Enantioselective Pd-Catalyzed Allyl-Allyl Cross Coupling

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Supporting Information

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General Information

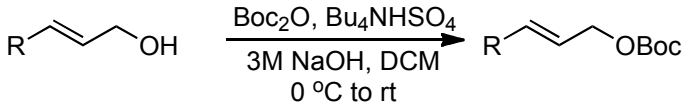
^{1}H NMR spectra were recorded on either a Varian Gemini-400 (400 MHz), a Varian Gemini-500 (500 MHz) or a Varian Inova-500 (500 MHz) spectrometer. Chemical shifts are reported in ppm with the solvent resonance as the internal standard ($CDCl_3$: 7.26 ppm). Data are reported as follows: chemical shift, integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, p = pentet, br = broad, m = multiplet, app = apparent), coupling constants (Hz), and assignment. ^{13}C NMR spectra were recorded on either a Varian Gemini-400 (100 MHz), or a Varian Gemini-500 (125 MHz) spectrometer with complete proton decoupling. Chemical shifts are reported in ppm with the solvent resonance as the internal standard ($CDCl_3$: 77.0 ppm). Infrared (IR) spectra were recorded on a Bruker alpha spectrophotometer, ν_{max} cm^{-1}. Bands are characterized as broad (br), strong (s), medium (m), and weak (w). High resolution mass spectrometry (ESI) was performed at the Mass Spectrometry Facility, Boston College.

Liquid Chromatography was performed using forced flow (flash chromatography) on silica gel (SiO_2, 230×450 Mesh) purchased from Silicycle. Thin Layer Chromatography was performed on 25 μm silica gel plates purchased from Silicycle. Visualization was performed using ultraviolet light (254 nm) or potassium permanganate ($KMnO_4$) in water. Analytical chiral gas-liquid chromatography (GLC) was performed on a Hewlett-Packard 6890 Series chromatograph equipped with a split mode capillary injection system, a flame ionization detector, and a Supelco β-Dex 120 column or a Supelco Chiraldex G-TA with helium as the carrier gas. Analytical chiral supercritical fluid chromatography (SFC) was performed on a Berger Instruments Supercritical Chromatograph equipped with an Alcott auto sampler and a Knauer UV detector with methanol as the modifier. Analytical high performance liquid chromatography (HPLC) was performed on an Agilent 1120 compact chromatograph equipped with gradient pump and variable wavelength detector. Optical rotations were measured on a Rudolph Analytical Research Autopol IV Polarimeter.

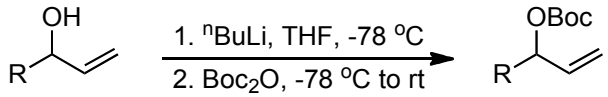
All reactions were conducted in oven- or flame-dried glassware under an inert atmosphere of nitrogen or argon. Tetrahydrofuran (THF) was purified using a Pure Solv MD-4 solvent purification system from Innovative Technology Inc. Ethyl acetate was purified by drying with calcium hydride and distilled under N_2. Tris(dibenzylideneacetone) dipalladium(0) [$Pd_2(dba)_3$], (R,R)-(-)-2,3-bis(*tert*-butylmethylphosphino)quinoxaline [(R,R)-QuinoxP*] and (R)-(+)-2,2'-bis(di-2-furanylphosphino)-6,6'-dimethoxy-1,1'-biphenyl [(R)-MeO-Fur-BIPHEP] as well as all the achiral bisphosphine ligands were purchased from Strem Chemicals, Inc. Allylboronic acid pinacol ester [allylB(pin)] was generously donated by Frontier Scientific, Inc. All other reagents were purchased from either Fisher or Aldrich and used without further purification.

Experimental Procedures

Preparation of Allylic Carbonates

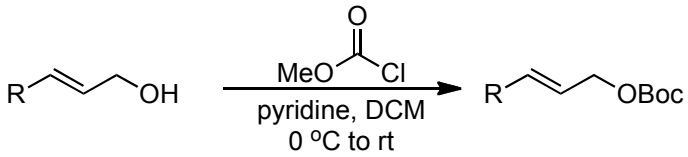


Representative Procedure A:[1] A round-bottomed flask with stir bar was charged with *p*-trifluoromethylcinnamyl alcohol (480 mg, 2.37 mmol) and methylene chloride (2 mL). To the resulting solution was added Boc_2O (570 mg, 2.61 mmol) and Bu_4NHSO_4 (16.0 mg, 0.047 mmol) at room temperature. The solution was cooled to 0 °C and aqueous NaOH (1.2 mL, 30% solution) was added dropwise. The solution was allowed to stir overnight. The reaction mixture was diluted with diethyl ether and water, and was then extracted into diethyl ether three times. The combined organics were washed with 1M HCl, water, then brine, and dried over $MgSO_4$, filtered, then concentrated *in vacuo*. The crude reaction mixture was purified on silica gel (22:1 hexanes: ethyl acetate) to afford 512 mg (72%) of a white solid. R_f = 0.28 (22:1 hexanes: ethyl acetate, stain in $KMnO_4$).



Representative Procedure B:[1] To a flame-dried round-bottomed flask with stir bar was added 1-(naphthalen-1-yl)prop-2-en-1-ol (530 mg, 2.88 mmol) and THF (7 mL). The solution was cooled to -78 °C (dry ice/acetone) and 1.18 mL (2.88 mmol) of a 2.45 M solution of butyllithium in hexane was added, dropwise. The solution was stirred for 30 minutes at -78 °C, Boc_2O (629 mg, 2.88 mmol) in 4 mL THF was added. The reaction was allowed to warm to room temperature, stirring overnight. The reaction mixture was diluted with 10 mL of diethyl ether and 7 mL of water, and the mixture was stirred 15 minutes. The organic layer was separated and the aqueous layer was extracted into diethyl ether three times. Combined organics were washed with brine then dried over $MgSO_4$, filtered, then concentrated *in vacuo*. The crude reaction mixture was purified on silica gel (24:1 hexanes: ethyl acetate) to afford 691 mg (84%) of a clear, colorless oil. R_f = 0.38 (20:1 hexanes: ethyl acetate, stain in $KMnO_4$).

[1] Trost, B. M.; Fraisse, P. L.; Ball, Z. T. *Angew. Chem. Int. Ed.* **2002**, *41*, 1059.



Representative Procedure C:[2] A flame-dried round-bottomed flask with stir bar was charged with (*E*)-dec-2-en-1-ol (1.56 g, 10.0 mmol), methylene chloride (20 mL) and pyridine (1.19 g, 15.0 mmol). The resulting solution was cooled to 0 °C (ice-water) and then methyl chloroformate (570 mg, 2.61 mmol) was added dropwise. The reaction was allowed to stir at this temperature for an hour and then warm up to room temperature for another 12 hours. At this time, water was added, and the organic layer was washed with methylene chloride three times. The combined organic layers were then washed with saturated $CuSO_4$, followed by saturated NH_4Cl and dried over Na_2SO_4, filtered, then concentrated. The crude reaction mixture was purified on silica gel (50:1 hexanes: ethyl acetate) to afford 2.10 g (99%) of a light yellow oil. R_f = 0.45 (8:1 hexanes: ethyl acetate, stain in $KMnO_4$).

Preparation of tert-butyl cinnamyl carbonate **(Table 1; Table 2, entry 1; Scheme 5).** From commercially available cinnamyl alcohol, procedure A was followed. Spectral data is in accordance with literature.[1]

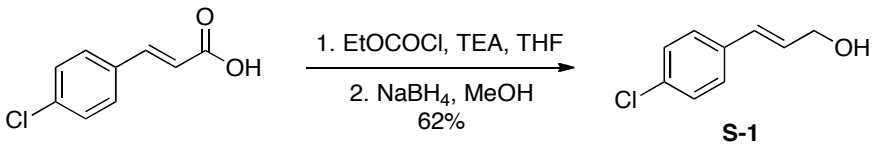
Preparation of cinnamyl methyl carbonate **(Table 2, entry 2; Scheme 3).** From commercially available cinnamyl alcohol, procedure C was followed. Spectral data is in accordance with literature.[3]

Preparation of tert-butyl (1-phenylallyl) carbonate **(Table 2, entry 3).** From commercially available 1-phenylprop-2-en-ol, procedure B was followed. Spectral data is in accordance with literature.[1]

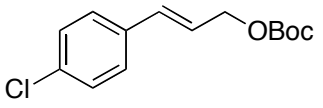
[2] Gnamm, C.; Frank, G.; Miller, N.; Stork, T.; Brödner, K.; Helmchen, G. *Synthesis* **2008**, *20*, 3331.

[3] Su, Y.; Jiao, N. *Org. Lett.* **2009**, *11*, 2980.

Preparation of (E)-tert-butyl (3-(4-chlorophenyl)allyl) carbonate. From allylic alcohol (**S-1**), synthesized as shown below,[4] procedure A was followed.

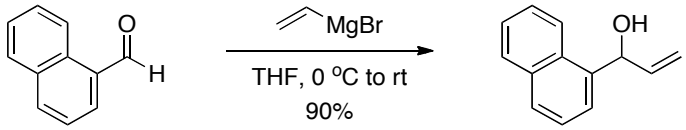


S-1

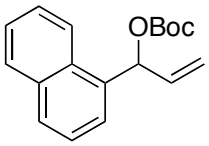


(*E*)-*tert*-butyl (3-(4-chlorophenyl)allyl) carbonate (Table 2, entry 4). ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.50 (9H, s, OC(C**H**$_3$)$_3$), 4.71 (2H, dd, *J* = 6.5 Hz, 1.5 Hz, C**H**$_2$OBoc), 6.27 (1H, dt, *J* = 16.0 Hz, 6.5 Hz, ArCH=C**H**), 6.62 (1H, d, *J* = 16.0 Hz, ArC**H**=CH), 7.28-7.32 (4H, m, Ar**H**); ^{13}C NMR (125 Hz, $CDCl_3$): δ 27.8, 67.2, 82.3, 123.7, 127.8, 128.8, 133.0, 133.8, 134.7, 153.3; IR (neat): 2980.9 (w), 1738.6 (s), 1491.6 (w), 1369.6 (m), 1252.8 (s), 1158.4 (s), 1117.2 (m), 1089.6 (m), 967.5 (w), 846.7 (m), 792.0 (w) cm^{-1}; HRMS (ESI+) for C_9H_8Cl [M-OBoc]: calculated: 151.0309, found: 151.0317; The crude reaction mixture was purified on silica gel (20:1 hexanes: ethyl acetate) to afford a clear, colorless oil (83%). R_f = 0.30 (20:1 hexanes: ethyl acetate, stain in $KMnO_4$).

Preparation of tert-butyl (1-(napthalen-1-yl)allyl) carbonate. From allylic alcohol (**S-2**), synthesized as shown below, procedure B was followed.



S-2



***tert*-butyl (1-(napthalen-1-yl)allyl) carbonate (Table 2, entry 5)**. ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.48 (9H, s, OC(C**H**$_3$)$_3$), 5.30 (1H, app dt, *J* = 10.5 Hz, 1.5 Hz, COCH=C**H**$_{cis}$), 5.35 (1H, app dt, *J* = 17.0 Hz, 1.5 Hz, COCH=C**H**$_{trans}$), 6.21 (1H, ddd, *J* = 17.0 Hz, 10.5 Hz, 5.5 Hz, COC**H**=CH$_2$), 6.78 (1H, d, *J* = 5.5 Hz, ArC**H**), 7.46-7.56 (3H, m, Ar**H**), 7.62 (1H, d, *J* = 7.0 Hz, Ar**H**), 7.83 (1H, d, *J* = 8.0 Hz, Ar**H**), 7.87 (1H, dd, *J* = 7.5 Hz, 1.5 Hz, Ar**H**), 8.12 (1H, d, *J* = 8.5 Hz, Ar**H**); ^{13}C NMR (125 Hz, $CDCl_3$): δ 27.8, 76.3, 82.4, 117.5, 123.6, 125.1, 125.3, 125.7, 126.3, 128.8, 128.9, 130.6, 133.8, 134.4, 135.8, 152.9; IR (neat): 2980.0 (w), 1736.1 (s), 1368.6 (w), 1271.4 (s), 1250.1 (s), 1154.7 (s), 1101.1 (m), 1082.8 (m), 965.0 (m), 930.1 (m), 882.7 (m), 846.4 (m), 775.2 (s), 435.4 (w) cm^{-1}; HRMS (TOF MS ES+) for $C_{18}H_{20}O_3Na$ [M+Na]: calculated: 307.1310, found: 307.1314; The crude reaction mixture was purified on silica gel (25:1 hexanes: ethyl acetate) to afford a clear, colorless oil (84%). R_f = 0.38 (20:1 hexanes: ethyl acetate, stain in $KMnO_4$).

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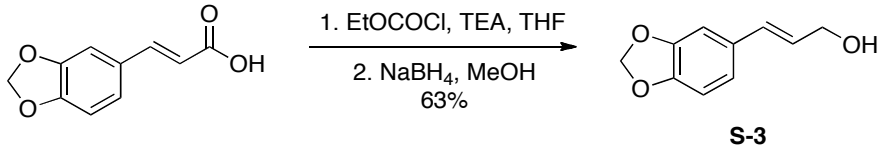
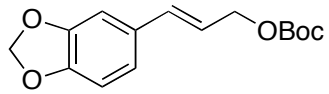
[4] Penjšević, J.; Šukalović, V.; Andrić, D.; Kostić-Rajačić, S.; Šoškic, V.; Roglić, G. *Arch. Pharm. Chem. Life. Sci.* **2007**, *340*, 456.

Preparation of (E)-3-(benzo[d][1,3]dioxol-5-yl)allyl tert-butyl carbonate. From allylic alcohol (**S-3**), synthesized as shown below,[4] procedure A was followed.

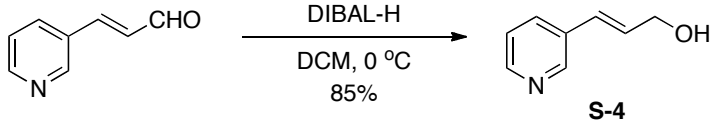


S-3

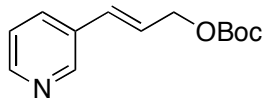


(*E*)-3-(benzo[*d*][1,3]dioxol-5-yl)allyl *tert*-butyl carbonate (Table 2, entry 6). ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.50 (9H, s, OC(C**H$_3$**)$_3$), 4.68 (2H, dd, *J* = 6.5 Hz, 1.5 Hz, C**H$_2$**OBoc), 5.96 (2H, s, OC**H$_2$**O), 6.12 (1H, dt, *J* = 15.5 Hz, 6.5 Hz, ArCH=C**H**), 6.57 (1H, d, *J* = 15.5 Hz, ArC**H**=CH), 6.75 (1H, d, *J* = 8.0 Hz, Ar**H**), 6.82 (1H, dd, *J* = 8.0 Hz, 1.5 Hz, Ar**H**), 6.92 (1H, d, *J* = 1.5 Hz, Ar**H**); ^{13}C NMR (100 Hz, $CDCl_3$): δ 27.8, 67.5, 82.2, 101.1, 105.8, 108.3, 121.0, 121.5, 130.6, 134.3, 147.6, 148.0, 153.3; IR (neat): 2979.3 (w), 1734.7 (s), 1490.1 (m), 1445.0 (m), 1368.6 (m), 1271.7 (s), 1245.5 (s), 1155.5 (s), 1124.0 (w), 1036.2 (s), 963.0 (m), 925.8 (m), 855.1 (s), 792.2 (m), 611.5 (w), 418.3 (w) cm^{-1}; HRMS (ESI+) for $C_{10}H_9O_2$ [M-OBoc]: calculated: 161.0597, found: 161.0604; The crude reaction mixture was purified on silica gel (20:1 hexanes: ethyl acetate) to afford a colorless oil (66%). R_f = 0.12 (20:1 hexanes: ethyl acetate, stain in $KMnO_4$).

Preparation of (E)-tert-butyl (3-(pyridin-3-yl)allyl) carbonate. From allylic alcohol (**S-4**), synthesized as shown below, procedure A was followed.

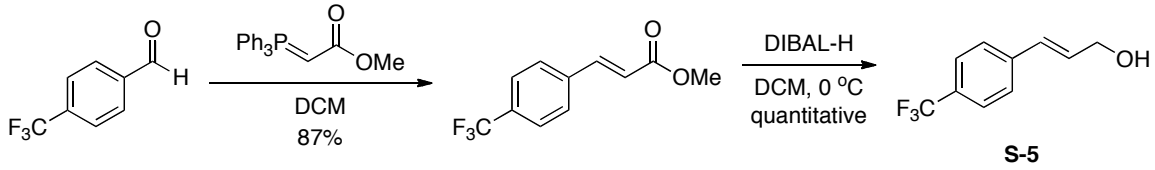


S-4

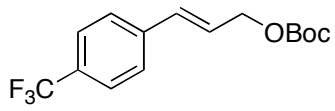


(*E*)-*tert*-butyl (3-(pyridin-3-yl)allyl) carbonate (Table 2, entry 7). ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.44 (9H, s, OC(C**H$_3$**)$_3$), 4.73 (2H, dd, *J* = 6.4 Hz, 1.2 Hz, C**H$_2$**OBoc), 6.36 (1H, dt, *J* = 16.0 Hz, 6.0 Hz, ArCH=C**H**), 6.65 (1H, d, *J* = 16.0 Hz, ArC**H**=CH), 7.23-7.26 (1H, m, Ar**H**), 7.69 (1H, app dt, *J* = 8.0 Hz, 1.6 Hz, Ar**H**), 8.48 (1H, dd, *J* = 4.8 Hz, 1.6 Hz, Ar**H**), 8.60 (1H, s, Ar**H**) ; ^{13}C NMR (100 Hz, $CDCl_3$): δ 27.8, 66.9, 82.5, 123.4, 125.4, 130.4, 131.8, 133.0, 148.5, 149.1, 153.2; IR (neat): 1736.2 (s), 1369.1 (m), 1251.6 (s), 1157.4 (s), 1114.9 (m), 968.2 (m), 861.5 (m), 791.8 (m), 707.0 (m) cm^{-1}; HRMS (ESI+) for $C_{13}H_{18}NO_3$ [M+H]: calculated: 236.1287, found: 236.1290; The crude reaction mixture was purified on silica gel (4:1 hexanes: ethyl acetate with 2% triethylamine) to afford a clear, colorless oil (55%). R_f = 0.12 (4:1 hexanes: ethyl acetate with 2% triethylamine, visualize by UV).

Preparation of (E)-tert-butyl(3-(4-(trifluoromethyl)phenyl)allyl) carbonate. From allylic alcohol (**S-5**), synthesized as shown below, procedure A was followed.

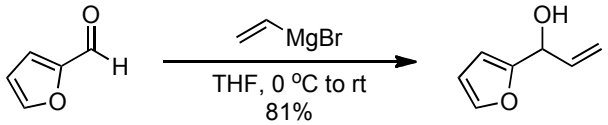


S-5

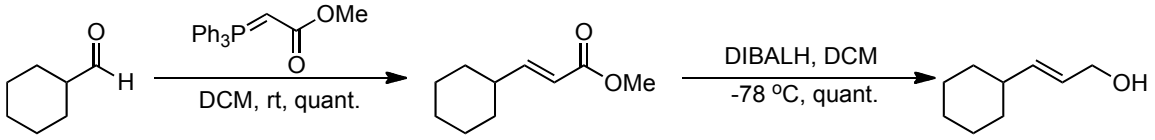


(*E*)-*tert*-butyl(3-(4-(trifluoromethyl)phenyl)allyl) carbonate (Table 2, entry 8). ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.51 (9H, s, OC(C**H$_3$**)$_3$), 4.74 (2H, dd, *J* = 6.4 Hz, 1.6 Hz, C**H$_2$**OBoc), 6.38 (1H, dt, *J* = 16.0 Hz, 6.0 Hz, ArCH=C**H**), 6.70 (1H, d, *J* = 16.0 Hz, ArC**H**=CH), 7.48 (2H, d, *J* = 8.4 Hz, Ar**H**), 7.58 (2H, d, *J* = 8.0 Hz, Ar**H**); ^{13}C NMR (100 Hz, $CDCl_3$): δ 27.8, 66.9, 82.5, 125.6 (q, *J* = 3.7 Hz) 125.7, 126.8, 129.7, 130.0, 132.5, 139.7, 153.3; IR (neat): 2981.7 (w), 1738.3, (s), 1615.8 (w), 1370.0 (w), 1323.7 (s), 1272.0 (s), 1251.8, (s), 1156.5 (s), 1117.7 (s), 1066.3 (s), 968.5 (m), 953.1 (m), 930.9 (w), 852.6 (m), 791.8 (m), 756.1 (w), 597.9 (w) cm^{-1}; HRMS (ESI+) for $C_{10}H_8F_3$ [M-OBoc]: calculated: 185.0573, found: 185.0579; The crude reaction mixture was purified on silica gel (22:1 hexanes: ethyl acetate) to afford a white solid (72%). R_f = 0.28 (22:1 hexanes: ethyl acetate, stain in $KMnO_4$).

Preparation of 1-(furan-2-yl)prop-2-en-1-ol. **(Table 2, entry 9).** The allylic alcohol was synthesized as shown below. Spectral data is in accordance with the literature.[5]



Preparation of (E)-tert-butyl (3-cyclohexylallyl) carbonate. **(Table 2, entry 10).** From allylic alcohol (**S-6**), synthesized as shown below, procedure A was followed. Spectral data is in accordance with the literature.[6]

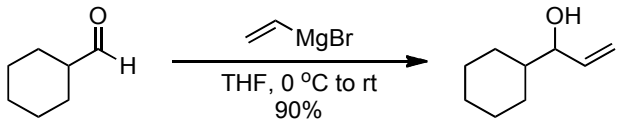


S-6

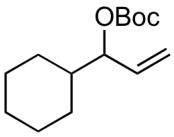
[5] Krauss, J.; Unterreitmeier, D. *Arch. Pharm. Chem. Life. Sci.* **2005**, *338*, 44.

[6] Weix, D.J.; Markovi, D.; Ueda, M.; Hartwig, J. F. *Org. Lett.* **2009**, *11*, 2944.

Preparation of tert-butyl (1-cyclohexylallyl) carbonate. From allylic alcohol (**S-7**), synthesized as shown below, procedure B was followed.

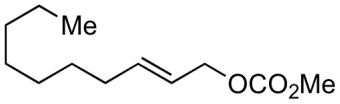


S-7



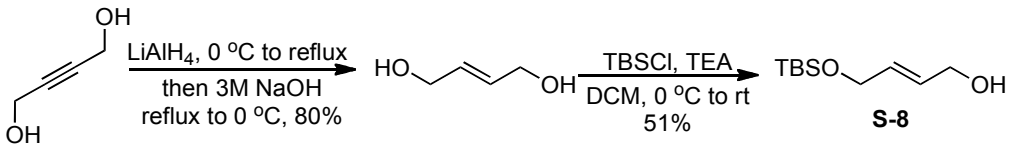
***tert*-butyl (1-cyclohexylallyl) carbonate (Table 2, entry 11)**. ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.88-1.30 (6H, m, Cy-**H**), 1.46 (9H, m, OC(C**H$_3$**)$_3$), 1.46-1.80 (5H, m, Cy-**H**), 4.67 (1H, app t, *J* = 7.2 Hz, CyC**H**O), 5.19 (1H, dt, *J* = 10.4, 1.2 Hz, CH=C**H**$_{cis}$), 5.22 (1H, dt, *J* =17.2, 1.2 Hz, CH=C**H**$_{trans}$), 5.75 (1H, ddd, *J* = 17.2, 10.4, 7.2 Hz, C**H**=CH$_2$); ^{13}C NMR (100 Hz, $CDCl_3$): δ 25.8, 25.9, 26.3, 27.8, 28.4, 28.5, 41.5, 81.6, 82.2, 117.7, 135.0, 153.2; IR (neat): 2980.5 (w), 2927.2 (m), 2854.3 (w), 1737.2 (s), 1451.4 (w), 1368.2 (w), 1272.7 (s), 1250.6 (s), 1160.9 (s), 958.3 (m), 855.0 (m), 792.1 (m) cm^{-1}; HRMS (ESI+) for C_9H_{15} [M-OBoc]: calculated: 123.1174, found: 123.1169; The crude reaction mixture was purified on silica gel (50:1 hexanes: ethyl acetate) to afford a clear, light yellow oil (48%). R_f = 0.52 (8:1 hexanes: ethyl acetate, stain in $KMnO_4$).

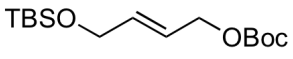
Preparation of (E)-dec-2-enyl methyl carbonate. From commercially available *trans*-2-decen-1-ol, procedure C was followed.



(*E*)-dec-2-enyl methyl carbonate (Table 2, entry 12). ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.86 (3H, t, *J* = 6.8 Hz, CH_2C**H$_3$**), 1.25-1.39 (10H, m, CH_3(C**H$_2$)$_5$**), 2.03 (2H, q, *J* =7.2 Hz, CH_2C**H$_2$**CH=CH), 3.75 (3H, s, OC**H$_3$**), 4.54 (2H, dd, *J* = 6.8, 0.8 Hz, CH=CHC**H$_2$**O), 5.55 (1H, dtt, *J* = 15.6, 6.4, 1.2 Hz, C**H**=CHCH$_2$O), 5.79 (1H, dt, *J* = 15.6, 7.8 Hz, CH=C**H**CH$_2$O); ^{13}C NMR (100 Hz, $CDCl_3$): δ 14.0, 22.6, 28.7, 29.02, 29.04, 31.7, 32.2, 54.5, 68.6, 123.1, 137.5, 155.6; IR (neat): 2955.9 (w), 2925.4 (m), 2855.0 (w), 1747.2 (s), 1441.6 (m), 1379.7 (w), 1252.5 (s), 943.0 (s), 792.0 (m) cm^{-1}; HRMS (ESI+) for $C_{10}H_{19}$ [M-OCO_2Me]: calculated: 137.1487, found: 139.1484; The crude reaction mixture was purified on silica gel (50:1 hexanes: ethyl acetate) to afford a clear, light yellow oil (99%). R_f = 0.45 (8:1 hexanes: ethyl acetate, stain in $KMnO_4$).

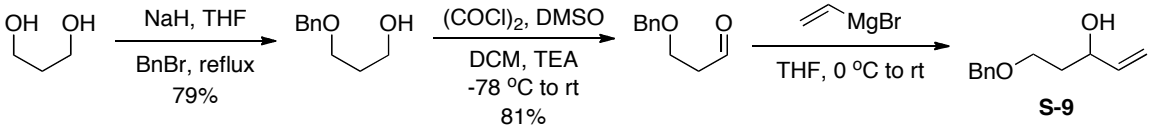
Preparation of (E)-tert-butyl 4-(tert-butyldimethylsilyloxyl)but-2-enyl carbonate. From allylic alcohol (**S-8**), synthesized as shown below, procedure A was followed.

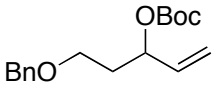




(*E*)-*tert*-butyl 4-(*tert*-butyldimethylsilyloxyl)but-2-enyl carbonate (Table 2, entry 13). ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.03 (6H, Si(C**H$_3$**)$_2$), 0.87 (9H, s, SiC(C**H$_3$**)$_3$), 1.45 (9H, s, OC(C**H$_3$**)$_3$), 4.14-4.16 (2H, m, SiOC**H$_2$**CH=CH), 4.52 (2H, d, *J* = 4.8 Hz, CH=CHC**H$_2$**OBoc), 5.75-5.87 (2H, m, C**H**=C**H**); ^{13}C NMR (100 Hz, $CDCl_3$): δ -5.4, 18.3, 25.8, 27.7, 62.7, 66.8, 81.9, 123.2, 134.4, 153.3; IR (neat): 2954.8 (w), 2930.6 (w), 2886.3 (w), 2857.0 (w), 1740.1 (s), 1462.0 (w), 1391.7 (w), 1368.6 (m), 1274.2 (s), 1161.9 (s), 1106.7 (s), 1050.4 (m), 833.6 (s), 774.7 (s) cm^{-1}; HRMS (ESI+) for $C_{10}H_{21}OSi$ [M-OBoc]: calculated: 185.1362, found: 185.1363; The crude reaction mixture was purified on silica gel (35:1 hexanes: ethyl acetate) to afford a clear, light yellow oil (83%). R_f = 0.41 (8:1 hexanes: ethyl acetate, stain in $KMnO_4$).

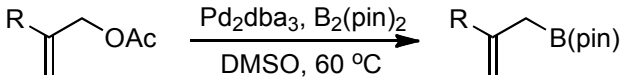
Preparation of 5-(benzyloxy)pent-1-en-3-yl tert-butyl carbonate. From allylic alcohol (**S-9**), synthesized as shown below, procedure B was followed.





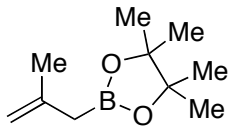
5-(benzyloxy)pent-1-en-3-yl *tert*-butyl carbonate (Table 2, entry 14). ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.48 (9H, s, OC(C**H$_3$**)$_3$), 1.86-1.94 (1H, m, BnOCH$_2$C**H$_A$**), 1.98-2.07 (1H, m, BnOCH$_2$C**H$_B$**), 3.49-3.58 (2H, m, BnOC**H$_2$**), 4.49 (2H, d, *J* = 0.8 Hz, PhC**H$_2$**O), 5.18-5.24 (1H, m, C**H**OBoc), 5.19 (1H, app dt, *J* = 10.4 Hz, 1.2 Hz, COCH=C**H$_{cis}$**), 5.29 (1H, app dt, *J* = 17.2 Hz, 1.2 Hz, COCH=C**H$_{trans}$**), 5.81 (1H, ddd, *J* = 17.2 Hz, 10.4 Hz, 6.8 Hz, COC**H**=CH$_2$), 7.26-7.35 (5H, m, Ph**H**); ^{13}C NMR (100 Hz, $CDCl_3$): δ 27.8, 34.5, 66.1, 73.1, 75.2, 82.0, 117.2, 127.6, 127.7, 128.4, 136.1, 138.3, 152.8; IR (neat): 2979.5 (w), 2931.1 (w), 2863.0 (w), 1738.5, (s), 1455.0 (w), 1368.4 (m), 1273.5 (s), 1253.6, (s), 1161.2 (s), 1100.8 (s), 990.0 (w), 931.3 (m), 854.1 (m), 792.3 (w), 737.7 (m), 697.9 (m) cm^{-1}; HRMS (ESI+) for $C_{17}H_{24}O_4$ [M+H]: calculated: 293.1753, found: 293.1751; The crude reaction mixture was purified on silica gel (100:1 hexanes: ethyl acetate) to afford a clear, colorless oil (59%). R_f = 0.48 (8:1 hexanes: ethyl acetate, stain in $KMnO_4$).

Representative Procedure for the Synthesis of Substituted AllylB(pin):[7]



A flame dried round-bottomed flask with stir bar was charged with tris(dibenzylideneacetone) dipalladium(0) (69.0 mg, 0.075 mmol) and $B_2(pin)_2$ (1.70 g, 6.60 mmol) in a dry-box under an argon atmosphere. The flask was sealed with a septum, and removed from the dry-box. Under an atmosphere of nitrogen, freshly distilled DMSO (18 mL) was added by syringe, followed by methallyl acetate (342 mg, 3.00 mmol). The reaction mixture was then heated to 60 °C in an oil bath for 12 hours. The reaction was diluted with diethyl ether and brine, and the aqueous layer was washed with diethyl ether three times. The combined organic layers were dried over Na_2SO_4, filtered, and concentrated. The crude reaction mixture was purified on silica gel (30:1 pentane: diethyl ether) to afford 338 mg (62%) of a clear, colorless oil. R_f = 0.35 (8:1 hexanes: ethyl acetate, stain in $KMnO_4$).

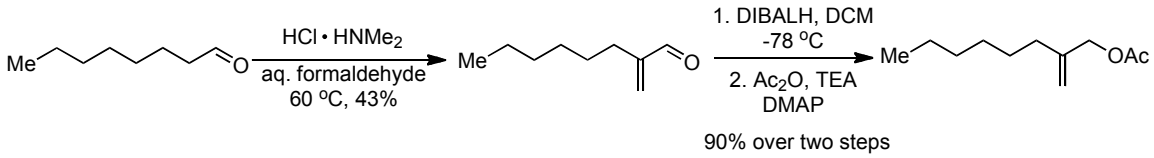
Preparation of 4,4,5,5-tetramethyl-2-(2-methallyl)-1,3,2-dioxaborolane. From commercially available methallyl acetate.

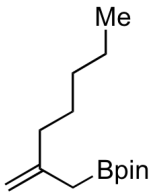


4,4,5,5-tetramethyl-2-(2-methallyl)-1,3,2-dioxaborolane (Scheme 5, equation 1). ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.25 (12H, s, $(C(CH_3)_2)_2$), 1.73 (2H, s, BCH_2), 1.77 (3H, m, $CH_2=CCH_3$), 4.66 (1H, m, $C=CH_AH_B$), 4.68 (1H, m, $C=CH_AH_B$); ^{13}C NMR (125 Hz, $CDCl_3$): 24.5, 24.6, 24.7, 83.3, 110.2, 142.9; IR (neat): 3414.2 (br), 2978.8 (m), 2929.3 (w), 1647.6, (w), 1475.2 (m), 1455.2 (m), 1372.3 (s), 1325.6 (s), 1272.3 (m), 1143.8, (s), 982.1 (m), 881.4 (m), 849.5 (s) cm^{-1}; HRMS (ESI+) for $C_{10}H_{20}BO_2$ [M+H]: calculated: 183.1556, found: 183.1558; The crude reaction mixture was purified on silica gel (50:1 pentane: ether) to afford 336 mg of a clear, colorless oil (62% yield). R_f = 0.35 (30:1 pentane: ether, stain in $KMnO_4$).

[7] Ishiyama, T.; Ahiko, T.; Miyaura, N. *Tetrahedron Lett*. **1996**, *37*, 6889.

Preparation of 4,4,5,5-tetramethyl-2-(2-methyleneoctyl)-1,3,2-dioxaborolane. From 2-methyeneoctyl acetate, synthesized as shown below.



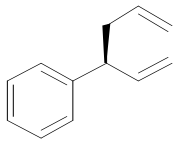


4,4,5,5-tetramethyl-2-(2-methyleneoctyl)-1,3,2-dioxaborolane (Scheme 5, equation 2). ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.23 (3H, t, J = 8.4 Hz, $CH_2C\mathbf{H_3}$), 1.24 (12H, s, $(C(C\mathbf{H_3})_2)_2$), 1.24-1.31 (6H, m, $CH_3(C\mathbf{H_2})_3$), 1.40-1.55 (2H, m, $C\mathbf{H_2}CH_2C{=}CH_2$), 1.71 (2H, s, $C\mathbf{H_2}$B(pin)), 2.04 (2H, t, J = 7.6 Hz, $C\mathbf{H_2}C{=}CH_2$), 4.69 (1H, m, $C{=}C\mathbf{H_A}H_B$), 4.70 (1H, m, $C{=}CH_A\mathbf{H_B}$); ^{13}C NMR (100 Hz, $CDCl_3$): δ 14.1, 22.6, 24.7, 27.6, 29.1, 31.8, 38.1, 83.2, 109.1, 146.8; IR (neat): 3072.7 (w), 2978.7 (m), 2957.2(m), 2927.7 (s), 2857.5 (m), 1743.9 (w), 1641.4 (w), 1466.8 (w), 1378.6 (m), 1325.2 (s), 1272.9 (w), 1144.4 (s), 878.4 (m), 848.4 (m) cm^{-1}; HRMS (ESI+) for $C_{15}H_{30}BO_2$ [M+H]: calculated: 253.2339, found: 253.2341; The crude reaction mixture was purified on silica gel (100:1 pentane: diethyl ether) to afford a clear, colorless oil (11%). R_f = 0.44 (8:1 hexanes: ethyl acetate, stain in $KMnO_4$).

Representative Procedure for Pd-Catalyzed Allyl-Allyl Cross Coupling

An oven-dried 2-dram vial equipped with a magnetic stir bar was charged with tris(dibenzylideneacetone) dipalladium(0) (4.6 mg, 0.005 mmol), (*R*)-(+)-2,2'-bis(di-2-furanylphosphino)-6,6'-dimethoxy-1,1'-biphenyl (5.4 mg, 0.010 mmol), and 0.20 mL of THF in a dry-box under an argon atmosphere. The vial was capped and stirred for 5 minutes, then *tert*-butyl cinnamyl carbonate (23.4 mg, 0.100mmol) was added, followed by allylboronic acid pinacol ester (20.1 mg, 0.120 mmol). The vial was sealed, removed from the dry-box, and allowed to stir at 60 °C for 12 hours. After this time period, the reaction vial was cooled to ambient temperature, diluted with diethyl ether, filtered through a plug of silica gel and concentrated *in vacuo*. Analysis of the unpurified reaction mixture using ^{1}H NMR was used to determine the branched to linear product ratio. Silica gel chromatography (pentane) afforded 11.4 mg (72%) of a colorless oil of the allyl-allyl coupling product as a mixture of isomers.

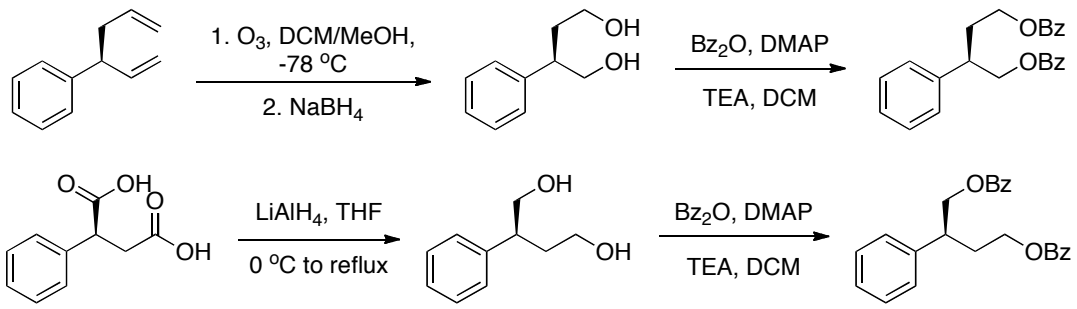
Characterization and Proof of Stereochemistry



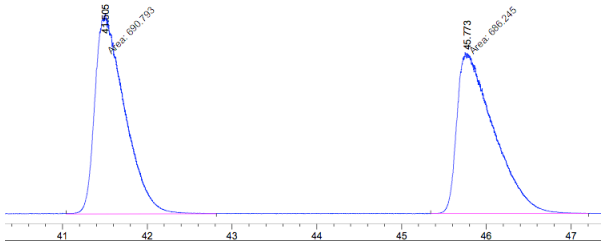
(*S*)-hexa-1,5-dien-3-ylbenzene. ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.47-2.51 (2H, m, CHC**H$_2$**CH=CH$_2$), 3.36 (1H, app q, *J* = 7.5 Hz, C**H**CH$_2$CH=CH$_2$), 4.96-5.07 (4H, m, CHCH=C**H$_2$** & CH$_2$CH=C**H$_2$**), 5.73 (1H, ddt, *J* = 17.0 Hz, 10.0 Hz, 7.5 Hz, CH$_2$C**H**=CH$_2$), 5.98 (1H, ddd, *J* = 17.0 Hz, 10.0 Hz, 7.5 Hz, CHC**H**=CH$_2$), 7.19-7.22 (3H, m, Ph**H**), 7.29-7.32 (2H, m, Ph**H**); ^{13}C NMR (100 MHz, $CDCl_3$): δ 39.7, 49.6, 114.4, 116.1, 126.3, 127.7, 128.4, 136.6, 141.6, 143.7; IR (neat): 3078.0 (w), 3028.0 (w), 3003.9 (w), 2977.9 (w), 2924.3 (w), 1631.0 (s), 1601.2 (w), 1492.2 (s), 1452.0 (s), 1415.0 (w), 1073.2 (w), 991.4 (m), 910.2 (s), 753.1 (m), 697.6 (s) cm^{-1}; HRMS (ESI+) for $C_{12}H_{15}$ [M+H]: calculated: 159.1174, found: 159.1176; $[\alpha]^{20}_{D}$ = +12.237 (*c* = 0.44, $CHCl_3$). The crude reaction mixture was purified on silica gel (pentane) to afford a clear, colorless oil (11.4 mg, 72% yield). R_f = 0.38 (pentane, stain in $KMnO_4$).

Proof of Stereochemistry:

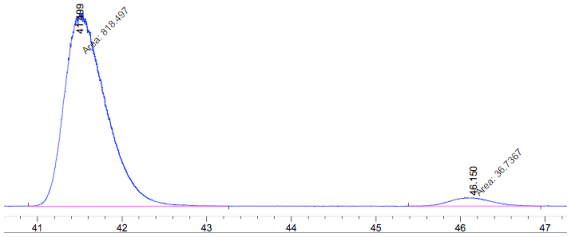
Enantioselectivity was determined by comparison of the title compound with racemic material prepared using 1,2-bis(diphenylphosphino)benzene as the achiral ligand in the allyl-allyl coupling reaction. Absolute stereochemistry was determined by converting the allyl-allyl coupling product to a dibenzoate by ozonolysis/reduction and benzoate protection of the corresponding diol, as shown below. *Via* chiral HPLC, the resulting dibenzoate was compared to authentic (*S*)-2-phenylbutane-1,4-diyl dibenzoate, which was derived from commercially available (*S*)-2-phenylsuccinic acid.



Chiral GLC (CD-GTA, Supelco, 60 °C, 25 psi) - analysis of title compound



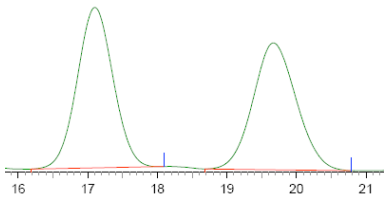
racemic



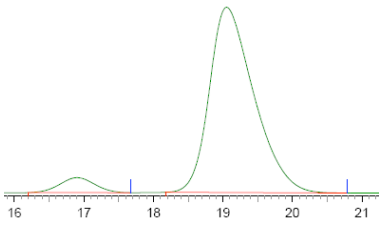
reaction product

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	41.499	MM	0.5666	818.49750	24.07773	95.70448
2	46.150	MM	0.5839	36.73672	1.04862	4.29552

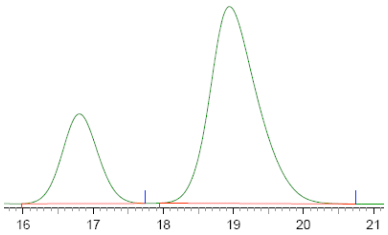
Chiral HPLC (OD-R, Chiralcel, 1 mL/min, 1% isopropanol, 254 nm) – analysis of 2-phenylbutane-1,4-diyl dibenzoate



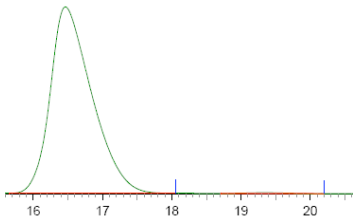
racemic



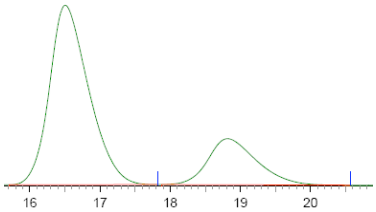
derived from reaction product



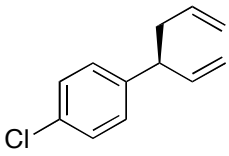
derivative + racemic



(*S*)-2-phenylbutane-1,4-diyl dibenzoate



(*S*)-2-phenylbutane-1,4-diyl dibenzoate + racemic

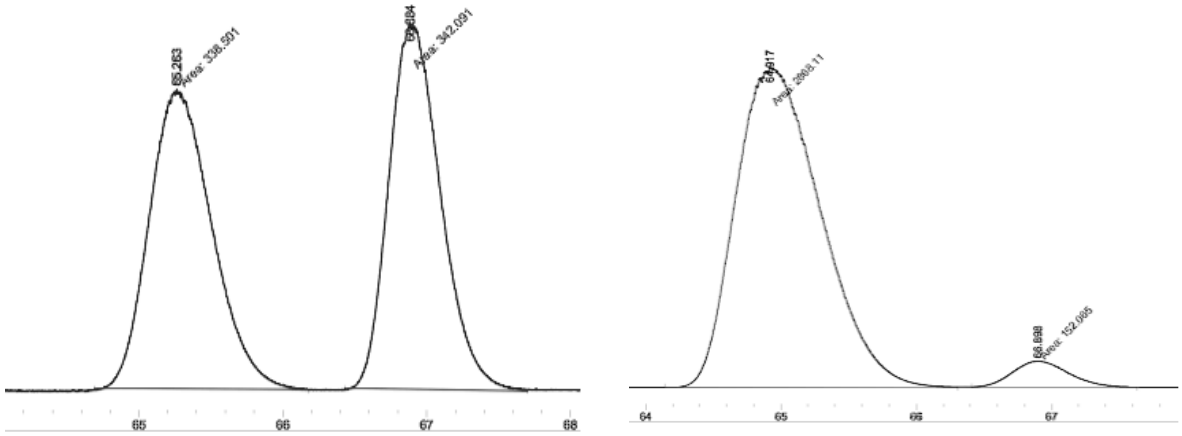


(*S*)-1-chloro-4-(hexa-1,5-dien-3-yl)benzene. ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.46 (2H, app dtd, *J* = 21.5 Hz, 14.0 Hz, 7.5 Hz, ArCHC**H$_2$**CH=CH$_2$), 3.34 (1H, app q, *J* = 7.5 Hz, ArC**H**CH=CH$_2$), 4.96-5.08 (4H, m, CH$_2$CH=C**H$_2$** & ArCHCH=C**H$_2$**), 5.69 (1H, app ddt, *J* = 17.0 Hz, 10.0 Hz, 7.0 Hz, CH$_2$C**H**=CH$_2$), 5.94 (1H, ddd, *J* = 17.5, 10.5, 7.5 Hz, ArCHC**H**=CH$_2$), 7.12 (2H, app dt, *J* = 8.5 Hz, 2.5 Hz, Ar**H**), 7.27 (2H, app dt, *J* = 9.0 Hz, 2.5 Hz, Ar**H**); ^{13}C NMR (125 Hz, $CDCl_3$): δ 39.6, 48.9, 114.8, 116.5, 128.5, 129.1, 132.0, 136.2, 141.1, 142.1; IR (neat): 3078.8 (w), 2978.4 (w), 2925.5 (w, br), 1640.1 (w), 1490.9 (s), 1406.8 (w), 1091.7 (s), 1014.4 (m), 922.2 (m), 913.9 (s), 826.9 (m), 523.8 (w) cm^{-1}; HRMS (ESI+) for $C_{12}H_{14}Cl$ [M+H]: calculated: 193.0784, found: 193.0793; $[\alpha]^{20}_{D}$ = +24.816 (*c* = 0.64, $CHCl_3$). The crude reaction mixture was purified on silica gel (pentane) to afford a clear, colorless oil (17.9 mg, 59% yield). R_f = 0.6 (pentane, stain in $KMnO_4$).

Proof of Stereochemistry:

Enantioselectivity was determined by comparison of the title compound with racemic material prepared using 1,2-bis(diphenylphosphino)benzene as the achiral ligand in the allyl-allyl coupling. The absolute stereochemistry was assigned by analogy.

Chiral GLC (CD-GTA, Supelco, 80 °C for 60 min, ramp 3 °C/min to 120 °C, 25 psi) – analysis of the title compound.



Racemic Reaction Product

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
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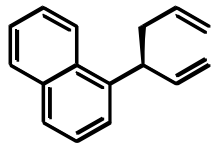
2	66.898	MM	0.4733	152.06499	5.35460	5.03496

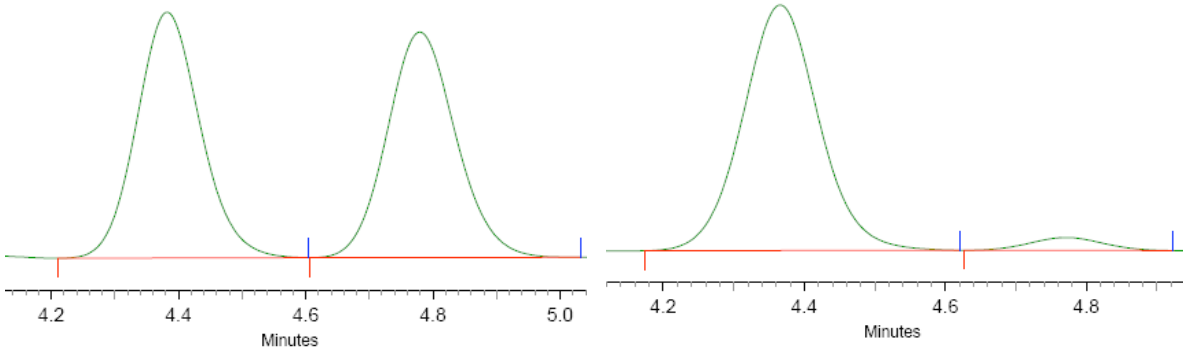


(*S*)-1-(hexa-1,5-dien-3-yl)naphthalene. ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.66-2.69 (2H, m, ArCHC**H$_2$**CH=CH$_2$), 4.23 (1H, app q, *J* = 7.0 Hz, ArC**H**CH=CH$_2$), 4.99-5.14 (4H, m, CH$_2$CH=C**H$_2$** & ArCHCH=C**H$_2$**), 5.83 (1H, app ddt, *J* = 17.0 Hz, 10.0 Hz, 7.0 Hz, CH$_2$C**H**=CH$_2$), 6.12 (1H, ddd, *J* = 17.0, 10.5, 7.0 Hz, ArCHC**H**=CH$_2$), 7.40 (1H, d, *J* = 7.0 Hz, Ar**H**), 7.44-7.54 (3H, m, Ar**H**), 7.74 (1H, d, *J* = 8.0 Hz, Ar**H**), 7.87 (1H, dd, *J* = 7.0 Hz, 0.5 Hz, Ar**H**), 8.12 (1H, d, *J* = 8.0 Hz, Ar**H**); ^{13}C NMR (100 Hz, $CDCl_3$): δ 39.3, 43.9, 115.0, 116.2, 123.4, 124.2, 125.4, 125.5, 125.8, 126.9, 128.9, 131.6, 134.0, 136.8, 139.7, 141.1; IR (neat): 3090.1 (w), 1638.2 (w), 992.1 (w), 911.9 (m), 796.3 (w), 776.4 (s) cm^{-1}; HRMS (ESI+) for $C_{16}H_{17}$ [M+H]: calculated: 209.1330, found: 209.1334; $[\alpha]^{20}_{D}$ = +26.602 (*c* = 0.89, $CHCl_3$). The crude reaction mixture was purified on silica gel (pentane) to afford a clear, colorless oil (27.1 mg, 87% yield). R_f = 0.29 (pentane, stain in $KMnO_4$).

Proof of Stereochemistry:

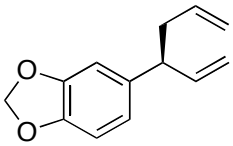
Enantioselectivity was determined by comparison of the title compound with racemic material prepared using 1,2-bis(diphenylphosphino)benzene as the achiral ligand in the allyl-allyl coupling. The absolute stereochemistry was assigned by analogy.

Chiral HPLC (OD-R, Chiraldex, 1 mL/min, 1% isopropanol in hexanes, 254 nm) – analysis of the title compound.



Racemic Reaction Product

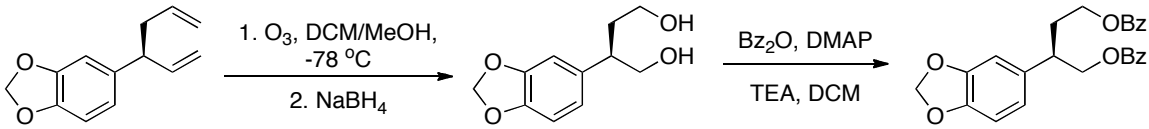
Retention Time	Area	Area %	Height	Height %
4.367	188084170	95.24	24956068	95.03
4.770	9408939	4.76	1303835	4.97



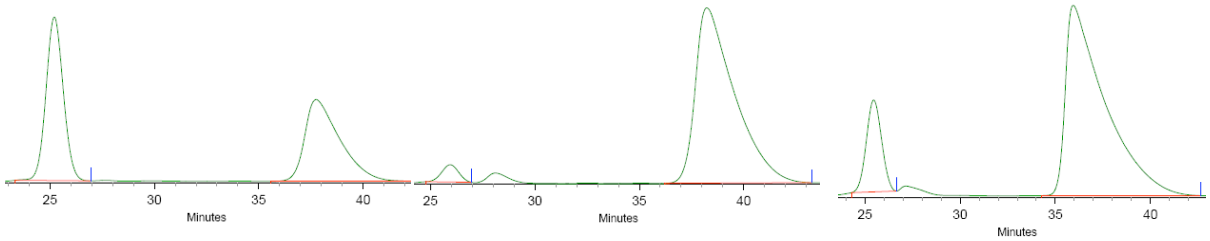
(*S*)-5-(hexa-1,5-dien-3-yl)benzo[*d*][1,3]dioxole. ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.38-2.50 (2H, m, ArCHC**H**$_2$CH=CH_2), 3.28 (1H, app q, *J* = 7.6 Hz, ArC**H**CH=CH_2), 4.95-5.06 (4H, m, CH_2CH=C**H**$_2$ & ArCHCH=C**H**$_2$), 5.71 (1H, app ddt, *J* = 16.8 Hz, 12.5 Hz, 6.8 Hz, CH_2C**H**=CH_2), 5.93 (2H, s, OC**H**$_2$O), 5.93 (1H, ddd, *J* = 17.6 Hz, 10.4 Hz, 7.6 Hz, ArCHC**H**=CH_2), 6.64 (1H, dd, *J* = 8.0 Hz, 1.6 Hz, Ar**H**), 6.69 (1H, d, *J* = 1.6 Hz, Ar**H**), 6.74 (1H, d, *J* = 8.0 Hz, Ar**H**); ^{13}C NMR (125 Hz, $CDCl_3$): δ 39.8, 49.3, 100.8, 108.0, 108.1, 114.2, 116.1, 120.6, 136.5, 137.7, 141.6, 145.9, 147.6; IR (neat): 2895.2 (w, br), 1639.0 (w), 1503.0 (s), 1486.6 (s), 1440.4 (m), 1245.3 (s), 1039.9 (s), 913.7 (s), 809.8 (w) cm^{-1}; HRMS (ESI+) for $C_{13}H_{15}O_2$ [M+H]: calculated: 203.1072, found: 203.1079; $[\alpha]^{20}_D$ = +22.830 (*c* = 0.69, $CHCl_3$). The crude reaction mixture was purified on silica gel (80:1 pentane:diethyl ether) to afford a clear, colorless oil (23.4 mg, 83% yield). R_f = 0.32 (60:1 pentane:diethyl ether, stain in $KMnO_4$).

Proof of Stereochemistry:

The title compound was subjected to ozonolysis and reduction. The resulting diol was protected with benzoic anhydride to afford the dibenzoate ester for HPLC analysis, as depicted below. The analogous racemic material was prepared via the same route using 1,2-bis(diphenylphosphino)benzene as achiral ligand in the allyl-allyl coupling reaction. The absolute stereochemistry was assigned by analogy.

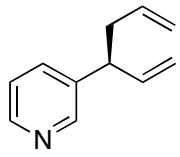


Chiral HPLC (OD-R, Chiraldex, 1 mL/min, 3% isopropanol in hexanes, 220 nm) – analysis of the dibenzoate ester.



Racemic Reaction Product Reaction Product + Racemic

Retention Time	Area	Area %	Height	Height %
25.947	9404811	4.10	166554	8.98
38.227	220148832	95.90	1687816	91.02

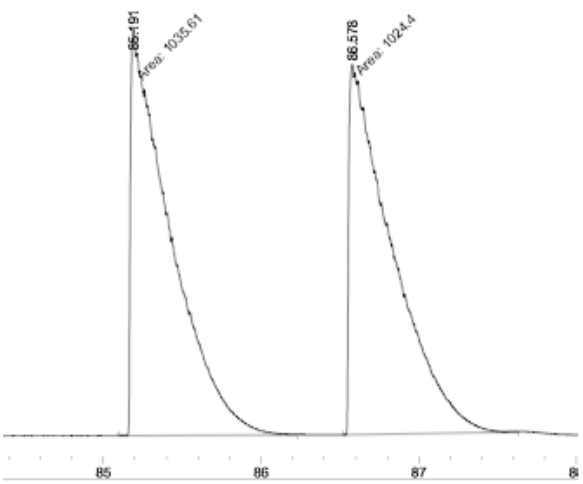


(*S*)-3-(hexa-1,5-dien-3-yl)pyridine. ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.44-2.57 (2H, m, ArCHC**H**$_2$CH=CH_2), 3.40 (1H, app q, *J* = 7.0 Hz, ArC**H**CH=CH_2), 4.98-5.01 (2H, m, CH_2CH=C**H**$_2$), 5.05 (1H, app dt, *J* = 17.0 Hz, 1.0 Hz, ArCHCH=C**H**$_{trans}$H), 5.11 (1H, app dt, *J* = 10.5 Hz, 1.0 Hz, ArCHCH=C**H**$_{cis}$H), 5.70 (1H, app ddt, *J* = 17.5 Hz, 10.5 Hz, 7.0 Hz, CH_2C**H**=CH_2), 5.97 (1H, ddd, *J* = 17.0 Hz, 10.5 Hz, 7.0 Hz, ArCHC**H**=CH_2), 7.23 (2H, m, Ar**H**), 7.50 (1H, app dt, *J* = 8.0 Hz, 2.0 Hz, Ar**H**), 8.46 (1H, s, Ar**H**); ^{13}C NMR (125 Hz, $CDCl_3$): δ 39.5, 46.9, 115.4, 116.9, 123.3, 135.1, 135.7, 138.8, 140.4, 147.8, 149.7; IR (neat): 3078.9 (w), 2925.5 (w), 1640.0 (w), 1574.5 (w), 1478.3 (w), 1423.6 (m), 1025.2 (w), 993.1 (m), 914.9 (s), 810.6 (w), 715.7 (s), 401.4 (w) cm^{-1}; HRMS (ESI+) for $C_{11}H_{14}N$ [M+H]: calculated: 160.1126, found: 160.1119; $[\alpha]^{20}_{D}$ = +21.938 (*c* = 0.550, $CHCl_3$). The crude reaction mixture was purified on silica gel pretreated with 2% triethylamine in column eluent (3:1 pentane:diethyl ether) to afford a clear, light yellow oil (12.5 mg, 52% yield). R_f = 0.17 (3:1 pentane:diethyl ether, visualize by UV).

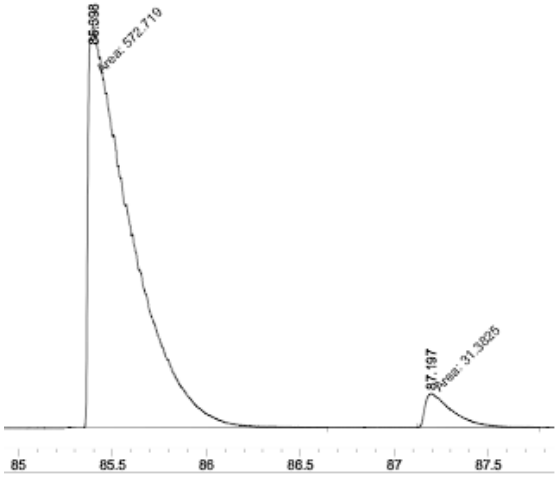
Proof of Stereochemistry:

Enantioselectivity was determined by comparison of the title compound with racemic material prepared using 1,2-bis(diphenylphosphino)benzene as the achiral ligand in the allyl-allyl coupling. The absolute stereochemistry was assigned by analogy.

Chiral GLC (CD-GTA, Supelco, 60 °C for 60 min, ramp 2 °C/min to 100 °C, 25 psi) – analysis of the title compound.

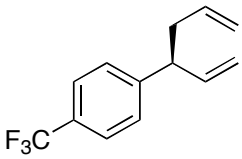


Racemic



Reaction Product

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	85.398	MM	0.2594	572.71948	36.80141	94.80510
2	87.197	MM	0.1692	31.38249	3.09128	5.19490

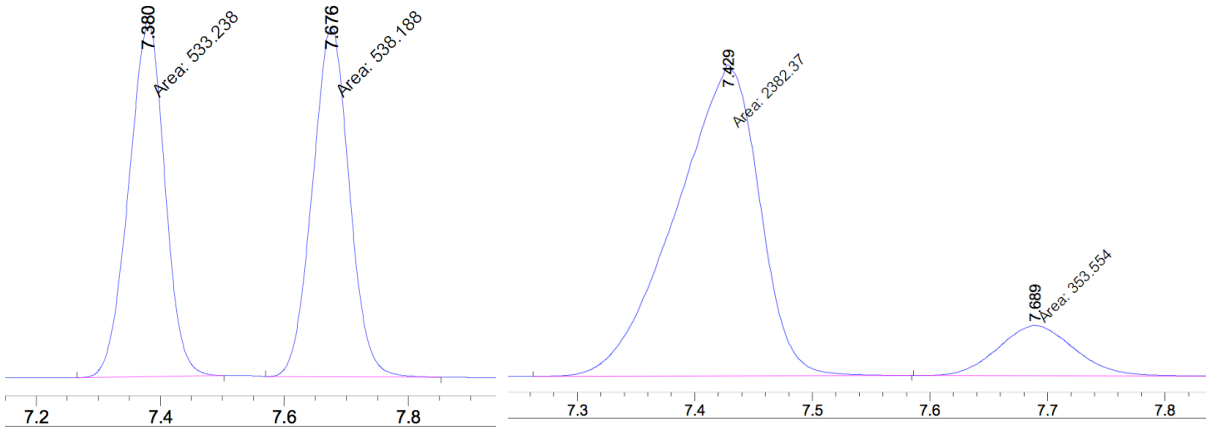


(*S*)-1-(hexa-1,5-dien-3-yl)-4-(trifluoromethyl)benzene. ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.44-2.56 (2H, m, ArCHC**H$_2$**CH=CH$_2$), 3.43 (1H, app q, *J* = 7.5 Hz, ArC**H**CH=CH$_2$), 4.97-5.11 (4H, m, CH$_2$CH=C**H$_2$** & ArCHCH=C**H$_2$**), 5.69 (1H, dddd, *J* = 17.0 Hz, 10.0 Hz, 7.0 Hz, 7.0 Hz, CH$_2$C**H**=CH$_2$), 5.96 (1H, ddd, *J* = 17.5, 10.5, 7.0 Hz, ArCHC**H**=CH$_2$), 7.30 (2H, dd, *J* = 8.0 Hz, 0.5 Hz, Ar**H**), 7.56 (2H, d, *J* = 8.0 Hz, Ar**H**); ^{13}C NMR (125 Hz, $CDCl_3$): δ 39.5, 49.4, 115.3, 116.7, 125.4 (q, *J* = 3.75 Hz), 128.1, 135.9, 140.6, 147.7; IR (neat): 2922.3 (s), 2851.2 (m), 2166.2 (m), 2036.7 (m), 2019.9 (m), 2004.6 (m), 1961.1 (w), 1325.7 (w), 485.1 (w), 453.4 (m), 438.2 (m), 421.5 (m) cm^{-1}; HRMS (ESI+) for $C_{12}H_{14}Cl$ [M+H]: calculated: 227.1048, found: 227.1047; $[\alpha]^{20}_D$ = +16.478 (*c* = 0.985, $CHCl_3$). The crude reaction mixture was purified on silica gel (pentane) to afford a clear, colorless oil (20.3 mg, 60% yield). R_f = 0.63 (pentane, stain in $KMnO_4$).

Proof of Stereochemistry:

Enantioselectivity was determined by comparison of the title compound with racemic material prepared using 1,2-bis(diphenylphosphino)benzene as the achiral ligand in the allyl-allyl coupling. The absolute stereochemistry was assigned by analogy.

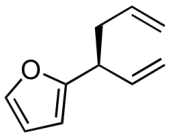
Chiral GLC (CD-GTA, Supelco, 100 °C, 25 psi) – analysis of the title compound.



Racemic

Reaction Product

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	7.429	MM	0.0821	2382.36694	483.47037	87.07733
2	7.689	MM	0.0752	353.55417	78.31371	12.92267

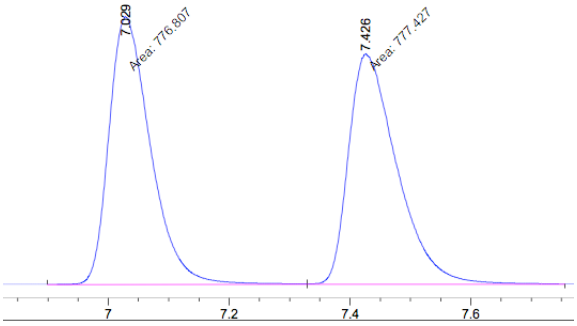


(*S*)-2-(hexa-1,5-dien-3-yl)furan. ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.43 (1H, ddd, *J* = 14.0, 7.5, 7.0 Hz, CHC**H$_a$**H$_b$CH=CH$_2$), 2.56 (1H, app dtt, *J* = 14.0, 7.0, 7.0, 1.5 Hz, CHCH$_a$**H$_b$**CH=CH$_2$), 3.47 (1H, app q, *J* = 7.5 Hz, FurC**H**CH=CH$_2$), 5.00-5.12 (4H, m, CHCH=C**H$_2$** & CH$_2$CH=C**H$_2$**), 5.75 (1H, app ddt, *J* = 17.0, 10.0, 7.0 Hz, CH$_2$C**H**=CH$_2$), 5.87 (1H, ddd, *J* = 17.0, 10.5, 8.0 Hz, CHC**H**=CH$_2$), 6.04 (1H, dt, *J* = 3.0, 1.0 Hz, Fur-**H**), 6.30 (1H, dd, *J* = 3.0, 2.0 Hz, Fur-**H**), 7.34 (1H, dd, *J* = 2.0, 1.0 Hz, Fur-**H**); ^{13}C NMR (125 Hz, $CDCl_3$): δ 37.7, 43.2, 105.1, 110.0, 115.7, 116.5, 132.9, 138.5, 141.2, 156.7; IR (neat): 2922.3 (s), 2851.9 (m), 1793.3 (w), 1727.3 (w), 1641.2 (w), 1462.8 (w), 1377.4 (w), 1274.1 (w), 1125.0 (w), 1077.4 (w), 823.4 (w) cm^{-1}; HRMS (ESI+) for $C_{10}H_{13}O$ [M+H]: calculated: 149.0966, found: 149.0968; $[\alpha]^{20}_D$ = +32.168 (*c* = 0.53, $CHCl_3$). The crude reaction mixture was purified on silica gel (pentane) to afford a clear, light yellow oil (9.5 mg, 64% yield). R_f = 0.56 (8:1 hexanes: ethyl acetate, stain in $KMnO_4$).

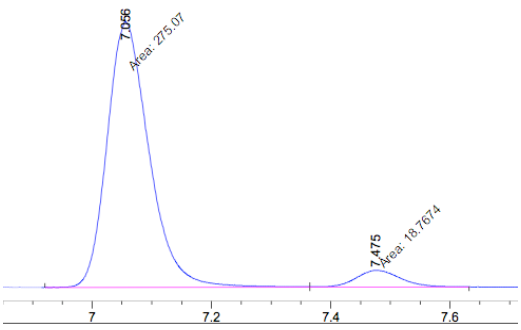
Proof of Stereochemistry:

Enantioselectivity was determined by comparison of the title compound with racemic material prepared using 1,2-bis(diphenylphosphino)benzene as the achiral ligand in the allyl-allyl coupling. The absolute stereochemistry was assigned by analogy.

Chiral GLC (CD-GTA, Supelco, 70 °C, 25 psi) – analysis of the title compound.

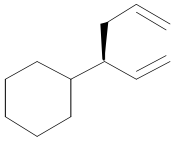


Racemic



Reaction product

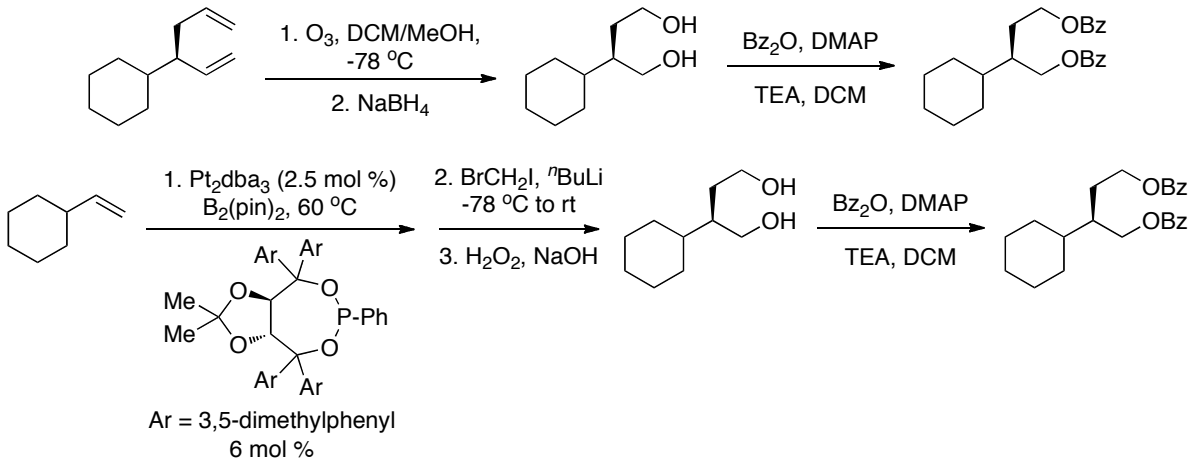
Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	7.056	MF	0.0811	275.07043	56.54135	93.61302
2	7.475	FM	0.0875	18.76736	3.57612	6.38698



(*S*)-hexa-1,5-dien-3-ylcyclohexane. ^{1}H NMR (500 Hz, $CDCl_3$): δ 0.87-1.30 (6H, m, (C**H**$_2$)$_3$), 1.61-1.73 (5H, m, C**H**$_2$C**H**C**H**$_2$), 1.89 (1H, m, C**H**CH=CH$_2$), 2.03-2.09 (1H, m, C**H**$_a$CH$_b$CH=CH$_2$), 2.19-2.24 (1H, m, CH$_a$C**H**$_b$CH=CH$_2$), 4.89-5.01 (4H, m, CHCH=C**H**$_2$ & CH$_a$CH$_b$CH=C**H**$_2$), 5.59 (1H, ddd, *J* = 19.5 Hz, 10.5 Hz, 9.5 Hz, CHC**H**=CH$_2$), 5.74 (1H, app ddt, *J* = 17.2 Hz, 10.4 Hz, 6.8 Hz, CH$_2$C**H**=CH$_2$); ^{13}C NMR (100 Hz, $CDCl_3$): δ 26.60, 26.63, 26.7, 29.4, 31.1, 36.4, 41.0, 49.8, 115.0, 115.2, 137.8, 140.9 ppm; IR (neat): 3075.2 (w), 2976.9 (w), 2921.2 (s), 2851.4 (s), 1639.8 (s), 1447.9 (m), 1419.4 (w), 993.9 (m), 908.2 (s), 704.9 (w) cm^{-1}; HRMS (ESI+) for $C_{12}H_{21}$ [M+H]: calculated: 165.1643, found: 165.1650; $[\alpha]^{20}_D$ = -4.322 (*c* = 0.62, $CHCl_3$). The crude reaction mixture was purified on silica gel (pentane) to afford a clear, colorless oil (10.3 mg, 63% yield of title compound). Mixture of branched to linear compounds: 10:1. R_f = 0.85 (8:1 hexane: ethyl acetate, stain in $KMnO_4$).

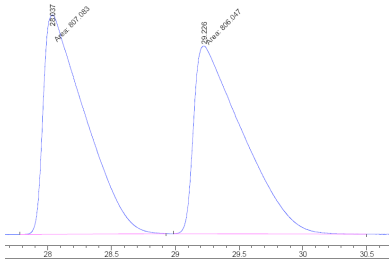
Proof of Stereochemistry:

Enantioselectivity was determined by comparison of the title compound with authentic racemic material prepared using 1,2-bis(diphenylphosphino)benzene as the achiral ligand in the allyl-allyl coupling reaction. Absolute stereochemistry was determined by converting the allyl-allyl coupling product to a dibenzoate, by ozonolysis/reduction and benzoate protection of the corresponding diol, as shown below. *Via* chiral HPLC the resulting dibenzoate was compared to (*R*)-2-cyclohexylbutane-1,4-diyl dibenzoate, which was prepared by diboration/homologation/oxidation of vinylcyclohexane, followed by dibenzoate protection as shown below.[8]

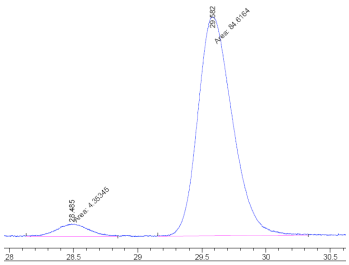


[8] Kliman, L. T.; Mlynarski, S. N.; Morken, J. P.; *J. Am. Chem. Soc.*, **2009**, *131*, 13210.

Chiral GLC (β-dex, Supelco, 80 °C, 25 psi) - analysis of title compound



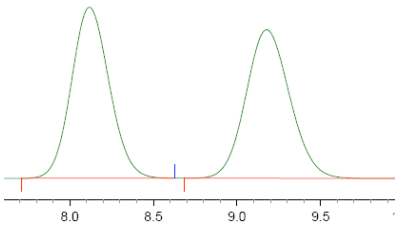
racemic



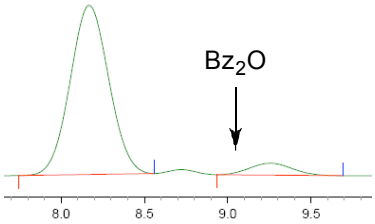
reaction product

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	28.486	MM	0.2998	4.78294	2.65896e-1	5.32266
2	29.580	MM	0.3141	85.07700	4.51380	94.67734

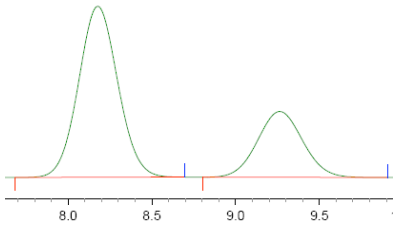
Chiral HPLC (OD-R, Chiralcel, 1 mL/min, 1% isopropanol, 220 nm) – analysis of 2-cyclohexylbutane-1,4-diyl dibenzoate



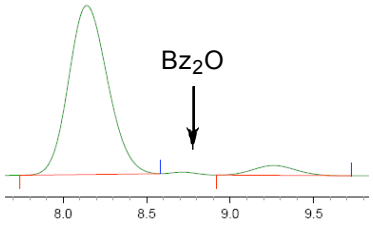
racemic



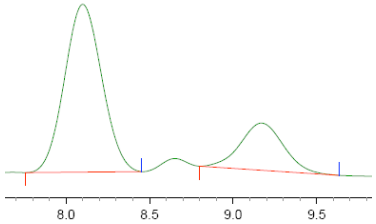
derived from reaction product



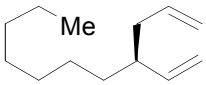
derivative + racemic



(*R*)-2-cyclohexylbutane-1,4-diyl dibenzoate



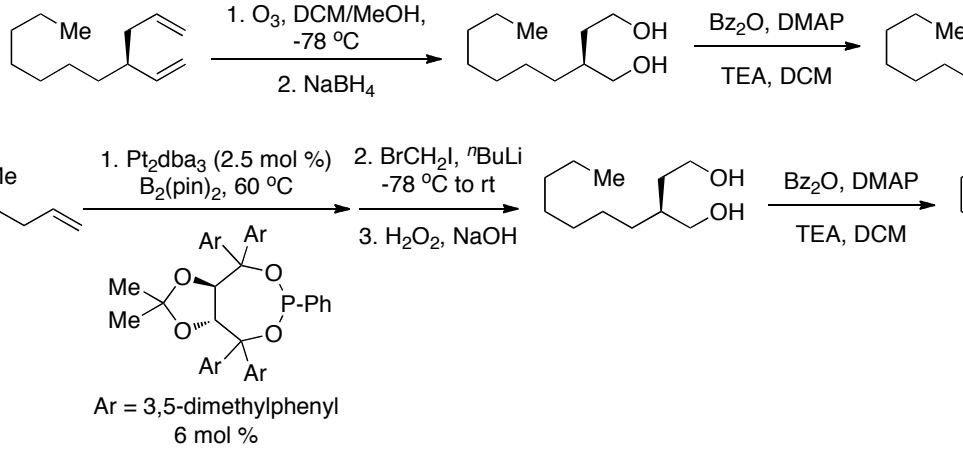
(*R*)-2-cyclohexylbutane-1,4-diyl dibenzoate + racemic



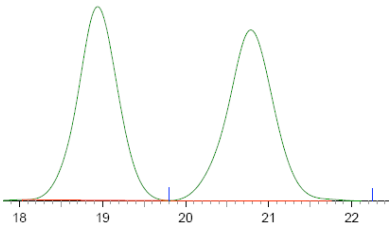
(*S*)-4-vinylundec-1-ene. ^{1}H NMR (400 Hz, $CDCl_3$): δ 0.88 (3H, t, *J* = 6.8 Hz, C**H$_3$**), 1.22-1.38 (12H, m, $CH_3(CH_2)_6$), 2.01-2.14 (3H, m, C**H**CH=CH_2 & C**H$_2$**CH=CH_2), 4.92-5.02 (4H, m, CHCH=C**H$_2$** & CH_2CH=C**H$_2$**), 5.58 (1H, ddd, *J* = 16.8 Hz, 10.4 Hz, 8.0 Hz, CHC**H**=CH_2), 5.76 (1H, app ddt, *J* = 17.2 Hz, 10.4 Hz, 6.8 Hz, CH_2C**H**=CH_2); ^{13}C NMR (100 Hz, $CDCl_3$): δ 14.1, 22.7, 27.1, 29.3, 29.7, 31.9, 34.2, 39.5, 43.7, 114.1, 115.5, 137.2, 142.8; IR (neat): 3077.2 (w), 2957.1 (m), 2923.3 (s), 2854.2 (m), 1641.0 (s), 1465.1 (s), 1419.4 (w), 1378.1 (w), 992.6 (m), 909.4 (s) cm^{-1}; HRMS (ESI+) for $C_{13}H_{25}$ [M+H]: calculated: 181.1956, found: 181.1958; $[\alpha]^{20}_D$ = -2.828 (*c* = 0.76, $CHCl_3$). The crude reaction mixture was purified on silica gel (pentane) to afford a clear, colorless oil (14.4 mg, 80% yield of title compound). Mixture of branched to linear compounds: 11:1. R_f = 0.86 (8:1 hexane: ethyl acetate).

Proof of Stereochemistry:

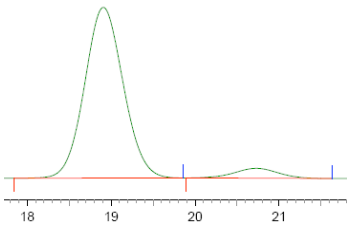
Enantioselectivity was determined by converting the allyl-allyl coupling product to a dibenzoate by ozonolysis/reduction and benzoate protection of the corresponding diol as shown below. *Via* chiral HPLC the resulting dibenzoate was compared to racemic material prepared using 1,2-bis(diphenylphosphino)benzene as the achiral ligand in the allyl-allyl coupling reaction. Absolute stereochemistry was determined by comparing the dibenzoate to authentic (*S*)-2-heptylbutane-1,4-diyl dibenzoate which was prepared by diboration/homologation/oxidation of 1-nonene, followed by dibenzoate protection as shown below.[8]



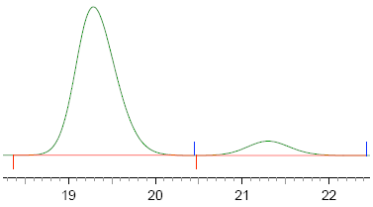
Chiral HPLC (OD-R, Chiralcel, 0.5 mL/min, 1% isopropanol, 220 nm) – analysis of 2-heptylbutane-1,4-diyl dibenzoate



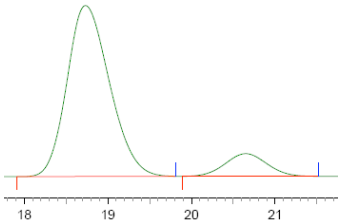
racemic



(*S*)-2-heptylbutane-1,4-diyl dibenzoate

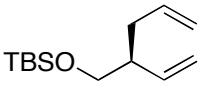


derived from reaction product



derivative + racemic

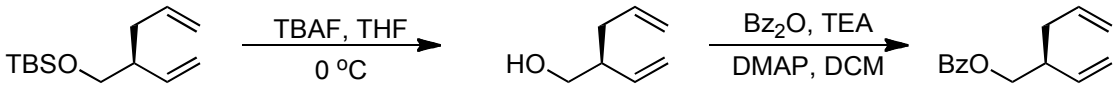
Retention Time	Area	Area %	Height	Height %
19.287	1013746288	90.51	30414501	91.21
21.297	106334887	9.49	2931252	8.79



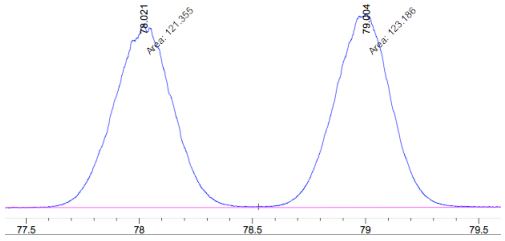
(*S*)-*tert*-butyldimethyl((2-vinylpent-4-en-1-yl)oxy)silane. ^{1}H NMR (500 MHz, $CDCl_3$): δ 0.04 (6H, s, Si(C**H$_3$**)$_2$), 0.89 (9H, s, Si(C(C**H$_3$**))$_3$), 2.04-2.09 (1H, m, CHC**H$_A$**H$_B$CH=CH$_2$), 2.24-2.33 (2H, m, CHCH$_A$**H$_B$**CH=CH$_2$ and CH$_2$C**H**CH$_A$H$_B$), 3.50-3.57 (2H, m, SiOC**H$_2$**), 4.98-5.06 (4H, m, CHCH=C**H$_2$** & CH$_2$CH=C**H$_2$**), 5.65-5.70 (1H, m, CHC**H**=CH$_2$), 5.78 (1H, app ddt, *J* = 17.0 Hz, 10.5 Hz, 7.0 Hz, CH$_2$C**H**=CH$_2$); ^{13}C NMR (125 Hz, $CDCl_3$): δ –5.4, -5.4, 18.3, 25.9, 35.3, 46.0, 65.9, 115.4, 115.8, 136.9, 139.7; IR (neat): 3077.8 (w), 2955.7 (m), 2928.7 (m), 2857.2 (m), 1730.7 (m), 1641.3, (w), 1470.9 (m), 1253.4 (s), 1097.4 (s), 992.6, (m), 1097.4 (s), 912.0 (s), 834.1 (s), 773.7 (s) cm^{-1}; HRMS (ESI+) for $C_{13}H_{27}OSi$ [M+H]: calculated: 227.1831, found: 227.1831; $[\alpha]^{20}_D$ = +6.254 (*c* = 1.227, $CHCl_3$). The crude reaction mixture was purified on silica gel (pentane, then 50:1 pentane: ether) to afford a clear, colorless oil (20.6 mg, 91% yield). R_f = 0.76 (8:1 hexanes: ethyl acetate, stain in $KMnO_4$).

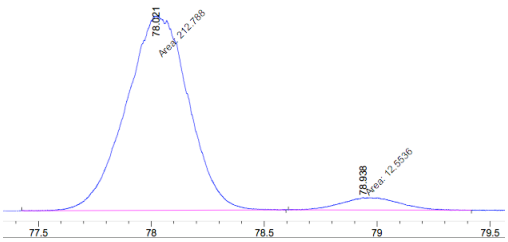
Proof of Stereochemistry:

Enantioselectivity was determined by converting the allyl-allyl coupling product to a benzoate by deprotection of TBS group and benzoate protection of the corresponding alcohol as shown below. *Via* chiral GLC the resulting benzoate was compared to racemic material prepared using 1,2-bis(diphenylphosphino)benzene as the achiral ligand in the allyl-allyl coupling reaction. The absolute stereochemistry was assigned by analogy.



Chiral GLC (CD-GTA, Supelco, 100 °C, 60 min, then 1°C/min to 130 °C, 25 psi) – analysis of benzoate.

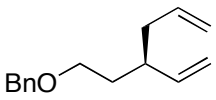




Racemic

Derived from reaction product

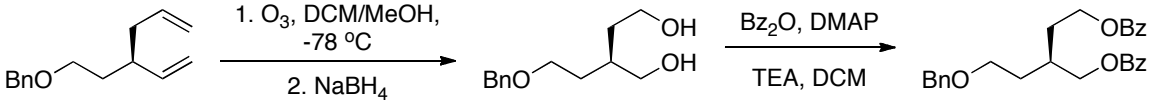
Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	78.021	MM	0.3222	212.78847	11.00840	94.42910
2	78.938	MM	0.3000	12.55359	6.97340e-1	5.57090



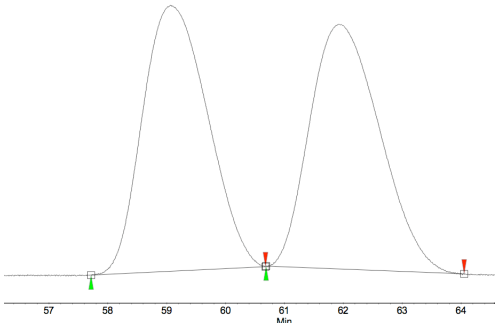
(*R*)-(((3-vinylhex-5-en-1-yl)oxy)methyl)benzene. ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.49-1.57 (1H, m, C**H$_A$**H$_B$CH=CH$_2$), 1.75-1.83 (1H, m, C**H$_B$** HCH=CH$_2$), 2.05-2.19 (2H, m, BnOCH$_2$C**H$_2$**), 2.28 (1H, app dtd, *J* = 13.6 Hz, 8.4 Hz, 5.2 Hz, C**H**CH=CH$_2$), 3.42-3.53 (2H, m, BnOC**H$_2$**), 4.49 (2H, d, *J* = 2.0 Hz, PhC**H$_2$**O), 4.94-5.04 (4H, m, CHCH=C**H$_2$** & CH$_2$CH=C**H$_2$**), 5.59 (1H, ddd, *J* = 17.2 Hz, 10.4 Hz, 8.8 Hz, CHC**H**=CH$_2$), 5.76 (1H, app dtt, *J* = 20.8 Hz, 12.0 Hz, 5.6 Hz, CH$_2$C**H**=CH$_2$), 7.25-7.34 (5H, m, Ph**H**); ^{13}C NMR (100 Hz, $CDCl_3$): δ 34.0, 39.5, 40.4, 68.3, 72.9, 114.8, 115.9, 127.5, 127.6, 128.3, 136.7, 138.6, 141.9; IR (neat): 3074.5 (w), 2926.0 (m), 2856.6 (m), 1640.7, (m), 1495.9 (w), 1453.9 (m), 1419.2 (w), 1363.5, (m), 1204.2 (w), 1101.6 (s), 1028.0 (w), 994.4 (m), 912.1 (s), 735.0 (s), 697.1 (s) cm^{-1}; HRMS (ESI+) for $C_{15}H_{21}O$ [M+H]: calculated: 217.1592, found: 217.1590; $[\alpha]^{20}_D$ = -11.355 (*c* = 1.18, $CHCl_3$). The crude reaction mixture was purified on silica gel (100:1 hexanes: ethyl acetate) to afford a clear, colorless oil (24.3 mg, 75% yield of title compound) as a mixture of coupling product and diene (90:10). R_f = 0.35 (100:1 hexanes: ethyl acetate, stain in $KMnO_4$).

Proof of Stereochemistry:

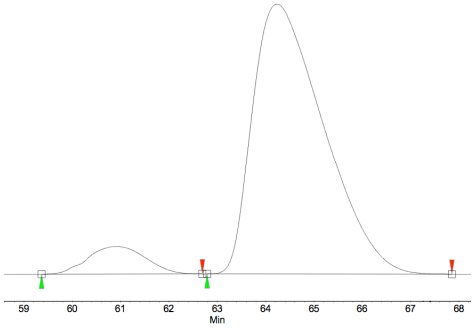
The title compound was subjected to ozonolysis and reduction. The resulting diol was protected with benzoic anhydride to afford the dibenzoate ester for HPLC analysis, as depicted below. The analogous racemic material was prepared via the same route using 1,2-bis(diphenylphosphino)benzene as achiral ligand in the allyl-allyl coupling reaction. The absolute stereochemistry was assigned by analogy.



Chiral SFC (AD-H, Chiralpak, 220nm, 1 mL/min, 1% MeOH, ramped 0.1% per minute to 5% MeOH, 150 bar, 50 °C) – analysis of the dibenzoate ester.

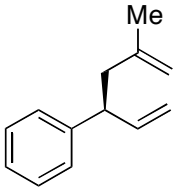


Racemic



Reaction Product

Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	59.36	60.90	62.69	0.00	7.64	493.9	682.7	7.636
2	UNKNOWN	62.79	64.23	67.85	0.00	92.36	4850.2	8258.5	92.364
Total						100.00	5344.2	8941.3	100.000

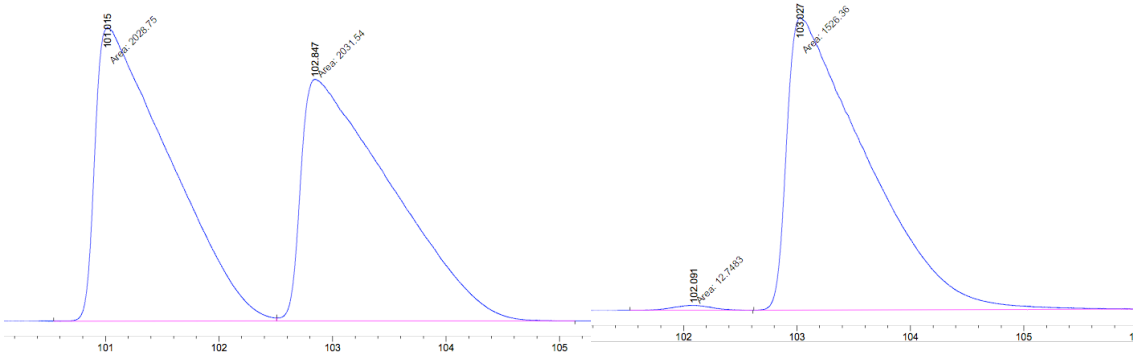


(*S*)-(5-methylhexa-hexa-1,5-dien-3-ylbenzene. ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.70 (3H, s, C**H$_3$**), 2.45 (2H, app dtd, *J* = 14.0 Hz, 14.0 Hz, 8.0 Hz, CHC**H$_2$**CH=CH$_2$), 3.51 (1H, app q, *J* = 7.5 Hz, PhC**H**CH$_2$), 4.64 (1H, m, MeC=C**H$_A$**), 4.72 (1H, m, MeC=C**H$_B$**), 4.98-5.04 (2H, m, CHCH=C**H$_2$**), 5.97 (1H, ddd, *J* = 17.0 Hz, 10.0 Hz, 7.0 Hz, CHC**H**=CH$_2$), 7.18-7.21 (3H, m, Ph**H**), 7.26-7.32 (2H, m, Ph**H**); ^{13}C NMR (125 MHz, $CDCl_3$): δ 22.4, 44.0, 47.8, 112.3, 114.1, 126.2, 127.7, 128.4, 141.8, 143.4, 144.0; IR (neat): 3075.8 (w), 3027.8 (w), 2970.2 (w), 1637.6 (w), 1601.2 (w), 1493.8 (w), 1451.7 (w), 1414.6 (m), 1373.9 (w), 990.8 (m), 911.9 (s), 887.6 (s), 752.2 (s) cm^{-1}; HRMS (ESI+) for $C_{13}H_{17}$ [M+H]: calculated: 173.1330, found: 173.1330; $[\alpha]^{20}_D$ = +27.681 (*c* = 0.987, $CHCl_3$). The crude reaction mixture was purified on silica gel (pentane) to afford a clear, colorless oil (13.6 mg, 79% yield). R_f = 0.48 (18:1 hexane: ethyl acetate, stain in $KMnO_4$).

Proof of Stereochemistry:

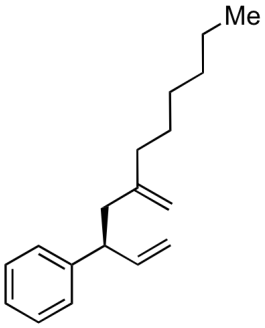
Enantioselectivity was determined by comparison of the title compound with racemic material prepared using 1,2-bis(diphenylphosphino)benzene as the achiral ligand in the allyl-allyl coupling. The absolute stereochemistry was assigned by analogy.

Chiral GLC (β-dex, Supelco, 80 °C, 25 psi) - analysis of title compound



Racemic Reaction Product

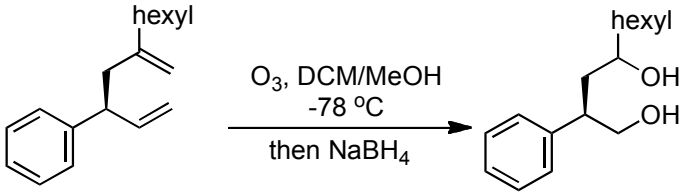
Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	102.091	MM	0.3999	12.74831	5.31307e-1	0.82829
2	103.027	MM	0.8084	1526.36267	31.46725	99.17171



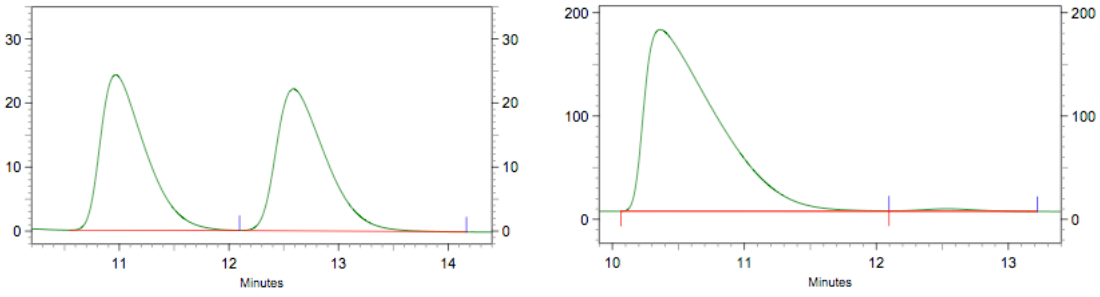
(*S*)-(5-methyleneundec-1-en-3-yl)benzene. ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.89 (3H, t, *J* = 7.2 Hz, CH_2CH_3), 1.24-1.44 (8H, m, $(CH_2)_4CH_3$), 1.97 (2H, t, *J* = 7.6 Hz, $(CH_2)_4CH_2CC=CH_2$), 2.44 (2H, d, *J* = 7.6 Hz, $PhCHCH_2C=CH_2$), 3.49 (1H, app q, *J* = 7.6 Hz, PhCH), 4.67 (1H, s, $C=CH_aH_b$), 4.73 (1H, s, $C=CH_aH_b$), 4.99 (1H, dt, *J* = 17.6, 1.2 Hz, $CH=CH_{trans}$), 5.02 (1H, dt, *J* = 10.4, 1.2 Hz, $CH=CH_{cis}$), 5.97 (1H, ddd, *J* = 17.6, 10.4, 7.2 Hz, $CH=CH_2$), 7.17-7.21 (3H, m, PhH), 7.28-7.31 (2H, m, PhH); ^{13}C NMR (100 Hz, $CDCl_3$): δ 13.0, 14.4, 27.9, 29.4, 32.1, 36.3, 42.4, 48.1, 111.4, 114.5, 126.5, 128.0, 128.7, 142.2, 144.5, 147.7; IR (neat): 3670.0 (w), 3028.1 (w), 2956.3 (m), 2926.8 (s), 2856.4 (m), 1642.9 (w), 1493.1 (w), 1453.0 (m), 1378.1 (w), 1074.4 (w), 990.6 (w), 912.3 (m), 891.0 (m), 753.2 (m), 673.0 (s) cm^{-1}; HRMS (ESI+) for $C_{18}H_{27}$ [M+H]: calculated: 243.2113, found: 243.2105; $[\alpha]^{20}_D$ = +24.191 (*c* = 0.75, $CHCl_3$). The crude reaction mixture was purified on silica gel (pentane) to afford a clear, colorless oil (18.9 mg, 78% yield). R_f = 0.68 (8:1 hexanes: ethyl acetate, stain in $KMnO_4$).

Proof of Stereochemistry:

Enantioselectivity was determined by converting the allyl-allyl coupling product to a diol by ozonolysis/reduction. *Via* chiral HPLC the resulting diol was compared to racemic material prepared using 1,2-bis(diphenylphosphino)benzene as the achiral ligand in the allyl-allyl coupling reaction. The absolute stereochemistry was assigned by analogy.



Chiral HPLC (AS-H, Chiralpak, 220nm, 1 mL/min, 2% isopropanol) – analysis of the diol.



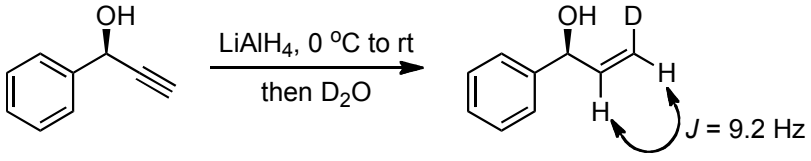
Racemic

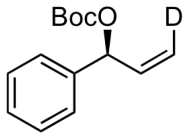
Derived from reaction product

Retention Time	Area	Area %	Height	Height %
10.363	110295188	98.88	2952849	98.61
12.533	1246973	1.12	41595	1.39

Deuterium-Labeling Study

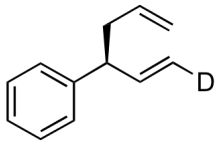
Preparation of (S)-(-)-tert-butyl cis-3-[^{2}H1]-1-phenylprop-2-enyl carbonate. From the deuterated allylic alcohol,[9] synthesized from commercially available (*S*)-1-phenylprop-2-yn-1-ol, >95% *ee*, procedure B was followed.



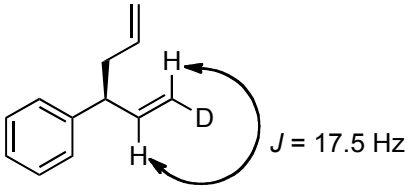


(*S*)-(-)-*tert*-butyl cis-3-[^{2}H1]-1-phenylprop-2-enyl carbonate. ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.47 (9H, s, OC(C**H$_3$**)$_3$), 5.24 (1H, app dt, *J* = 9.2, 4.0 Hz, CH=C**H**D), 6.01-6.04 (2H, m, C**H**=CHD & PhC**H**OBoc), 7.26-7.38 (5H, m, Ph**H**); ^{13}C NMR (100 Hz, $CDCl_3$): δ 27.8, 71.2, 82.3, 116.9 [t, ^1J(C, ^{2}H) = 23.8 Hz], 127.0, 128.2, 128.5, 136.1, 138.7, 152.7; IR (neat): 2980.8 (w), 2933.3 (w), 1739.3 (s), 1495.0 (w), 1394.3 (m), 1312.3 (s), 1273.8 (s), 1252.5 (s), 1086.0 (m), 966.7 (w), 894.8 (m), 698.8 (m) cm^{-1}; HRMS (ESI+) for C_9H_8D [M-OBoc]: calculated: 118.0767, found: 118.0768; $[\alpha]^{20}_D$ = -29.776 (*c* = 0.97, $CHCl_3$). The crude reaction mixture was purified on silica gel (50:1 hexanes: ethyl acetate) to afford a clear, light yellow oil (93%). R_f = 0.56 (8:1 hexanes: ethyl acetate, stain in $KMnO_4$).

Allyl-allyl coupling of deuterium-labeled starting material utilizing allylboronic acid pinacol ester: The representative procedure for allyl-allyl coupling was applied.



(*S*)-*trans*-1-[^{2}H1]-hexa-1,5-dien-ylbenzene. ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.49 (2H, m, C**H$_2$**CH=CH$_2$), 3.36 (1H, app q, *J* = 7.0 Hz, PhC**H**), 4.96-5.04 (3H, m, CH=C**H$_2$** & CH=C**H**D), 5.73 (1H, ddt, *J* = 17.5, 10.0, 7.0 Hz, CH$_2$C**H**=CH$_2$), 5.98 (1H, dd, *J* = 17.5, 7.5 Hz, CHC**H**=CHD), 7.19-7.32 (3H, m, Ph-**H**), 7.29-7.38 (2H, m, Ph-**H**); ^{13}C NMR (100 Hz, $CDCl_3$): δ 39.7, 49.6, 114.1 [t, ^1J(C, ^{2}H) = 23.8 Hz], 116.1, 126.3, 127.7, 128.4, 136.6, 141.4, 143.7; IR (neat): 3077.2 (w), 3028.3 (w), 3003.2 (w), 2924.5 (w, br), 2857.1 (w, br), 1640.2 (w), 1600.4 (w), 1451.8 (w), 1415.7 (w), 979.3 (m), 911.7 (s), 747.1 (m) cm^{-1}; HRMS (ESI+) for $C_{12}H_{14}D$ [M+H]: calculated: 160.1237, found: 160.1233; $[\alpha]^{20}_D$ = +18.858 (*c* = 0.88, $CHCl_3$). The crude reaction mixture was purified on silica gel (pentane) to afford a clear, colorless oil (11.7 mg, 77% yield). R_f = 0.79 (8:1 hexanes: ethyl acetate, stain in $KMnO_4$).



[9] Kang, M. J.; Jang, J. S.; Lee, S. G. *Tetrahedron Lett.* **1995**, *36*, 8829.

OBoc
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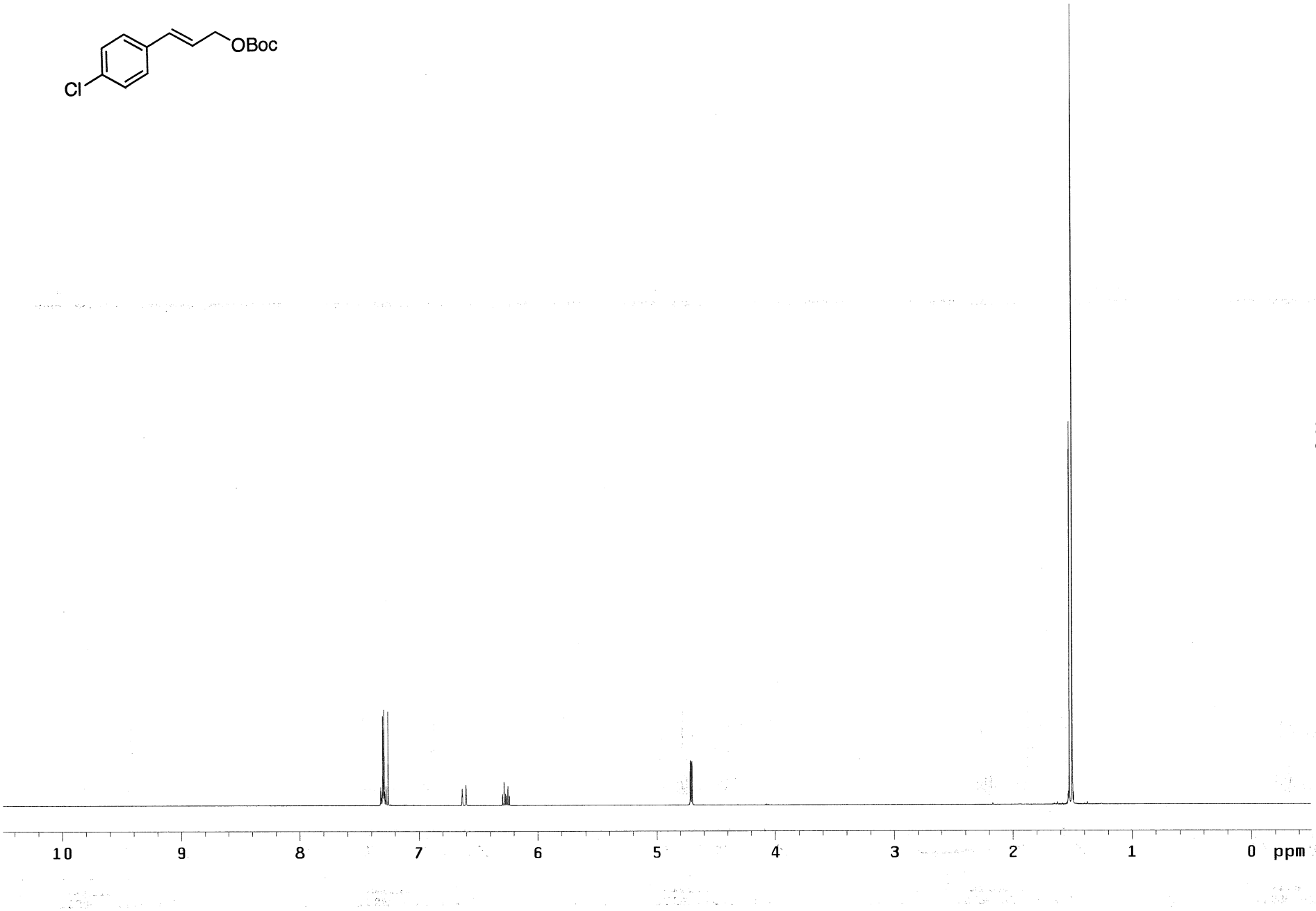
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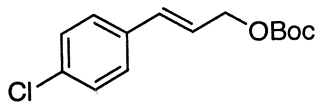
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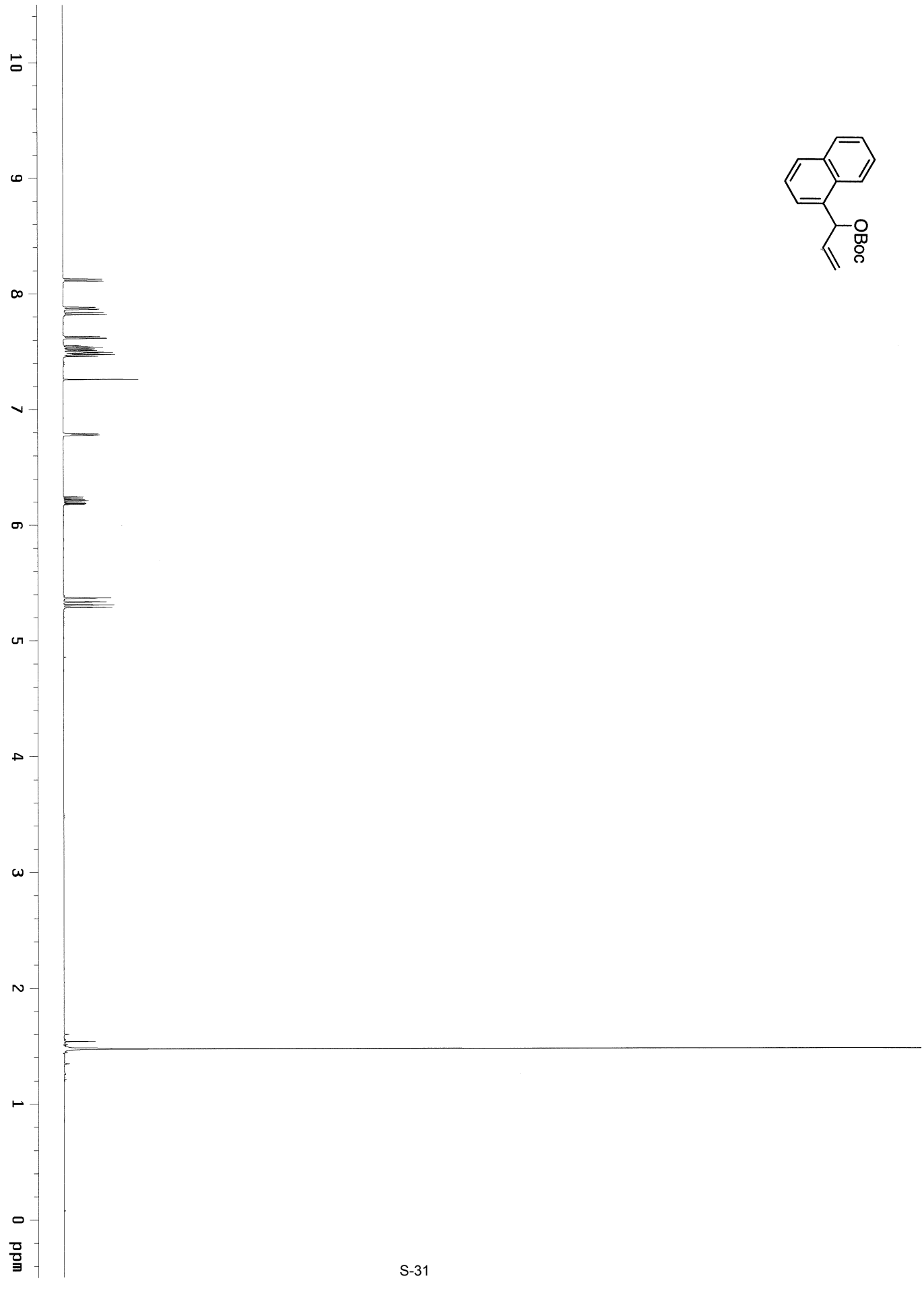
OBoc
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ppm

OBoc

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100
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60
40
20
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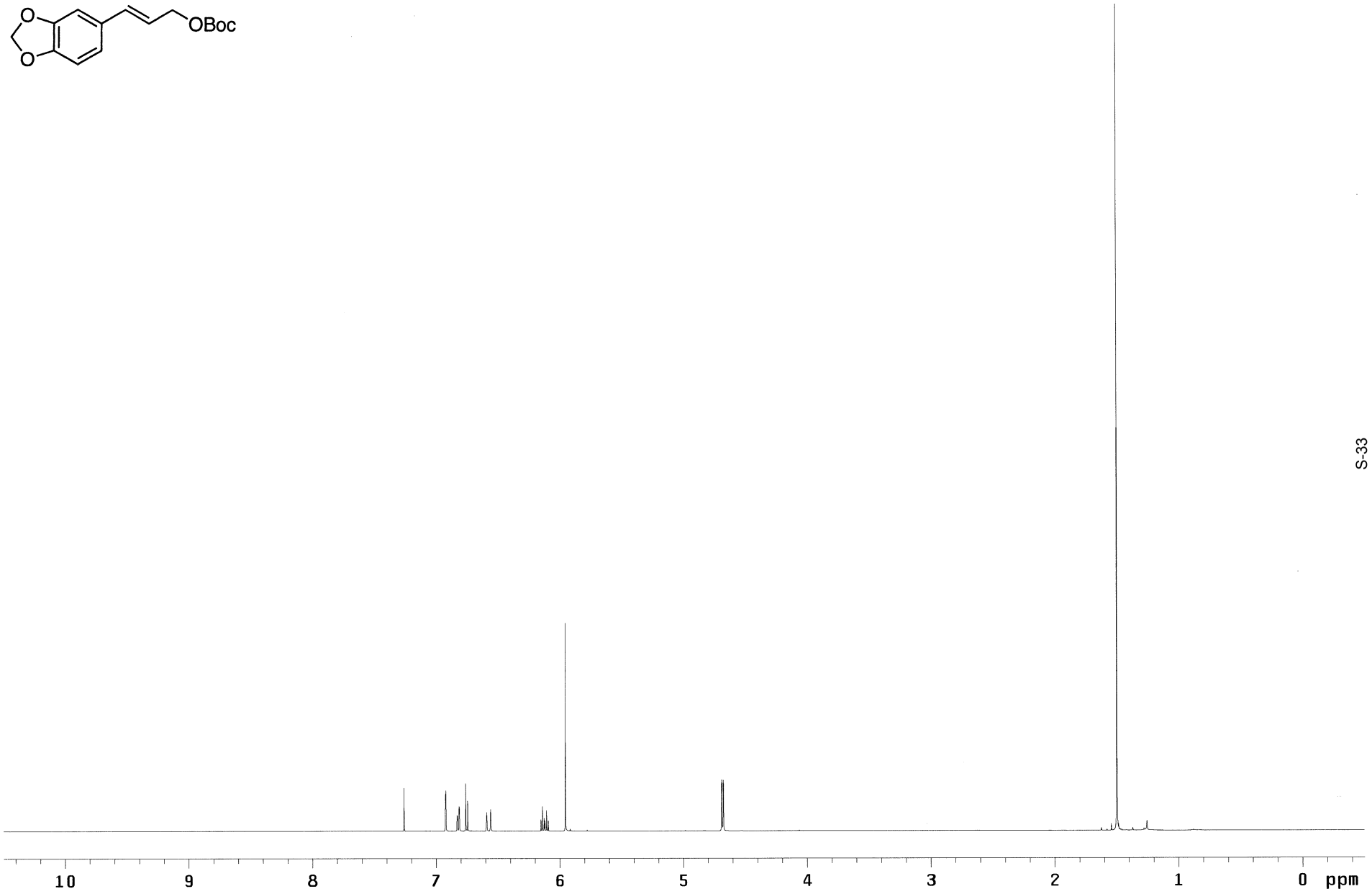
ppm

O
O
OBoc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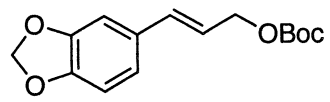
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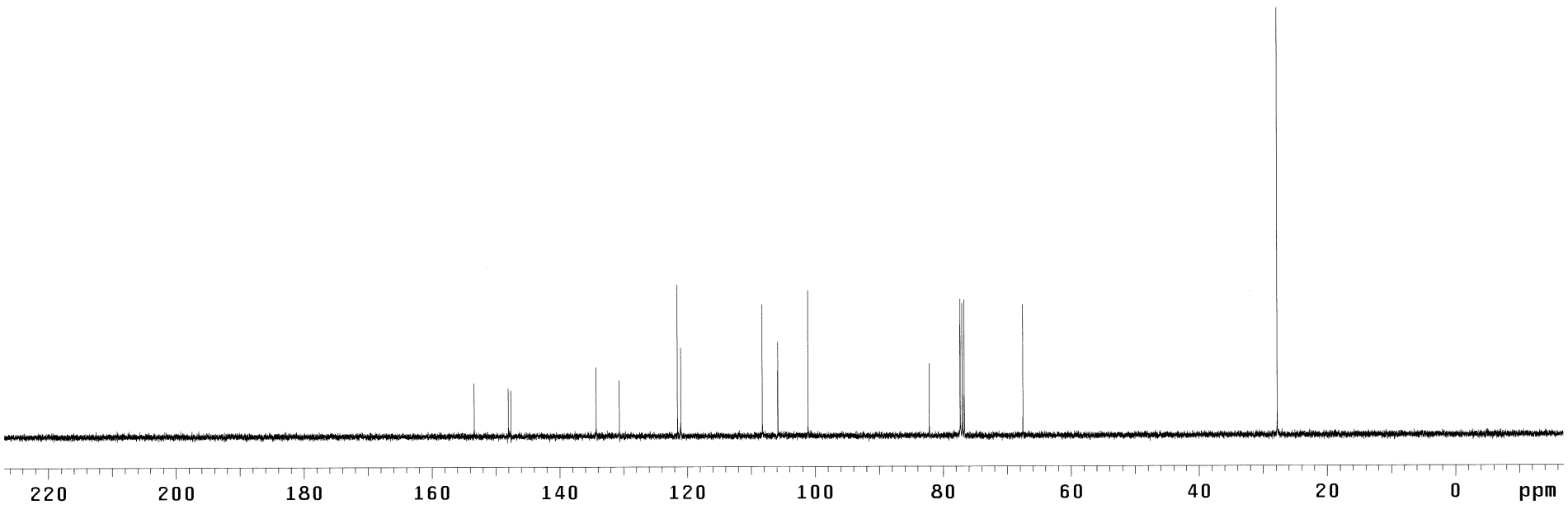
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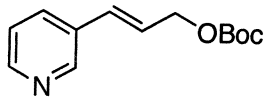
ppm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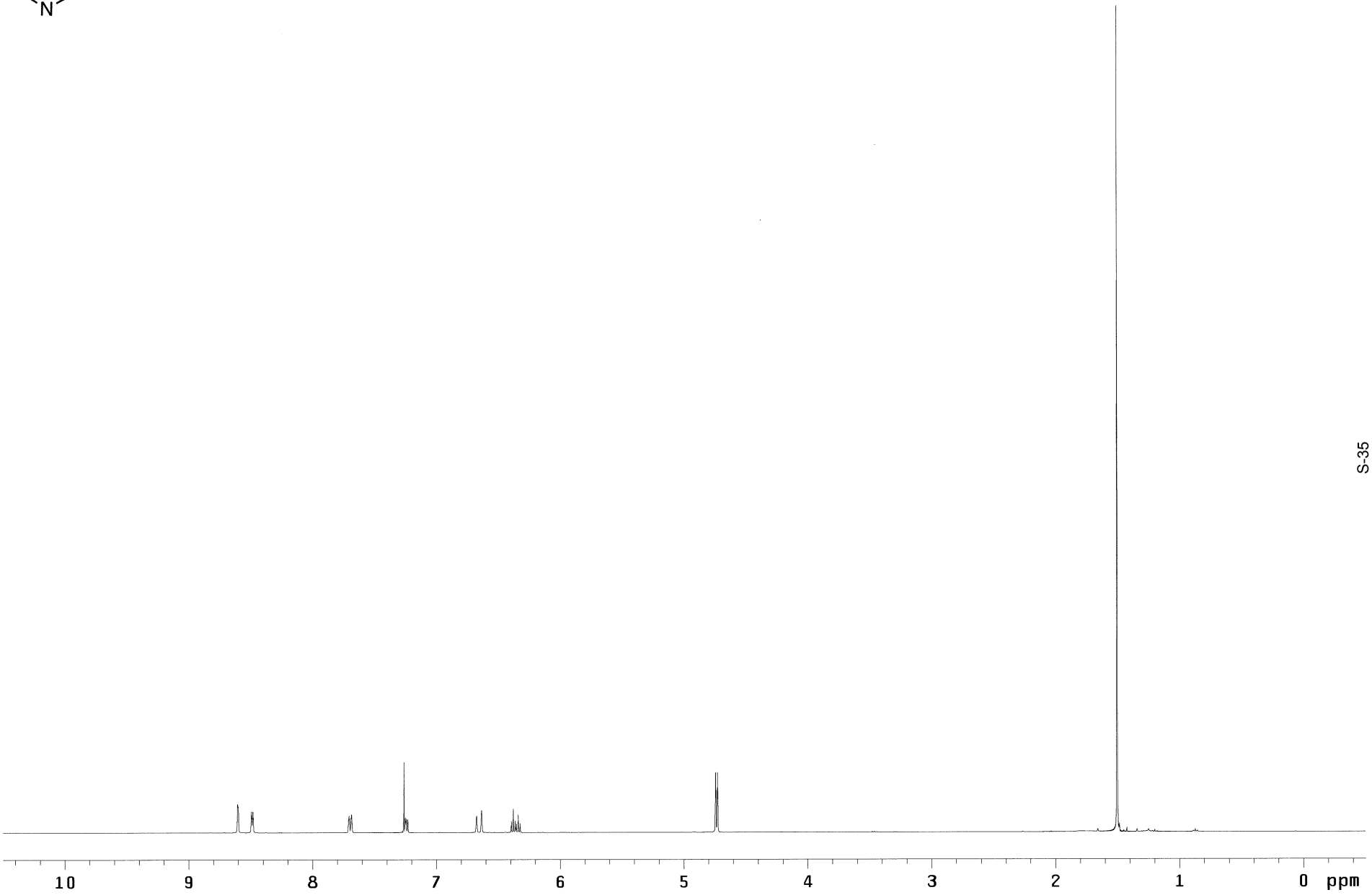
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4
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2
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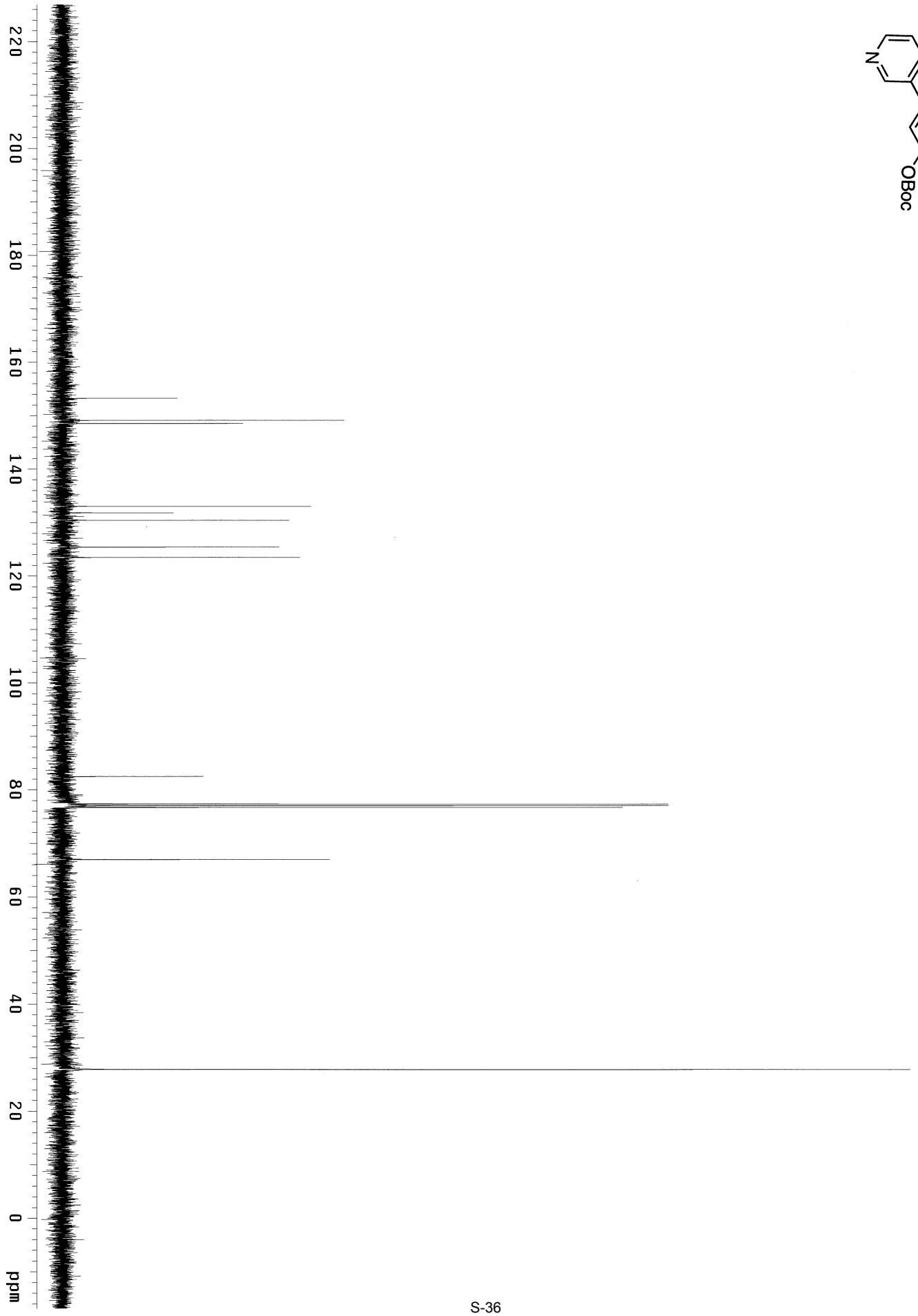
0
ppm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200
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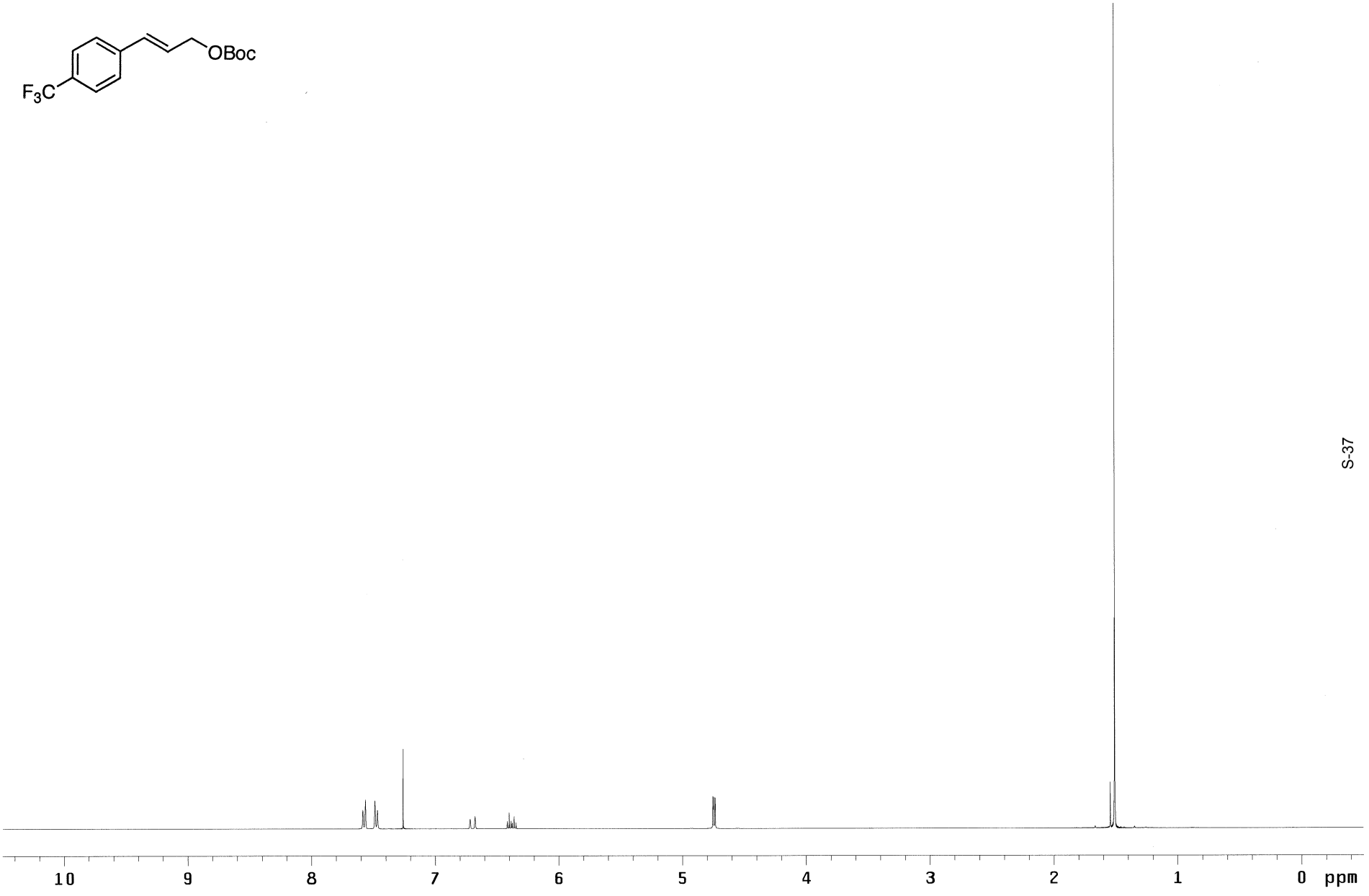
120
100
80
60
40
20
0
ppm

OBoc
F3C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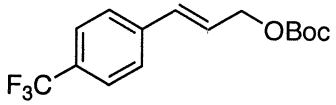
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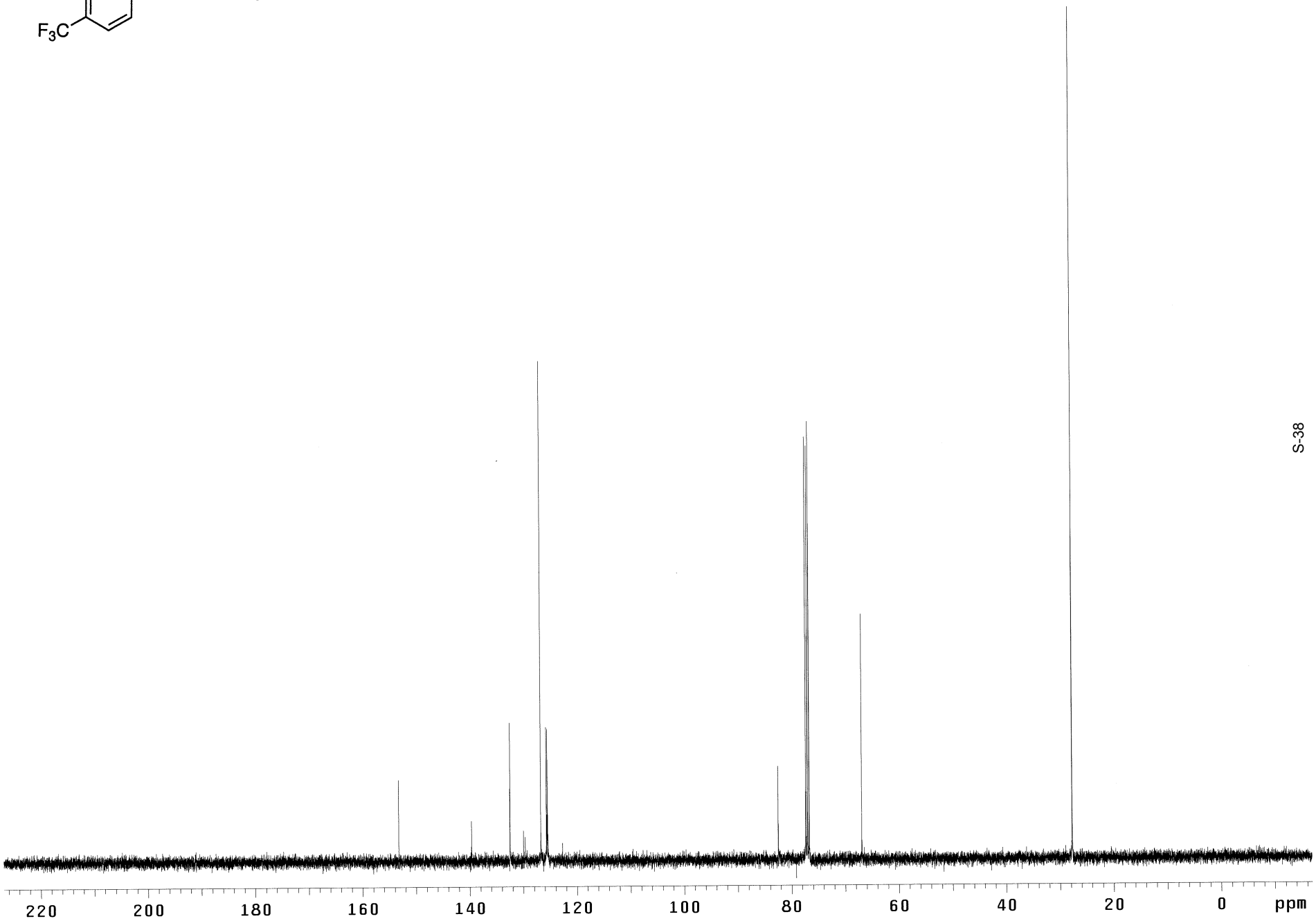
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200
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140
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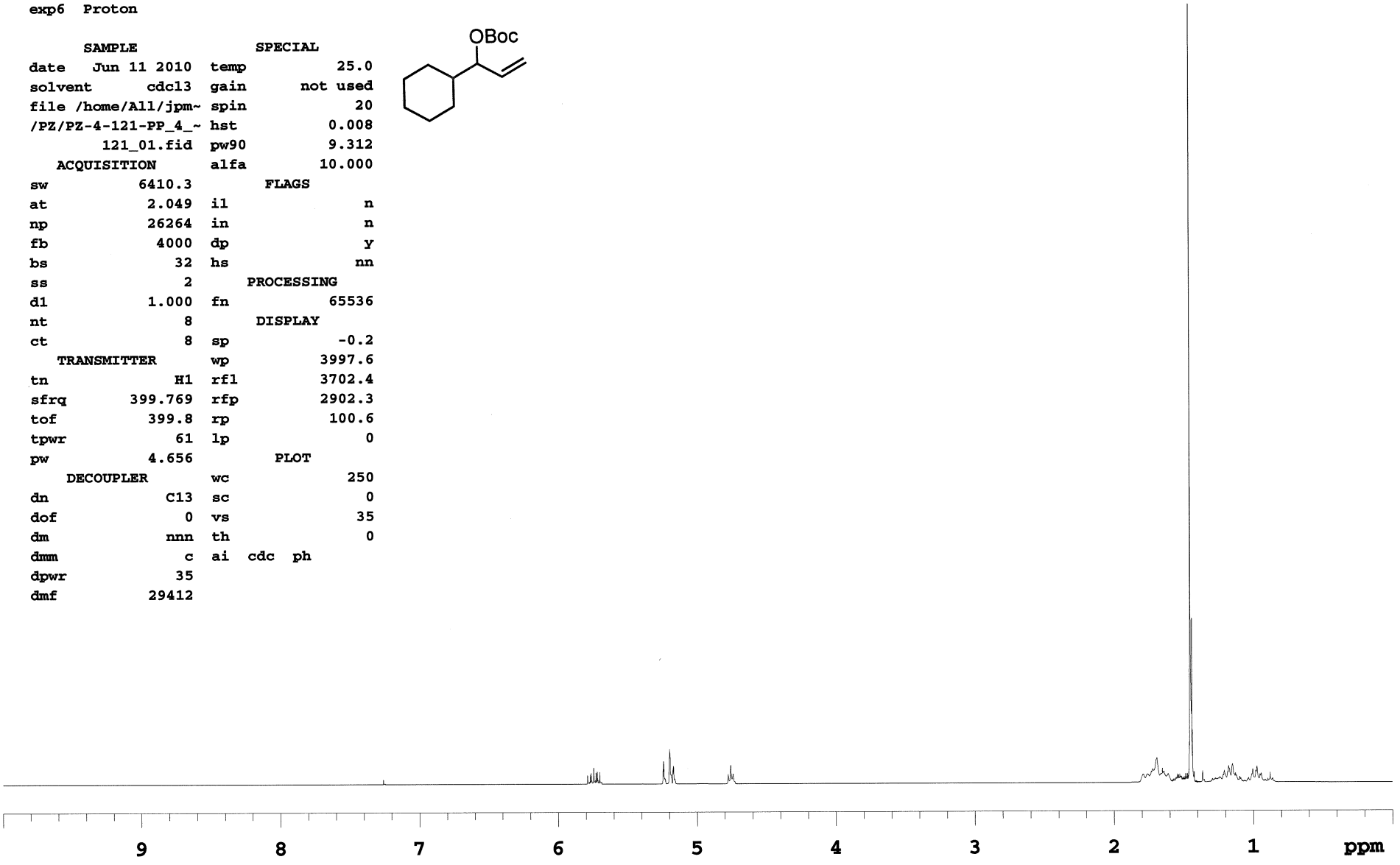
80
60
40
20
0
ppm

PZ-4-121-PP

exp6 Proton

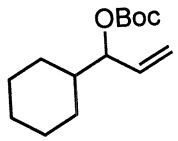
SAMPLE		SPECIAL	
date	Jun 11 2010	temp	25.0
solvent	cdcl3	gain	not used
file	/home/All/jpm~	spin	20
/PZ/PZ-4-121-PP_4_~		hst	0.008
	121_01.fid	pw90	9.312
ACQUISITION		alfa	10.000
sw	6410.3	FLAGS	
at	2.049	il	n
np	26264	in	n
fb	4000	dp	y
bs	32	hs	nn
ss	2	PROCESSING	
d1	1.000	fn	65536
nt	8	DISPLAY	
ct	8	sp	-0.2
TRANSMITTER		wp	3997.6
tn	H1	rfl	3702.4
sfrq	399.769	rfp	2902.3
tof	399.8	rp	100.6
tpwr	61	lp	0
pw	4.656	PLOT	
DECOUPLER		wc	250
dn	C13	sc	0
dof	0	vs	35
dm	nnn	th	0
dmm	c	ai cdc ph	
dpwr	35		
dmf	29412		

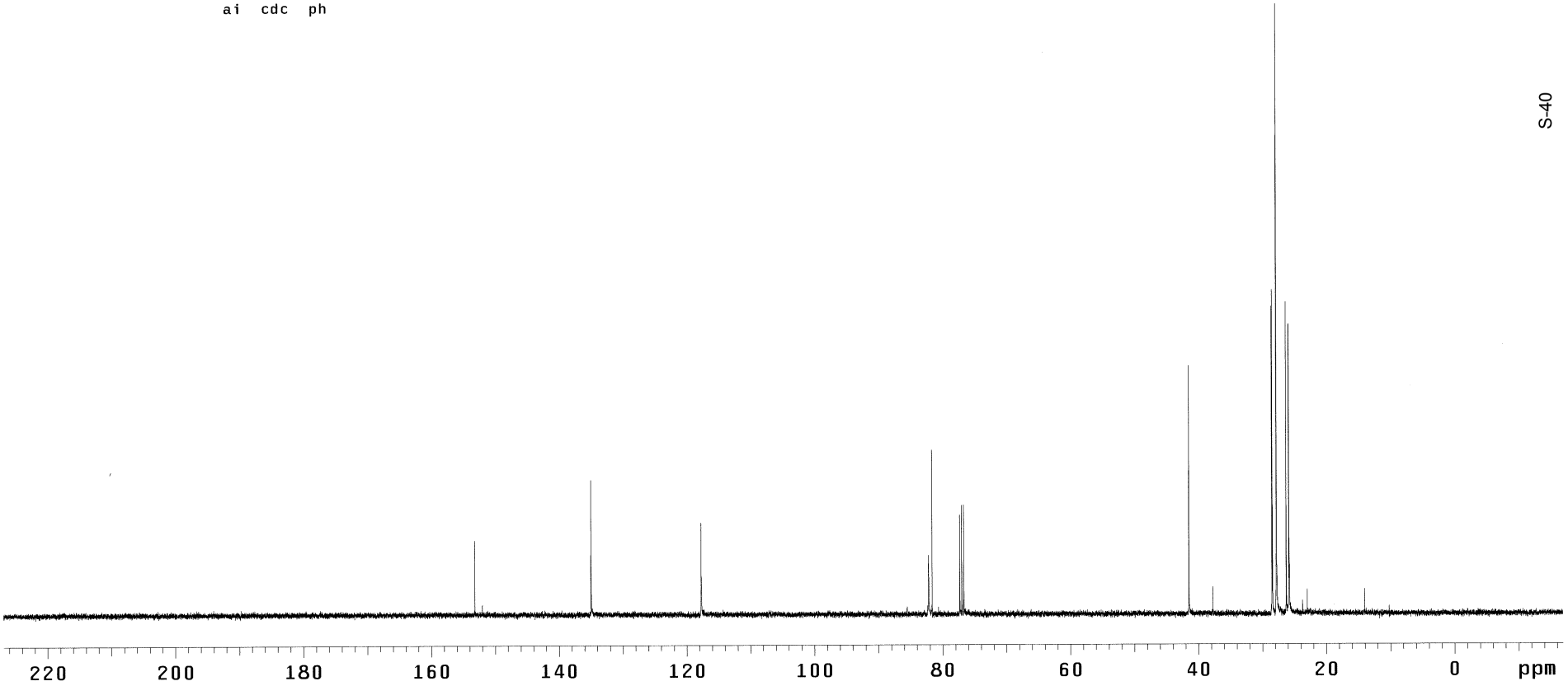


PZ-4-121-PP-C13

exp10 Carbon

SAMPLE		SPECIAL	
date	Jun 8 2010	temp	25.0
solvent	cdcl3	gain	30
file	exp	spin	20
ACQUISITION		hst	0.008
sw	24509.8	pw90	9.300
at	1.300	alfa	10.000
np	63750	FLAGS	
fb	17000	il	n
bs	32	in	n
d1	1.000	dp	y
nt	5000	hs	nn
ct	128	PROCESSING	
TRANSMITTER		lb	0.50
tn	C13	fn	not used
sfrq	100.532	DISPLAY	
tof	1028.1	sp	-1699.4
tpwr	57	wp	24509.1
pw	4.650	rfl	9440.3
DECOUPLER		rfp	7740.1
dn	H1	rp	-149.1
dof	0	lp	0
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	40	sc	0
dmf	10086	vs	9454
		th	15
		ai	cdc ph

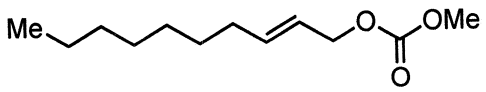




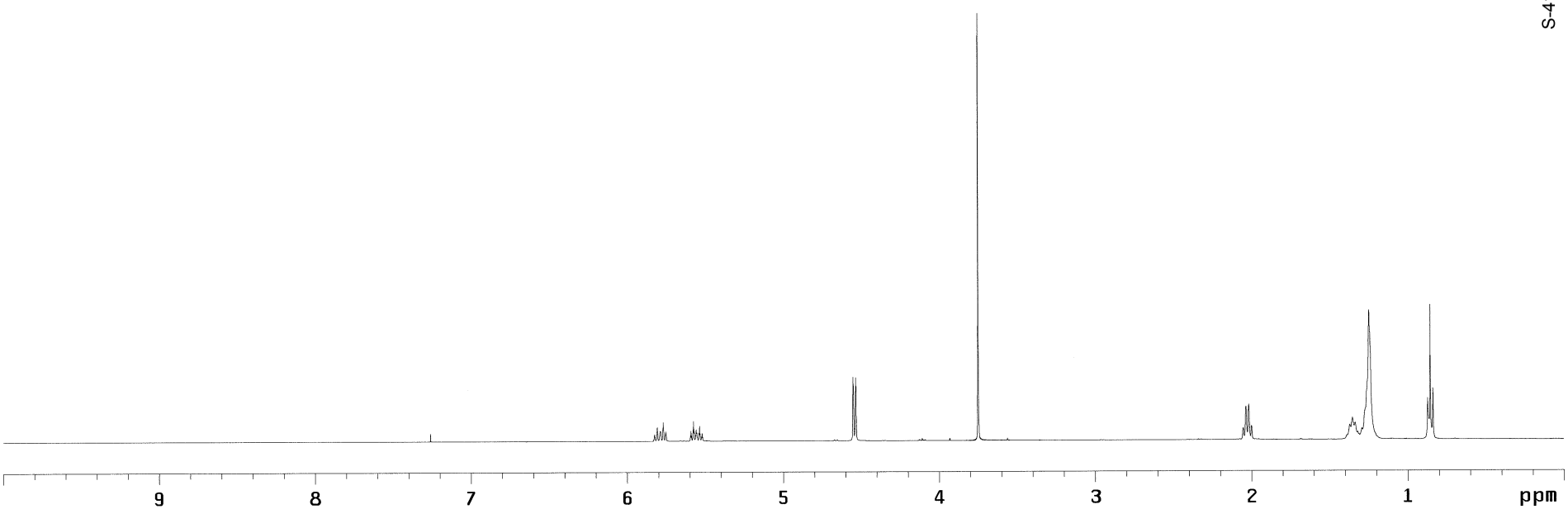
PZ-4-55-P

Sample: PZ-4-55-P
File: exp

Pulse Sequence: s2pul
 Solvent: cdcl3
 Temp. 25.0 C / 298.1 K
Operator: jpm
VNMRS-400 "nmr14"

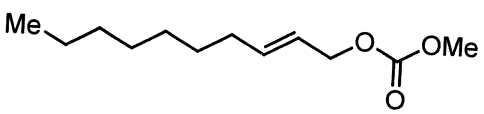


 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.049 sec
 Width 6410.3 Hz
 16 repetitions
OBSERVE H1, 399.7662704 MHz
DATA PROCESSING
 Resol. enhancement -0.0 Hz
FT size 65536
Total time 0 min, 55 sec



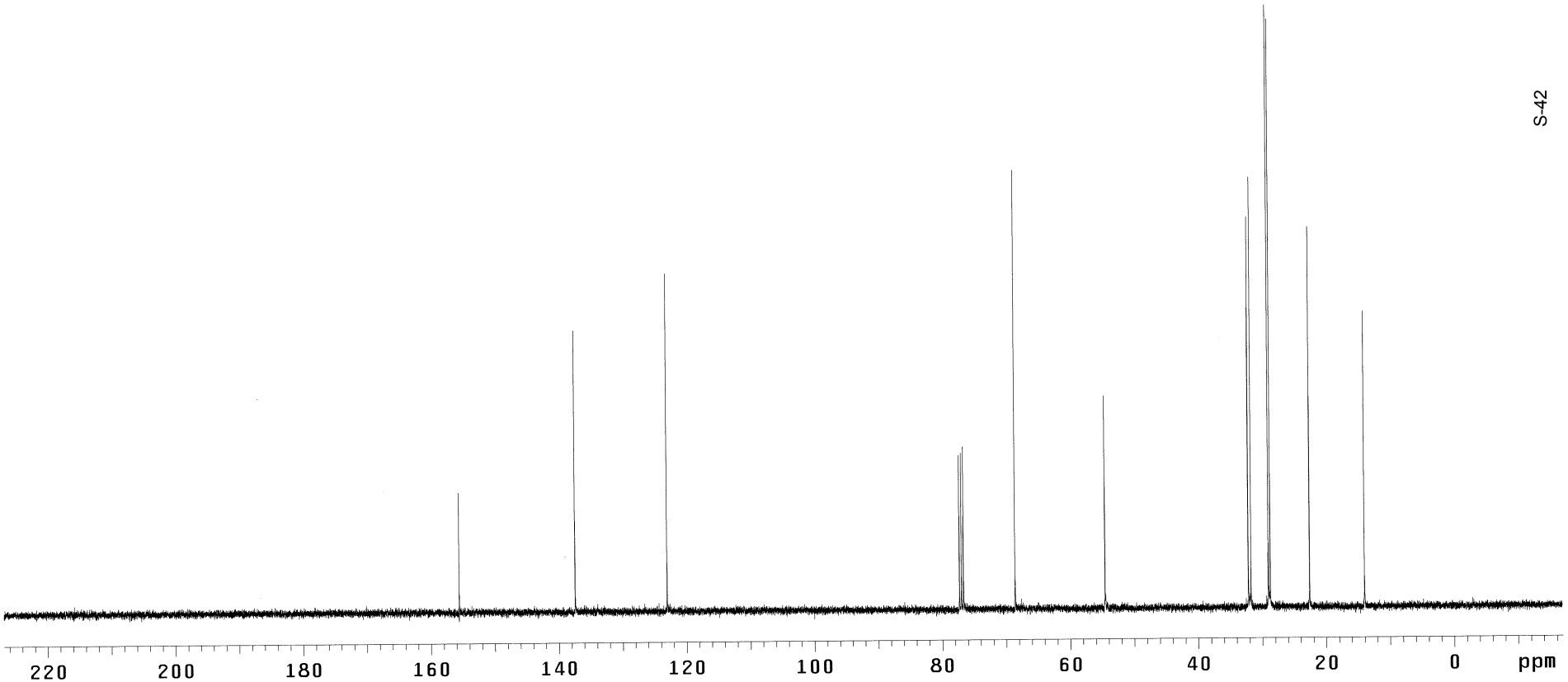
PZ-4-55-P-C13

Sample: PZ-4-55-P-C13
File: exp



Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: jpm
VNMRS-400 "nmr14"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
64 repetitions
OBSERVE C13, 100.5213117 MHz
DECOUPLE H1, 399.7682756 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 38 min, 21 sec

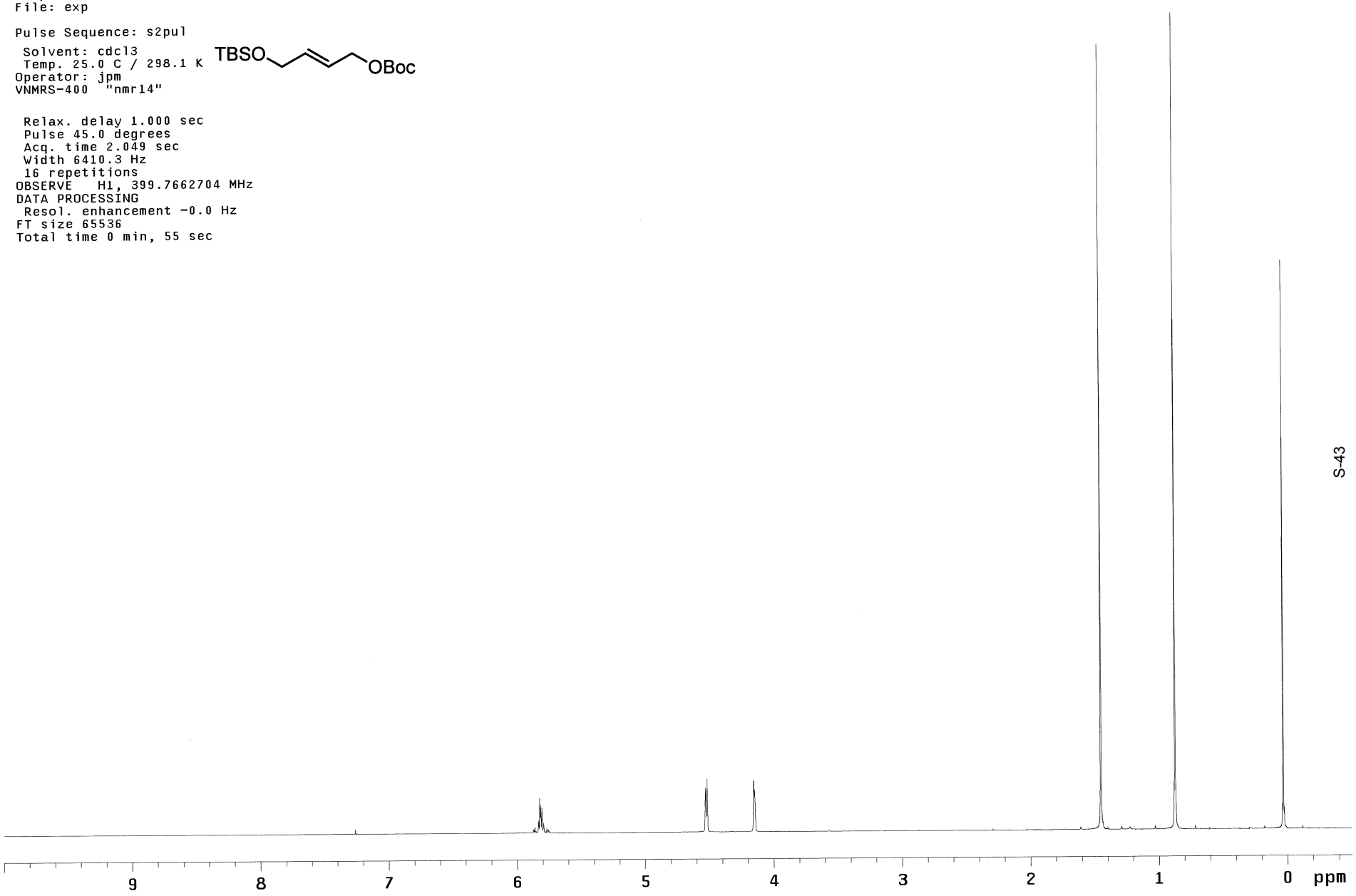


PZ-4-114-P

Sample: PZ-4-114-P
File: exp

Pulse Sequence: s2pul
 Solvent: cdcl3
 Temp. 25.0 C / 298.1 K
Operator: jpm
VNMRS-400 "nmr14"

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.049 sec
 Width 6410.3 Hz
 16 repetitions
OBSERVE   H1, 399.7662704 MHz
DATA PROCESSING
 Resol. enhancement -0.0 Hz
FT size 65536
Total time 0 min, 55 sec

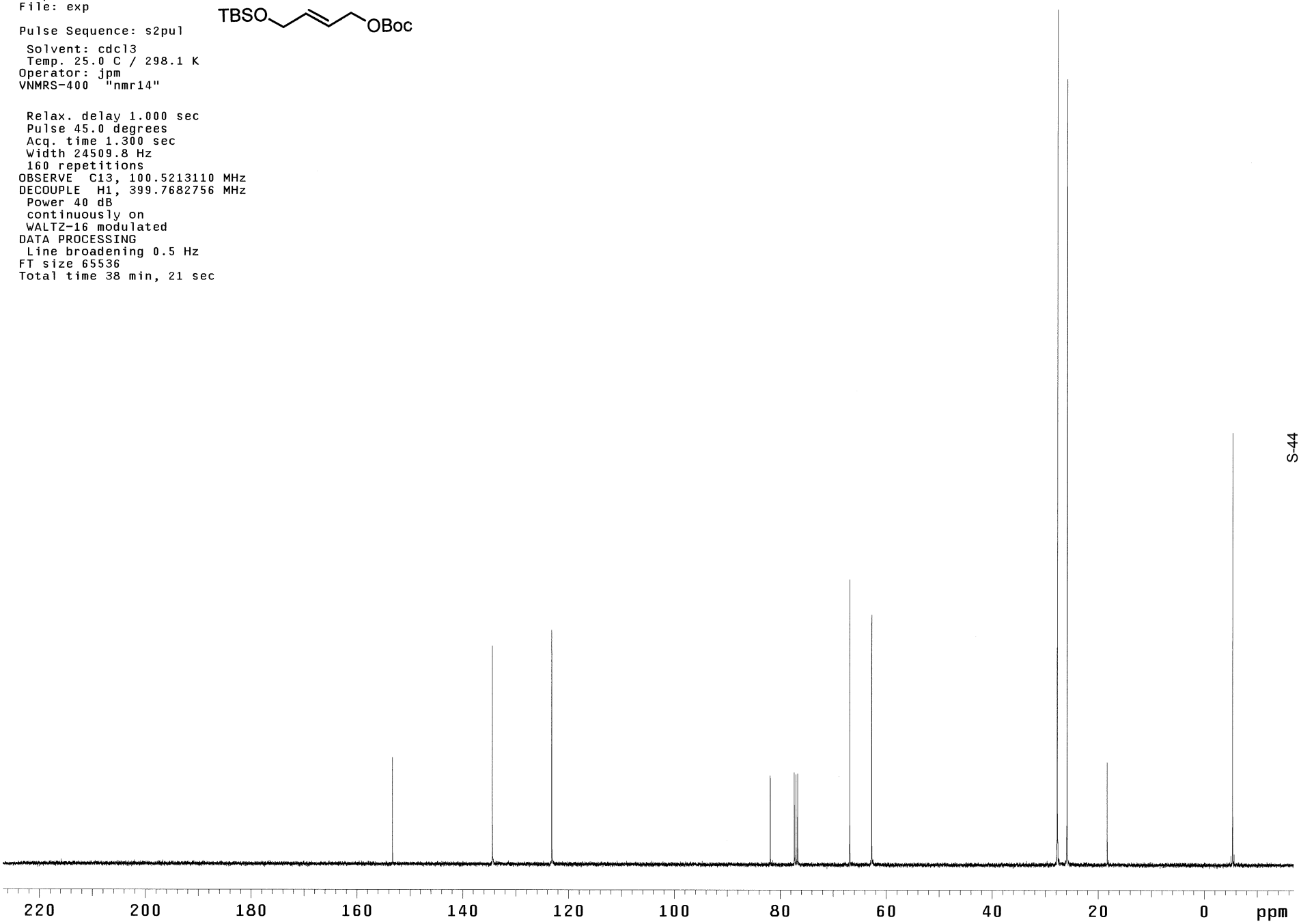


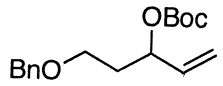
PZ-4-114-P-C13

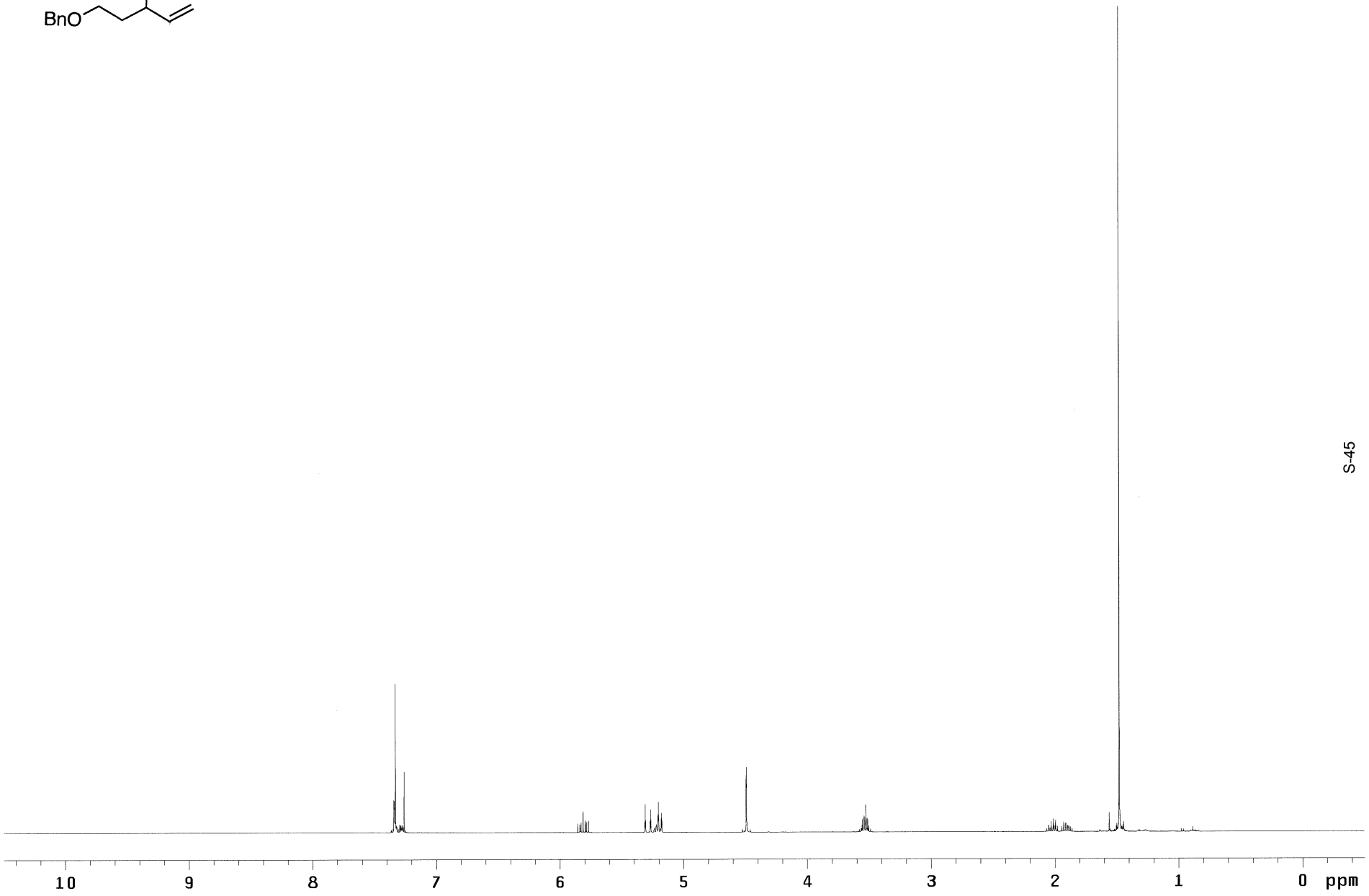
Sample: PZ-4-114-P
File: exp

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: jpm
VNMRS-400 "nmr14"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
160 repetitions
OBSERVE C13, 100.5213110 MHz
DECOUPLE H1, 399.7682756 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 38 min, 21 sec




OBoc
BnO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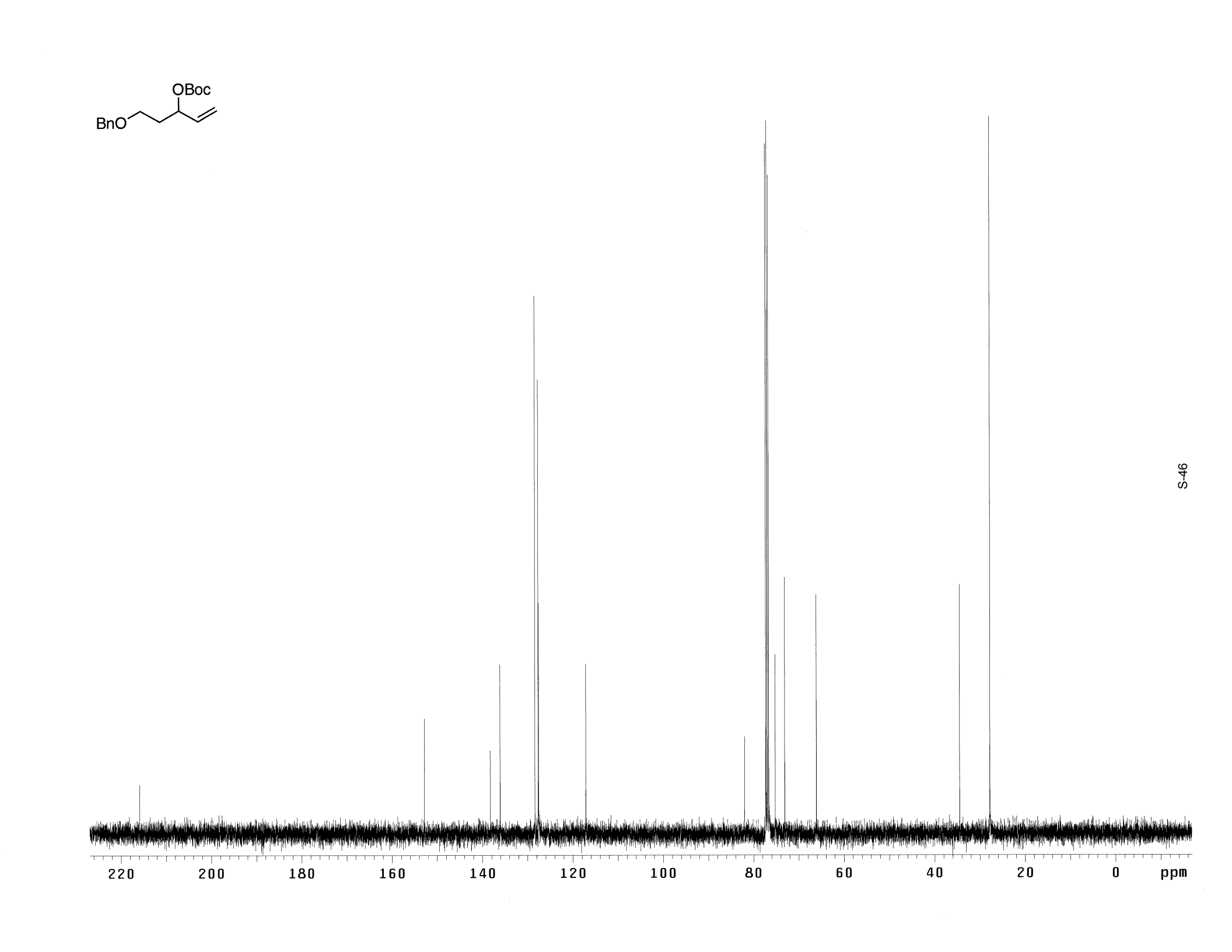
0
ppm


OBoc
BnO
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60
40
20
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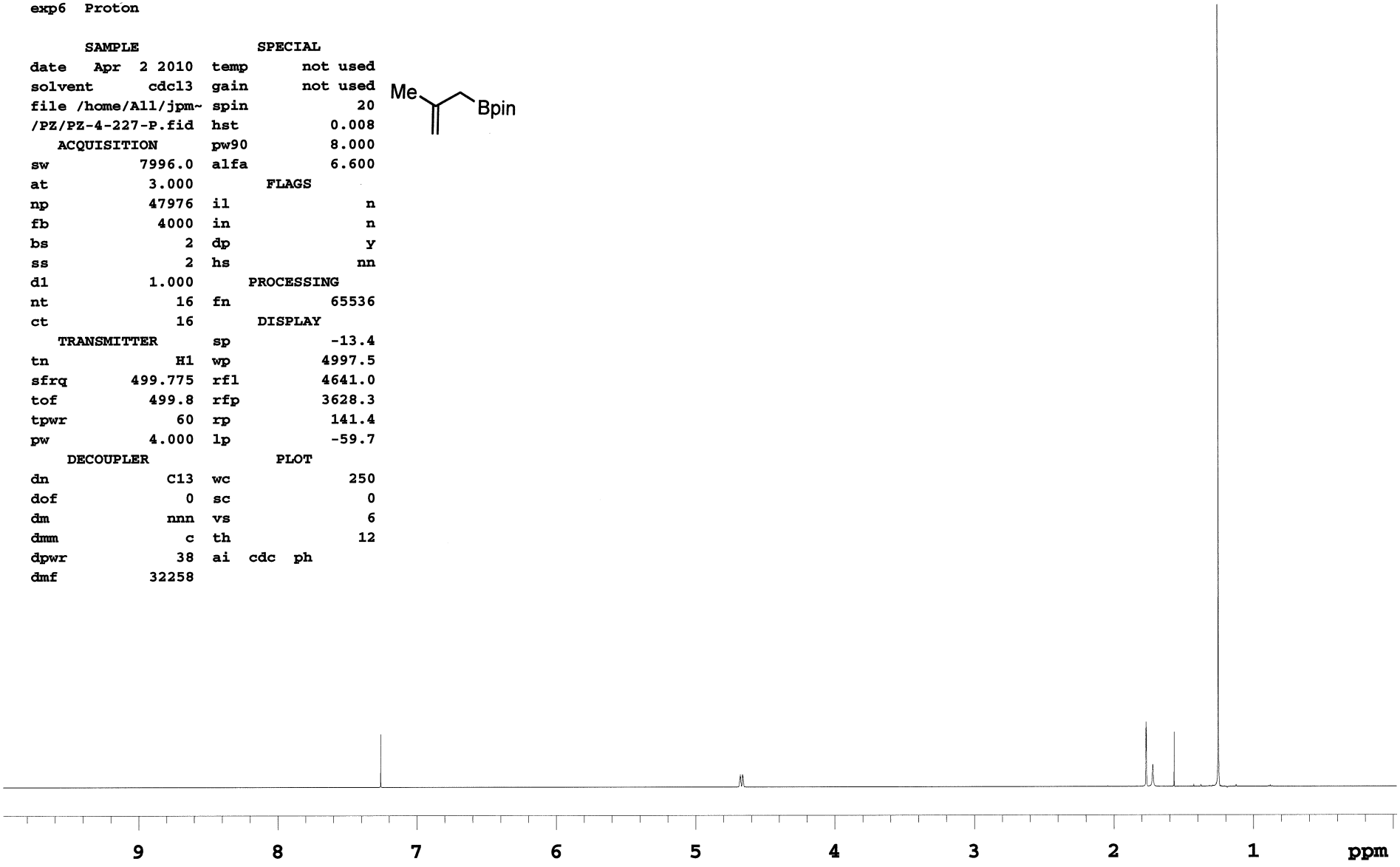
ppm
S-46

PZ-4-227-P

exp6 Proton

SAMPLE		SPECIAL	
date	Apr 2 2010	temp	not used
solvent	cdcl3	gain	not used
file	/home/All/jpm~	spin	20
	/PZ/PZ-4-227-P.fid	hst	0.008
ACQUISITION		pw90	8.000
sw	7996.0	alfa	6.600
at	3.000	FLAGS	
np	47976	il	n
fb	4000	in	n
bs	2	dp	y
ss	2	hs	nn
d1	1.000	PROCESSING	
nt	16	fn	65536
ct	16	DISPLAY	
TRANSMITTER		sp	-13.4
tn	H1	wp	4997.5
sfrq	499.775	rf1	4641.0
tof	499.8	rfp	3628.3
tpwr	60	rp	141.4
pw	4.000	lp	-59.7
DECOUPLER		PLOT	
dn	C13	wc	250
dof	0	sc	0
dm	nnn	vs	6
dmm	c	th	12
dpwr	38	ai cdc ph	
dmf	32258		



PZ-4-227-P-C13

exp1 Carbon

SAMPLE		SPECIAL	
date	Apr 18 2010	temp	25.0
solvent	cdcl3	gain	30
file	/home/All/jpm~	spin	20
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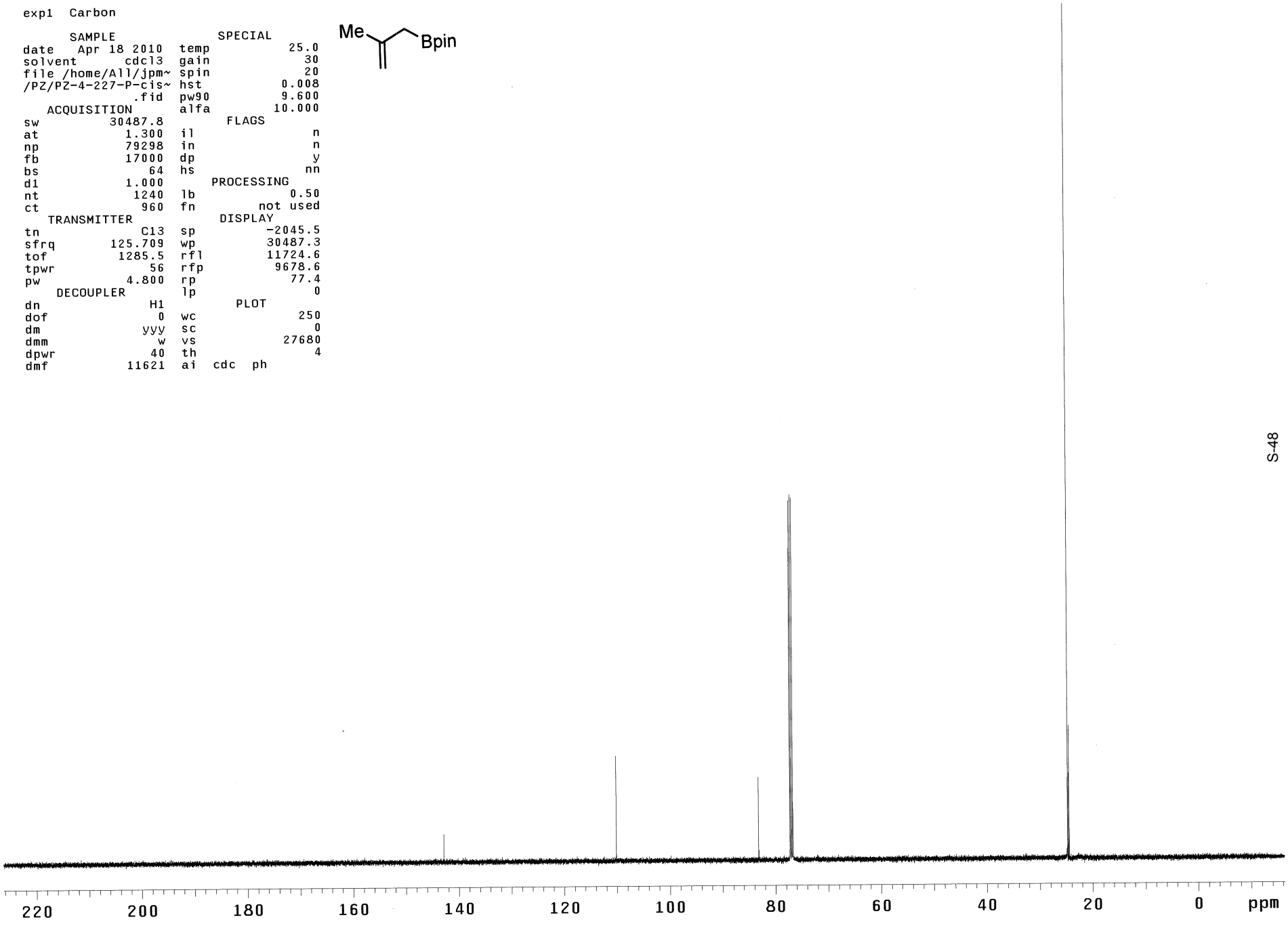
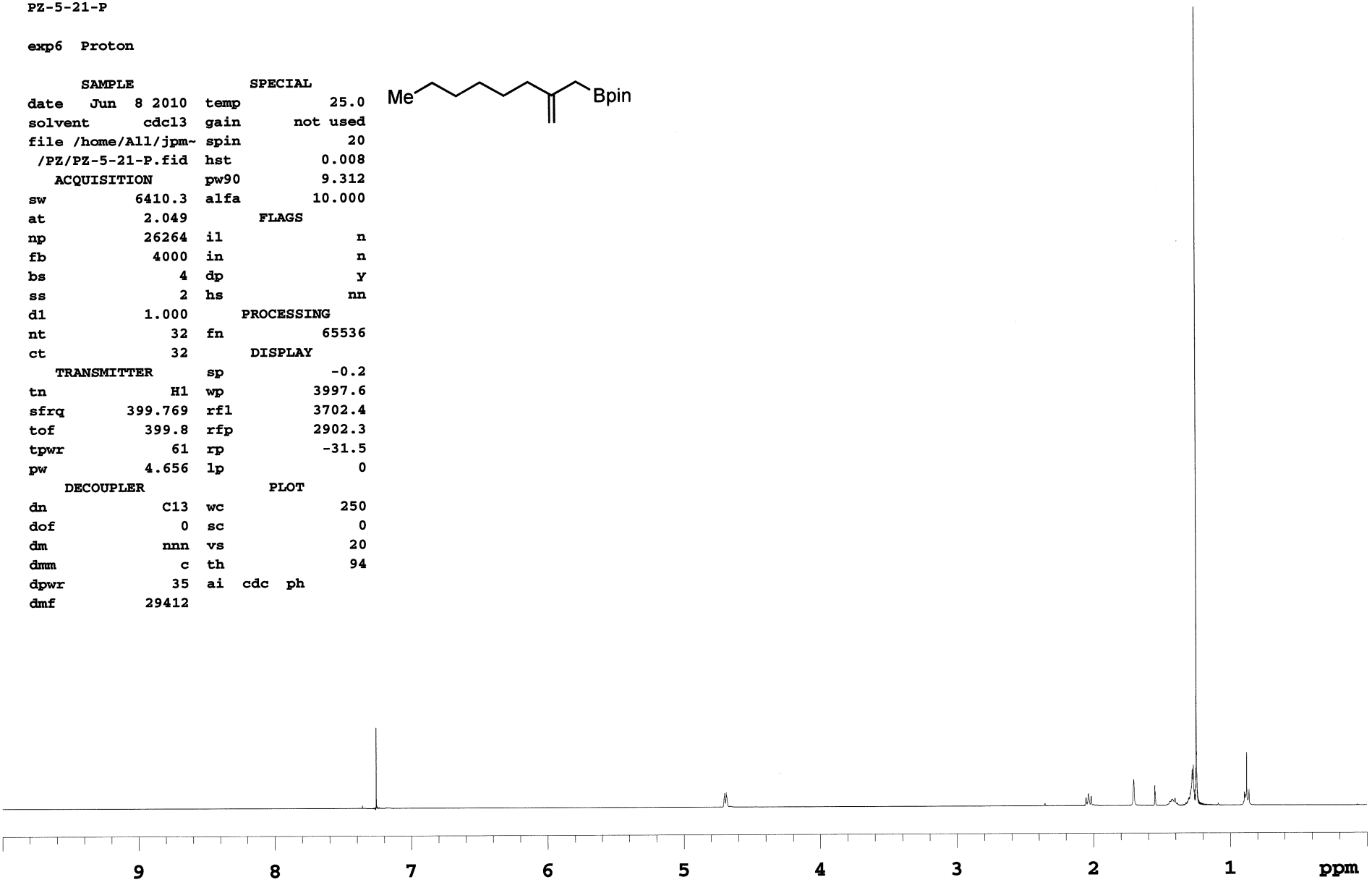
		pw90	9.600
ACQUISITION		alfa	10.000
sw	30487.8	FLAGS	
at	1.300	il	n
np	79298	in	n
fb	17000	dp	y
bs	64	hs	nn
d1	1.000	PROCESSING	
nt	1240	lb	0.50
ct	960	fn	not used
TRANSMITTER		DISPLAY	
tn	C13	sp	-2045.5
sfrq	125.709	wp	30487.3
tof	1285.5	rfl	11724.6
tpwr	56	rfp	9678.6
pw	4.800	rp	77.4
DECOUPLER		lp	0
dn	H1	PLOT	
dof	0	wc	250
dm	yyy	sc	0
dmm	w	vs	27680
dpwr	40	th	4
dmf	11621	ai cdc ph	



PZ-5-21-P

exp6 Proton

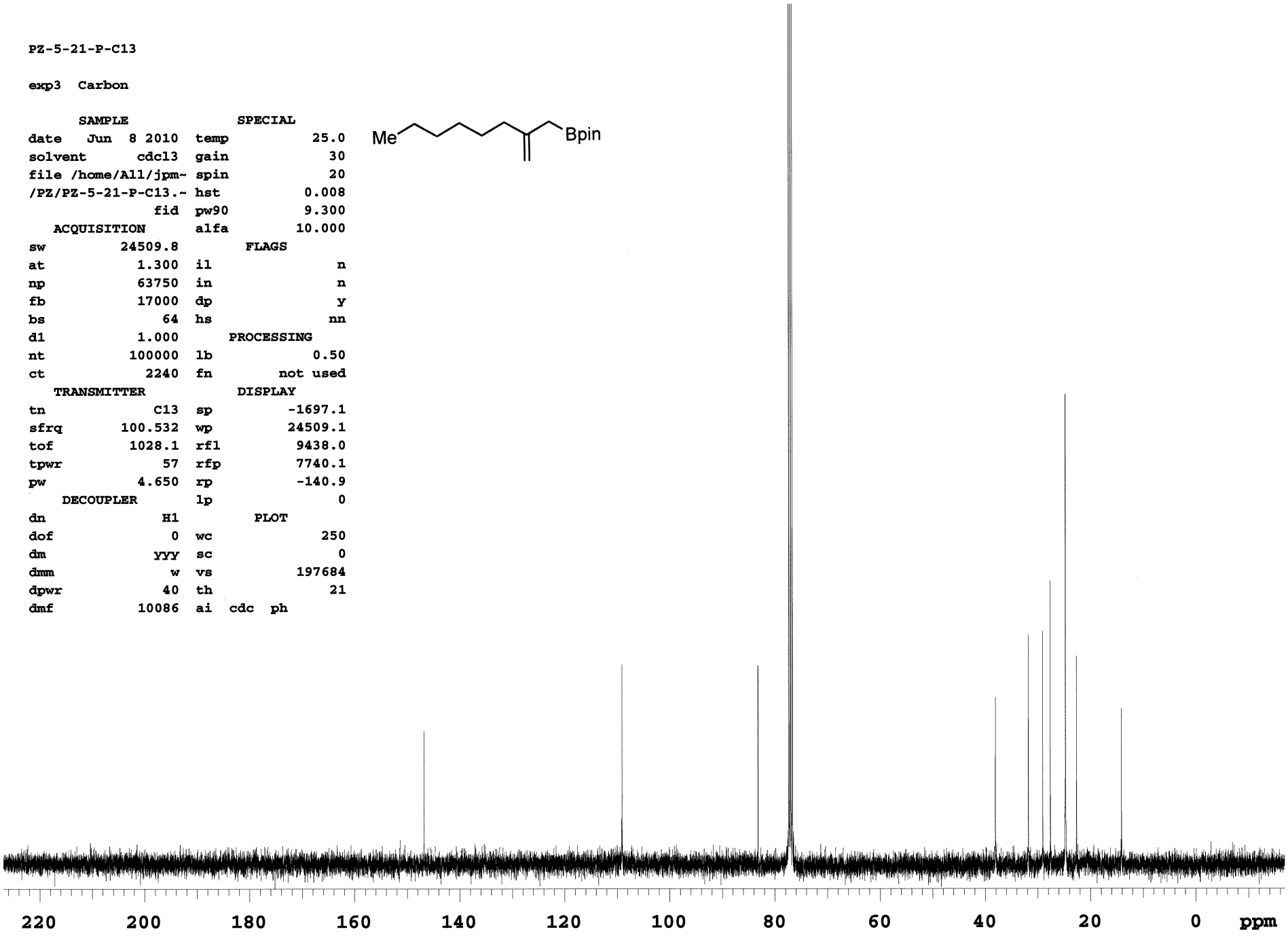
SAMPLE		SPECIAL	
date	Jun 8 2010	temp	25.0
solvent	cdcl3	gain	not used
file	/home/All/jpm~	spin	20
	/PZ/PZ-5-21-P.fid	hst	0.008
ACQUISITION		pw90	9.312
sw	6410.3	alfa	10.000
at	2.049	FLAGS	
np	26264	il	n
fb	4000	in	n
bs	4	dp	y
ss	2	hs	nn
d1	1.000	PROCESSING	
nt	32	fn	65536
ct	32	DISPLAY	
TRANSMITTER		sp	-0.2
tn	H1	wp	3997.6
sfrq	399.769	rf1	3702.4
tof	399.8	rfp	2902.3
tpwr	61	rp	-31.5
pw	4.656	lp	0
DECOUPLER		PLOT	
dn	C13	wc	250
dof	0	sc	0
dm	nnn	vs	20
dmm	c	th	94
dpwr	35	ai cdc ph	
dmf	29412		



PZ-5-21-P-C13

exp3 Carbon

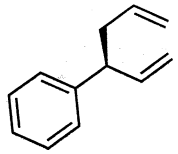
SAMPLE		SPECIAL	
date	Jun 8 2010	temp	25.0
solvent	cdcl3	gain	30
file	/home/All/jpm~	spin	20
/PZ/PZ-5-21-P-C13.~		hst	0.008
	fid	pw90	9.300
ACQUISITION		alfa	10.000
sw	24509.8	FLAGS	
at	1.300	il	n
np	63750	in	n
fb	17000	dp	y
bs	64	hs	nn
d1	1.000	PROCESSING	
nt	100000	lb	0.50
ct	2240	fn	not used
TRANSMITTER		DISPLAY	
tn	C13	sp	-1697.1
sfrq	100.532	wp	24509.1
tof	1028.1	rfl	9438.0
tpwr	57	rfp	7740.1
pw	4.650	rp	-140.9
DECOUPLER		lp	0
dn	H1	PLOT	
dof	0	wc	250
dm	yyy	sc	0
dmm	w	vs	197684
dpwr	40	th	21
dmf	10086	ai cdc ph	

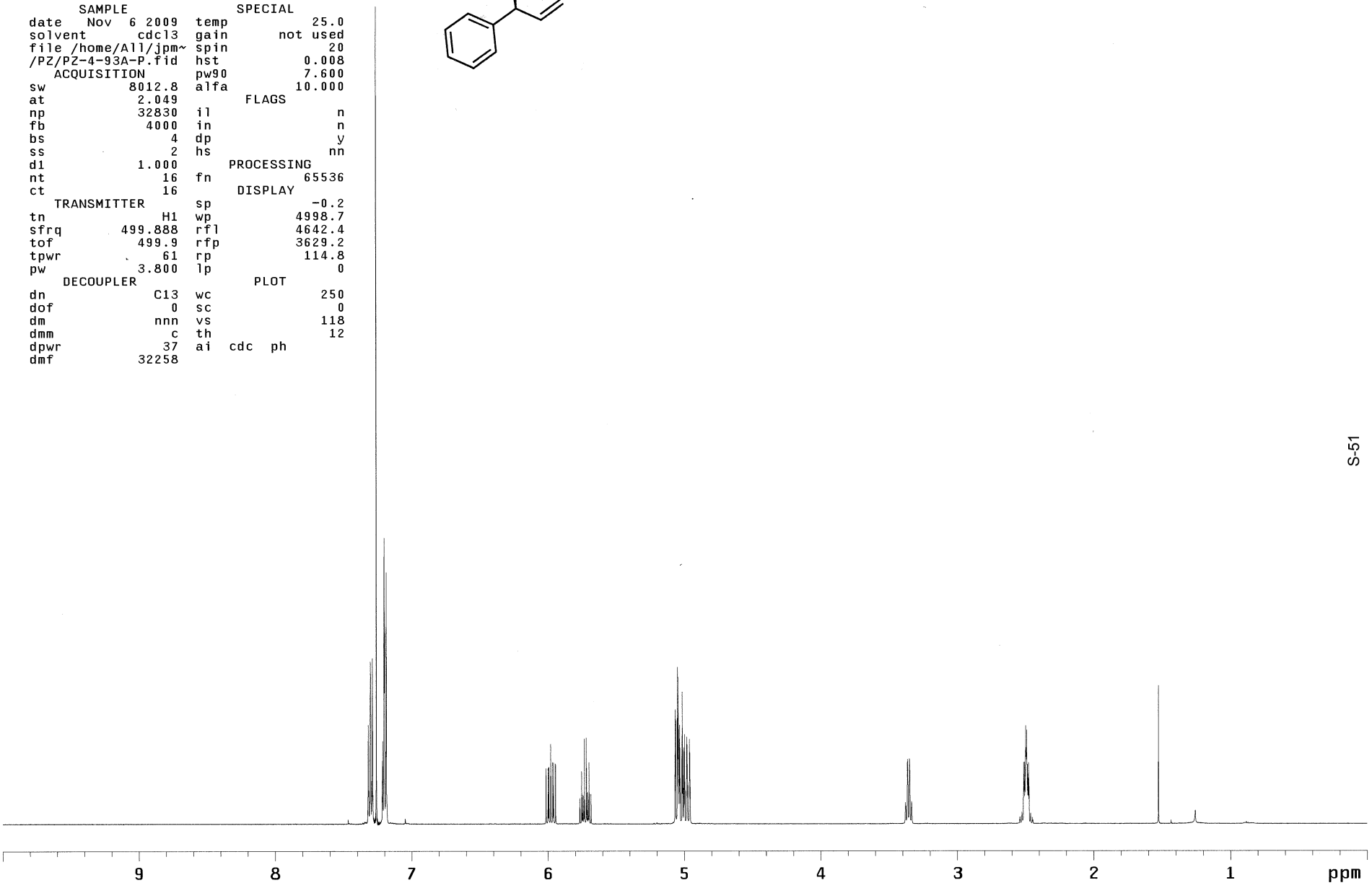


PZ-4-93A-P

exp1 Proton

SAMPLE		SPECIAL	
date	Nov 6 2009	temp	25.0
solvent	cdcl3	gain	not used
file	/home/All/jpm~	spin	20
	/PZ/PZ-4-93A-P.fid	hst	0.008
ACQUISITION		pw90	7.600
sw	8012.8	alfa	10.000
at	2.049	FLAGS	
np	32830	il	n
fb	4000	in	n
bs	4	dp	y
ss	2	hs	nn
d1	1.000	PROCESSING	
nt	16	fn	65536
ct	16	DISPLAY	
TRANSMITTER		sp	-0.2
tn	H1	wp	4998.7
sfrq	499.888	rfl	4642.4
tof	499.9	rfp	3629.2
tpwr	61	rp	114.8
pw	3.800	lp	0
DECOUPLER		PLOT	
dn	C13	wc	250
dof	0	sc	0
dm	nnn	vs	118
dmm	c	th	12
dpwr	37	ai cdc ph	
dmf	32258		

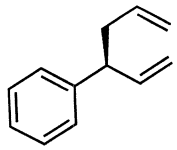


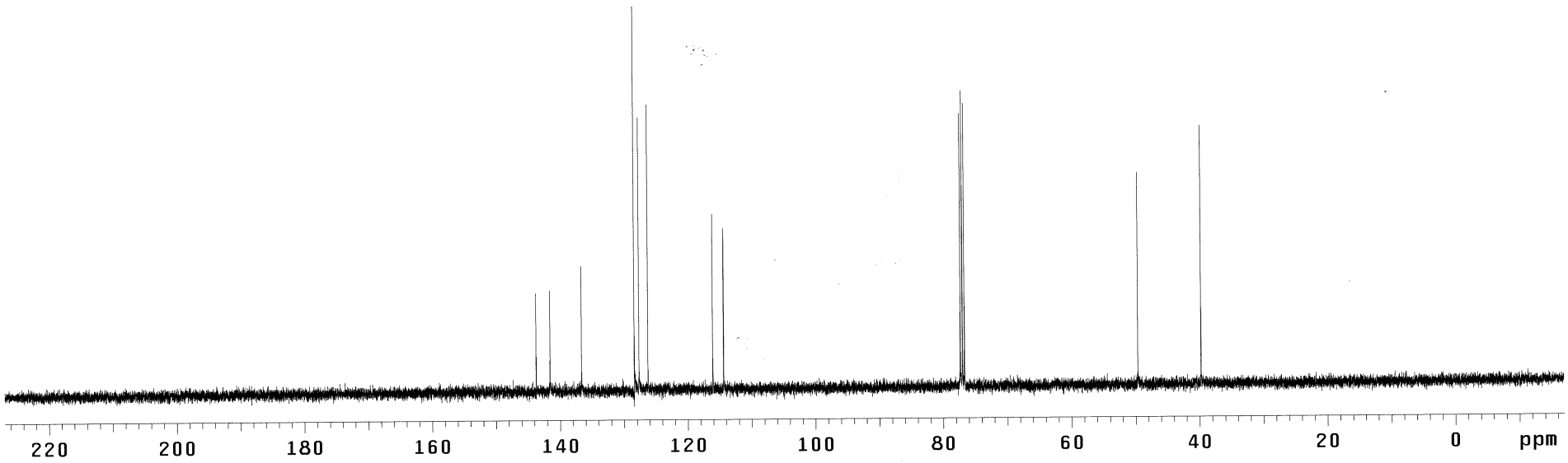


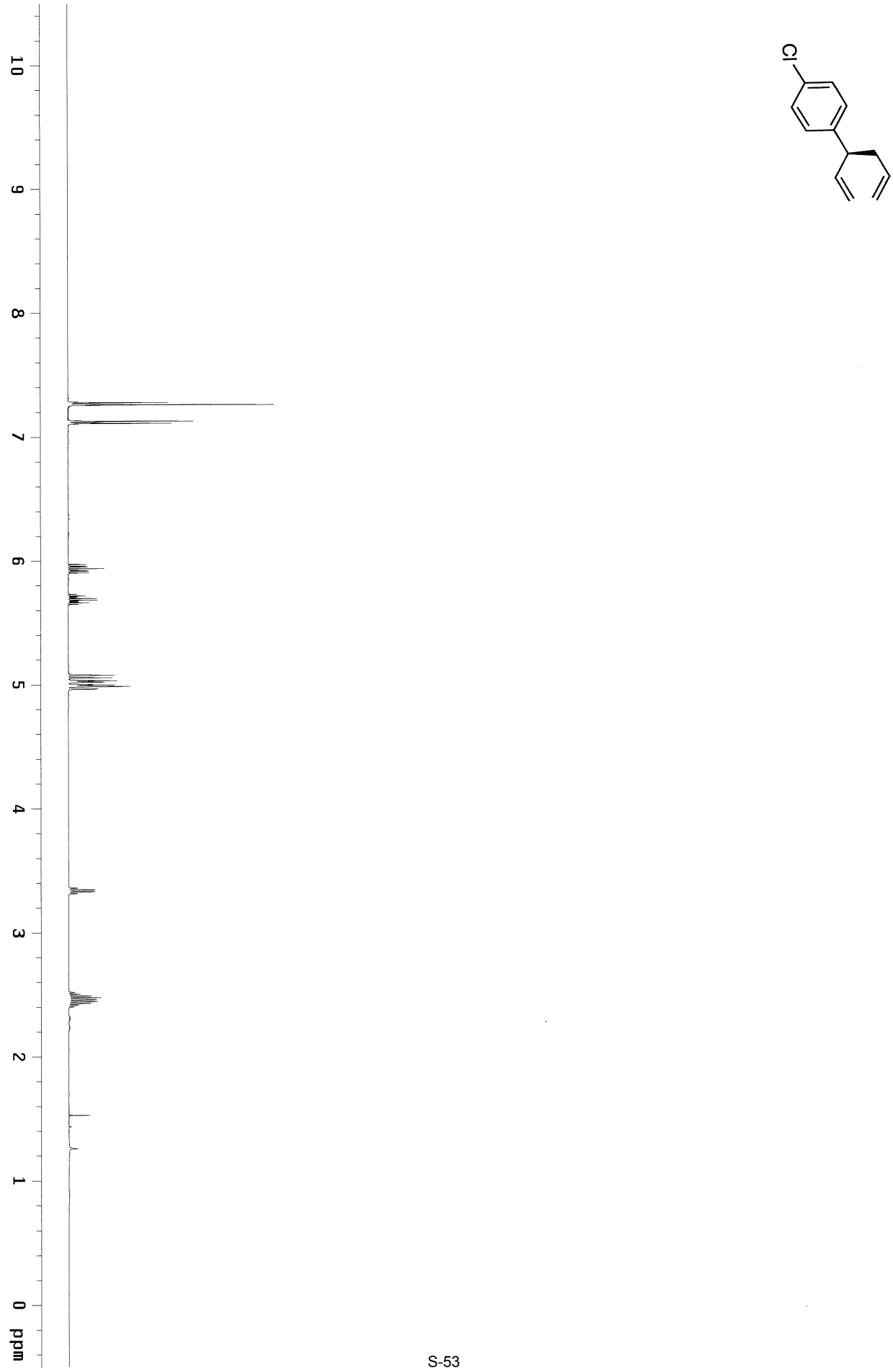
PZ-4-38C-P-C13

exp1 Carbon

SAMPLE		SPECIAL	
date	Sep 25 2009	temp	25.0
solvent	cdcl3	gain	30
file	/home/All/jpm~	spin	not used
/PZ/PZ-4-38C-P-C13~	.fid	hst	0.008
		pw90	9.300
ACQUISITION		alfa	10.000
sw	24509.8	FLAGS	
at	1.300	il	n
np	63750	in	n
fb	17000	dp	y
bs	16	hs	nn
d1	1.000	PROCESSING	
nt	256	lb	0.50
ct	256	fn	not used
TRANSMITTER		DISPLAY	
tn	C13	sp	-1698.6
sfrq	100.532	wp	24509.1
tof	1028.1	rfl	9439.5
tpwr	57	rfp	7740.1
pw	4.650	rp	47.2
DECOUPLER		lp	0
dn	H1	PLOT	
dof	0	wc	250
dm	yyy	sc	0
dmm	w	vs	32723
dpwr	40	th	19
dmf	10086	ai ph	

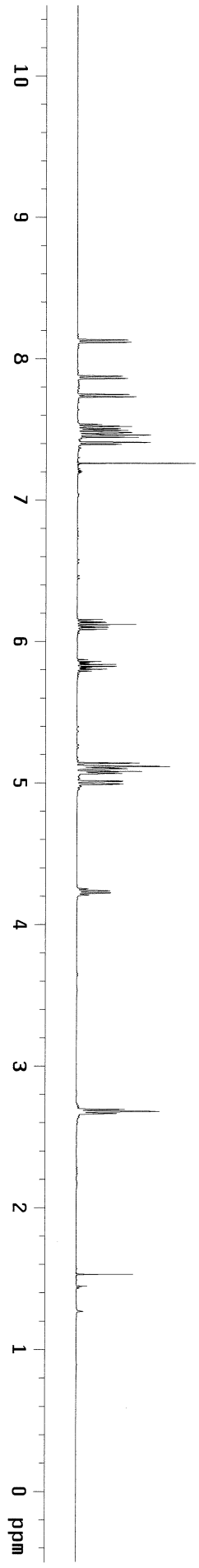





Cl
10
9
8
7
6
5
4
3
2
1
0
ppm

220
200
180
160
140
120
100
80
60
40
20
0
ppm

Cl


10
9
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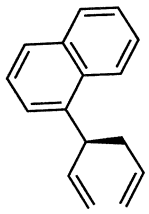
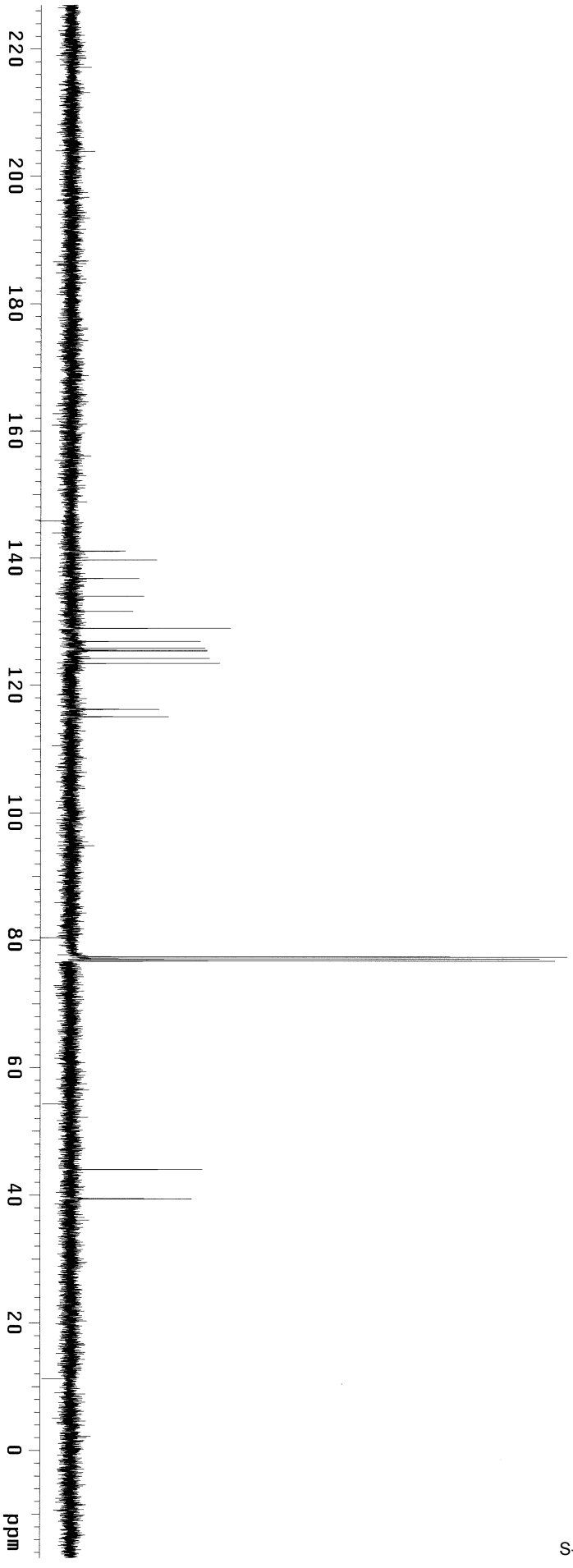
7
6
5
4
3
2
1
0
ppm

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200
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140
120
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80
60
40
20
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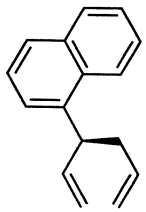
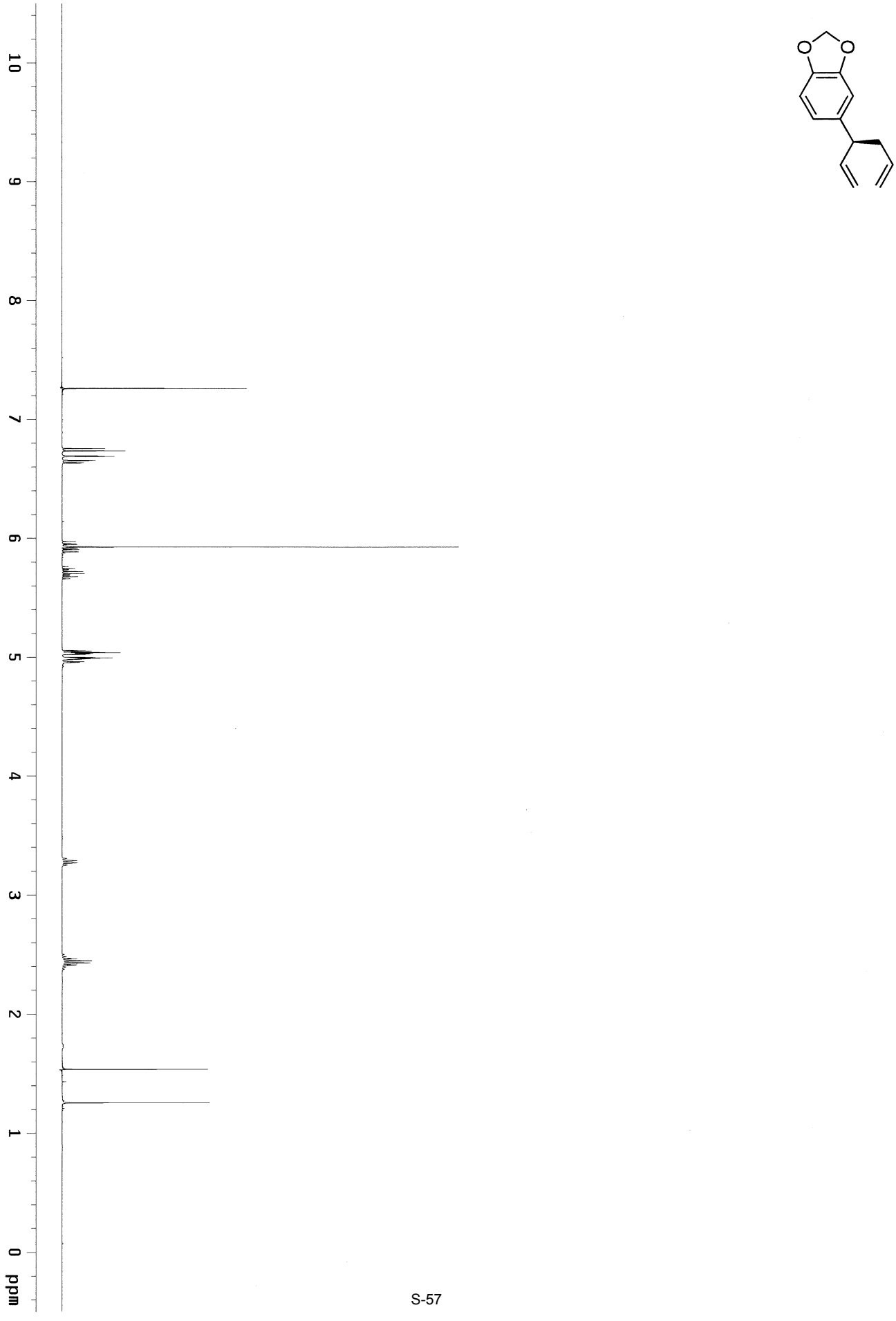
ppm

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6
5
4
3
2
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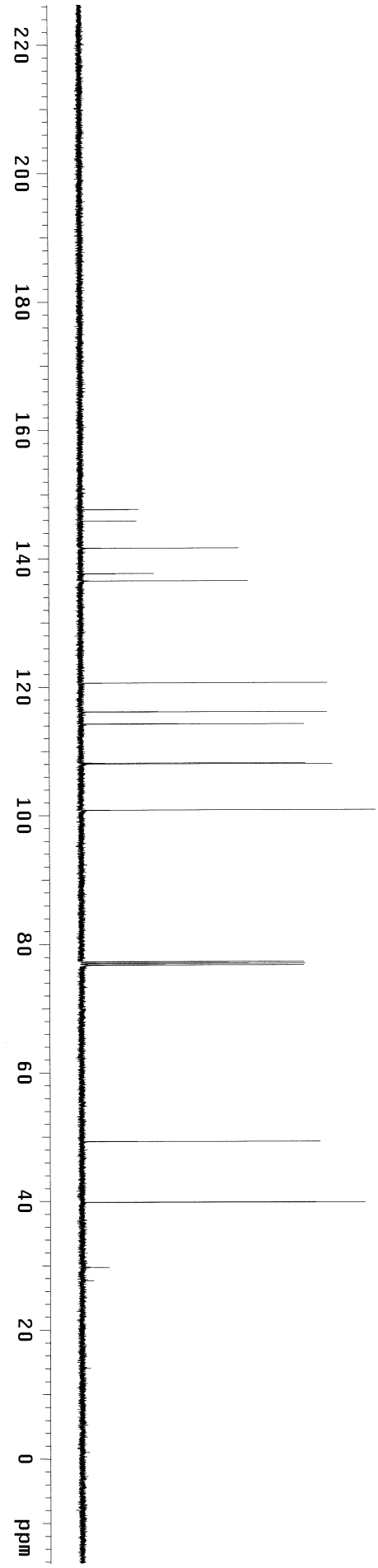
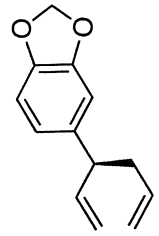
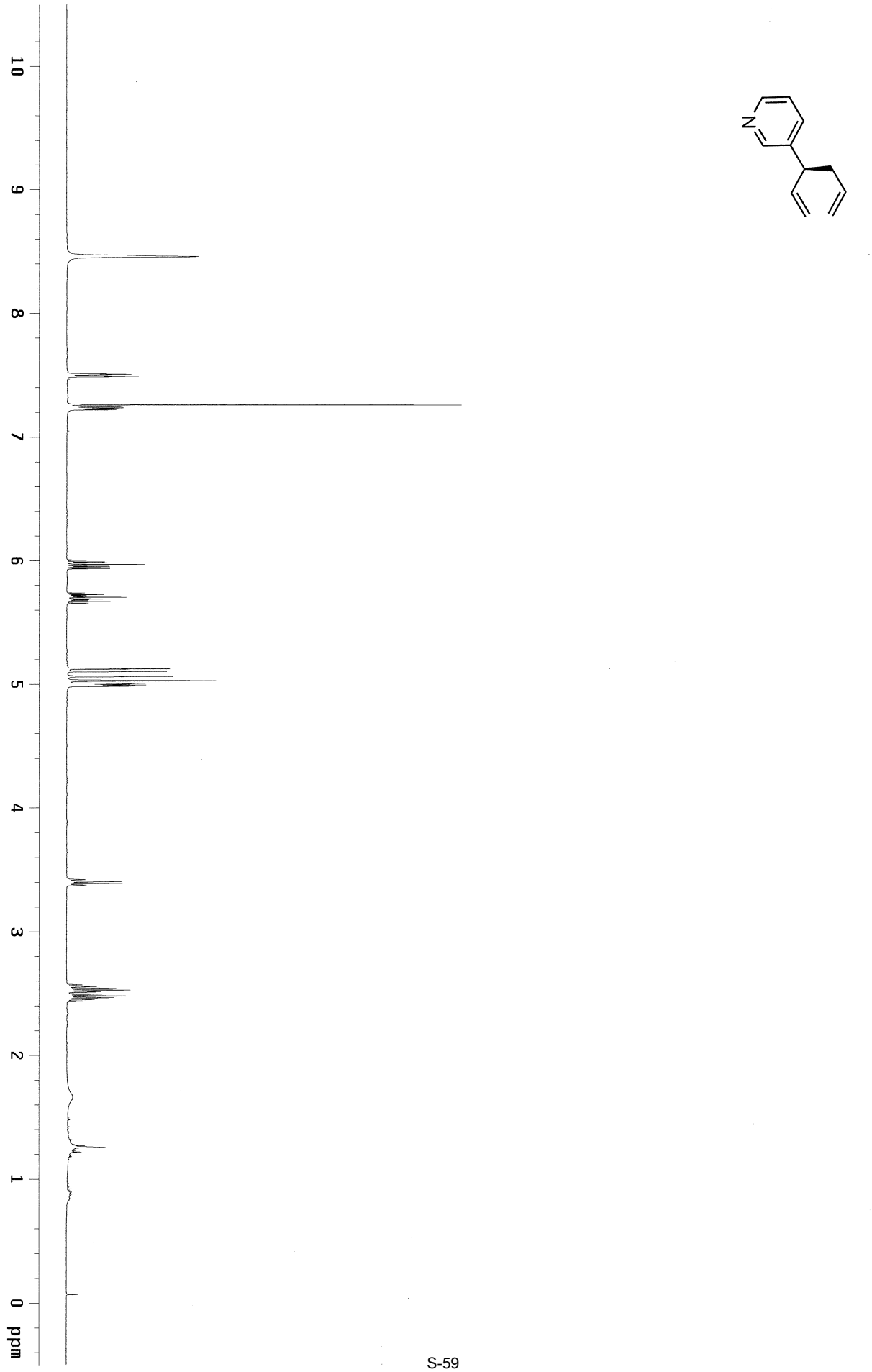
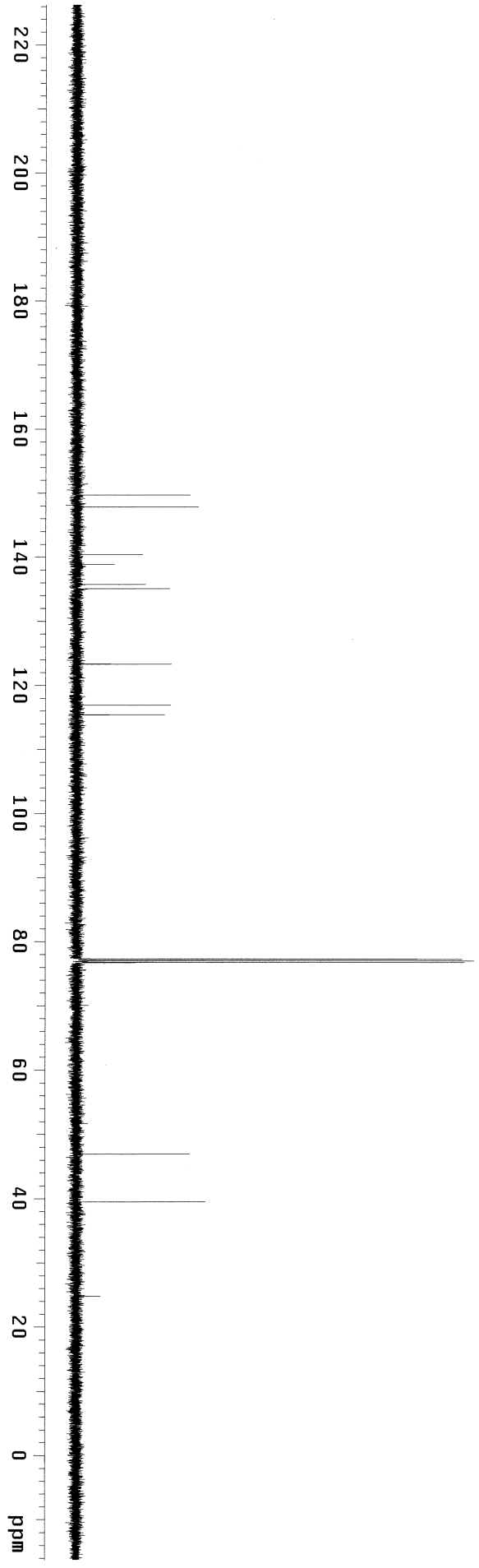
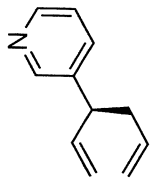
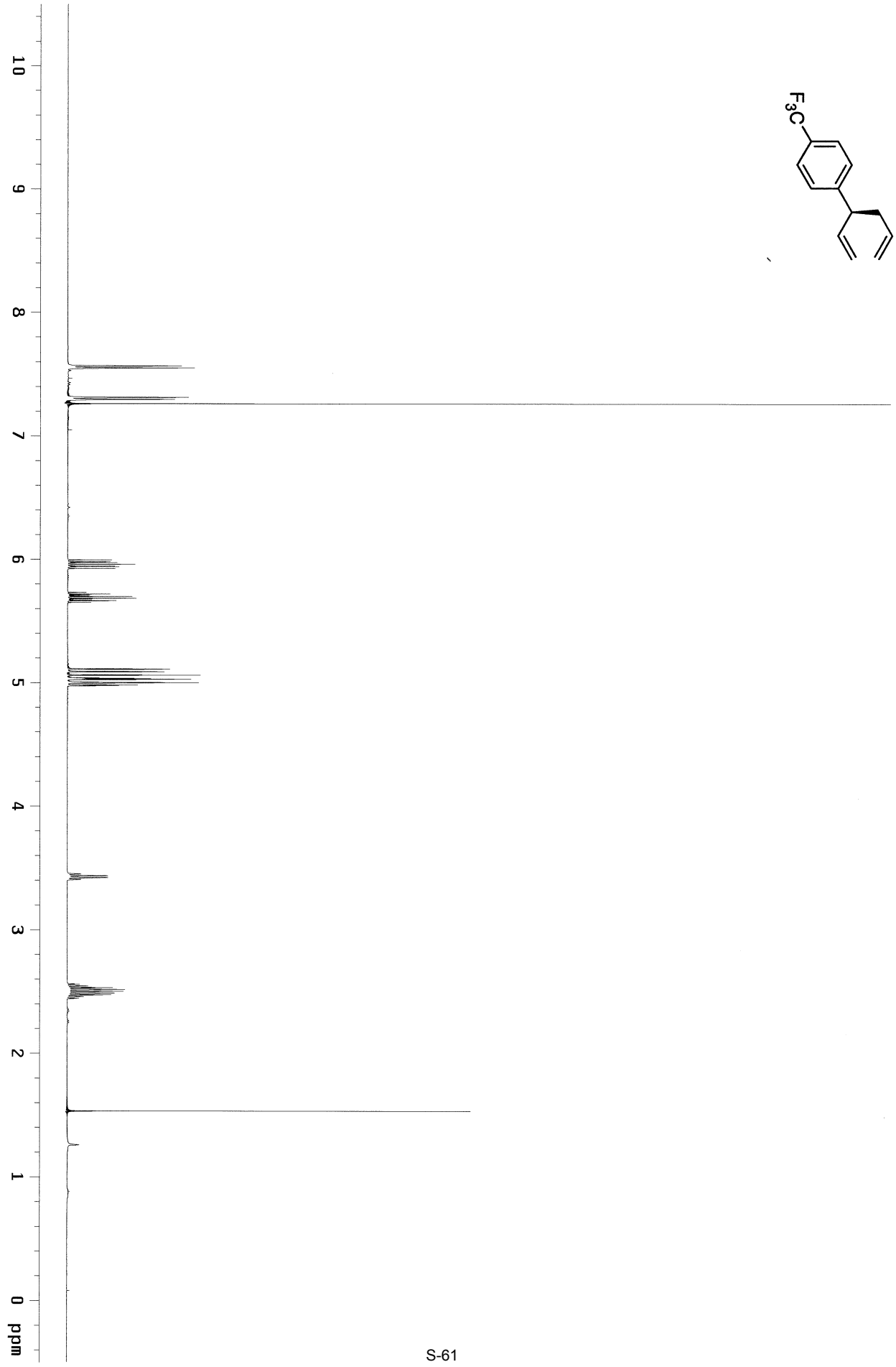

220
200
180
160
140
120
100
80
60
40
20
0
ppm




10
9
8
7
6
5
4
3
2
1
0
ppm
N


220
200
180
160
140
120
100
80
60
40
20
0
ppm


N


F3C
10
9
8
7
6
5
4
3
2
1
0
ppm

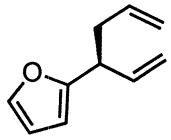
220
200
180
160
140
120
100
80
60
40
20
0
ppm

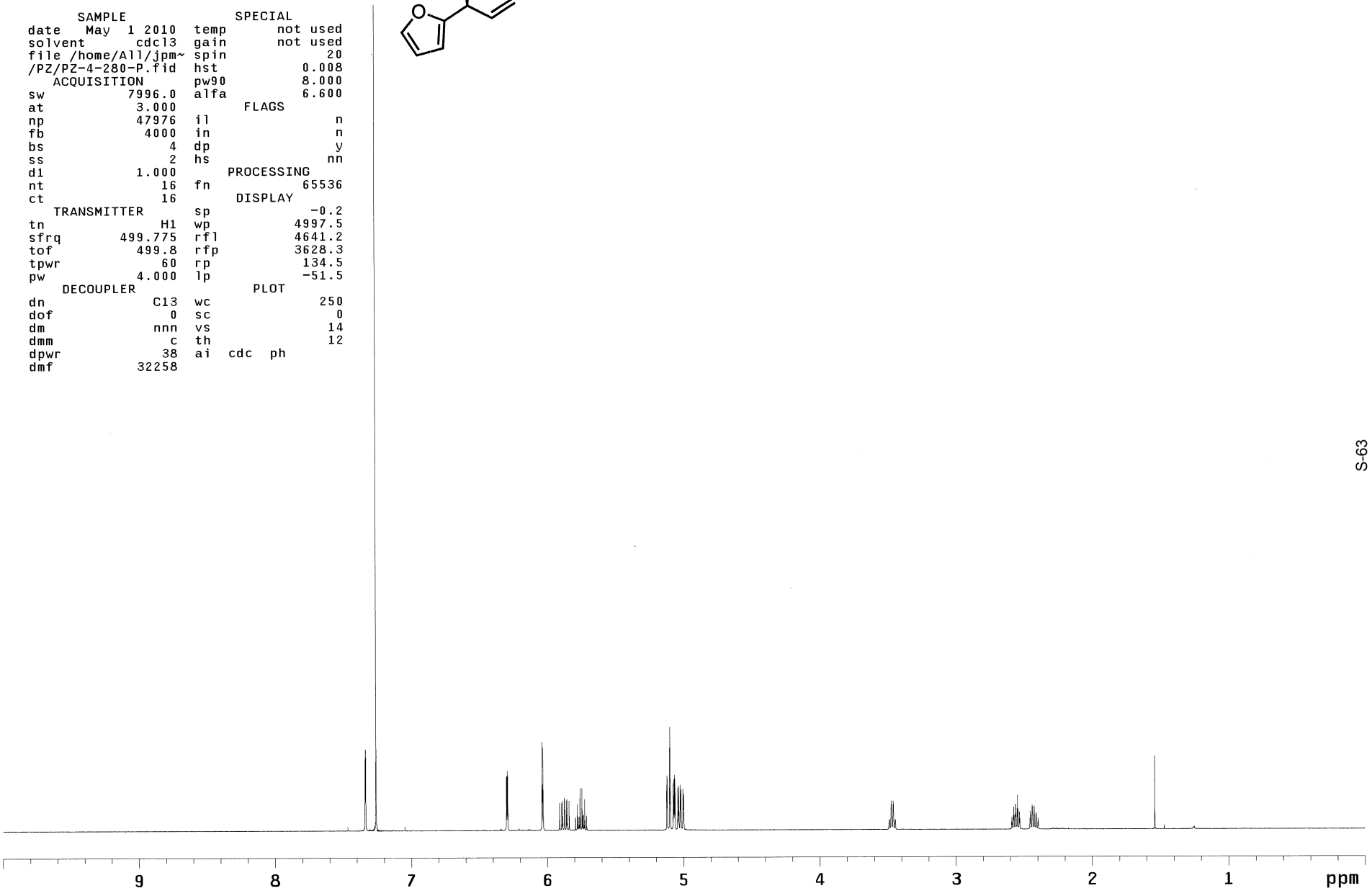
F3C

PZ-4-280-P

exp2 Proton

SAMPLE		SPECIAL	
date	May 1 2010	temp	not used
solvent	cdcl3	gain	not used
file	/home/All/jpm~	spin	20
	/PZ/PZ-4-280-P.fid	hst	0.008
ACQUISITION		pw90	8.000
sw	7996.0	alfa	6.600
at	3.000	FLAGS	
np	47976	il	n
fb	4000	in	n
bs	4	dp	y
ss	2	hs	nn
d1	1.000	PROCESSING	
nt	16	fn	65536
ct	16	DISPLAY	
TRANSMITTER		sp	-0.2
tn	H1	wp	4997.5
sfrq	499.775	rfl	4641.2
tof	499.8	rfp	3628.3
tpwr	60	rp	134.5
pw	4.000	lp	-51.5
DECOUPLER		PLOT	
dn	C13	wc	250
dof	0	sc	0
dm	nnn	vs	14
dmm	c	th	12
dpwr	38	ai cdc ph	
dmf	32258		

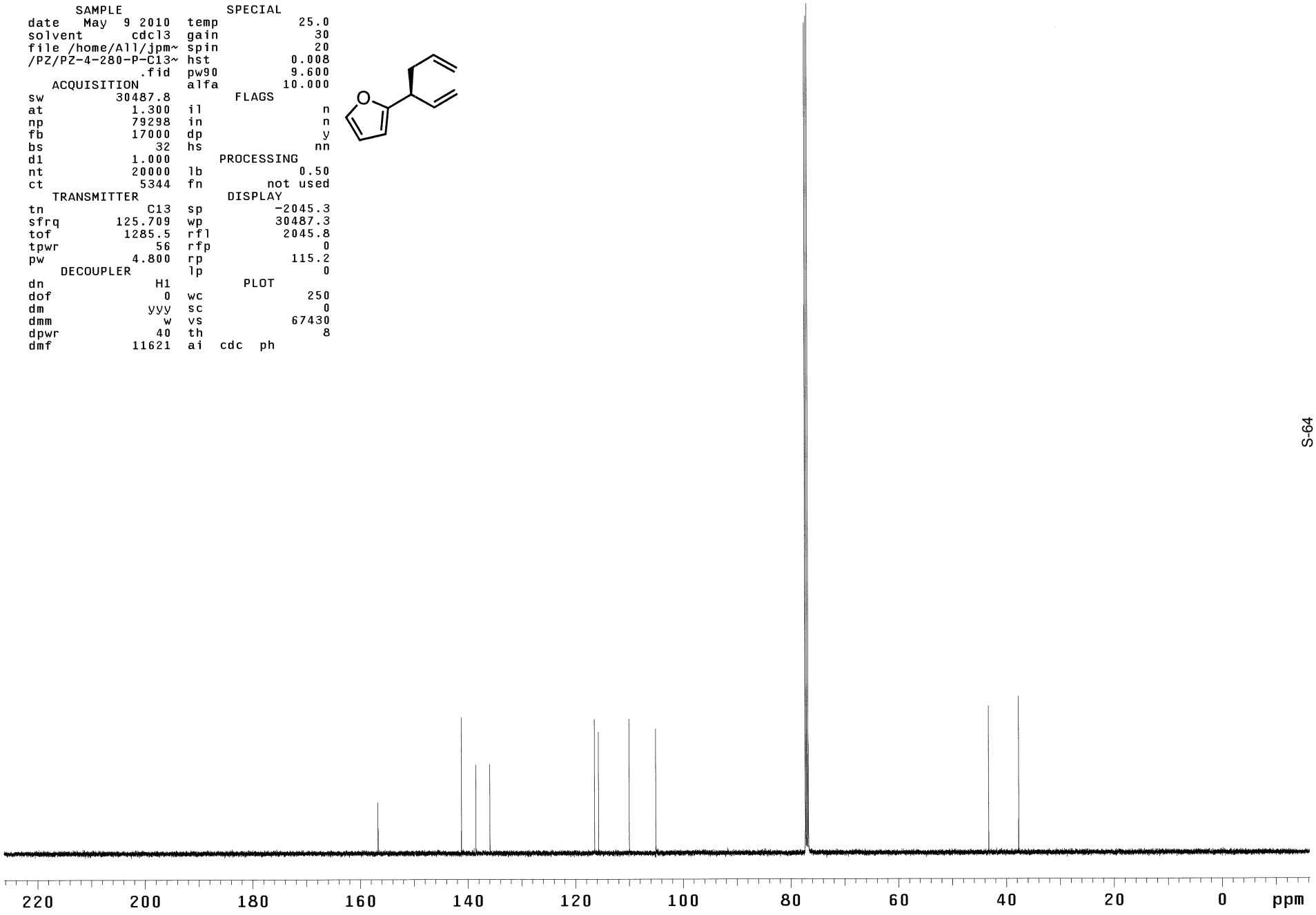




PZ-4-280-P-C13

exp2 Carbon

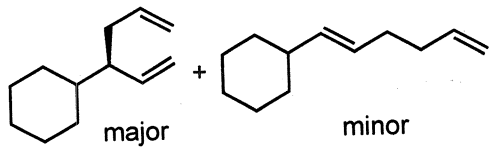
SAMPLE		SPECIAL	
date	May 9 2010	temp	25.0
solvent	cdcl3	gain	30
file	/home/All/jpm~	spin	20
	/PZ/PZ-4-280-P-C13~	hst	0.008
	.fid	pw90	9.600
ACQUISITION		alfa	10.000
sw	30487.8	FLAGS	
at	1.300	il	n
np	79298	in	n
fb	17000	dp	y
bs	32	hs	nn
d1	1.000	PROCESSING	
nt	20000	lb	0.50
ct	5344	fn	not used
TRANSMITTER		DISPLAY	
tn	C13	sp	-2045.3
sfrq	125.709	wp	30487.3
tof	1285.5	rfl	2045.8
tpwr	56	rfp	0
pw	4.800	rp	115.2
DECOUPLER		lp	0
dn	H1	PLOT	
dof	0	wc	250
dm	yyy	sc	0
dmm	w	vs	67430
dpwr	40	th	8
dmf	11621	ai cdc ph	



PZ-4-123-P

exp1 Proton

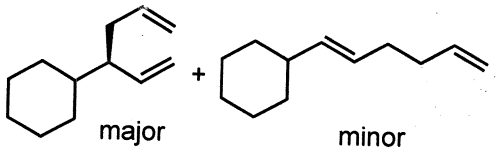
SAMPLE		SPECIAL	
date	Dec 10 2009	temp	25.0
solvent	cdcl3	gain	not used
file	/home/All/jpm~	spin	20
	/PZ/PZ-4-123-P.fid	hst	0.008
ACQUISITION		pw90	8.000
sw	7996.0	alfa	6.600
at	3.000	FLAGS	
np	47976	il	n
fb	4000	in	n
bs	4	dp	y
ss	2	hs	nn
d1	1.000	PROCESSING	
nt	16	fn	65536
ct	16	DISPLAY	
TRANSMITTER		sp	-0.2
tn	H1	wp	4997.5
sfrq	499.775	rfl	4640.5
tof	499.8	rfp	3628.3
tpwr	60	rp	-89.3
pw	4.000	lp	-48.7
DECOUPLER		PLOT	
dn	C13	wc	250
dof	0	sc	0
dm	nnn	vs	25
dmm	c	th	12
dpwr	38	ai cdc ph	
dmf	32258		

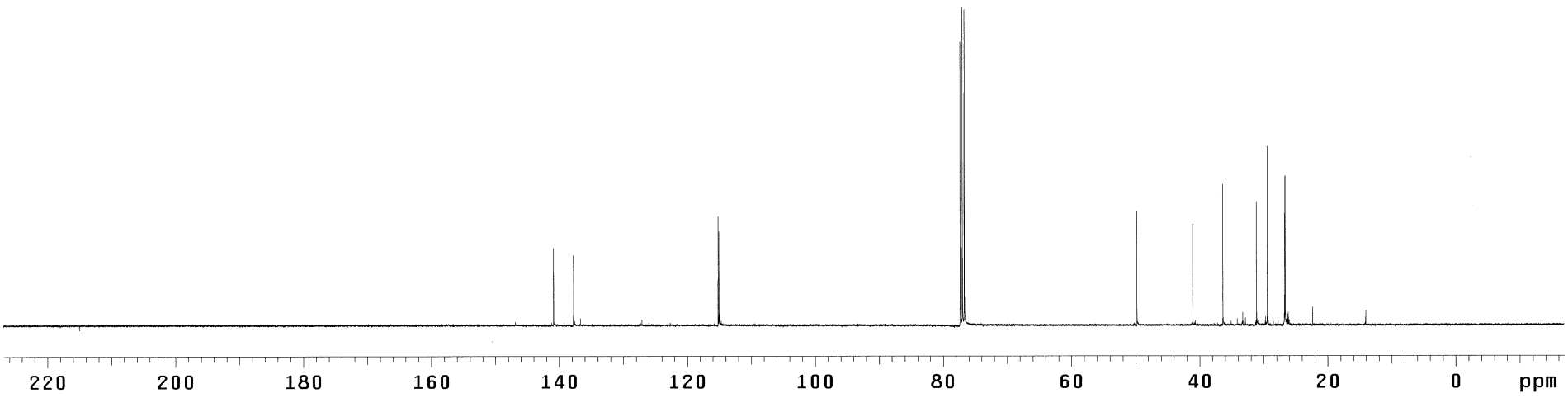


PZ-4-151-P-C13

exp1 Carbon

SAMPLE		SPECIAL	
date	Jan 23 2010	temp	25.0
solvent	cdcl3	gain	30
file	/home/All/jpm~	spin	not used
/PZ/PZ-4-151-P-C13~	.fid	hst	0.008
		pw90	9.300
ACQUISITION		alfa	10.000
sw	24509.8	FLAGS	
at	1.300	il	n
np	63750	in	n
fb	17000	dp	y
bs	16	hs	nn
d1	1.000	PROCESSING	
nt	25600	lb	0.50
ct	12592	fn	not used
TRANSMITTER		DISPLAY	
tn	C13	sp	-1697.1
sfrq	100.532	wp	24509.1
tof	1028.1	rfl	9438.0
tpwr	57	rfp	7740.1
pw	4.650	rp	-131.5
DECOUPLER		lp	0
dn	H1	PLOT	
dof	0	wc	250
dm	yyy	sc	0
dmm	w	vs	19223
dpwr	40	th	20
dmf	10086	ai cdc ph	

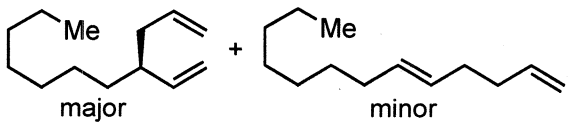


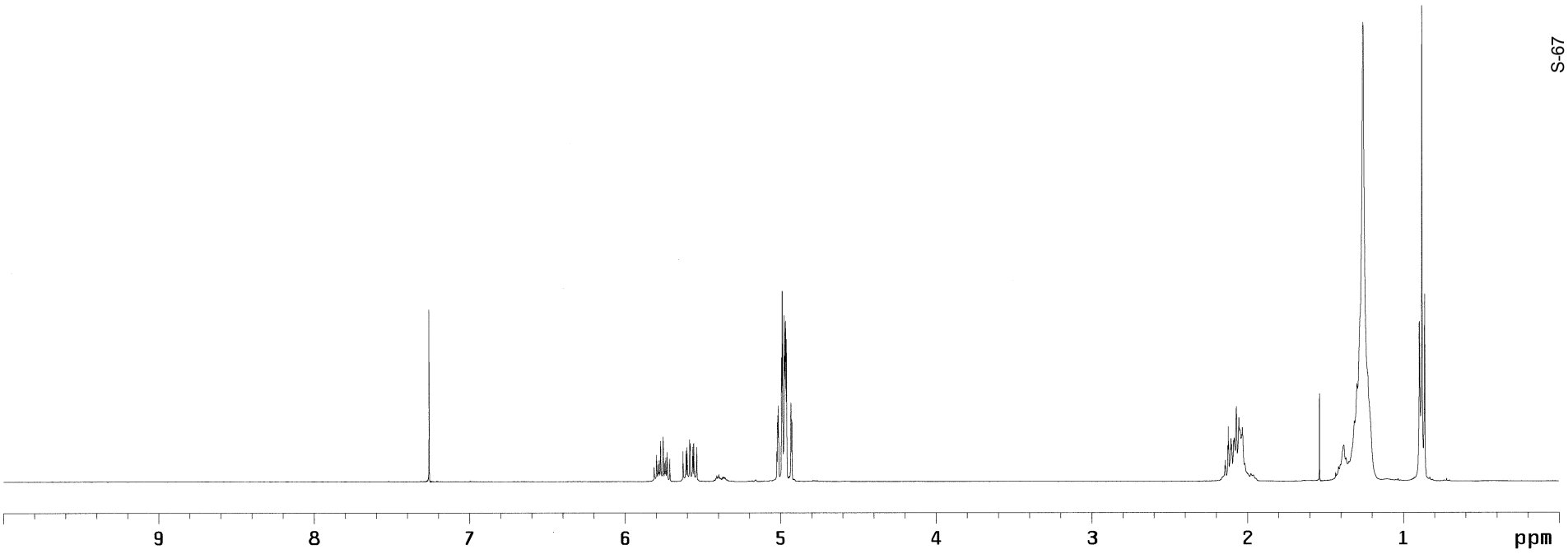


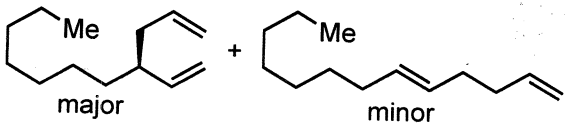
PZ-4-133-P

exp1 Proton

SAMPLE		SPECIAL	
date	Jan 14 2010	temp	25.0
solvent	cdcl3	gain	not used
file	/home/All/jpm~	spin	not used
	/PZ/PZ-4-133-P-con~	hst	0.008
	centrate.fid	pw90	11.300
ACQUISITION		alfa	10.000
sw	6410.3	FLAGS	
at	2.049	il	n
np	26264	in	n
fb	4000	dp	y
bs	4	hs	nn
ss	2	PROCESSING	
d1	1.000	fn	65536
nt	16	DISPLAY	
ct	16	sp	-0.2
TRANSMITTER		wp	3997.6
tn	H1	rfl	3702.2
sfrq	399.769	rfp	2902.3
tof	399.8	rp	119.0
tpwr	59	lp	0
pw	5.650	PLOT	
DECOUPLER		wc	250
dn	C13	sc	0
dof	0	vs	200
dm	nnn	th	12
dmm	c	ai cdc ph	
dpwr	35		
dmf	29412		



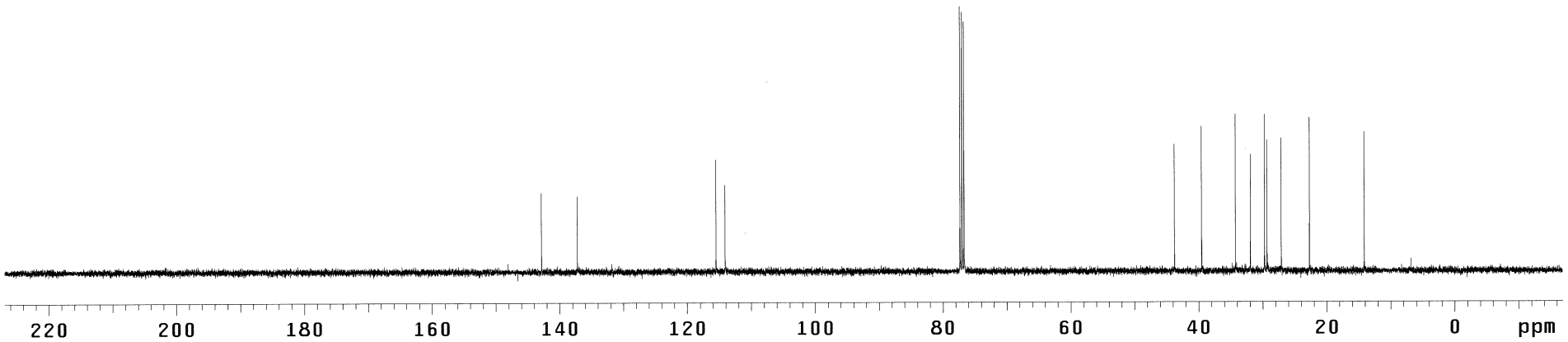




PZ-4-133-P

exp1 Carbon

SAMPLE		SPECIAL	
date	Jan 14 2010	temp	25.0
solvent	cdcl3	gain	30
file	/home/All/jpm~	spin	not used
	/PZ/PZ-4-133-P-C13~	hst	0.008
	.fid	pw90	9.300
ACQUISITION		alfa	10.000
sw	24509.8	FLAGS	
at	1.300	il	n
np	63750	in	n
fb	17000	dp	y
bs	16	hs	nn
d1	1.000	PROCESSING	
nt	5000	lb	0.50
ct	1488	fn	not used
TRANSMITTER		DISPLAY	
tn	C13	sp	-1697.9
sfrq	100.532	wp	24509.1
tof	1028.1	rfl	9438.8
tpwr	57	rfp	7740.1
pw	4.650	rp	154.9
DECOUPLER		lp	0
dn	H1	PLOT	
dof	0	wc	250
dm	yyy	sc	0
dmm	w	vs	18000
dpwr	40	th	22
dmf	10086	ai cdc ph	



PZ-4-226A-P

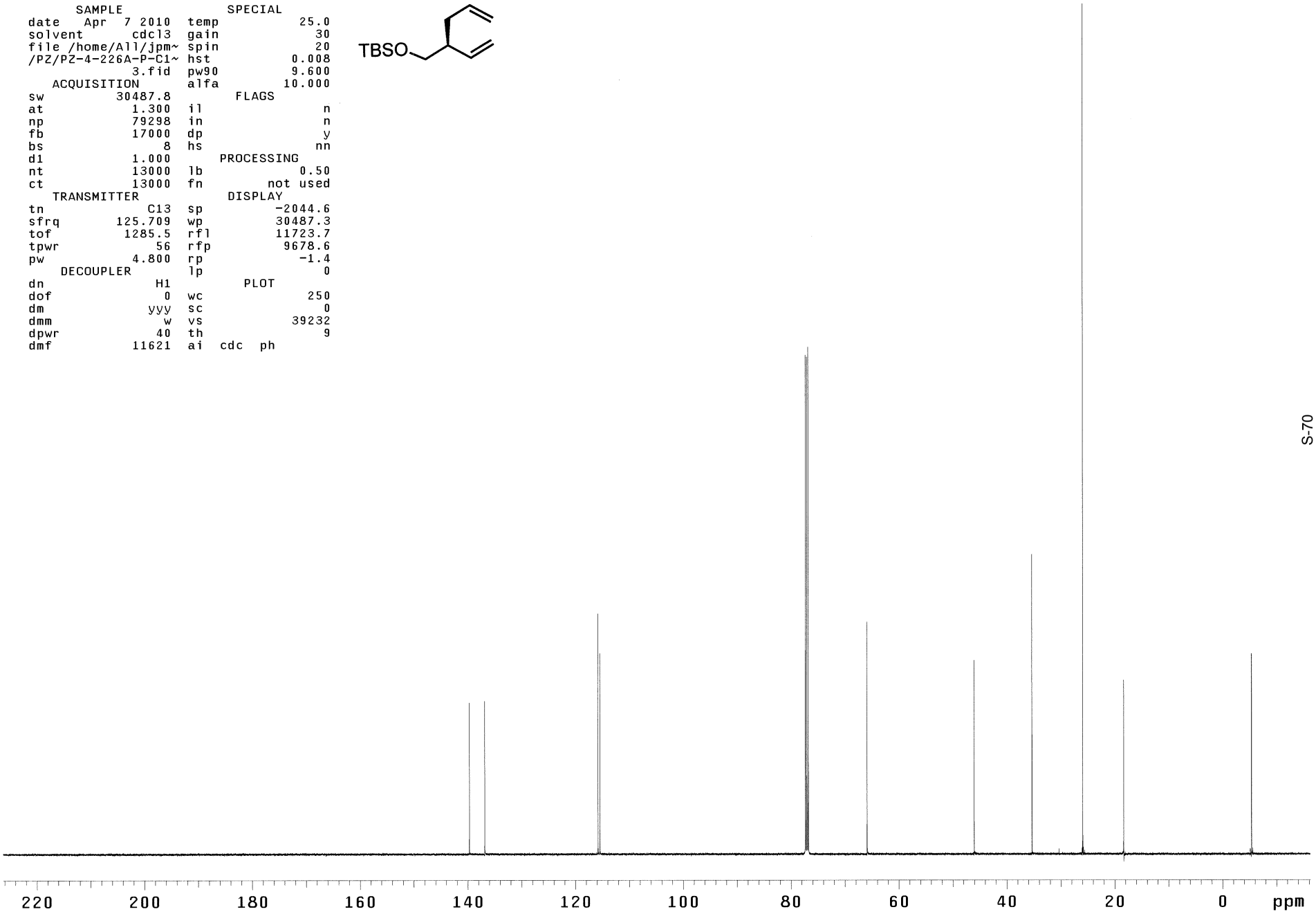
exp2 Proton

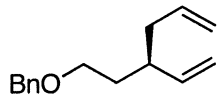
SAMPLE		SPECIAL	
date	Apr 7 2010	temp	25.0
solvent	cdcl3	gain	not used
file	/home/All/jpm~	spin	20
	/PZ/PZ-4-226A-P.fi~	hst	0.008
	d	pw90	7.600
ACQUISITION		alfa	10.000
sw	8012.8	FLAGS	
at	2.049	il	n
np	32830	in	n
fb	4000	dp	y
bs	4	hs	nn
ss	2	PROCESSING	
d1	1.000	fn	65536
nt	32	DISPLAY	
ct	28	sp	-256.2
TRANSMITTER		wp	5248.6
tn	H1	rfl	4642.4
sfrq	499.888	rfp	3629.2
tof	499.9	rp	17.7
tpwr	61	lp	0
pw	3.800	PLOT	
DECOUPLER		wc	250
dn	C13	sc	0
dof	0	vs	72
dm	nnn	th	12
dmm	c	ai cdc ph	
dpwr	37		
dmf	32258		

PZ-4-226A-P-C13

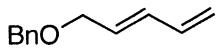
exp2 Carbon

SAMPLE		SPECIAL	
date	Apr 7 2010	temp	25.0
solvent	cdcl3	gain	30
file	/home/All/jpm~	spin	20
	/PZ/PZ-4-226A-P-C1~	hst	0.008
	3.fid	pw90	9.600
ACQUISITION		alfa	10.000
sw	30487.8	FLAGS	
at	1.300	il	n
np	79298	in	n
fb	17000	dp	y
bs	8	hs	nn
d1	1.000	PROCESSING	
nt	13000	lb	0.50
ct	13000	fn	not used
TRANSMITTER		DISPLAY	
tn	C13	sp	-2044.6
sfrq	125.709	wp	30487.3
tof	1285.5	rfl	11723.7
tpwr	56	rfp	9678.6
pw	4.800	rp	-1.4
DECOUPLER		lp	0
dn	H1	PLOT	
dof	0	wc	250
dm	yyy	sc	0
dmm	w	vs	39232
dpwr	40	th	9
dmf	11621	ai cdc ph	

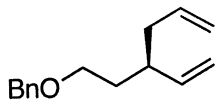


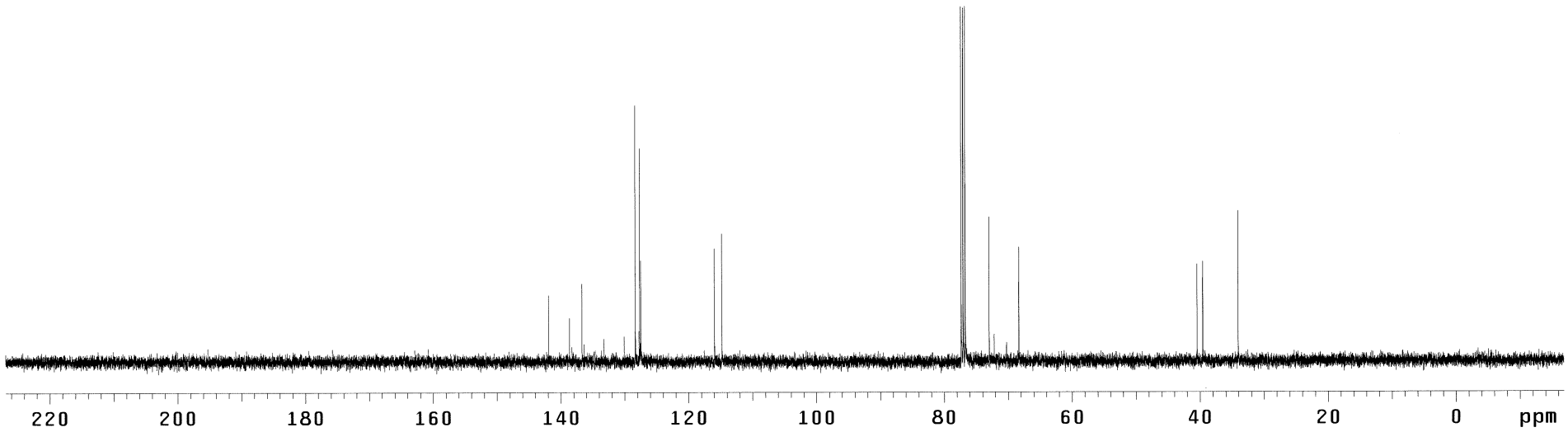


A, Major



B, Minor

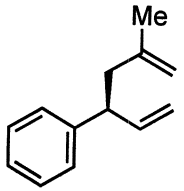

BnO

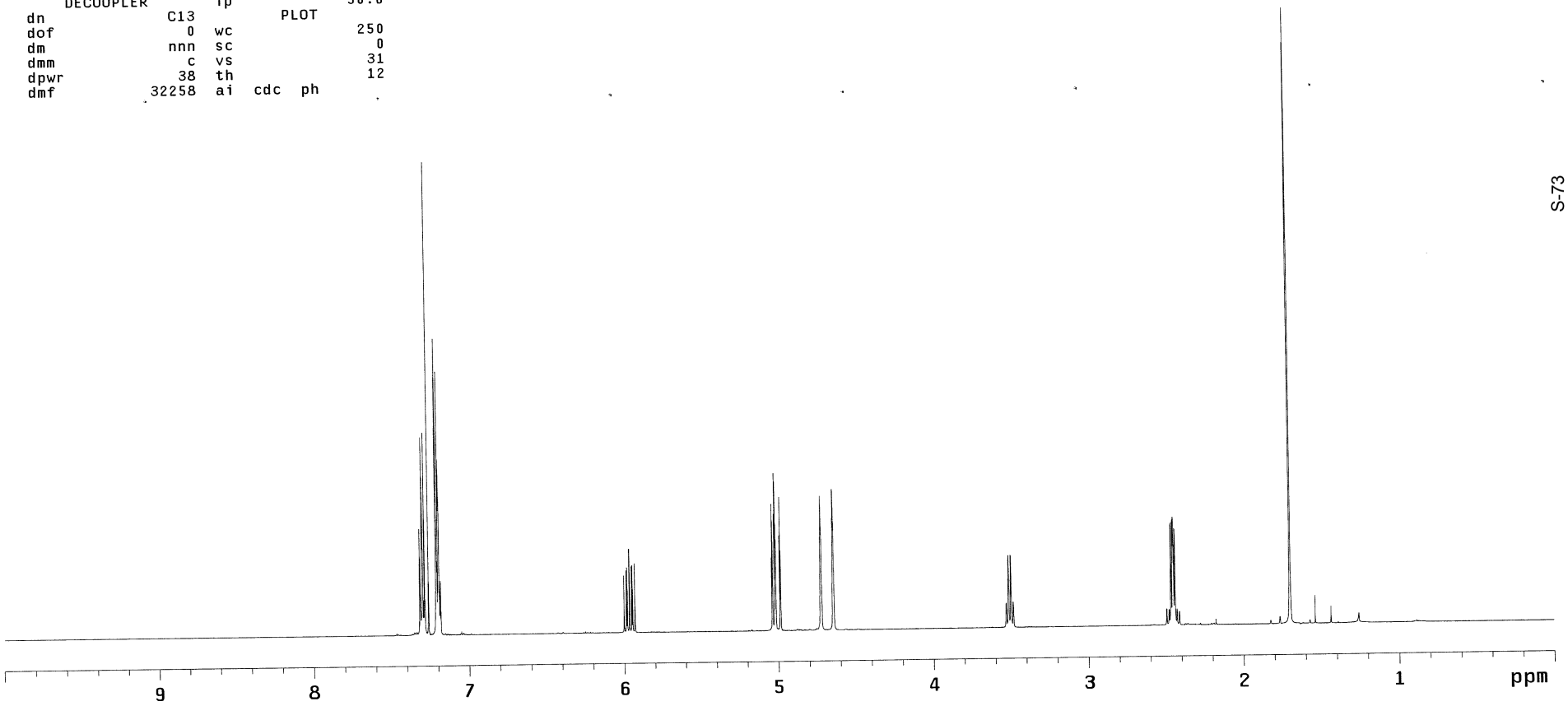

220
200
180
160
140
120
100
80
60
40
20
0
ppm

PZ-4-245B-P

exp10 Proton

SAMPLE		SPECIAL	
date	Apr 12 2010	temp	25.0
solvent	cdcl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	7996.0	pw90	8.000
at	3.000	alfa	6.600
np	47976	**FLAGS**	
fb	4000	il	n
bs	2	in	n
ss	2	dp	y
d1	1.000	hs	nn
nt	16	**PROCESSING**	
ct	16	fn	65536
TRANSMITTER		**DISPLAY**	
tn	H1	sp	-0.2
sfrq	499.775	wp	4997.5
tof	499.8	rfl	4641.0
tpwr	60	rfp	3628.3
pw	4.000	rp	140.2
DECOUPLER		lp	-50.6
dn	C13	**PLOT**	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	31
dpwr	38	th	12
dmf	32258	ai	cdc ph

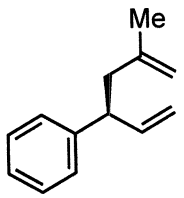


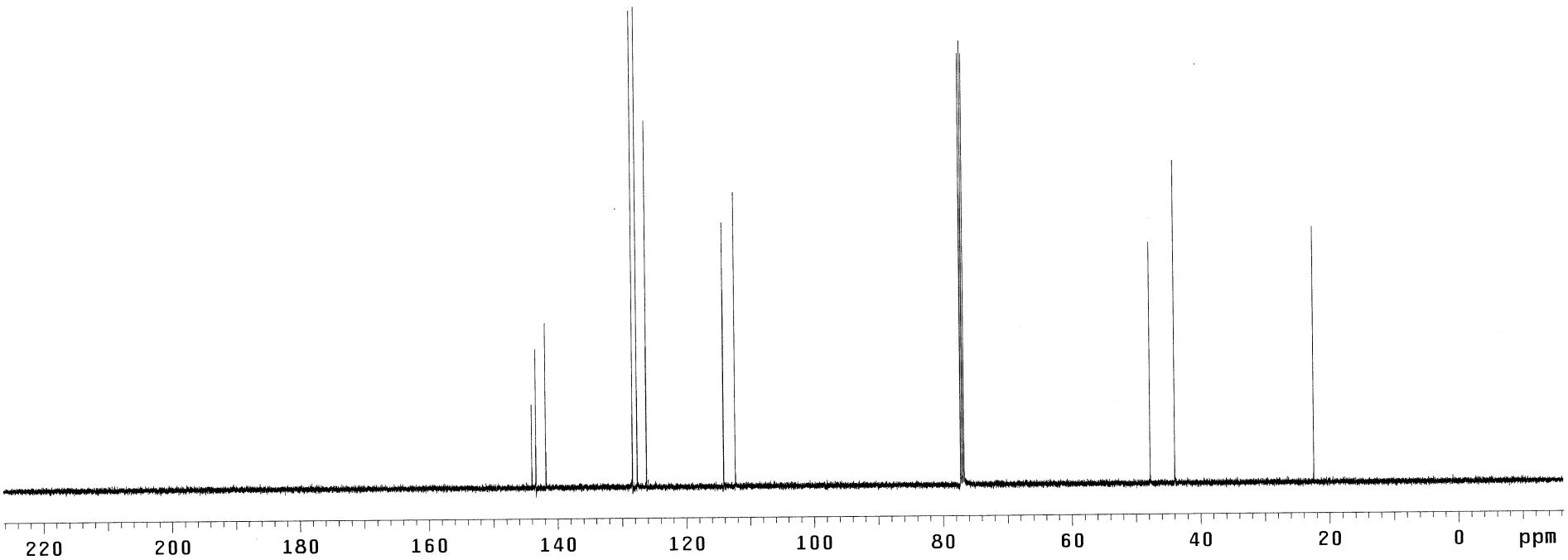


PZ-4-245B-P-C13

exp4 Carbon

SAMPLE		SPECIAL	
date	Apr 18 2010	temp	25.0
solvent	cdcl3	gain	30
file	exp	spin	20
ACQUISITION		hst	0.008
sw	30487.8	pw90	9.600
at	1.300	alfa	10.000
np	79298	FLAGS	
fb	17000	il	n
bs	64	in	n
d1	1.000	dp	y
nt	1000	hs	nn
ct	1000	PROCESSING	
TRANSMITTER		lb	0.50
tn	C13	fn	not used
sfrq	125.709	DISPLAY	
tof	1285.5	sp	-2045.3
tpwr	56	wp	30487.3
pw	4.800	rfl	2045.8
DECOUPLER		rfp	0
dn	H1	rp	76.9
dof	0	lp	-14.2
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	40	sc	0
dmf	11621	vs	28102
		th	9
		ai	cdc ph

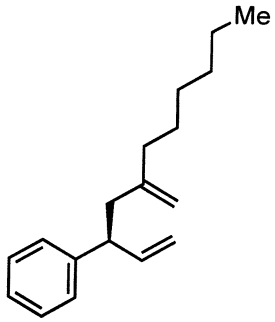


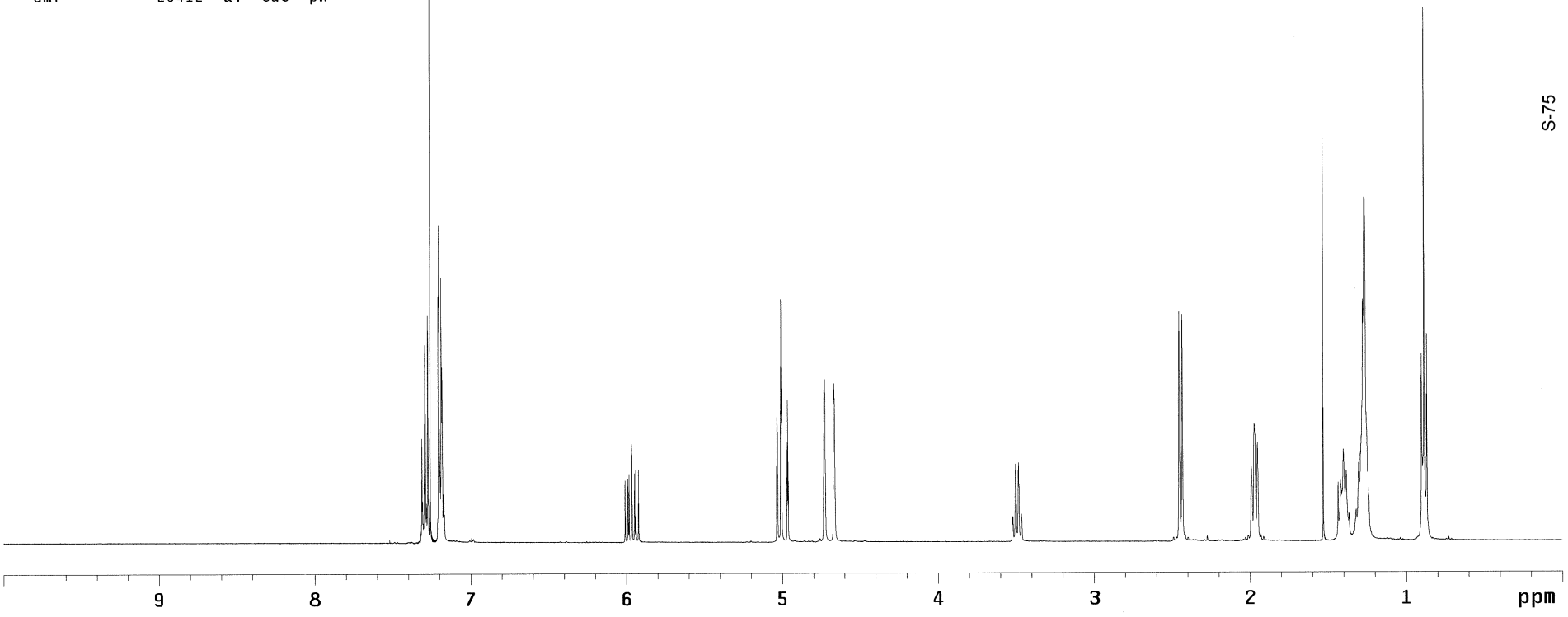


PZ-5-36-P

exp8 Proton

SAMPLE		SPECIAL	
date	Jun 7 2010	temp	25.0
solvent	cdcl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	6410.3	pw90	9.312
at	2.049	alfa	10.000
np	26264	FLAGS	
fb	4000	il	n
bs	4	in	n
ss	2	dp	y
d1	1.000	hs	nn
nt	32	PROCESSING	
ct	32	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-0.2
sfrq	399.769	wp	3997.6
tof	399.8	rfl	3702.4
tpwr	61	rfp	2902.3
pw	4.656	rp	-45.5
DECOUPLER		lp	0
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	200
dpwr	35	th	5
dmf	29412	ai cdc ph	

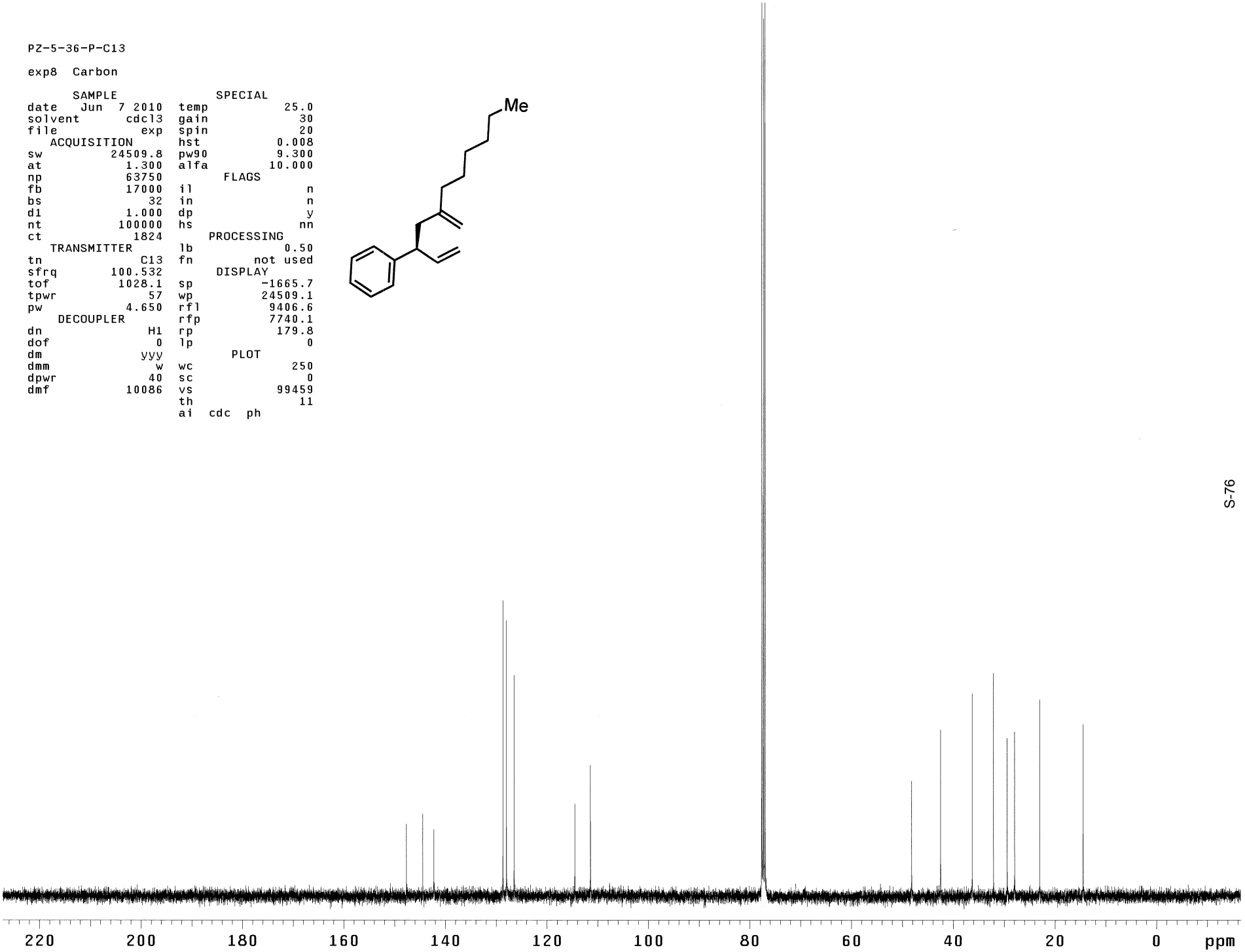




PZ-5-36-P-C13

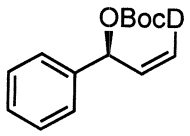
exp8 Carbon

SAMPLE		SPECIAL	
date	Jun 7 2010	temp	25.0
solvent	cdcl3	gain	30
file	exp	spin	20
ACQUISITION		hst	0.008
sw	24509.8	pw90	9.300
at	1.300	alfa	10.000
np	63750	FLAGS	
fb	17000	il	n
bs	32	in	n
d1	1.000	dp	y
nt	100000	hs	nn
ct	1824	PROCESSING	
TRANSMITTER		lb	0.50
tn	C13	fn	not used
sfrq	100.532	DISPLAY	
tof	1028.1	sp	-1665.7
tpwr	57	wp	24509.1
pw	4.650	rfl	9406.6
DECOUPLER		rfp	7740.1
dn	H1	rp	179.8
dof	0	lp	0
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	40	sc	0
dmf	10086	vs	99459
		th	11
		ai cdc ph	

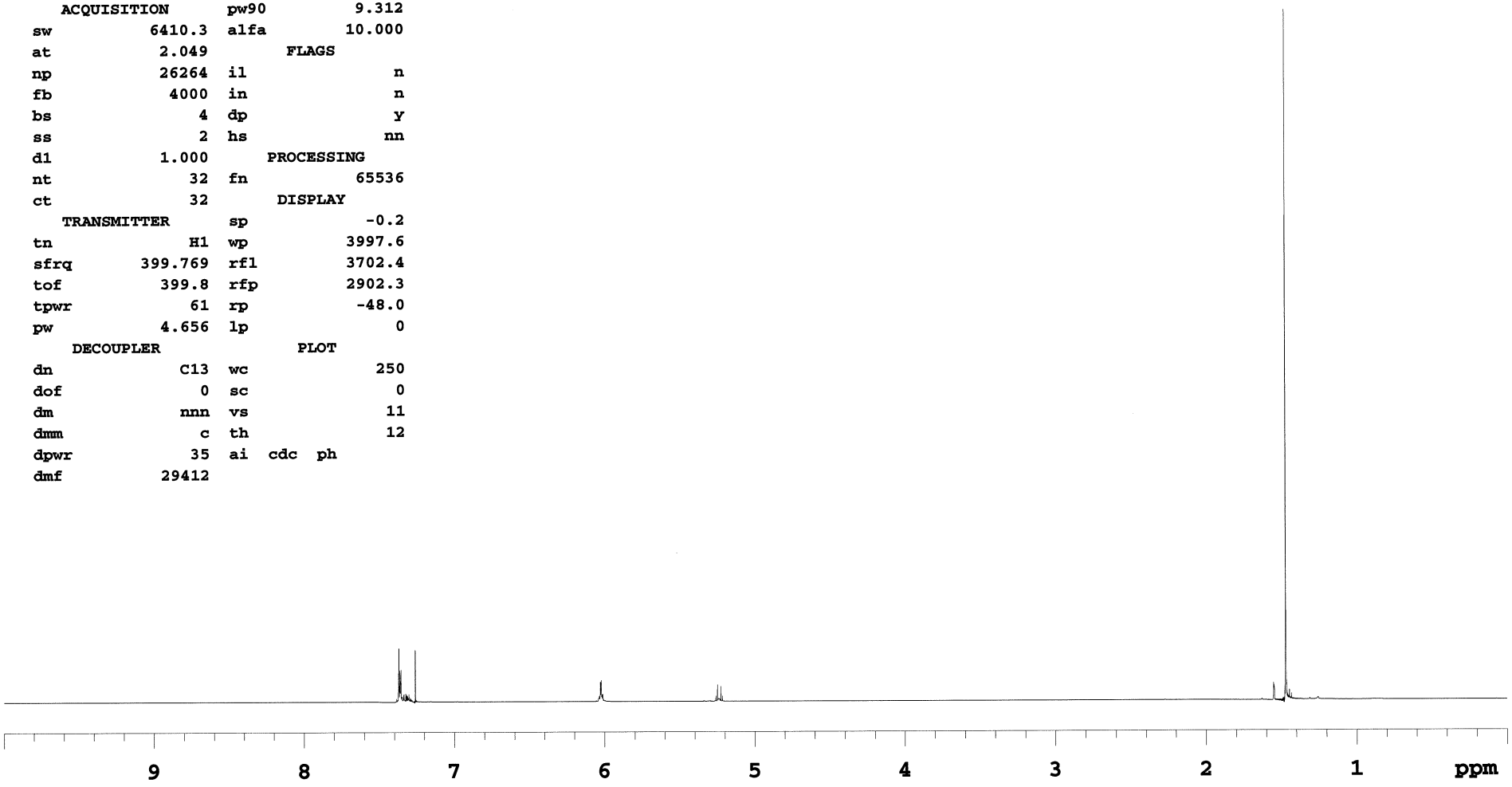


PZ-4-310-P

exp6 Proton



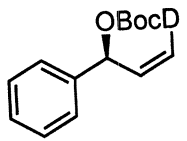
SAMPLE		SPECIAL	
date	Jun 7 2010	temp	25.0
solvent	cdcl3	gain	not used
file	/home/All/jpm~	spin	20
	/PZ/PZ-4-310-P.fid	hst	0.008
ACQUISITION		pw90	9.312
sw	6410.3	alfa	10.000
at	2.049	FLAGS	
np	26264	il	n
fb	4000	in	n
bs	4	dp	y
ss	2	hs	nn
d1	1.000	PROCESSING	
nt	32	fn	65536
ct	32	DISPLAY	
TRANSMITTER		sp	-0.2
tn	H1	wp	3997.6
sfrq	399.769	rfl	3702.4
tof	399.8	rfp	2902.3
tpwr	61	rp	-48.0
pw	4.656	lp	0
DECOUPLER		PLOT	
dn	C13	wc	250
dof	0	sc	0
dm	nnn	vs	11
dmm	c	th	12
dpwr	35	ai cdc ph	
dmf	29412		

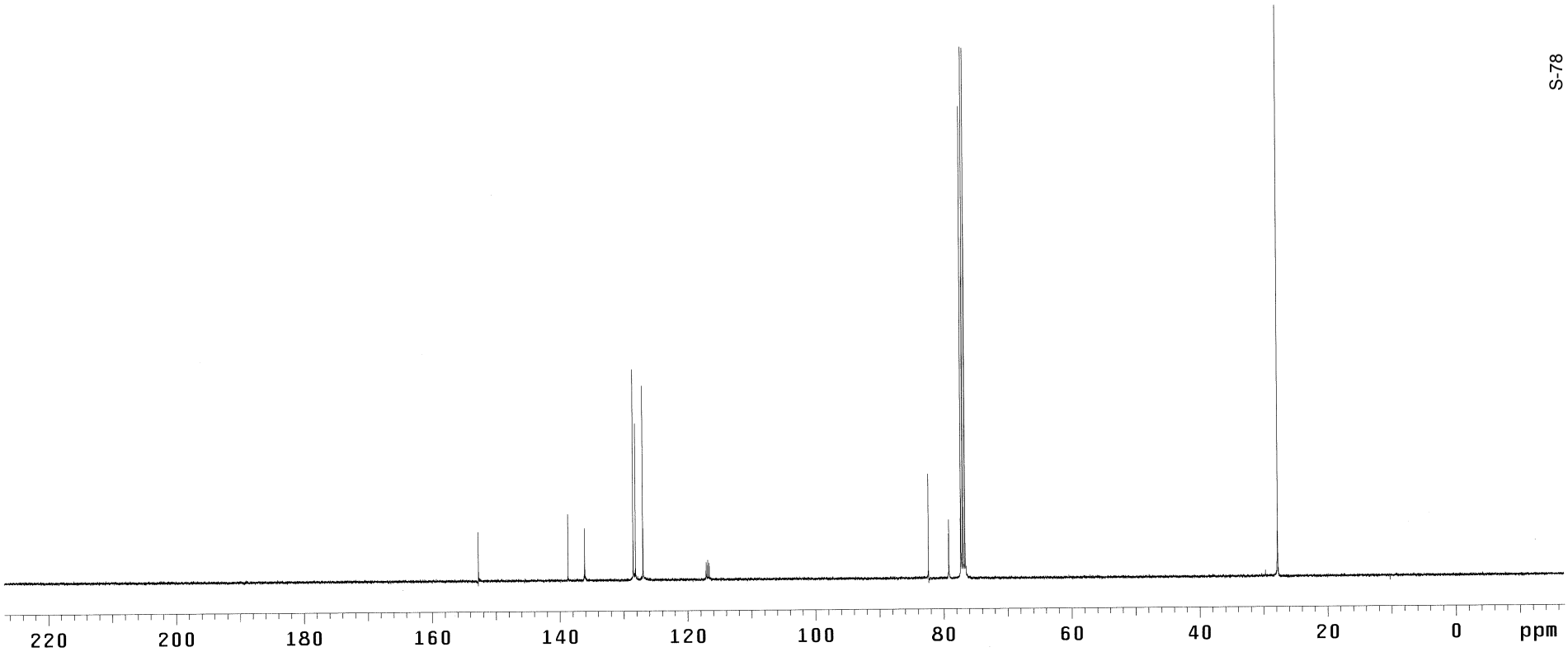


PZ-4-310-P-C13

exp8 Carbon

SAMPLE		SPECIAL	
date	Jun 7 2010	temp	25.0
solvent	cdcl3	gain	30
file	/home/jpm/PZ/~	spin	20
	PZ-4-310-P-C13.fid	hst	0.008
ACQUISITION		pw90	9.300
sw	24509.8	alfa	10.000
at	1.300	FLAGS	
np	63750	il	n
fb	17000	in	n
bs	32	dp	y
d1	1.000	hs	nn
nt	100000	PROCESSING	
ct	15296	lb	0.50
TRANSMITTER		fn	not used
tn	C13	DISPLAY	
sfrq	100.532	sp	-1697.9
tof	1028.1	wp	24509.1
tpwr	57	rfl	9438.8
pw	4.650	rfp	7740.1
DECOUPLER		rp	173.5
dn	H1	lp	0
dof	0	PLOT	
dm	yyy	wc	250
dmm	w	sc	0
dpwr	40	vs	42481
dmf	10086	th	12
		ai	cdc ph

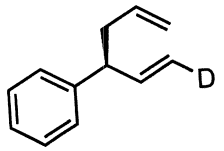


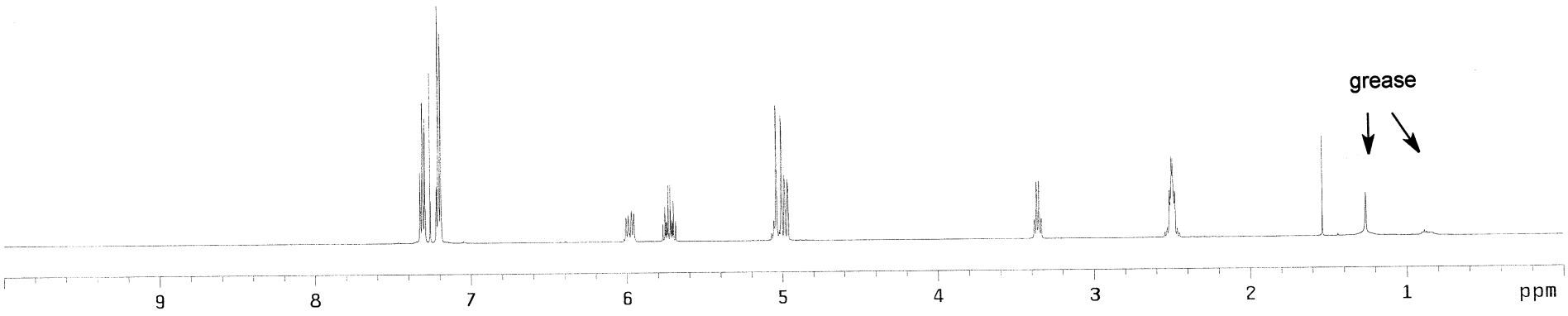


PZ-5-39-P

exp1 Proton

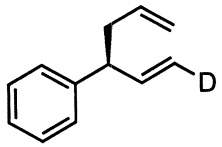
SAMPLE		SPECIAL	
date	Jun 9 2010	temp	not used
solvent	cdcl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	7996.0	pw90	8.000
at	3.000	alfa	6.600
np	47976	FLAGS	
fb	4000	il	n
bs	4	in	n
ss	2	dp	y
d1	1.000	hs	nn
nt	16	PROCESSING	
ct	16	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-0.2
sfrq	499.775	wp	4997.5
tof	499.8	rfl	4640.7
tpwr	60	rfp	3628.3
pw	4.000	rp	122.0
DECOUPLER		lp	-54.4
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	19
dpwr	38	th	12
dmf	32258	ai cdc ph	





PZ-5-17-P-C13

exp3 Carbon



SAMPLE		SPECIAL	
date	Jun 6 2010	temp	25.0
solvent	cdcl3	gain	30
file	/home/All/jpm~	spin	20
/PZ/PZ-5-17-P-C13.~		hst	0.008
	fid	pw90	9.300
ACQUISITION		alfa	10.000
sw	24509.8	FLAGS	
at	1.300	il	n
np	63750	in	n
fb	17000	dp	y
bs	32	hs	nn
d1	1.000	PROCESSING	
nt	100000	lb	0.50
ct	16800	fn	not used
TRANSMITTER		DISPLAY	
tn	C13	sp	-1697.9
sfrq	100.532	wp	24509.1
tof	1028.1	rfl	9438.8
tpwr	57	rfp	7740.1
pw	4.650	rp	170.0
DECOUPLER		lp	0
dn	H1	PLOT	
dof	0	wc	250
dm	yyy	sc	0
dmm	w	vs	44541
dpwr	40	th	1
dmf	10086	ai cdc ph