Supporting Information for:

Endoperoxide Synthesis by Photocatalytic Aerobic [2+2+2] Cycloadditions

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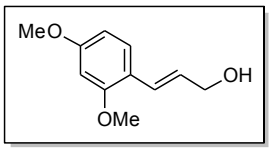
I. General information

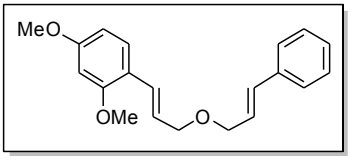
THF and CH_2Cl_2 were purified by elution through alumina as described by Grubbs.[1] $MeNO_2$ was purified by distillation from $MgSO_4$ prior to use. A 200 W (3980 lumens) GE Crystal Clear light bulb was used for all photochemical reactions depicted in Tables 1 and 2. Flash column chromatography was performed with Silicycle 40-63 Å silica (230-400 mesh). 9,10-Dicyanoanthracene (DCA), 5,10,15,20-tetraphenyl-21H,23H-porphine (TPP) and 2,4,6-triphenylpyrylium tetrafluoroborate (TPT) were purchased from Aldrich and used without further purification. $Ru(bpz)_3(PF_6)_2$[2] and $Ru(bpy)_3(PF_6)_2$[3] were prepared as previously described. Diastereomer ratios for all compounds were determined by ^1H NMR analysis of the unpurified reaction mixture. ^1H and ^{13}C NMR data for all previously uncharacterized compounds were obtained using Varian Inova-500 and Varian Unity-500 spectrometers and are referenced to TMS (0.0 ppm), $CDCl_3$ (77.0 ppm), and CD_3CN (118.6 ppm), respectively. IR spectral data were obtained using a Bruker Vector 22 spectrometer (thin film on NaCl). Melting points were obtained using a Mel-Temp II (Laboratory Devices, Inc., USA) melting point apparatus. Mass spectrometry was performed with a Waters (Micromass) AutoSpec®. These facilities are funded by the NSF (CHE-9974839, CHE-9304546) and the University of Wisconsin.

II. Synthesis of cyclization substrates

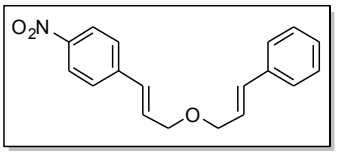
The substrates for entries 1–4, 7, 8, 10, 11, 13, and 15 were prepared as previously described.[3] The substrate for entry 6 was prepared as described by Nair.[4]



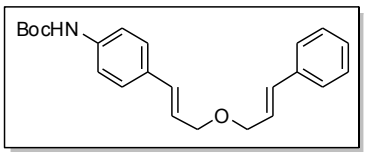
***o,p*-Dimethoxycinnamyl alcohol.** A solution of ethyl 2,4-dimethoxycinnamate (4.39 g, 18.5 mmol) in 37 mL CH_2Cl_2 was placed in a 250 mL round-bottomed flask. The solution was cooled to –78 °C, and a 1.0 M solution of DIBAL-H in hexanes (46.3 mL, 46.3 mmol) was added over 5 minutes. The reaction was then stirred for 2 h at –78 °C. After warming to 0 °C, the reaction was quenched with a saturated aqueous solution of Rochelle's salt. The mixture was stirred and allowed to warm to room temperature overnight. The layers were separated, and the aqueous layer was extracted with three portions of Et_2O. The combined organic fractions were dried over $MgSO_4$ and concentrated by rotary evaporation. The residue was purified by chromatography on SiO_2 (8:1 to 2:1 hexanes:EtOAc) to give 3.39 g (17.4 mmol, 94% yield) of a yellow oil. IR (neat) 3477, 2939, 1110 cm^{-1}. ^1H NMR: (500 MHz, $CDCl_3$) δ 7.35 (d, J = 8.4 Hz, 1H), 6.83 (d, J = 15.9 Hz, 1H), 6.47 (dd, J = 8.4, 2.5 Hz, 1H), 6.44 (d, J = 2.5 Hz, 1H), 6.28 (dt, J = 15.9 Hz, 6.6, 1H), 4.29 (m, 2H), 3.83 (s, 3H), 3.81 (s, 3H), 1.46 (m, 1H). ^{13}C NMR: (125 MHz, $CDCl_3$) δ 160.5, 157.9, 127.8, 127.0, 126.2, 118.7, 104.8, 98.4, 64.4, 55.4, 55.3. HRMS (EI) calculated for $[C_{11}H_{14}O_3]^+$ requires m/z 194.0938, found m/z 194.0939.



***(E)-o,p*-Dimethoxycinnamyl *(E)*-cinnamyl ether** (Table 2, Entry 5). A 50 mL round-bottomed flask was charged with 60% NaH (575 mg, 14.4 mmol) and 5 mL THF. A solution of 2,4-dimethoxycinnamyl alcohol (2.00 g, 10.3 mmol) in 7.5 mL THF was added slowly. The reaction was stirred at room temperature for 30 min and then cooled to 0 °C. A solution of cinnamyl bromide (2.42 g, 12.3 mmol) in 8 mL of THF was added, and the reaction was warmed to room temperature. After 24 h, the reaction was quenched with water and the aqueous layer was extracted with two portions of Et_2O. The combined organic fractions were dried over $MgSO_4$ and concentrated via rotary evaporation. Purification by chromatography on SiO_2 (10:1 to 4:1 hexanes:EtOAc) afforded 2.91 g (9.40 mmol, 91% yield) of a yellow oil. IR (neat) 3003, 2938, 1209 cm^{-1}. ^1H NMR: (500 MHz, $CDCl_3$) δ 7.39–7.20 (m, 6H), 6.86 (d, J = 16.0 Hz, 1H), 6.62 (d, J = 16.0 Hz, 1H), 6.46 (dd, J = 8.6, 2.5 Hz, 1H), 6.42 (d, J = 2.5 Hz, 1H), 6.32 (dt, J = 16.0, 6.2 Hz, 1H), 6.23 (dt, J = 16.0, 6.2 Hz, 1H), 4.18 (d, J = 6.2 Hz, 4H), 3.8 (s, 3H), 3.78 (s, 3H). ^{13}C NMR: (125 MHz, $CDCl_3$) δ 160.4, 157.8, 136.7, 132.3, 128.4, 127.7, 127.6, 127.5, 126.4, 126.2, 124.2, 118.7, 104.7, 98.3, 71.5, 70.4, 55.3, 55.2;. HRMS (EI) calculated for $[C_{20}H_{22}O_3]^+$ requires m/z 310.1564, found m/z 310.1549.

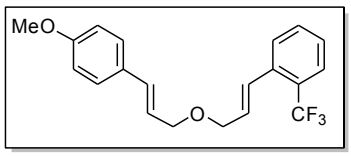


***(E)-p*-Nitrocinnamyl *(E)*-cinnamyl ether.** A solution of 4-nitrocinnamyl alcohol (1.20 g, 6.70 mmol) and cinnamyl bromide (1.98 g, 10.0 mmol) in 27 mL DMF was placed in a 100 mL round-bottomed flask. The solution was cooled to 0 °C and 60% NaH (402 mg, 10.0 mmol) was added slowly. The mixture was allowed to warm to room temperature and stirred for 7 h. The reaction was quenched by slowly adding a saturated aqueous solution of NH_4Cl and the aqueous layer was extracted with two portions of Et_2O. The combined organic fractions were dried over $MgSO_4$ and concentrated via rotary evaporation. Purification by chromatography on SiO_2 (20:1 to 4:1 hexanes:EtOAc) gave 885 mg (3.00 mmol, 45% yield) of a red oil. IR (neat) 3080, 1343, 1110 cm^{-1}. ^1H NMR: (500 MHz, $CDCl_3$) δ 8.11 (d, J = 9.3 Hz, 2H), 7.44 (d, J = 9.3 Hz, 2H), 7.40–7.20 (m, 5H), 6.68 (d, J = 16.0 Hz, 1H), 6.64 (d, J = 16.0 Hz, 1H), 6.45 (dt, J = 16.0, 5.5 Hz, 1H), 6.31 (dt, J = 16.0, 5.5 Hz, 1H), 4.22–4.19 (m, 4H). ^{13}C NMR: (125 MHz, $CDCl_3$) δ 146.6, 143.0, 136.4, 132.5, 131.1, 129.2, 128.4, 127.6, 126.7, 126.3, 125.5, 123.7, 71.0, 69.8. HRMS (EI) calculated for $[C_{18}H_{17}NO_3]^+$ requires m/z 195.1208, found m/z 195.1215.

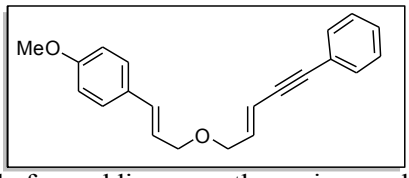


***(E)-p-N*-Boc-Aminocinnamyl *(E)*-cinnamyl ether** (Table 2, Entry 9). A solution of $Cu(acac)_2$ (213 mg, 0.81 mmol) in 10 mL EtOH was placed in a 100 mL round-bottomed flask. $NaBH_4$ (300 mg, 7.93 mmol) was added, and the mixture was stirred for 10 min. The flask was then placed

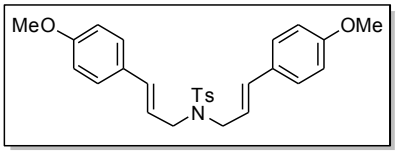
in a water bath and vigorously stirred while a suspension of *(E)-p*-nitrocinnamyl *(E)*-cinnamyl ether (1.20 g, 4.06 mmol) in 15 mL of EtOH was added dropwise. After 1 h, additional $NaBH_4$ (161 mg, 4.25 mmol) was added, and the mixture was stirred for 12 h before being quenched with water. The aqueous phase was extracted with two portions of EtOAc, and the combined organic fractions were dried over $MgSO_4$ and concentrated via rotary evaporation. Purification by flash column chromatography (20:1 to 1:1 hexanes:EtOAc) afforded the free amine. This material was dissolved in 20 mL 1,4-dioxane in a 100 mL round-bottomed flask. Triethylamine (0.54 mL, 3.90 mmol) and Boc_2O (851 mg, 3.90 mmol) were added, and the reaction was stirred at room temperature for 18 h. The solution was concentrated via rotary evaporation, and the residue was eluted through a short plug of SiO_2 with Et_2O. The eluent was concentrated by rotary evaporation, and the residue was purified by chromatography on SiO_2 (20:1 to 4:1 hexanes:EtOAc) to afford 325 mg (0.912 mmol, 22% yield) of a yellow solid (mp = 82–84 °C). IR (neat) 3329, 1727, 1522 cm^{-1}. ^1H NMR: (500 MHz, $CDCl_3$) δ 7.40–7.22 (m, 9H), 6.64 (d, J = 15.9 Hz, 1H), 6.57 (d, J = 15.9 Hz, 1H), 6.51 (s, 1H), 6.32 (dt, J = 15.9, 5.9 Hz, 1H), 6.23 (dt, J = 15.9, 5.9 Hz, 1H), 4.21–4.18 (m, 4H), 1.52 (s, 9H). ^{13}C NMR: (125 MHz, $CDCl_3$) δ 152.6, 137.8, 136.7, 132.5, 132.2, 131.6, 128.5, 127.6, 127.1, 126.5, 126.0, 124.5, 118.4, 80.6, 70.8, 70.7, 28.3. HRMS (EI) calculated for $[C_{23}H_{27}O_3N+Na]^+$ requires m/z 388.1884, found m/z 388.1880.



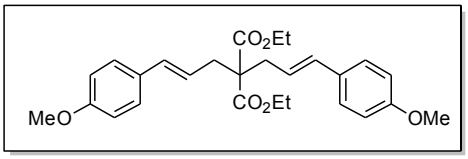
***(E)-p*-Methoxycinnamyl *(E)-o*-trifluoromethylcinnamyl ether** (Table 2, Entry 12). A solution of 2-trifluoromethyl cinnamyl alcohol[5] (1.69 g, 8.36 mmol) in 20 mL THF was placed in a 250 mL round-bottomed flask. 60% NaH (402 mg, 10.1 mmol) was added, and the reaction was stirred for 1 h. The flask was then cooled to 0 °C, and a solution of *p*-methoxycinnamyl bromide (2.85 g, 12.5 mmol) in 15 mL of THF was added slowly. The solution was allowed to warm to room temperature and stirred for 48 h. The reaction was quenched with water and the aqueous layer was extracted with two portions of Et_2O. The combined organic fractions were washed with brine, dried over $MgSO_4$, and concentrated via rotary evaporation. Purification by chromatography on SiO_2 (20:1 to 10:1 hexanes:EtOAc) afforded 1.45 g (4.2 mmol, 50% yield) of a colorless oil. IR (neat) 3004, 1514, 1315 cm^{-1}. ^1H NMR: (500 MHz, $CDCl_3$) δ 7.62 (m, 2H), 7.47 (t, J = 7.5 Hz, 1H), 7.34–7.20 (m, 3H), 7.02 (d, J = 15.7 Hz, 1H), 6.85 (d, J = 8.6 Hz, 2H), 6.58 (d, J = 15.7 Hz, 1H), 6.28 (dt, J = 15.7, 5.9 Hz, 1H), 6.18 (dt, J = 15.7, 5.9 Hz, 1H), 4.22 (d, J = 5.9Hz, 2H), 4.18 (d, J = 5.9 Hz, 2H), 3.78 (s, 3H). ^{13}C NMR: (125 MHz, $CDCl_3$) δ 159.3, 135.9, 132.5, 131.8, 130.6, 129.4, 128.2, 127.7, 127.5, 127.3, 127.0, 125.7 (q, J = 5.8 Hz), 124.3 (q, J= 273.9 Hz), 123.5, 114.0, 70.9, 70.2, 55.2. HRMS (EI) calculated for $[C_{20}H_{19}O_2F_3]^+$ requires m/z 348.1332, found m/z 348.1346.



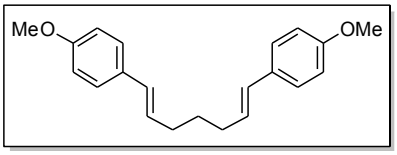
***(E)-p*-Methoxycinnamyl *(E)*-5-phenylpent-2-en-4-yn-1-yl ether** (Table 2, Entry 14). A suspension of 60% NaH (290 mg, 7.20 mmol) in 20 mL THF was placed in a 100 mL round-bottomed flask. The flask was cooled to 0 °C, and (*E*)-5-phenylpent-2-en-4-yn-1-ol[6] (1.14 g, 7.20 mmol) was added. The mixture was stirred for 30 min before adding *p*-methoxycinnamyl bromide (1.63 g, 7.20 mmol). The reaction was allowed to warm to room temperature. After stirring for 12 h, the reaction was cooled to 0 °C and quenched by adding a saturated aqueous solution of NH_4Cl. The aqueous layer was extracted with two portions of EtOAc. The combined organic fractions were washed with brine, dried over $MgSO_4$, and concentrated via rotary evaporation. Purification by chromatography on SiO_2 (20:1 to 8:1 hexanes:EtOAc) afforded 1.65 g (5.42 mmol, 75% yield) of the title product as a yellow solid (mp = 49–51 °C). IR (neat) 2935, 2198, 1513 cm^{-1}. ^1H NMR: (500 MHz, $CDCl_3$) δ 7.45–7.30 (m, 7H), 6.86 (d, J = 9.0 Hz, 2H), 6.57 (d, J = 16.1 Hz, 1H), 6.3 (dt, J = 16.01, 5.6 Hz, 1H), 6.16 (dt, J = 16.0, 6.0 Hz, 1H), 5.98 (dt, J = 16.0, 1.7 Hz, 1H), 4.16 (dd, J = 6.0, 1.3 Hz, 2H), 4.13 (dd, J = 5.6, 1.7 Hz, 2H), 3.81 (s, 3H). ^{13}C NMR: (125 MHz, $CDCl_3$) δ 159.3, 139.5, 132.4, 131.5, 129.4, 128.3, 128.2, 127.7, 123.4, 123.2, 114.0, 111.7, 90.0, 87.4, 71.1, 69.7, 55.3. HRMS (EI) calculated for $[C_{21}H_{20}O_2]^+$ requires m/z 304.1458, found m/z 304.1469.



(*E*,*E*)-*N*,*N*-Bis-(*p*-methoxycinnamyl) toluenesulfonamide (Table 2, Entry 16). A suspension of 60% NaH (568 mg, 14.2 mmol) in 30 mL DMF was placed in a 250 mL round-bottomed flask. The mixture was cooled to 0 °C and a solution of *N*-*p*-methoxycinnamyl *p*-toluenesulfonamide[7] (1.5 g, 4.73 mmol) in 30 mL of DMF was added dropwise over 10 min. The reaction was warmed to room temperature and stirred for 30 min before a solution of *p*-methoxycinnamyl bromide (2.69 g, 11.8 mmol) in 35 mL of DMF was added. After 24 h, the reaction was carefully quenched with a saturated aqueous solution of NH_4Cl and the aqueous layer was extracted with two portions of Et_2O. The combined organic fractions were dried over $MgSO_4$ and concentrated via rotary evaporation. Purification by flash column chromatography on SiO_2 (20:1 to 2:1 hexanes:EtOAc) afforded 1.29 g (2.8 mmol, 59% yield) of the title product as a yellow solid (mp = 92–94 °C). IR (neat) 3033, 1512, 1251 cm^{-1}. ^1H NMR: (500 MHz, $CDCl_3$) δ 7.75 (d, J = 7.8 Hz, 2H), 7.30 (d, J = 7.8 Hz, 2H), 7.18 (d, J = 8.9 Hz, 4H), 6.82 (d, J = 8.9 Hz, 4H), 6.36 (d, J = 16.0 Hz, 2H), 5.82 (dt, J = 16.0, 6.7 Hz, 2H), 3.98 (d, J = 6.7 Hz, 4H), 3.80 (s, 6H), 2.43 (s, 3H). ^{13}C NMR: 159.4, 143.2, 137.6, 133.5, 129.7, 129.0, 127.6, 127.3, 121.6, 114.0, 55.3, 49.0, 21.5. HRMS (EI) calculated for $[C_{27}H_{29}NO_4S+Na]^+$ requires m/z 486.1710, found m/z 486.1700.



(*E*,*E*)-Diethyl 2,2-bis(*p*-methoxycinnamyl)malonate (Table 2, Entry 17). Diethyl malonate (1.00 g, 6.24 mmol) was added to a suspension of 60% NaH (929 mg, 23.2 mmol) in 16 mL THF. The mixture was stirred for 1 h then cooled to 0 °C. A solution of *p*-methoxycinnamyl bromide (3.54 g, 15.6 mmol) in 15 mL of DMF was added. After 12 h, the reaction was quenched with water and the aqueous layer was extracted with two portions of Et_2O. The combined organic fractions were dried over $MgSO_4$ and concentrated via rotary evaporation. Purification by chromatography on SiO_2 (15:1 to 10:1 hexanes:EtOAc) afforded 2.45 g (5.41 mmol, 87% yield) of a white solid (mp = 74–75 °C). IR (neat) 1729, 1512, 1249 cm^{-1}. ^1H NMR: (500 MHz, $CDCl_3$) δ 7.32 (d, J = 9.1 Hz, 4H), 6.83 (d, J = 9.1 Hz, 4H), 6.39 (d, J = 15.7 Hz, 2H), 5.93 (dt, J = 15.9, 7.6 Hz, 2H), 4.20 (q, J = 7.2 Hz, 4H), 3.80 (s, 6H), 2.81 (dd, J = 7.6, 1 Hz, 4H), 1.24 (t, J = 7.2 Hz, 6H). ^{13}C NMR: (125 MHz, $CDCl_3$) δ 170.9, 159.1, 133.4, 130.0, 127.3, 121.7, 113.9, 61.3, 58.1, 55.3, 36.5, 14.2. HRMS (EI) calculated for $[C_{27}H_{32}O_6+H]^+$ requires m/z 453.2272, found m/z 453.2276.

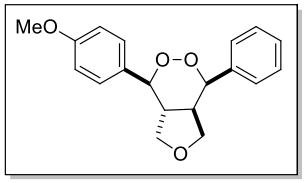


(*E*,*E*)-1,7-bis(4-methoxyphenyl)hepta-1,6-diene (Table 2, Entry 18). An aqueous solution of glutaraldehyde (50% w/w, 1.00 g, 4.99 mmol) and a solution of 4-methoxybenzyl triphenylphosphonium bromide (5.80 g, 12.5 mmol) in 20 mL THF were placed in a 100 mL round-bottomed flask and cooled to 0 °C. KO*t*-Bu (1.68 g, 15.0 mmol) was added slowly over 5 min. The resulting orange suspension was stirred at room temperature for 18 h before being diluted with water and extracted with three portions of Et_2O. The combined organic fractions were dried over $MgSO_4$ and concentrated via rotary evaporation. Purification by chromatography on SiO_2 (20:1 to 10:1 hexanes:EtOAc) afforded a mixture of isomeric bis(styrene)s. This material was dissolved in 50 mL of benzene in 100 mL round-bottomed flask, to which was added thiophenol (0.022 mL, 0.211 mmol) and AIBN (34.7 mg, 0.211 mmol). The solution was heated at reflux for 24 h. Upon cooling to room temperature, the solution was diluted with hexanes and washed with two portions of saturated aqueous $NaHCO_3$ and one portion of brine. The organic layer was then dried over $MgSO_4$ and concentrated by rotary evaporation. Purification by chromatography on SiO_2 (gradient, 25:1 to 20:1 hexanes/EtOAc) gave 207 mg (0.670 mmol, 13% yield) of the title product as a white solid (mp = 91–93 °C). IR (neat) 3004, 2916, 1250 cm^{-1}. ^1H NMR: (500 MHz, $CDCl_3$) δ 7.24 (d, J = 8.9 Hz, 4H), 6.80 (d, J = 8.9 Hz, 4H), 6.32 (d, J = 15.9 Hz, 2H), 6.06 (dt, J = 15.9, 7 Hz, 2H), 3.72 (s, 6H), 2.21 (q, J = 7 Hz, 4H), 1.60 (p, J = 7.3 Hz,

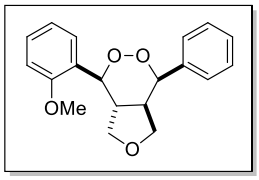
2H). ^{13}C NMR: (125 MHz, $CDCl_3$) δ 158.6, 130.7, 129.2, 128.8, 127.0, 113.9, 55.2, 32.9, 29.0. HRMS (EI) calculated for $[C_{21}H_{24}O_2]^+$ requires m/z 308.1672, found m/z 308.1668.

III. Photocycloadditions

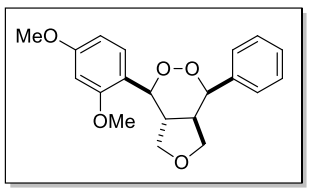
General Procedure: An oven-dried 135 mL glass pressure vessel containing a magnetic stirbar was charged with bis(alkene) substrate, $Ru(bpz)_3(PF_6)_2$ (0.005 or 0.02 equiv), and $MeNO_2$. The vessel was fitted with a regulator and pressurized to 60 psi with O_2 gas. The reaction was cooled in a 5 °C water bath and irradiated with a 200 W incandescent bulb. After consumption of the substrate, the reaction mixture was eluted through a short pad of SiO_2 using EtOAc and CH_2Cl_2. The solvent was removed by rotary evaporation, and purification of the residue by chromatography on SiO_2 (8:1 to 2:1 hexanes:EtOAc) afforded the [2+2+2] cycloadduct.



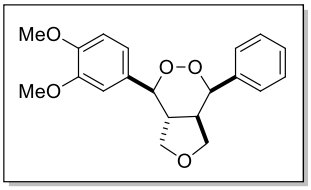
(Table 2, entry 1). Prepared according to the General Procedure using 100 mg (0.357 mmol) of bis(alkene), 1.5 mg (1.8 μmol) of $Ru(bpz)_3(PF_6)_2$, 17.8 mL of $MeNO_2$ and an irradiation time of 30 min. Purification by flash column chromatography afforded the cycloadduct as an oil. Experiment 1: 100.6 mg (0.322 mmol, 90% yield, dr = 6:1), Experiment 2: 104.0 mg (0.333 mmol, 94% yield, dr = 6:1). IR (neat) 1253, 732, 702 cm^{-1}. ^{1}H NMR: (500 MHz) δ 7.50 (d, J = 7.8 Hz, 2H), 7.39-7.35 (m, 2H), 7.33-7.30 (m, 3H), 6.91 (d, J = 8.8 Hz, 2H), 5.56 (d, J = 5.8 Hz, 1H), 5.04 (d, J = 10 Hz, 1H), 4.17 (t, J = 7.4 Hz, 1H), 3.82–3.77 (m, 4H), 3.43 (dd, J = 10.8, 8.0 Hz, 1H), 3.25 (dd, J = 11.4, 7.6 Hz, 1H), 2.88-2.79 (m, 1H), 2.66-2.57 (m, 1H). ^{13}C NMR: (125 MHz, CD_3CN) δ 161.8, 139.1, 130.72, 130.66, 129.5, 128.9, 128.7, 115.4, 88.5, 84.4, 69.8, 68.7, 56.4, 47.7, 42.3;. HRMS (EI) calculated for $[C_{19}H_{20}O_4]^+$ requires m/z 312.1456, found m/z 312.1457.



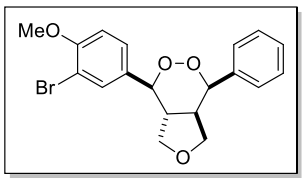
(Table 2, entry 3). Prepared according to the General Procedure using 100 mg (0.357 mmol) of bis(alkene), 6.2 mg (7.0 μmol) of $Ru(bpz)_3(PF_6)_2$, 17.8 mL of $MeNO_2$ and an irradiation time of 24 h. Purification by flash column chromatography afforded the cycloadduct as an oil. Experiment 1: 52.0 mg (0.167 mmol, 47% yield, dr = 10:1), Experiment 2: 47.1 mg (0.151 mmol, 42% yield, dr = 10:1). IR (neat) 756, 735, 702 cm^{-1}. ^{1}H NMR: (500 MHz) δ 7.50–7.28 (m, 7H), 7.00 (t, J = 7.9 Hz, 1H), 6.90 (d, J = 8.3 Hz, 1H), 5.62 (d, J = 6.3 Hz, 1H), 5.54 (d, J = 9.8 Hz, 1H), 4.10 (t, J = 7.3 Hz, 1H), 3.85–3.76 (m, 4H), 3.56 (dd, J = 10.9, 8.1 Hz, 1H), 3.13 (dd, J = 11.2, 7.7 Hz, 1H), 2.92 (m, 1H), 2.64 (m, 1H). ^{13}C NMR: (125 MHz, $CDCl_3$) δ 156.7, 136.7, 129.9, 128.3, 127.8, 127.3, 127.0, 120.8, 120.8, 110.5, 83.1, 81.7, 68.72, 68.69, 55.3, 46.0, 41.8;. HRMS (EI) calculated for $[C_{19}H_{20}O_4]^+$ requires m/z 312.1456, found m/z 312.1437.



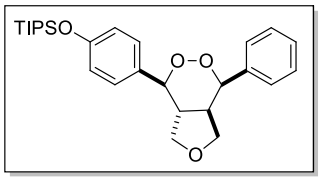
(Table 2, entry 5). Prepared according to the General Procedure using 100 mg (0.322 mmol) of bis(alkene), 1.4 mg (1.6 μmol) of $Ru(bpz)_3(PF_6)_2$, 16 mL of $MeNO_2$ and an irradiation time of 2 h. Purification by flash column chromatography afforded the cycloadduct as an oil. Experiment 1: 75.4 mg (0.220 mmol, 68% yield, dr = 4:1), Experiment 2: 71.7 mg (0.209 mmol, 65% yield, dr = 4:1). IR (neat) 1210, 735, 702 cm^{-1}. ^{1}H NMR: (500 MHz, $CDCl_3$) δ 7.49 (d, J = 7.2 Hz, 2H), 7.40–7.28 (m, 4H), 6.51 (dd, J = 8.5, 2.4 Hz, 1H), 6.46 (d, J = 2.4 Hz, 1H), 5.58 (d, J = 6 Hz, 1H), 5.48 (d, J = 9.7 Hz, 1H), 4.14 (t, J = 7.4 Hz, 1H), 3.86–3.81 (m, 7H), 3.48 (dd, J = 10.8, 8.1 Hz, 1H), 3.20 (dd, J = 11.3, 7.6 Hz, 1H), 2.92-2.83 (m, 1H), 2.71-2.61 (m, 1H). ^{13}C NMR: (125 MHz, $CDCl_3$) δ 161.5, 158.5, 137.0, 128.6, 128.1, 127.6, 127.1, 118.2, 104.4, 98.5, 83.1, 81.4, 68.8, 68.4, 55.43, 55.35, 46.4, 40.9. HRMS (EI) calculated for $[C_{20}H_{22}O_5]^+$ requires m/z 342.1462, found m/z 342.1444.



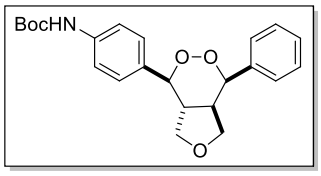
(Table 2, entry 6). Prepared according to the General Procedure using 100 mg (0.322 mmol) of bis(alkene), 1.4 mg (1.6 μmol) of $Ru(bpz)_3(PF_6)_2$, 16 mL of $MeNO_2$ and an irradiation time of 1 h. Purification by flash column chromatography afforded the cycloadduct as an oil. Experiment 1: 100.9 mg (0.295 mmol, 92% yield, dr = 7:1), Experiment 2: 99.5 mg (0.291 mmol, 90% yield, dr = 7:1). IR (neat) 1261, 731, 702 cm^{-1}. ^1H NMR: (500 MHz) δ 7.50 (d, J = 7.5 Hz, 2H), 7.39–7.29 (m, 3H), 6.96–6.91 (m, 2H), 6.87 (d, J = 8.2 Hz, 1H), 5.58 (d, J = 5.8Hz, 1H), 5.05 (d, J = 9.9 Hz, 1H), 4.18 (t, J = 7.3 Hz, 1H), 3.90 (s, 3H), 3.88 (s, 3H), 3.83 (t, J = 7.8 Hz, 1H), 3.46 (dd, J = 10.8, 7.9 Hz, 1H), 3.26 (dd, J = 11.3, 7.6 Hz, 1H), 2.85 (m, 1H), δ 2.63 (m, 1H). ^{13}C NMR: (125 MHz, $CDCl_3$) δ 149.9, 149.2, 137.0, 129.3, 128.2, 127.7, 127.1, 120.0, 111.1, 111.0, 87.6, 83.1, 68.9, 68.2, 55.91, 55.89, 46.4, 41.1. HRMS (EI) calculated for $[C_{20}H_{22}O_5]^+$ requires m/z 342.1462, found m/z 342.1472.



(Table 2, entry 7). Prepared according to the General Procedure using 100 mg (0.279 mmol) of bis(alkene), 4.8 mg (5.6 μmol) of $Ru(bpz)_3(PF_6)_2$, 14 mL of $MeNO_2$ and an irradiation time of 12 h. Purification by flash column chromatography afforded the cycloadduct as an oil. Experiment 1: 70.5 mg (0.196 mmol, 65% yield, dr = 8:1), Experiment 2: 69.5 mg (0.194 mmol, 64% yield, dr = 8:1). IR (neat) 1262, 732, 702 cm^{-1}. ^1H NMR: (500 MHz) δ 7.57 (d, J = 2.2 Hz, 1H), 7.50 (d, J = 7.5 Hz, 2H), 7.39 (t, J = 7.6 Hz, 2H), 7.32 (m, 2H), 6.91 (d, J = 8.47, 1H), 5.59 (d, J = 5.9 Hz, 1H), 5.01 (d, J = 10.1 Hz, 1H), 4.17 (t, J = 7.3 Hz, 1H), 3.91 (s, 3H), 3.82 (t, J = 7.7 Hz, 1H), 3.46 (dd, J = 10.7, 7.9 Hz, 1H), 3.24 (dd, J = 11.4, 7.7 Hz, 1H), 2.85 (m, 1H), 2.59 (m, 1H). ^{13}C NMR: (125 MHz, $CDCl_3$) δ 13C NMR: (125.7 MHz, CDCl3) δ 156.6, 136.7, 132.5, 130.6, 128.3, 127.84, 127.80, 127.0, 112.0, 111.9, 86.6, 83.2, 68.9, 68.0, 56.3, 46.3, 41.4. HRMS (EI) calculated for $[C_{19}H_{19}O_4Br]^+$ requires m/z 390.0462, found m/z 390.0461.

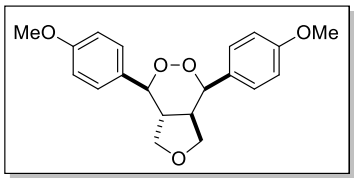


(Table 2, entry 8). Prepared according to the General Procedure using 100 mg (0.237 mmol) of bis(alkene), 1.0 mg (1.2 μmol) of $Ru(bpz)_3(PF_6)_2$, 11.8 mL of $MeNO_2$ and an irradiation time of 2 h. Purification by flash column chromatography afforded the cycloadduct as an oil. Experiment 1: 77.1 mg (0.170 mmol, 72% yield, dr = 10:1), Experiment 2: 82.8 mg (0.182 mmol, 77% yield, dr = 10:1). IR (neat) 737, 701, 684 cm^{-1}. ^1H NMR: (500 MHz) δ 7.50 (d, J = 7.5 Hz, 2H), 7.39–7.28 (m, 3H), 7.23 (d, J = 8.6 Hz, 2H), 6.88 (d, J = 8.6 Hz, 2H), 5.55 (d, J = 6.0 Hz, 1H), 5.02 (d, J = 9.8 Hz, 1H), 4.16 (t, J = 7.3 Hz, 1H), 3.76 (t, J = 7.8 Hz, 1H), 3.43 (dd, J = 10.8, 8.0 Hz, 1H), 3.24 (dd, J = 11.3, 7. 6 Hz, 1H), 2.82 (m, 1H), 2.59 (m, 1H), 1.26 (septet, J = 7.5 Hz, 3H), 1.1 (d, J = 7.5 Hz, 18H). ^{13}C NMR: (125 MHz, $CDCl_3$) δ 157.0, 137.1, 129.4, 128.9, 128.2, 127.6, 127.1, 120.1, 87.5, 83.2, 68.9, 68.1, 46.5, 41.4, 17.9, 12.6. HRMS (EI) calculated for $[C_{27}H_{38}O_4Si]^+$ requires m/z 454.2534, found m/z 454.2549.

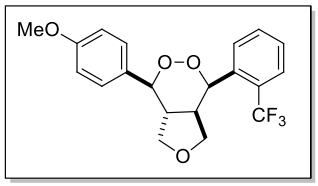


(Table 2, entry 9). Prepared according to the General Procedure using 100 mg (0.281 mmol) of bis(alkene), 1.2 mg (1.4 μmol) of $Ru(bpz)_3(PF_6)_2$, 14 mL of $MeNO_2$ and an irradiation time of 30 min. Purification by flash column chromatography afforded the cycloadduct as an oil. Experiment 1: 103.7 mg (0.267 mmol, 95% yield, dr = 6:1), Experiment 2: 104.6 mg (0.269 mmol, 96% yield, dr = 6:1). IR (neat) 3333, 2876, 2885 cm^{-1}. ^1H NMR: (500 MHz) δ 7.48 (d, J = 7.5 Hz, 2H), 7.40-7.35 (m, 4H), 7.39-7.28 (m, 3H), 6.57 (s, 1H), 5.58 (d, J = 5.9 Hz, 1H), 5.04 (d, J = 9.8 Hz, 1H), 4.17 (t, J = 7.3 Hz, 1H), 3.79 (t, J = 7.9 Hz, 1H), 3.45 (dd, J = 10.7, 8.0 Hz, 1H), 3.24 (dd, J = 11.4, 7.7 Hz, 1H), 2.89-2.80 (m, 1H), 2.66-2.56 (m, 1H), 1.51 (s, 9H). ^{13}C NMR: (125 MHz, $CDCl_3$) δ 152.5, 139.4, 136.9, 131.4, 128.33, 128.25, 127.7, 127.1, 118.6, 87.3, 83.2,

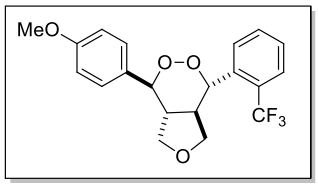
80.8, 68.9, 68.1, 46.4, 41.3, 28.3. HRMS (EI) calculated for $[C_{23}H_{27}O_5N+Na]^+$ requires m/z 420.1782, found m/z 420.1790.



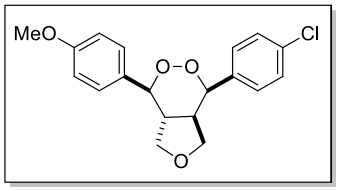
(Table 2, entry 11). Prepared according to the General Procedure using 100 mg (0.322 mmol) of bis(alkene), 1.4 mg (1.6 μmol) of $Ru(bpz)_3(PF_6)_2$, 16.1 mL of $MeNO_2$ and an irradiation time of 30 min. Purification by flash column chromatography afforded the cycloadduct as an oil. Experiment 1: 83.0 mg (0.242 mmol, 75% yield, dr >10:1), Experiment 2: 82.1 mg (0.239 mmol, 74% yield, dr >10:1). IR (neat) 1159, 732, 702 cm^{-1}. ^1H NMR: (500 MHz) δ 7.42 (d, J = 8.6 Hz, 2H), 7.32 (d, J = 8.6 Hz, 2H), 6.91 (d, J = 8.6 Hz, 2H), 6.90 (d, J = 8.6 Hz, 2H), 5.52 (d, J = 5.7 Hz, 1H), 5.04 (d, J = 9.9 Hz, 1H), 4.14 (t, J = 7.3 Hz, 1H), 3.83–3.80 (m, 7H), 3.44 (dd, J = 10.8, 8 Hz, 1H), 3.23 (dd, J = 11.2, 7.5 Hz, 1H), 2.86-2.77 (m, 1H), 2.69-2.60 (m, 1H). ^{13}C NMR: (125 MHz, $CDCl_3$) δ 160.3, 159.0, 129.1, 129.03, 128.95, 128.4, 114.2, 113.6, 87.3, 82.9, 68.9, 68.1, 55.3, 55.2, 46.7, 41.1. HRMS (EI) calculated for $[C_{20}H_{22}O_5]^+$ requires m/z 342.1462, found m/z 342.1453.



(Table 2, entry 12). Prepared according to the General Procedure using 100 mg (0.287 mmol) of bis(alkene), 1.2 mg (1.4 μmol) $Ru(bpz)_3(PF_6)_2$, 14.4 mL of $MeNO_2$ and an irradiation time of 2 h. Purification by flash column chromatography afforded the cycloadduct as an oil. Experiment 1: 90.3 mg (0.237 mmol, 83% yield, dr = 4:1), Experiment 2: 88.4 mg (0.232 mmol, 81% yield, dr = 4:1). IR (neat) 1312, 1120, 773 cm^{-1}. ^1H NMR: (500 MHz) δ 8.22 (d, J = 7.5 Hz, 1H), 7.68 (d, J = 7.8 Hz, 1H), 7.59 (t, J = 7.5 Hz, 1H), 7.45 (t, J = 7.5 Hz, 1H), 7.40 (d, J = 8.6 Hz, 2H), 6.97 (2, J = 8.8 Hz, 2H), 5.99 (d, J = 7.2 Hz, 1H), 5.07 (d, J = 9.7 Hz, 1H), 4.02 (td, J = 7.7, 1.7 Hz, 1H), 3.93 (t, J = 7.7 Hz, 1H), 3.84 (s, 3H), 3.53 (dd, J = 10.6, 7.9 Hz, 1H), 3.00 (m, 1H), 2.91–2.79 (m, 2H). ^{13}C NMR: (125 MHz, $CDCl_3$) δ 160.2, 135.3, 132.1, 130.3, 128.8, 128.4, 128.2, 127.4 (q, J = 30.2 Hz), 126.2 (q, J = 5.8 Hz), 124.1 (t, J = 274.5 Hz), 114.3, 86.8, 78.8 (q, J = 2.7 Hz), 68.9 (q, J = 3.9 Hz), 68.7, 55.3, 45.5, 41.3. HRMS (E1) calculated for $[C_{20}H_{19}O_4F_3]^+$ requires m/z 380.1230, found m/z 380.1237.

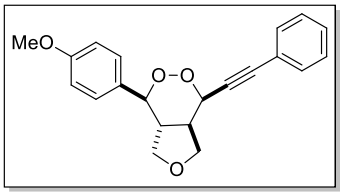


(Table 2, entry 12, minor diastereomer). IR (neat) 1315, 1253, 1037 cm^{-1}. ^1H NMR: (500 MHz) δ 7.73 (t, J = 7.6 Hz, 2H), 7.64 (t, J = 7.6 Hz, 1H), 7.50 (t, J = 7.6 Hz, 1H), 7.38 (d, J = 8.6 Hz, 2H), 6.94 (d, J = 8.6 Hz, 2H), 5.59 (d, J = 9.8 Hz, 1H), 5.18 (d, J = 10.0 Hz, 1H), 3.96 (t, J = 7.6 Hz, 1H), 3.84 (s, 3H), 3.77 (t, J = 7.5 Hz, 1H), 3.60 (dd, J = 11.0, 8.1 Hz, 1H), 3.55 (dd, J = 11.1, 8.0 Hz, 1H), 2.75 (m, 1H), 2.62 (m, 1H). ^{13}C NMR: (125 MHz, $CDCl_3$) δ 160.6, 135.1, 132.4, 129.3, 129.2, 128.54, 128.51, 127.8, 126.0 (q, J = 5.5 Hz), 123.9 (q, J = 274.5 Hz), 114.2, 87.1, 81.9, 68.2, 68.0, 55.3, 50.6, 48.5. HRMS (E1) calculated for $[C_{20}H_{19}O_4F_3]^+$ requires m/z 380.1230, found m/z 380.1212.

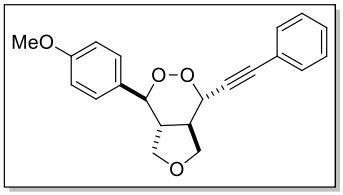


(Table 2, entry 13). Prepared according to the General Procedure using 100 mg (0.318 mmol) of bis(alkene), 1.4 mg (1.6 μmol) of $Ru(bpz)_3(PF_6)_2$, 15.9 mL of $MeNO_2$ and an irradiation time of 2 h. Purification by flash column chromatography afforded the cycloadduct as an oil. Experiment 1: 94.0 mg (0.271 mmol, 85% yield, dr = 5:1), Experiment 2: 95.4 mg (0.275 mmol, 87% yield, dr = 5:1). IR (neat) 1252, 1062, 848 cm^{-1}. ^1H NMR: (500 MHz) δ 7.44 (d, J = 8.6 Hz, 2H), 7.35 (2, J = 8.6 Hz, 2H), 7.29 (d, J = 8.6 Hz, 2H), 6.91 (d, J = 8.6 Hz, 2H), 5.53 (d, J = 5.9 Hz, 1H), 5.04 (d, J = 10.0 Hz, 1H), 4.18 (t, J = 7.3 Hz, 1H), 3.83–3.77 (m, 4H), 3.43 (dd, J = 10.9, 8.1 Hz, 1H), 3.24 (dd, J = 11.4, 7.6 Hz, 1H), 2.82 (m, 1H), 2.55 (m, 1H). ^{13}C NMR:

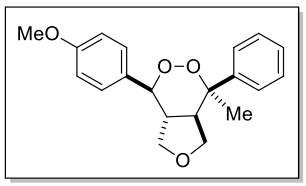
(125 MHz, CD_3CN) δ 161.9, 138.0, 134.2, 130.8, 130.45, 130.39, 129.5, 115.4, 88.6, 83.8, 69.7, 68.6, 56.4, 47.6, 42.2;. HRMS (EI) calculated for $[C_{19}H_{19}O_4Cl]^+$ requires m/z 346.0967, found m/z 346.0985.



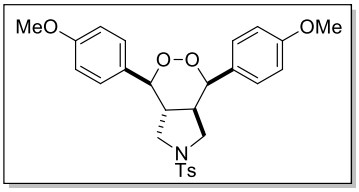
(Table 2, entry 14). Prepared according to the General Procedure using 100 mg (0.329 mmol) of bis(alkene), 1.4 mg (1.6 μmol) of $Ru(bpz)_3(PF_6)_2$, 16.6 mL of $MeNO_2$ and an irradiation time of 2 h. Purification by flash column chromatography afforded the cycloadduct as an oil. Experiment 1: 88 mg (0.262 mmol, 80% yield, dr = 2:1), Experiment 2: 92.1 mg (0.274 mmol, 83% yield, dr = 2:1). IR (neat) 1253, 758, 691 cm^{-1}. ^1H NMR: (500 MHz) δ 7.51 (m, 2H), 7.38–7.30 (m, 5H), 6.89 (d, J = 8.7 Hz, 2H), 5.34 (d, J = 4.7 Hz, 1H), 5.02 (d, J = 10.0 Hz, 1H), 4.17 (t, J = 7.4 Hz, 1H), 3.89 (t, J = 7.7 Hz, 1H), 3.80 (s, 3H), 3.74 (dd, J = 11.0, 7.1 Hz, 1H), 3.46 (dd, J = 11.4, 7.9 Hz, 1H), 2.96 (m, 1H), 2.65 (m, 1H). ^{13}C NMR: (125 MHz, $CDCl_3$) δ 160.5, 132.0, 129.2, 128.9, 128.4, 128.3, 122.0, 114.1, 89.0, 87.2, 82.8, 73.3, 68.8, 68.0, 55.3, 46.6, 43.5. HRMS (EI) calculated for $[C_{21}H_{20}O_4]^+$ requires m/z 336.1456, found m/z 336.1464.



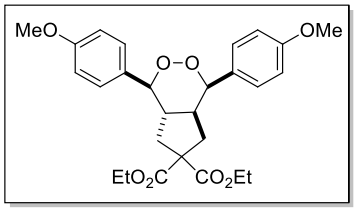
(Table 2, entry 14, minor diastereomer). IR (neat) 1252, 759, 691 cm^{-1}. ^1H NMR: (500 MHz) δ 7.46 (m, 2H), 7.38–7.28 (m, 5H), 6.90 (d, J = 8.7 Hz, 2H), 5.05 (d, J = 9.5 Hz, 2H), 4.23 (t, J = 7.3 Hz, 1H), 3.92 (t, J = 7.3 Hz, 1H), 3.81 (s, 3H), 3.69 (dd, J = 10.7, 7.8 Hz, 1H), 3.51 (dd, J = 10.7, 7.9 Hz, 1H), 2.55 (m, 2H). ^{13}C NMR: (125 MHz, $CDCl_3$) δ 160.6, 132.0, 129.21, 129.17, 128.4, 127.7, 121.5, 114.2, 88.8, 86.8, 82.7, 75.1, 68.4, 68.3, 55.3, 49.8, 48.1. HRMS (EI) calculated for $[C_{21}H_{20}O_4]^+$ requires m/z 336.1456, found m/z 336.1461.



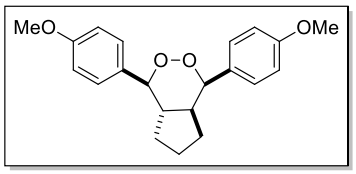
(Table 2, entry 15). Prepared according to the General Procedure using 100 mg (0.340 mmol) of bis(alkene), 5.9 mg (6.8 μmol) of $Ru(bpz)_3(PF_6)_2$, 17 mL of $MeNO_2$ and an irradiation time of 2 h. Purification by flash column chromatography afforded the cycloadduct as an oil. Experiment 1: 88.1 mg (0.270 mmol, 79% yield, dr >10:1), Experiment 2: 90.5 mg (0.277 mmol, 82% yield, dr >10:1). IR (neat) 1251, 738, 704 cm^{-1}. ^1H NMR: (500 MHz) δ 7.49 (m, 2H), 7.37 (m, 2H), 7.29 (m, 1H), 7.21 (d, J = 8.6 Hz, 2H), 6.86 (d, J = 8.6 Hz, 2H), 4.97 (d, J = 9.9 Hz, 1H), 4.28 (t, J = 7.0 Hz, 1H), 3.78 (s, 3H), 3.72 (t, J = 8.0 Hz, 1H), 3.66 (dd, J = 11.4, 7.4 Hz, 1H), 3.42 (dd, J = 10.6, 8.1 Hz, 1H), 2.51 (m, 1H), 2.30 (m, 1H), 1.61 (s, 3H). ^{13}C NMR: (125 MHz, $CDCl_3$) δ 160.3, 140.6, 129.1, 128.6, 127.9, 127.1, 126.9, 114.1, 87.3, 86.1, 68.8, 68.2, 55.2, 53.4, 43.0, 28.5. HRMS (EI) calculated for $[C_{20}H_{22}O_4]^+$ requires m/z 326.1613, found m/z 326.1616.



(Table 2, entry 16). Prepared according to the General Procedure using 100 mg (0.230 mmol) of bis(alkene), 1.0 mg (1.1 μmol) of $Ru(bpz)_3(PF_6)_2$, 11.5 mL of $MeNO_2$ and an irradiation time of 2 h. Purification by flash column chromatography afforded the cycloadduct as an oil. Experiment 1: 80.0 mg (0.171 mmol, 75% yield, dr >10:1), Experiment 2: 76.2 mg (0.163 mmol, 71% yield, dr >10:1). IR (neat) 1163, 733, 665 cm^{-1}. ^1H NMR: (500 MHz) δ 7.57 (d, J = 8.2 Hz, 2H), 7.35 (d, J = 8.6 Hz, 2H), 7.25 (d, J = 8.2 Hz, 2H), 7.20 (d, J = 8.6 Hz, 2H), 6.91–6.86 (m, 4H), 5.32 (d, J = 5.7 Hz, 1H), 4.86 (d, J = 9.9 Hz, 1H), 3.83 (s, 3H), 3.82 (s, 3H), 3.66 (dd, J = 8.9, 7.5 Hz, 1H), 3.34 (dd, J = 9.8, 7.2 Hz, 1H), 2.82 (m, 2H), 2.47 (m, 1H), 2.43 (s, 3H), 2.28 (m, 1H). ^{13}C NMR: (125 MHz, $CDCl_3$) δ 160.5, 159.2, 143.5, 134.2, 129.7, 129.05, 128.96, 128.5, 128.4, 127.1, 114.2, 113.7, 87.0, 82.7, 55.3, 55.2, 49.5, 48.9, 45.1, 39.7, 21.4;. HRMS (EI) calculated for $[C_{27}H_{29}NO_6S+Na]^+$ requires m/z 518.1608, found m/z 518.1614.

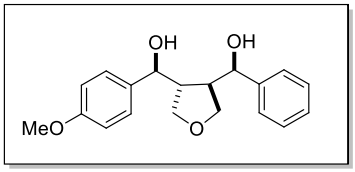


(Table 2, entry 17). Prepared according to the General Procedure using 100 mg (0.220 mmol) of bis(alkene), 1.0 mg (1.1 μmol) of $Ru(bpz)_3(PF_6)_2$, 11 mL of $MeNO_2$ and an irradiation time of 2 h. Purification by flash column chromatography afforded the cycloadduct as an oil. Experiment 1: 97.4 mg (0.201 mmol, 91% yield, dr >10:1), Experiment 2: 98.8 mg (0.204 mmol, 92% yield, dr >10:1). IR (neat) 1253, 1034, 733 cm^{-1}. ^1H NMR: (500 MHz) δ 7.49 (d, J = 8.6 Hz, 2H), 7.32 (d, J = 8.6 Hz, 2H), 6.93–6.87 (m, 4H), 5.37 (d, J = 5.7 Hz, 1H), 4.93 (d, J = 9.8 Hz, 1H), 4.18 (m, 2H), 4.06 (m, 2H), 3.82 (s, 3H), 3.81 (s, 3H), 2.64 (dd, J = 12.8, 6.1 Hz, 1H), 2.49 (m, 1H), 2.33 (dd, J = 13.1, 7.1 Hz, 1H), 2.26 (m, 1H), 1.74 (dd, J = 13.0, 11.6 Hz, 1H), 1.53 (t, J = 13.0 Hz, 1H), 1.23 (t, J = 7.2 Hz, 3H), 1.13 (t, J = 7.2 Hz, 3H). ^{13}C NMR: (125 MHz, $CDCl_3$) δ 172.2, 171.9, 160.2, 158.9, 129.4, 129.3, 128.9, 128.8, 114.1, 113.5, 89.5, 84.8, 61.6, 61.5, 57.4, 55.25, 55.18, 46.2, 40.8, 36.5, 35.1, 14.0, 13.8. HRMS (EI) calculated for $[C_{27}H_{32}O_8+Na]^+$ requires m/z 507.1990, found m/z 507.2000.

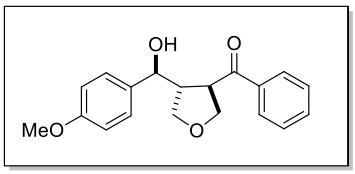


(Table 2, entry 18). Prepared according to the General Procedure using 100 mg (0.325 mmol) of bis(alkene), 1.4 mg (1.6 μmol) of $Ru(bpz)_3(PF_6)_2$, 16 mL of $MeNO_2$ and an irradiation time of 2 h. Purification by flash column chromatography afforded the cycloadduct as an oil. Experiment 1: 88.0 mg (0.259 mmol, 80% yield, >10:1dr), Experiment 2: 86.9 mg (0.256 mmol, 79% yield, >10:1dr). IR (neat) 1250, 1032, 743 cm^{-1}. ^1H NMR: (500 MHz) δ 7.53 (d, J = 9.0 Hz, 2H), 7.32 (d, J = 9.0 Hz, 2H), 6.90 (d, J = 9.0 Hz, 4H), 5.39 (d, J = 5.8 Hz, 1H), 4.91 (d, J = 10 Hz, 1H), 3.82 (s, 3H), 3.81 (s, 3H), 2.27 (m, 1H), 2.03 (m, 1H), 1.87 (m, 1H), 1.66 (m, 1H), 1.55 (m, 2H), 1.19 (m, 1H), 1.06 (m, 1H). ^{13}C NMR: (125 MHz, $CDCl_3$) δ 160.0, 158.7, 130.5, 129.8, 129.3, 129.0, 113.9, 113.2, 90.7, 85.9, 55.24, 55.18, 47.4, 41.4, 27.3, 25.8, 21.2. HRMS (EI) calculated for $[C_{21}H_{24}O_4]^+$ requires m/z 340.1670, found m/z 340.1657.

IV. Endoperoxide opening reactions



Compound 6. A 2 dram vial equipped with a stirbar was charged with 139 mg (0.440 mmol) of **5**. AcOH (2.2 mL) and zinc dust (144 mg, 2.2 mmol) were added, the vial was sealed with a Teflon cap, and the reaction was stirred at room temperature. After 2 h, the reaction mixture was concentrated by rotary evaporation and then eluted through a short plug of silica using EtOAc. The eluent was concentrated by rotary evaporation, and the residue was purified by chromatography on SiO_2 (2:1 to 1:1 hexanes/EtOAc) to afford 126 mg (0.40 mmol, 91% yield) of the desired product as a colorless solid (mp = 150–151 °C). ^1H NMR: (500 MHz, $CDCl_3$) δ 7.34 (m, 4H), 7.28 (m, 1H), 7.19 (d, J = 8.6 Hz, 2H), 6.85 (d, J = 8.6 Hz, 2H), 4.79 (s, 1H), 4.48 (d, J = 9.0 Hz, 1H), 3.88 (dd, J = 9.0, 7.7 Hz, 1H), 3.80 (m, 4H), 3.79 (dd, J = 9.1, 6.6 Hz, 1H), 3.49 (dd, J = 9.1, 7.8 Hz, 1H), 3.39 (dd, J = 9.3, 6.1 Hz, 1H), 3.03 (s, 1H), 2.77 (m, 1H), 2.53 (m, 1H). ^{13}C NMR: (125 MHz, $CDCl_3$) δ 159.4, 142.4, 134.9, 128.4, 127.7, 127.6, 126.7, 114.0, 76.3, 74.4, 71.0, 70.2, 55.3, 49.8, 48.1. HRMS (E1) calculated for $[C_{19}H_{22}O_4+Na]^+$ requires m/z 337.1411, found m/z 337.1409.



Compound 7. A 2 dram vial equipped with a stirbar was charged with 74.0 mg (0.240 mmol) of endoperoxide **5**. Dry CH_2Cl_2 (1.2 mL) and NEt_3 (10 μL, 0.07 mmol) were added sequentially, the vial was sealed with a Teflon cap, and the reaction was stirred at room temperature. After 13 h, the reaction mixture was concentrated by rotary evaporation and the crude reaction mixture was purified by chromatography on SiO_2 (2:1 to 1:1 hexanes/EtOAc) to afford 69.0 mg (0.22 mmol, 92% yield) of a colorless oil. ^1H NMR: (500 MHz, $CDCl_3$) δ 7.95 (d, J = 7.8 Hz, 2H), 7.56 (t, J = 7.2 Hz, 1H), 7.44 (t, J = 7.5 Hz, 2H), 7.27 (d, J = 8.7 Hz, 2H), 6.85 (d, J = 8.7 Hz, 2H), 4.61 (d, J = 9.0 Hz, 1H), 4.26 (m, 2H), 3.89 (m, 1H), 3.78 (s, 3H), 3.74 (dd,

J = 9.0, 7.2 Hz, 1H), 3.60 (dd, J = 9.0, 4.6 Hz, 1H), 3.16 (m, 1H), 2.33 (s, 1H). ^{13}C NMR: (125 MHz, $CDCl_3$) δ 200.0, 159.4, 136.4, 134.9, 133.2, 128.62, 128.60, 127.6, 114.0, 75.7, 71.3, 70.8, 55.2, 50.6, 49.7. HRMS (E1) calculated for $[C_{19}H_{20}O_4+Na]^+$ requires m/z 335.1254, found m/z 335.1256.

V. Representative NOE data

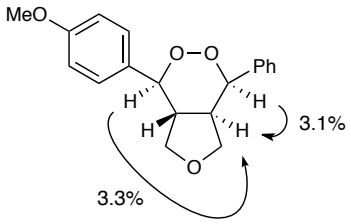


Table 2, entry 1

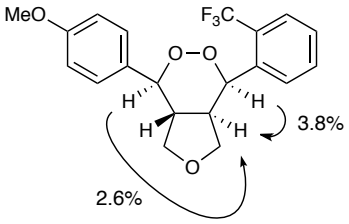


Table 2, entry 12

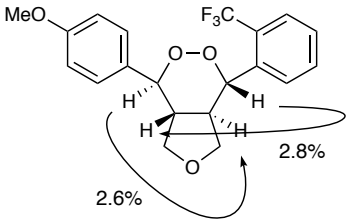


Table 2, entry 12
minor diastereomer

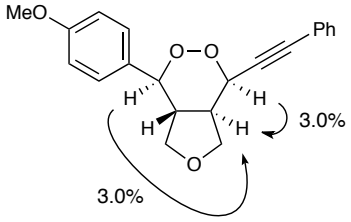


Table 2, entry 14

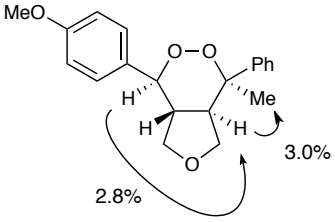


Table 2, entry 15

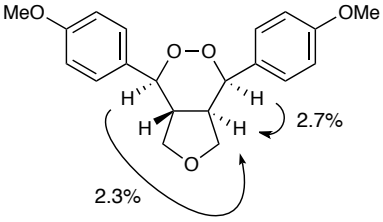


Table 2, entry 17

VI. Cytotoxicity screening data

Table S-1. Tabulated cytotoxicity data for representative endoperoxides from Table 2.

Compound	**IC50** (μM)	**SE**
Table 2, Entry 1	>100	NA
Table 2, Entry 3	27.64	1.27
Table 2, Entry 5	>100	NA
Table 2, Entry 6	77.72	1.82
Table 2, Entry 7	38.71	2.66
Table 2, Entry 11	>100	NA
Table 2, Entry 12	13.18	0.63
Table 2, Entry 13	44.30	1.89
Table 2, Entry 14	4.56	0.19
Table 2, Entry 16	17.00	0.46

As an initial demonstration of their potential biological activity, a selection of the compounds reported in Table 2 were assayed for cytotoxicity in human prostate cancer cells (Du145s). Cells were treated for 72 hours with each test compound and then the amount of live cells was quantified using an ATP determining assay reagent, Cell Titer Glo (Promega). The IC_{50} was determined using curve fitting software, xlfit 5.0 (IDBS), to determine the concentration at which point 50% cytotoxicity was observed. Working stocks were prepared at a final concentration of 100X in anhydrous DMSO. Serial dilutions were made in DMSO in 96-well polypropylene plates using the Precision XS liquid handler (BioTek, Inc.). Compounds were divided equally into the corresponding wells of a 384-well plate in all 4 quadrants using a Biomek FX liquid handler with 96 channel pipetting head (Beckman-Coulter, Inc.). Compounds

were stored at –20 °C in 100% DMSO until the day of the assay(s). Freeze-thaw cycles are minimized to a maximum of 10 per plate.

All cell lines were maintained as previously reported.[8] Cells were harvested by trypsinization using 0.25% trypsin and 0.1% EDTA and then counted in a Cellometer Auto T4 cell counter (Nexcelom, inc), before dilution for assay plating. Cell plating, compound handling and assay set up were performed as previously reported (Langenhan, et al) except that the cells were plated in 50 µL volumes in 384-well clear bottom, tissue culture plates (Corning-Costar, Inc). Compounds were added from the 384-well compound stock plates at a 1:100 dilution using a Biomek FX liquid handler equipped with a 384-channel head (Beckman Coulter, Inc.). 15 µL of cell titer-glo reagent (Promega Corporation, Inc.) was added and incubated for 10 min at room temperature with gentle agitation to lyse the cells.

IC_{50}s for cytotoxicity assays were determined cell-titer glo using xlfit 5.0 as previously reported in Langenhan, et al. The IC_{50} determined using the best fit curve for dosage response is reported as the final IC_{50}. The best fit curve is defined as the curve that has the lowest standard error. If the standard error exceeds 10% of the IC_{50}, the assay is deemed a failure and repeated.

VII. References

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[2]Lin, S.; Ischay, M. A.; Fry, C. G.; Yoon, T. P. *J. Am. Chem. Soc.* **2011**, *133*, 19350–19353.
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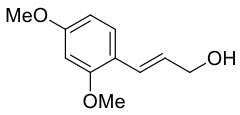
[7]Ghorai, M. K.; Kumar, A.; Das, K. *Org. Lett.* **2007,** *9*, 5441–5444.
[8]Langenhan, J. M.; Peters, N. R.; Guzei, I. A.; Hoffmann, F. M.; Thorson, J. S. *Proc. Nat. Acad. Sci.* **2005**, *102*, 12305–12310.

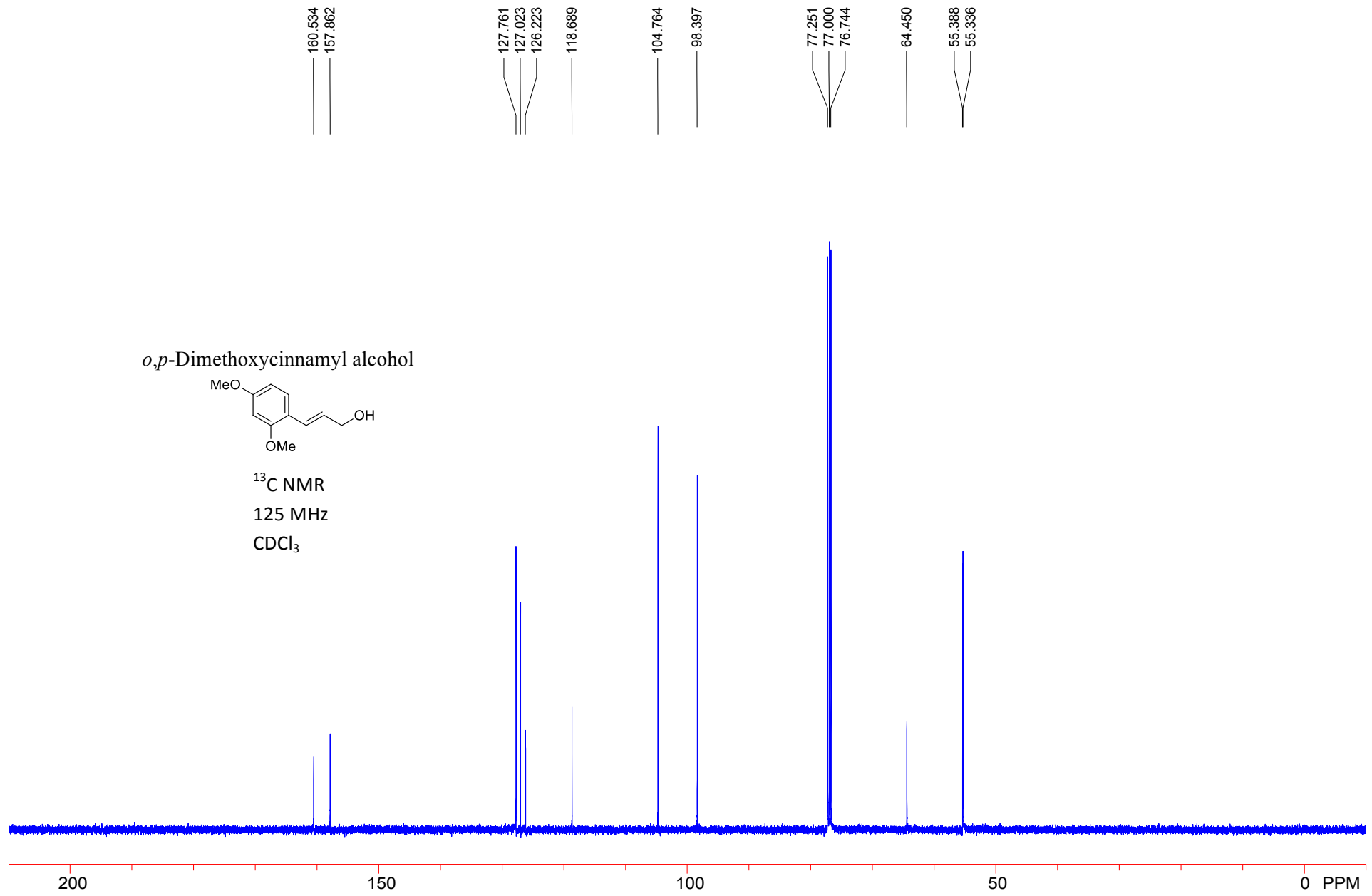
o,p-Dimethoxycinnamyl alcohol



^{1}H NMR

500 MHz

$CDCl_3$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77.000
76.744
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55.388
55.336
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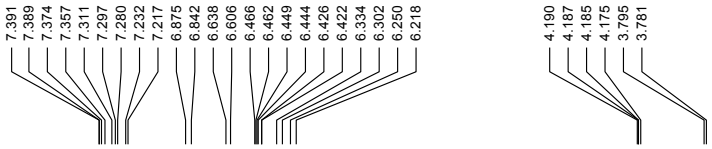
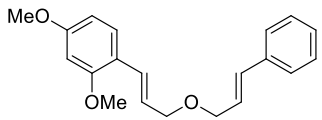
OH
OMe
13C NMR
125 MHz
CDCl3
200
150
100
50
0 PPM



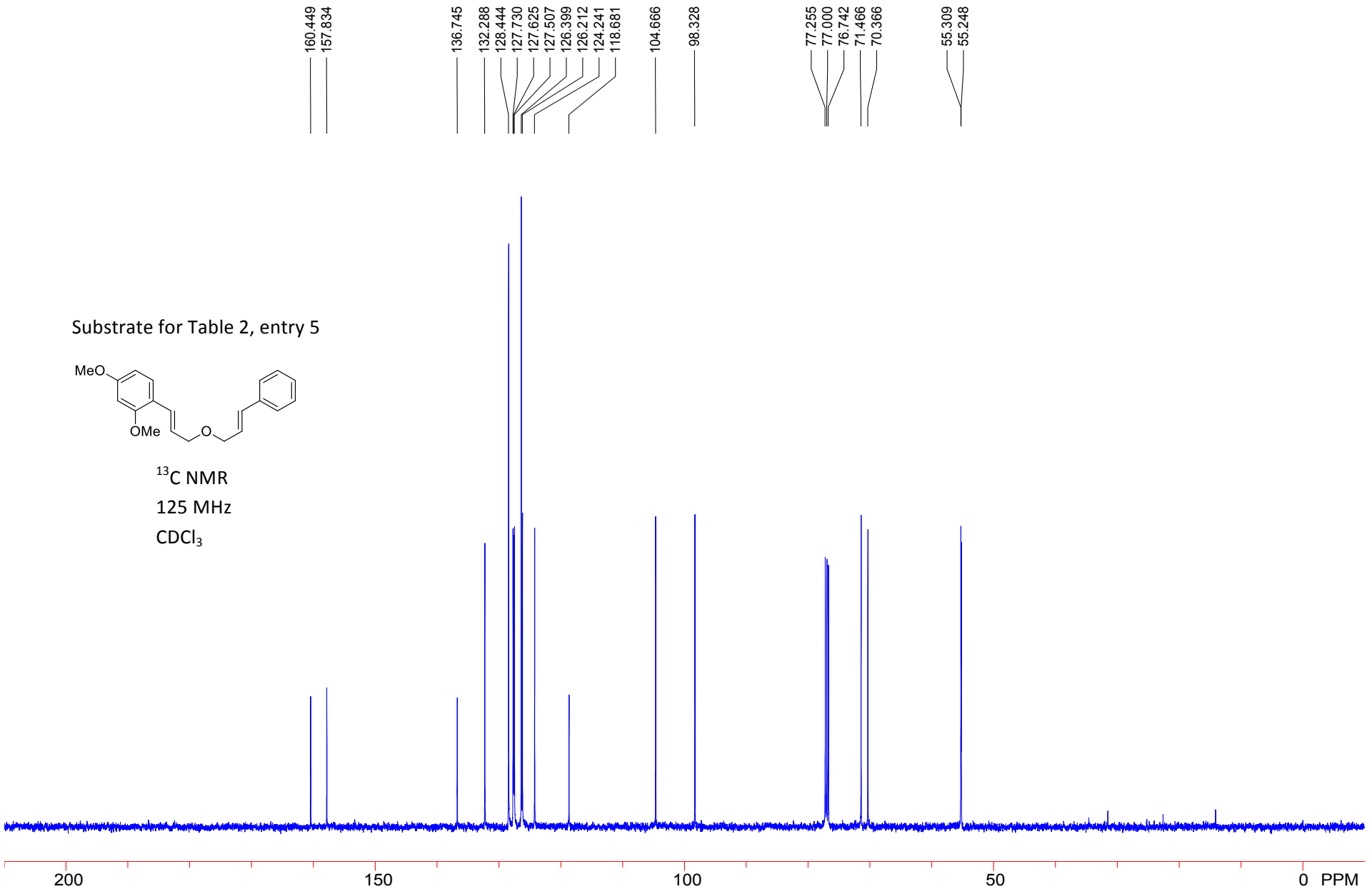
Substrate for Table 2, entry 5



^{1}H NMR

500 MHz

$CDCl_3$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126.399
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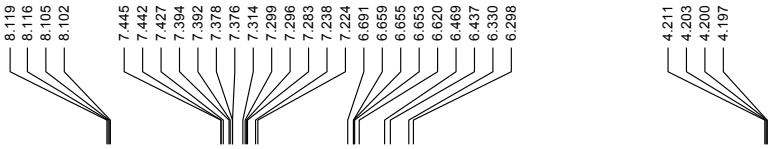
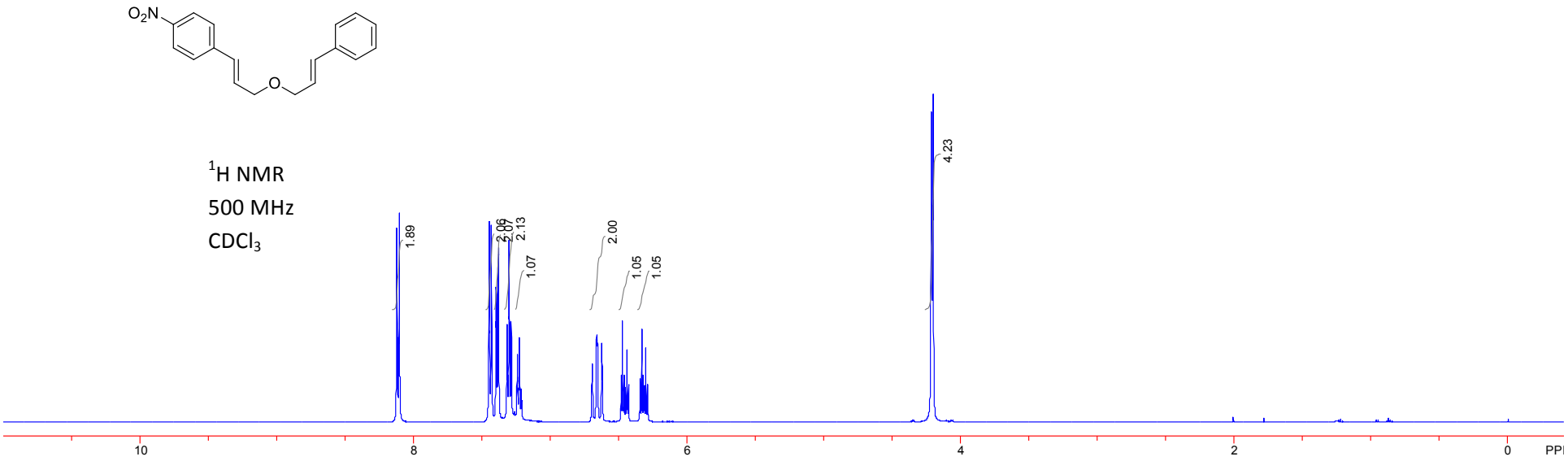
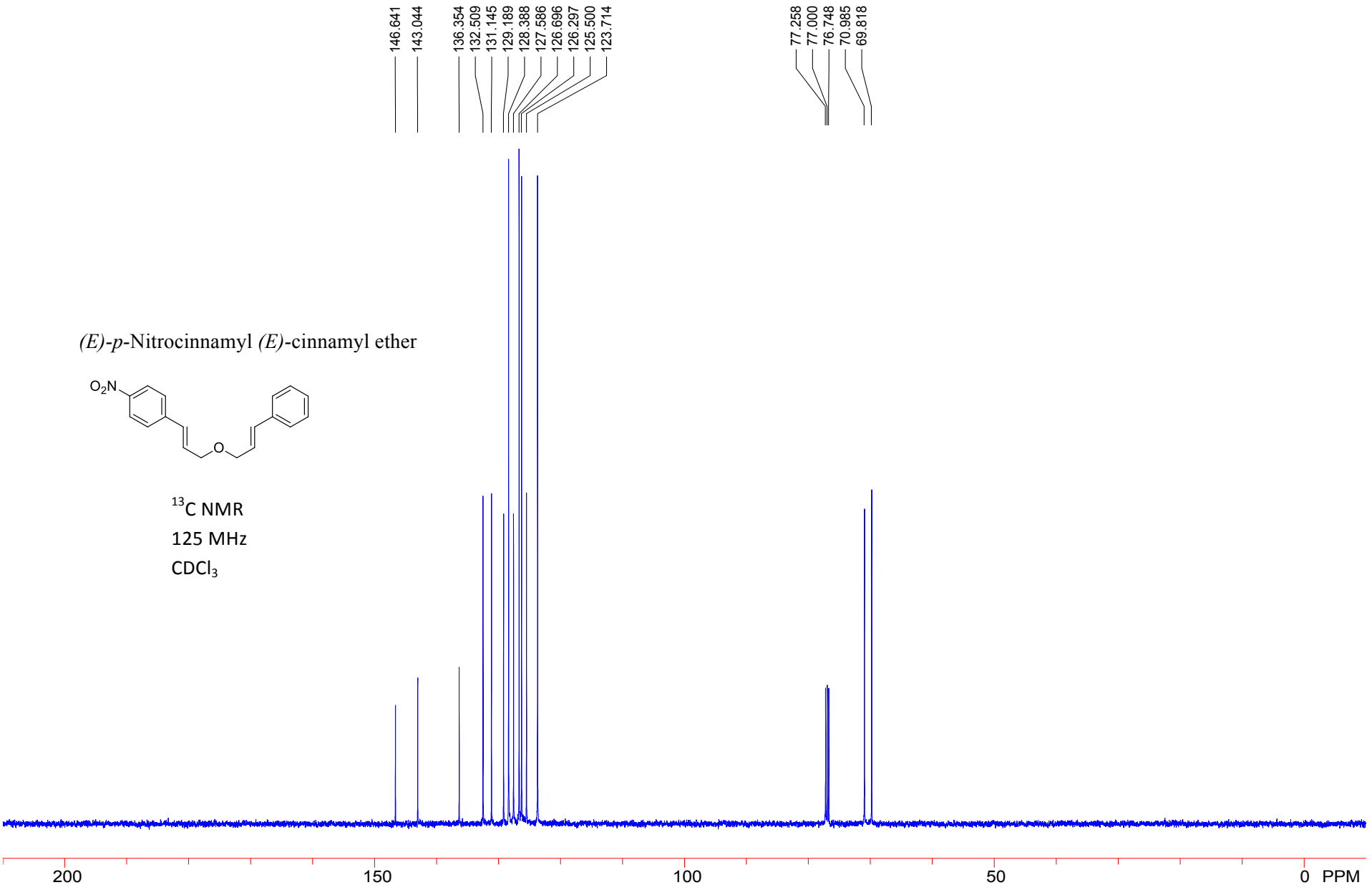
104.666
98.328
77.255
77.000
76.742
71.466
70.366
55.309
55.248
Substrate for Table 2, entry 5
MeO
OMe
O
13C NMR
125 MHz
CDCl3
200
150
100
50
0 PPM



(E)-*p*-Nitrocinnamyl *(E)*-cinnamyl ether

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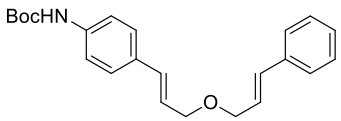
131.145
129.189
128.388
127.586
126.696
126.297
125.500
123.714
77.258
77.000
76.748
70.985
69.818
(E)-p-Nitrocinnamyl *(E)*-cinnamyl ether
O_2N
O
^{13}C NMR
125 MHz
$CDCl_3$
200
150
100
50
0 PPM

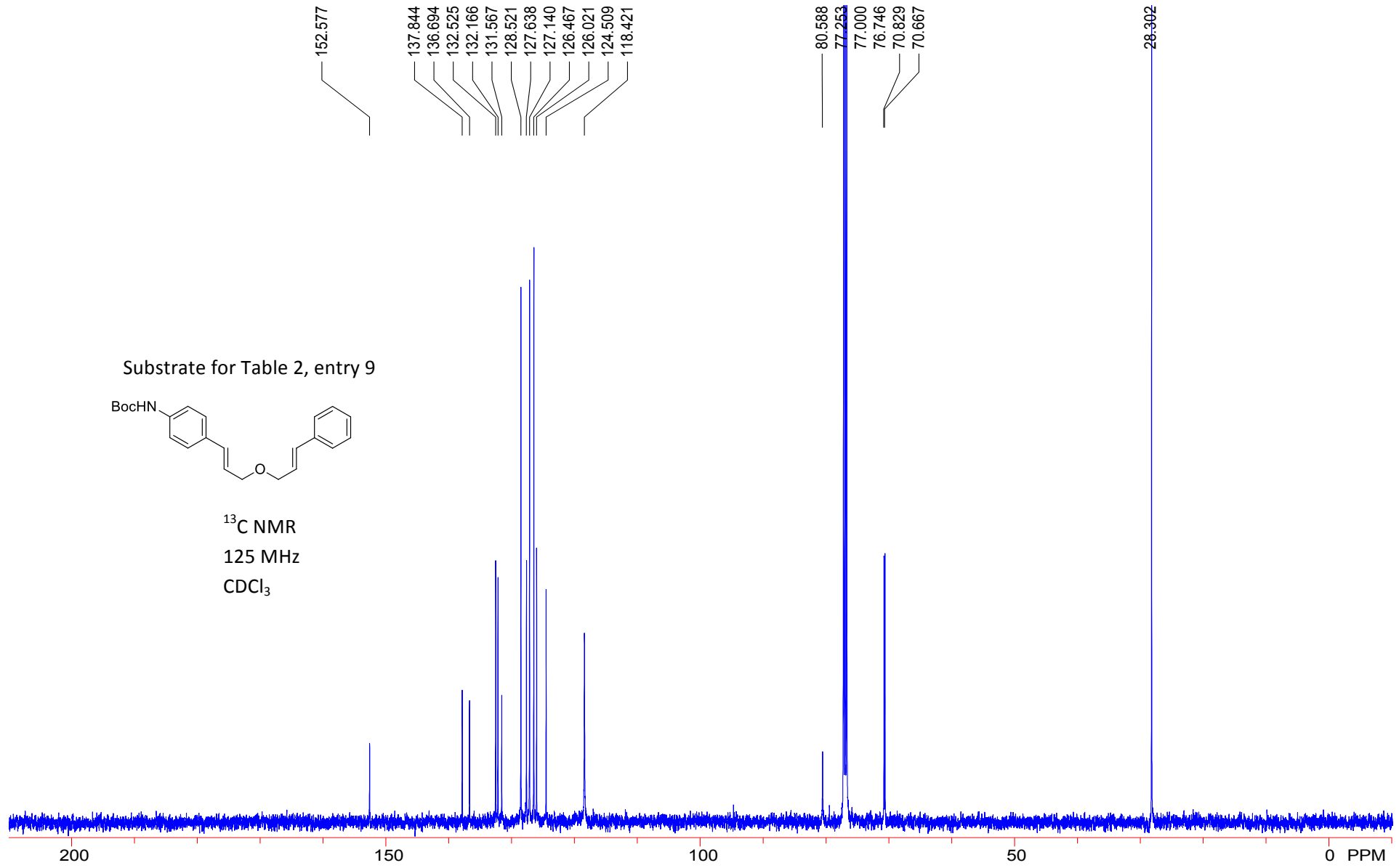
Substrate for Table 2, entry 9



^{1}H NMR

500 MHz

$CDCl_3$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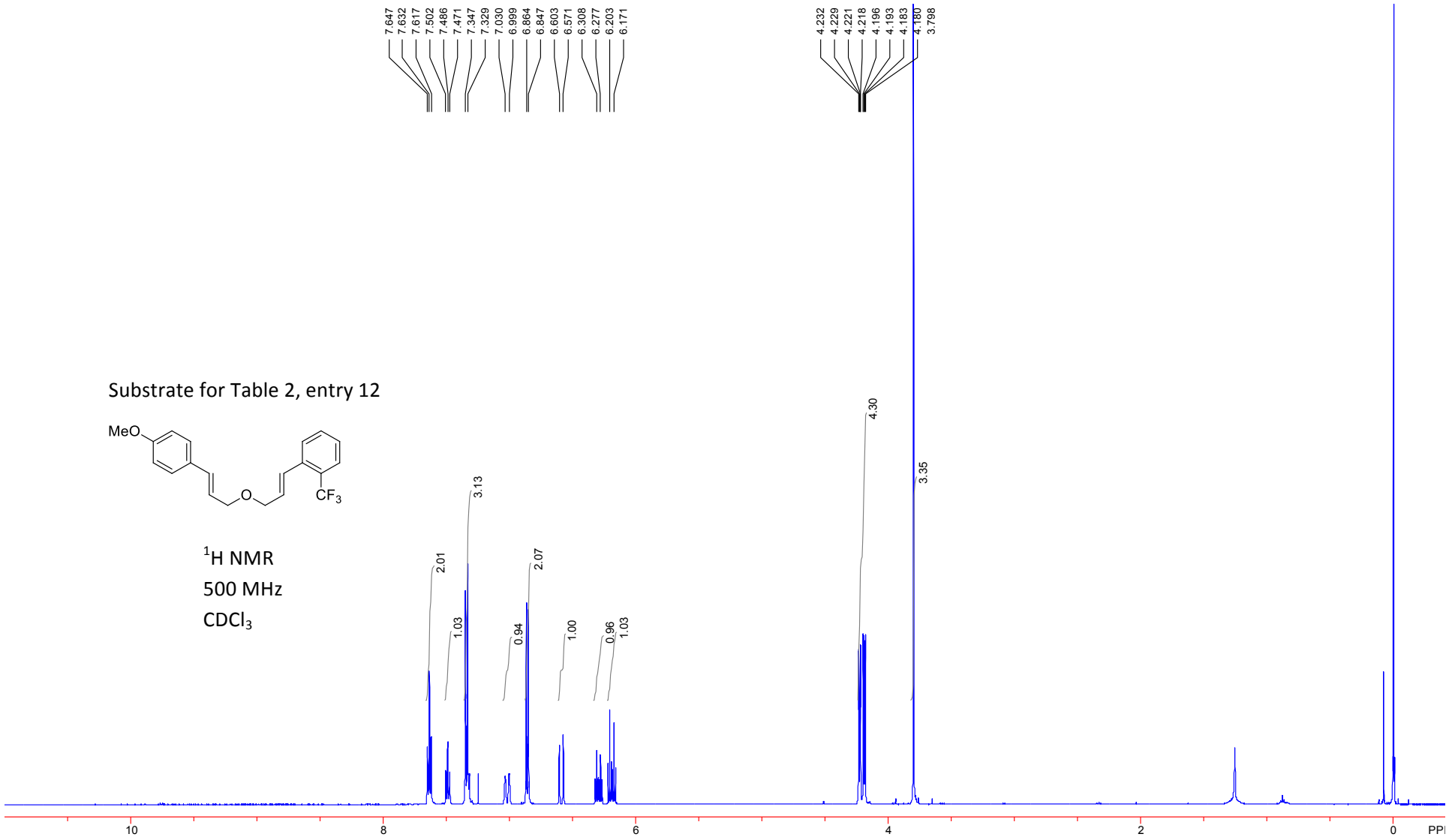
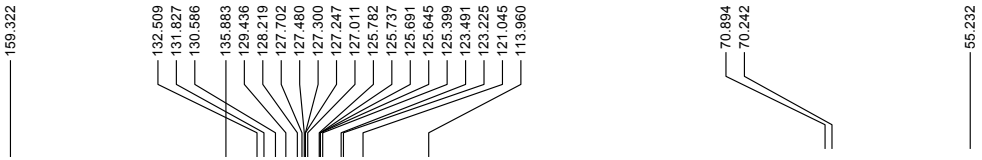
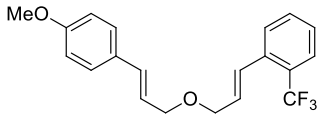
132.166
131.567
128.521
127.638
127.140
126.467
126.021
124.509
118.421
80.588
77.253
77.000
76.746
70.829
70.667
28.302
Substrate for Table 2, entry 9
BocHN
O
13C NMR
125 MHz
CDCl3
200
150
100
50
0 PPM


7.647
7.632
7.617
7.502
7.486
7.471
7.347
7.329
7.030
6.999
6.864
6.847
6.603
6.571
6.308
6.277
6.203
6.171
4.232
4.229
4.221
4.218
4.196
4.193
4.183
4.180
3.798
Substrate for Table 2, entry 12
MeO
O
CF3
1H NMR
500 MHz
CDCl3
2.01
1.03
3.13
0.94
2.07
1.00
0.96
1.03
4.30
3.35
10
8
6
4
2
0
PPI



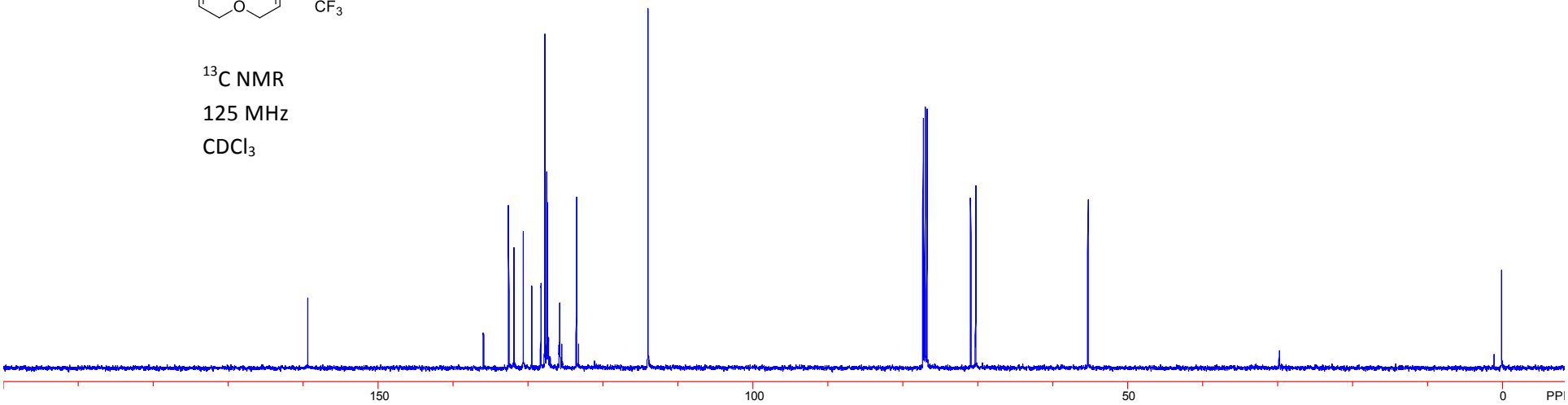
Substrate for Table 2, entry 12



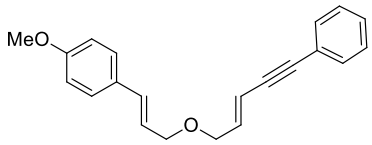
^{13}C NMR

125 MHz

$CDCl_3$



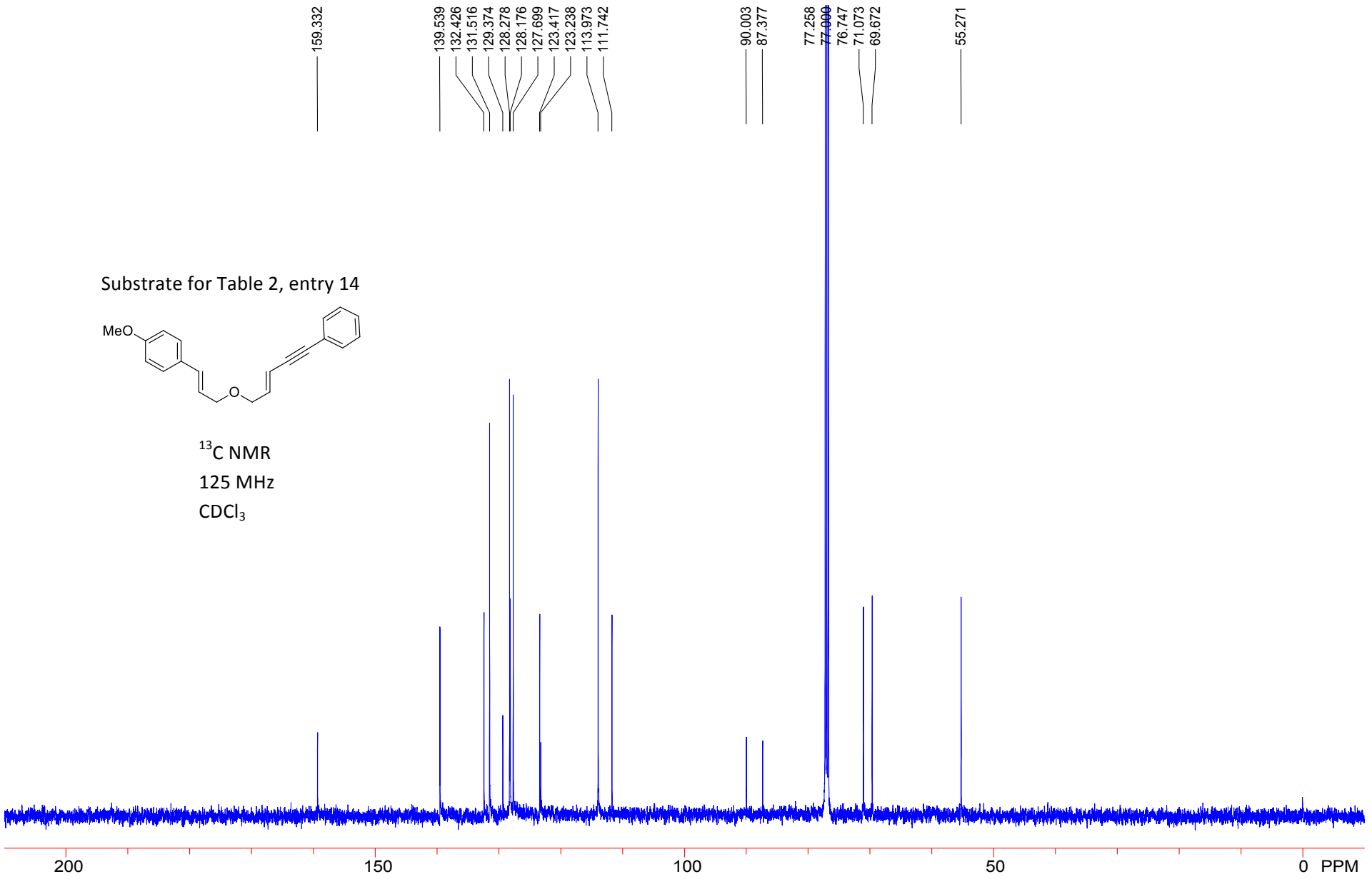
Substrate for Table 2, entry 14



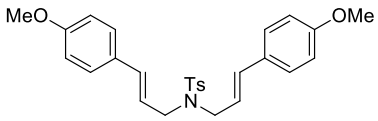
^{1}H NMR

500 MHz

$CDCl_3$


159.332
139.539
132.426
131.516
129.374
128.278
128.176
127.699
123.417
123.238
113.973
111.742
90.003
87.377
77.258
77.000
76.747
71.073
69.672
55.271
Substrate for Table 2, entry 14
MeO
O
^{13}C NMR
125 MHz
$CDCl_3$
200
150
100
50
0 PPM

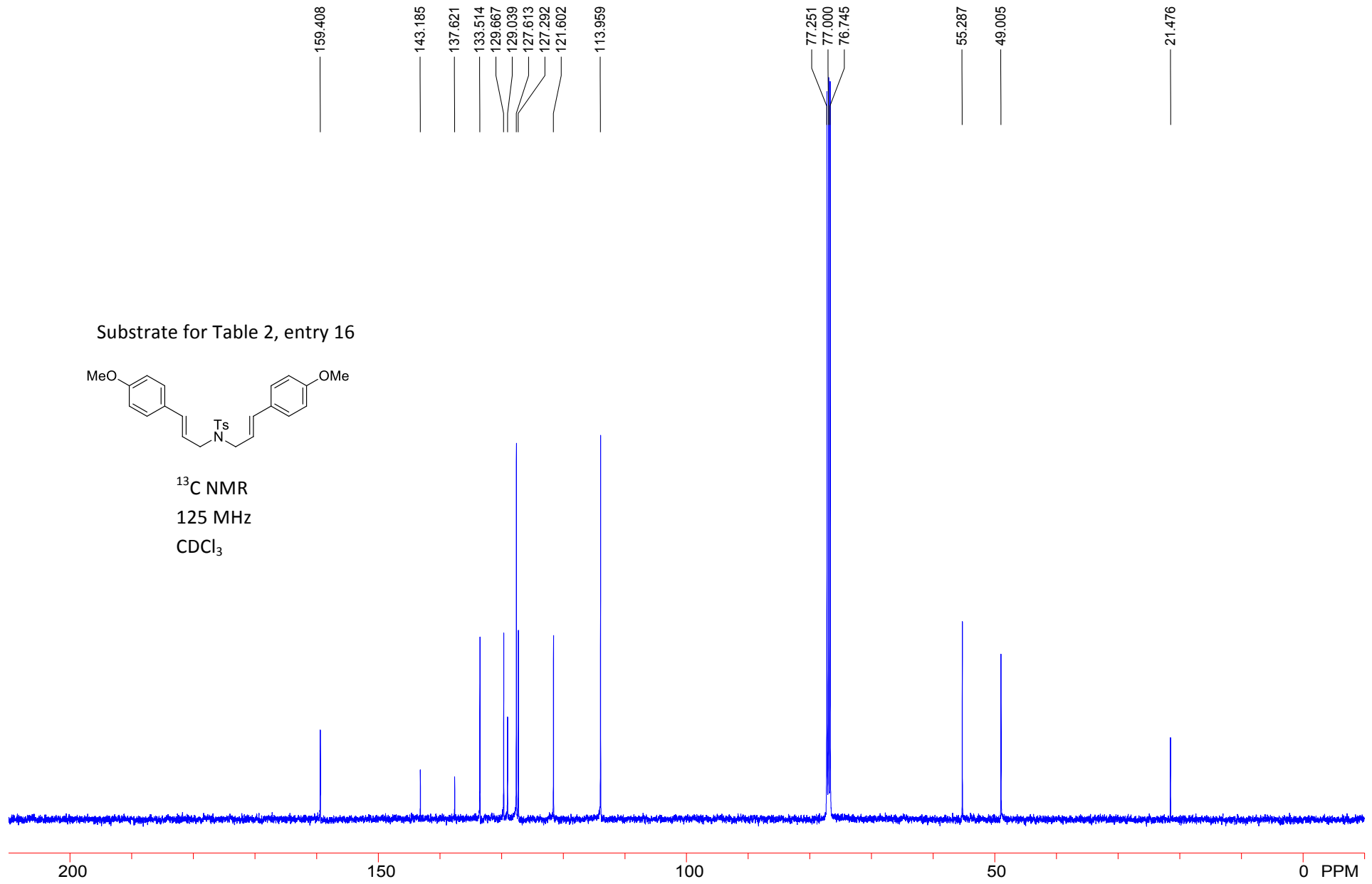
Substrate for Table 2, entry 16



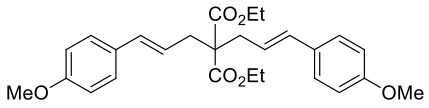
^{1}H NMR

500 MHz

$CDCl_3$


159.408
143.185
137.621
133.514
129.667
129.039
127.613
127.292
121.602
113.959
77.251
77.000
76.745
55.287
49.005
21.476
Substrate for Table 2, entry 16
MeO
OMe
Ts
N
^{13}C NMR
125 MHz
$CDCl_3$
200
150
100
50
0
PPM

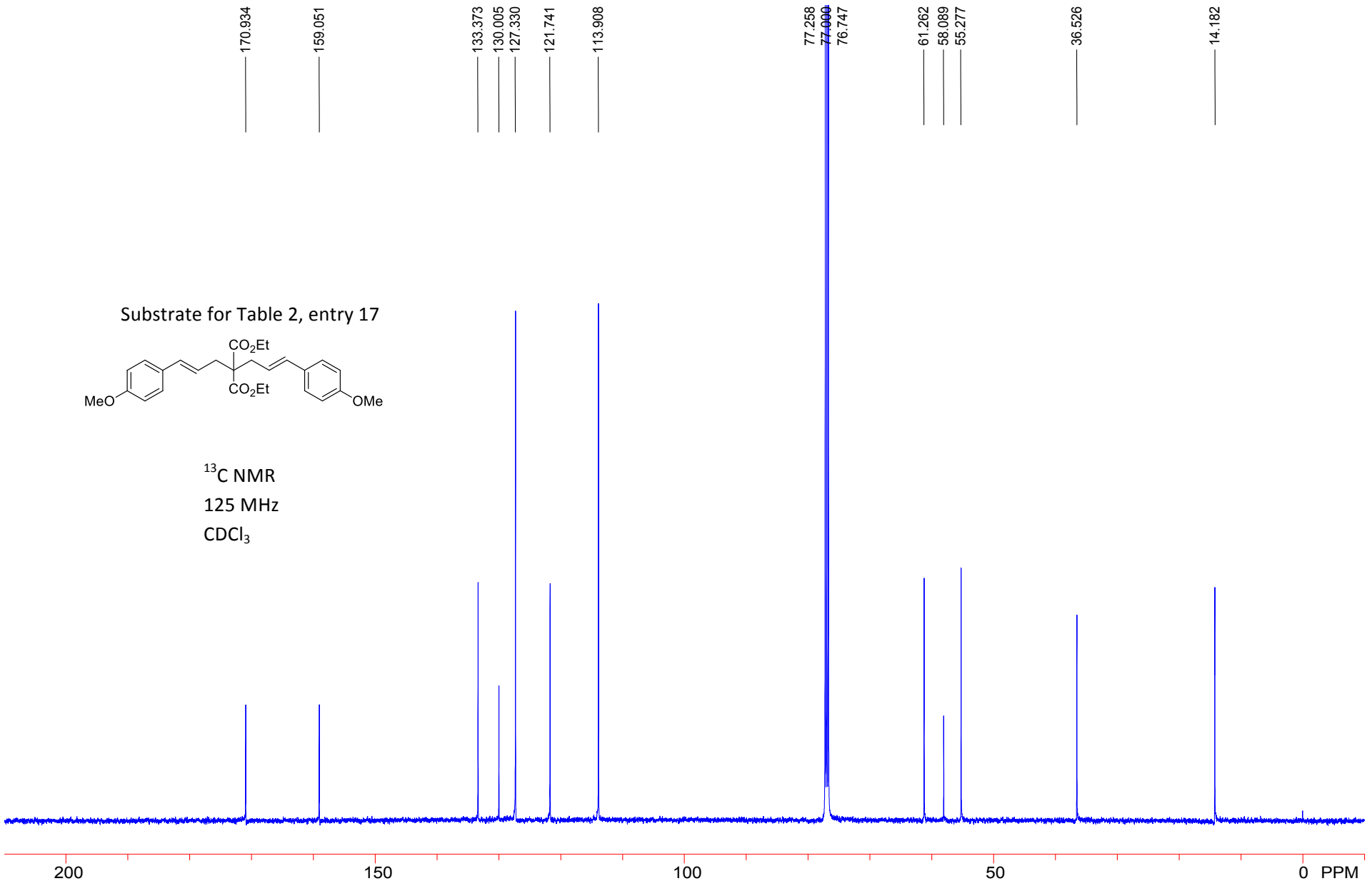
Substrate for Table 2, entry 17



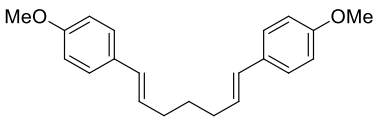
^{1}H NMR

500 MHz

$CDCl_3$


170.934
159.051
133.373
130.005
127.330
121.741
113.908
77.258
77.000
76.747
61.262
58.089
55.277
36.526
14.182
Substrate for Table 2, entry 17
CO2Et
CO2Et
MeO
OMe
13C NMR
125 MHz
CDCl3
200
150
100
50
0 PPM

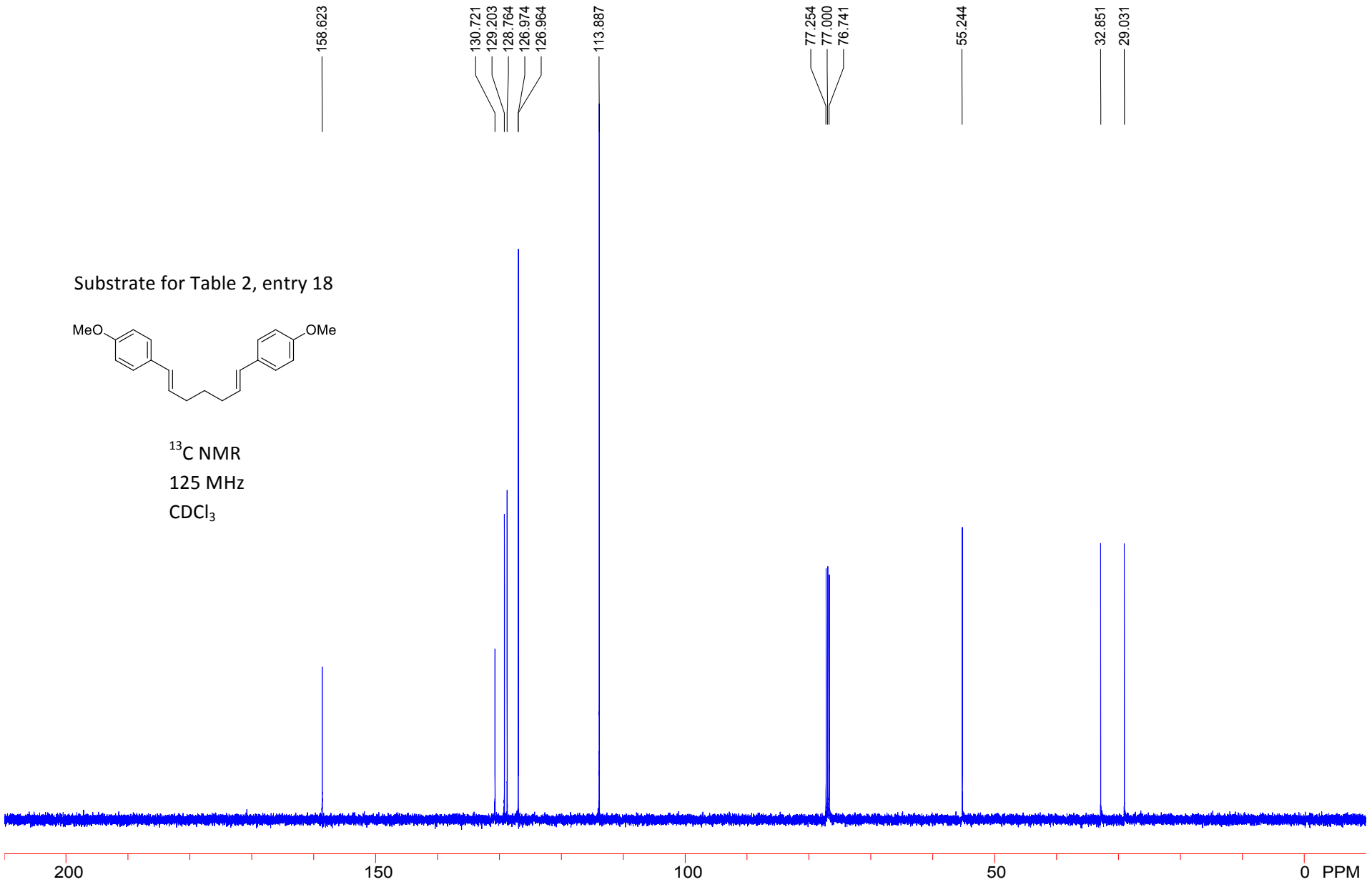
Substrate for Table 2, entry 18

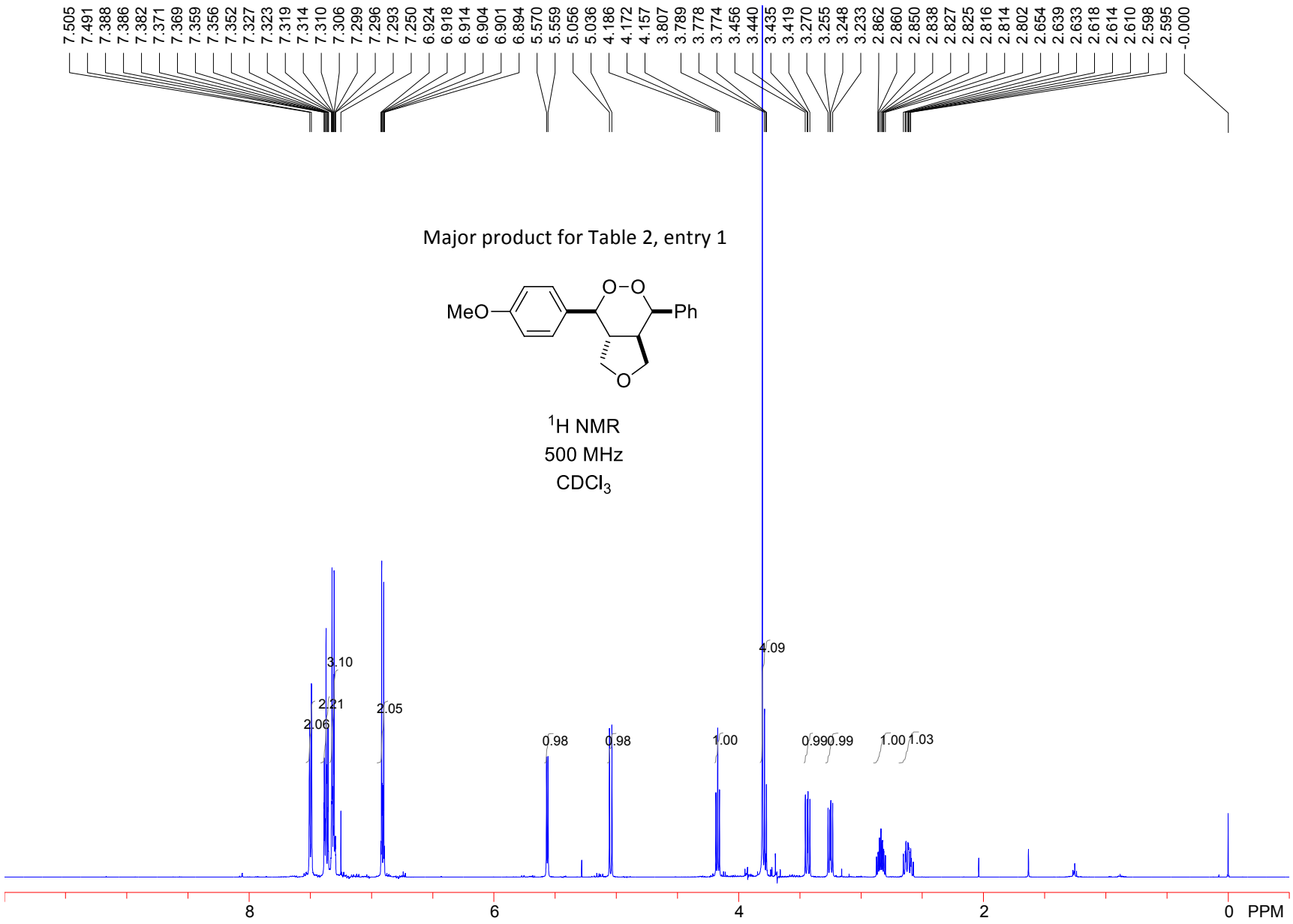


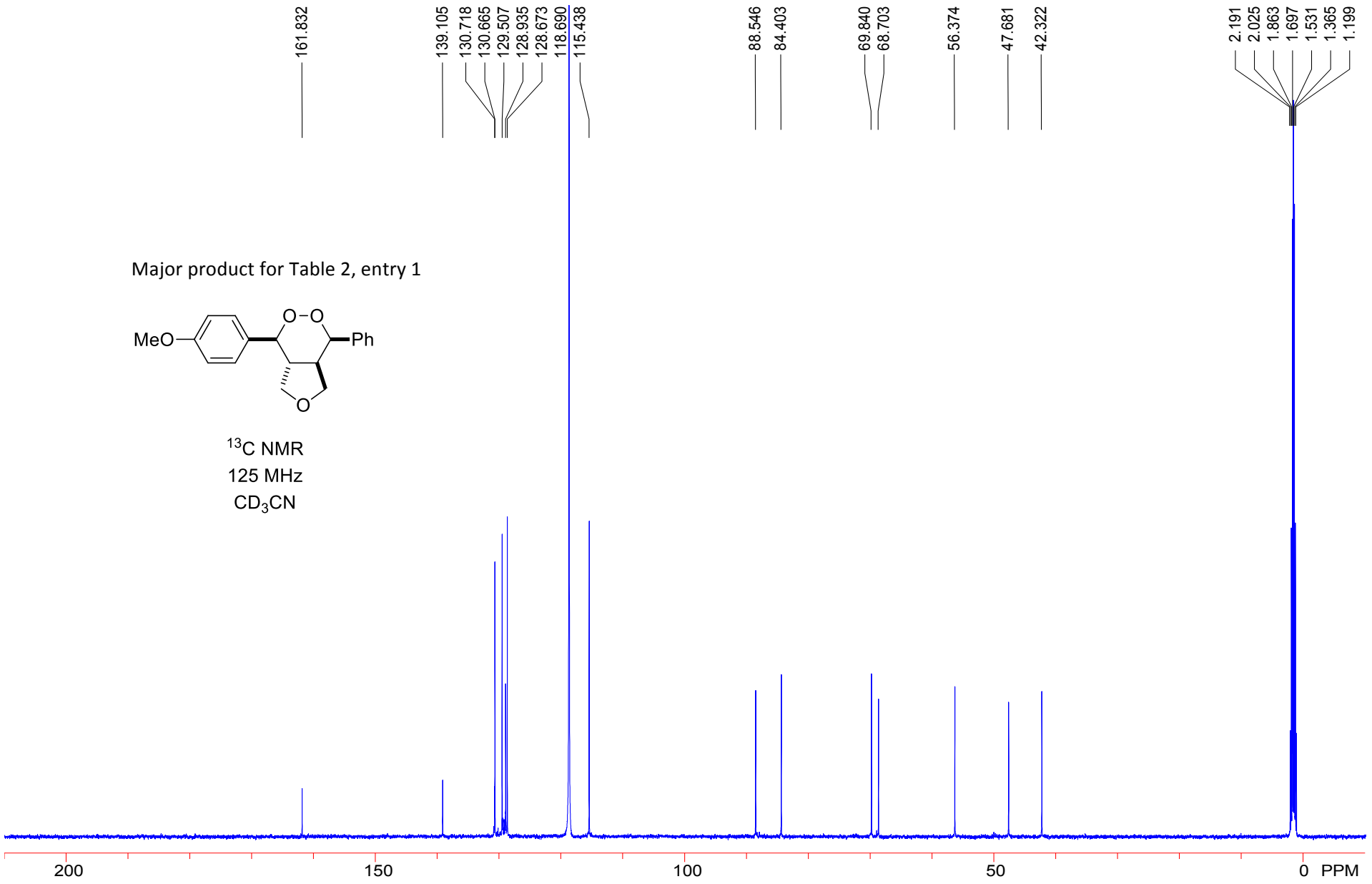
^{1}H NMR

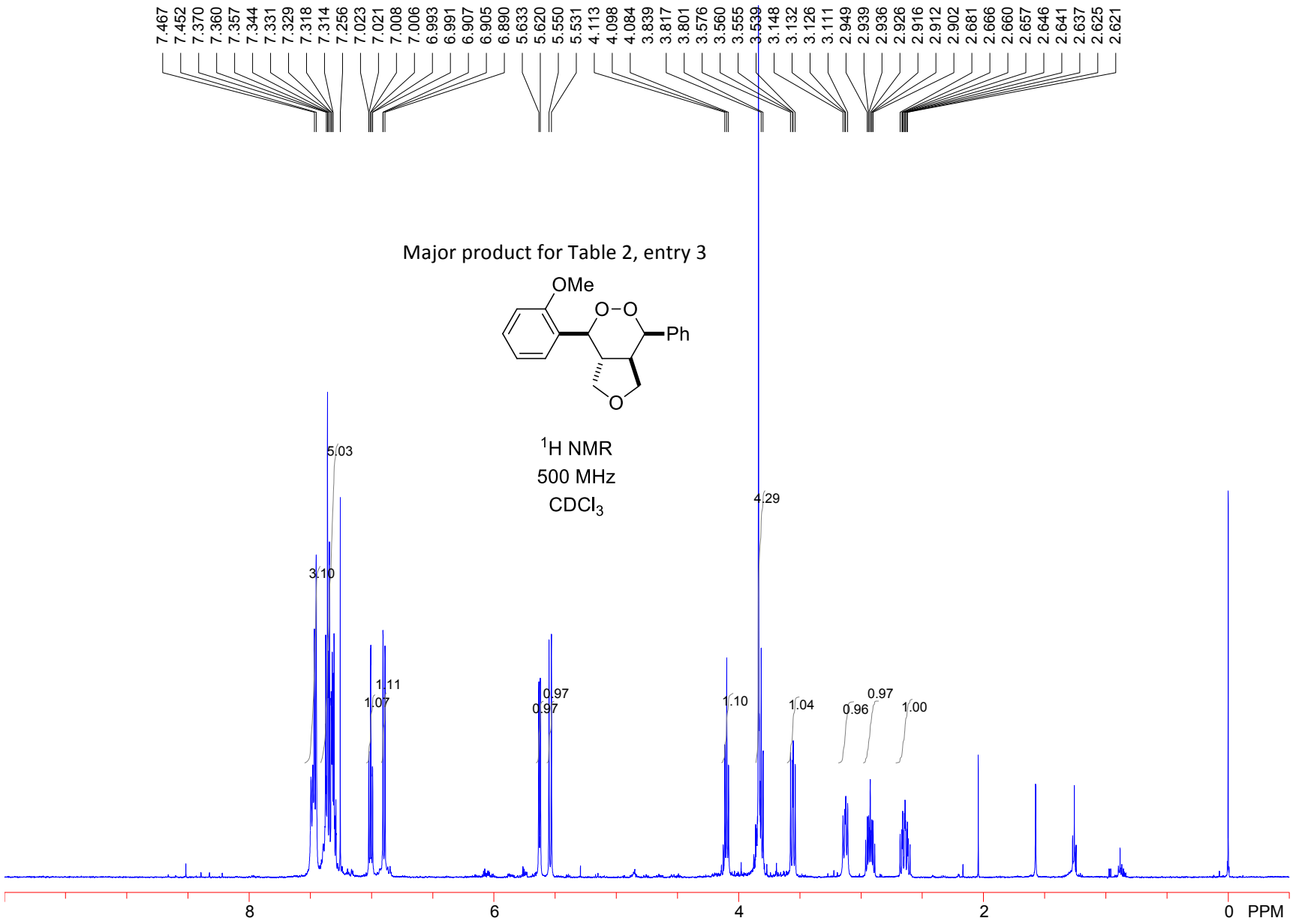
500 MHz

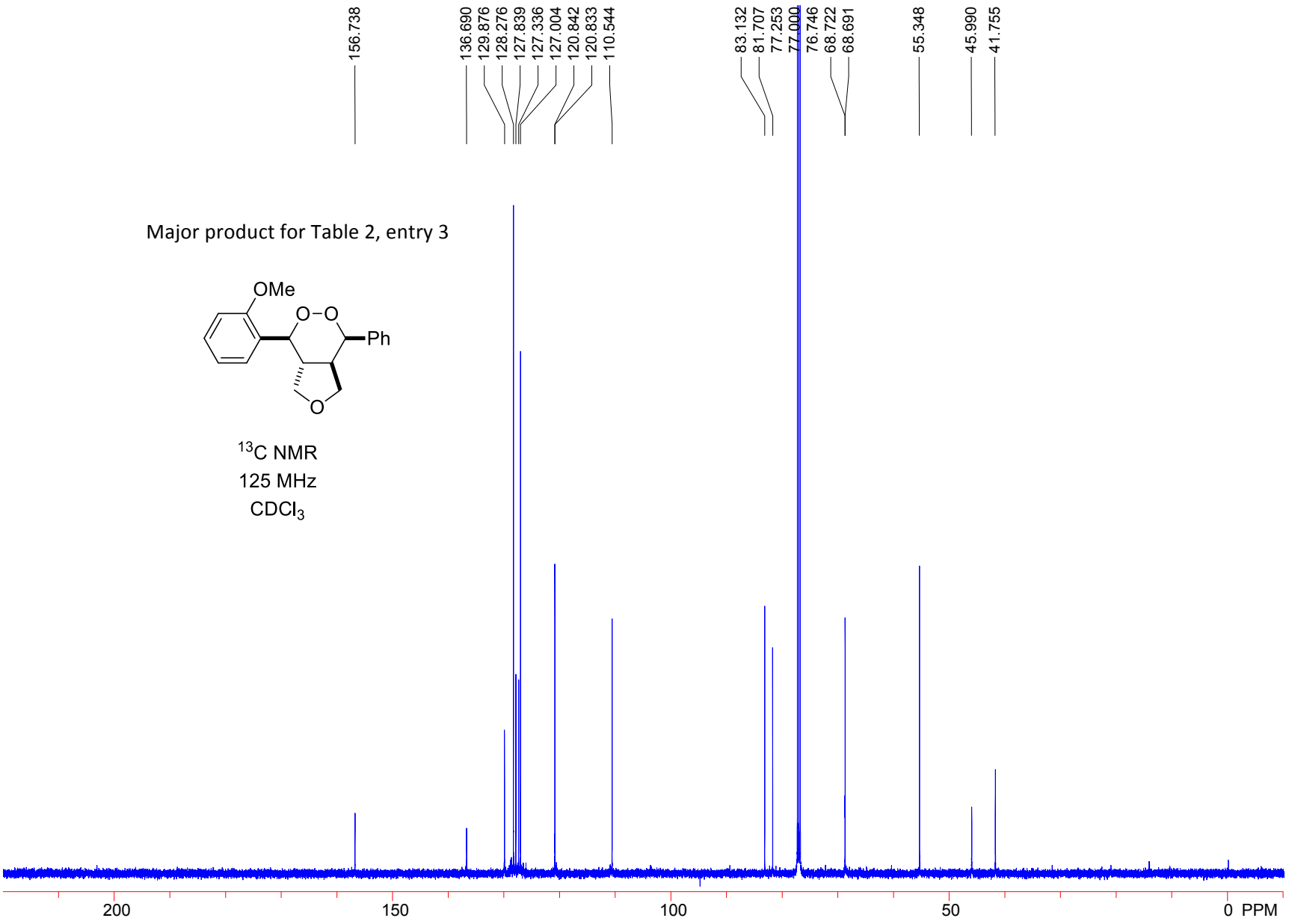
$CDCl_3$

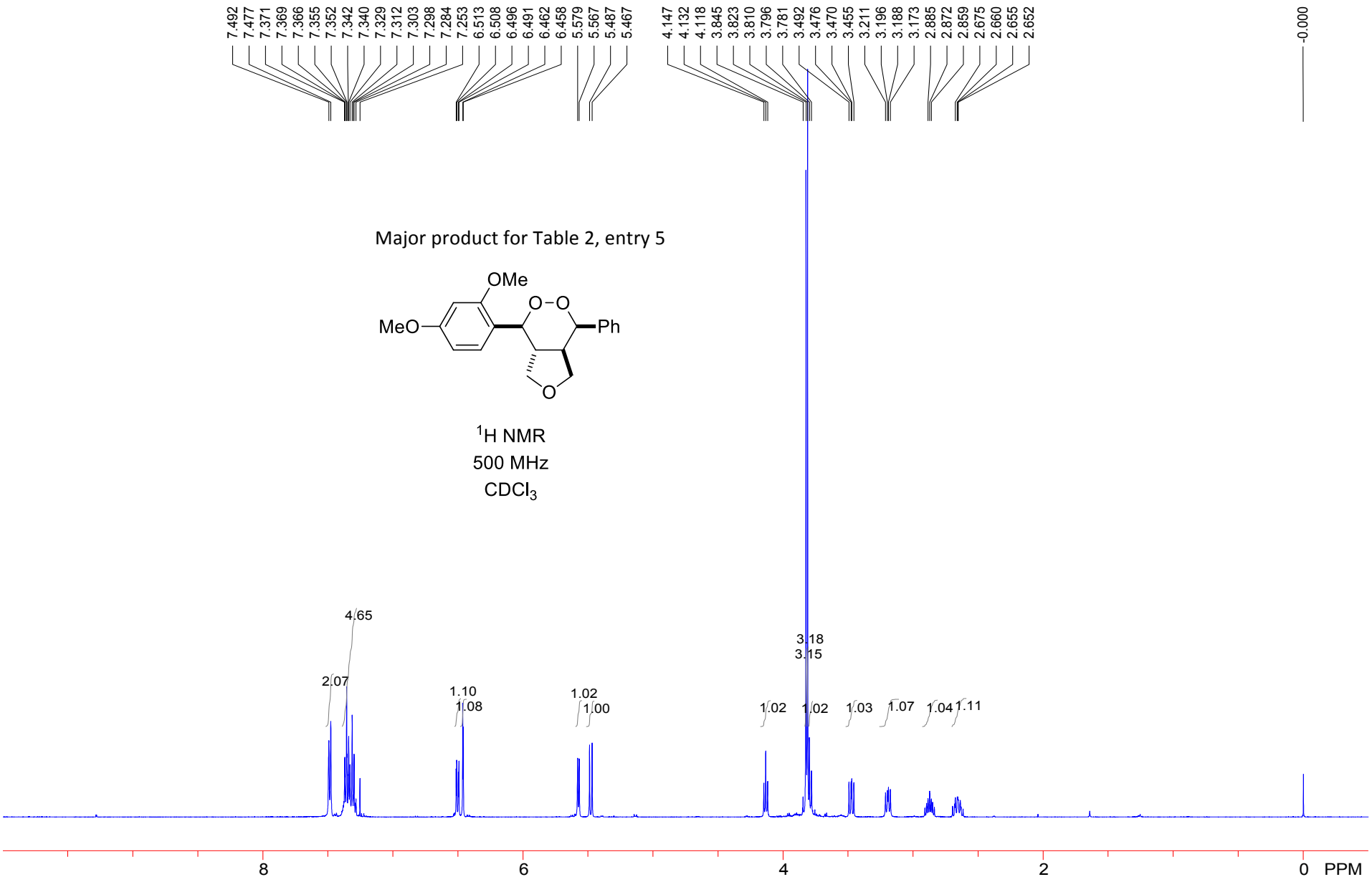

158.623
130.721
129.203
128.764
126.974
126.964
113.887
77.254
77.000
76.741
55.244
32.851
29.031
Substrate for Table 2, entry 18
MeO
OMe
13C NMR
125 MHz
CDCl3
200
150
100
50
0 PPM

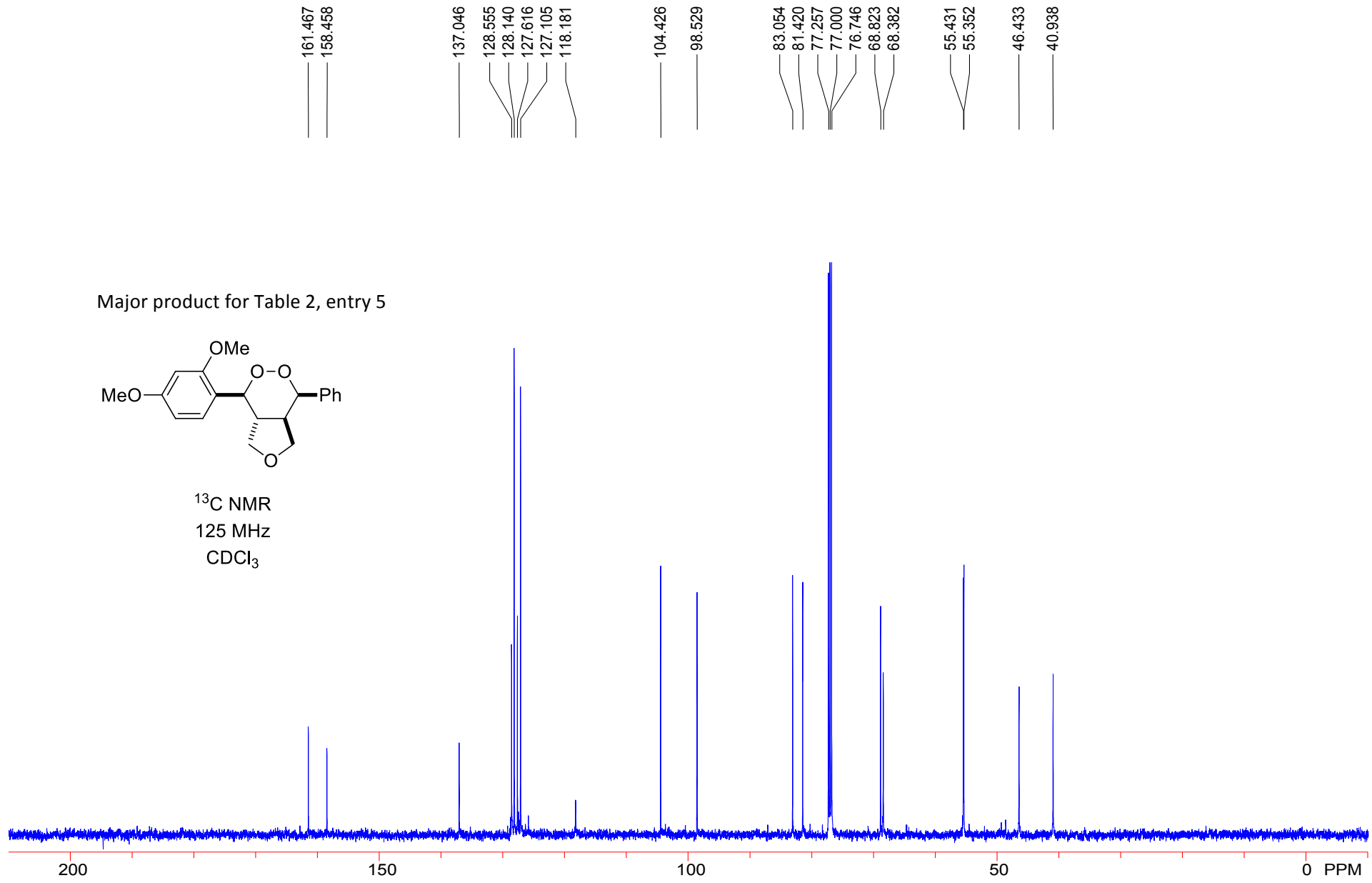

7.505
7.491
7.388
7.386
7.382
7.371
7.369
7.359
7.356
7.352
7.327
7.323
7.319
7.314
7.310
7.306
7.299
7.296
7.293
7.250
6.924
6.918
6.914
6.904
6.901
6.894
5.570
5.559
5.056
5.036
4.186
4.172
4.157
3.807
3.789
3.778
3.774
3.456
3.440
3.435
3.419
3.270
3.255
3.248
3.233
2.862
2.860
2.850
2.838
2.827
2.825
2.816
2.814
2.802
2.654
2.639
2.633
2.618
2.614
2.610
2.598
2.595
-0.000
Major product for Table 2, entry 1
MeO
O–O
Ph
O
1H NMR
500 MHz
CDCl3
3.10
2.21
2.06
2.05
0.98
0.98
1.00
4.09
0.99
0.99
1.00
1.03
8
6
4
2
0
PPM

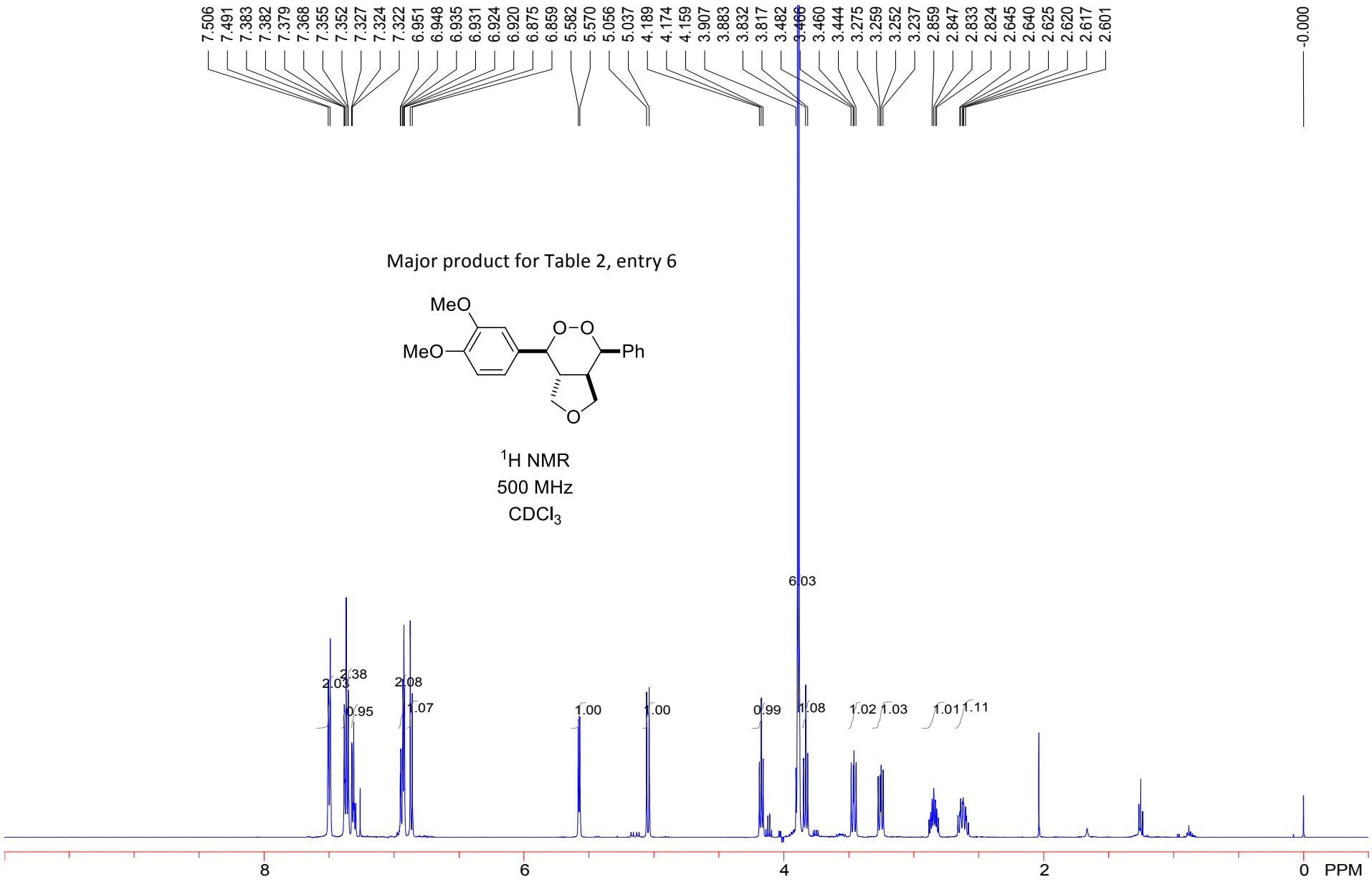

161.832
139.105
130.718
130.665
129.507
128.935
128.673
118.690
115.438
88.546
84.403
69.840
68.703
56.374
47.681
42.322
2.191
2.025
1.863
1.697
1.531
1.365
1.199
Major product for Table 2, entry 1
MeO
O
O
Ph
O
^{13}C NMR
125 MHz
CD_3CN
200
150
100
50
0 PPM

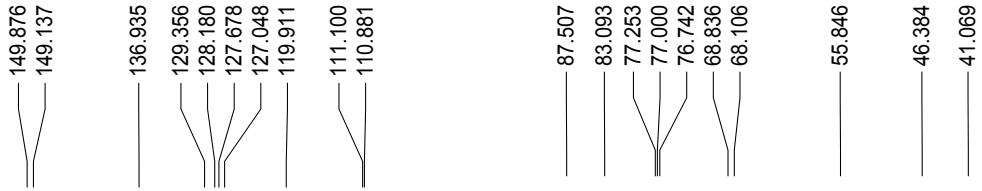

7.467
7.452
7.370
7.360
7.357
7.344
7.331
7.329
7.318
7.314
7.256
7.023
7.021
7.008
7.006
6.993
6.991
6.907
6.905
6.890
5.633
5.620
5.550
5.531
4.113
4.098
4.084
3.839
3.817
3.801
3.576
3.560
3.555
3.539
3.148
3.132
3.126
3.111
2.949
2.939
2.936
2.926
2.916
2.912
2.902
2.681
2.666
2.660
2.657
2.646
2.641
2.637
2.625
2.621
Major product for Table 2, entry 3
OMe
O–O
Ph
O
1H NMR
500 MHz
CDCl3
5.03
3.10
1.11
1.07
0.97
0.97
4.29
1.10
1.04
0.96
0.97
1.00
8
6
4
2
0
PPM


156.738
136.690
129.876
128.276
127.839
127.336
127.004
120.842
120.833
110.544
83.132
81.707
77.253
77.000
76.746
68.722
68.691
55.348
45.990
41.755
Major product for Table 2, entry 3
OMe
O–O
Ph
O
^{13}C NMR
125 MHz
$CDCl_3$
200
150
100
50
0 PPM

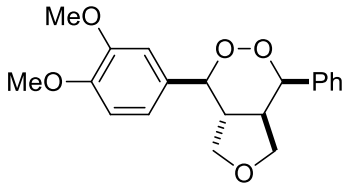

7.492
7.477
7.371
7.369
7.366
7.355
7.352
7.342
7.340
7.329
7.312
7.303
7.298
7.284
7.253
6.513
6.508
6.496
6.491
6.462
6.458
5.579
5.567
5.487
5.467
4.147
4.132
4.118
3.845
3.823
3.810
3.796
3.781
3.492
3.476
3.470
3.455
3.211
3.196
3.188
3.173
2.885
2.872
2.859
2.675
2.660
2.655
2.652
-0.000
Major product for Table 2, entry 5
OMe
O–O
MeO
Ph
O
1H NMR
500 MHz
CDCl3
4.65
2.07
1.10
1.08
1.02
1.00
3.18
3.15
1.02
1.02
1.03
1.07
1.04
1.11
8
6
4
2
0
PPM


161.467
158.458
137.046
128.555
128.140
127.616
127.105
118.181
104.426
98.529
83.054
81.420
77.257
77.000
76.746
68.823
68.382
55.431
55.352
46.433
40.938
Major product for Table 2, entry 5
OMe
O–O
MeO
Ph
O
13C NMR
125 MHz
CDCl3
200
150
100
50
0 PPM

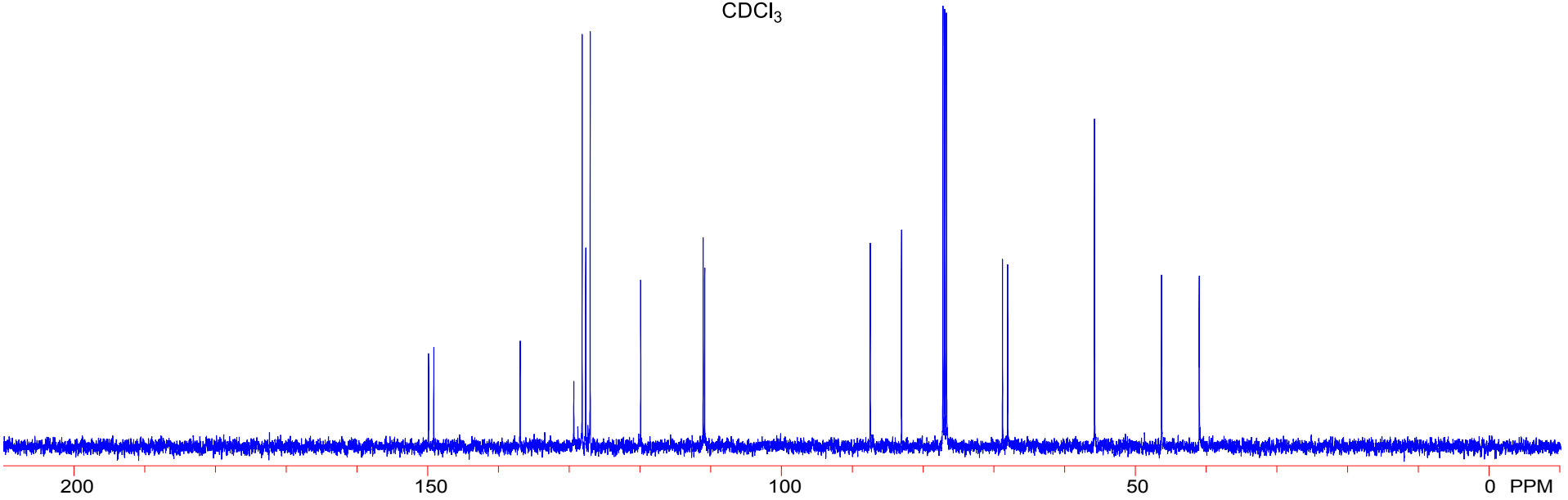

7.506
7.491
7.383
7.382
7.379
7.368
7.355
7.352
7.327
7.324
7.322
6.951
6.948
6.935
6.931
6.924
6.920
6.875
6.859
5.582
5.570
5.056
5.037
4.189
4.174
4.159
3.907
3.883
3.832
3.817
3.482
3.466
3.460
3.444
3.275
3.259
3.252
3.237
2.859
2.847
2.833
2.824
2.645
2.640
2.625
2.620
2.617
2.601
-0.000
Major product for Table 2, entry 6
MeO
MeO
O−O
Ph
O
1H NMR
500 MHz
CDCl3
6.03
2.03
2.38
2.08
0.95
1.07
1.00
1.00
0.99
1.08
1.02
1.03
1.01
1.11
8
6
4
2
0
PPM

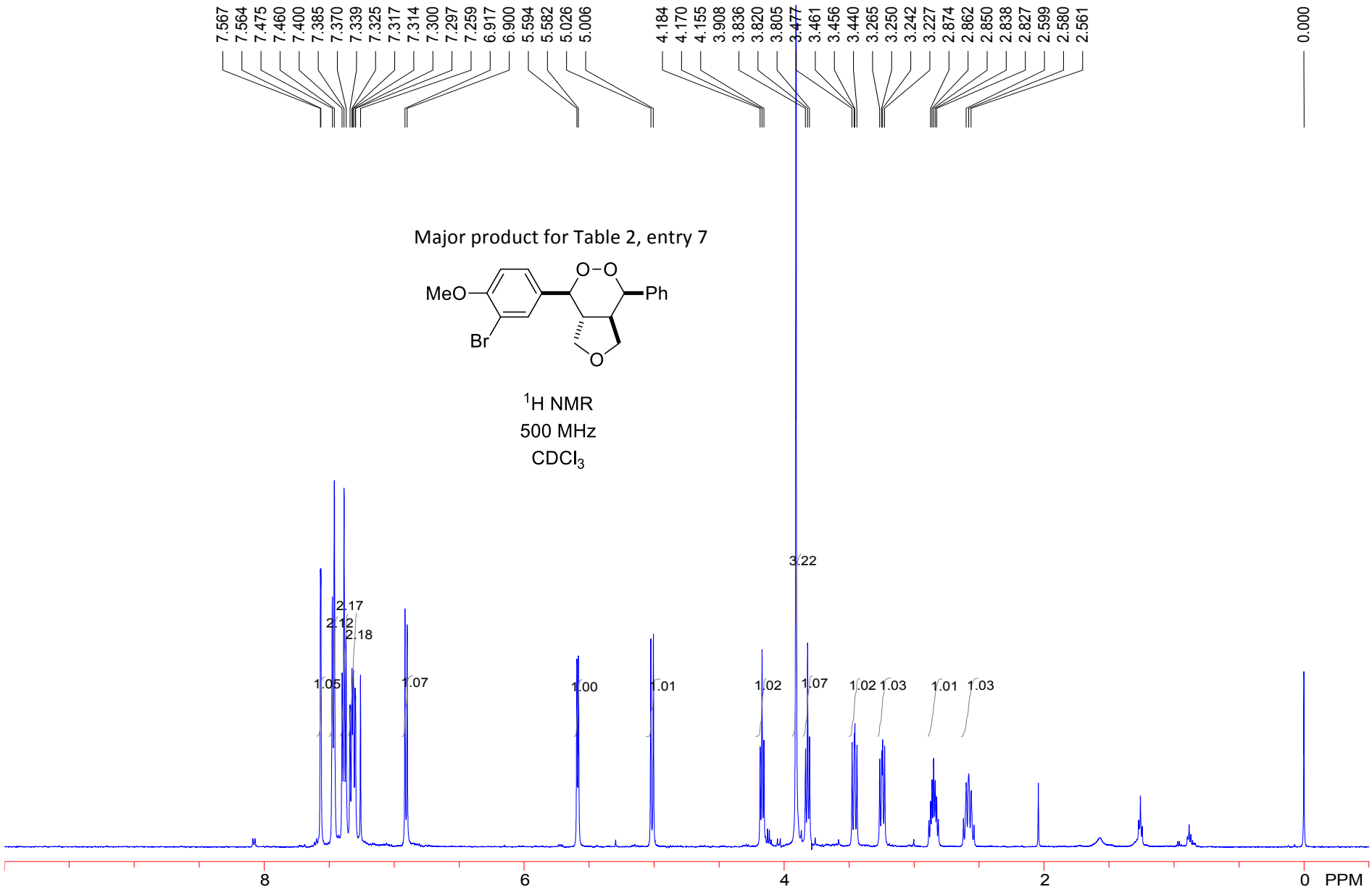


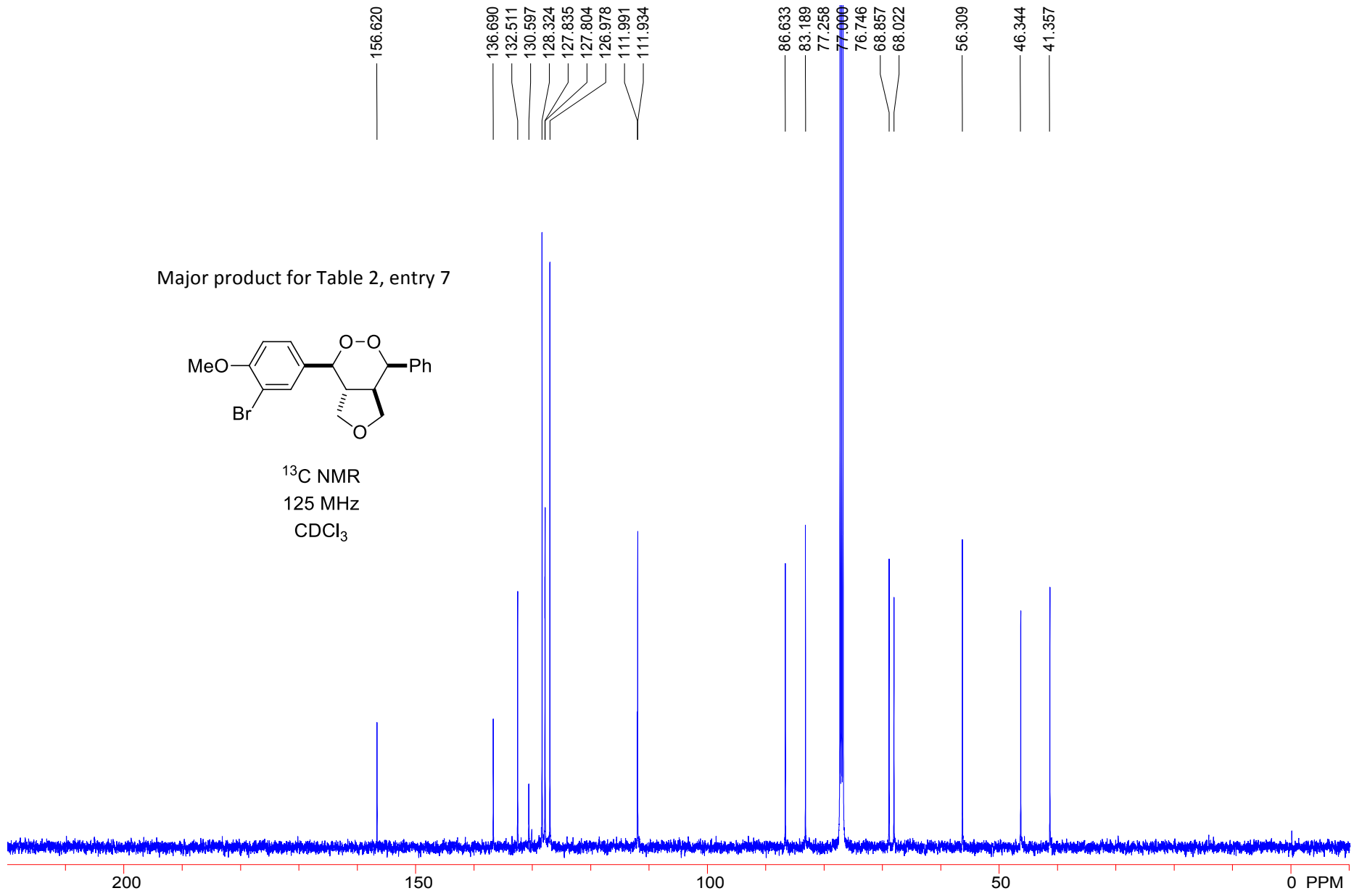
Major product for Table 2, entry 6



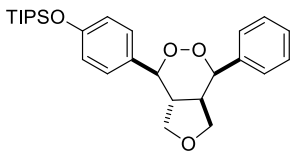
^{13}C NMR
125 MHz
$CDCl_3$




7.567
7.564
7.475
7.460
7.400
7.385
7.370
7.339
7.325
7.317
7.314
7.300
7.297
7.259
6.917
6.900
5.594
5.582
5.026
5.006
4.184
4.170
4.155
3.908
3.836
3.820
3.805
3.477
3.461
3.456
3.440
3.265
3.250
3.242
3.227
2.874
2.862
2.850
2.838
2.827
2.599
2.580
2.561
0.000
Major product for Table 2, entry 7
MeO
Br
O–O
Ph
O
1H NMR
500 MHz
CDCl3
3.22
2.17
2.12
2.18
1.05
1.07
1.00
1.01
1.02
1.07
1.02
1.03
1.01
1.03
8
6
4
2
0
PPM


156.620
136.690
132.511
130.597
128.324
127.835
127.804
126.978
111.991
111.934
86.633
83.189
77.258
77.000
76.746
68.857
68.022
56.309
46.344
41.357
Major product for Table 2, entry 7
MeO
Br
O
O
Ph
O
^{13}C NMR
125 MHz
$CDCl_3$
200
150
100
50
0 PPM

Major product for Table 2, entry 8

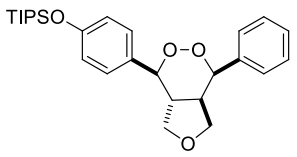


^{1}H NMR

500 MHz

$CDCl_3$

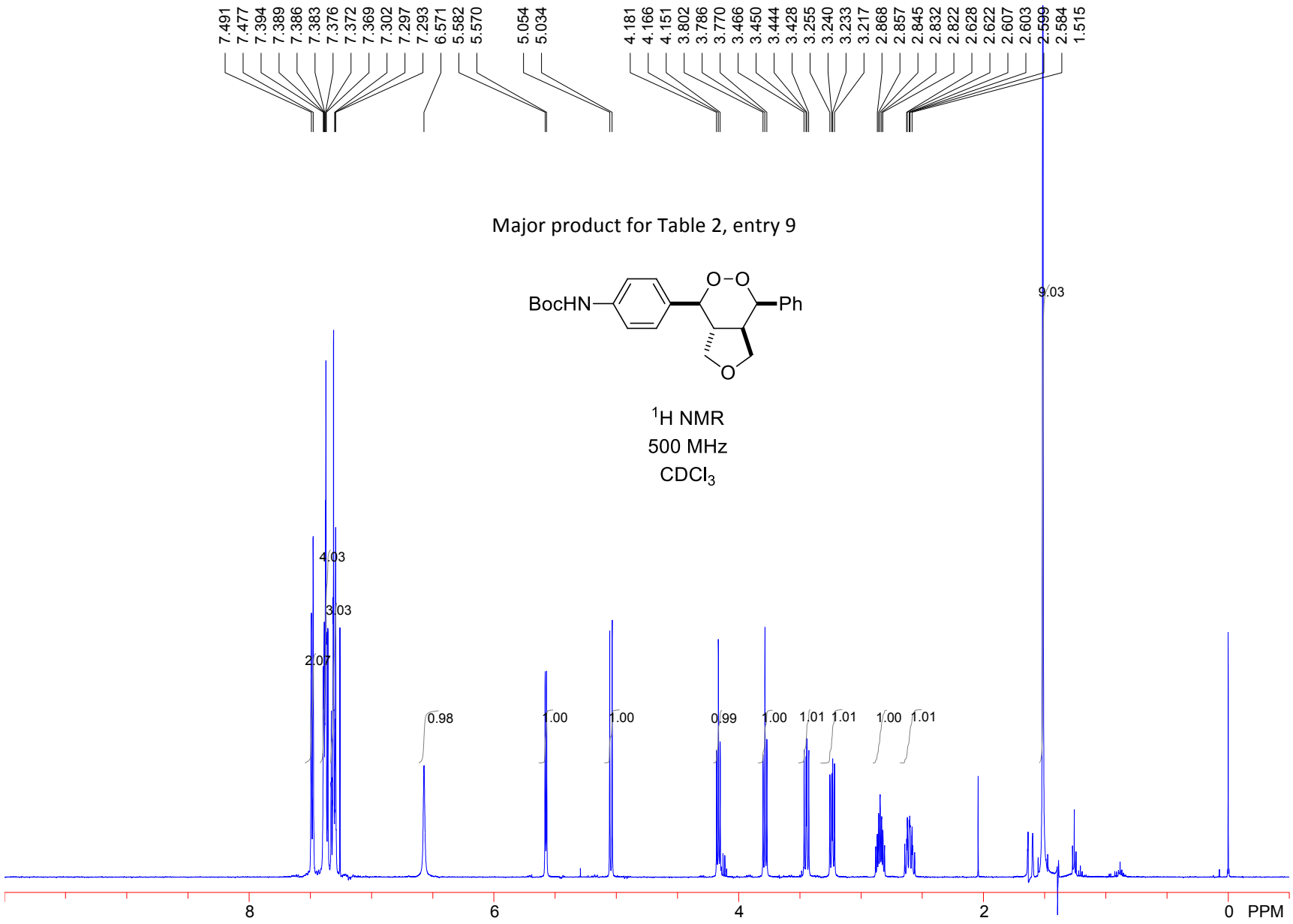
Major product for Table 2, entry 8

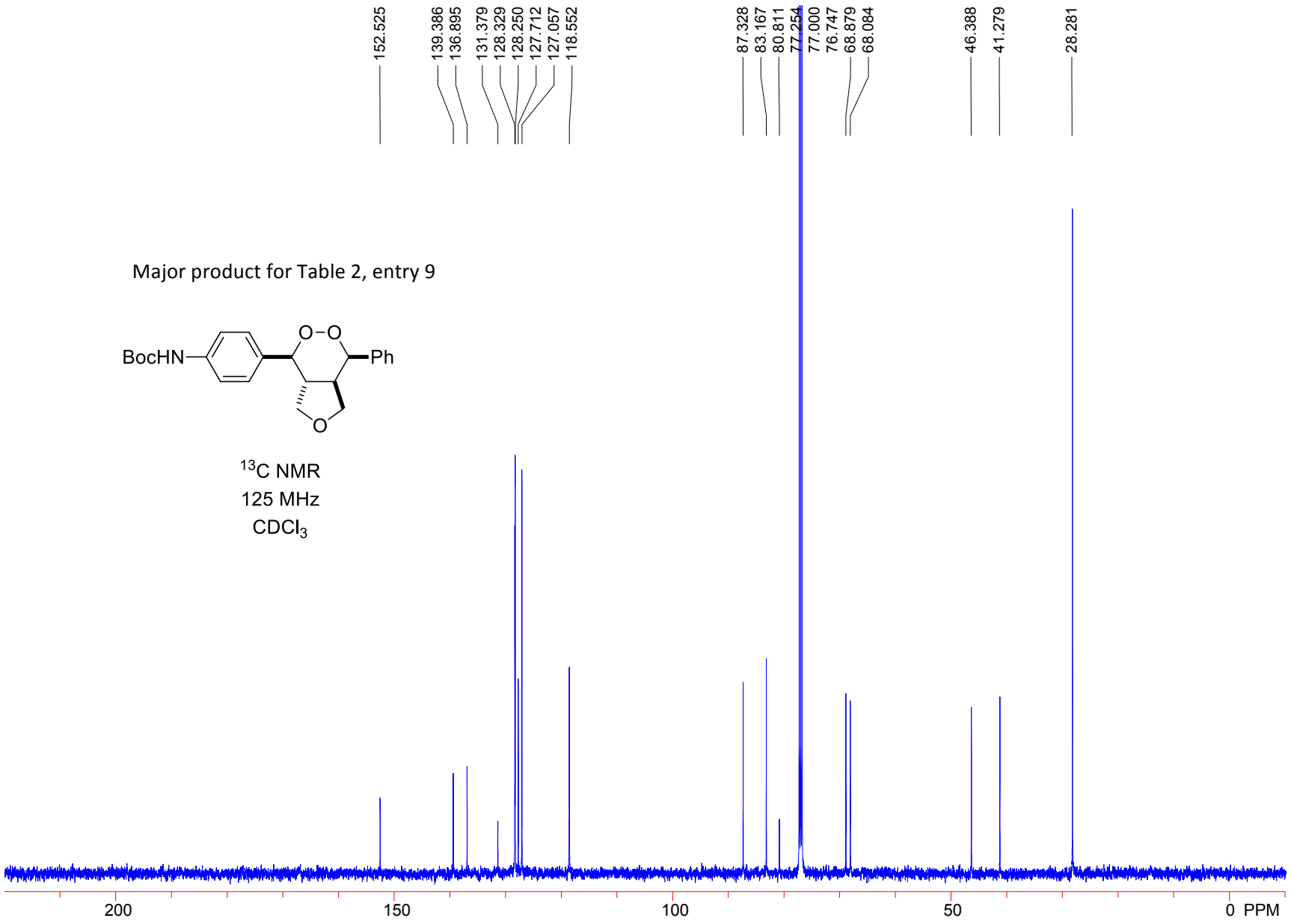


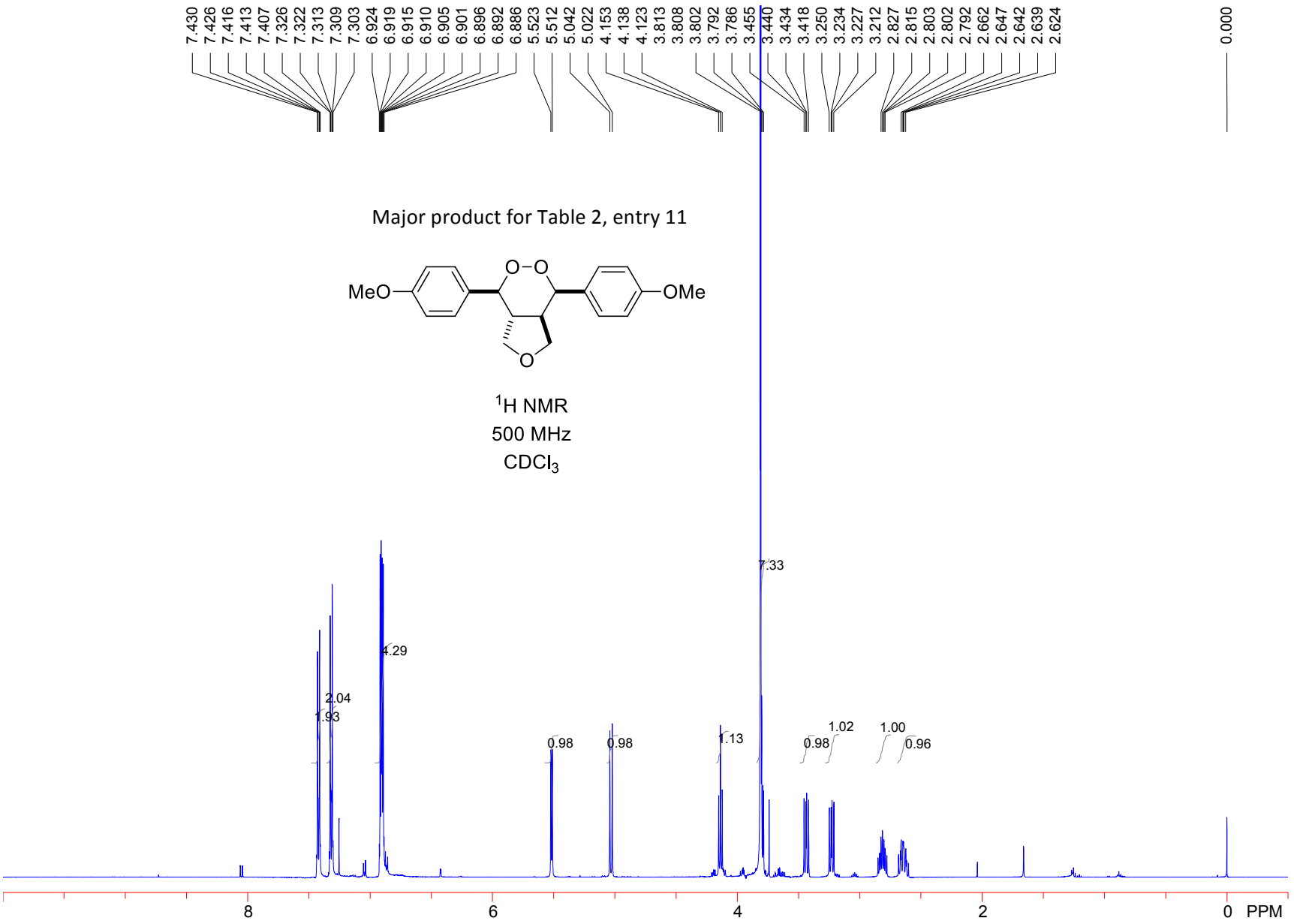
^{13}C NMR

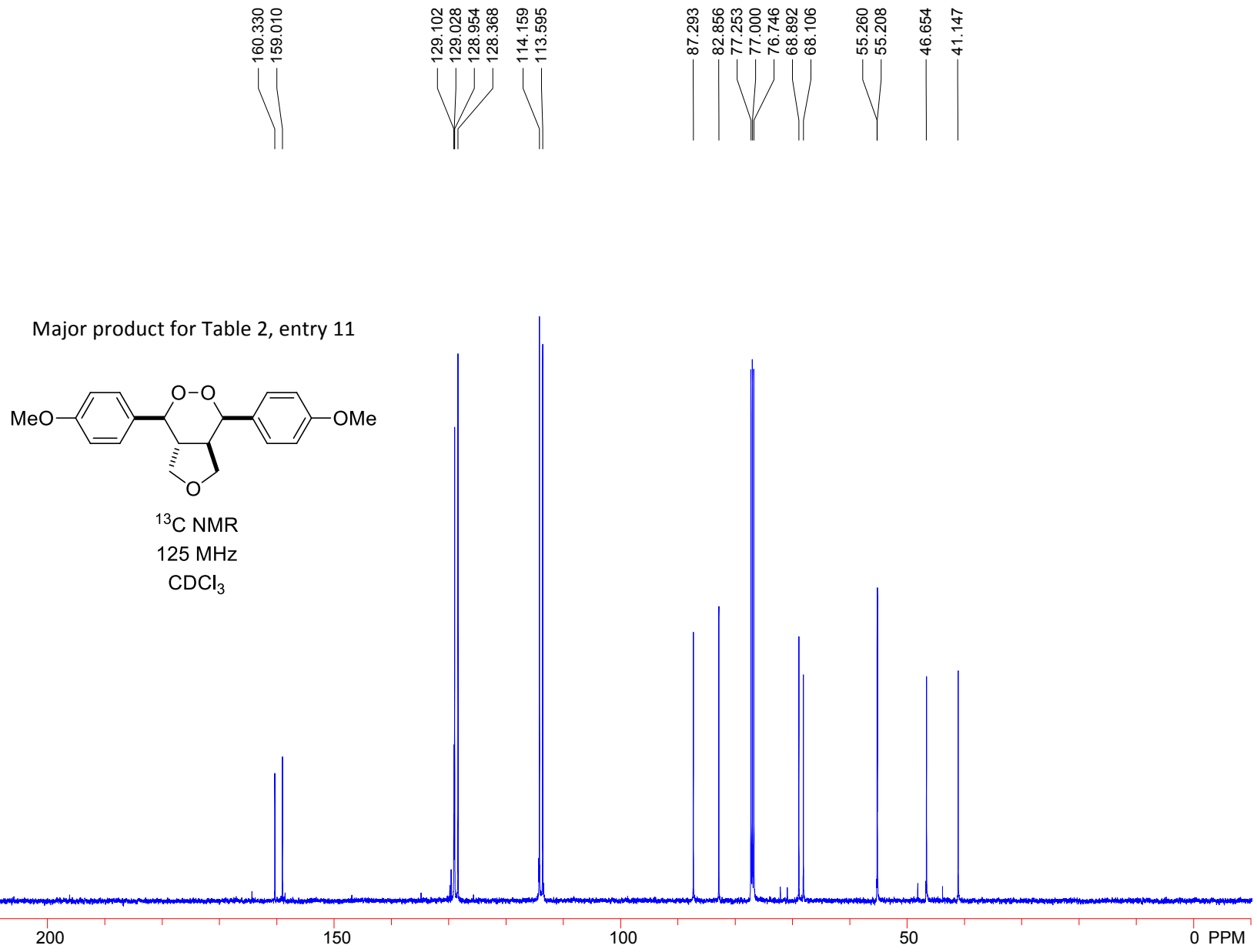
125 MHz

$CDCl_3$

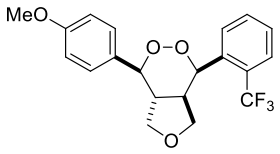

7.491
7.477
7.394
7.389
7.386
7.383
7.376
7.372
7.369
7.302
7.297
7.293
6.571
5.582
5.570
5.054
5.034
4.181
4.166
4.151
3.802
3.786
3.770
3.466
3.450
3.444
3.428
3.255
3.240
3.233
3.217
2.868
2.857
2.845
2.832
2.822
2.628
2.622
2.607
2.603
2.599
2.584
1.515
Major product for Table 2, entry 9
BocHN
O–O
Ph
O
^{1}H NMR
500 MHz
$CDCl_3$
9.03
4.03
3.03
2.07
0.98
1.00
1.00
0.99
1.00
1.01
1.01
1.00
1.01
8
6
4
2
0
PPM


152.525
139.386
136.895
131.379
128.329
128.250
127.712
127.057
118.552
87.328
83.167
80.811
77.254
77.000
76.747
68.879
68.084
46.388
41.279
28.281
Major product for Table 2, entry 9
BocHN
O–O
Ph
O
^{13}C NMR
125 MHz
$CDCl_3$
200
150
100
50
0 PPM


7.430
7.426
7.416
7.413
7.407
7.326
7.322
7.313
7.309
7.303
6.924
6.919
6.915
6.910
6.905
6.901
6.896
6.892
6.886
5.523
5.512
5.042
5.022
4.153
4.138
4.123
3.813
3.808
3.802
3.792
3.786
3.455
3.440
3.434
3.418
3.250
3.234
3.227
3.212
2.827
2.815
2.803
2.802
2.792
2.662
2.647
2.642
2.639
2.624
0.000
Major product for Table 2, entry 11
MeO
O–O
OMe
O
1H NMR
500 MHz
CDCl3
7.33
4.29
2.04
1.93
0.98
0.98
1.13
0.98
1.02
1.00
0.96
8
6
4
2
0
PPM


160.330
159.010
129.102
129.028
128.954
128.368
114.159
113.595
87.293
82.856
77.253
77.000
76.746
68.892
68.106
55.260
55.208
46.654
41.147
Major product for Table 2, entry 11
MeO
O–O
OMe
O
^{13}C NMR
125 MHz
$CDCl_3$
200
150
100
50
0 PPM

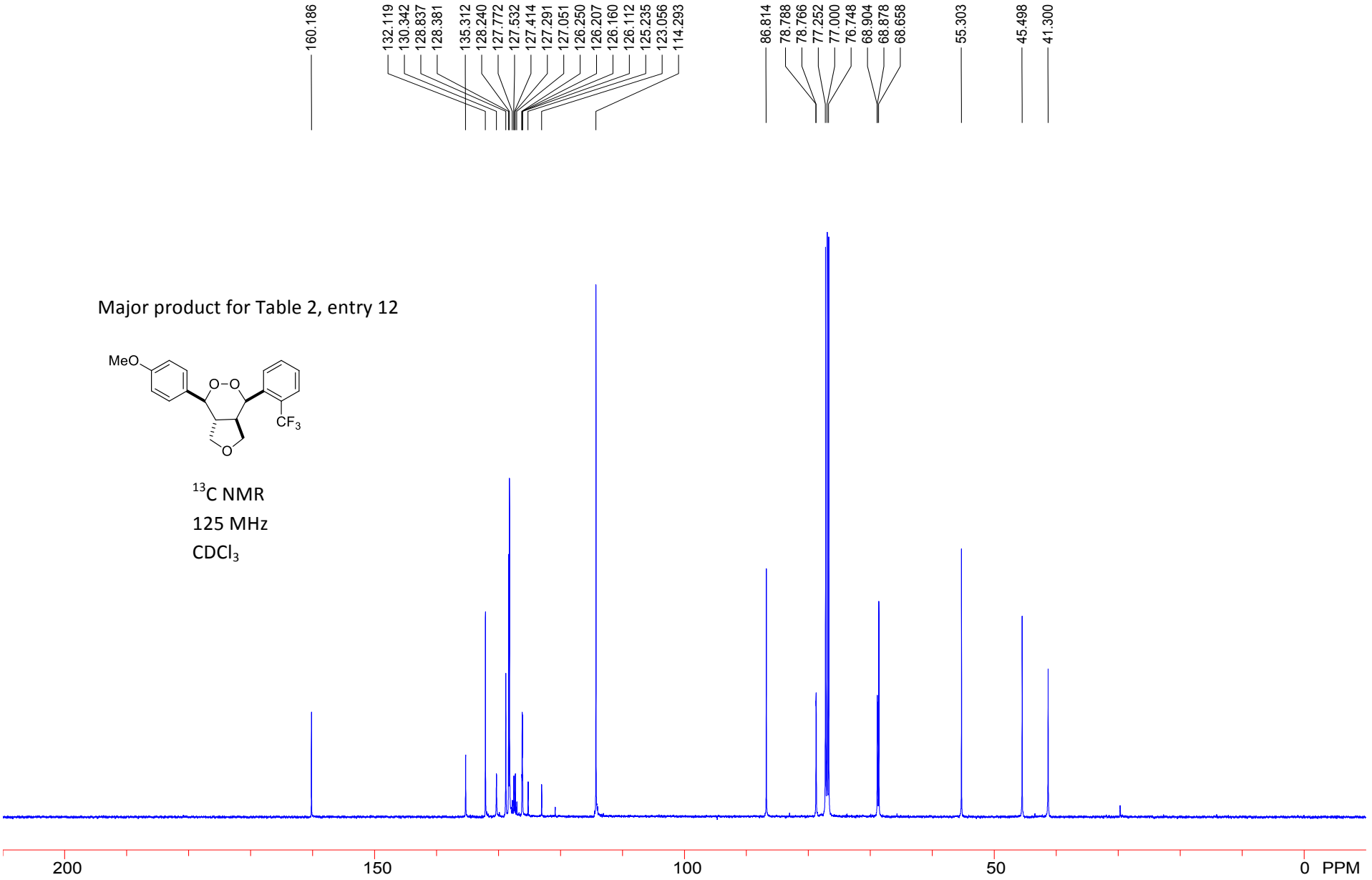
Major product for Table 2, entry 12



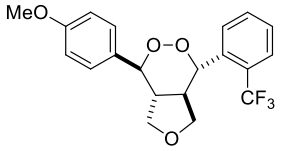
^{1}H NMR

500 MHz

$CDCl_3$


160.186
132.119
130.342
128.837
128.381
135.312
128.240
127.772
127.532
127.414
127.291
127.051
126.250
126.207
126.160
126.112
125.235
123.056
114.293
86.814
78.788
78.766
77.252
77.000
76.748
68.904
68.878
68.658
55.303
45.498
41.300
Major product for Table 2, entry 12
MeO
O-O
CF3
O
13C NMR
125 MHz
CDCl3
200
150
100
50
0 PPM

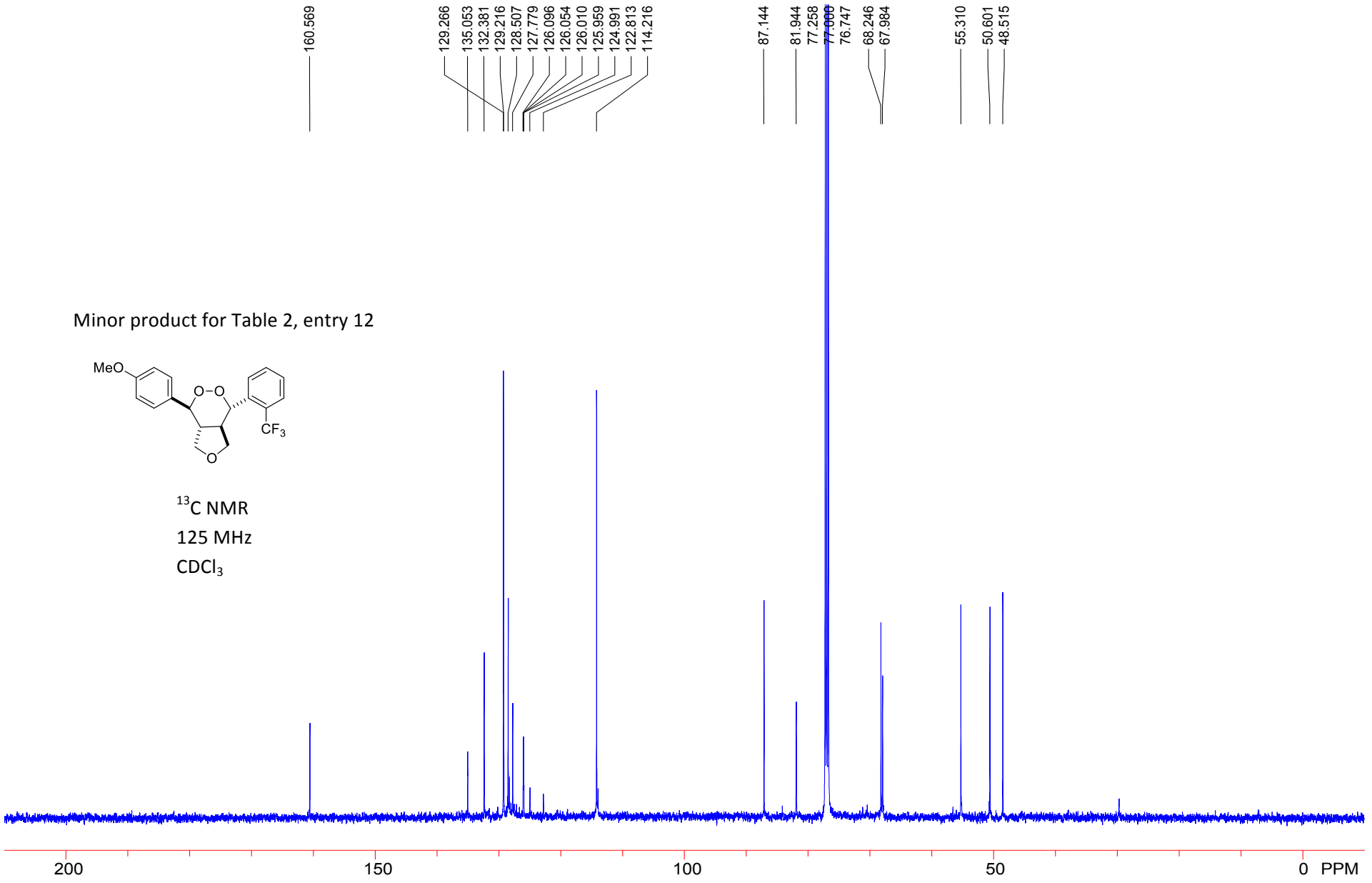
Minor product for Table 2, entry 12



^{1}H NMR

500 MHz

$CDCl_3$



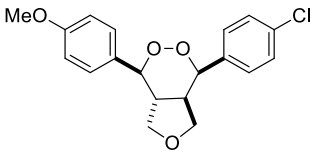
Minor product for Table 2, entry 12

^{13}C NMR

125 MHz

$CDCl_3$

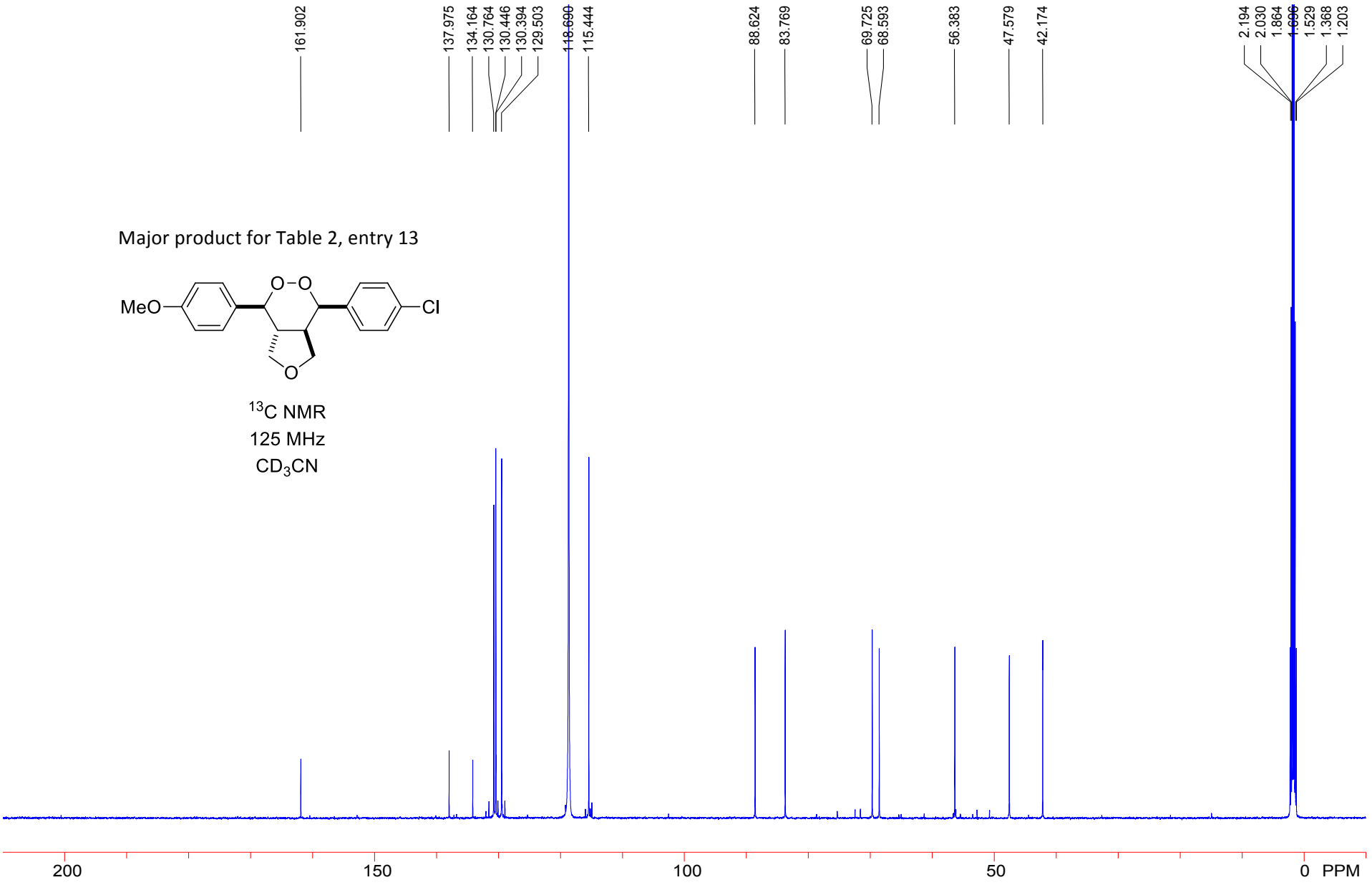
Major product for Table 2, entry 13



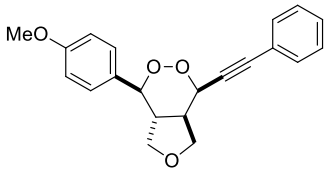
^{1}H NMR

500 MHz

$CDCl_3$


161.902
137.975
134.164
130.764
130.446
130.394
129.503
118.690
115.444
88.624
83.769
69.725
68.593
56.383
47.579
42.174
2.194
2.030
1.864
1.529
1.368
1.203
Major product for Table 2, entry 13
MeO
O–O
Cl
O
^{13}C NMR
125 MHz
CD_3CN
200
150
100
50
0 PPM

Major product for Table 2, entry 14

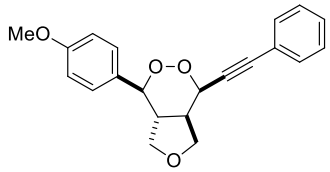


^{1}H NMR

500 MHz

$CDCl_3$

Major product for Table 2, entry 14

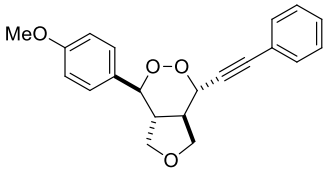


^{13}C NMR

125 MHz

$CDCl_3$

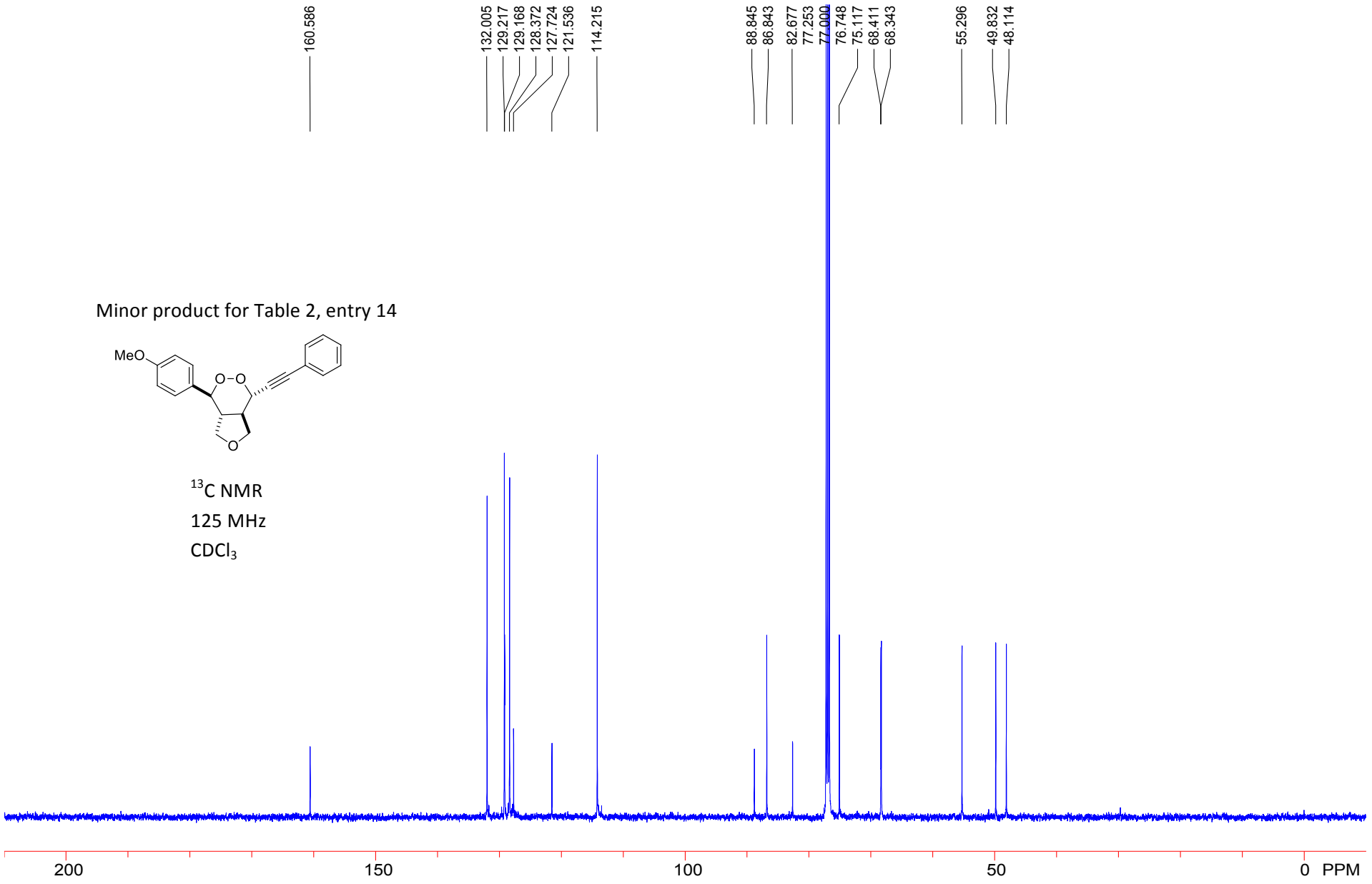
Minor product for Table 2, entry 14



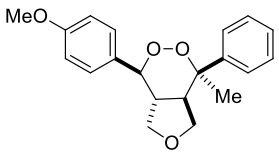
^{1}H NMR

500 MHz

$CDCl_3$


160.586
132.005
129.217
129.168
128.372
127.724
121.536
114.215
88.845
86.843
82.677
77.253
77.000
76.748
75.117
68.411
68.343
55.296
49.832
48.114
Minor product for Table 2, entry 14
MeO
O
O
O
^{13}C NMR
125 MHz
$CDCl_3$
200
150
100
50
0 PPM

Major product for Table 2, entry 15

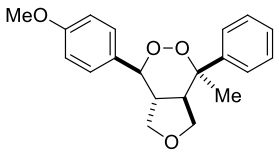


^{1}H NMR

500 MHz

$CDCl_3$

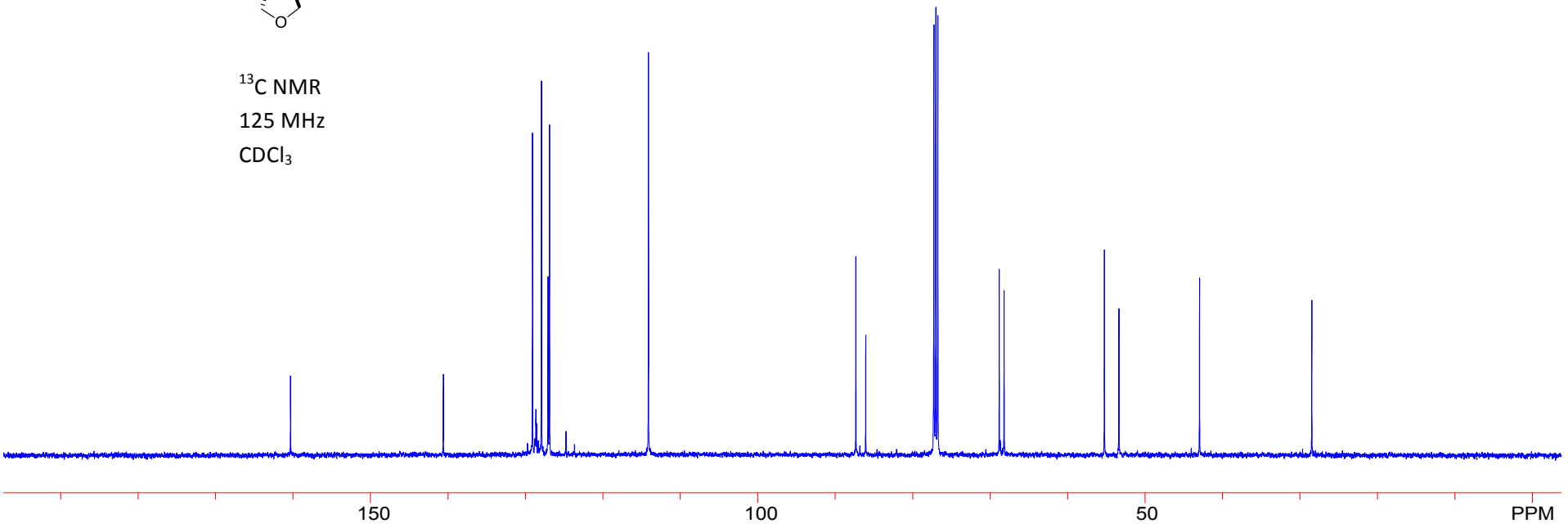
Major product for Table 2, entry 15



^{13}C NMR

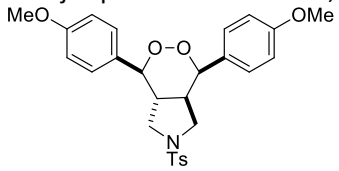
125 MHz

$CDCl_3$



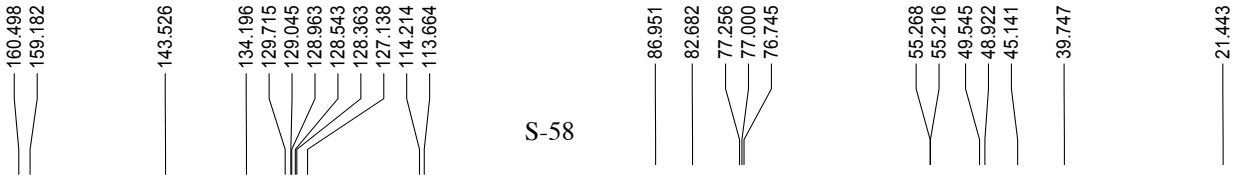
Major product for Table 2, entry 16

Major product for Table 2, entry 16



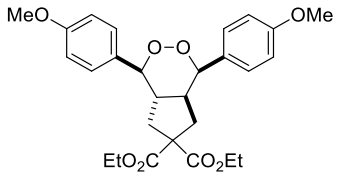
^{1}H NMR
500 MHz
$CDCl_3$

^{13}C NMR
125 MHz
$CDCl_3$



Major product for Table 2, entry 17

Major product for Table 2, entry 17



^{13}C NMR

125 MHz

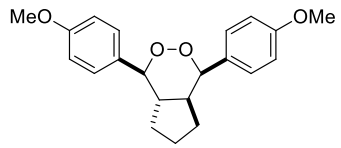
$CDCl_3$

^{1}H NMR

500 MHz

$CDCl_3$

Major product for Table 2, entry 18

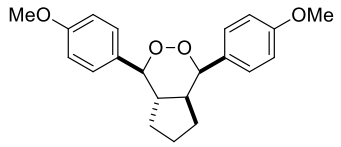


^{1}H NMR

500 MHz

$CDCl_3$

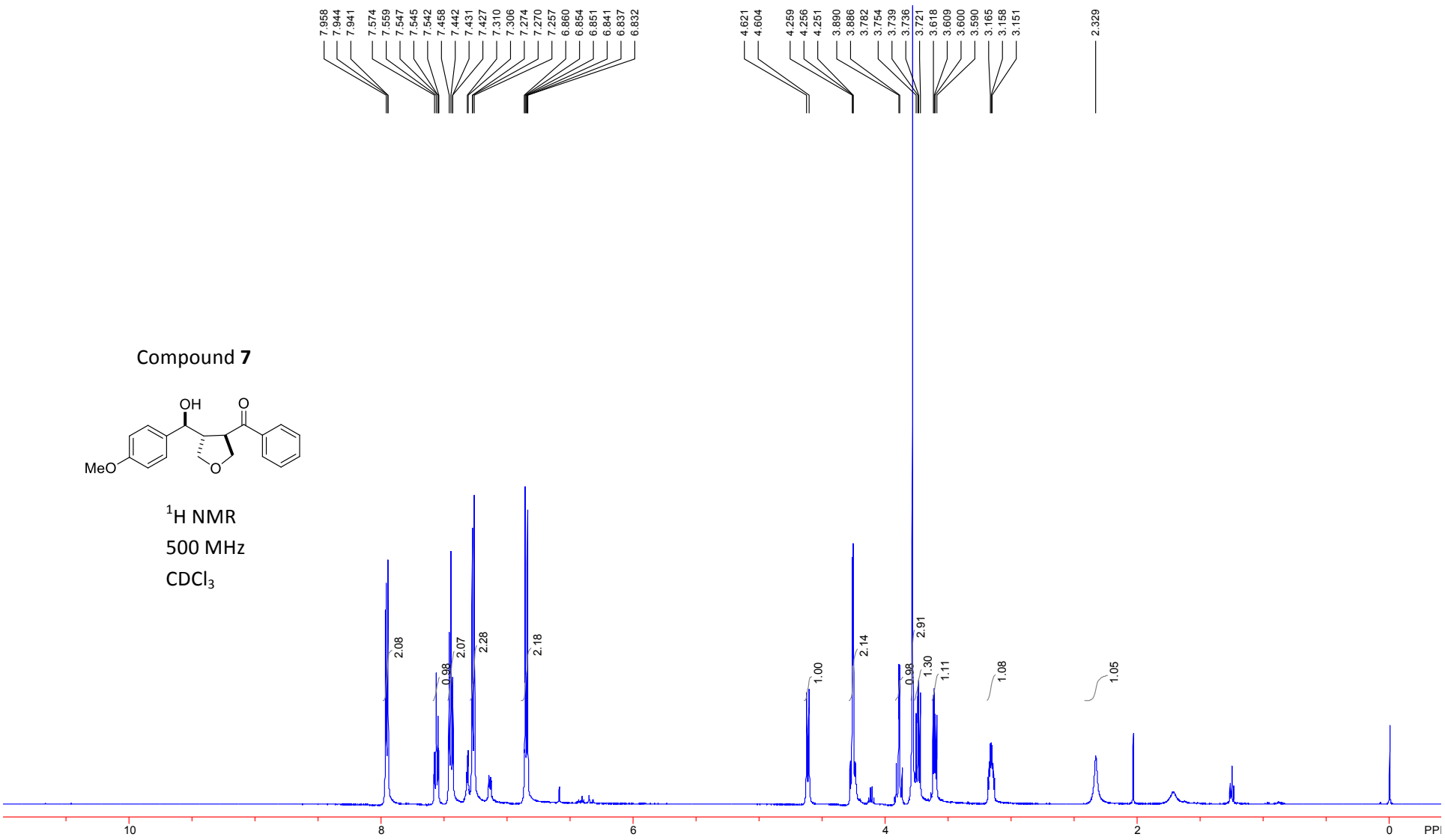
Major product for Table 2, entry 18

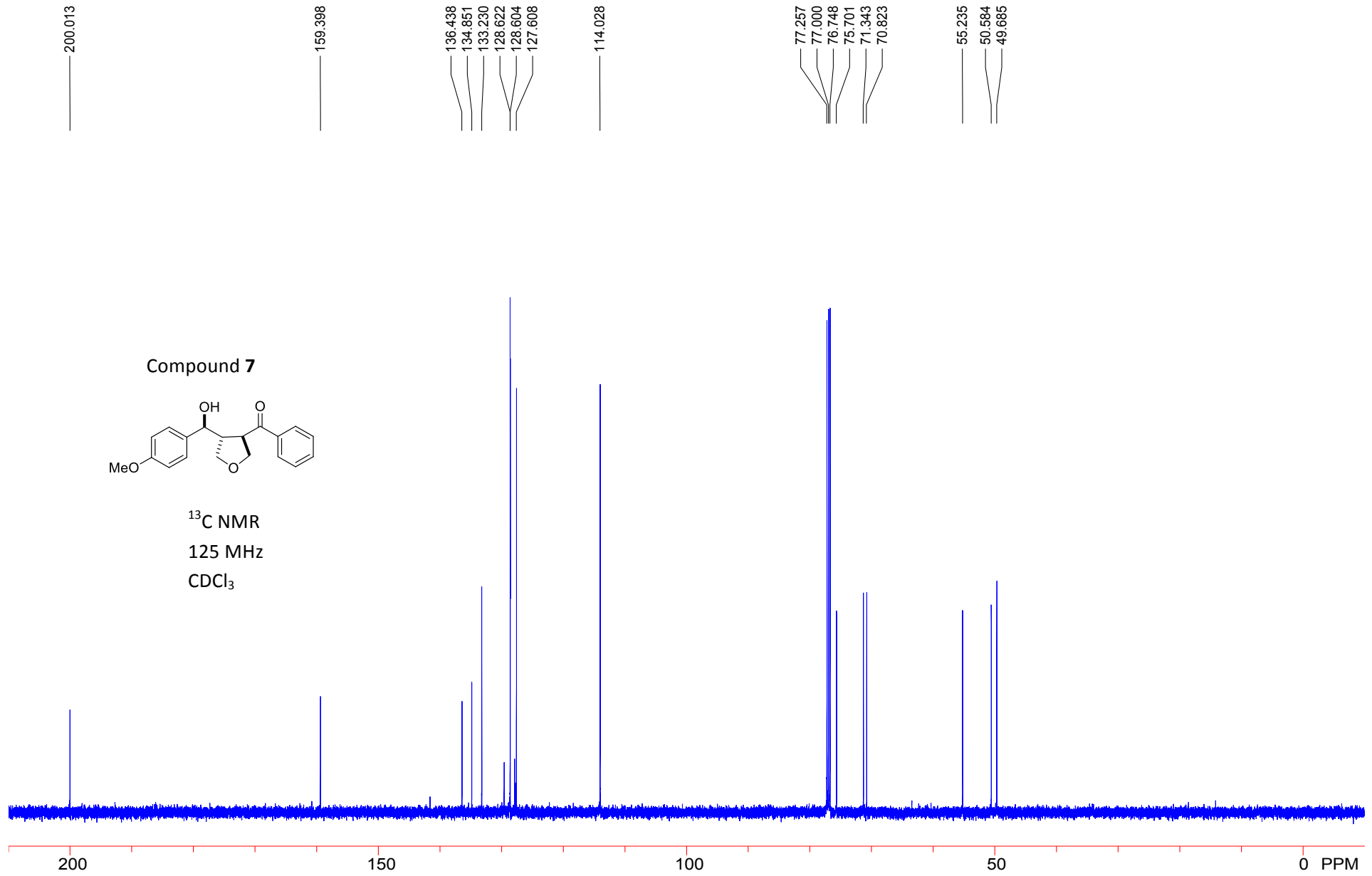


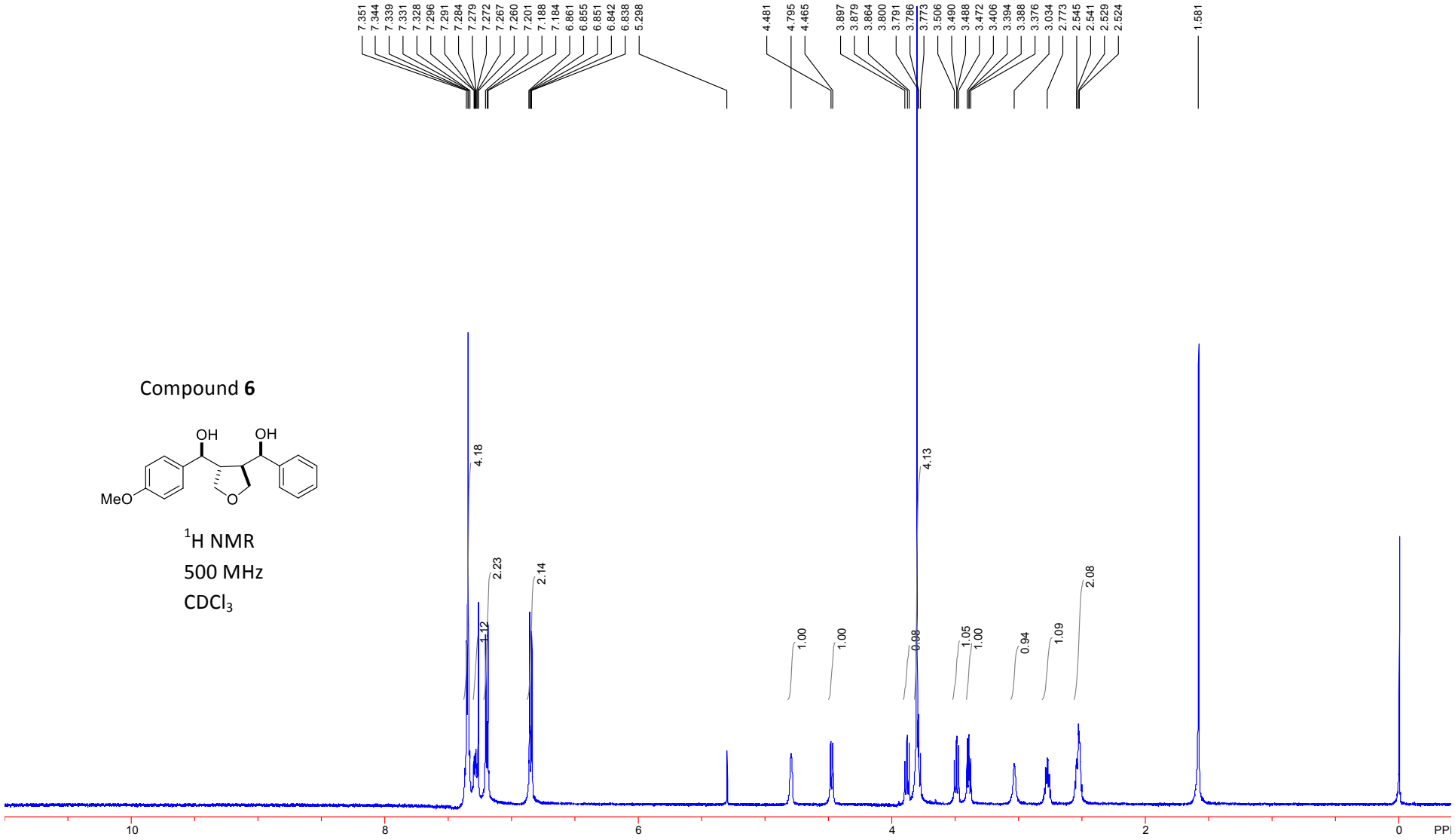
^{13}C NMR

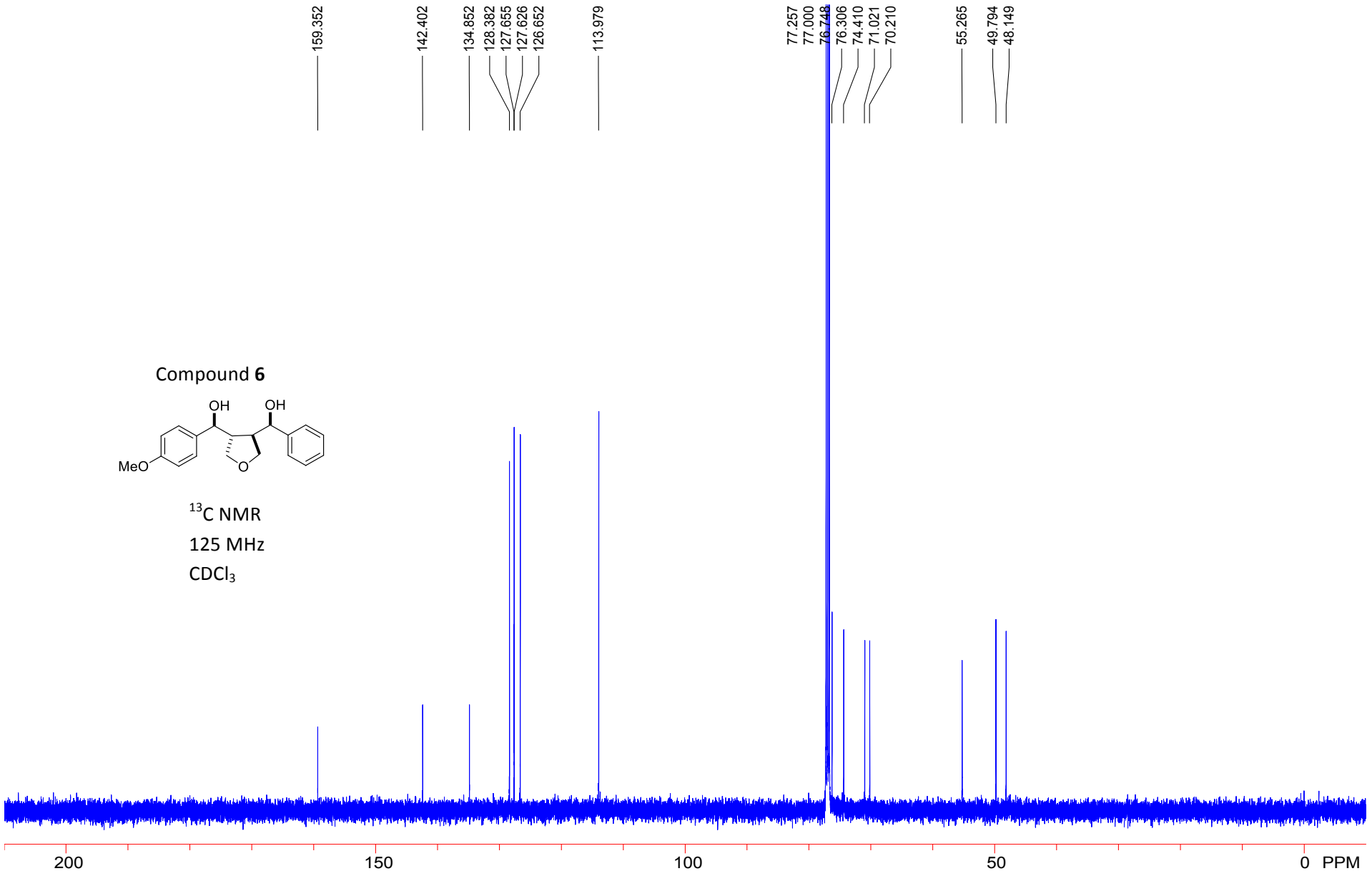
125 MHz

$CDCl_3$


7.958
7.944
7.941
7.574
7.559
7.547
7.545
7.542
7.458
7.442
7.431
7.427
7.310
7.306
7.274
7.270
7.257
6.860
6.854
6.851
6.841
6.837
6.832
4.621
4.604
4.259
4.256
4.251
3.890
3.886
3.782
3.754
3.739
3.736
3.721
3.618
3.609
3.600
3.590
3.165
3.158
3.151
2.329
Compound 7
OH
O
MeO
O
1H NMR
500 MHz
CDCl3
2.08
0.98
2.07
2.28
2.18
1.00
2.14
0.98
2.91
1.30
1.11
1.08
1.05
10
8
6
4
2
0
PPI


200.013
159.398
136.438
134.851
133.230
128.622
128.604
127.608
114.028
77.257
77.000
76.748
75.701
71.343
70.823
55.235
50.584
49.685
Compound 7
OH
O
MeO
O
13C NMR
125 MHz
CDCl3
200
150
100
50
0 PPM


7.351
7.344
7.339
7.331
7.328
7.296
7.291
7.284
7.279
7.272
7.267
7.260
7.201
7.188
7.184
6.861
6.855
6.851
6.842
6.838
5.298
4.481
4.795
4.465
3.897
3.879
3.864
3.800
3.791
3.786
3.773
3.506
3.490
3.488
3.472
3.406
3.394
3.388
3.376
3.034
2.773
2.545
2.541
2.529
2.524
1.581
Compound 6
OH
OH
MeO
O
1H NMR
500 MHz
CDCl3
4.18
2.23
1.12
2.14
1.00
1.00
4.13
0.08
1.05
1.00
0.94
1.09
2.08
10
8
6
4
2
0
PPI


159.352
142.402
134.852
128.382
127.655
127.626
126.652
113.979
77.257
77.000
76.748
76.306
74.410
71.021
70.210
55.265
49.794
48.149
Compound 6
OH
OH
MeO
O
13C NMR
125 MHz
CDCl3
200
150
100
50
0 PPM