(18)	B1-C5	1.6524(18)
C1-C2	1.5390(18)	C1-C8	1.5420(19)
C1-H1A	1.0	C2-C3	1.5393(18)
C2-H2A	0.99	C2-H2B	0.99
C3-C4	1.5417(17)	C3-H3A	0.99
C3-H3B	0.99	C4-C5	1.5402(17)
C4-H4A	0.99	C4-H4B	0.99
C5-C6	1.5371(18)	C5-H5A	1.0
C6-C7	1.5427(18)	C6-H6A	0.99
C6-H6B	0.99	C7-C8	1.544(2)
C7-H7A	0.99	C7-H7B	0.99
C8-H8A	0.99	C8-H8B	0.99
C9-C10	1.5320(17)	C9-H9A	0.99
C9-H9B	0.99	C10-C11	1.5269(17)
C10-H10A	0.99	C10-H10B	0.99
C11-C12	1.5226(18)	C11-H11A	0.99
C11-H11B	0.99	C12-H12A	0.98
C12-H12B	0.98	C12-H12C	0.98
C13-C14	1.5328(18)	C13-H13A	0.99
C13-H13B	0.99	C14-C15	1.523(2)
C14-H14A	0.99	C14-H14B	0.99
C15-C16	1.520(2)	C15-H15A	0.99
C15-H15B	0.99	C16-H16A	0.98
C16-H16B	0.98	C16-H16C	0.98
C17-C18	1.488(5)	C17-O1	1.508(3)
C17-H17A	0.99	C17-H17B	0.99
C18-O2	1.380(4)	C18-H18A	0.99
C18-H18B	0.99	C19-O2	1.444(3)
C19-C20	1.463(7)	C19-H19A	0.99
C19-H19B	0.99	C20-O3	1.431(5)
C20-H20A	0.99	C20-H20B	0.99
C21-O3	1.423(6)	C21-C22	1.475(7)
C21-H21A	0.99	C21-H21B	0.99
C22-O4	1.416(3)	C22-H22A	0.99
C22-H22B	0.99	C23-O4	1.484(4)
C23-C24	1.484(5)	C23-H23A	0.99

C23-H23B	0.99	C24-O1	1.353(3)
C24-H24A	0.99	C24-H24B	0.99
C17A-O1	1.391(3)	C17A-C18A	1.507(5)
C17A-H17C	0.99	C17A-H17D	0.99
C18A-O2	1.464(4)	C18A-H18C	0.99
C18A-H18D	0.99	C19A-O2	1.444(3)
C19A-C20A	1.527(6)	C19A-H19C	0.99
C19A-H19D	0.99	C20A-O3	1.422(6)
C20A-H20C	0.99	C20A-H20D	0.99
C21A-O3	1.433(5)	C21A-C22A	1.520(6)
C21A-H21C	0.99	C21A-H21D	0.99
C22A-O4	1.475(3)	C22A-H22C	0.99
C22A-H22D	0.99	C23A-O4	1.376(3)
C23A-C24A	1.510(5)	C23A-H23C	0.99
C23A-H23D	0.99	C24A-O1	1.503(3)
C24A-H24C	0.99	C24A-H24D	0.99
C25-O5	1.4429(18)	C25-C26	1.492(2)
C25-H25A	0.99	C25-H25B	0.99
C26-O6	1.4166(18)	C26-H26A	0.99
C26-H26B	0.99	C27-O6	1.4444(18)
C27-C28	1.489(2)	C27-H27A	0.99
C27-H27B	0.99	C28-O7	1.4124(18)
C28-H28A	0.99	C28-H28B	0.99
C29-O7	1.454(2)	C29-C30	1.498(2)
C29-H29A	0.99	C29-H29B	0.99
C30-O8	1.4155(18)	C30-H30A	0.99
C30-H30B	0.99	C31-O8	1.4474(18)
C31-C32	1.496(2)	C31-H31A	0.99
C31-H31B	0.99	C32-O5	1.4164(17)
C32-H32A	0.99	C32-H32B	0.99

Table 5 Bond Angles (°) for complex **41.**

O1-Li1-O5	80.94(9)	O1-Li1-O6	83.29(9)
O5-Li1-O6	72.08(8)	O1-Li1-O4	72.75(8)
O5-Li1-O4	83.45(9)	O6-Li1-O4	148.14(12)
O1-Li1-O2	72.79(8)	O5-Li1-O2	142.85(12)
O6-Li1-O2	79.06(8)	O4-Li1-O2	112.18(10)
O1-Li1-O3	112.15(10)	O5-Li1-O3	145.22(12)
O6-Li1-O3	139.14(12)	O4-Li1-O3	71.12(8)

O2-Li1-O3	70.81(8)	O1-Li1-O7	146.95(12)
O5-Li1-O7	109.08(10)	O6-Li1-O7	70.93(8)
O4-Li1-O7	138.28(12)	O2-Li1-O7	82.08(8)
O3-Li1-O7	78.00(8)	O1-Li1-O8	142.16(12)
O5-Li1-O8	71.20(8)	O6-Li1-O8	110.67(10)
O4-Li1-O8	78.96(8)	O2-Li1-O8	142.86(12)
O3-Li1-O8	80.73(8)	O7-Li1-O8	68.78(7)
C1-B1-C13	111.72(10)	C1-B1-C9	109.96(10)
C13-B1-C9	109.75(10)	C1-B1-C5	102.62(10)
C13-B1-C5	110.29(10)	C9-B1-C5	112.36(10)
C2-C1-C8	112.55(11)	C2-C1-B1	110.64(10)
C8-C1-B1	110.75(11)	C2-C1-H1A	107.6
C8-C1-H1A	107.6	B1-C1-H1A	107.6
C1-C2-C3	114.31(11)	C1-C2-H2A	108.7
C3-C2-H2A	108.7	C1-C2-H2B	108.7
C3-C2-H2B	108.7	H2A-C2-H2B	107.6
C2-C3-C4	115.10(10)	C2-C3-H3A	108.5
C4-C3-H3A	108.5	C2-C3-H3B	108.5
C4-C3-H3B	108.5	H3A-C3-H3B	107.5
C5-C4-C3	115.62(10)	C5-C4-H4A	108.4
C3-C4-H4A	108.4	C5-C4-H4B	108.4
C3-C4-H4B	108.4	H4A-C4-H4B	107.4
C6-C5-C4	112.66(10)	C6-C5-B1	110.29(10)
C4-C5-B1	111.01(10)	C6-C5-H5A	107.5
C4-C5-H5A	107.5	B1-C5-H5A	107.5
C5-C6-C7	114.49(11)	C5-C6-H6A	108.6
C7-C6-H6A	108.6	C5-C6-H6B	108.6
C7-C6-H6B	108.6	H6A-C6-H6B	107.6
C6-C7-C8	115.34(11)	C6-C7-H7A	108.4
C8-C7-H7A	108.4	C6-C7-H7B	108.4
C8-C7-H7B	108.4	H7A-C7-H7B	107.5
C1-C8-C7	115.69(11)	C1-C8-H8A	108.4
C7-C8-H8A	108.4	C1-C8-H8B	108.4
C7-C8-H8B	108.4	H8A-C8-H8B	107.4
C10-C9-B1	116.74(10)	C10-C9-H9A	108.1
B1-C9-H9A	108.1	C10-C9-H9B	108.1
B1-C9-H9B	108.1	H9A-C9-H9B	107.3
C11-C10-C9	113.88(11)	C11-C10-H10A	108.8
C9-C10-H10A	108.8	C11-C10-H10B	108.8

C9-C10-H10B	108.8	H10A-C10-H10B	107.7
C12-C11-C10	114.46(11)	C12-C11-H11A	108.6
C10-C11-H11A	108.6	C12-C11-H11B	108.6
C10-C11-H11B	108.6	H11A-C11-H11B	107.6
C11-C12-H12A	109.5	C11-C12-H12B	109.5
H12A-C12-H12B	109.5	C11-C12-H12C	109.5
H12A-C12-H12C	109.5	H12B-C12-H12C	109.5
C14-C13-B1	116.66(11)	C14-C13-H13A	108.1
B1-C13-H13A	108.1	C14-C13-H13B	108.1
B1-C13-H13B	108.1	H13A-C13-H13B	107.3
C15-C14-C13	114.82(12)	C15-C14-H14A	108.6
C13-C14-H14A	108.6	C15-C14-H14B	108.6
C13-C14-H14B	108.6	H14A-C14-H14B	107.5
C16-C15-C14	113.96(13)	C16-C15-H15A	108.8
C14-C15-H15A	108.8	C16-C15-H15B	108.8
C14-C15-H15B	108.8	H15A-C15-H15B	107.7
C15-C16-H16A	109.5	C15-C16-H16B	109.5
H16A-C16-H16B	109.5	C15-C16-H16C	109.5
H16A-C16-H16C	109.5	H16B-C16-H16C	109.5
C18-C17-O1	110.6(3)	C18-C17-H17A	109.5
O1-C17-H17A	109.5	C18-C17-H17B	109.5
O1-C17-H17B	109.5	H17A-C17-H17B	108.1
O2-C18-C17	106.8(3)	O2-C18-H18A	110.4
C17-C18-H18A	110.4	O2-C18-H18B	110.4
C17-C18-H18B	110.4	H18A-C18-H18B	108.6
O2-C19-C20	110.8(3)	O2-C19-H19A	109.5
C20-C19-H19A	109.5	O2-C19-H19B	109.5
C20-C19-H19B	109.5	H19A-C19-H19B	108.1
O3-C20-C19	108.8(3)	O3-C20-H20A	109.9
C19-C20-H20A	109.9	O3-C20-H20B	109.9
C19-C20-H20B	109.9	H20A-C20-H20B	108.3
O3-C21-C22	112.1(4)	O3-C21-H21A	109.2
C22-C21-H21A	109.2	O3-C21-H21B	109.2
C22-C21-H21B	109.2	H21A-C21-H21B	107.9
O4-C22-C21	105.3(3)	O4-C22-H22A	110.7
C21-C22-H22A	110.7	O4-C22-H22B	110.7
C21-C22-H22B	110.7	H22A-C22-H22B	108.8
O4-C23-C24	114.7(3)	O4-C23-H23A	108.6
C24-C23-H23A	108.6	O4-C23-H23B	108.6

C24-C23-H23B	108.6	H23A-C23-H23B	107.6
O1-C24-C23	105.1(3)	O1-C24-H24A	110.7
C23-C24-H24A	110.7	O1-C24-H24B	110.7
C23-C24-H24B	110.7	H24A-C24-H24B	108.8
O1-C17A-C18A	104.3(3)	O1-C17A-H17C	110.9
C18A-C17A-H17C	110.9	O1-C17A-H17D	110.9
C18A-C17A-H17D	110.9	H17C-C17A-H17D	108.9
O2-C18A-C17A	112.2(3)	O2-C18A-H18C	109.2
C17A-C18A-H18C	109.2	O2-C18A-H18D	109.2
C17A-C18A-H18D	109.2	H18C-C18A-H18D	107.9
O2-C19A-C20A	104.5(3)	O2-C19A-H19C	110.8
C20A-C19A-H19C	110.8	O2-C19A-H19D	110.8
C20A-C19A-H19D	110.8	H19C-C19A-H19D	108.9
O3-C20A-C19A	109.5(3)	O3-C20A-H20C	109.8
C19A-C20A-H20C	109.8	O3-C20A-H20D	109.8
C19A-C20A-H20D	109.8	H20C-C20A-H20D	108.2
O3-C21A-C22A	105.9(3)	O3-C21A-H21C	110.5
C22A-C21A-H21C	110.5	O3-C21A-H21D	110.5
C22A-C21A-H21D	110.5	H21C-C21A-H21D	108.7
O4-C22A-C21A	108.9(3)	O4-C22A-H22C	109.9
C21A-C22A-H22C	109.9	O4-C22A-H22D	109.9
C21A-C22A-H22D	109.9	H22C-C22A-H22D	108.3
O4-C23A-C24A	104.5(3)	O4-C23A-H23C	110.9
C24A-C23A-H23C	110.9	O4-C23A-H23D	110.9
C24A-C23A-H23D	110.9	H23C-C23A-H23D	108.9
O1-C24A-C23A	112.0(2)	O1-C24A-H24C	109.2
C23A-C24A-H24C	109.2	O1-C24A-H24D	109.2
C23A-C24A-H24D	109.2	H24C-C24A-H24D	107.9
O5-C25-C26	111.09(12)	O5-C25-H25A	109.4
C26-C25-H25A	109.4	O5-C25-H25B	109.4
C26-C25-H25B	109.4	H25A-C25-H25B	108.0
O6-C26-C25	105.96(12)	O6-C26-H26A	110.5
C25-C26-H26A	110.5	O6-C26-H26B	110.5
C25-C26-H26B	110.5	H26A-C26-H26B	108.7
O6-C27-C28	111.26(12)	O6-C27-H27A	109.4
C28-C27-H27A	109.4	O6-C27-H27B	109.4
C28-C27-H27B	109.4	H27A-C27-H27B	108.0
O7-C28-C27	106.47(12)	O7-C28-H28A	110.4
C27-C28-H28A	110.4	O7-C28-H28B	110.4

C27-C28-H28B	110.4	H28A-C28-H28B	108.6
O7-C29-C30	111.81(12)	O7-C29-H29A	109.3
C30-C29-H29A	109.3	O7-C29-H29B	109.3
C30-C29-H29B	109.3	H29A-C29-H29B	107.9
O8-C30-C29	106.35(12)	O8-C30-H30A	110.5
C29-C30-H30A	110.5	O8-C30-H30B	110.5
C29-C30-H30B	110.5	H30A-C30-H30B	108.6
O8-C31-C32	111.41(12)	O8-C31-H31A	109.3
C32-C31-H31A	109.3	O8-C31-H31B	109.3
C32-C31-H31B	109.3	H31A-C31-H31B	108.0
O5-C32-C31	106.76(12)	O5-C32-H32A	110.4
C31-C32-H32A	110.4	O5-C32-H32B	110.4
C31-C32-H32B	110.4	H32A-C32-H32B	108.6
C17A-O1-C24A	111.7(2)	C24-O1-C17	113.2(3)
C24-O1-Li1	119.61(17)	C17A-O1-Li1	117.80(15)
C24A-O1-Li1	108.47(16)	C17-O1-Li1	108.49(16)
C18-O2-C19	115.3(3)	C19A-O2-C18A	111.3(2)
C18-O2-Li1	116.23(17)	C19-O2-Li1	111.34(16)
C19A-O2-Li1	114.82(15)	C18A-O2-Li1	106.54(16)
C21-O3-C20	115.7(3)	C20A-O3-C21A	113.7(3)
C20A-O3-Li1	110.0(2)	C21-O3-Li1	107.9(2)
C20-O3-Li1	113.4(2)	C21A-O3-Li1	114.4(2)
C23A-O4-C22A	113.6(2)	C22-O4-C23	110.5(2)
C23A-O4-Li1	117.39(15)	C22-O4-Li1	114.00(16)
C22A-O4-Li1	110.62(14)	C23-O4-Li1	105.64(16)
C32-O5-C25	113.33(11)	C32-O5-Li1	118.00(11)
C25-O5-Li1	109.74(10)	C26-O6-C27	113.41(11)
C26-O6-Li1	115.88(11)	C27-O6-Li1	110.45(11)
C28-O7-C29	113.66(12)	C28-O7-Li1	112.56(11)
C29-O7-Li1	110.78(10)	C30-O8-C31	113.61(11)
C30-O8-Li1	115.71(11)	C31-O8-Li1	105.82(10)

Table 6 Torsion Angles (°) for complex **41.**

C13-B1-C1-C2	54.68(14)	C9-B1-C1-C2	176.80(11)
C5-B1-C1-C2	-63.46(13)	C13-B1-C1-C8	-179.81(10)
C9-B1-C1-C8	-57.69(13)	C5-B1-C1-C8	62.05(12)
C8-C1-C2-C3	-68.08(14)	B1-C1-C2-C3	56.41(15)
C1-C2-C3-C4	-42.51(16)	C2-C3-C4-C5	40.86(16)
C3-C4-C5-C6	71.60(14)	C3-C4-C5-B1	-52.66(14)

C1-B1-C5-C6	-64.29(12)	C13-B1-C5-C6	176.56(10)
C9-B1-C5-C6	53.77(13)	C1-B1-C5-C4	61.31(12)
C13-B1-C5-C4	-57.84(13)	C9-B1-C5-C4	179.37(10)
C4-C5-C6-C7	-68.47(14)	B1-C5-C6-C7	56.19(14)
C5-C6-C7-C8	-41.44(16)	C2-C1-C8-C7	72.15(14)
B1-C1-C8-C7	-52.28(15)	C6-C7-C8-C1	39.61(16)
C1-B1-C9-C10	169.42(11)	C13-B1-C9-C10	-67.30(14)
C5-B1-C9-C10	55.79(15)	B1-C9-C10-C11	177.01(11)
C9-C10-C11-C12	-179.09(11)	C1-B1-C13-C14	60.62(14)
C9-B1-C13-C14	-61.62(14)	C5-B1-C13-C14	174.07(11)
B1-C13-C14-C15	-168.62(11)	C13-C14-C15-C16	-176.16(13)
O1-C17-C18-O2	55.4(5)	O2-C19-C20-O3	55.7(4)
O3-C21-C22-O4	61.9(5)	O4-C23-C24-O1	53.3(5)
O1-C17A-C18A-O2	-57.4(4)	O2-C19A-C20A-O3	-61.3(4)
O3-C21A-C22A-O4	-59.2(4)	O4-C23A-C24A-O1	-55.5(4)
O5-C25-C26-O6	-55.30(15)	O6-C27-C28-O7	-60.08(16)
O7-C29-C30-O8	-58.33(16)	O8-C31-C32-O5	-58.44(15)
C23-C24-O1-C17	-164.3(3)	C23-C24-O1-Li1	-34.4(4)
C18A-C17A-O1-C24A	164.4(3)	C18A-C17A-O1-Li1	37.9(3)
C23A-C24A-O1-C17A	-84.5(3)	C23A-C24A-O1-Li1	46.9(3)
C18-C17-O1-C24	86.9(4)	C18-C17-O1-Li1	-48.4(4)
C17-C18-O2-C19	-168.6(3)	C17-C18-O2-Li1	-35.6(4)
C20-C19-O2-C18	86.2(4)	C20-C19-O2-Li1	-49.0(4)
C20A-C19A-O2-C18A	167.0(3)	C20A-C19A-O2-Li1	45.9(3)
C17A-C18A-O2-C19A	-77.6(3)	C17A-C18A-O2-Li1	48.3(3)
C19A-C20A-O3-C21A	-83.0(4)	C19A-C20A-O3-Li1	46.8(4)
C22-C21-O3-C20	82.8(4)	C22-C21-O3-Li1	-45.4(4)
C19-C20-O3-C21	-160.2(3)	C19-C20-O3-Li1	-34.8(4)
C22A-C21A-O3-C20A	166.0(3)	C22A-C21A-O3-Li1	38.4(4)
C24A-C23A-O4-C22A	168.7(3)	C24A-C23A-O4-Li1	37.5(3)
C21-C22-O4-C23	-165.0(3)	C21-C22-O4-Li1	-46.2(4)
C21A-C22A-O4-C23A	-82.7(3)	C21A-C22A-O4-Li1	51.8(3)
C24-C23-O4-C22	78.5(4)	C24-C23-O4-Li1	-45.2(4)
C31-C32-O5-C25	168.22(11)	C31-C32-O5-Li1	37.99(15)
C26-C25-O5-C32	-84.64(15)	C26-C25-O5-Li1	49.62(14)
C25-C26-O6-C27	163.65(12)	C25-C26-O6-Li1	34.36(15)
C28-C27-O6-C26	-81.61(16)	C28-C27-O6-Li1	50.39(15)
C27-C28-O7-C29	166.01(12)	C27-C28-O7-Li1	39.04(15)
C30-C29-O7-C28	-81.03(16)	C30-C29-O7-Li1	46.87(15)

C29-C30-O8-C31	163.61(12)	C29-C30-O8-Li1	40.89(16)
C32-C31-O8-C30	-79.61(15)	C32-C31-O8-Li1	48.41(14)

Table 7 Hydrogen Atom Coordinates and Isotropic Displacement Parameters ($Å^2$) for complex **41.**

	x/a	y/b	z/c	U(eq)
H1A	0.7436	0.6437	0.5088	0.018
H2A	0.5534	0.6038	0.5496	0.021
H2B	0.5239	0.6665	0.5016	0.021
H3A	0.4090	0.6461	0.6399	0.02
H3B	0.4734	0.7102	0.6357	0.02
H4A	0.5202	0.6801	0.7817	0.017
H4B	0.5473	0.6132	0.7516	0.017
H5A	0.7384	0.6524	0.7993	0.014
H6A	0.8238	0.7409	0.7516	0.019
H6B	0.6863	0.7553	0.7860	0.019
H7A	0.7423	0.8045	0.6431	0.023
H7B	0.6061	0.7743	0.6418	0.023
H8A	0.6796	0.7464	0.5051	0.023
H8B	0.8197	0.7362	0.5467	0.023
H9A	0.9636	0.6236	0.6089	0.017
H9B	0.9390	0.6895	0.6443	0.017
H10A	0.9692	0.6580	0.7980	0.018
H10B	0.9873	0.5908	0.7649	0.018
H11A	1.1756	0.6189	0.6936	0.018
H11B	1.1579	0.6856	0.7283	0.018
H12A	1.3095	0.6345	0.8227	0.03
H12B	1.2043	0.5873	0.8494	0.03
H12C	1.1936	0.6552	0.8815	0.03
H13A	0.7887	0.5465	0.7215	0.018
H13B	0.6569	0.5544	0.6656	0.018
H14A	0.7716	0.5419	0.5256	0.023
H14B	0.8908	0.5227	0.5898	0.023
H15A	0.7715	0.4405	0.6439	0.027
H15B	0.6578	0.4589	0.5737	0.027
H16A	0.7733	0.3802	0.5126	0.053
H16B	0.8988	0.4198	0.5186	0.053
H16C	0.7843	0.4377	0.4487	0.053
H17A	0.0185	0.7850	0.7201	0.028
H17B	0.0645	0.7735	0.8249	0.028

H18A	-0.0634	0.8601	0.8097	0.029
H18B	0.0750	0.8757	0.8527	0.029
H19A	-0.0345	0.9667	0.7834	0.035
H19B	0.0090	0.9810	0.6819	0.035
H20A	0.1417	1.0297	0.7862	0.032
H20B	0.1718	0.9686	0.8401	0.032
H21A	0.3799	1.0116	0.8012	0.032
H21B	0.4342	0.9854	0.7088	0.032
H22A	0.4995	0.9269	0.8356	0.033
H22B	0.3544	0.9136	0.8559	0.033
H23A	0.4770	0.8184	0.8351	0.026
H23B	0.4416	0.7884	0.7380	0.026
H24A	0.2969	0.7559	0.8390	0.026
H24B	0.2661	0.8230	0.8695	0.026
H17C	0.1544	0.8339	0.8642	0.022
H17D	0.0931	0.7705	0.8359	0.022
H18C	-0.0258	0.8150	0.7141	0.024
H18D	-0.0563	0.8485	0.8074	0.024
H19C	0.1127	0.9290	0.8465	0.023
H19D	-0.0212	0.9569	0.8122	0.023
H20C	0.0795	1.0074	0.6901	0.028
H20D	0.1415	1.0282	0.7874	0.028
H21C	0.3145	0.9591	0.8480	0.028
H21D	0.3736	1.0173	0.8022	0.028
H22C	0.4870	0.9561	0.7010	0.025
H22D	0.5248	0.9381	0.8052	0.025
H23C	0.3664	0.8633	0.8669	0.022
H23D	0.4957	0.8320	0.8388	0.022
H24C	0.3840	0.7658	0.7408	0.022
H24D	0.3265	0.7612	0.8401	0.022
H25A	0.2218	0.7359	0.5980	0.028
H25B	0.2846	0.7299	0.5006	0.028
H26A	0.1459	0.8012	0.4365	0.03
H26B	0.0644	0.7501	0.4833	0.03
H27A	-0.0669	0.8813	0.5609	0.034
H27B	-0.0684	0.8417	0.4695	0.034
H28A	0.1018	0.8975	0.4160	0.033
H28B	-0.0154	0.9405	0.4351	0.033
H29A	0.2384	1.0160	0.5381	0.035

H29B	0.1738	1.0054	0.4380	0.035
H30A	0.3078	0.9234	0.4157	0.031
H30B	0.3911	0.9817	0.4394	0.031
H31A	0.5268	0.8714	0.5807	0.029
H31B	0.5270	0.8926	0.4758	0.029
H32A	0.3565	0.8285	0.4379	0.029
H32B	0.4716	0.7910	0.4844	0.029

4. X-ray Crystallographic Data for Complex 42

Table 1 Crystal data and structure refinement for complex **42.**

Empirical formula	$C_{28}H_{56}B_2Li_2O_6$
Formula weight	524.22
Temperature/K	101(2)
Crystal system	triclinic
Space group	P-1
a/Å	8.3308(4)
b/Å	9.3831(5)
c/Å	10.3351(5)
α/°	98.430(2)
β/°	106.326(2)
γ/°	90.671(2)
Volume/Å^3	765.74(7)
Z	1
ρ_{calc}g/cm^3	1.137
μ/mm^{-1}	0.074
F(000)	288
Crystal size/mm^3	0.536 × 0.482 × 0.429
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.40 to 52.84
Index ranges	$-10 \le h \le 10$, $-11 \le k \le 11$, $-8 \le l \le 12$
Reflections collected	16573
Independent reflections	3134 [R_{int} = 0.0187]
Data/restraints/parameters	3134/4/184
Goodness-of-fit on F^2	1.0323
Final R indexes [I>=2σ (I)]	R_1 = 0.0530, wR_2 = 0.1448
Final R indexes [all data]	R_1 = 0.0576, wR_2 = 0.1492
Largest diff. peak/hole / e Å^{-3}	0.561/-0.448

Table 2. Atomic coordinates and equivalent isotropic atomic displacement parameters (Å^2) for complex **42.** U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x/a	**y/b**	**z/c**	**U(eq)**

B1	0.47622(19)	0.57092(17)	0.30244(16)	0.0138(3)
O1	0.65189(12)	0.59106(11)	0.39875(10)	0.0172(3)
O2	0.41479(13)	0.41488(10)	0.28563(10)	0.0169(3)
O3	0.86952(15)	0.77137(14)	0.66953(11)	0.0300(3)
Li1	0.6796(3)	0.6374(3)	0.5846(3)	0.0234(6)
C1	0.35809(18)	0.66972(15)	0.37579(14)	0.0157(3)
C2	0.4244(2)	0.82837(16)	0.40416(15)	0.0200(3)
C3	0.4461(2)	0.88905(16)	0.27956(16)	0.0235(3)
C4	0.5258(2)	0.78653(16)	0.18794(16)	0.0211(3)
C5	0.45935(17)	0.62777(15)	0.15862(14)	0.0151(3)
C6	0.27561(19)	0.60278(17)	0.07020(15)	0.0213(3)
C7	0.14488(19)	0.66845(18)	0.13730(16)	0.0239(4)
C8	0.17471(18)	0.64691(16)	0.28675(16)	0.0197(3)
C9	0.7859(2)	0.5337(2)	0.35341(18)	0.0300(4)
C10	0.4606(2)	0.30831(17)	0.1932(2)	0.0302(4)
C11	0.9612(3)	0.7951(3)	0.81120(19)	0.0469(6)
C12	0.1017(3)	0.9019(2)	0.8296(2)	0.0476(6)
C13	0.0654(5)	0.9640(5)	0.7096(5)	0.0386(8)
C13A	0.1092(7)	0.9063(6)	0.6775(6)	0.0386(8)
C14	0.9392(3)	0.8682(2)	0.6011(2)	0.0412(5)

Table 3. Anisotropic atomic displacement parameters ($Å^2$) for complex **42.**

The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U_{11} + ... + 2 h k a^* b^* U_{12}]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
B1	0.0142(7)	0.0138(7)	0.0141(7)	0.0018(6)	0.0052(6)	-0.0003(6)
O1	0.0130(5)	0.0235(5)	0.0155(5)	0.0024(4)	0.0050(4)	0.0015(4)
O2	0.0213(5)	0.0128(5)	0.0186(5)	0.0010(4)	0.0100(4)	0.0004(4)
O3	0.0275(6)	0.0410(7)	0.0180(6)	0.0084(5)	-0.0004(5)	-0.0167(5)
Li1	0.0230(13)	0.0300(14)	0.0170(12)	0.0039(10)	0.0055(10)	-0.0072(11)
C1	0.0179(7)	0.0151(7)	0.0152(7)	0.0022(5)	0.0067(5)	0.0002(5)
C2	0.0251(8)	0.0151(7)	0.0200(7)	-0.0024(5)	0.0093(6)	0.0003(6)
C3	0.0326(9)	0.0141(7)	0.0253(8)	0.0014(6)	0.0115(7)	-0.0019(6)
C4	0.0290(8)	0.0170(7)	0.0196(7)	0.0027(6)	0.0110(6)	-0.0038(6)
C5	0.0171(7)	0.0143(7)	0.0141(7)	0.0003(5)	0.0057(5)	-0.0006(5)
C6	0.0209(8)	0.0251(8)	0.0157(7)	0.0015(6)	0.0024(6)	0.0007(6)
C7	0.0172(7)	0.0294(8)	0.0229(8)	0.0040(6)	0.0021(6)	0.0033(6)
C8	0.0156(7)	0.0202(7)	0.0247(8)	0.0026(6)	0.0085(6)	0.0023(5)
C9	0.0166(8)	0.0447(10)	0.0282(9)	0.0003(7)	0.0084(7)	0.0067(7)
C10	0.0402(10)	0.0153(7)	0.0428(10)	-0.0022(7)	0.0279(8)	0.0011(7)
C11	0.0395(11)	0.0719(15)	0.0212(9)	0.0113(9)	-0.0054(8)	-0.0242(10)

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C12	0.0451(12)	0.0393(11)	0.0417(12)	0.0010(9)	-0.0114(9)	-0.0113(9)
C13	0.0336(19)	0.029(2)	0.051(2)	0.0086(17)	0.0088(13)	-0.0103(14)
C13A	0.0336(19)	0.029(2)	0.051(2)	0.0086(17)	0.0088(13)	-0.0103(14)
C14	0.0390(11)	0.0512(12)	0.0331(10)	0.0150(9)	0.0068(8)	-0.0212(9)

Table 4. Bond lengths (Å) for complex **42.**

B1-O1	1.5125(18)	B1-O2	1.5145(18)
B1-C5	1.622(2)	B1-C1	1.624(2)
O1-C9	1.4104(18)	O1-Li1	1.854(3)
O2-C10	1.4112(18)	O2-Li1	1.853(3)
O3-C11	1.431(2)	O3-C14	1.438(2)
O3-Li1	1.924(3)	Li1-O2	1.853(3)
C1-C2	1.5391(19)	C1-C8	1.540(2)
C1-H1A	1.0	C2-C3	1.536(2)
C2-H2A	0.99	C2-H2B	0.99
C3-C4	1.538(2)	C3-H3A	0.99
C3-H3B	0.99	C4-C5	1.5399(19)
C4-H4A	0.99	C4-H4B	0.99
C5-C6	1.541(2)	C5-H5A	1.0
C6-C7	1.537(2)	C6-H6A	0.99
C6-H6B	0.99	C7-C8	1.538(2)
C7-H7A	0.99	C7-H7B	0.99
C8-H8A	0.99	C8-H8B	0.99
C9-H9A	0.98	C9-H9B	0.98
C9-H9C	0.98	C10-H10A	0.98
C10-H10B	0.98	C10-H10C	0.98
C11-C12	1.484(3)	C11-H11A	0.99
C11-H11B	0.99	C12-C13	1.406(5)
C12-C13A	1.598(6)	C12-H12A	0.99
C12-H12B	0.99	C12-H13D	1.17(3)
C13-C14	1.480(5)	C13-H13A	0.99
C13-H13B	0.99	C13A-C14	1.426(5)
C13A-H13C	1.006(19)	C13A-H13D	0.722(15)
C14-H14A	0.99	C14-H14B	0.99

Table 5. Bond angles (°) for complex **42.**

O1-B1-O2	108.83(11)	O1-B1-C5	113.52(11)

O2-B1-C5	113.42(11)	O1-B1-C1	106.97(11)
O2-B1-C1	107.74(11)	C5-B1-C1	105.97(11)
C9-O1-B1	119.30(11)	C9-O1-Li1	119.11(13)
B1-O1-Li1	118.84(12)	C10-O2-B1	120.54(11)
C10-O2-Li1	120.42(13)	B1-O2-Li1	117.63(12)
C11-O3-C14	108.64(14)	C11-O3-Li1	126.03(13)
C14-O3-Li1	125.29(13)	O2-Li1-O1	138.60(16)
O2-Li1-O3	110.51(14)	O1-Li1-O3	110.78(13)
C2-C1-C8	112.99(12)	C2-C1-B1	109.34(11)
C8-C1-B1	109.66(11)	C2-C1-H1A	108.2
C8-C1-H1A	108.2	B1-C1-H1A	108.2
C3-C2-C1	115.15(12)	C3-C2-H2A	108.5
C1-C2-H2A	108.5	C3-C2-H2B	108.5
C1-C2-H2B	108.5	H2A-C2-H2B	107.5
C2-C3-C4	114.39(12)	C2-C3-H3A	108.7
C4-C3-H3A	108.7	C2-C3-H3B	108.7
C4-C3-H3B	108.7	H3A-C3-H3B	107.6
C3-C4-C5	115.45(12)	C3-C4-H4A	108.4
C5-C4-H4A	108.4	C3-C4-H4B	108.4
C5-C4-H4B	108.4	H4A-C4-H4B	107.5
C4-C5-C6	113.93(12)	C4-C5-B1	108.91(11)
C6-C5-B1	108.72(11)	C4-C5-H5A	108.4
C6-C5-H5A	108.4	B1-C5-H5A	108.4
C7-C6-C5	115.56(12)	C7-C6-H6A	108.4
C5-C6-H6A	108.4	C7-C6-H6B	108.4
C5-C6-H6B	108.4	H6A-C6-H6B	107.5
C6-C7-C8	114.42(12)	C6-C7-H7A	108.7
C8-C7-H7A	108.7	C6-C7-H7B	108.7
C8-C7-H7B	108.7	H7A-C7-H7B	107.6
C7-C8-C1	115.31(12)	C7-C8-H8A	108.4
C1-C8-H8A	108.4	C7-C8-H8B	108.4
C1-C8-H8B	108.4	H8A-C8-H8B	107.5
O1-C9-H9A	109.5	O1-C9-H9B	109.5
H9A-C9-H9B	109.5	O1-C9-H9C	109.5
H9A-C9-H9C	109.5	H9B-C9-H9C	109.5
O2-C10-H10A	109.5	O2-C10-H10B	109.5
H10A-C10-H10B	109.5	O2-C10-H10C	109.5
H10A-C10-H10C	109.5	H10B-C10-H10C	109.5
O3-C11-C12	107.55(16)	O3-C11-H11A	110.2

C12-C11-H11A	110.2	O3-C11-H11B	110.2
C12-C11-H11B	110.2	H11A-C11-H11B	108.5
C13-C12-C11	106.1(2)	C11-C12-C13A	103.6(2)
C13-C12-H12A	110.5	C11-C12-H12A	110.5
C13-C12-H12B	110.5	C11-C12-H12B	110.5
H12A-C12-H12B	108.7	C11-C12-H13D	121.5(11)
C13A-C12-H13D	24.5(9)	C12-C13-C14	108.5(3)
C12-C13-H13A	110.0	C14-C13-H13A	110.0
C12-C13-H13B	110.0	C14-C13-H13B	110.0
H13A-C13-H13B	108.4	C14-C13A-C12	101.4(3)
C14-C13A-H13C	111.(3)	C12-C13A-H13C	108.(3)
C14-C13A-H13D	142.(2)	C12-C13A-H13D	42.(3)
H13C-C13A-H13D	94.(4)	C13A-C14-O3	108.7(3)
O3-C14-C13	106.0(2)	O3-C14-H14A	110.5
C13-C14-H14A	110.5	O3-C14-H14B	110.5
C13-C14-H14B	110.5	H14A-C14-H14B	108.7

Table 6. Torsion angles (°) for complex **42.**

O2-B1-O1-C9	72.71(16)	C5-B1-O1-C9	-54.62(17)
C1-B1-O1-C9	-171.15(13)	O2-B1-O1-Li1	-88.38(15)
C5-B1-O1-Li1	144.29(14)	C1-B1-O1-Li1	27.76(17)
O1-B1-O2-C10	-81.95(16)	C5-B1-O2-C10	45.44(18)
C1-B1-O2-C10	162.40(13)	O1-B1-O2-Li1	84.54(15)
C5-B1-O2-Li1	-148.08(13)	C1-B1-O2-Li1	-31.11(16)
C9-O1-Li1-O2	-119.3(2)	B1-O1-Li1-O2	41.8(3)
C9-O1-Li1-O3	56.3(2)	B1-O1-Li1-O3	-142.55(13)
O1-B1-C1-C2	59.08(14)	O2-B1-C1-C2	175.95(11)
C5-B1-C1-C2	-62.34(14)	O1-B1-C1-C8	-176.52(11)
O2-B1-C1-C8	-59.65(14)	C5-B1-C1-C8	62.06(14)
C8-C1-C2-C3	-68.54(16)	B1-C1-C2-C3	53.88(17)
C1-C2-C3-C4	-43.17(19)	C2-C3-C4-C5	43.47(19)
C3-C4-C5-C6	67.38(17)	C3-C4-C5-B1	-54.17(17)
O1-B1-C5-C4	-54.87(15)	O2-B1-C5-C4	-179.77(11)
C1-B1-C5-C4	62.24(14)	O1-B1-C5-C6	-179.54(11)
O2-B1-C5-C6	55.56(15)	C1-B1-C5-C6	-62.43(14)
C4-C5-C6-C7	-67.07(17)	B1-C5-C6-C7	54.59(16)
C5-C6-C7-C8	-43.35(19)	C6-C7-C8-C1	42.28(19)
C2-C1-C8-C7	69.26(16)	B1-C1-C8-C7	-52.99(16)
C14-O3-C11-C12	-4.8(3)	Li1-O3-C11-C12	177.24(18)

O3-C11-C12-C13	14.5(3)	O3-C11-C12-C13A	-14.2(3)
C11-C12-C13-C14	-18.4(4)	C11-C12-C13A-C14	27.9(4)
C12-C13A-C14-O3	-31.9(4)	C11-O3-C14-C13A	24.7(3)
Li1-O3-C14-C13A	-157.2(3)	C11-O3-C14-C13	-6.3(3)
Li1-O3-C14-C13	171.8(2)	C12-C13-C14-O3	15.7(4)

Table 7. Hydrogen atomic coordinates and isotropic atomic displacement parameters ($Å^2$) for complex **42.**

	x/a	**y/b**	**z/c**	**U(eq)**
H1A	0.3653	0.6375	0.4652	0.019
H2A	0.3464	0.8883	0.4422	0.024
H2B	0.5341	0.8379	0.4749	0.024
H3A	0.5166	0.9801	0.3118	0.028
H3B	0.3348	0.9126	0.2241	0.028
H4A	0.6482	0.7905	0.2316	0.025
H4B	0.5076	0.8225	0.0998	0.025
H5A	0.5308	0.5693	0.1102	0.018
H6A	0.2645	0.6433	-0.0149	0.026
H6B	0.2492	0.4975	0.0443	0.026
H7A	0.0328	0.6249	0.0836	0.029
H7B	0.1442	0.7732	0.1332	0.029
H8A	0.1339	0.5477	0.2879	0.024
H8B	0.1065	0.7146	0.3286	0.024
H9A	0.7778	0.5585	0.2630	0.045
H9B	0.7806	0.4285	0.3479	0.045
H9C	0.8923	0.5743	0.4178	0.045
H10A	0.5268	0.2378	0.2435	0.045
H10B	0.5274	0.3538	0.1445	0.045
H10C	0.3594	0.2594	0.1275	0.045
H11A	1.0049	0.7036	0.8399	0.056
H11B	0.8875	0.8325	0.8672	0.056
H12A	1.1102	0.9766	0.9099	0.057
H12B	1.2090	0.8538	0.8433	0.057
H13A	1.1685	0.9756	0.6816	0.046
H13B	1.0211	1.0604	0.7256	0.046
H13C	1.142(6)	1.008(3)	0.672(5)	0.046
H13D	1.167(3)	0.907(3)	0.744(2)	0.046

	x/a	y/b	z/c	U(eq)
H14A	0.8507	0.9251	0.5501	0.049
H14B	0.9924	0.8136	0.5362	0.049

5. NMR Spectra

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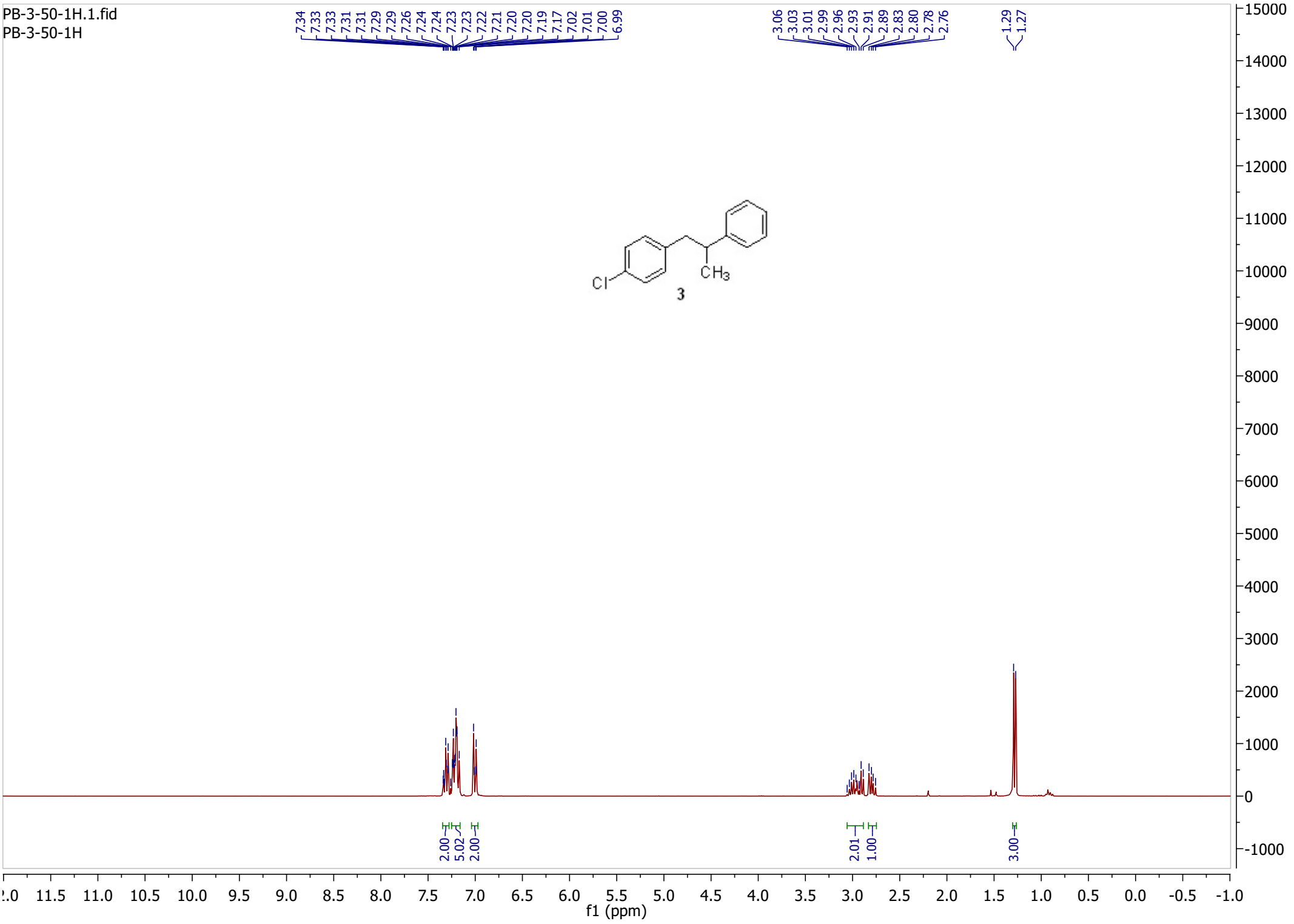
PB-3-50-1H



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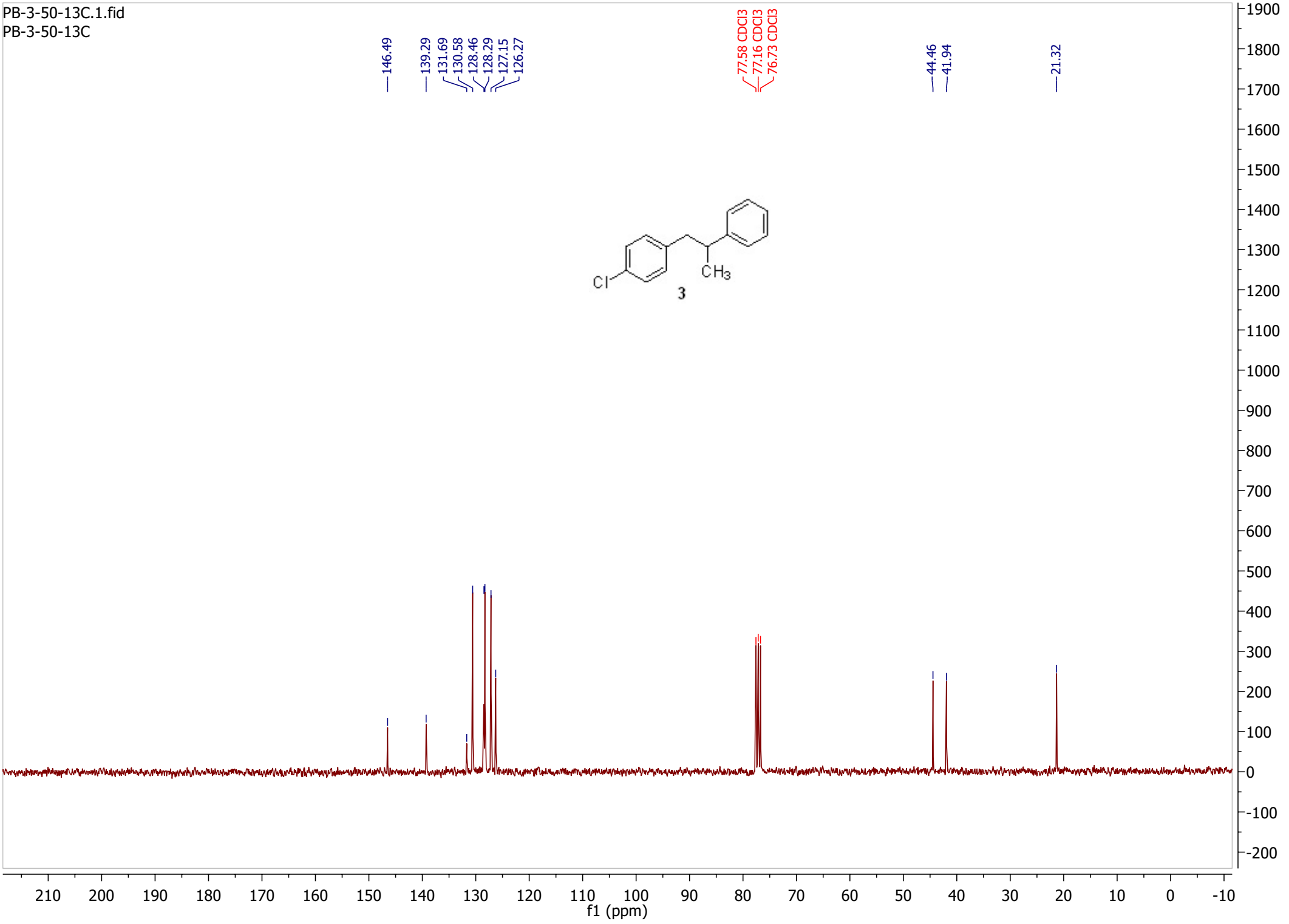

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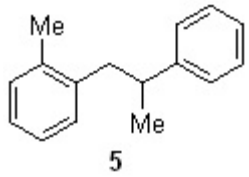

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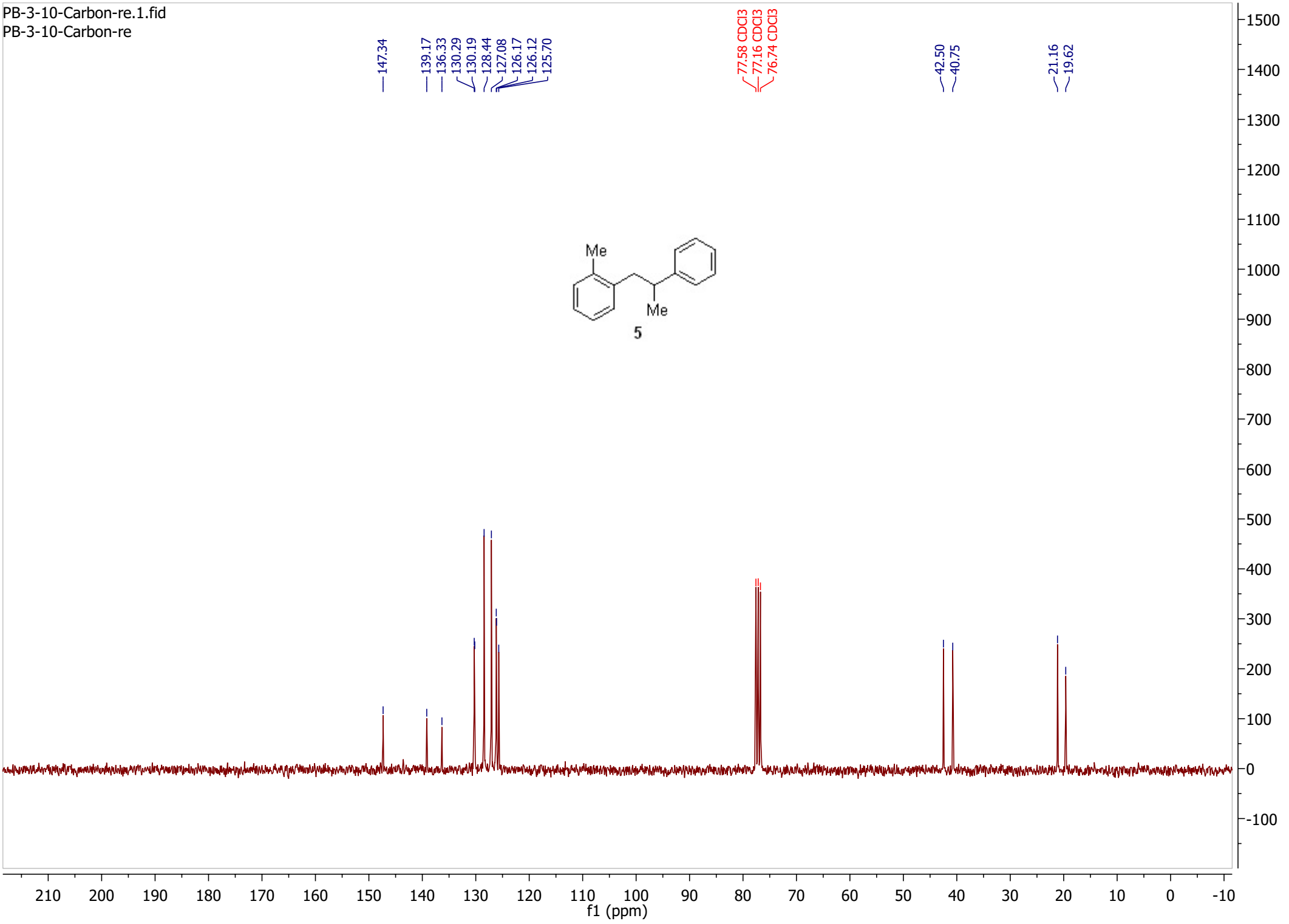

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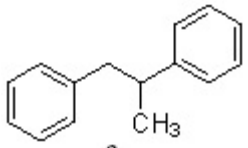

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f1 (ppm)

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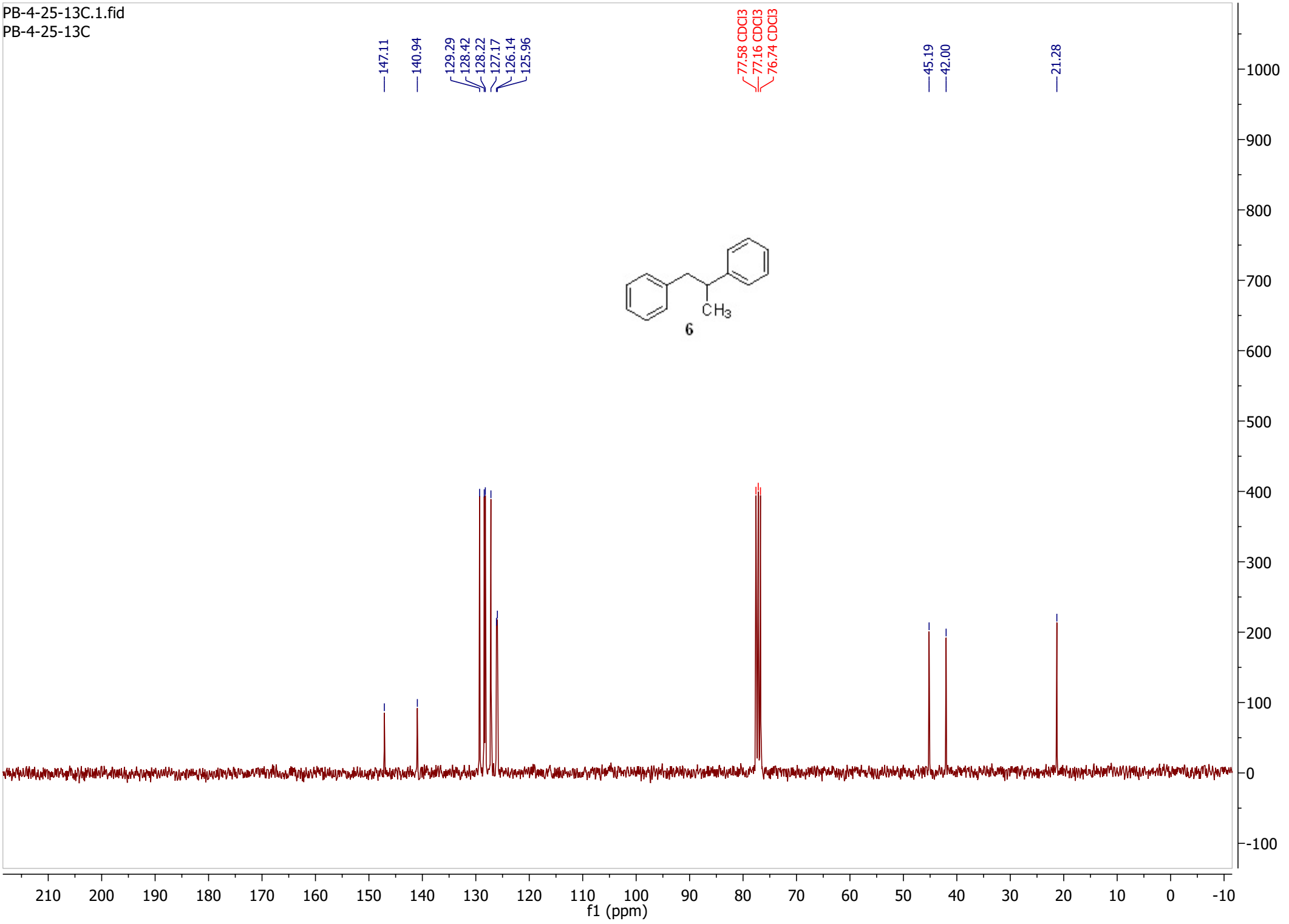

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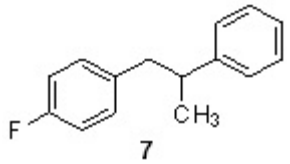

7

f1 (ppm)

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PB-4-26-13C

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130.62
130.52
128.45
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114.79
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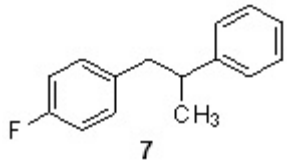
77.16 CDCl3
76.74 CDCl3
44.34
42.13
21.28



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f1 (ppm)

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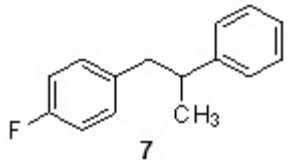

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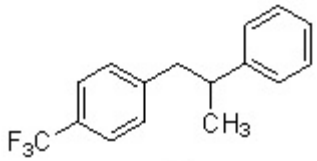
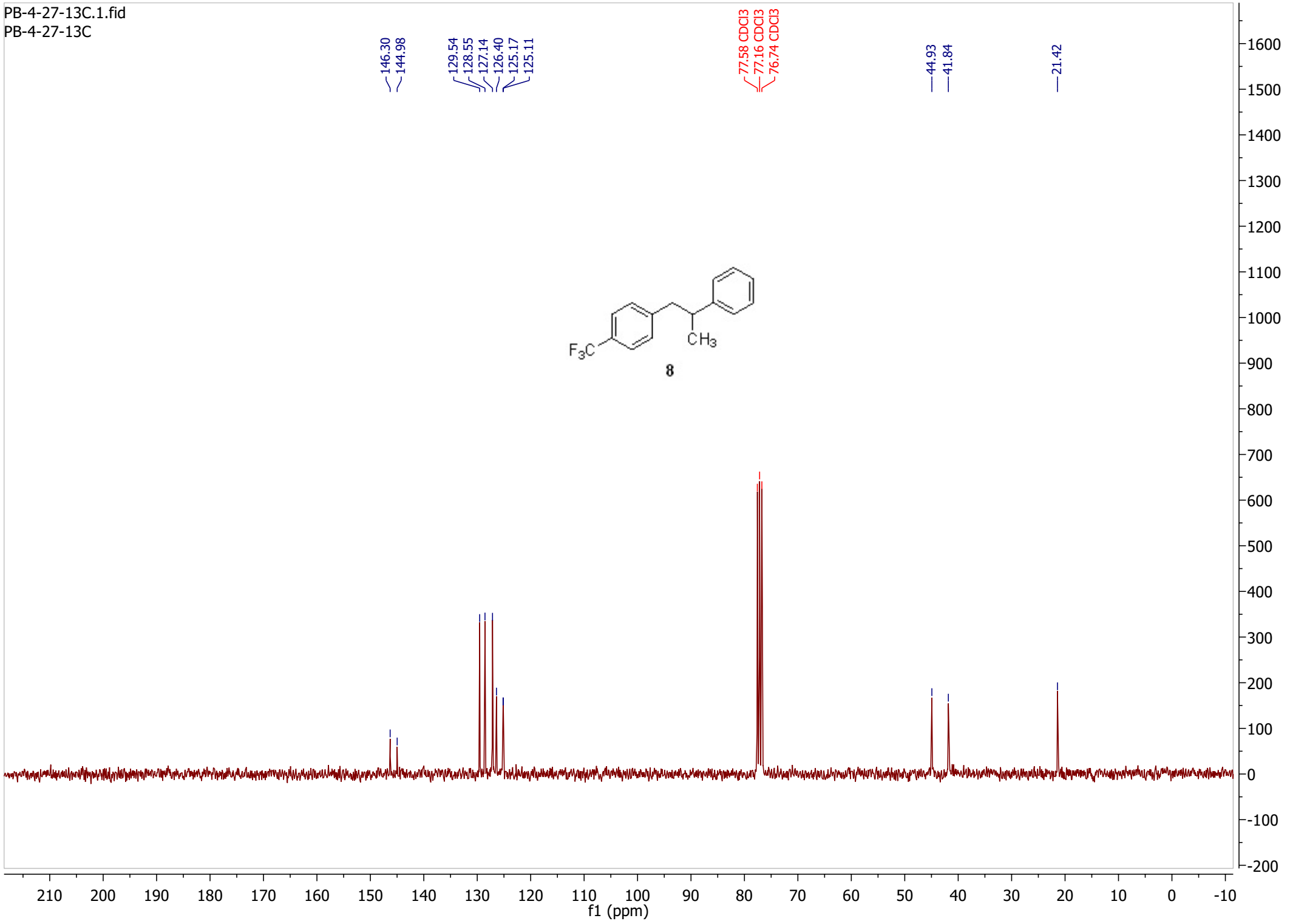
PB-4-27-1H.1.fid
PB-4-27-1H



8

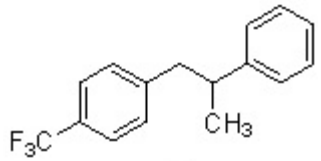
f1 (ppm)

PB-4-27-13C.1.fid
PB-4-27-13C



PB-4-27-19F.1.fid
PB-4-27-19F

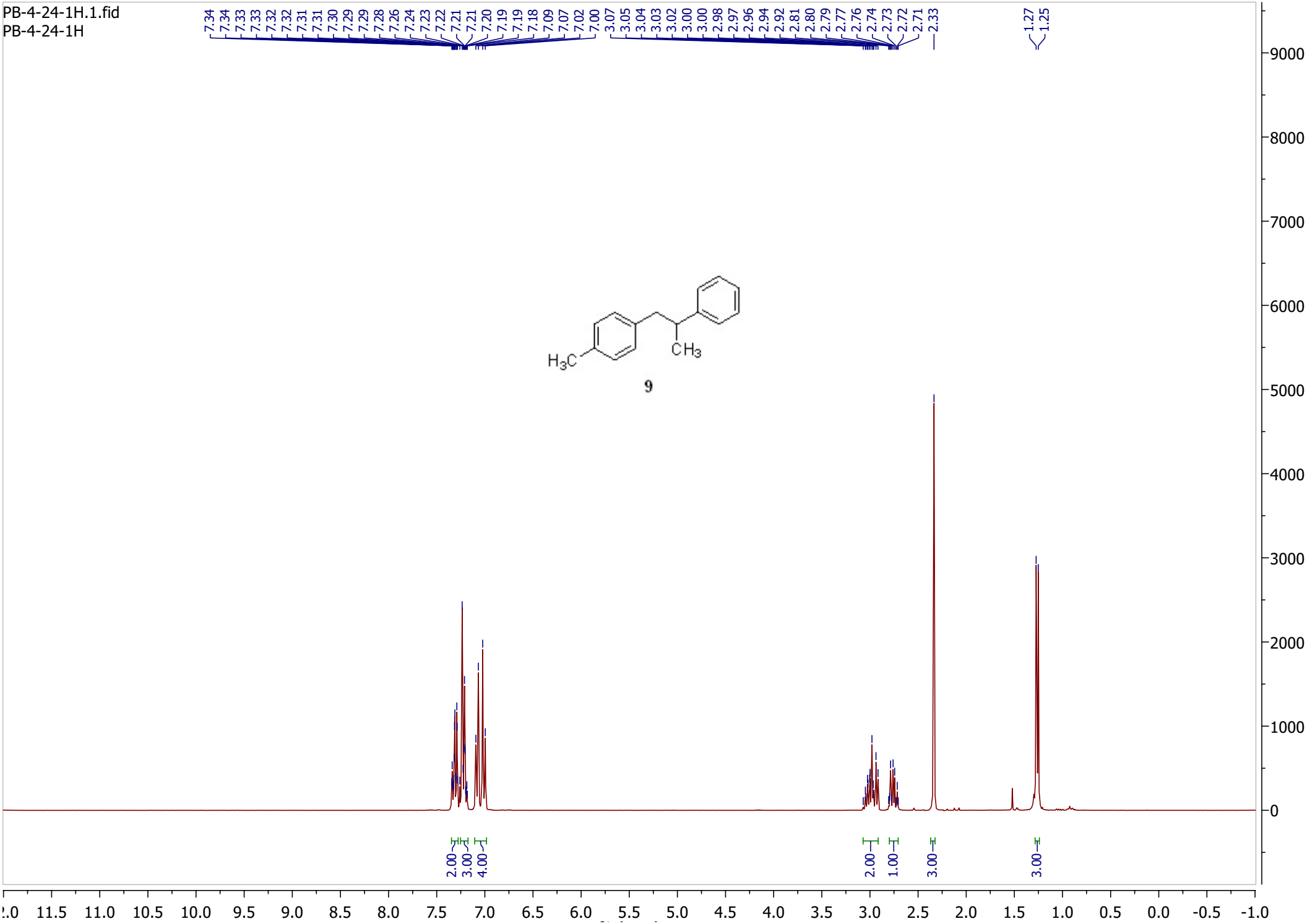
-60.70



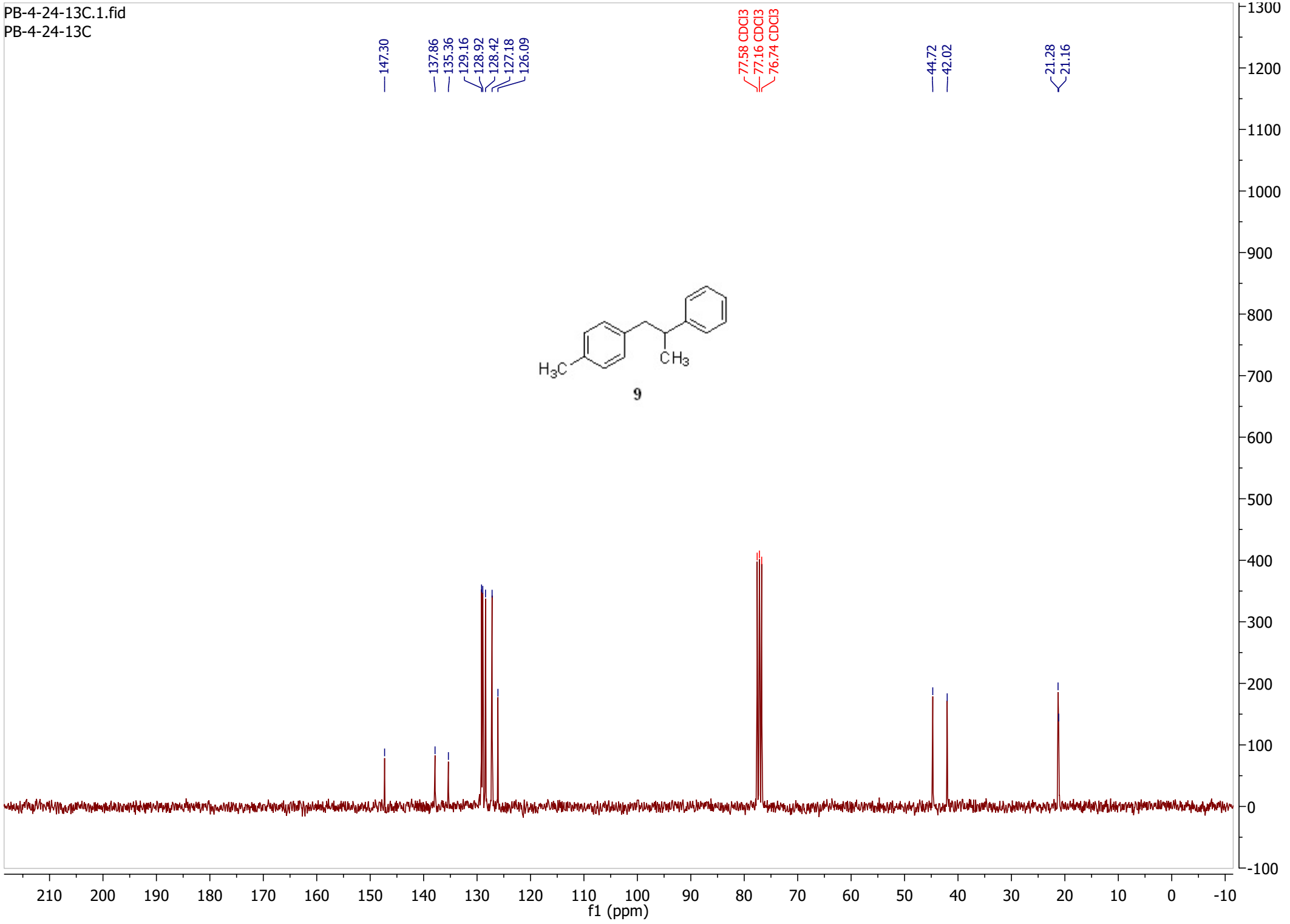
8

f1 (ppm)

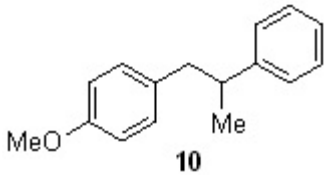
PB-4-24-1H.1.fid
PB-4-24-1H



PB-4-24-13C.1.fid
PB-4-24-13C



PB-3-25-1H-1.1.fid
PB-3-25-1H-1

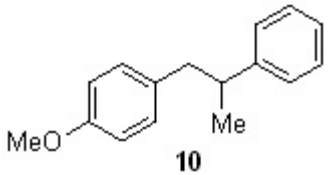


10

f1 (ppm)

PB-3-25-13C-1.1.fid
PB-3-25-13C-1

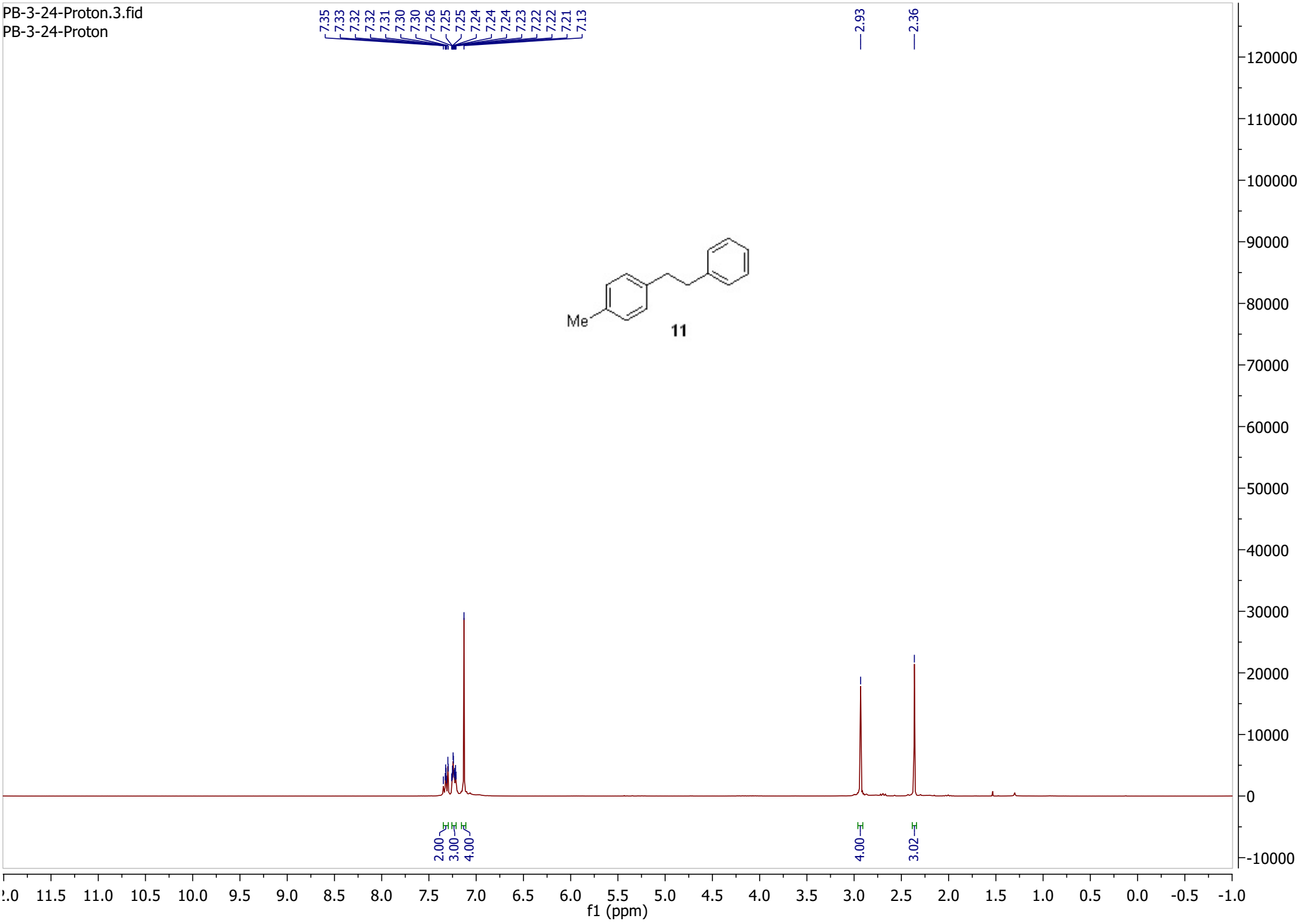
157.89
147.18
133.04
130.16
128.39
127.20
126.08
113.61
77.59 CDCl3
77.16 CDCl3
76.74 CDCl3
55.32
44.26
42.16
21.23



10

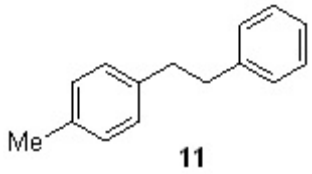
f1 (ppm)

PB-3-24-Proton.3.fid
PB-3-24-Proton



PB-3-24-Carbon.3.fid
PB-3-9-Carbon

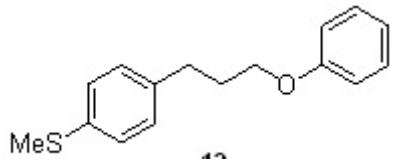
142.05
138.86
135.47
129.15
128.58
128.44
126.00
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
38.23
37.65
21.17



11

f1 (ppm)

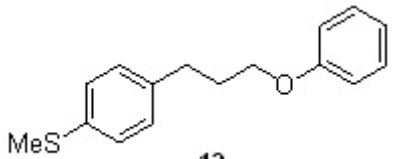
PB-3-57-Proton-4.1.fid
PB-3-57-Proton-4



12

PB-3-57-carbon-4.1.fid
PB-3-57-carbon-4

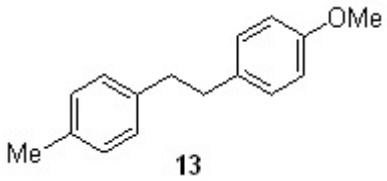
159.09
138.75
135.58
129.55
129.20
127.28
120.73
114.63
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
66.74
31.73
30.95
16.41



12

f1 (ppm)

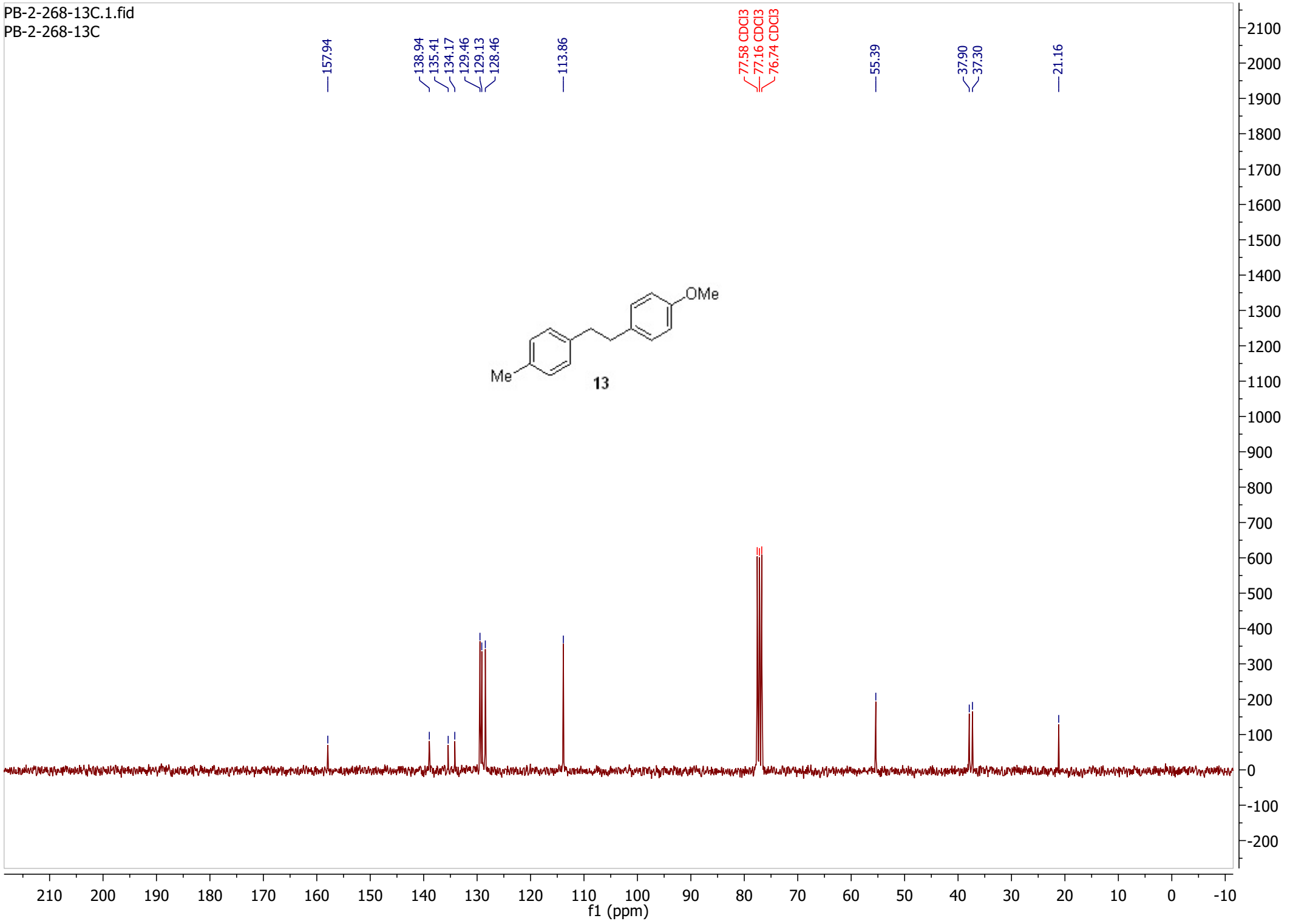
PB-2-268-proton.1.fid
PB-2-268-proton



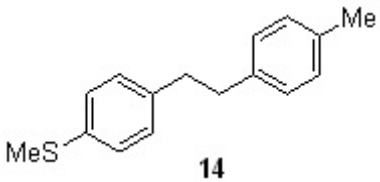
13

f1 (ppm)

PB-2-268-13C.1.fid
PB-2-268-13C



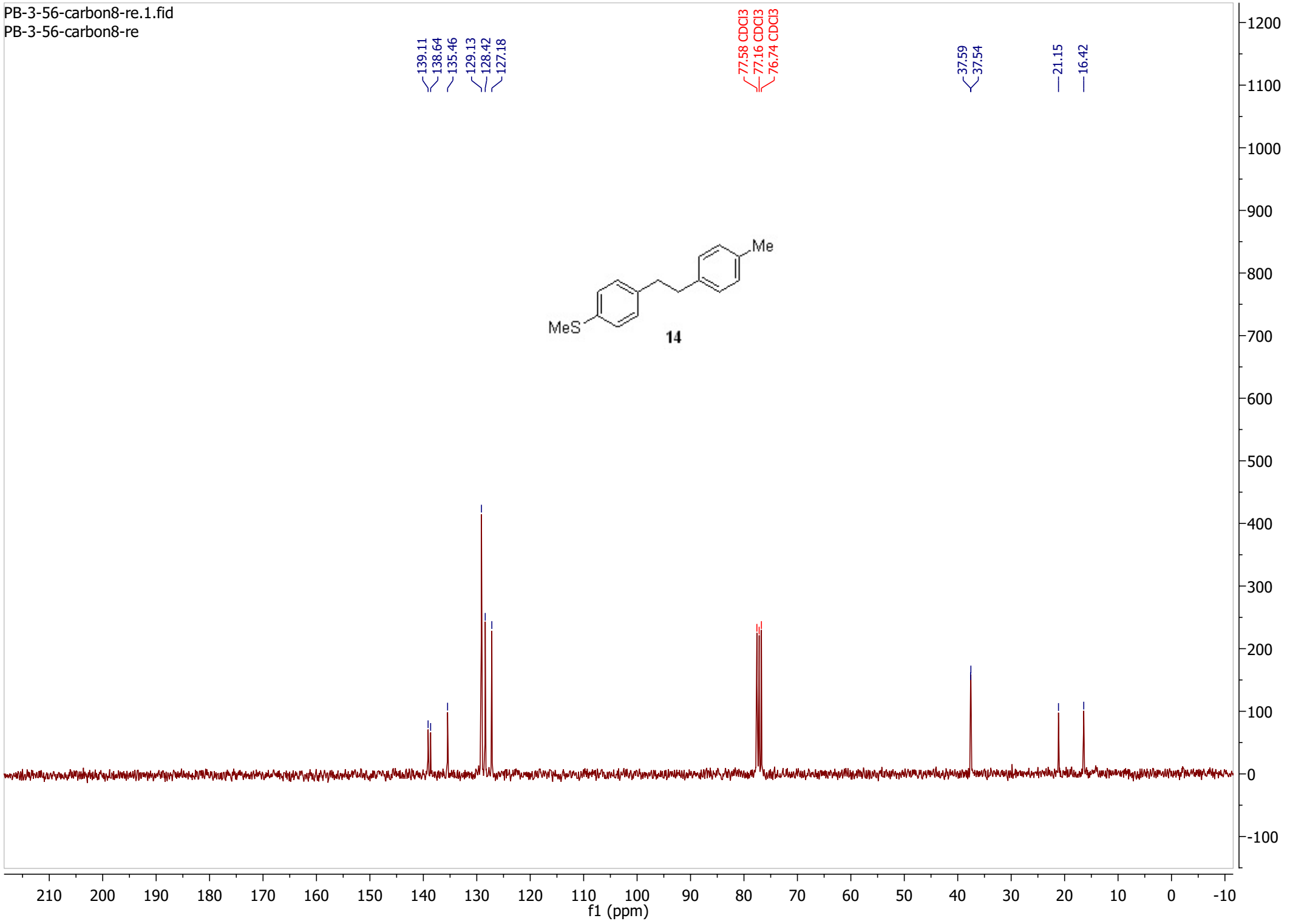
PB-3-56-proton8.1.fid
PB-3-56-proton8



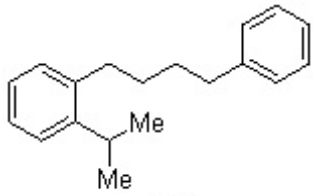
14

f1 (ppm)

PB-3-56-carbon8-re.1.fid
PB-3-56-carbon8-re



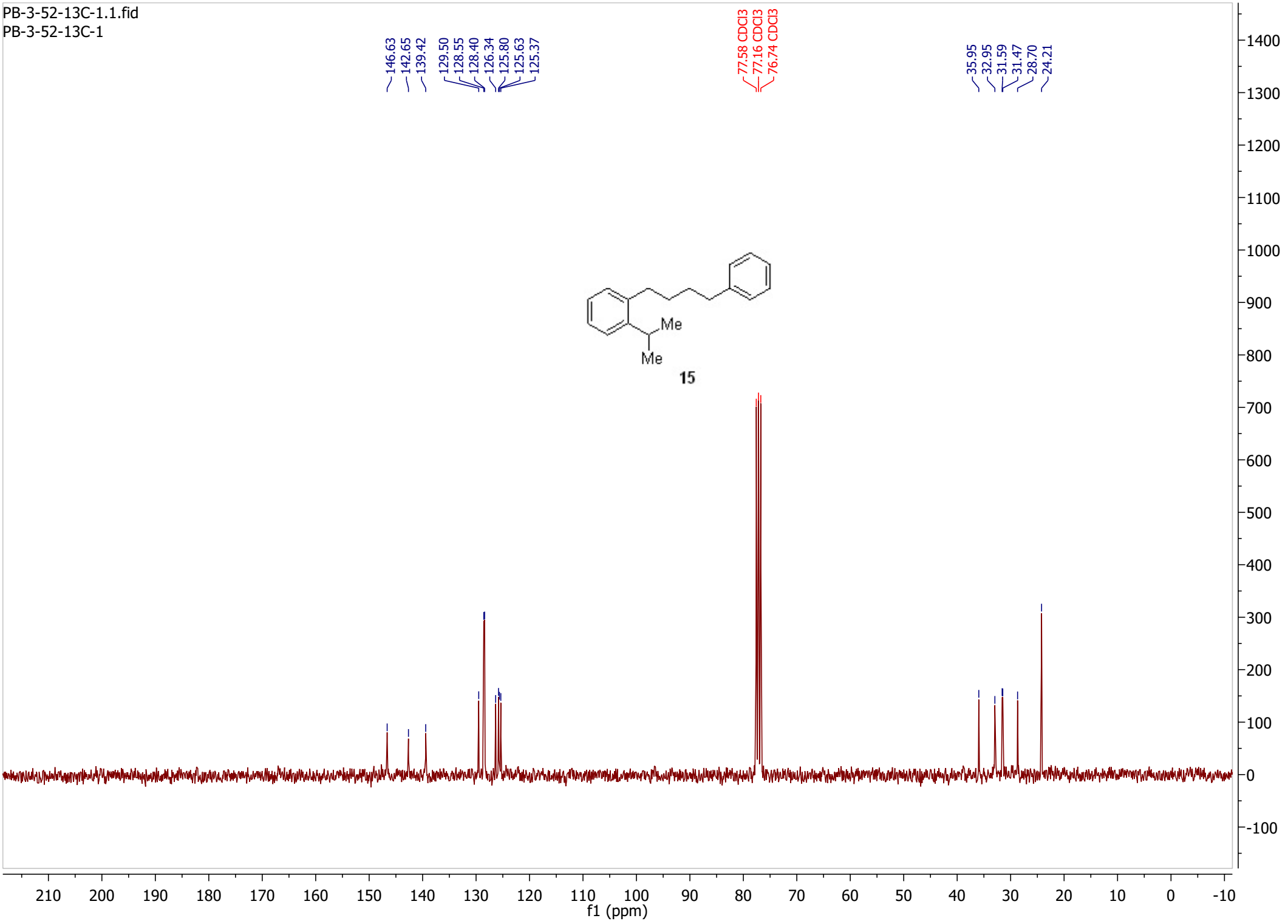
PB-3-52-1H-2.1.fid
PB-3-52-1H-2



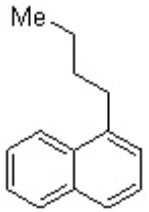
15

f1 (ppm)

PB-3-52-13C-1.1.fid
PB-3-52-13C-1



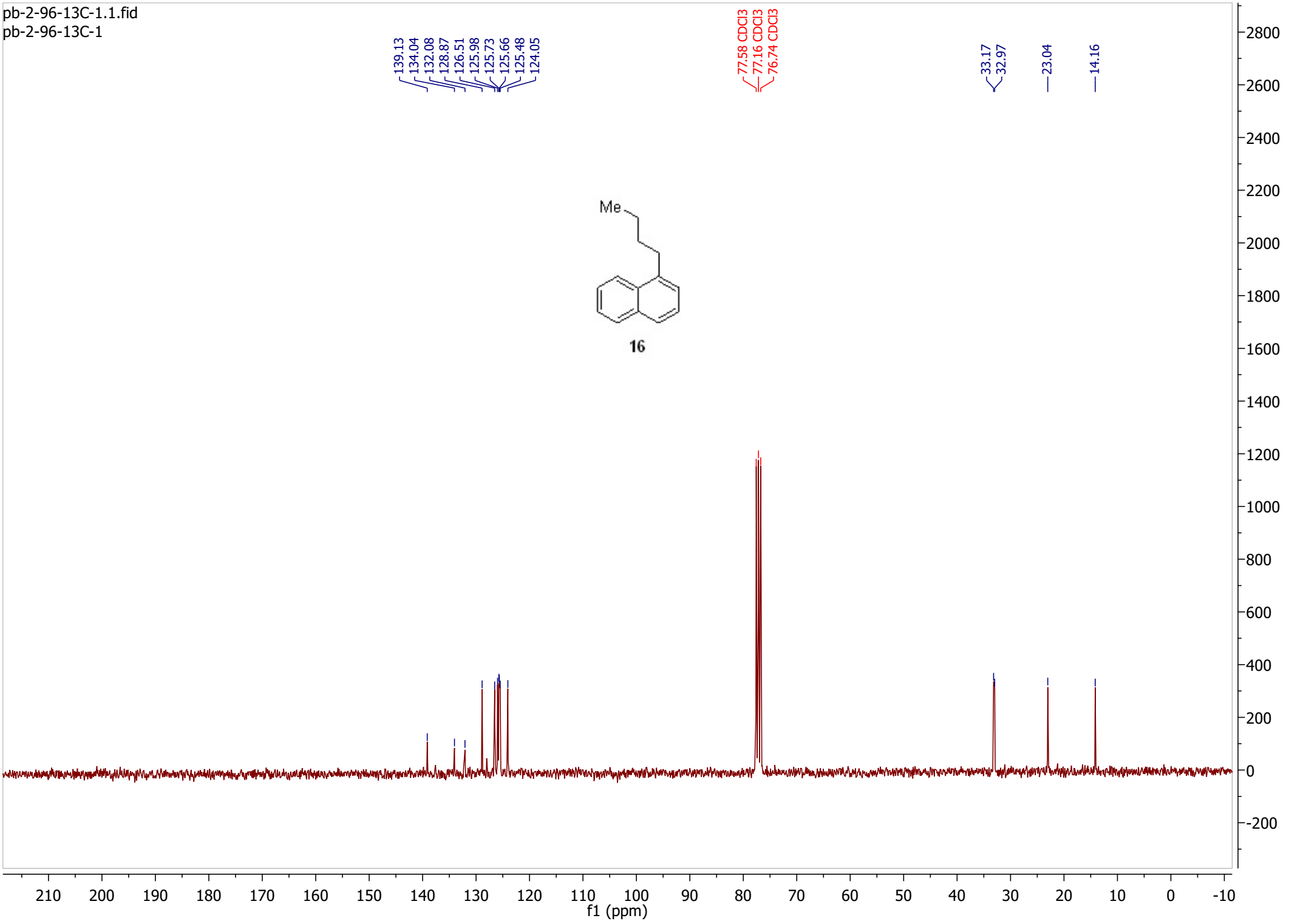
pb-2-96-proton-1.1.fid
pb-2-96-proton-1



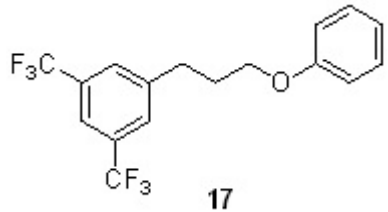
16

f1 (ppm)

pb-2-96-13C-1.1.fid
pb-2-96-13C-1



PB-2-253-prot.1.fid
PB-2-253-prot

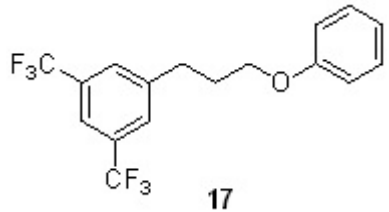


17

f1 (ppm)

PB-2-253-13C.1.fid
PB-2-253-13C

158.88
144.23
132.46
132.02
131.59
131.15
129.67
128.83
125.37
121.76
121.07
120.30
120.24
120.19
114.62
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
66.52
32.28
30.68

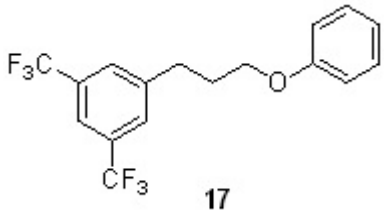


17

f1 (ppm)

PB-2-253-19F.1.fid
PB-2-253-19F

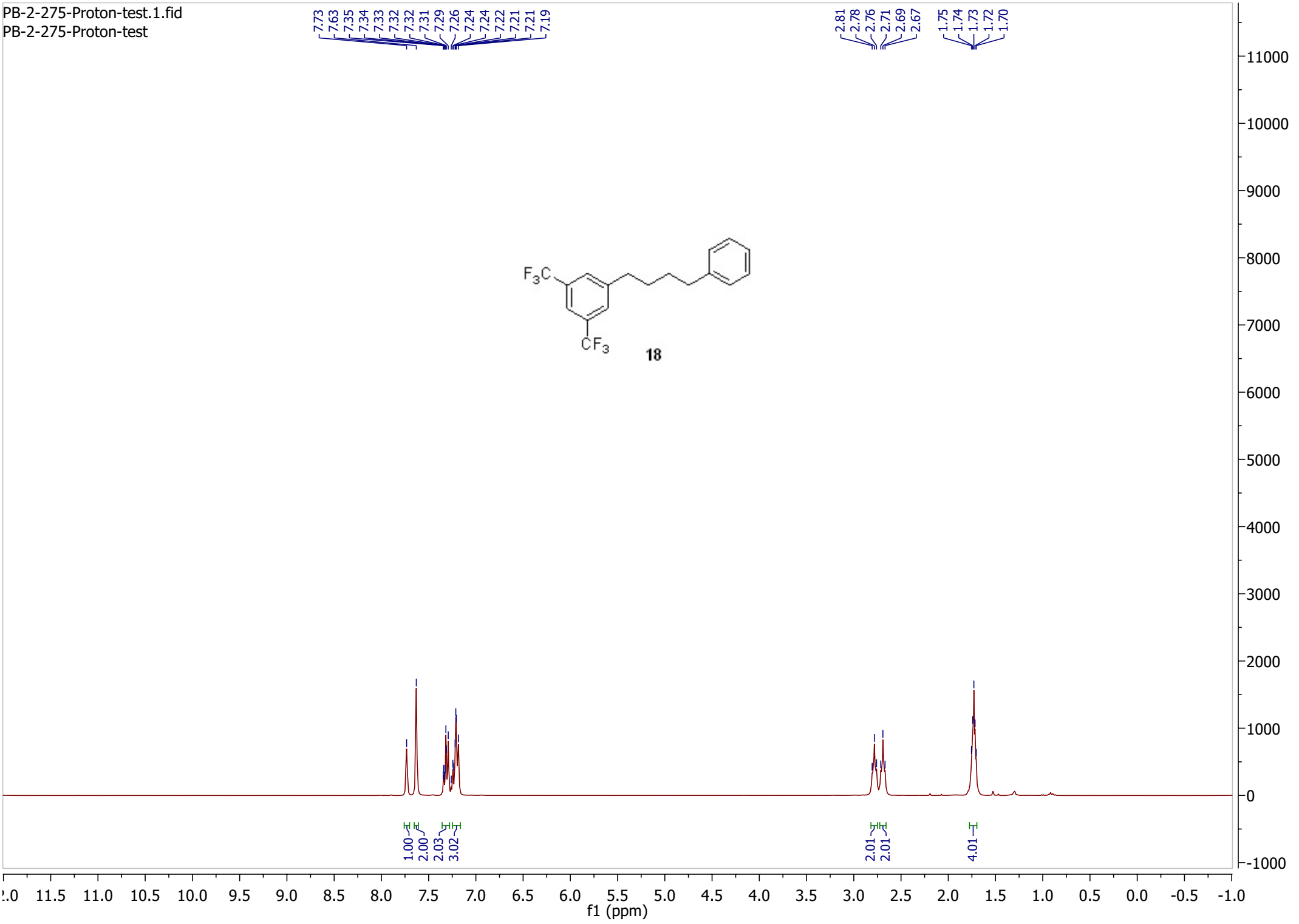
-61.29



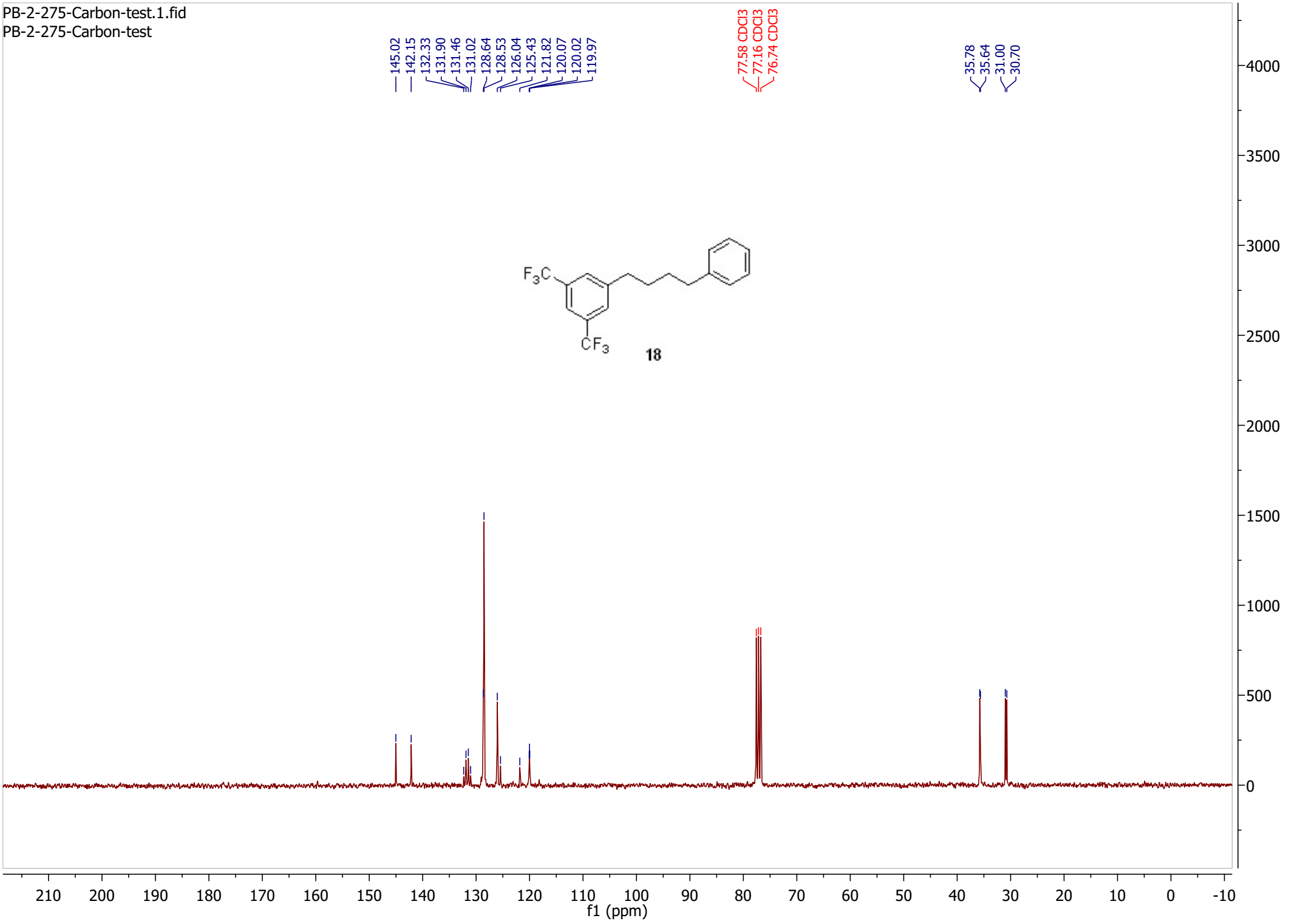
17

f1 (ppm)

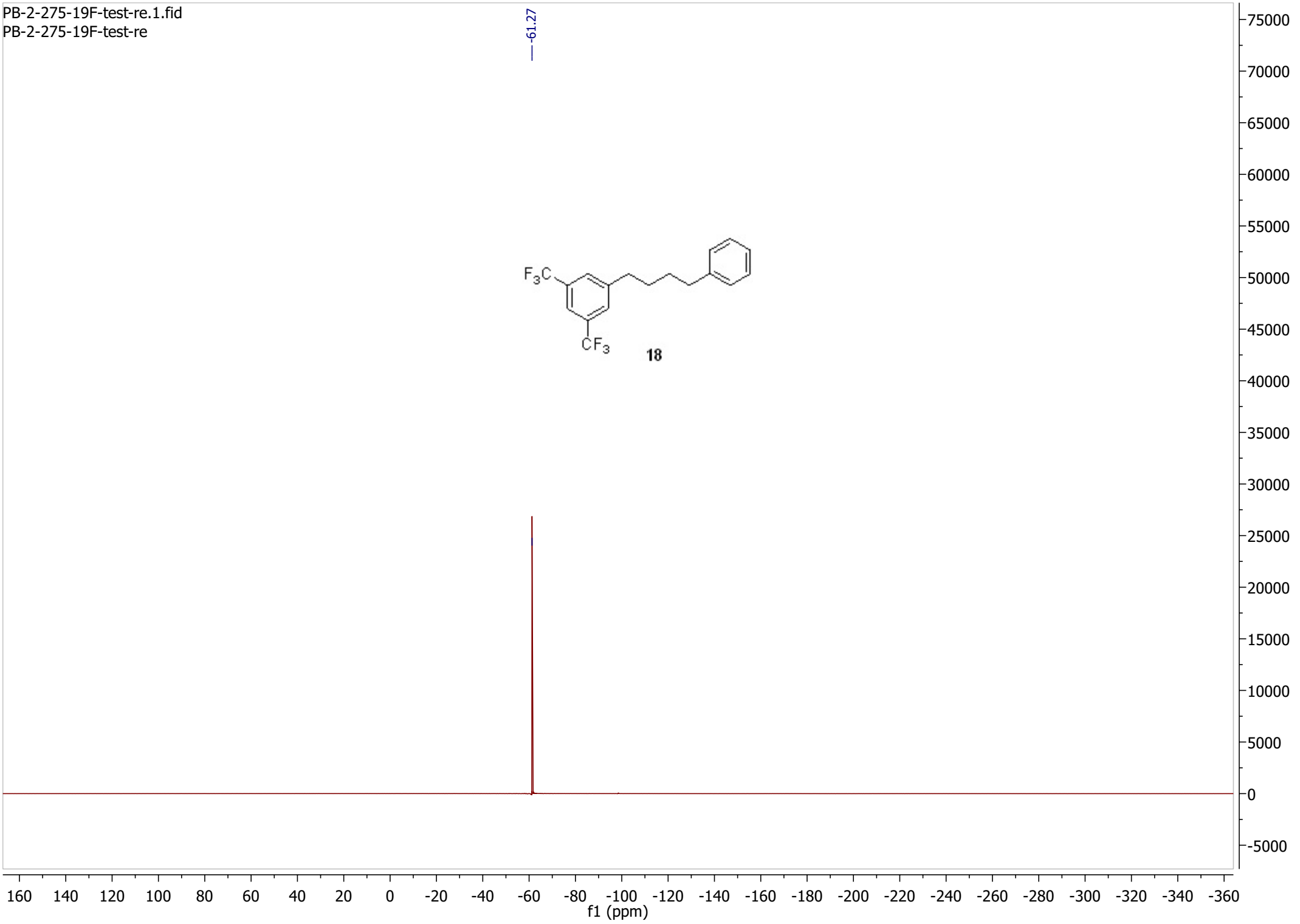
PB-2-275-Proton-test.1.fid
PB-2-275-Proton-test



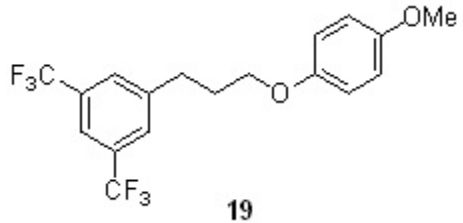
PB-2-275-Carbon-test.1.fid
PB-2-275-Carbon-test



PB-2-275-19F-test-re.1.fid
PB-2-275-19F-test-re

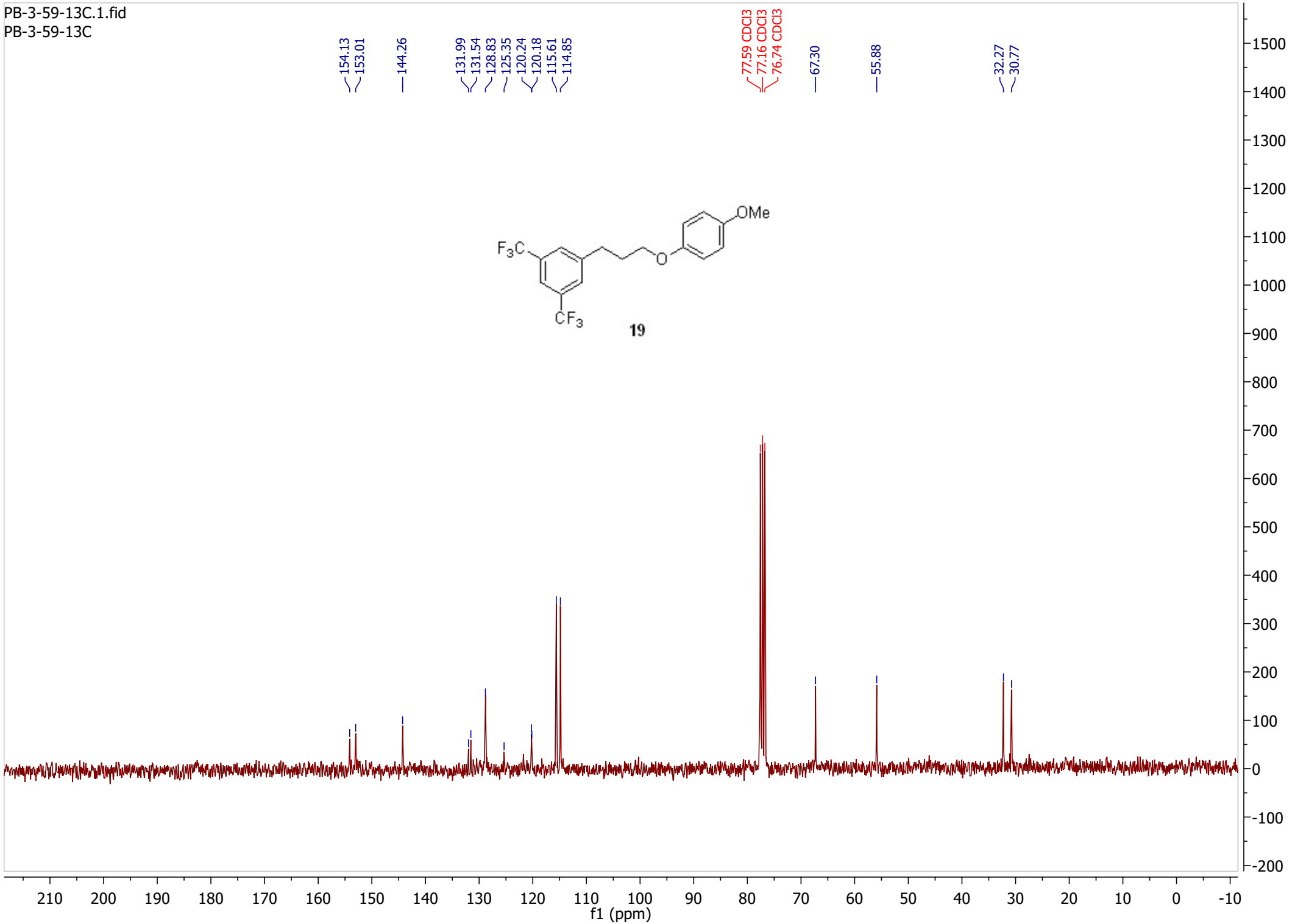


PB-3-59-1H-3.1.fid
PB-3-59-1H-3



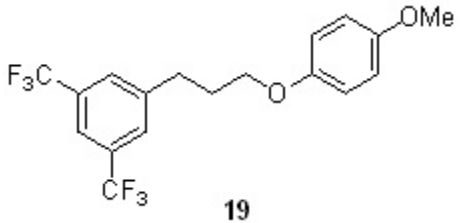
19

PB-3-59-13C.1.fid
PB-3-59-13C



PB-3-59-19F.1.fid
PB-3-59-19F

-61.22



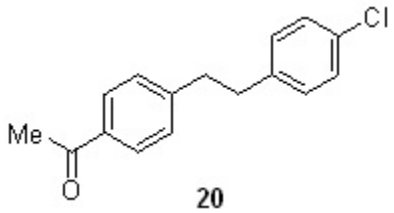
19

4500
4000
3500
3000
2500
2000
1500
1000
500
0

0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190

f1 (ppm)

PB-3-unk-3-proton.1.fid
PB-3-unk-3-proton

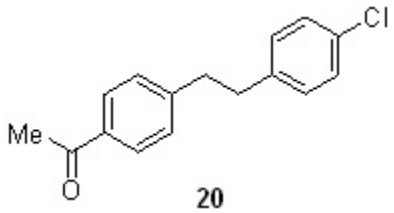


20

f1 (ppm)

PB-3-unk-3-Carbon.1.fid
PB-3-unk-3-Carbon

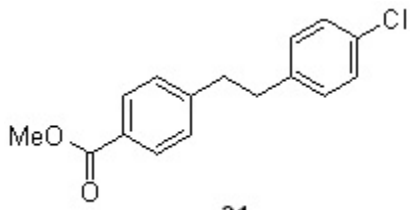
197.81
146.96
139.50
135.28
131.86
129.87
128.77
128.59
128.52
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
37.68
36.72
26.62



20

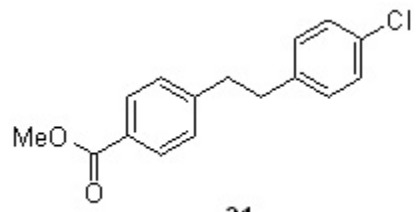
f1 (ppm)

PB-3-80-proton-1.1.fid
PB-3-80-proton-1



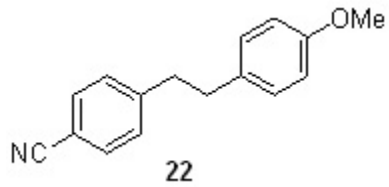
21

PB-3-80-13C-re.1.fid
PB-3-80-13C-re



21

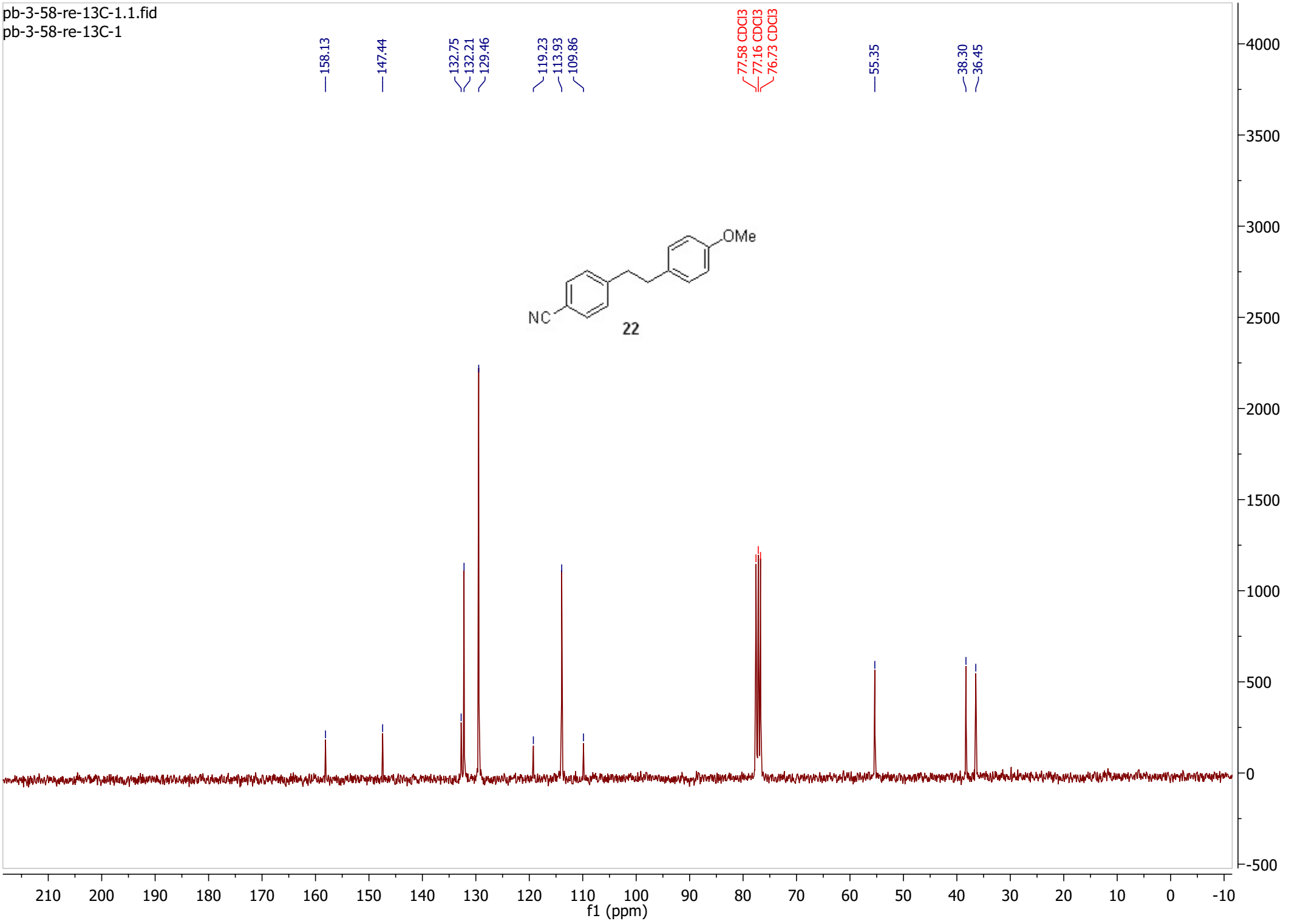
pb-3-58-re-1H-1.1.fid
pb-3-58-re-1H-1



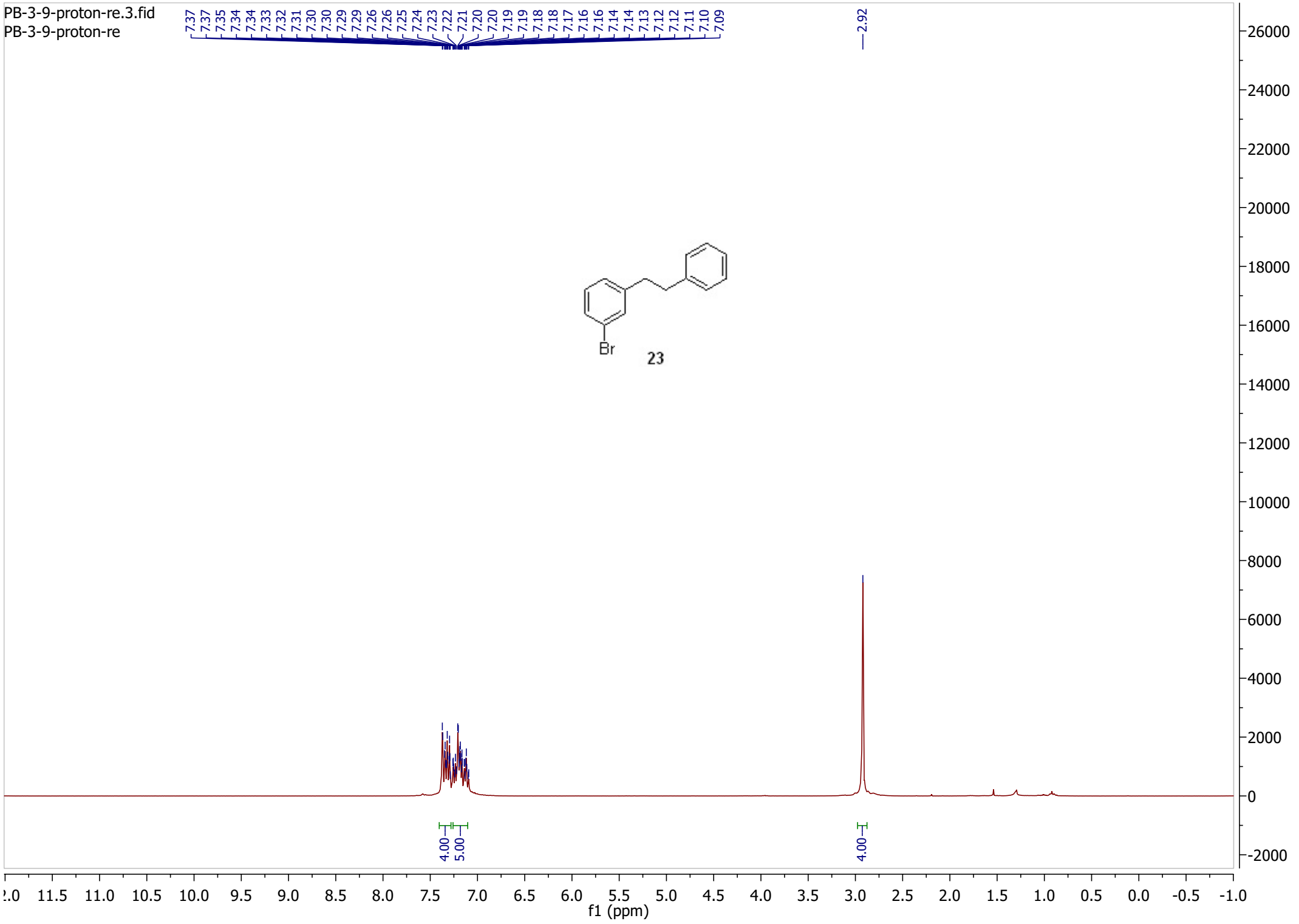
22

f1 (ppm)

pb-3-58-re-13C-1.1.fid
pb-3-58-re-13C-1

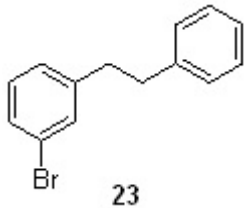


PB-3-9-proton-re.3.fid
PB-3-9-proton-re



PB-3-9-Carbon-re.3.fid
PB-3-9-Carbon-re

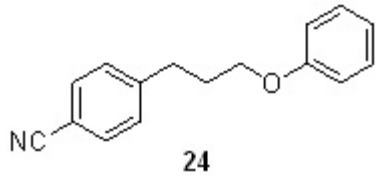
144.17
141.31
131.64
130.00
129.17
128.54
127.27
126.22
122.50
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
37.78
37.68



23

f1 (ppm)

PB-3-30-1H-3

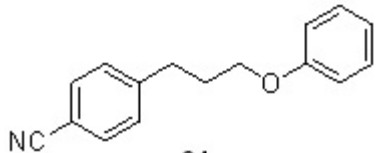


24

f1 (ppm)

PB-3-30-13C-re.1.fid
PB-3-30-13C-re

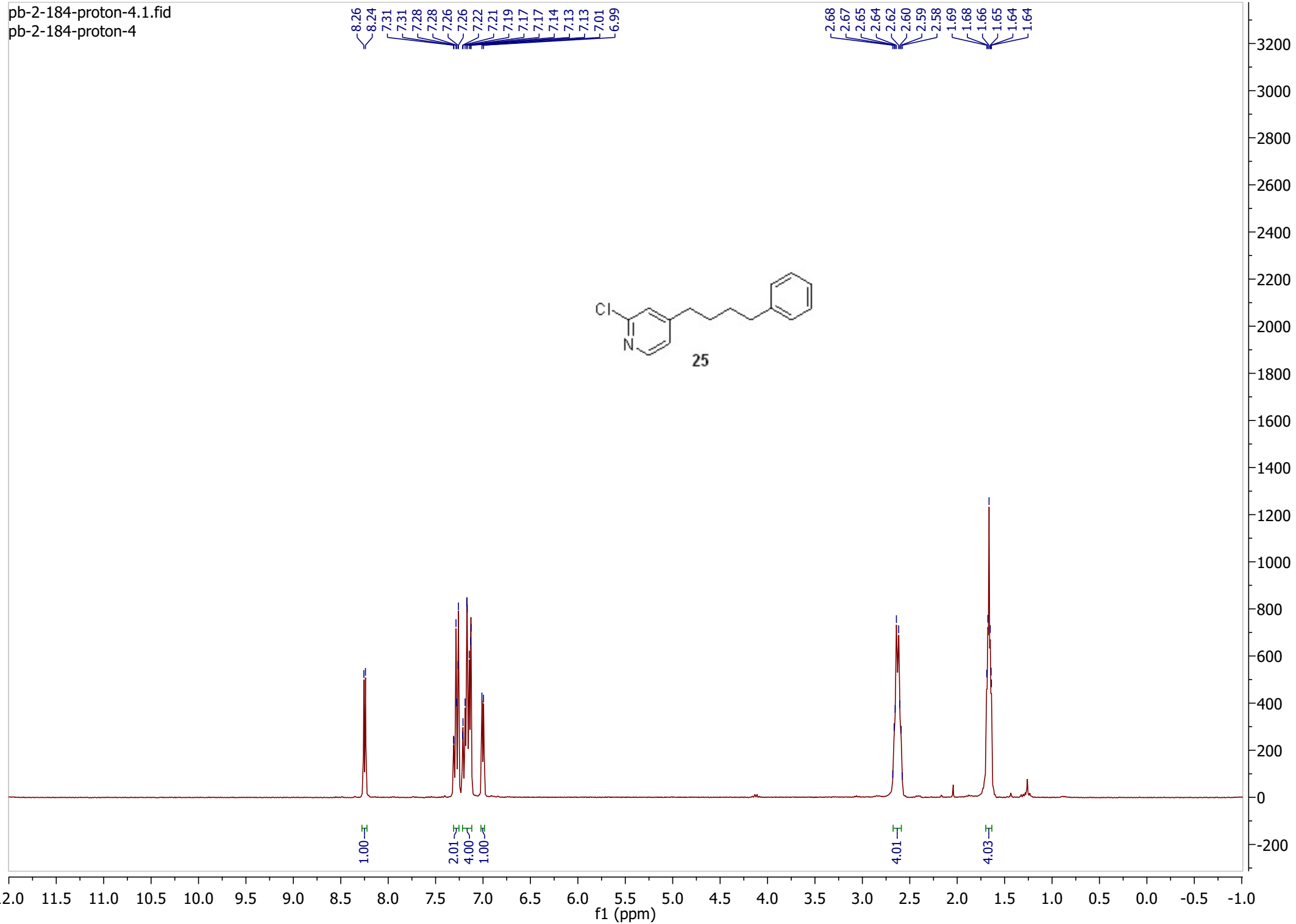
158.88
147.39
132.37
129.61
129.46
120.94
119.16
114.56
110.01
77.59 CDCl3
77.16 CDCl3
76.74 CDCl3
66.40
32.55
30.51



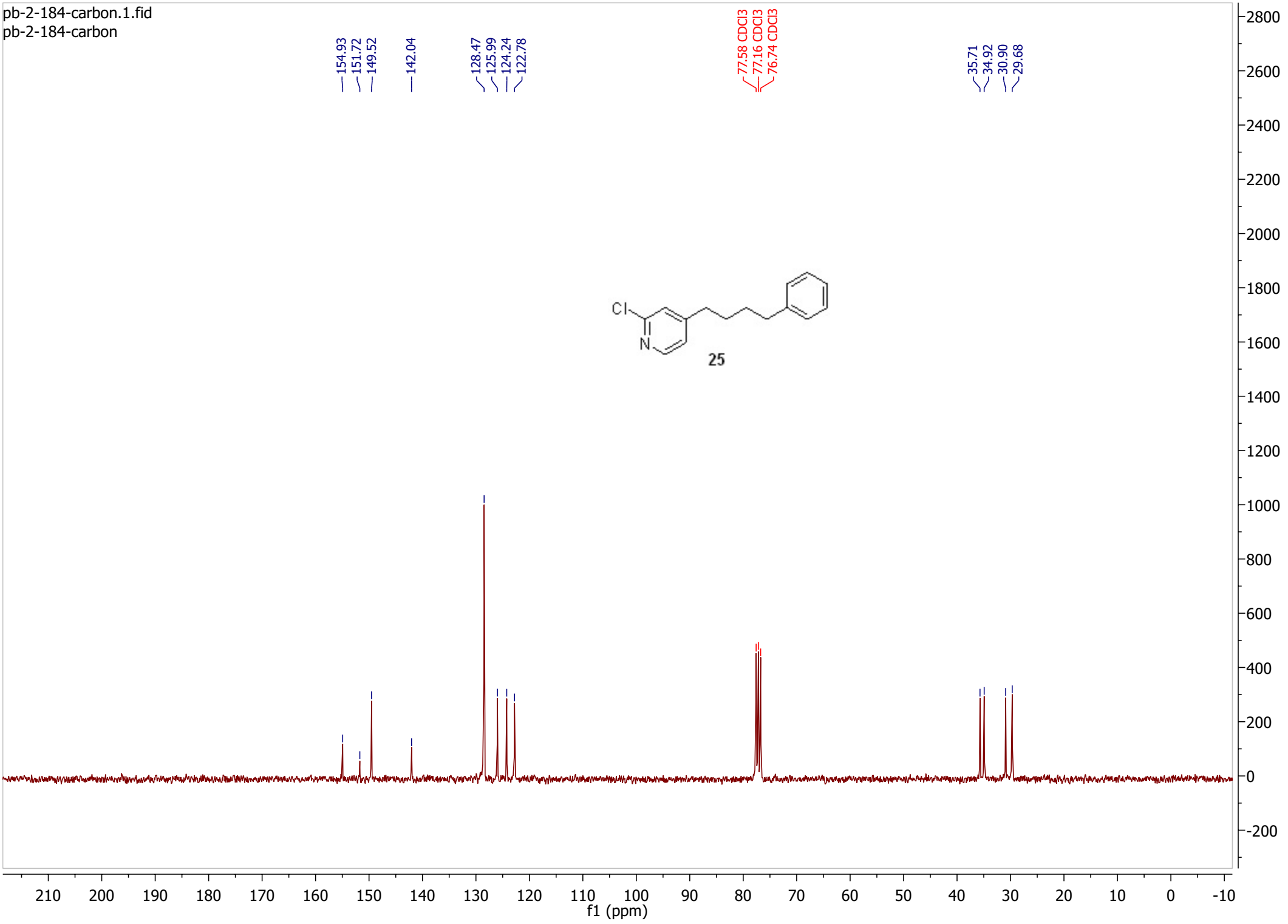
24

f1 (ppm)

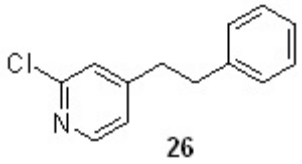
pb-2-184-proton-4.1.fid
pb-2-184-proton-4



pb-2-184-carbon.1.fid
pb-2-184-carbon



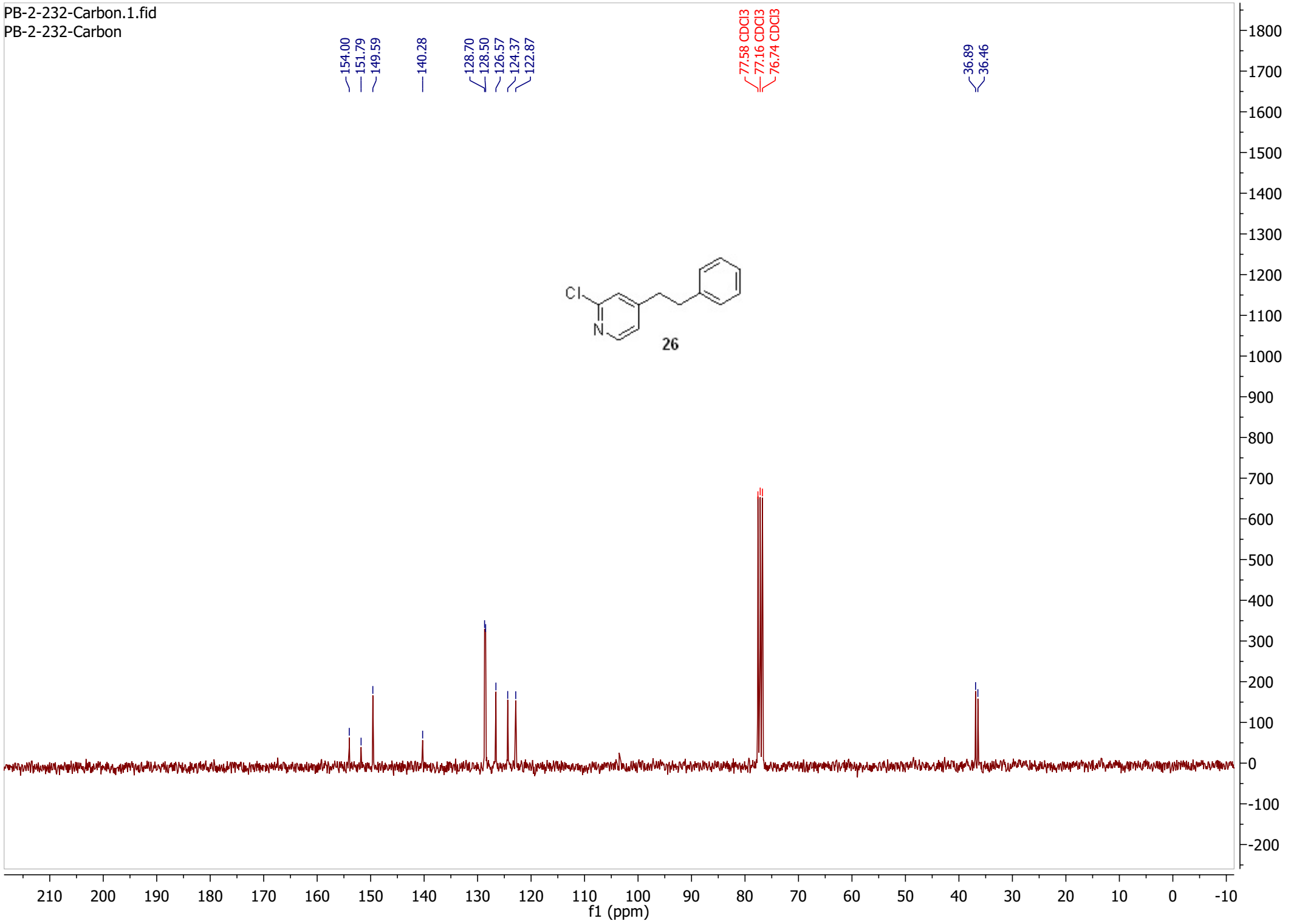
PB-2-232-proton.1.fid
PB-2-232-proton



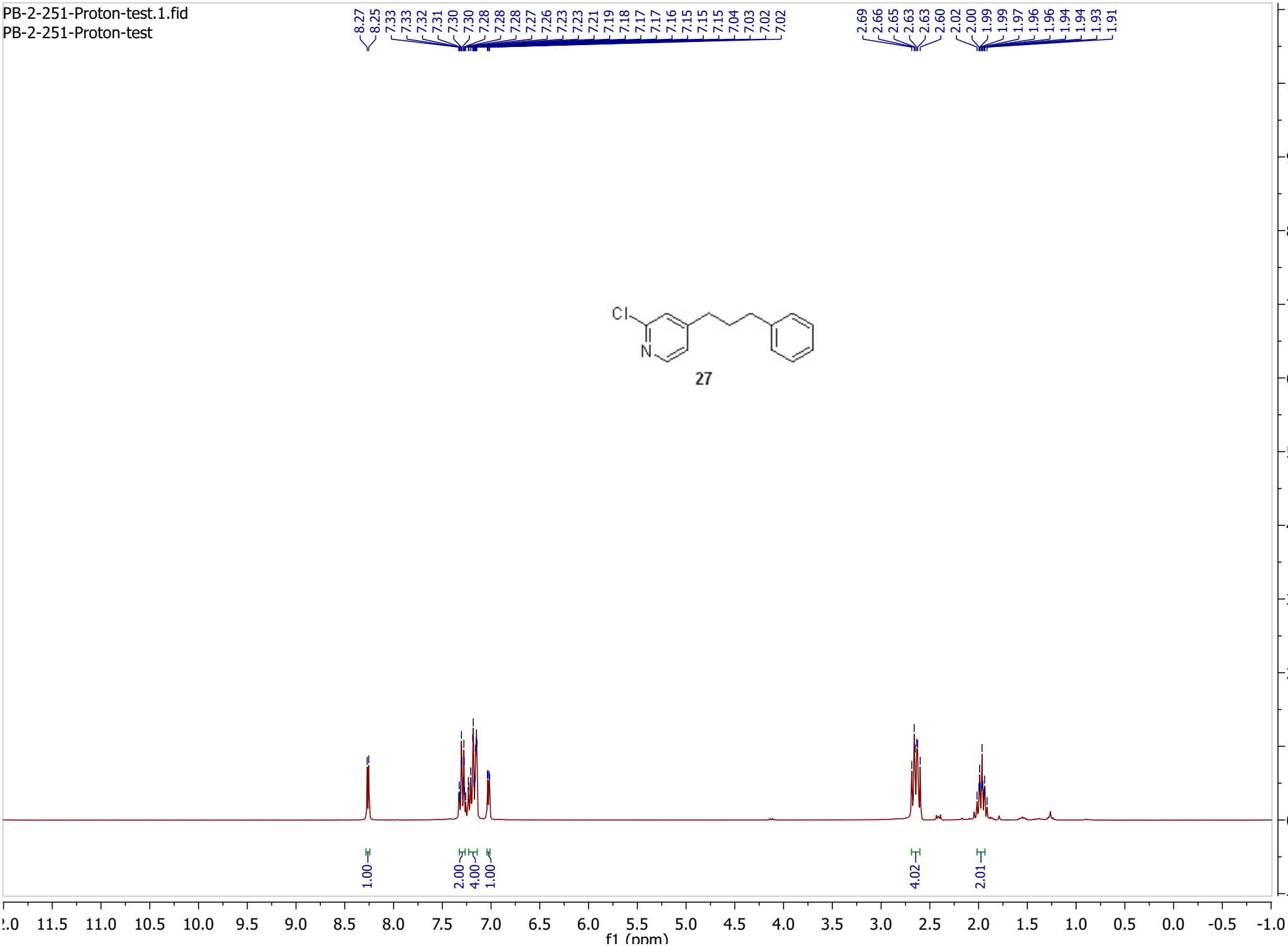
26

f1 (ppm)

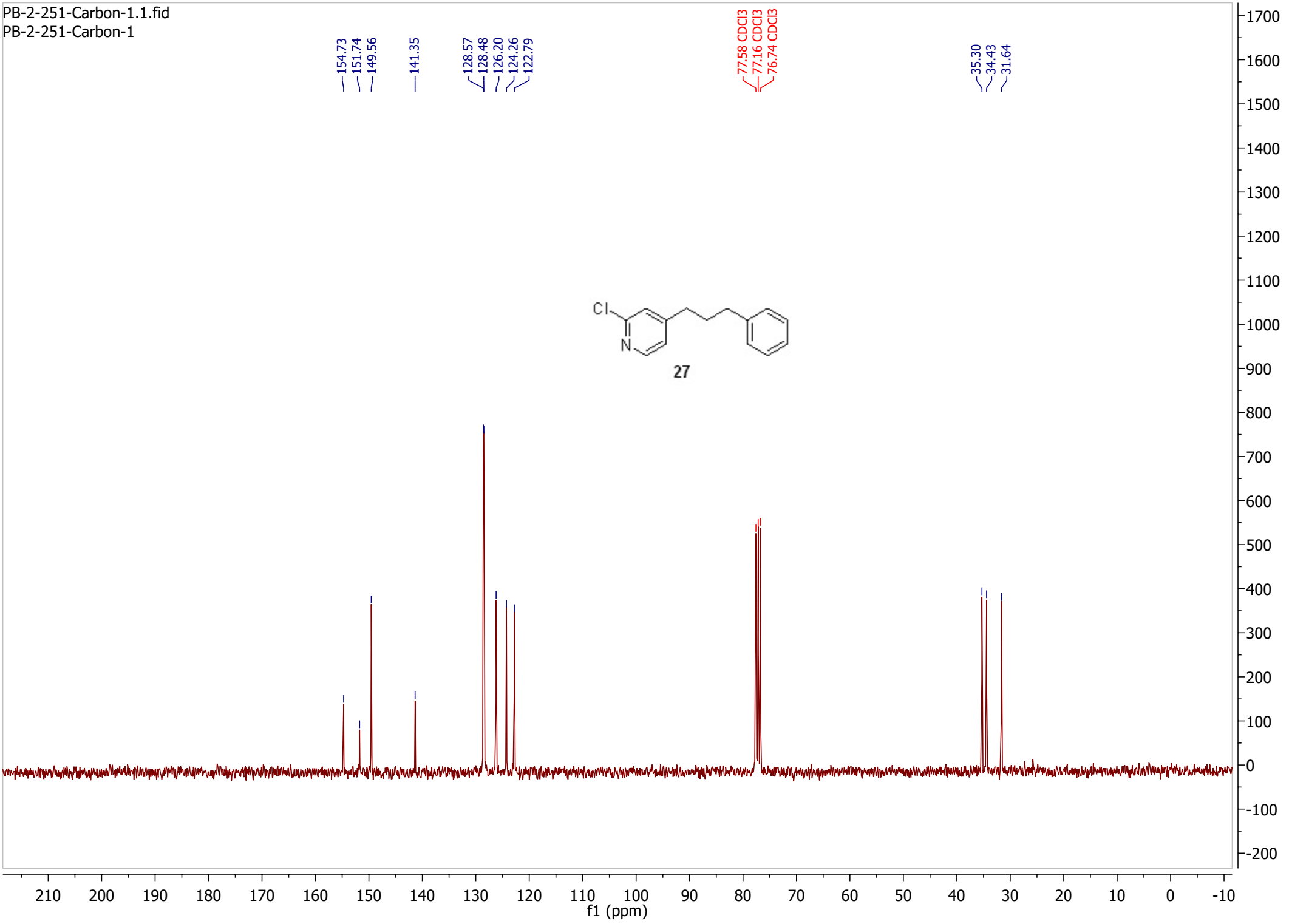
PB-2-232-Carbon.1.fid
PB-2-232-Carbon

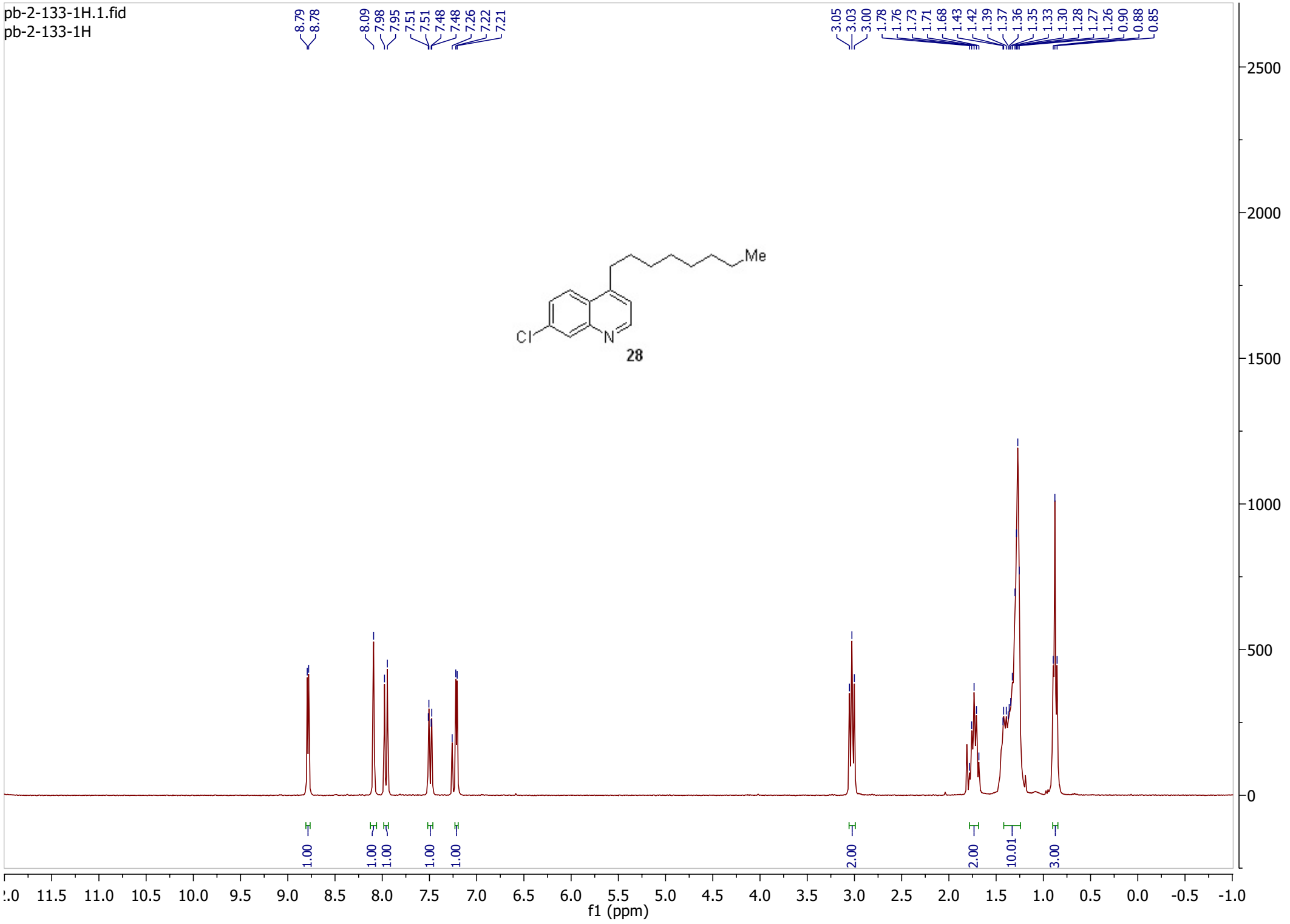


PB-2-251-Proton-test.1.fid
PB-2-251-Proton-test

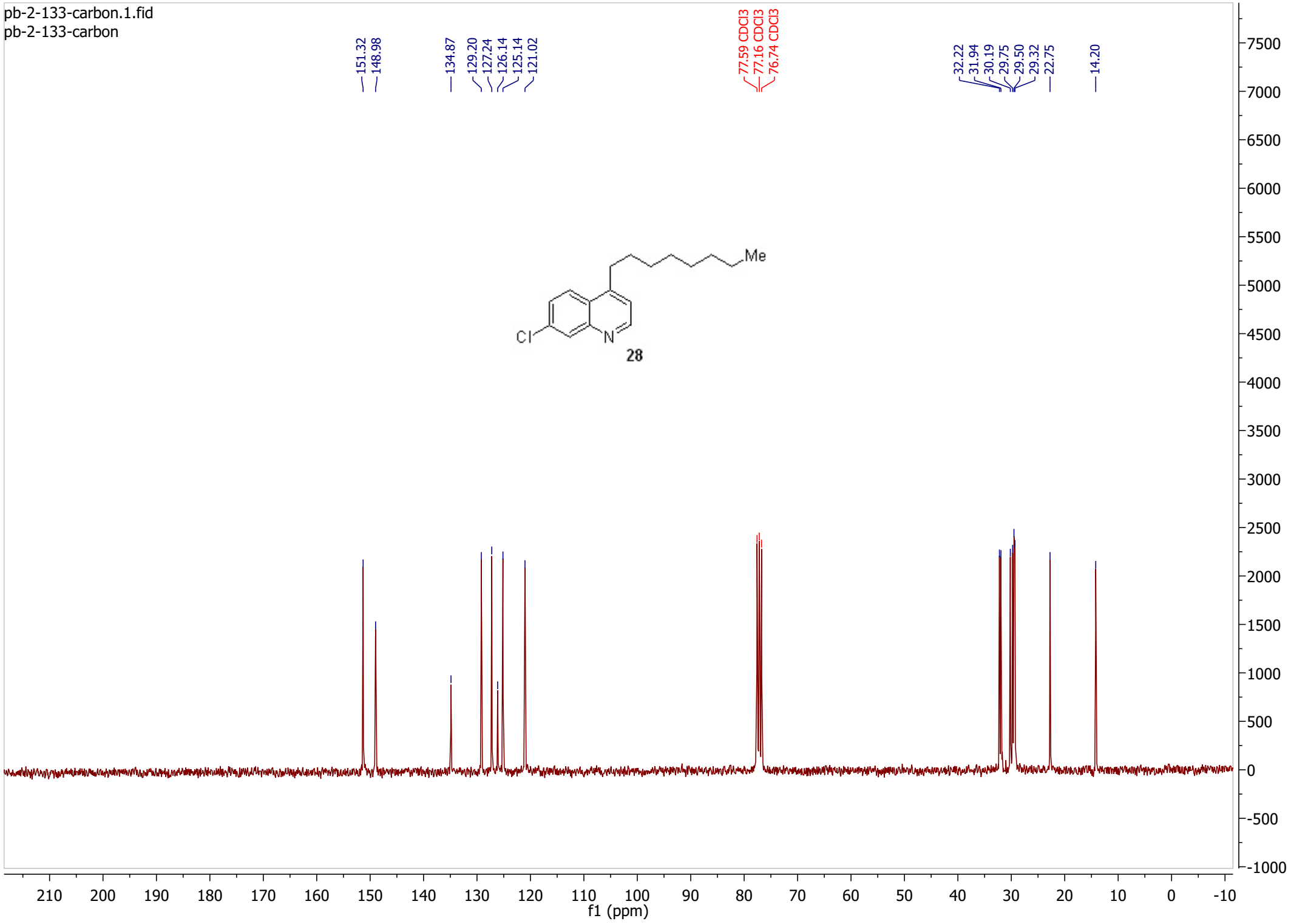


PB-2-251-Carbon-1.1.fid
PB-2-251-Carbon-1

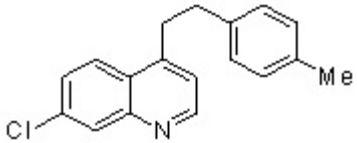



pb-2-133-1H.1.fid
pb-2-133-1H
8.79
8.78
8.09
7.98
7.95
7.51
7.51
7.48
7.48
7.26
7.22
7.21
3.05
3.03
3.00
1.78
1.76
1.73
1.71
1.68
1.43
1.42
1.39
1.37
1.36
1.35
1.33
1.30
1.28
1.27
1.26
0.90
0.88
0.85
Me
Cl
N
28
2500
2000
1500
1000
500
0
1.00
1.00
1.00
1.00
1.00
2.00
2.00
10.01
3.00
f1 (ppm)

pb-2-133-carbon.1.fid
pb-2-133-carbon



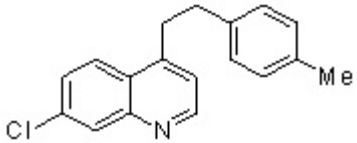
pb-2-269-1H-1.1.fid
pb-2-269-1H-1



29

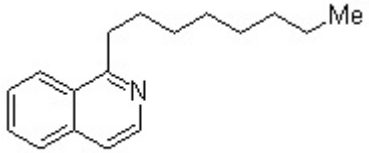
f1 (ppm)

pb-2-269-carbon.1.fid
pb-2-269-carbon



29

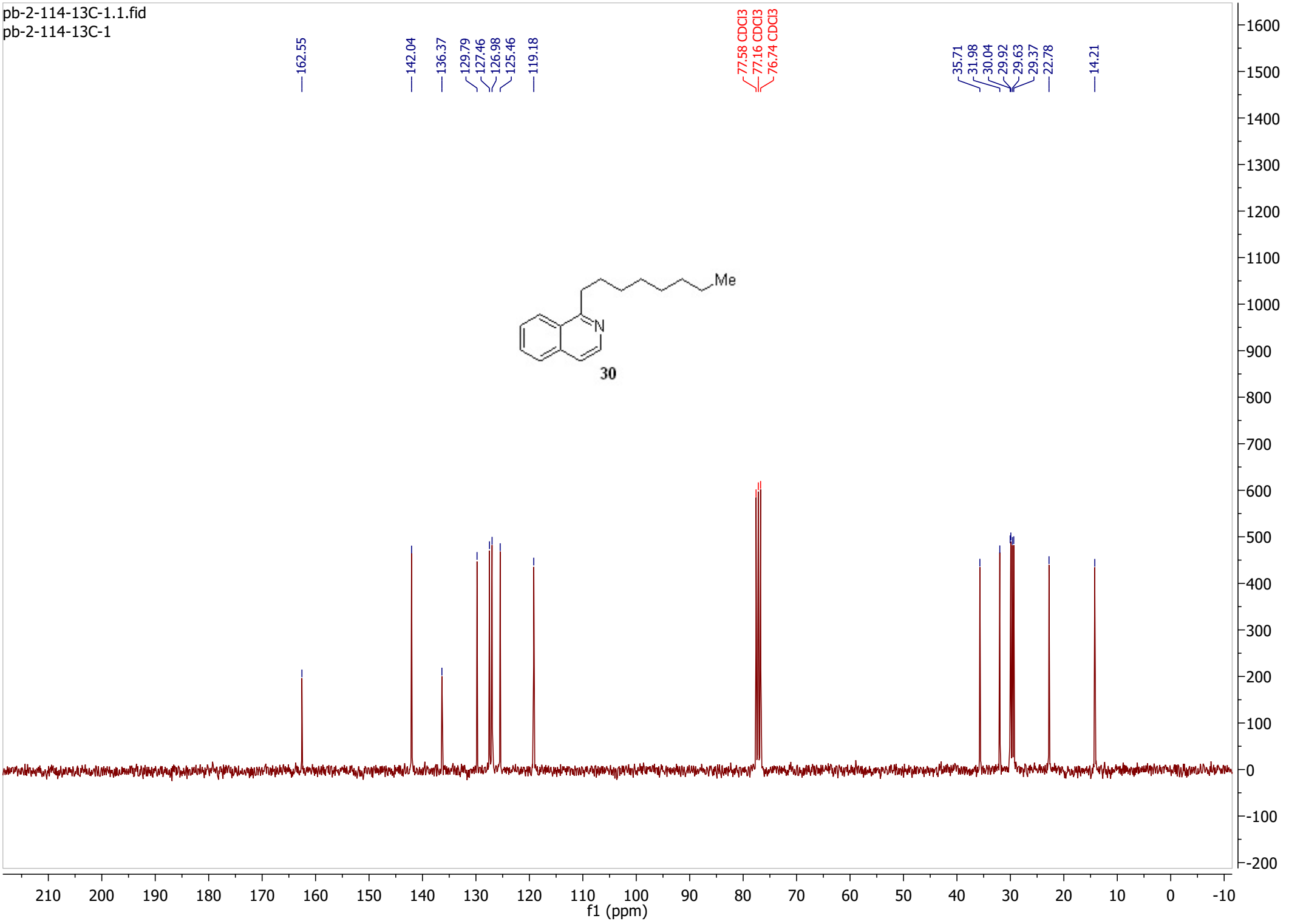
pb-2-114-proton.1.fid
pb-2-114-proton



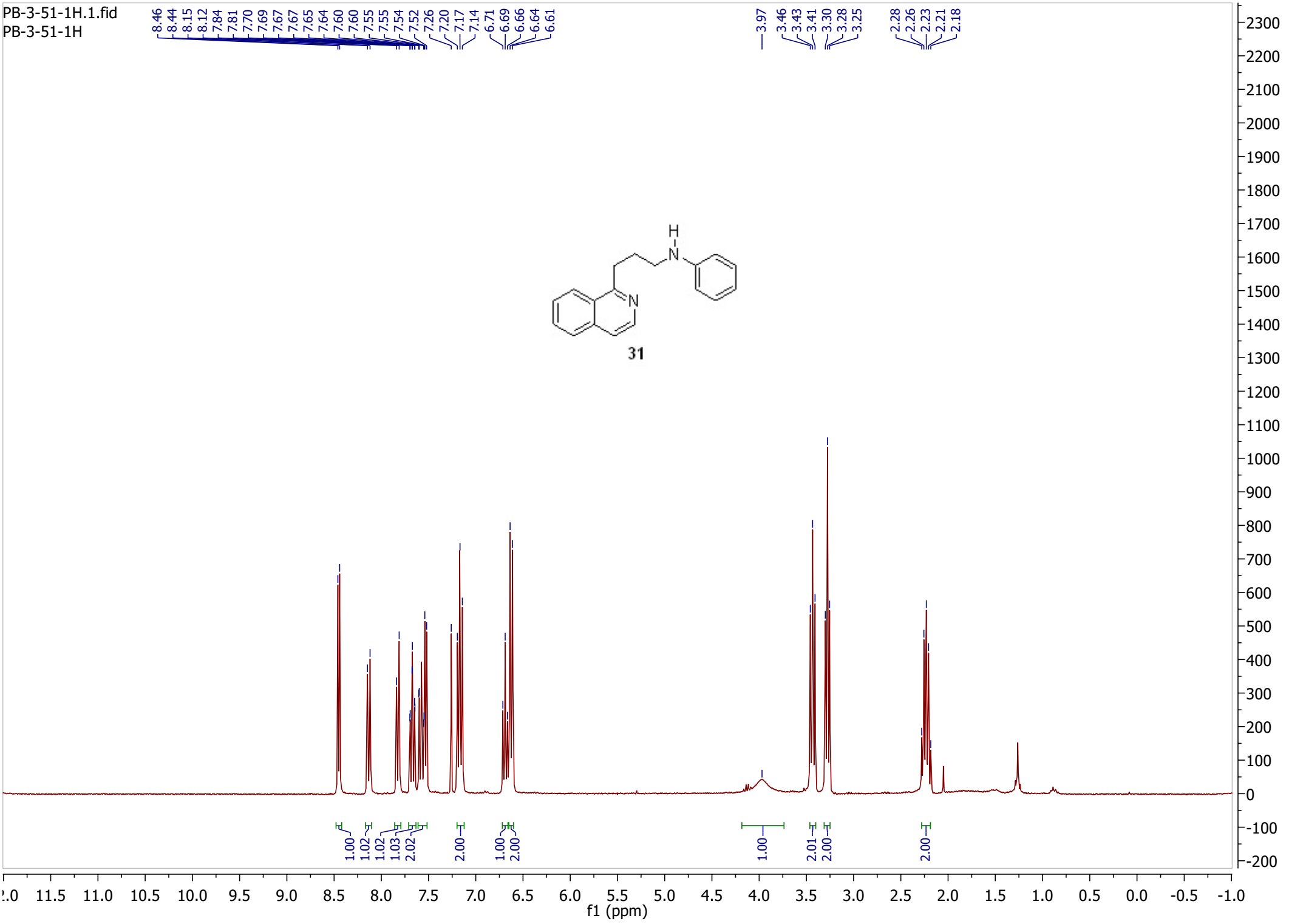
30

f1 (ppm)

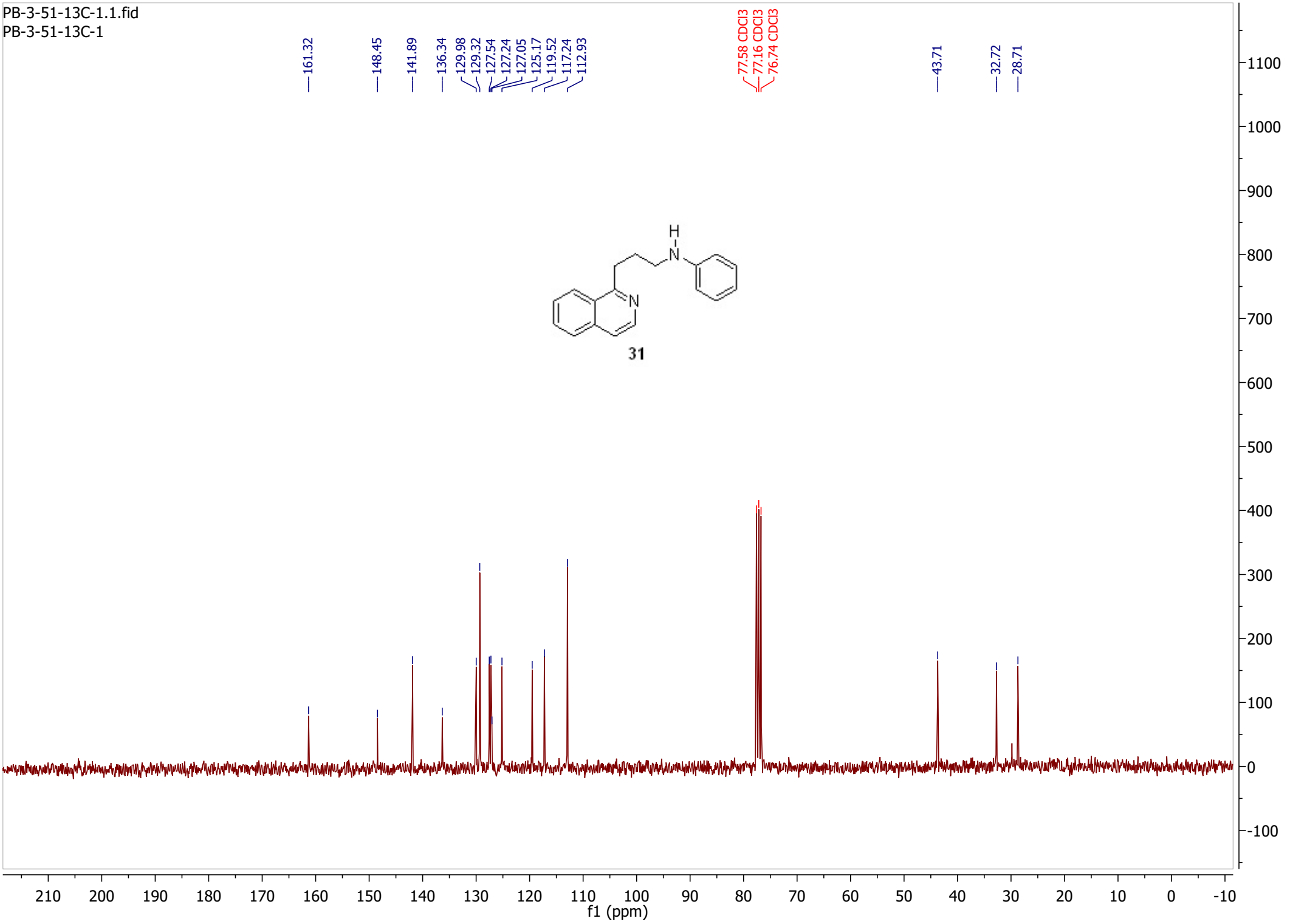
pb-2-114-13C-1.1.fid
pb-2-114-13C-1



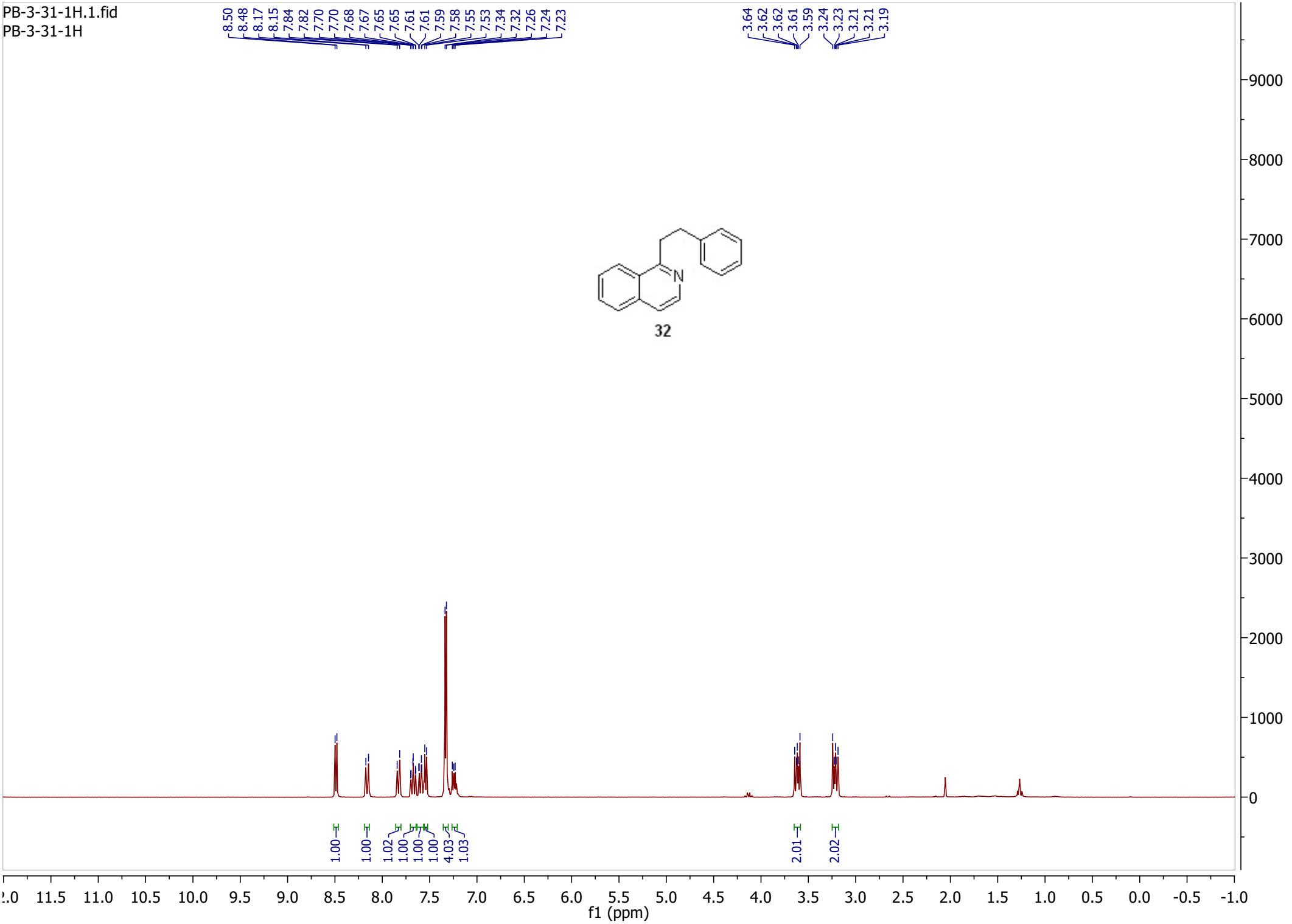
PB-3-51-1H.1.fid
PB-3-51-1H



PB-3-51-13C-1.1.fid
PB-3-51-13C-1

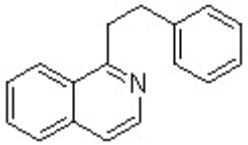


PB-3-31-1H.1.fid
PB-3-31-1H



PB-3-31-13C.1.fid
PB-3-31-13C

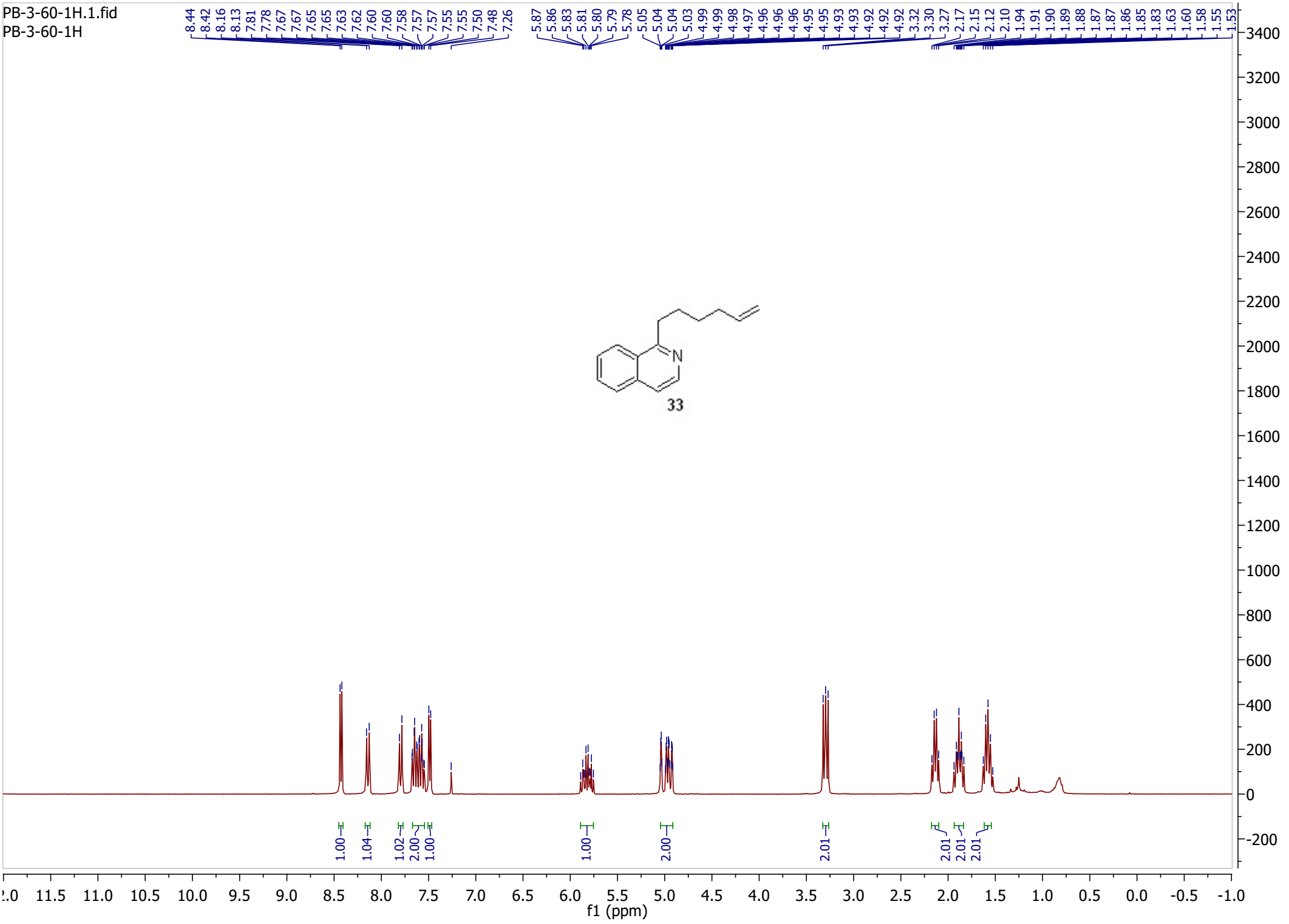
161.13
142.04
136.35
129.93
128.60
128.56
127.54
127.21
127.04
126.17
125.17
119.54
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
37.38
35.59



32

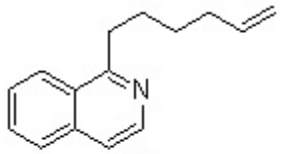
f1 (ppm)

PB-3-60-1H.1.fid
PB-3-60-1H



PB-3-60-13C-1.1.fid
PB-3-60-13C-1

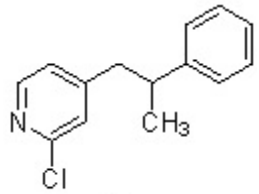
162.30
142.02
138.90
136.37
129.86
127.50
127.04
125.43
119.27
114.61
77.59 CDCl3
77.16 CDCl3
76.74 CDCl3
35.47
33.77
29.33
29.23



33

f1 (ppm)

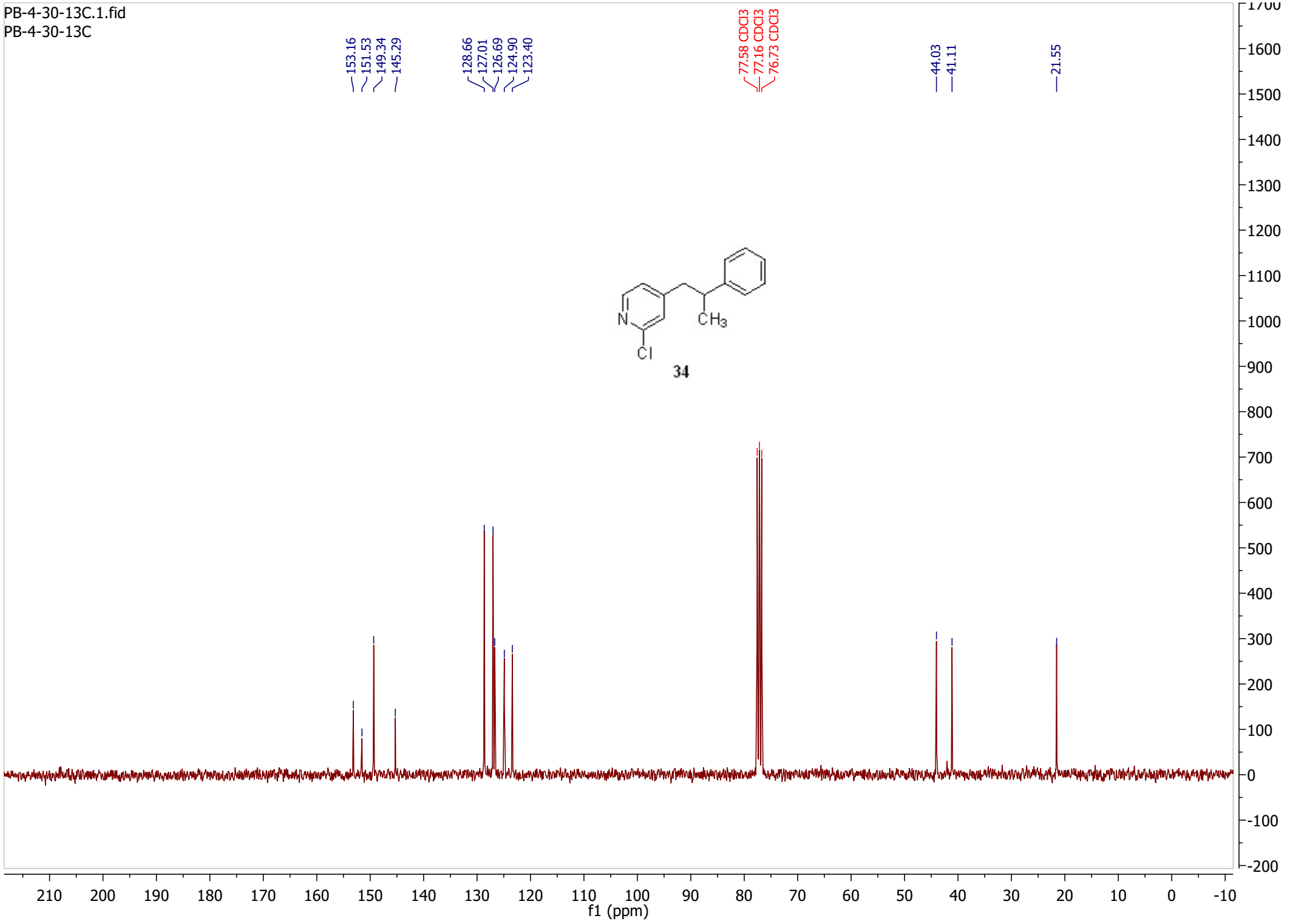
PB-4-30-tt-91-1H-re.1.fid
PB-4-30-tt-91-1H-re



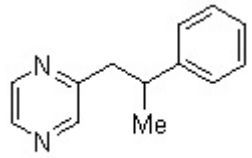
34

f1 (ppm)

PB-4-30-13C.1.fid
PB-4-30-13C



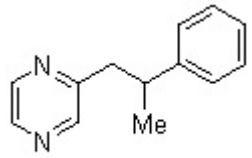
pb-2-186-proton-5.1.fid
pb-2-186-proton-5



35

pb-2-186-13C.1.fid
pb-2-186-13C

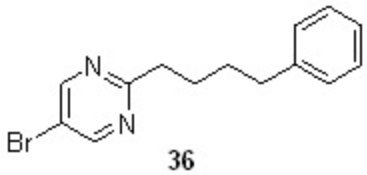
156.36
145.87
145.26
144.17
142.31
128.62
127.03
126.51
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
44.32
40.33
21.64



35

f1 (ppm)

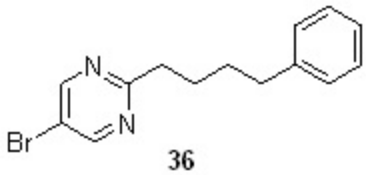
PB-2-276-1H-2.1.fid
PB-2-276-1H-2



36

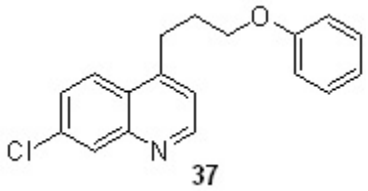
f1 (ppm)

PB-2-276-13C.1.fid
PB-2-276-13C



36

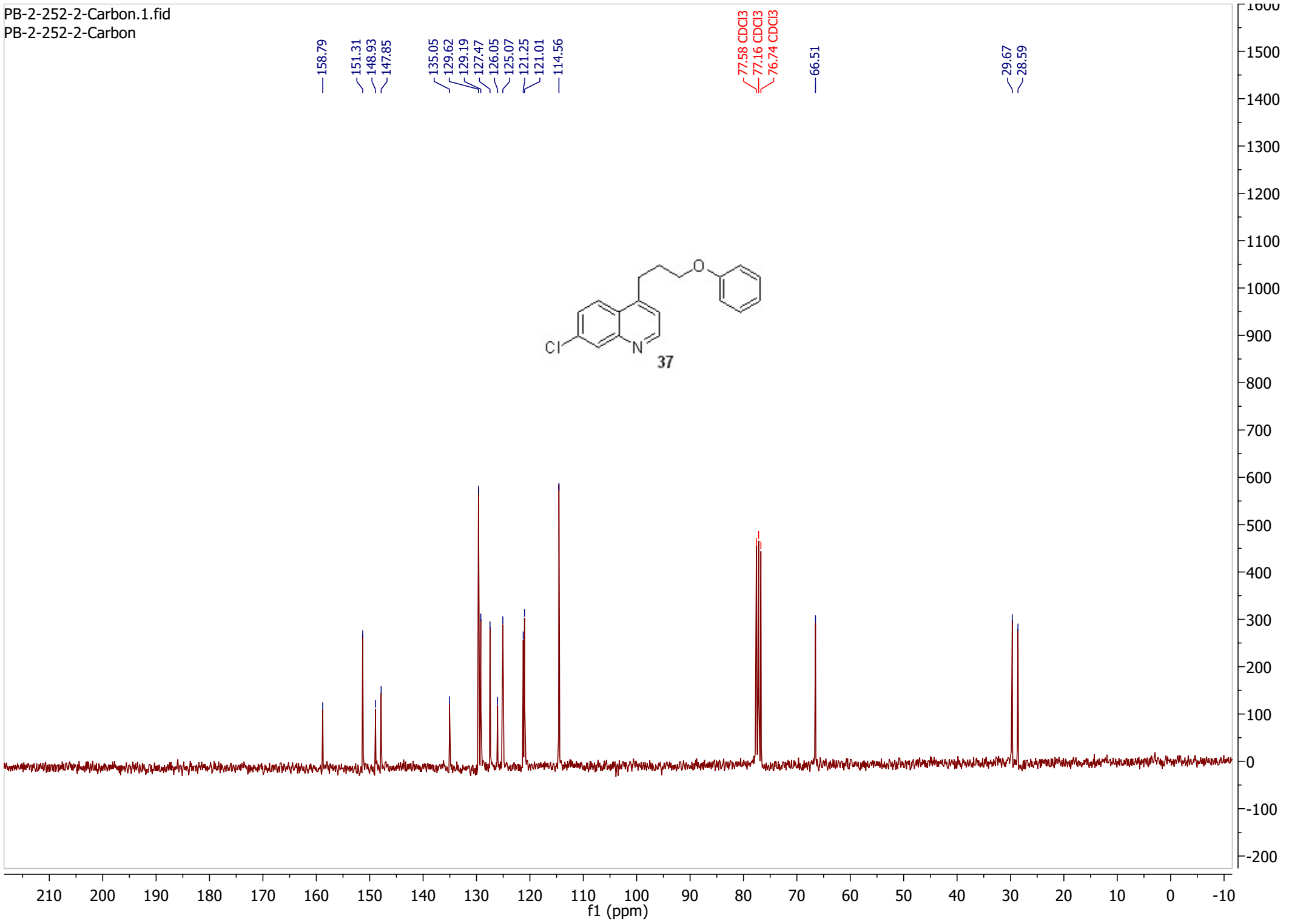
PB-2-252-2-Proton.1.fid
PB-2-252-2-Proton



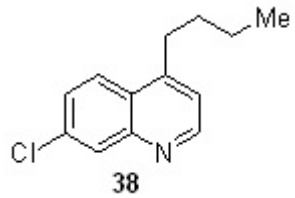
37

f1 (ppm)

PB-2-252-2-Carbon.1.fid
PB-2-252-2-Carbon



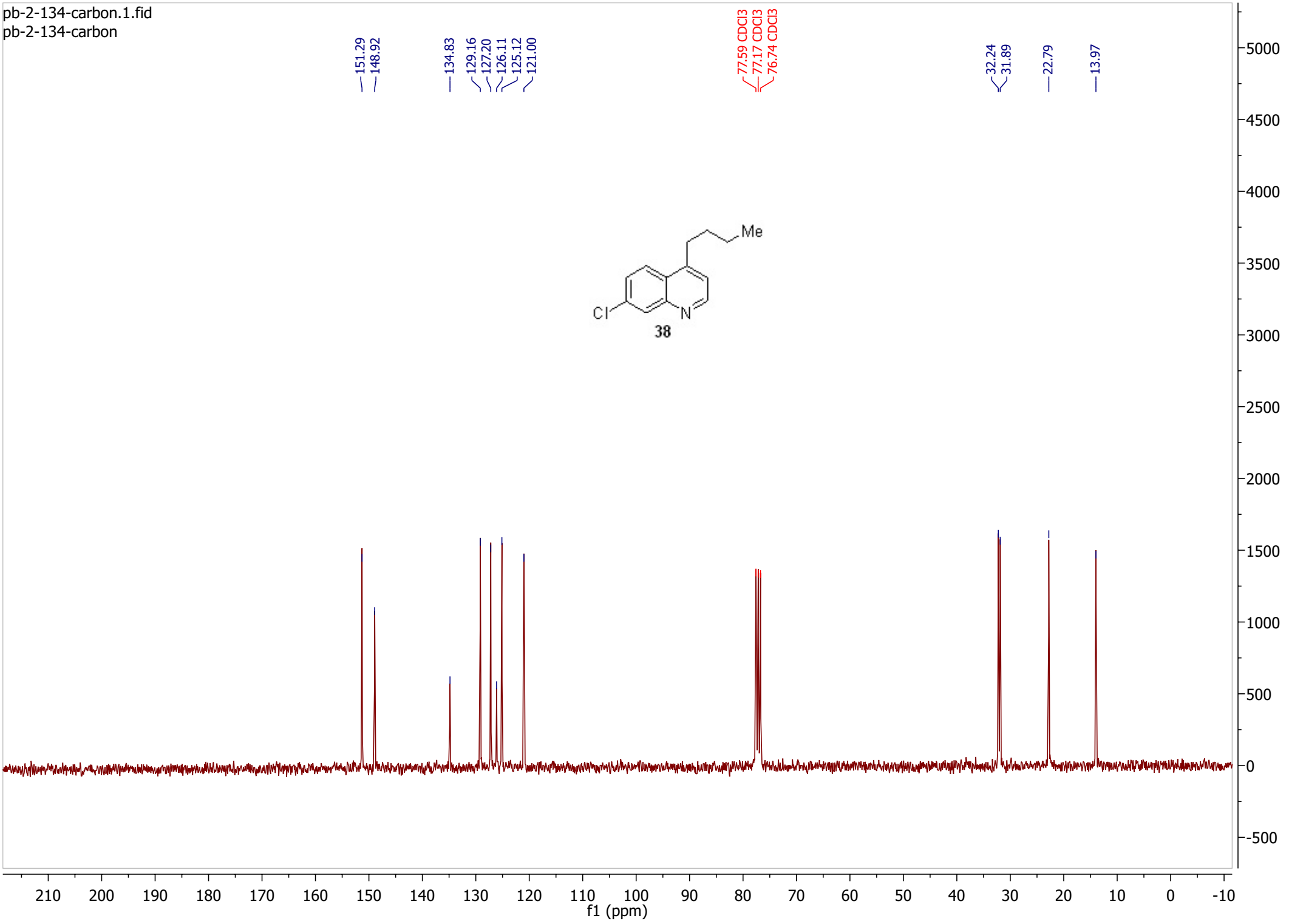
pb-2-134-proton.1.fid
pb-2-134-proton

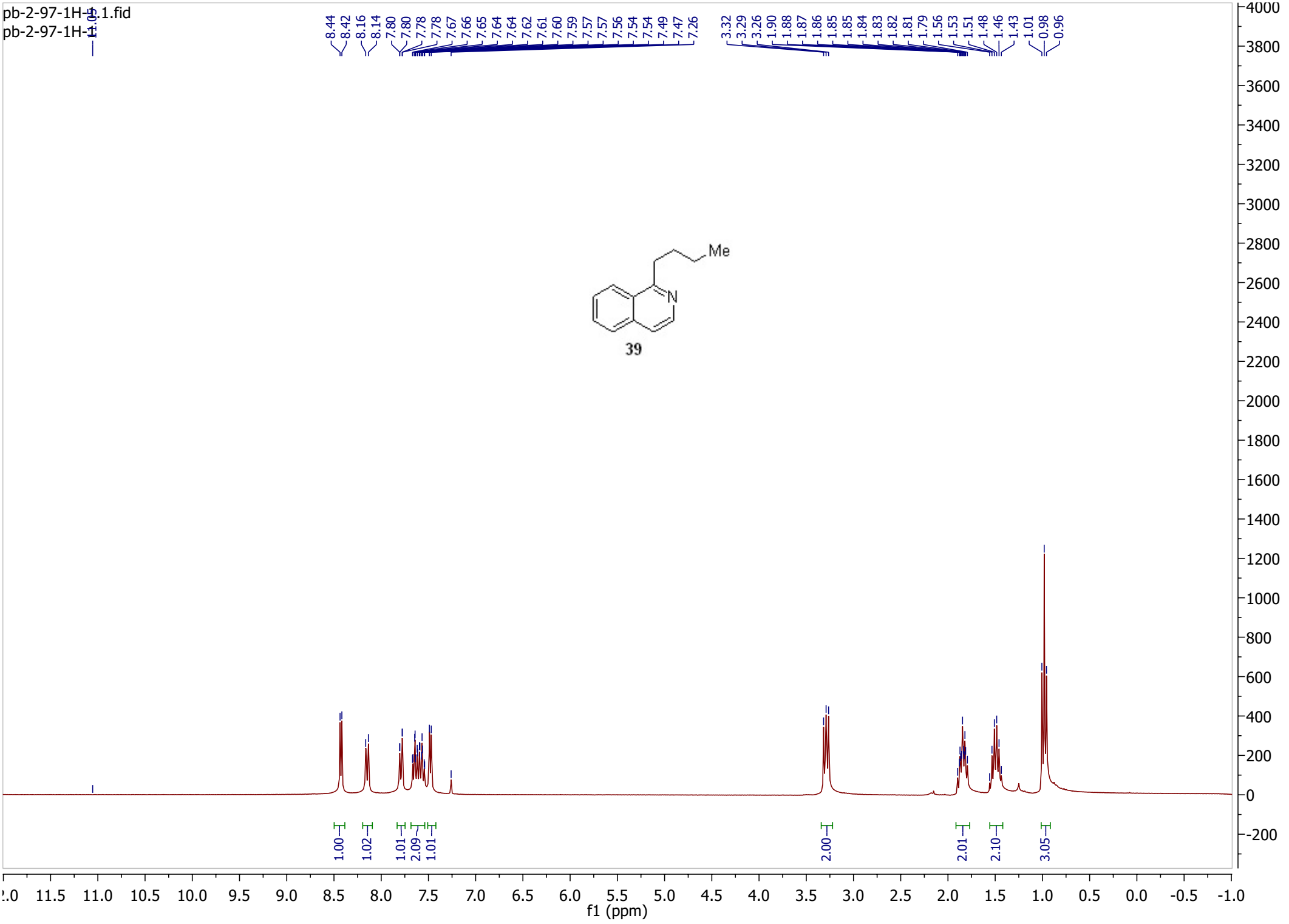


38

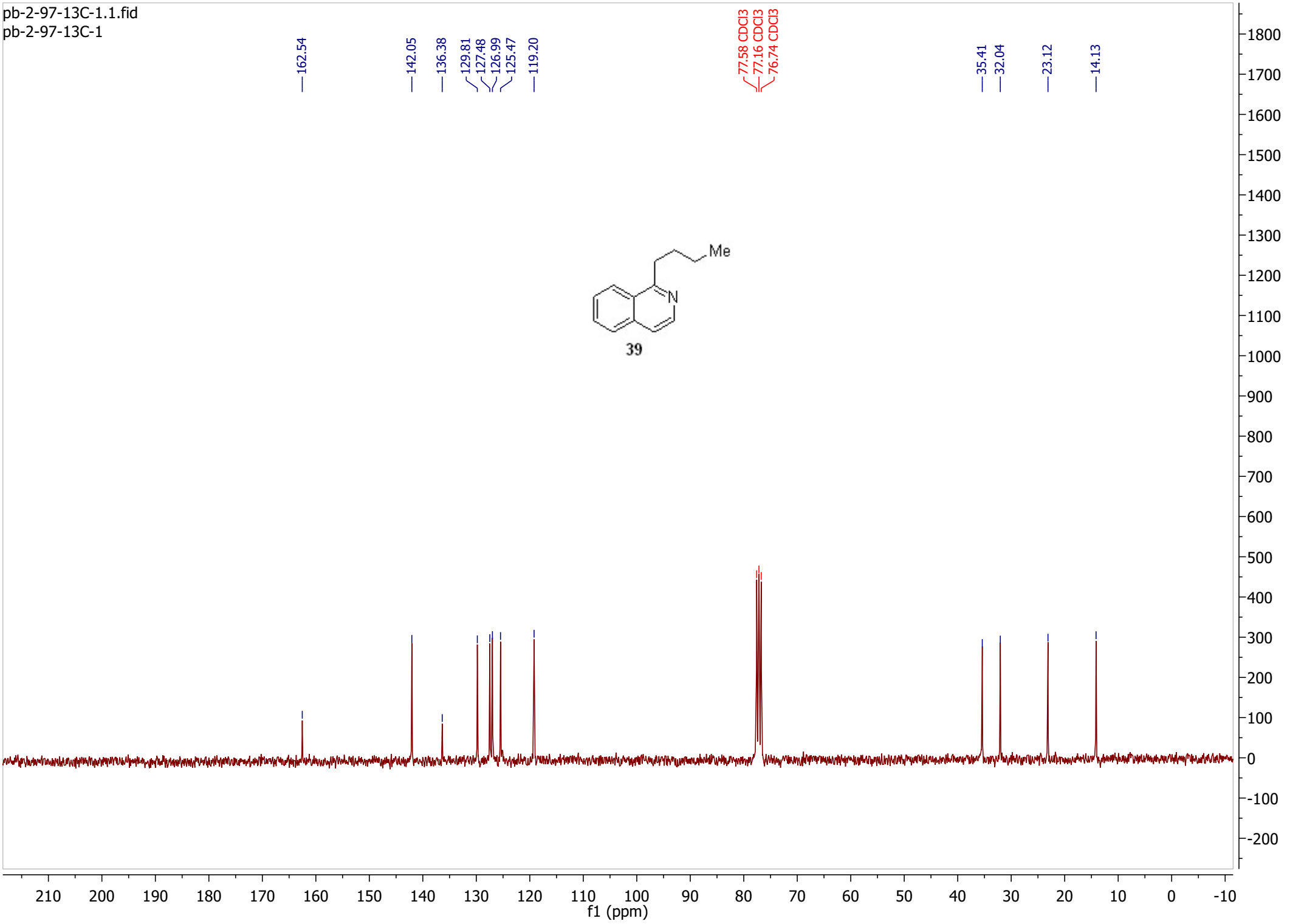
f1 (ppm)

pb-2-134-carbon.1.fid
pb-2-134-carbon

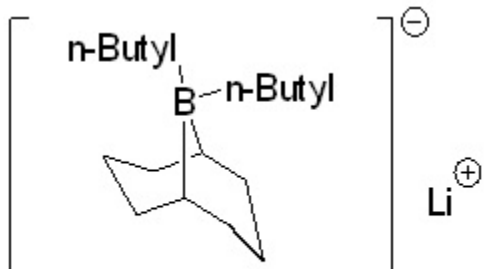



pb-2-97-1H-1.1.fid
pb-2-97-1H-1
8.44
8.42
8.16
8.14
7.80
7.80
7.78
7.78
7.67
7.66
7.65
7.64
7.64
7.62
7.61
7.60
7.59
7.57
7.57
7.56
7.54
7.54
7.49
7.47
7.26
3.32
3.29
3.26
1.90
1.88
1.87
1.86
1.85
1.85
1.84
1.83
1.82
1.81
1.79
1.56
1.53
1.51
1.48
1.46
1.43
1.01
0.98
0.96
Me
N
39
1.00
1.02
1.01
2.09
1.01
2.00
2.01
2.10
3.05
f1 (ppm)

pb-2-97-13C-1.1.fid
pb-2-97-13C-1



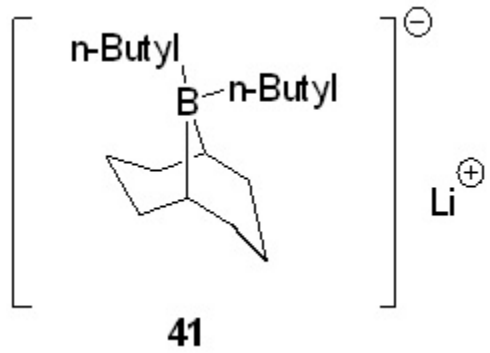
PB-3-33-1H-re.1.fid
PB-3-33-1H-re



41

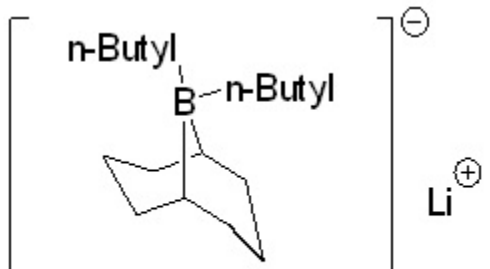
f1 (ppm)

PB-3-33-2n-butyl-9bbn-comp-13C.1.fid
PB-3-33-2n-butyl-9bbn-comp-13C



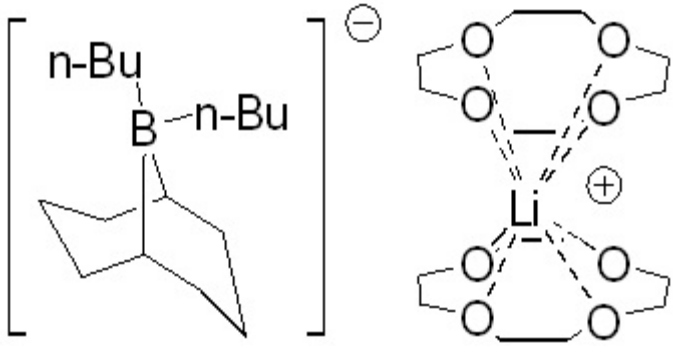
41

PB-3-33-11B-re.1.fid
PB-3-33-11B-re



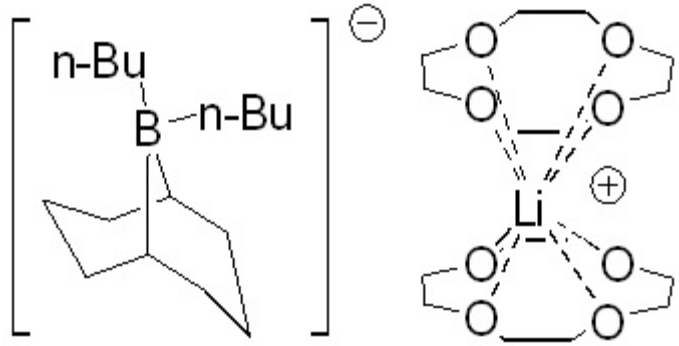
41

PB-3-251-1H.1.fid
PB-3-251-1H



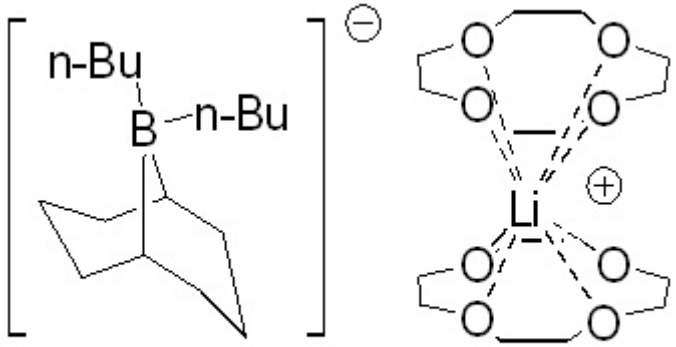
41·2(12-Crown-4)

PB-3-251-13C-re.1.fid
PB-3-251-13C-re



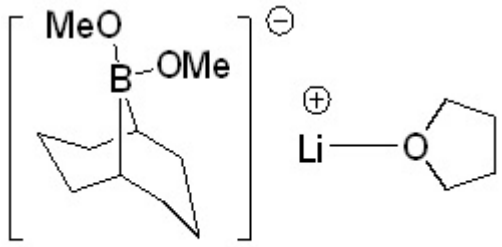
41·2(12-Crown-4)

PB-3-251-11B.1.fid
PB-3-251-11B



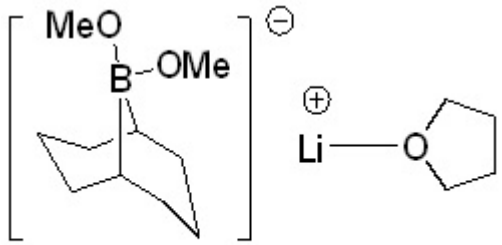
41·2(12-Crown-4)

PB-4-6-THF-pent-wash-1H.1.fid
PB-4-6-THF-pent-wash-1H



42 (THF adduct dimer)

PB-4-6-13C.1.fid
PB-4-6-13C

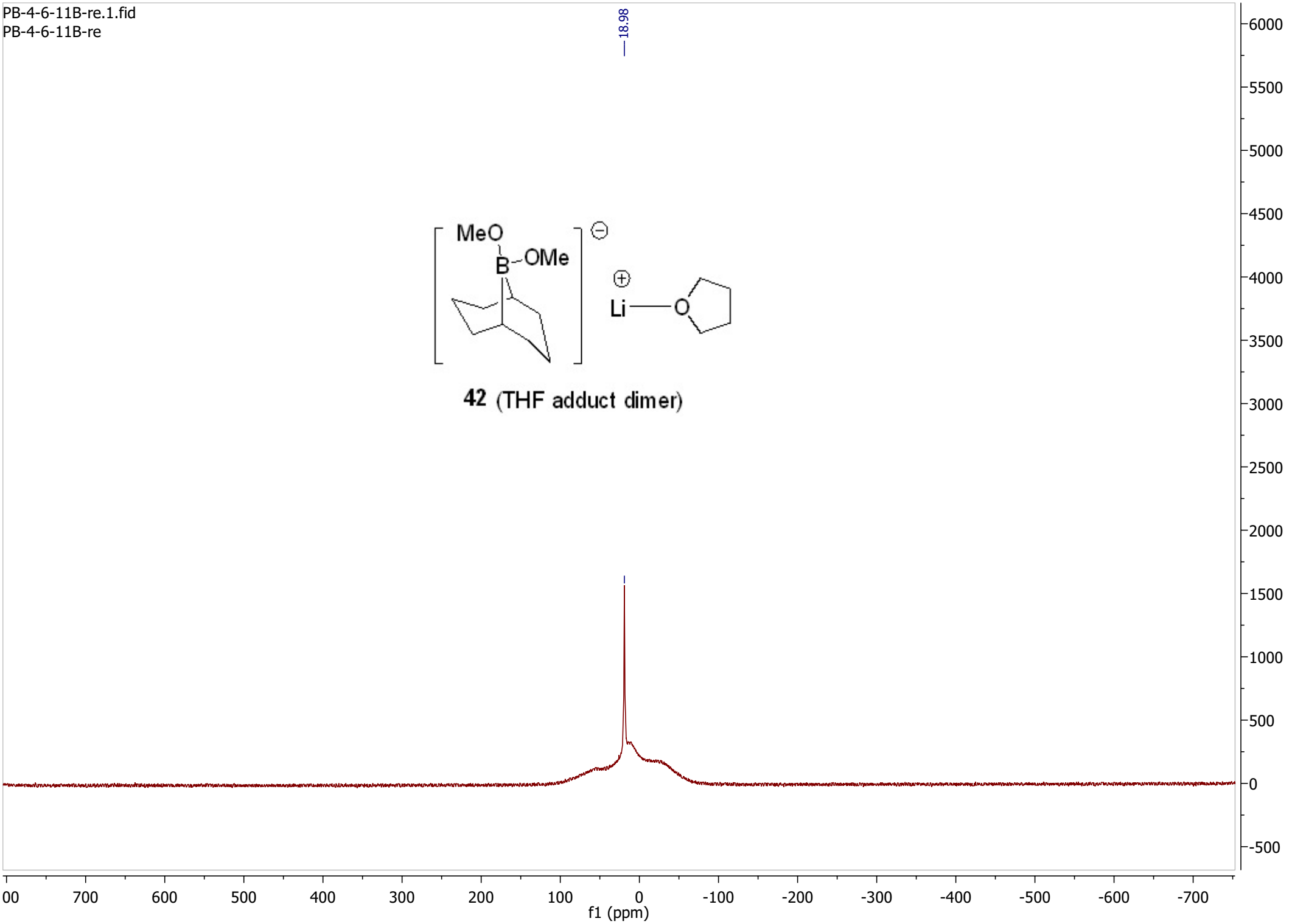


42 (THF adduct dimer)

67.00
47.17
40.34 DMSO
40.06 DMSO
39.78 DMSO
39.50 DMSO
39.22 DMSO
38.95 DMSO
38.67 DMSO
32.69
26.38
25.12

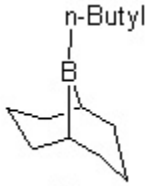
f1 (ppm)

PB-4-6-11B-re.1.fid
PB-4-6-11B-re



42 (THF adduct dimer)

PB-3-226-pent-lay-1H.1.fid
PB-3-226-pent-lay-1H



43

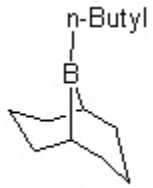
f1 (ppm)

PB-3-226-pent-laye-13C.1.fid
PB-3-226-pent-laye-13C

77.58 CDCl3
77.16 CDCl3
76.74 CDCl3

33.32
31.12
26.87
26.08
23.43

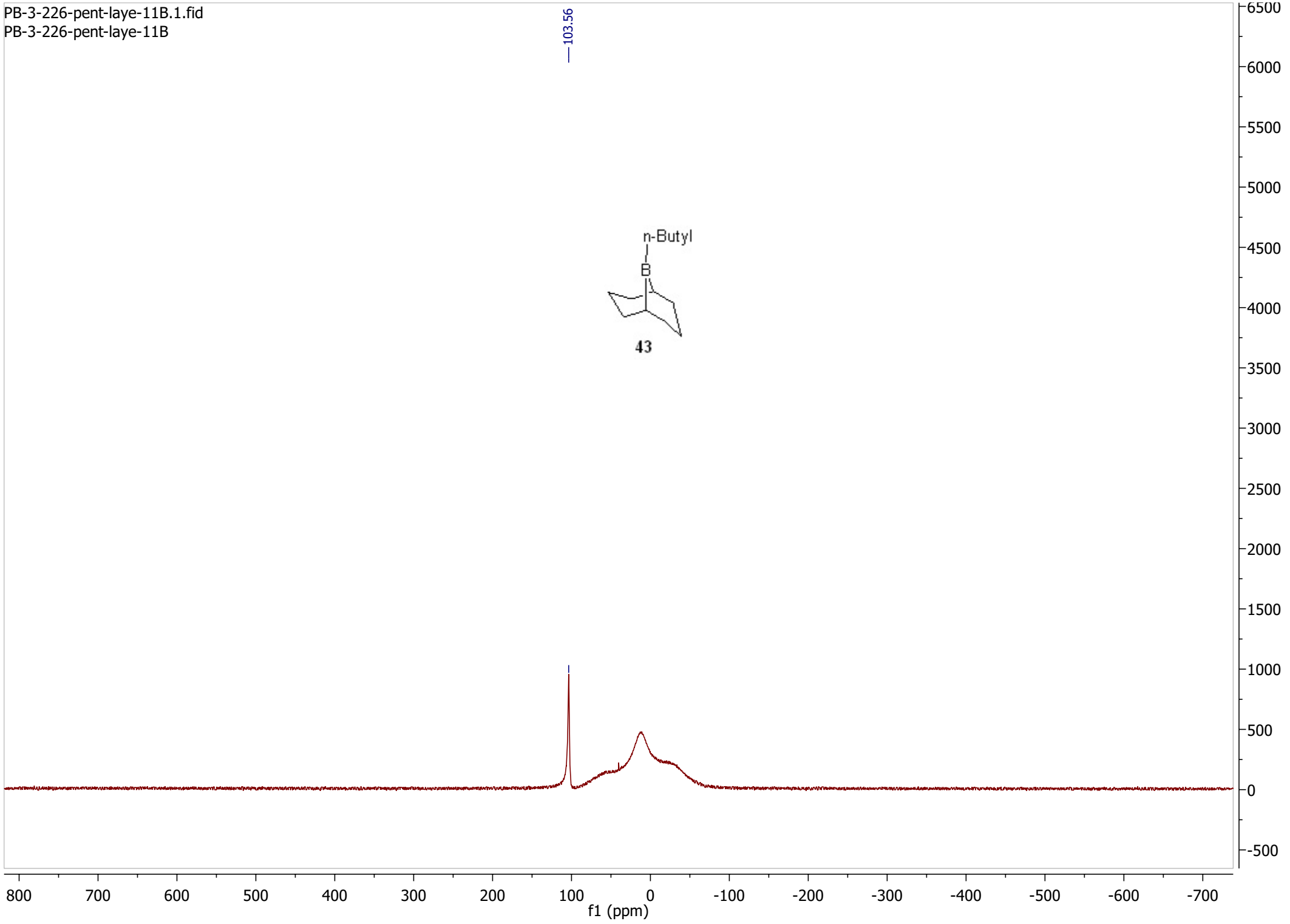
14.26



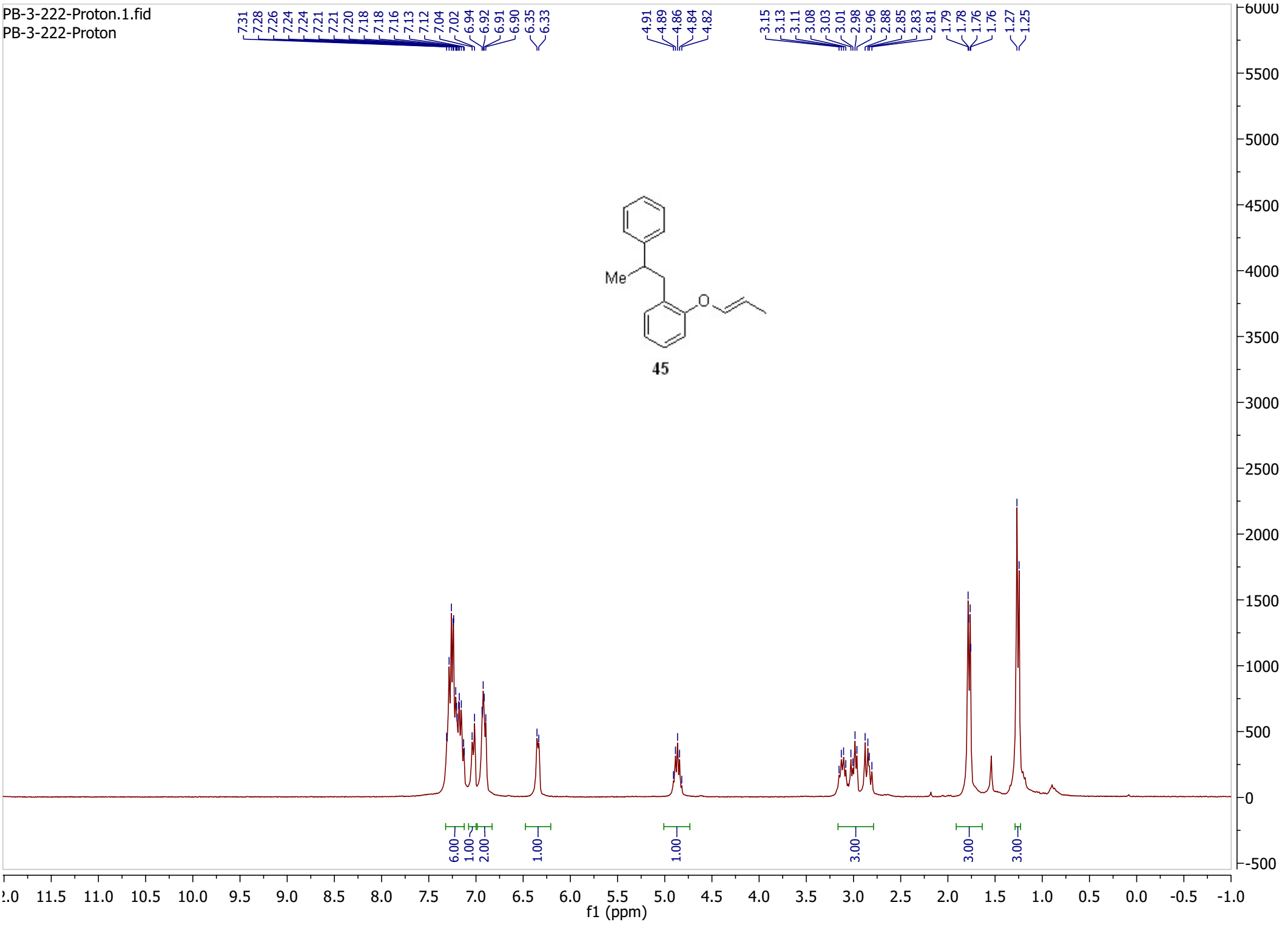
43

f1 (ppm)

PB-3-226-pent-laye-11B.1.fid
PB-3-226-pent-laye-11B

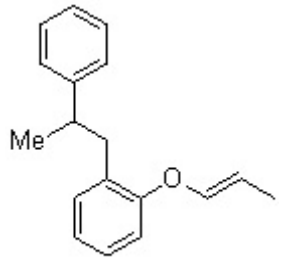


PB-3-222-Proton.1.fid
PB-3-222-Proton



PB-3-222-13C-re.1.fid
PB-3-222-13C-re

155.78
147.55
141.57
131.36
130.38
128.36
127.33
127.13
126.03
122.22
114.82
106.68
77.59 CDCl3
77.17 CDCl3
76.74 CDCl3
40.53
39.60
21.06
9.63



45

f1 (ppm)