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Supporting Information

Promoter Free Allylation of Trichloroacetimidates with Allyltributylstannanes Under Thermal Conditions to Access the Common 1,1'-Diarylbutyl Pharmacophore

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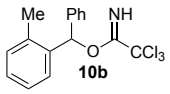
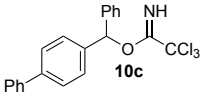
General Procedure for Trichloroacetimidate Synthesis from an alcohol. A flame dried 25 mL round bottom flask was charged with the alcohol starting material (1 equiv) under argon. Dry DCM was then added to form a 0.5 M solution, and the flask was cooled to 0°C. 1,8-Diazabicyclo[5.4.0]undec-7-ene (0.2 equiv) was added to the solution, followed by trichloroacetonitrile (1.5 equiv). The reaction was monitored by TLC. After no alcohol starting material was observed, the reaction mixture was concentrated *in vacuo*. Purification by silica gel column chromatography was performed using the listed solvent system to provide the desired trichloroacetimidate. Trichloroacetimidates **10b**, **10c**, **10e**, **10f**, **10g**, **10i**, **10j**, **10k**, **10l**, and **10o** were prepared by the general procedure. Trichloroacetimidates **10a**,[1] **10d**,[2] **10h**,[3] **10m,**[4] **10n,**[5] **10p**[6] and **10q**[7] were prepared as reported previously in the literature.



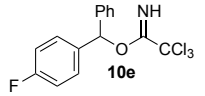
10b

(2-Methylphenyl)(phenyl)methyl 2,2,2-trichloroacetimidate (10b). Purified by silica gel chromatography (5% ethyl acetate/ 1% triethylamine/ 94% hexanes); Clear oil (2.38 g, 92%); TLC R_f = 0.59 (10% ethyl acetate/ 90% hexanes); IR (CH_2Cl_2) 3339, 3053, 3032, 2983, 1664, 1286, 1265, 1075, 913, 741 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.39 (brs, 1H), 7.46-7.43 (m, 1H), 7.38-7.28 (m, 5H), 7.24-7.16 (m, 3H), 7.12 (s, 1H), 2.37 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.3, 138.9, 137.5, 136.2, 130.5, 128.4, 128.1, 128.0, 127.3, 127.2, 126.1, 91.6, 79.0, 19.4. Anal. calcd for $C_{16}H_{14}Cl_3NO$: C, 56.09; H, 4.12; N, 4.09. Found: C, 56.00; H, 3.96; N, 4.44.



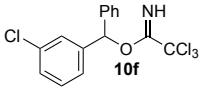
10c

[1,1'-Biphenyl]-4-yl](phenyl)methyl 2,2,2-trichloroacetimidate (10c). Purified by silica gel chromatography (1% triethylamine/ 99% hexanes); White solid (0.35 g, 75%); IR (CH_2Cl_2) 3339, 3052, 1664, 1493, 1265, 1075, 984, 798, 739, 703 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.42 (brs, 1H), 7.58-7.29 (m, 14H), 6.97 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.4, 141.0, 140.7, 139.8, 138.8, 128.9, 128.6, 128.2, 127.5, 127.4, 127.2, 127.0, 91.7, 81.3 (one aromatic resonance was unresolved). Anal. calcd for $C_{21}H_{16}Cl_3NO$: C, 62.32; H, 3.99; N, 3.46. Found: C, 62.50; H, 3.98; N, 3.59.



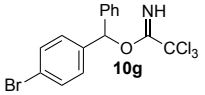
10e

(3-Fluorophenyl)(phenyl)methyl 2,2,2-trichloroacetimidate (10e). Purified by silica gel chromatography (5% ethyl acetate/ 1% triethylamine/ 94% hexanes); Yellow oil (1.28 g, 90%); TLC R_f = 0.52 (10% ethyl acetate/ 90% hexanes); IR (CH_2Cl_2) 3054, 1666, 1076, 999 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.42 (brs, 1H), 7.42-7.30 (m, 7H), 7.03 (tt, *J* = 9.8 Hz, 2.9, 2H), 6.93 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 162.4 (d, *J* = 245.2 Hz), 161.3, 160.8, 139.64, 135.60, 128.9 (d, *J* = 8.2 Hz), 128.6, 128.1, 126.8, 115.5 (d, *J* = 21.8 Hz), 91.5, 80.7. Anal. Calcd for $C_{15}H_{11}Cl_3FNO$: C, 51.98; H, 3.20; N, 4.04. Found: C, 52.06; H, 3.02; N, 4.06.



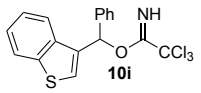
10f

(3-Chlorophenyl)(phenyl)methyl 2,2,2-trichloroacetimidate (10f). Purified by silica gel chromatography (5% ethyl acetate/ 1% triethylamine/ 94% hexanes); Viscous oil (1.23 g, 78%); TLC R_f = 0.65 (10% ethyl acetate/ 90% hexanes); IR (CH_2Cl_2) 3341, 1664, 1354, 1266, 1007, 791, 720, 701 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.44 (brs, 1H), 7.41 (d, *J* = 8.9 Hz, 3H), 7.37-7.21 (m, 6H), 6.90 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.2, 141.9, 139.1, 134.5, 129.9, 128.7, 128.4, 128.3, 127.1, 127.0, 125.1, 91.4, 80.6. Anal. Calcd for $C_{15}H_{11}Cl_4NO$: C, 49.62; H, 3.05; N, 3.86. Found: C, 49.85; H, 3.25; N, 4.00.



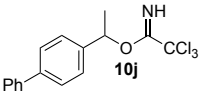
10g

(4-Bromophenyl)(phenyl)methyl 2,2,2-trichloroacetimidate (10g). Purified by silica gel chromatography (5% ethyl acetate/ 1% triethylamine/ 94% hexanes); White solid (2.08 g, 90%); TLC R_f = 0.59 (10% ethyl acetate/ 90% hexanes); IR (CH_2Cl_2) 3054, 2986, 2305, 1667, 1487, 1421, 1265, 705 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.43 (brs, 1H), 7.48 (d, *J* = 8.5 Hz, 2H), 7.34-7.29 (m, 7H), 6.89 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.2, 139.2, 138.8, 131.7, 128.7, 128.6, 128.3, 126.9, 122.1, 91.4, 80.7. Anal. Calcd for $C_{15}H_{11}BrCl_3NO$: C, 44.21; H, 2.72; N, 3.44. Found: C, 44.18; H, 2.98; N, 3.76.



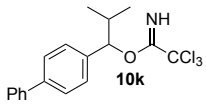
10i

(1-Benzothiophen-3-yl)(phenyl)methyl 2,2,2-trichloroacetimidate (10i). Purified by silica gel chromatography (5% ethyl acetate/ 1% triethylamine/ 94% hexanes); Viscous oil (0.34 g, 61%); IR (CH_2Cl_2) 3335, 1664, 1264, 702 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.49 (brs, 1H), 7.86-7.84 (m, 1H), 7.78-7.75 (m, 1H), 7.52 (d, *J* = 7.0 Hz, 1H), 7.40-7.32 (m, 7H), 7.25 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.4, 140.7, 138.2, 137.3, 134.5, 128.6, 128.5, 127.3, 125.9, 124.7, 124.3, 122.9, 122.7, 91.6, 77.2. Anal. Calcd for $C_{17}H_{12}Cl_3NOS$: C, 53.08; H, 3.14; N, 3.64. Found: C, 53.29; H, 2.92; N, 4.01.



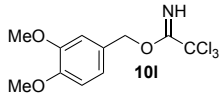
10j

1-(1,1'-Biphenyl]-4-yl)ethyl 2,2,2-trichloroacetimidate (10j). Purified by silica gel chromatography (5% ethyl acetate/ 1% triethylamine/ 94% hexanes); Off white solid (1.06 g, 81%); IR (CH_2Cl_2) 3333, 1661, 1661, 798, 765, 702 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.33 (brs, 1H), 7.60-7.57 (m, 4H), 7.49 (d, *J* = 8.2 Hz, 2H), 7.43 (t, *J* = 7.3 Hz, 2H), 7.36-7.32 (m, 1H), 6.03 (q, *J* = 6.5 Hz, 1H), 1.69 (d, *J* = 6.6 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 161.7, 140.9, 140.8, 140.4, 128.8, 127.4, 127.3, 127.1, 126.3, 91.8, 77.0, 22.1. Anal. Calcd for $C_{16}H_{14}Cl_3NO$: C, 56.09; H, 4.12; N, 4.09. Found: C, 56.00; H, 3.96; N, 4.44.



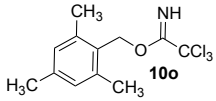
10k

1-(1,1'-Biphenyl]-4-yl)-2-methylpropyl 2,2,2-trichloroacetimidate (10k). Purified by silica gel chromatography (10% ethyl acetate/ 1% triethylamine/ 89% hexanes); White solid (2.00 g, 66%), TLC R_f = 0.42 (10% ethyl acetate/ 90% hexanes); IR (DCM) 3339, 2967, 1661, 1487, 1300, 1058, 990 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.16 (s,1H), 7.53-7.49 (m, 4H), 7.37-7.34 (m, 4H), 7.28-7.26 (m, 1H), 5.52 (d, *J* = 7.1 Hz, 1H), 2.16 (octet, *J* = 6.8 Hz, 1H), 1.02 (d, *J* = 6.6 Hz, 3H), 0.86 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.8, 140.8, 140.7, 138.2, 128.8, 127.4, 127.3, 127.2, 126.9, 92.0, 85.6, 34.4, 19.0, 18.3. Anal. Calcd for $C_{18}H_{18}Cl_3NO$: C, 58.32; H, 4.89; N, 3.78. Found: C, 58.50; H, 4.69; N, 3.90.



10l

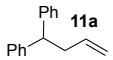
3,4-Dimethoxybenzyl 2,2,2-trichloroacetimidate (10l). Yellow oil (3.00 g, 90%); TLC R_f = 0.28 (20% ethyl acetate/ 80% hexanes); Purified by silica gel chromatography (30% ethyl acetate/ 1% triethylamine/ 69% hexanes); ^1H NMR (400 MHz, $CDCl_3$) δ 8.38 (brs, 1H), 7.01-6.98 (m, 2H), 6.87 (d, *J* = 8.1 Hz, 1H), 5.29 (s, 2H), 3.89 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.4, 149.1, 149.0, 127.9, 120.8, 111.3, 111.0, 91.5, 70.8, 55.85, 55.83. Anal. Calcd for $C_{11}H_{12}Cl_3NO_3$: C, 42.27; H, 3.87; N, 4.48. Found: C, 42.39; H, 3.59; N, 4.59.



10o

(2,4,6-Trimethylphenyl)methyl 2,2,2-trichloroacetimidate (10o). Purified by silica gel chromatography (5% ethyl acetate/ 1% triethylamine/ 94% hexanes); Viscous oil (1.23 g, 78%); TLC R_f = 0.65 (10% ethyl acetate/ 90% hexanes); ^1H NMR (400 MHz, $CDCl_3$) δ 8.35 (brs, 1H), 6.90 (s, 2H), 5.33 (s, 2H), 2.39 (s, 6H), 2.29 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.1, 138.7, 138.6, 129.0, 128.6, 91.6, 66.2, 21.1, 19.5. Anal. Calcd for $C_{12}H_{14}Cl_3NO$: C, 48.92; H, 4.79; N, 4.75. Found: C, 49.29; H, 4.47; N, 4.93.

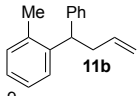
General procedure for the allylation of benzylic trichloroacetimidates with allyltributylstannane.
The trichloroacetimidate (1 eq) was dissolved in dry $MeNO_2$ (0.25 M) under argon in a flame dried round bottom flask. Allyltributylstannane (2 eq) was added to this flask and the mixture was heated to reflux. The reaction was monitored by TLC. After the imidate was consumed, the solution was allowed to cool to rt and a KF solution (aq., 0.1M) was added. The mixture was poured into water extracted with ethyl acetate. The combined organic extracts were then dried with Na_2SO_4, filtered, and concentrated. Purification by silica gel column chromatography provided the reported allylation product.



11a

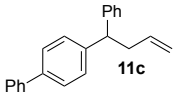
(1-Phenylbut-3-en-1-yl)benzene (11a).[8] Purified by silica gel chromatography (1% ethyl acetate/ 99% hexanes); Clear oil (0.127 g, 71%); TLC R_f = 0.59 (10% ethyl acetate/ 90% hexanes); ^1H NMR (400 MHz, $CDCl_3$) δ 7.31- 7.24 (m, 8H), 7.20-7.16 (m, 2H), 5.79-5.68 (m, 1H), 5.04 (dd, *J* = 17.2, 1.7 Hz, 1H), 4.96 (dt, *J* = 10.1, 1.0 Hz, 1H), 4.02

(t, *J* = 7.9 Hz, 1H), 2.85-2.82 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.5, 136.9, 128.4, 128.0, 126.2, 116.3, 51.3, 40.0.



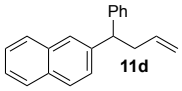
11b

1-Methyl-2-(1-phenylbut-3-en-1-yl)benzene (11b).[9] Purified by silica gel chromatography (100% hexanes); Clear oil (0.05 g, 77%); TLC R_f = 0.83 (10% ethyl acetate/ 90% hexanes); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.33-7.10 (m, 9H), 5.81-5.68 (m, 1H), 5.06-4.93 (m, 2H), 4.19 (t, *J* = 7.7 Hz, 2H), 2.78 (t, *J* = 7.7 Hz, 1H), 2.25 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 144.1, 142.2, 137.0, 136.3, 130.5, 128.3, 126.9, 126.1, 126.0, 126.0, 116.2, 47.0, 40.4, 19.9.



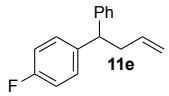
11c

4-(1-Phenylbut-3-en-1-yl)-1,1'-biphenyl (11c). Purified by silica gel chromatography (0.5% ethyl acetate/ 99.5% hexanes); Clear oil (0.05 g, 71%); TLC R_f = 0.68 (10% ethyl acetate/ 90% hexanes); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 (d, J = 7.6 Hz, 2H), 7.42 (d, *J* = 8.0 Hz, 2H), 7.32 (t, *J* = 7.4 Hz, 2H), 7.24-7.20 (m, 7H), 7.12-7.09 (m, 1H), 5.72-5.62 (m, 1H), 4.98 (d, *J* = 17.1 Hz, 1H), 4.89 (d, *J* = 10.2 Hz, 1H), 3.97 (t, *J* = 7.8 Hz, 1H), 2.77 (t, *J* = 7.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.4, 142.6, 139.9, 138.0, 135.7, 127.7, 127.4, 127.3, 126.9, 126.1, 126.02, 126.00, 125.2, 115.3, 49.9, 38.9. Anal. Calcd for $C_{22}H_{20}$: C, 92.91; H, 7.09. Found: C, 92.62; H, 7.29.



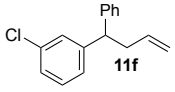
11d

2-(1-Phenylbut-3-en-1-yl)naphthalene (11d).[10] Purified by silica gel chromatography (1% ethyl acetate/99% hexanes); clear oil (0.08 g, 78%); TLC R_f = 0.72 (10% ethyl acetate/ 90% hexanes); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (t, *J* = 7.1 Hz, 2H), 7.74 (d, *J* = 8.6 Hz, 1H), 7.70 (s, 1H), 7.43 (dd, *J* = 6.8, 1.44 Hz, 2H), 7.33 (dd, *J* = 8.5, 1.6 Hz, 1H), 7.28 (d, *J* = 4.3 Hz, 4H), 7.18 (sextet, *J* = 4 Hz, 1H), 5.81- 5.71 (m, 1H), 5.06 (dd, *J* = 17.1, 1.5 Hz, 1H), 4.95 (dd, *J* = 10.2, 0.6 Hz, 1H), 4.17 (t, *J* = 7.84 Hz, 1H), 2.98-2.86 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.4, 141.9, 136.8, 133.5, 132.2, 128.4, 128.1, 127.8, 127.6, 126.8, 126.3, 126.03, 126.00, 125.4, 116.4, 51.3 (2C), 39.8.



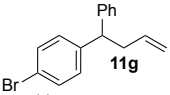
11e

1-Fluoro-4-(1-phenylbut-3-en-1-yl)benzene (11e).[11] Purified by silica gel chromatography (100% hexanes); clear oil (0.035 g, 36%); TLC R_f = 0.77 (10% ethyl acetate/ 90% hexanes); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30- 7.16 (m, 7H), 6.96 (t, *J* = 8.7 Hz, 2H), 5.75-5.64 (m, 1H), 5.02 (dd, *J* = 17.1, 1.7 Hz, 1H), 4.96 (d, *J* = 10.2 Hz, 1H), 3.99 (t, *J* = 7.9 Hz, 1H); 2.81-2.76 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.6 (d, *J* = 243.0 Hz), 144.4, 140.2, 136.6, 129.3 (d, *J* = 8.1 Hz), 128.5, 127.8, 126.3, 116.5, 115.1 (d, *J* = 21.0 Hz), 50.5, 40.1.



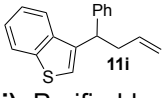
11f

1-Chloro-3-(1-phenylbut-3-en-1-yl)benzene (11f). Purified by silica gel chromatography (0.5% ethyl acetate/ 99.5% hexanes); Clear oil (0.035 g, 52%); TLC R_f = 0.74 (10% ethyl acetate/ 90% hexanes); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.32-7.09 (m, 9H), 5.76-5.62 (m, 1H), 5.03 (dq, *J* = 17.1, 1.5 Hz, 1H), 4.98-4.93 (m, 1H), 3.98 (t, *J* = 7.9 Hz, 1H), 2.79 (t, *J* = 7.0 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 146.6, 143.7, 136.3, 134.2, 129.7, 128.6, 128.1, 127.9, 126.5, 126.4, 126.2, 116.7, 51.0, 39.7. Anal. Calcd for $C_{16}H_{15}Cl$: C, 79.17; H, 6.23. Found: C, 79.04; H, 6.34.



11g

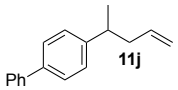
1-Bromo-4-(1-phenylbut-3-en-1-yl)benzene (11g).[11] Purified by silica gel chromatography (0.5% ethyl acetate/ 99.5% hexanes); clear oil (0.045 g, 64%); TLC R_f = 0.82 (10% ethyl acetate/ 90% hexanes); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (d, *J* = 8.4 Hz, 2H), 7.30-7.25 (m, 2H), 7.19 (d, *J* = 7.4, Hz, 3H), 7.10 (d, *J* = 8.3 Hz, 2H), 5.74-5.64 (m, 1H), 5.02 (dd, *J* = 17.1, 1.6 Hz, 1H), 4.96 (dd, *J* = 10.2, 0.7 Hz, 1H), 3.97 (t, *J* = 7.9 Hz, 1H), 2.84-2.72 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.9, 143.5, 136.4, 131.5, 129.8, 128.5, 127.8, 126.4, 120.0, 116.7, 50.6, 39.8.



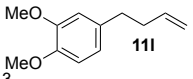
11i

3-(1-Phenylbut-3-en-1-yl)-1-benzothiophene (11i). Purified by silica gel chromatography (1% ethyl acetate/ 99% hexanes); Clear oil (0.030 g, 42%); TLC R_f = 0.41 (10% ethyl acetate/ 90% hexanes); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83-7.81 (m, 1H), 7.62- 60 (m, 1H), 7.29-7.26 (m, 7H), 7.21-7.15 (m, 1H), 5.85 (m, 1H), 5.07 (dd, *J* = 17.1, 1.6 Hz, 1H), 4.99 (d, *J* = 10.2 Hz, 1H), 4.36 (t, *J* = 7.6 Hz, 1H), 3.00- 2.93 (m, 1H), 2.88-2.78 (m, 1H). ^{13}C NMR (100 MHz,

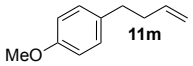
$CDCl_3$) δ 143.2, 140.5, 138.8, 138.7, 136.6, 128.5, 128.0, 126.5, 124.2, 123.9, 122.8, 122.3, 121.8, 116.6, 45.4, 40.4 . Anal. Calcd for $C_{18}H_{16}S$: C, 81.77; H, 6.10. Found: C, 81.40; H, 5.91.



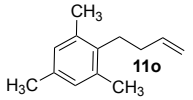
4-(Pent-4-en-2-yl)-1,1'-biphenyl (11j).[12] Purified by silica gel chromatography (1% ethyl acetate/ 99% hexanes); clear oil (0.020 g, 31%); TLC R_f = 0.52 (100% hexanes); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (d, *J* = 7.1 Hz, 2H), 7.53 (d, *J* = 8.3 Hz, 2H), 7.42 (t, *J* = 7.3 Hz, 2H), 7.34-7.23 (m, 3H), 5.80-5.70 (m, 1H), 5.04-4.96 (m, 2H), 2.84 (sextet, *J* = 7.0 Hz, 1H), 2.46-2.39 (m, 1H), 2.35-2.28 (m, 1H), 1.28 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.1, 146.2, 141.1, 138.9, 137.1, 128.7, 127.4, 127.1, 127.0, 116.0, 42.6, 39.5, 21.5.



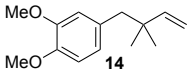
4-(But-3-en-1-yl)-1,2-dimethoxybenzene (11l).[13] Purified by silica gel chromatography (5% ethyl acetate/ 95% hexanes); Clear oil (0.045 g, 61%); TLC R_f = 0.41 (10% ethyl acetate/ 90% hexanes); ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.80 (d, *J* = 8.4 Hz, 1H), 6.73 (d, *J* = 7.2 Hz, 2H), 5.91-5.81 (m, 1H), 5.05 (dd, *J* = 17.1, 1.6 Hz, 1H), 4.98 (dd, *J* = 10.2, 0.6 Hz, 1H), 3.87 (s, 3H), 3.86 (s, 3H), 2.66 (t, *J* = 7.4 Hz, 2H), 2.36 (q, *J* = 6.8 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 148.8, 147.2, 138.2, 134.6, 120.2, 114.9, 111.8, 111.2, 55.9, 55.8, 35.7, 35.0.



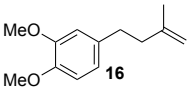
1-(But-3-en-1-yl)-4-methoxybenzene (11m).[10] Purified by silica gel chromatography (2% ethyl acetate/ 98% hexanes); Clear oil (0.050 g, 43%); TLC R_f= 0.72 (10% ethyl acetate/ 90% hexanes); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.02 (d, *J* = 8.3 Hz, 2H), 6.75 (d, *J* = 8.5 Hz , 2H), 5.82-5.73 (m, 1H), 4.95 (d, *J* = 17.1 Hz, 1H), 4.89 (d, *J* = 10.2 Hz, 1H), 3.70 (s, 3H), 2.57 (t, *J* = 7.4 Hz, 2H), 2.26 (d, *J* = 6.7 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.8, 138.2, 134.0, 129.3, 114.9, 113.7, 55.3, 35.8, 34.5.



2-(But-3-en-1-yl)-1,3,5-trimethylbenzene (11o).[14] Purified by silica gel chromatography (100% pentane); Clear oil (0.015 g, 13%); TLC R_f = 0.38 (100% hexanes); ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.83 (brs, 2H), 5.98-5.88 (m, 1H), 5.08 (dd, *J* = 17.1, 1.6 Hz, 1H), 4.99 (d, *J* = 10.2 Hz, 1H), 2.69-2.65 (m, 2H), 2.29 (s, 9H), 2.20-2.18 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.6, 136.0, 135.6, 135.0, 128.9, 114.5, 33.3, 28.9, 20.8, 19.7.

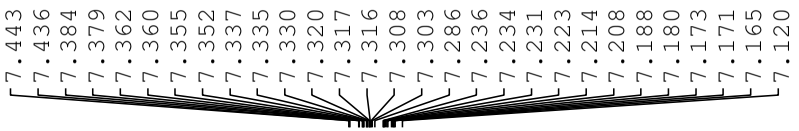


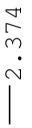
4-(2,2-Dimethylbut-3-en-1-yl)-1,2-dimethoxybenzene (14). Purified by silica gel chromatography (2% ethyl acetate/ 98% hexanes); Clear oil (0.036 g, 50%); TLC R_f = 0.61 (10% ethyl acetate/ 90% hexanes); ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.70-6.57 (m, 3H), 5.78 (dd, *J* = 17.5, 10.8 Hz, 1H), 4.85-4.76 (m, 2H), 3.78 (s, 6H), 2.45 (s, 2H), 0.92 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.3, 148.0, 147.3, 131.5, 122.6, 114.1, 110.5 (2C), 55.8, 48.7, 37.7, 26.5. Anal. Calcd for $C_{14}H_{20}O_2$: C, 76.33; H, 9.15. Found: C, 75.93; H, 9.19.

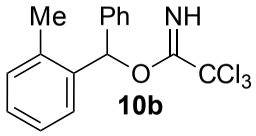


1,2-Dimethoxy-4-(3-methylbut-3-en-1-yl)benzene (16). Purified by silica gel chromatography (2% ethyl acetate/ 98% hexanes); Clear oil (0.035 g, 45%); TLC R_f = 0.61 (10% ethyl acetate/ 90% hexanes); ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.79-6.73 (m, 3H), 4.73 (s, 1H), 4.71 (s, 1H), 3.86 (s, 3H), 3.84 (s, 3H), 2.69 (t, *J* = 7.6 Hz, 2H), 2.29 (t, *J* = 7.6 Hz, 2H), 1.76 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.8, 147.2, 145.4, 134.9, 120.1, 111.7, 111.2, 110.2, 55.9, 55.8, 39.8, 33.9, 22.6. Anal. Calcd for $C_{13}H_{18}O_2$: C, 75.69; H, 8.80. Found: C, 75.49; H, 8.93.

NSM-5-84Final.1







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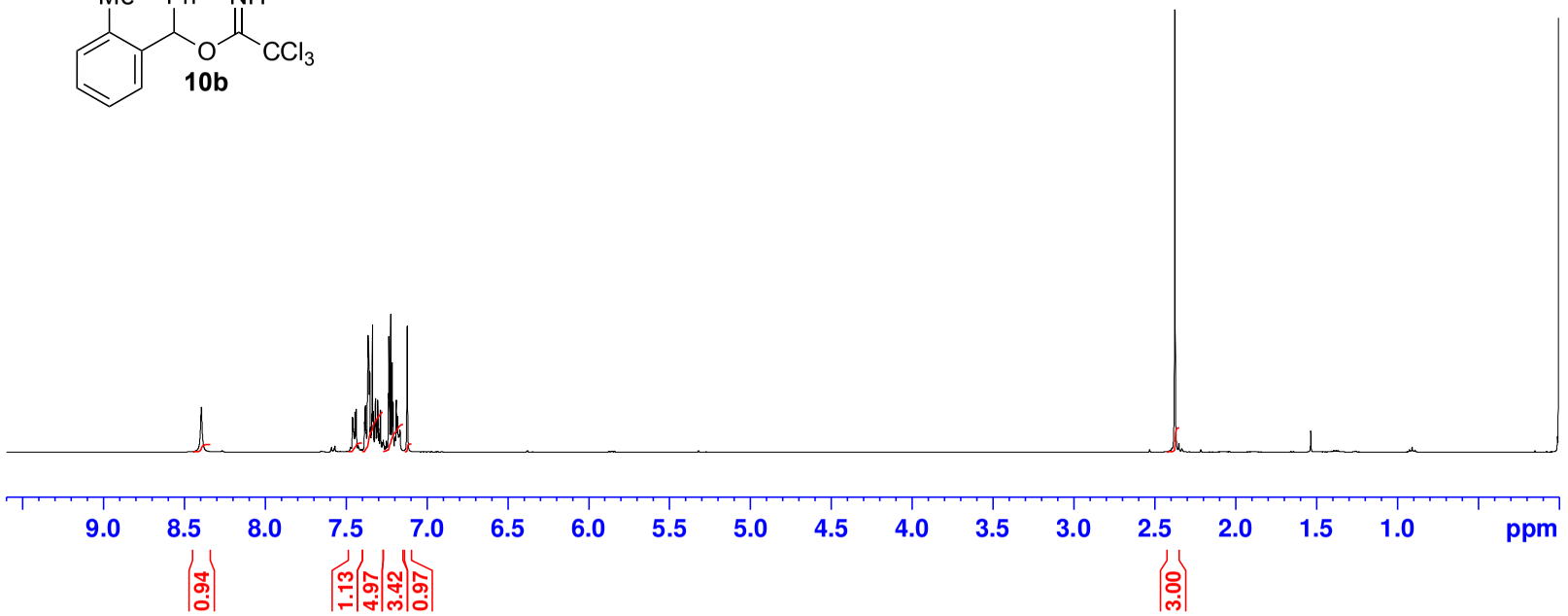
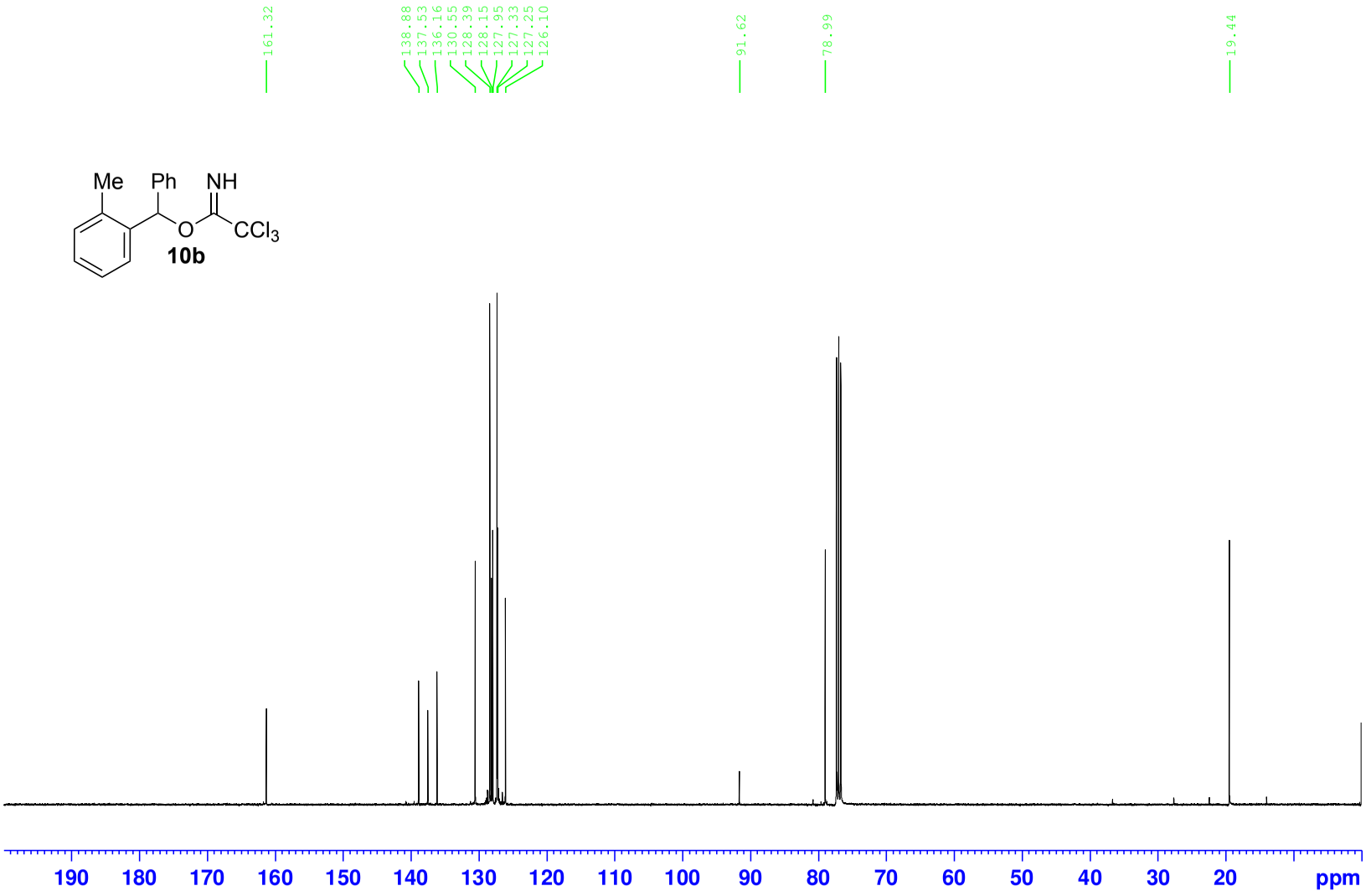


NSM-5-84FinalC13



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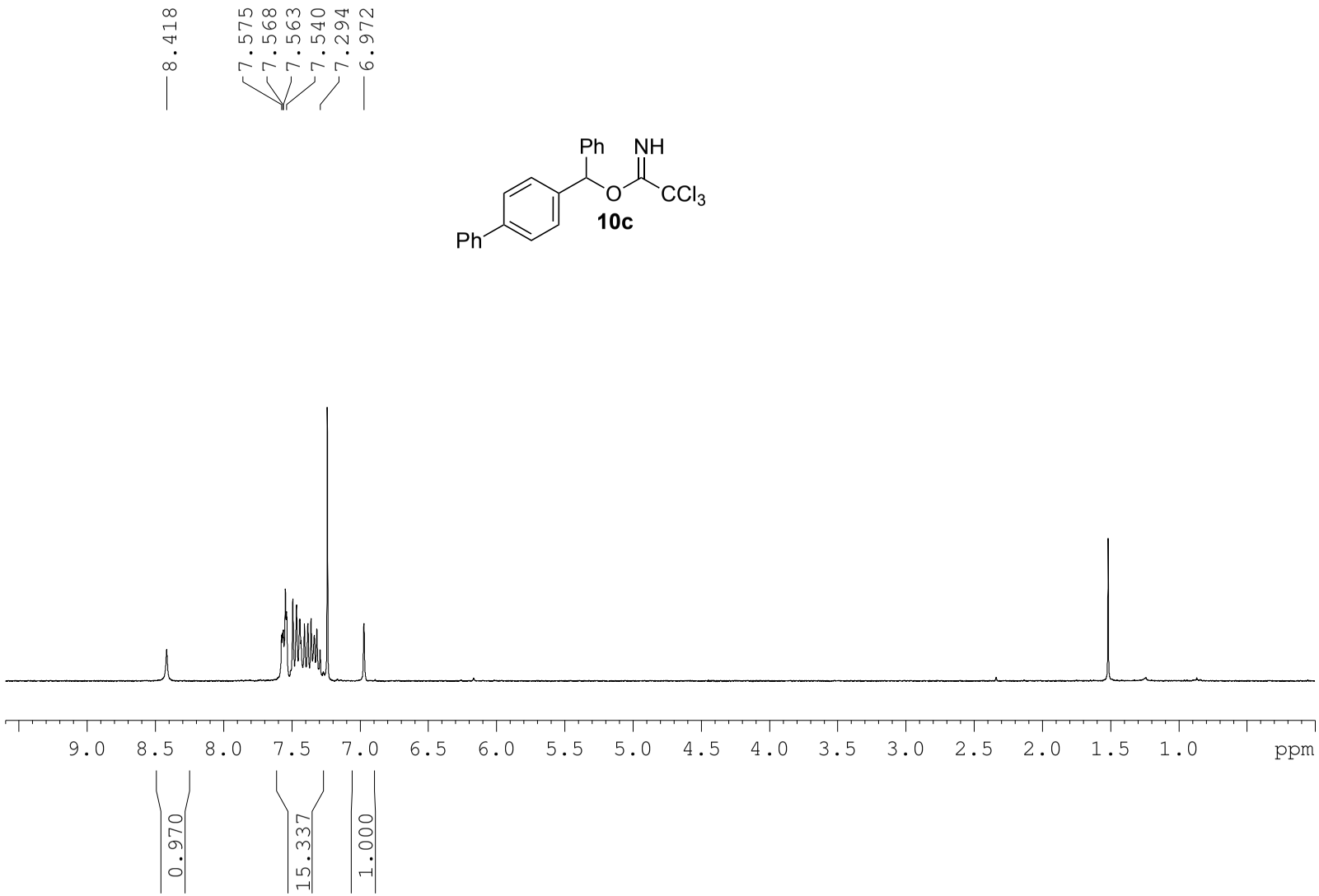
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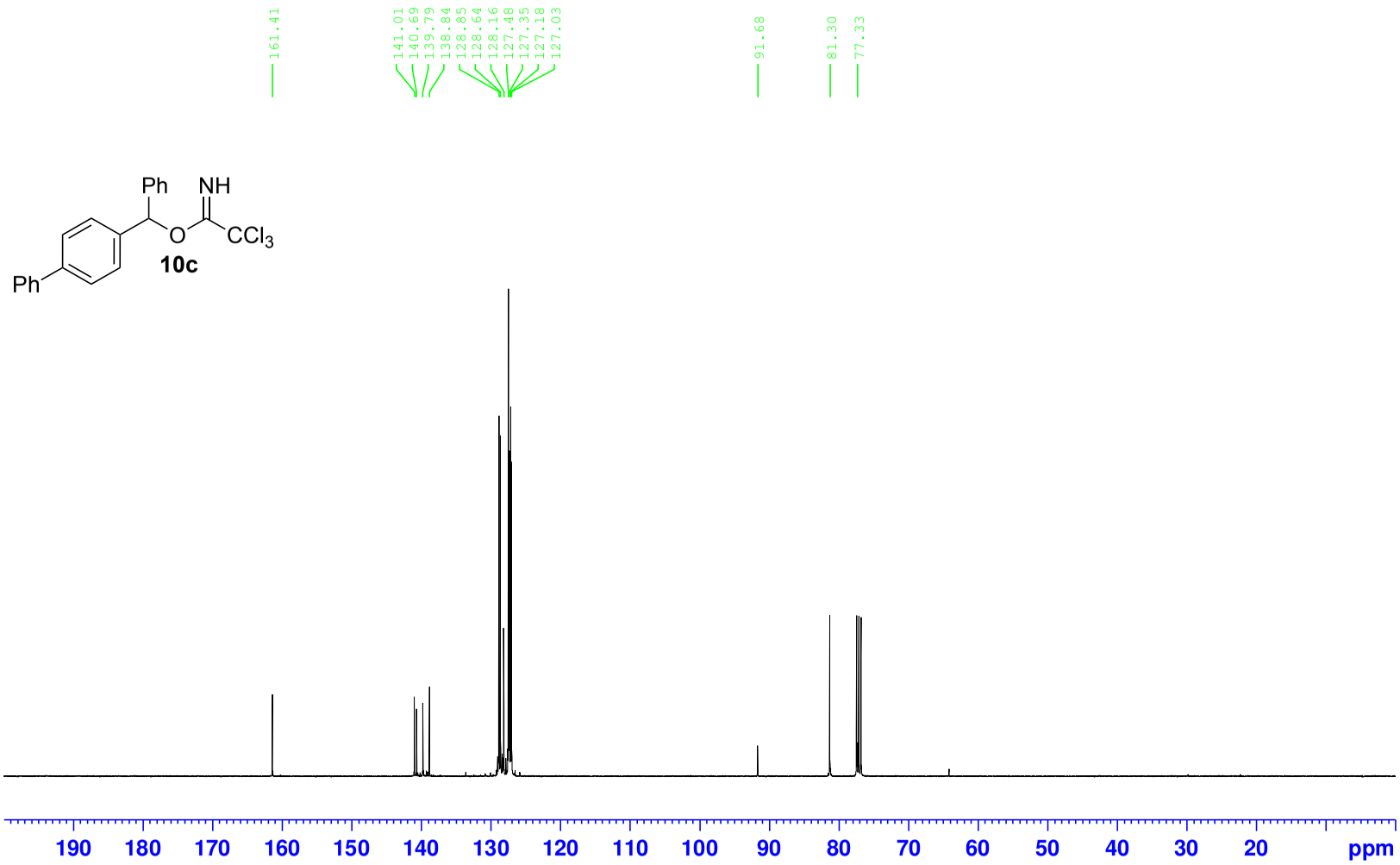

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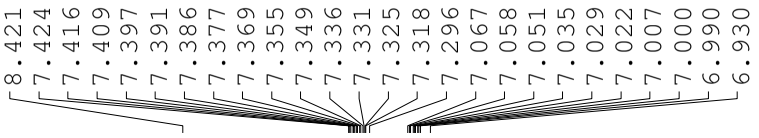

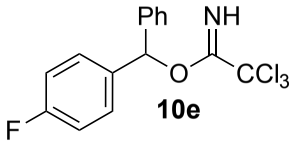

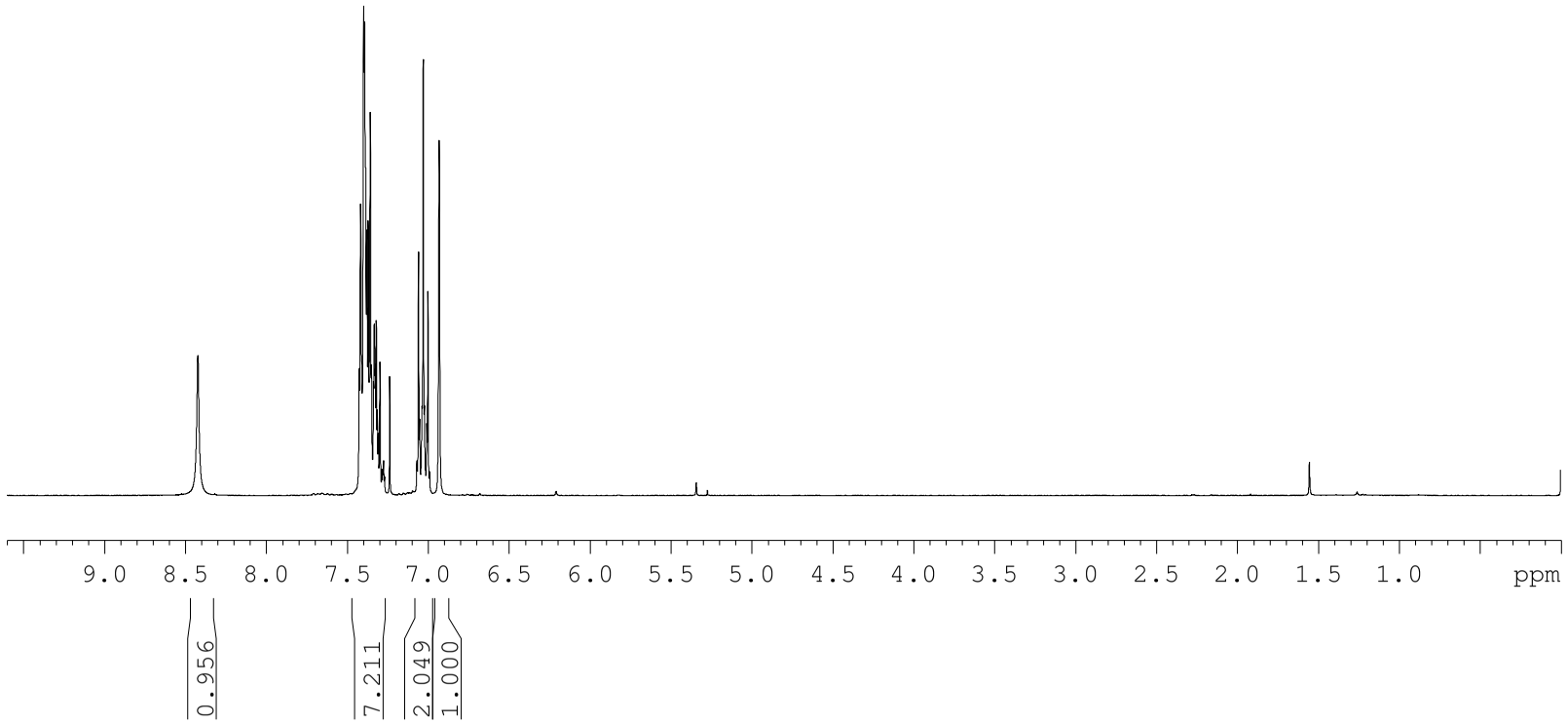

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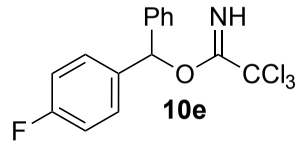

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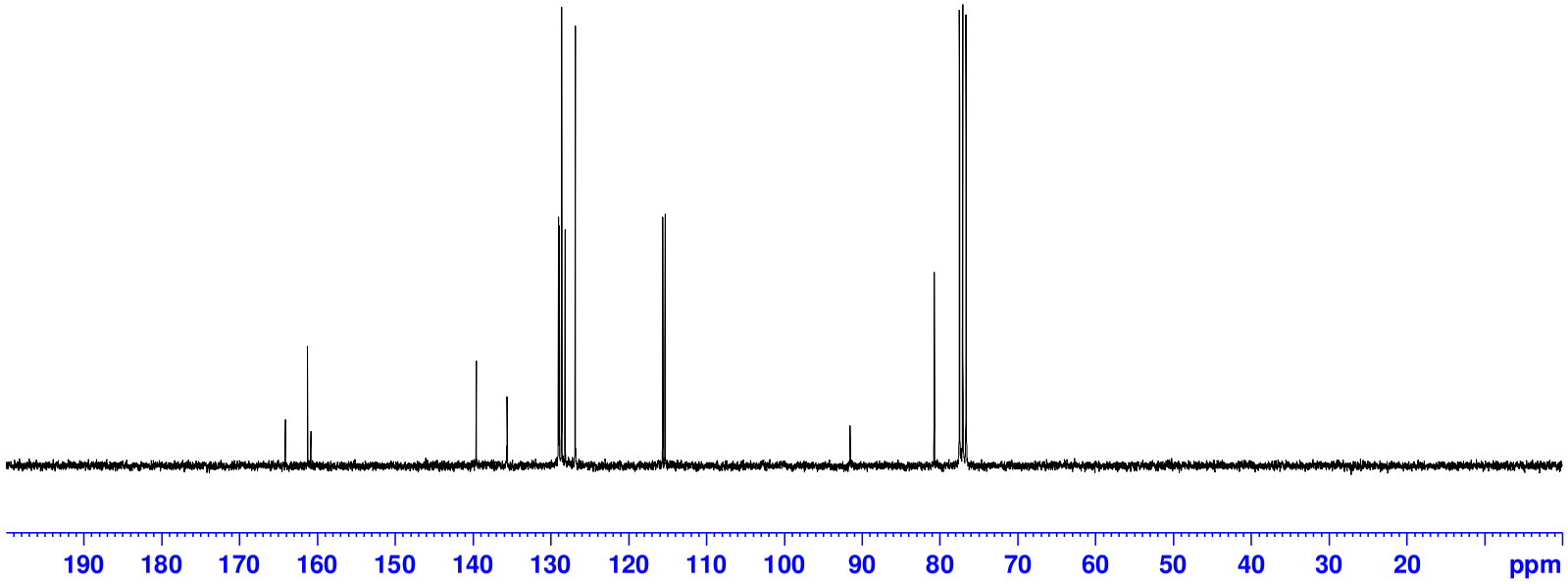

NSM-6-113Final

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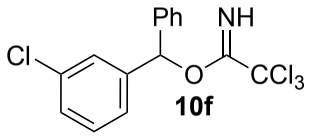

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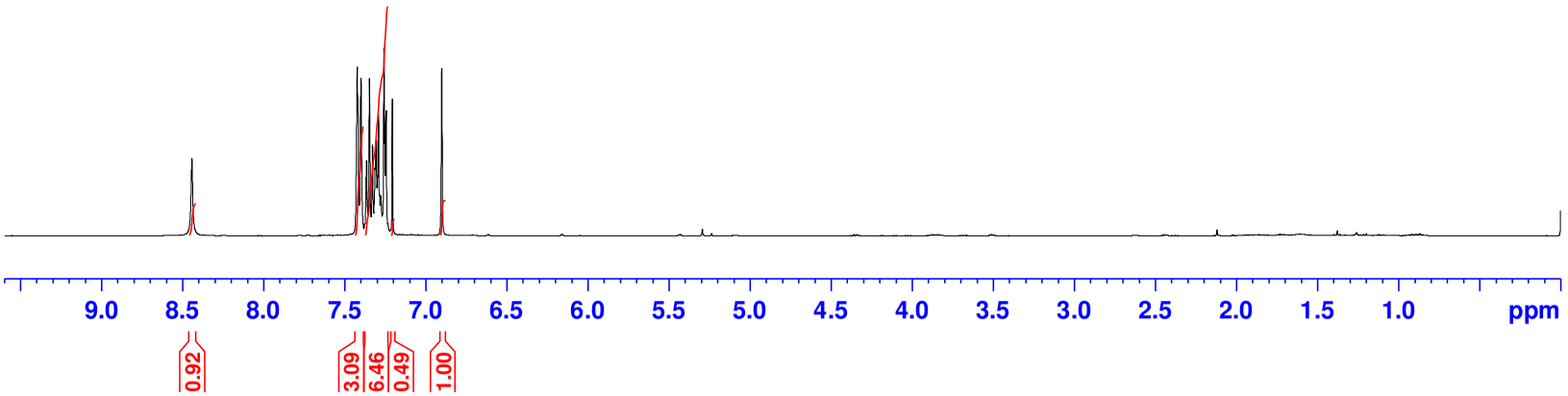

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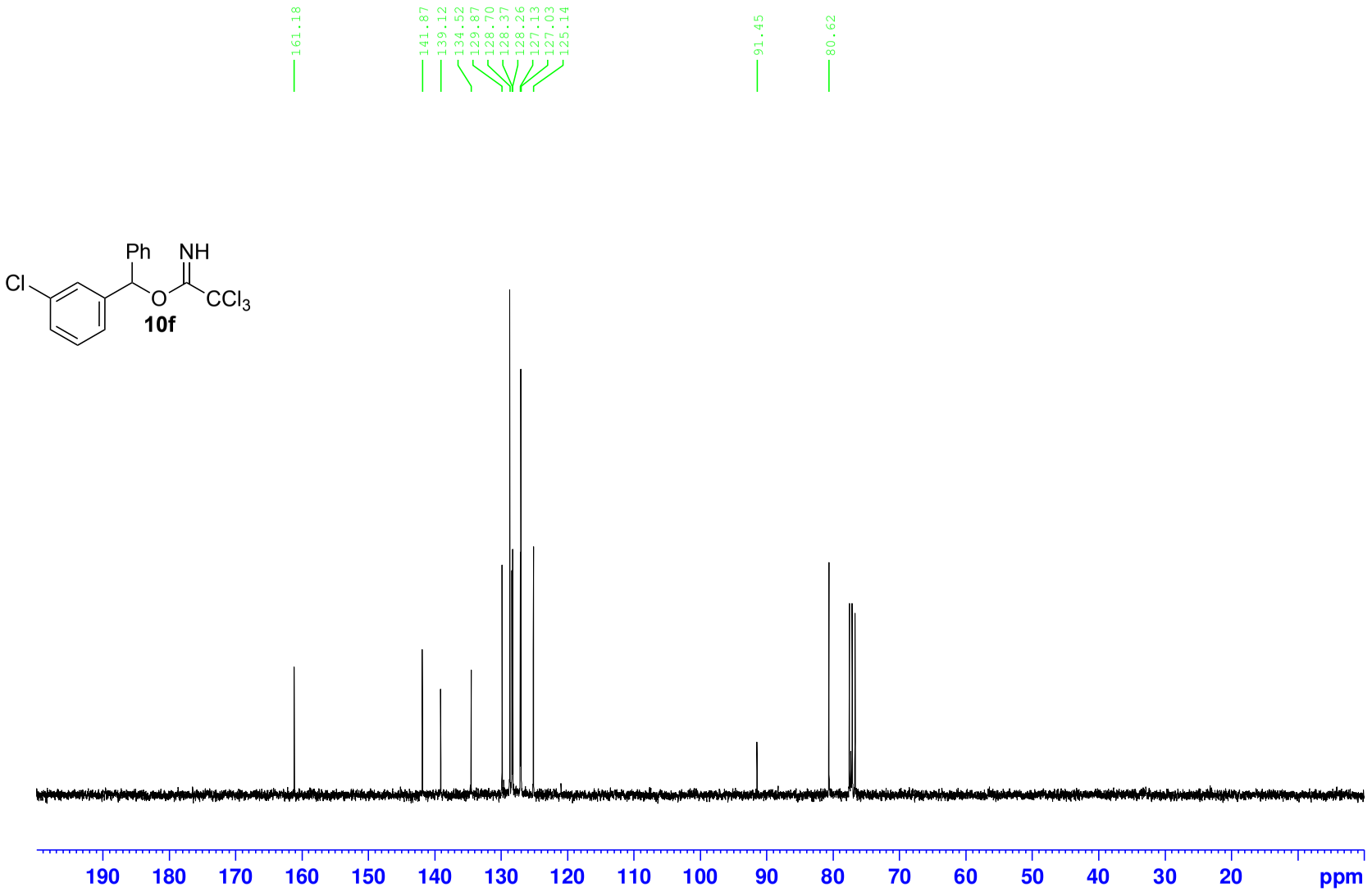

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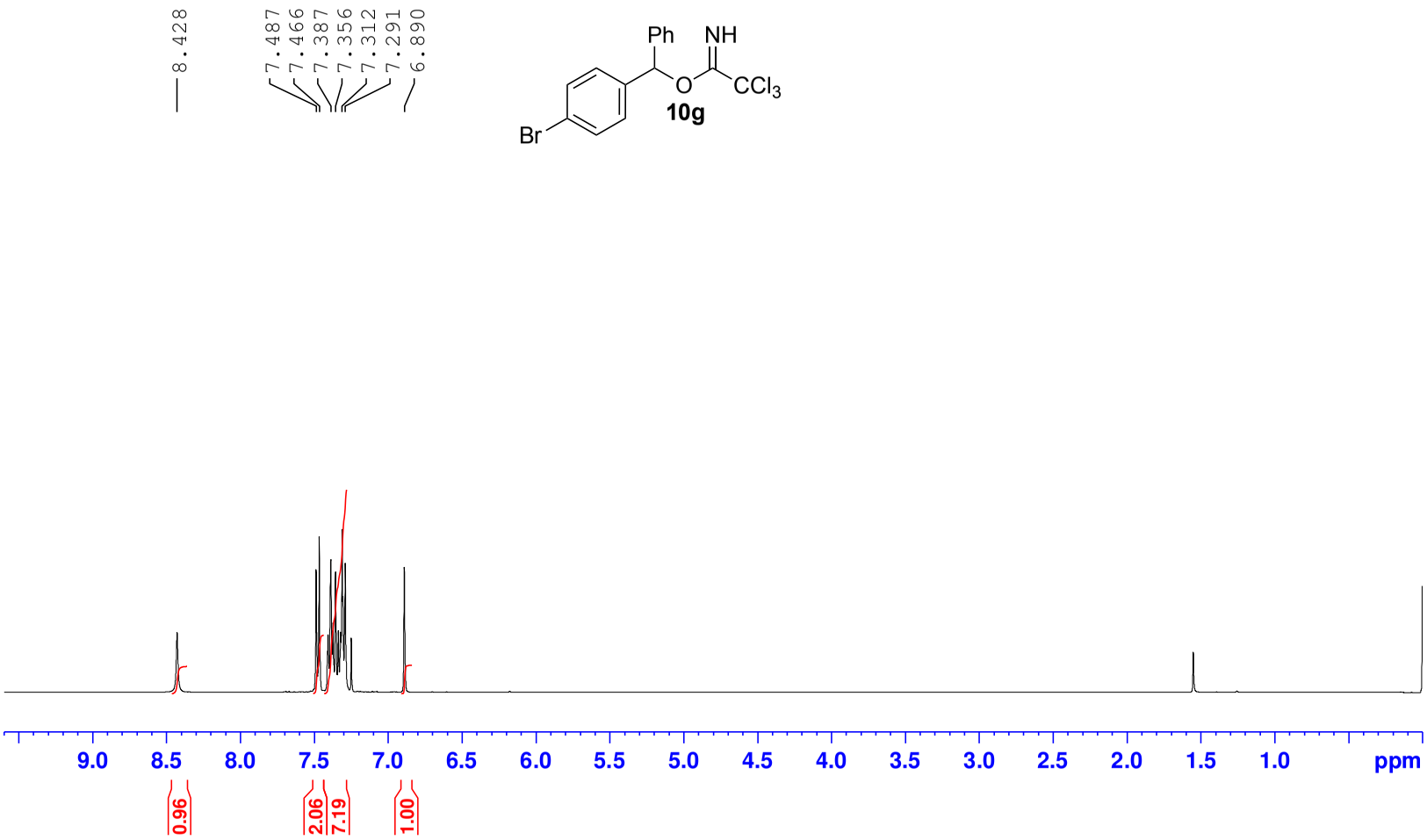

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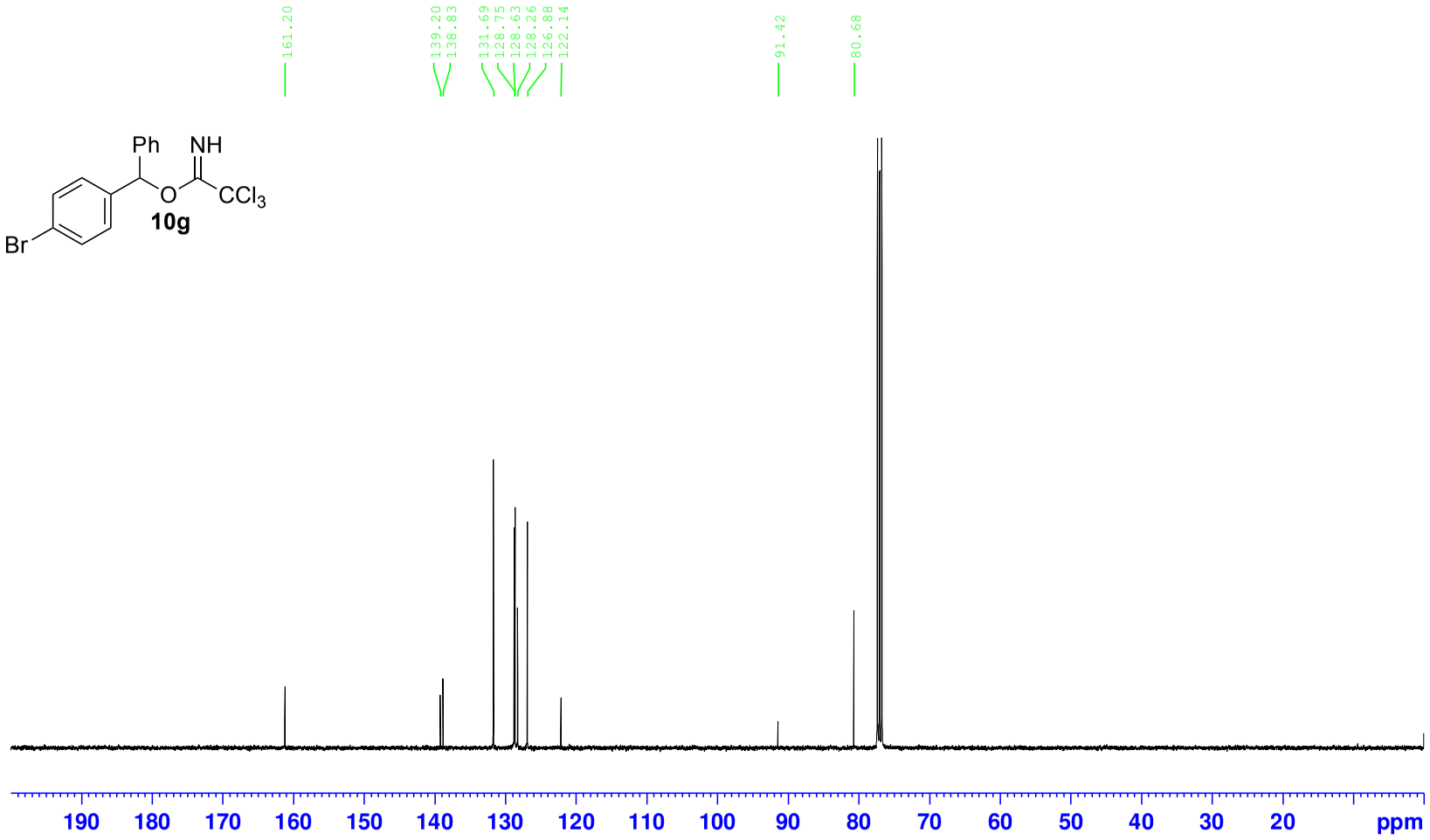

NSM-6-108Final

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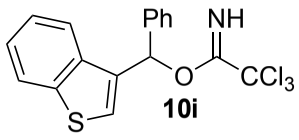

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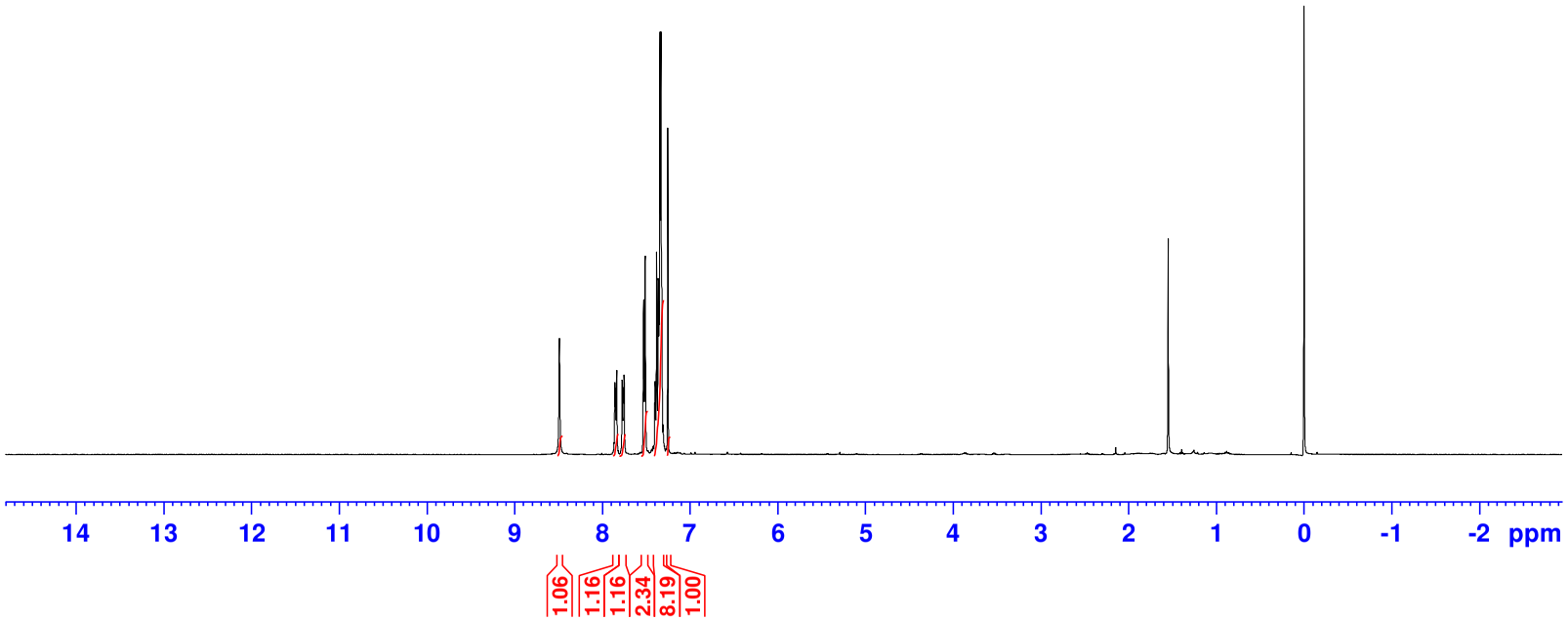

NSM-6-108C13.3

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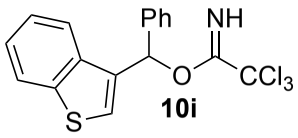

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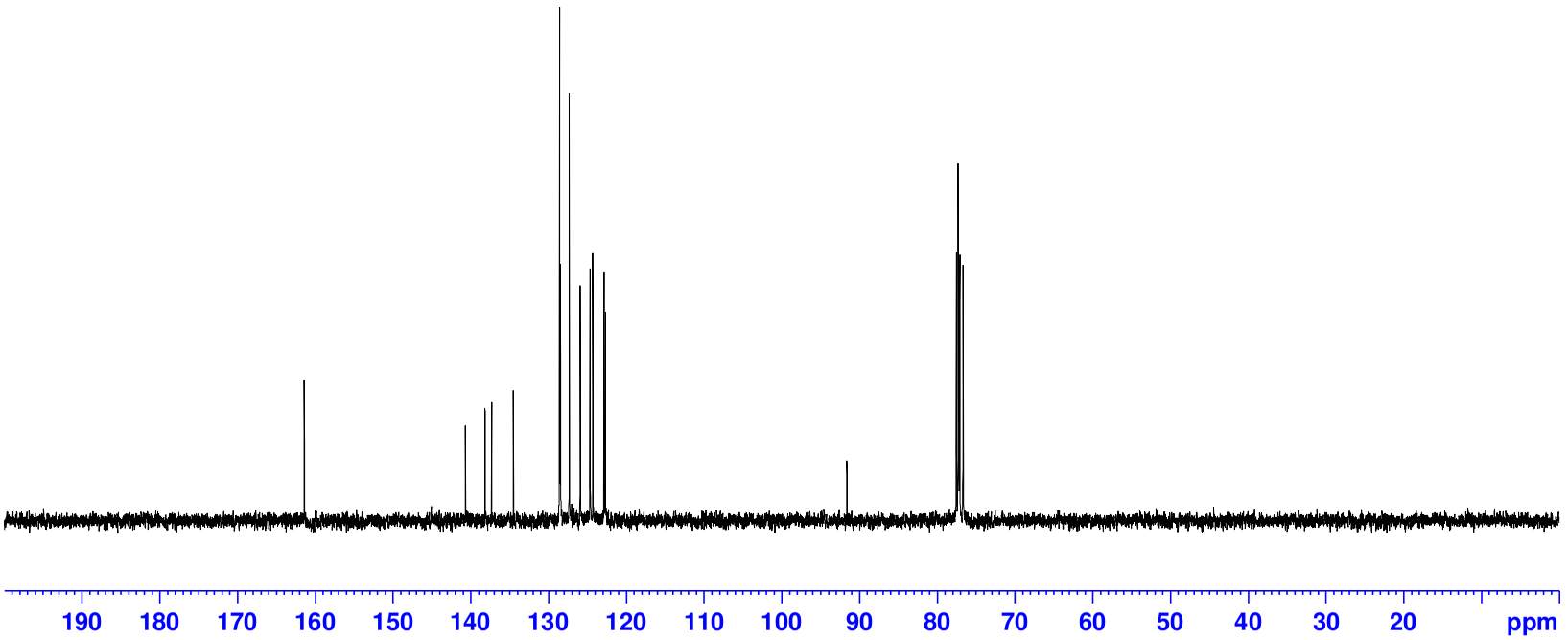

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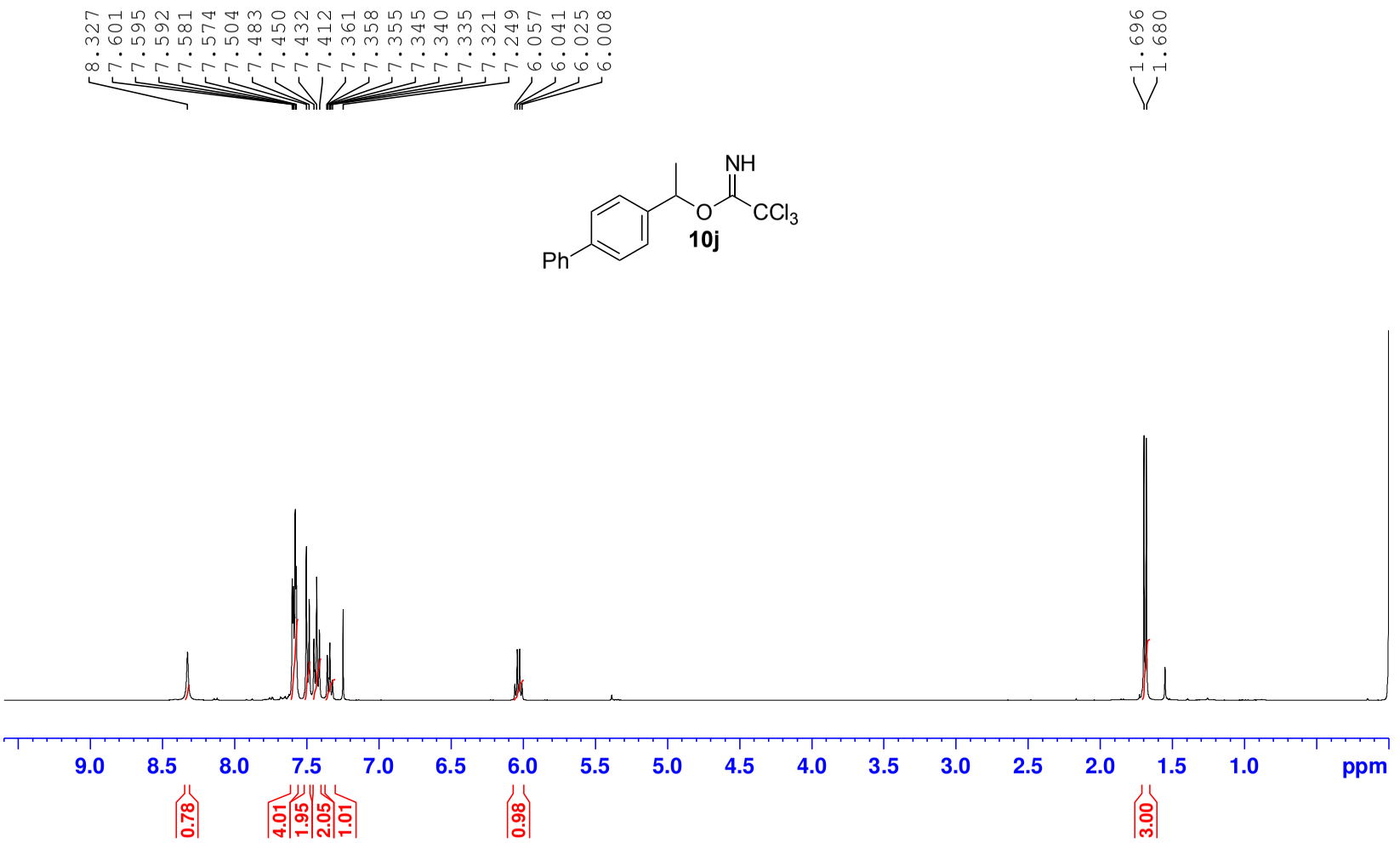

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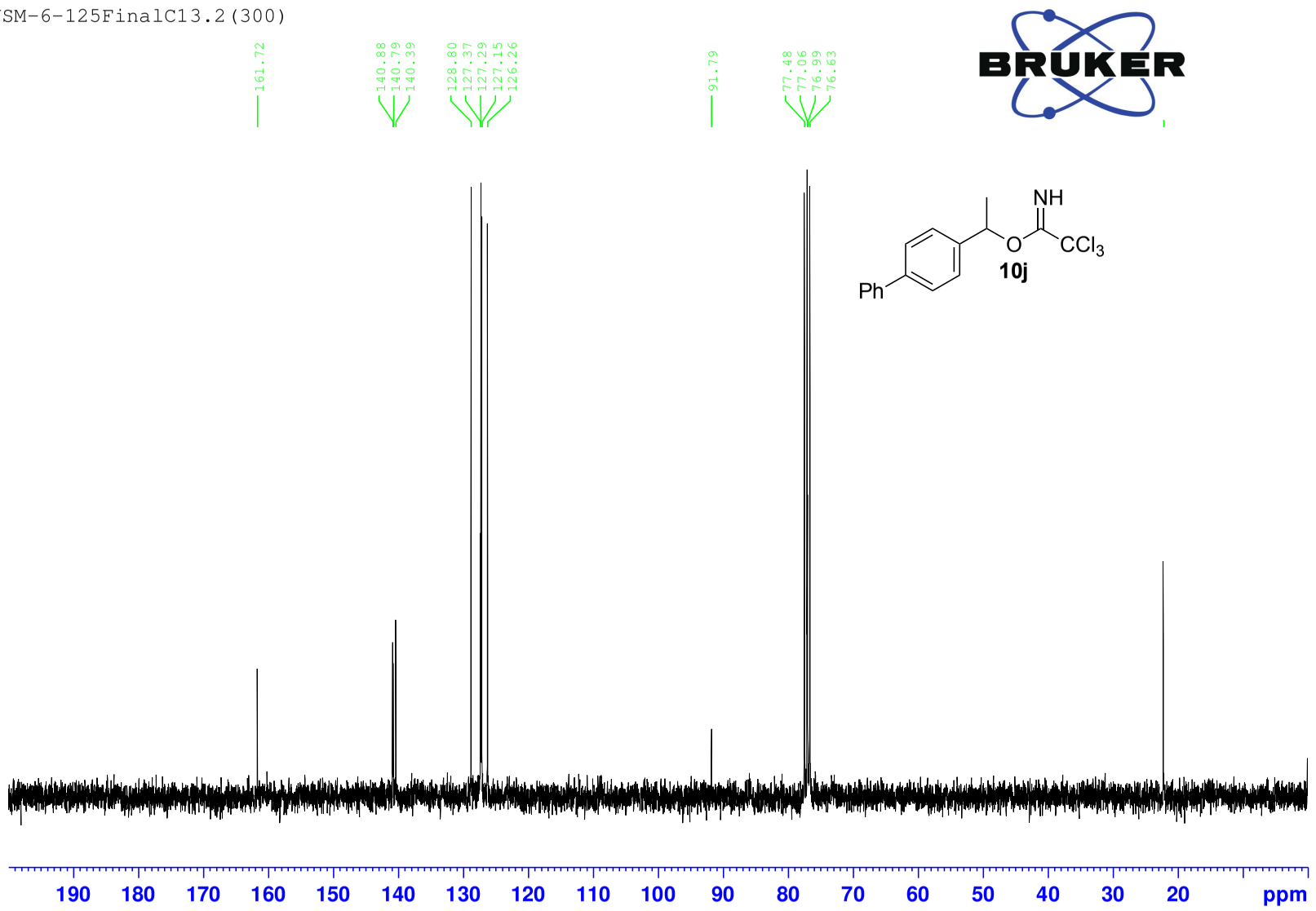

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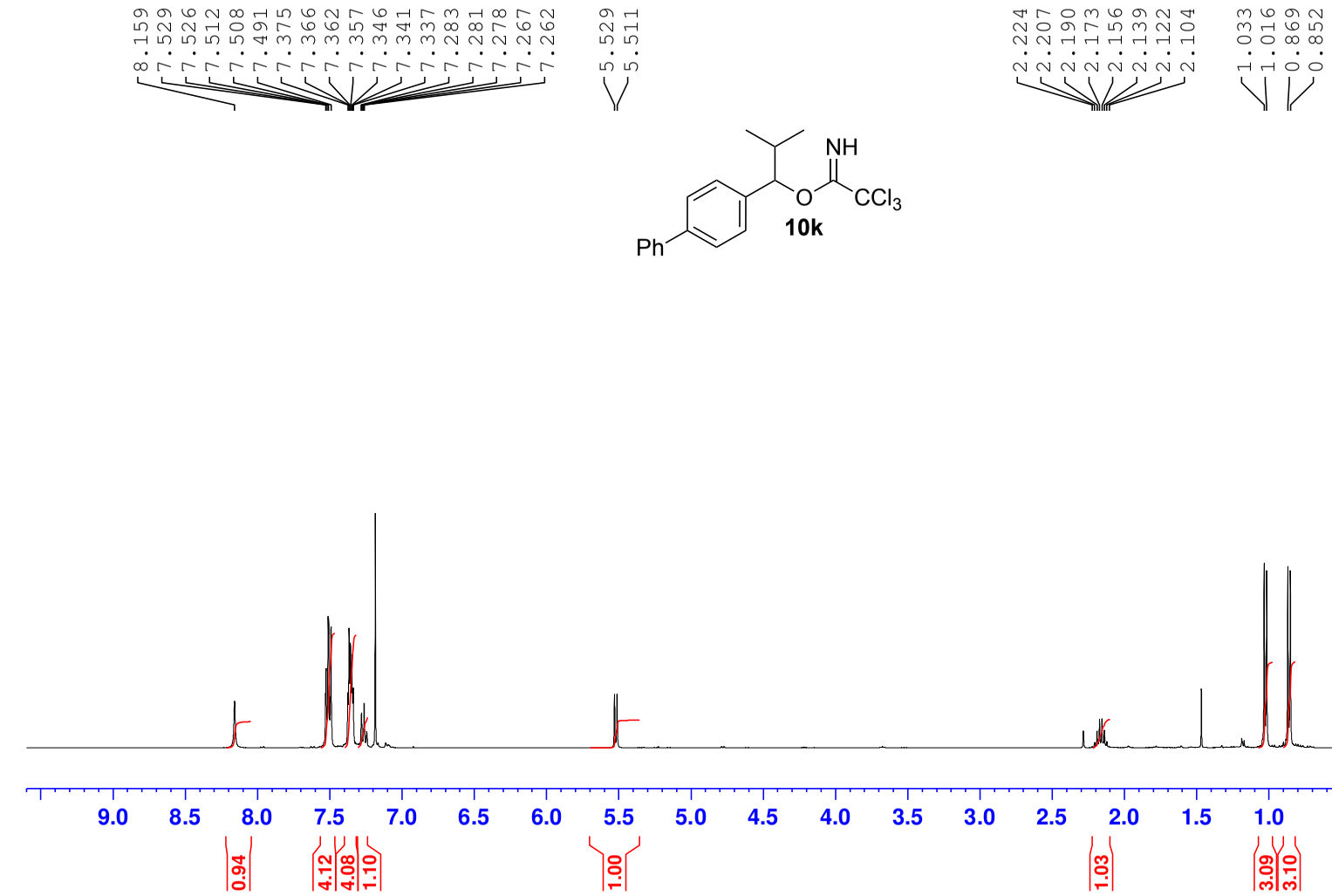

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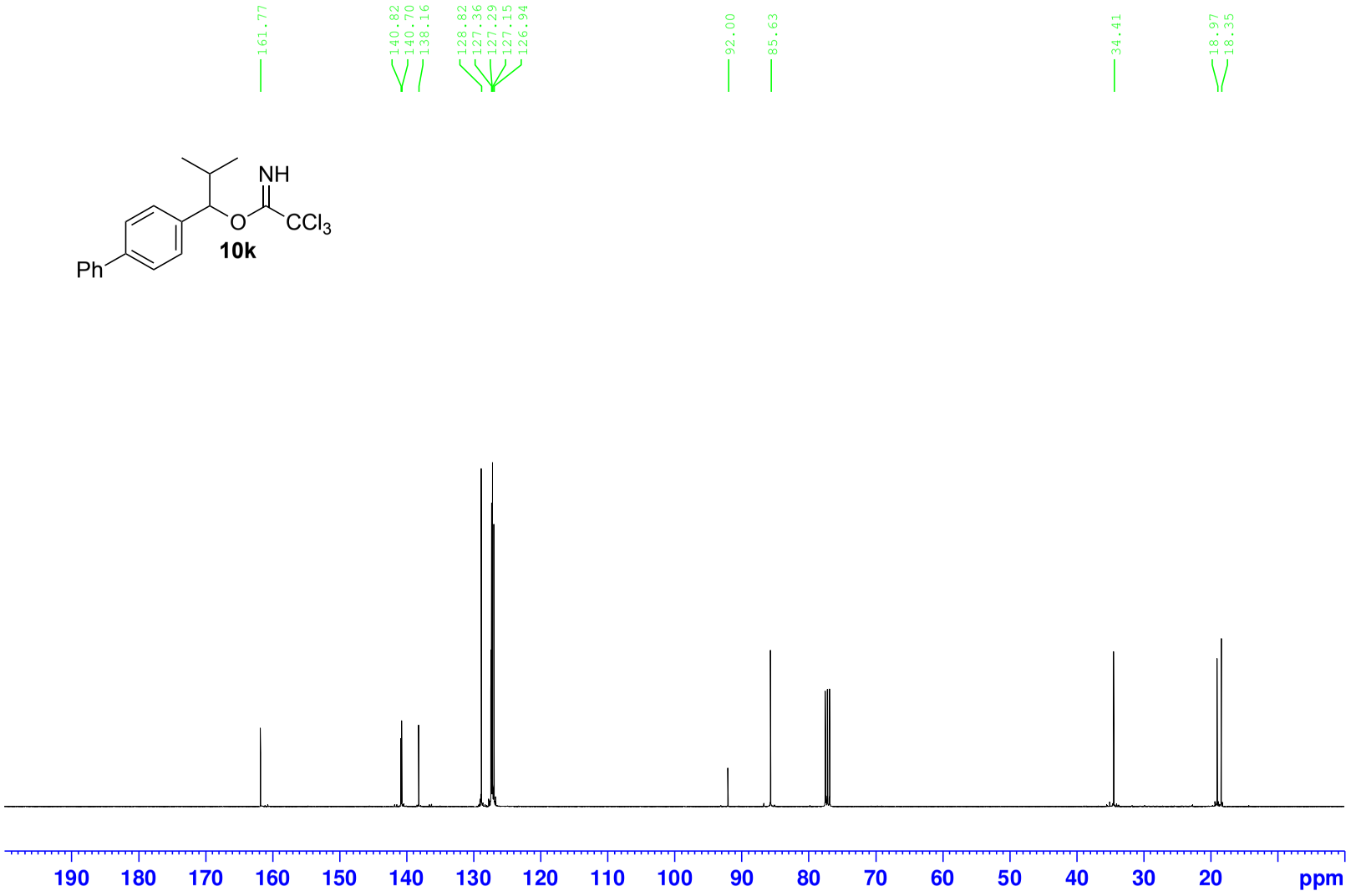

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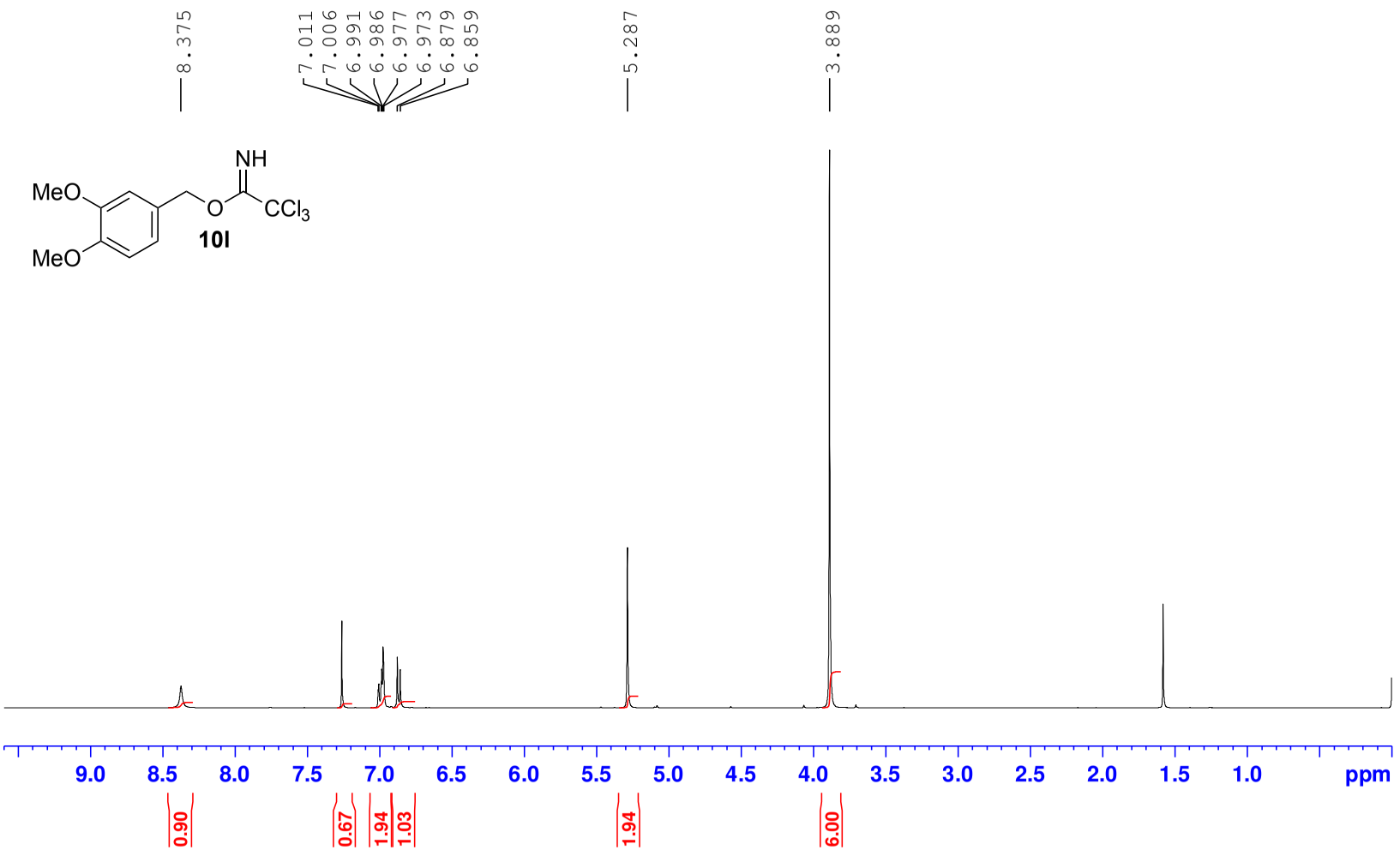
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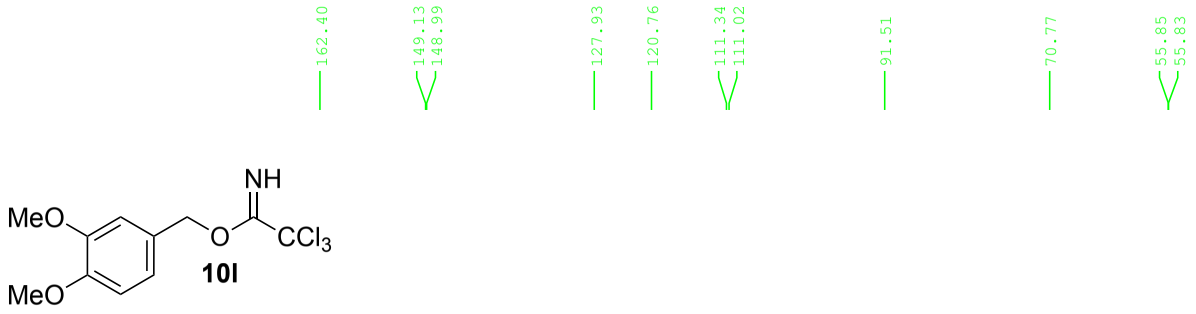

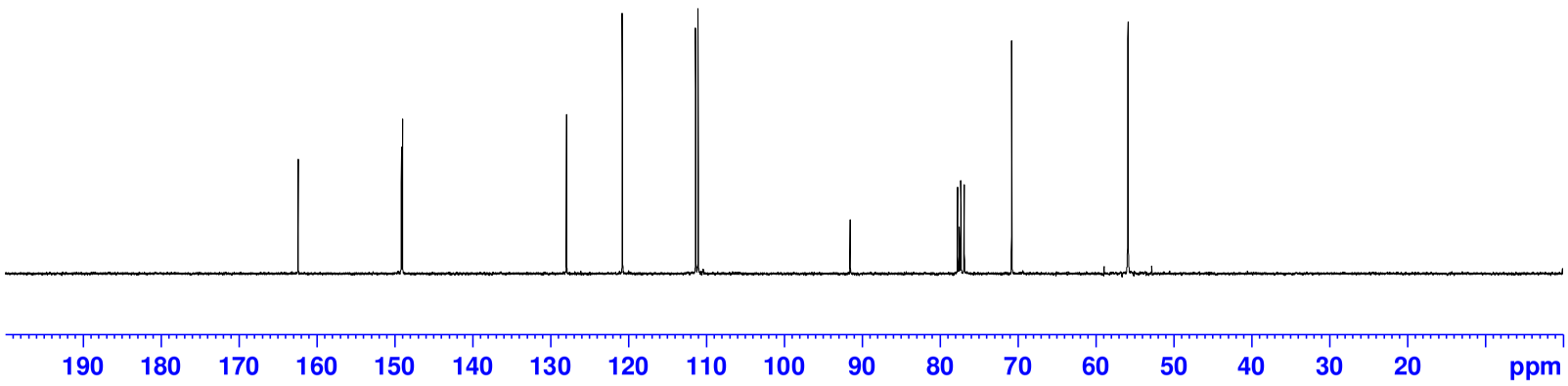

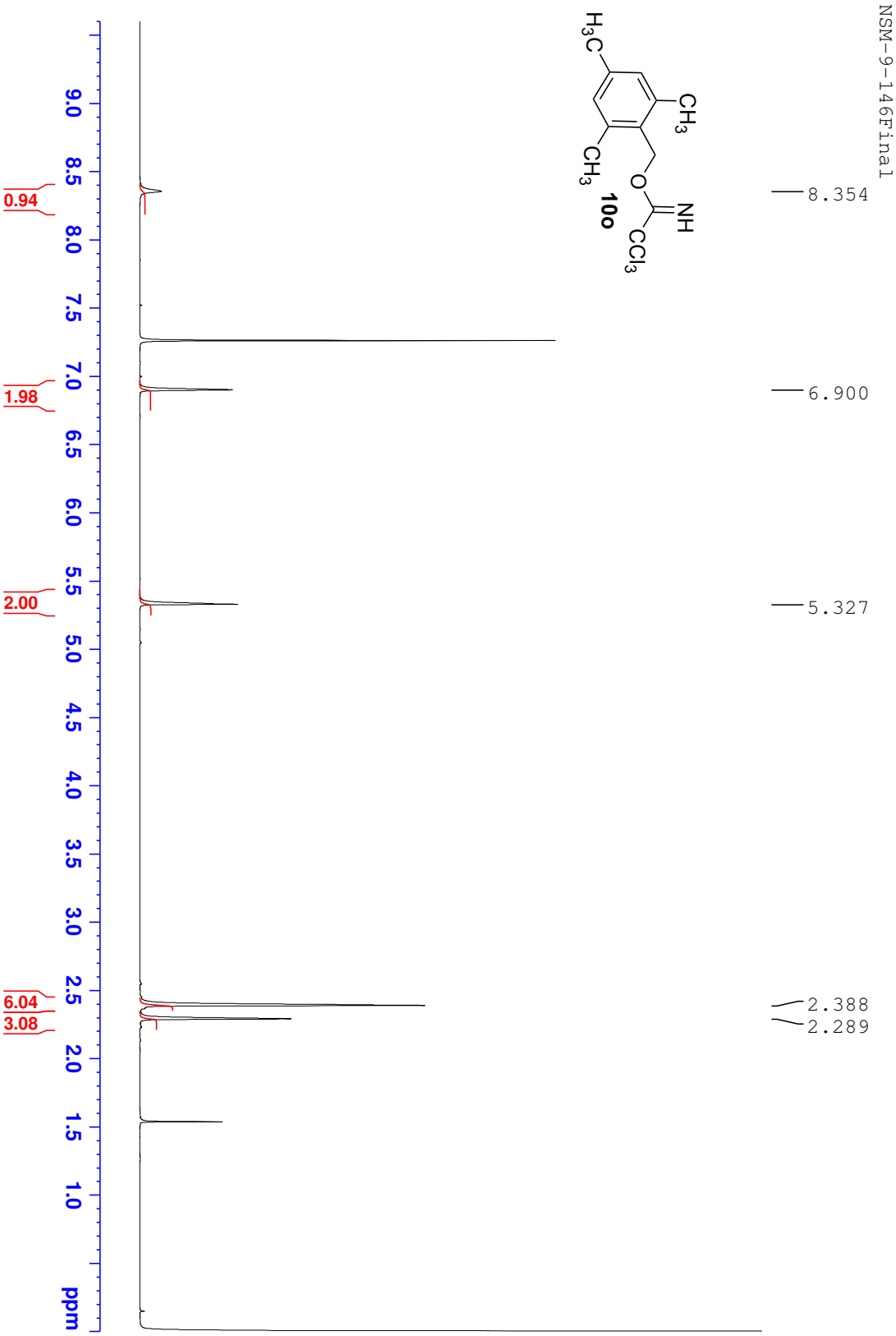
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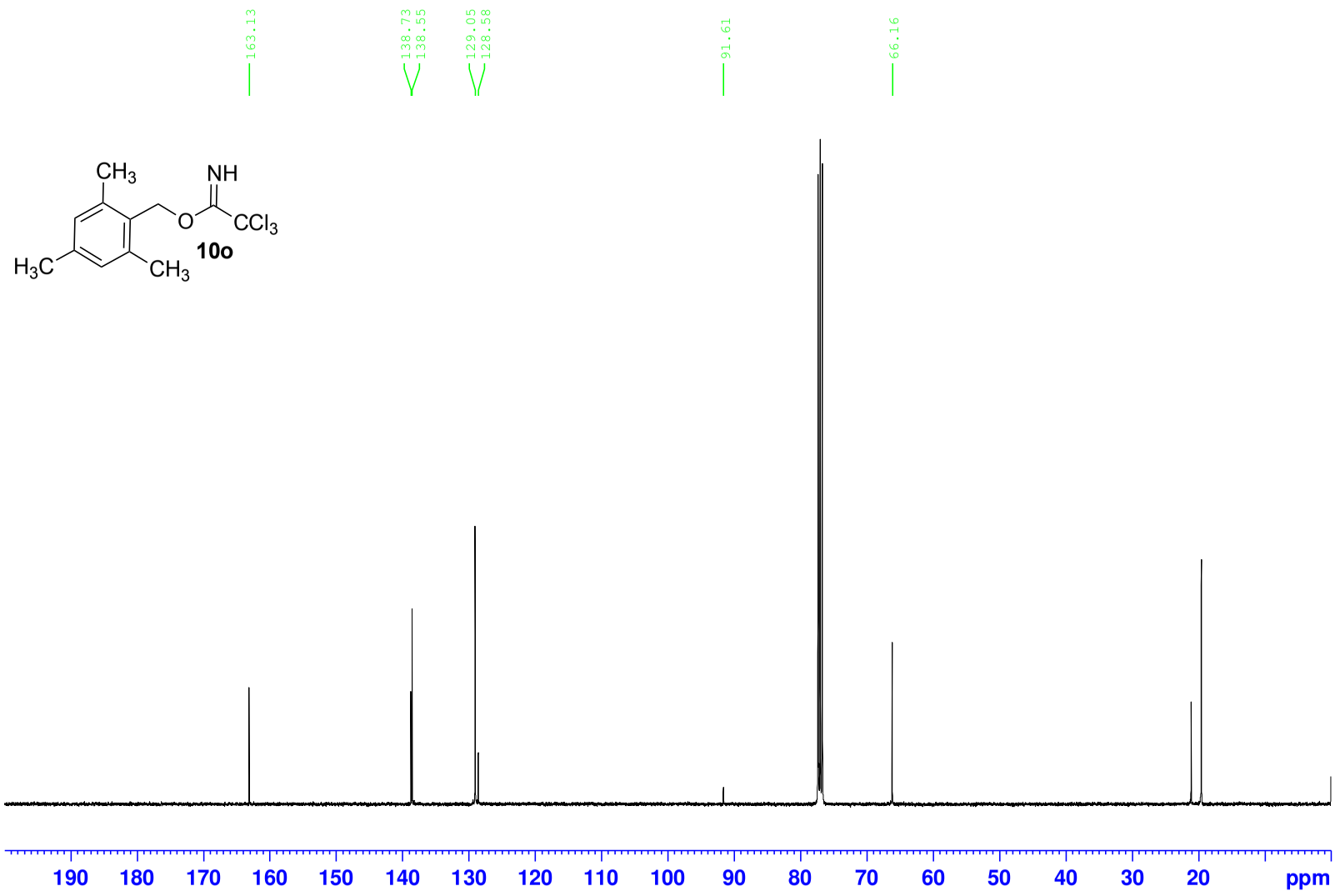
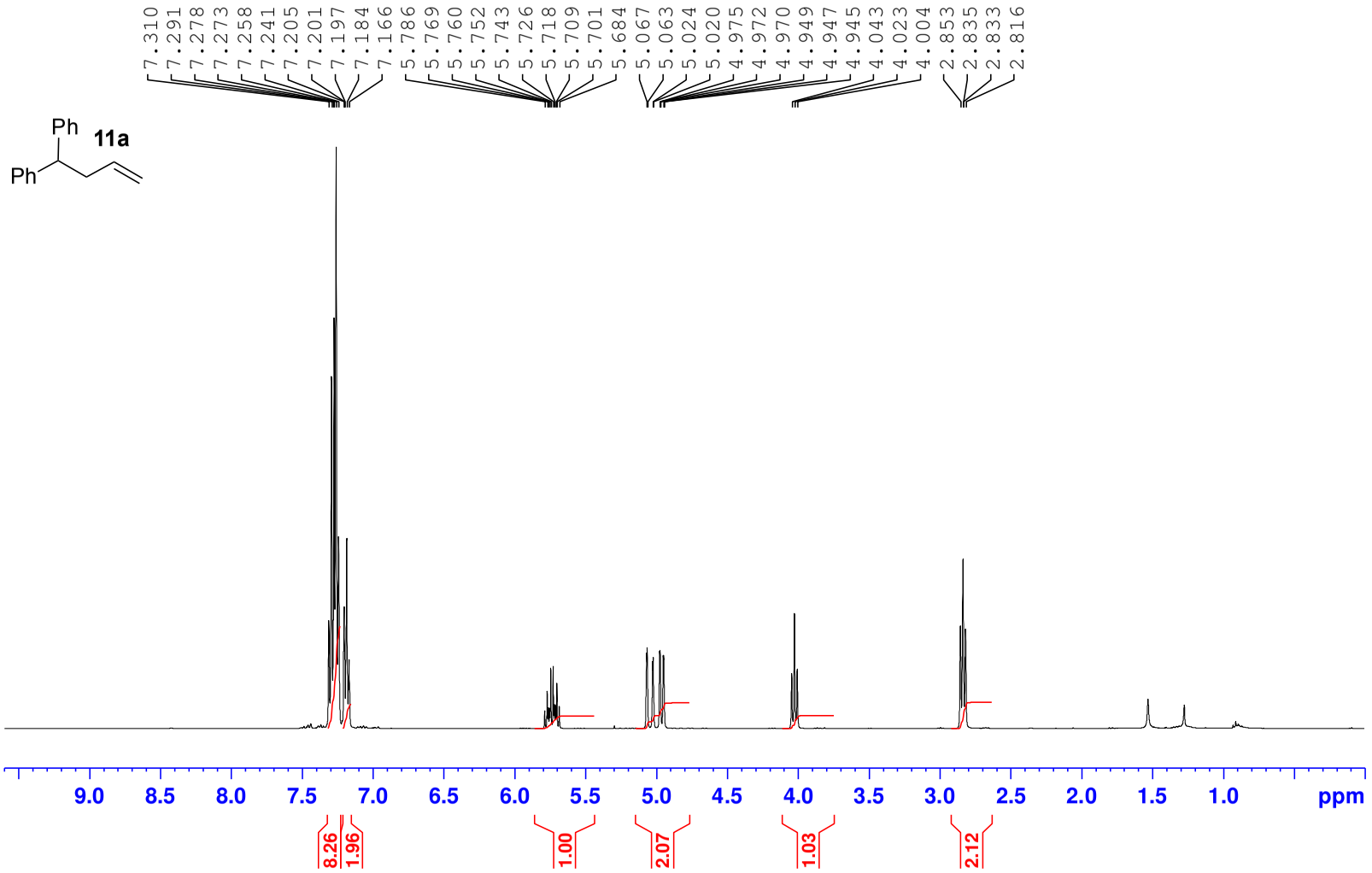
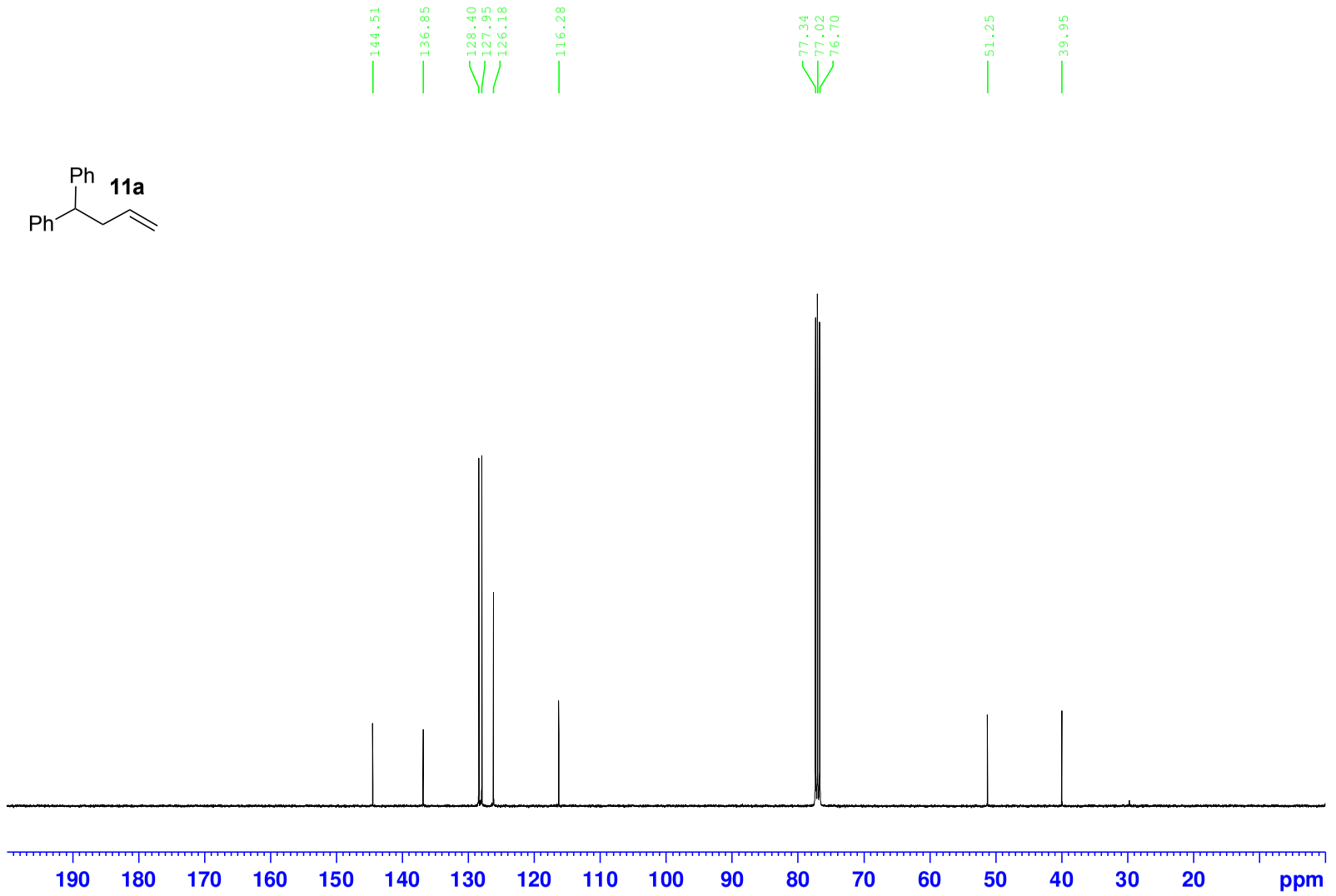


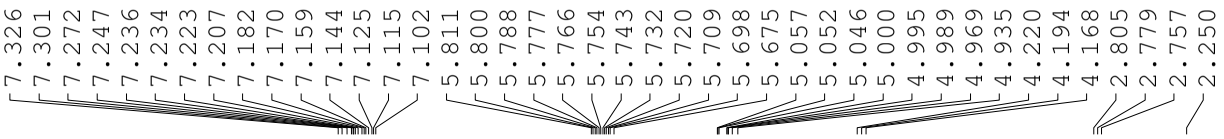
AMM-1-31.1Final

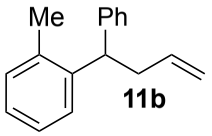


AMM-1-31.1FinalC13.400

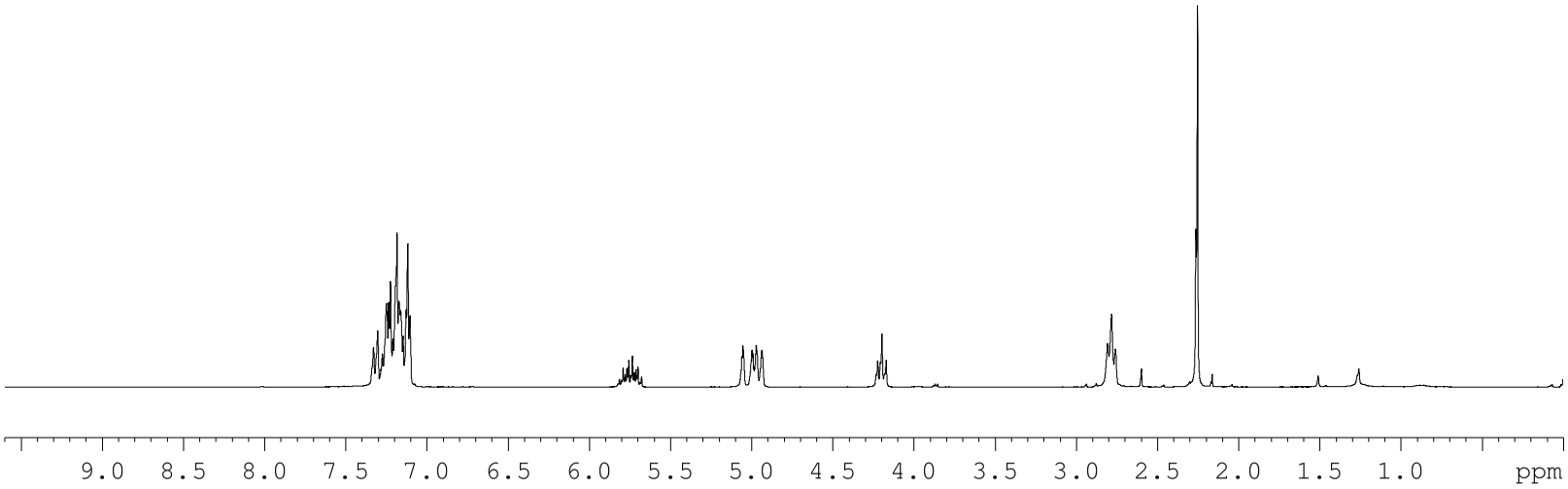


NSM-9-134Final.4



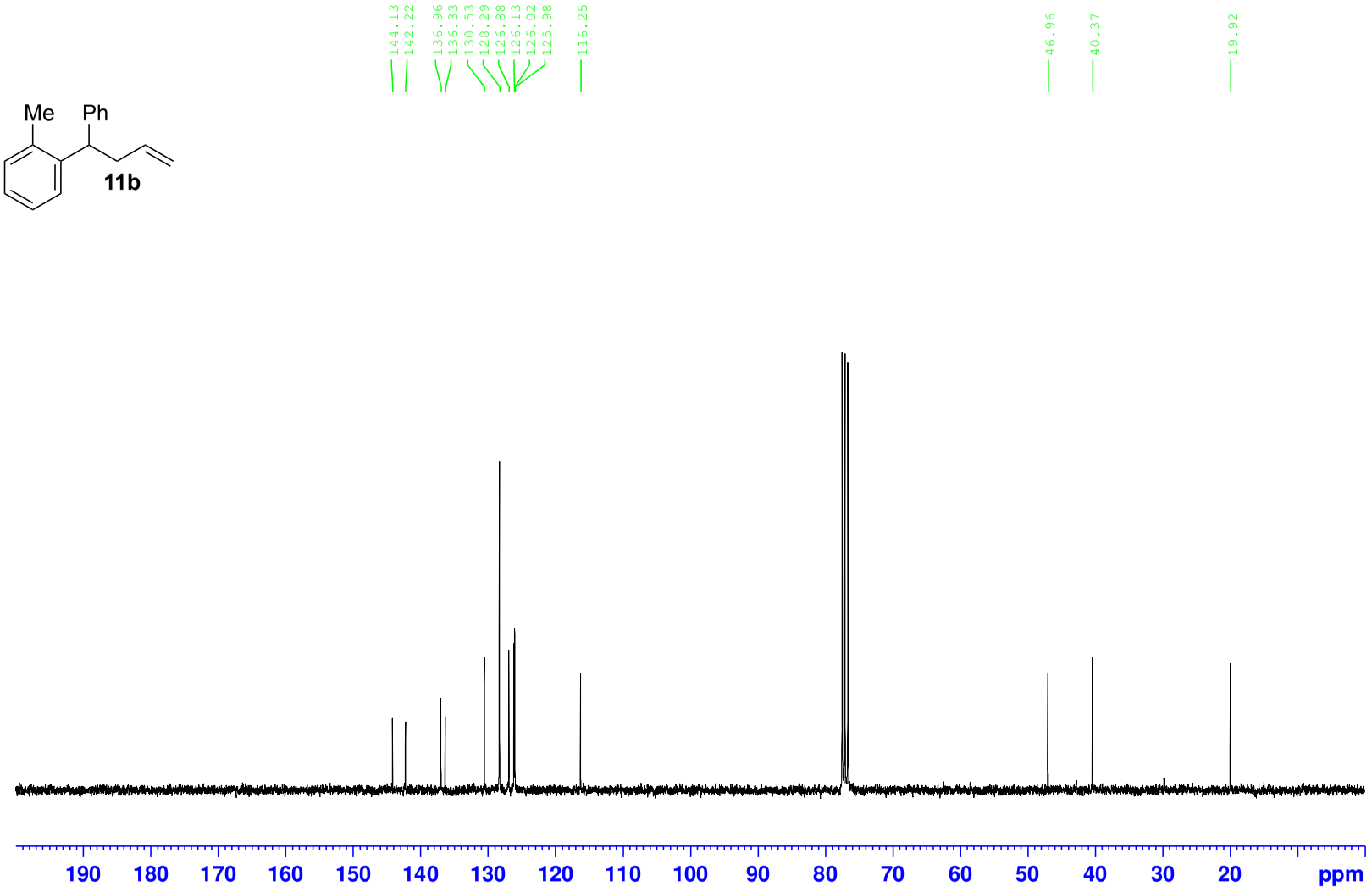


11b

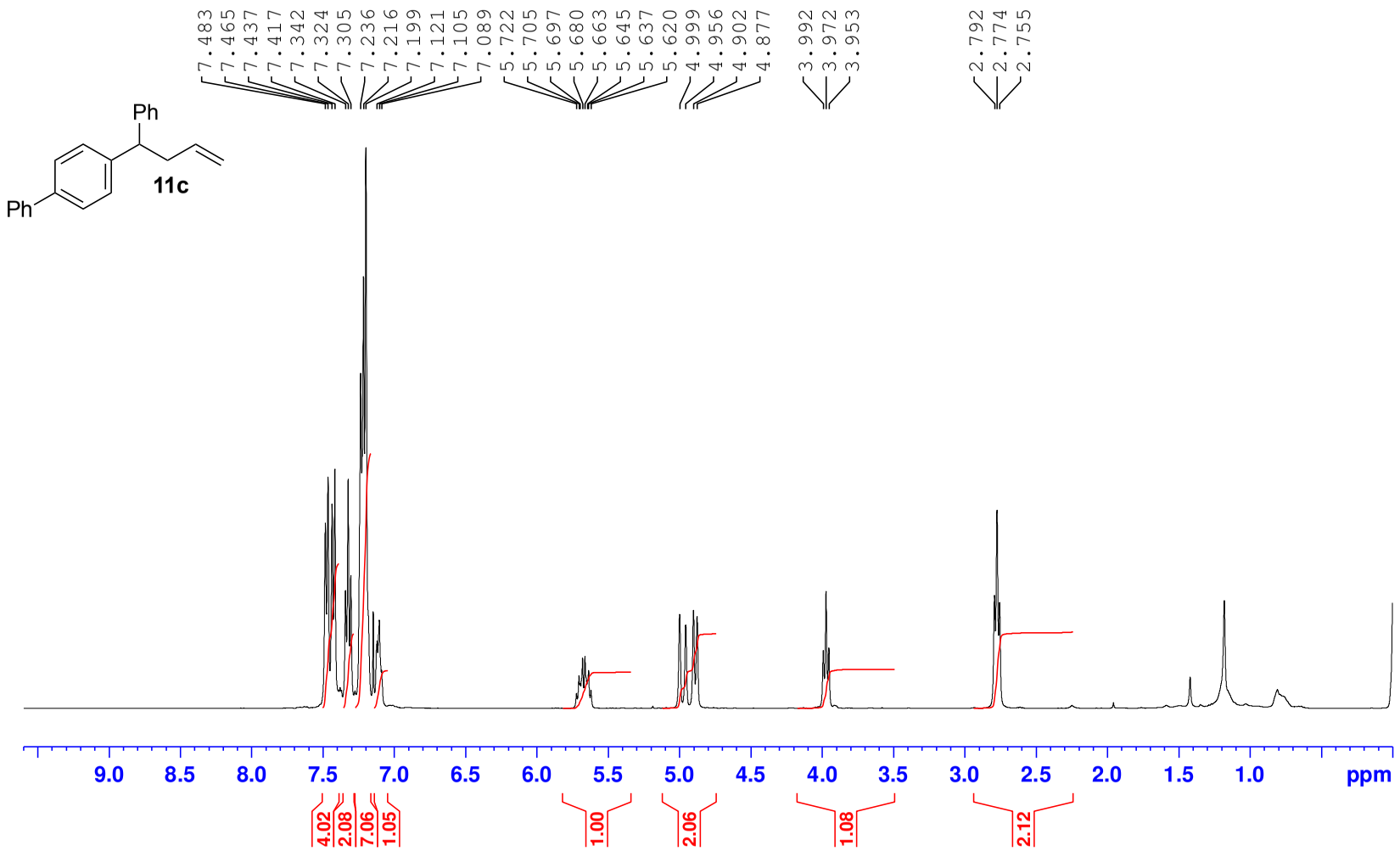


NSM-9-134FinalC13

11b

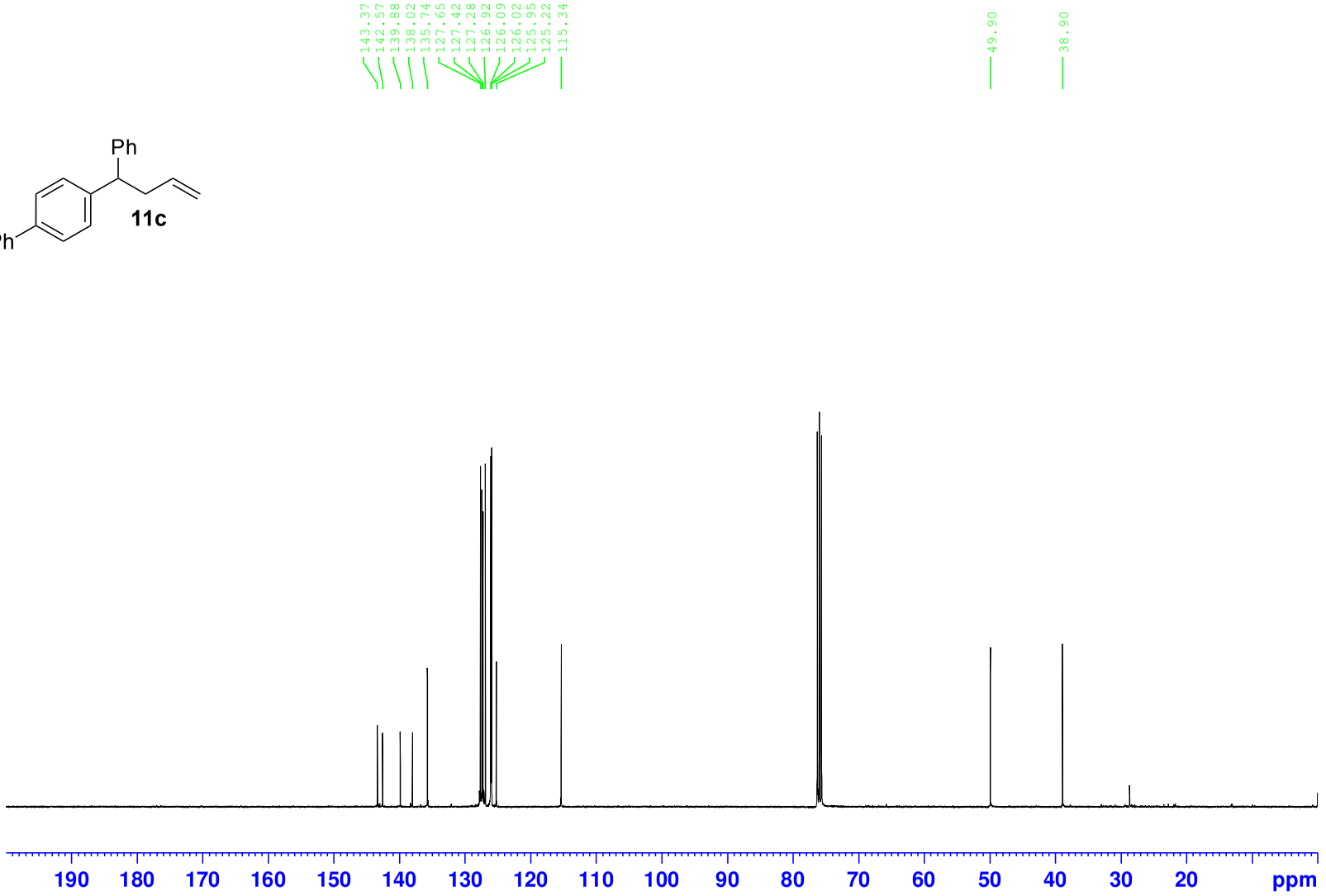


NSM-9-108final.4

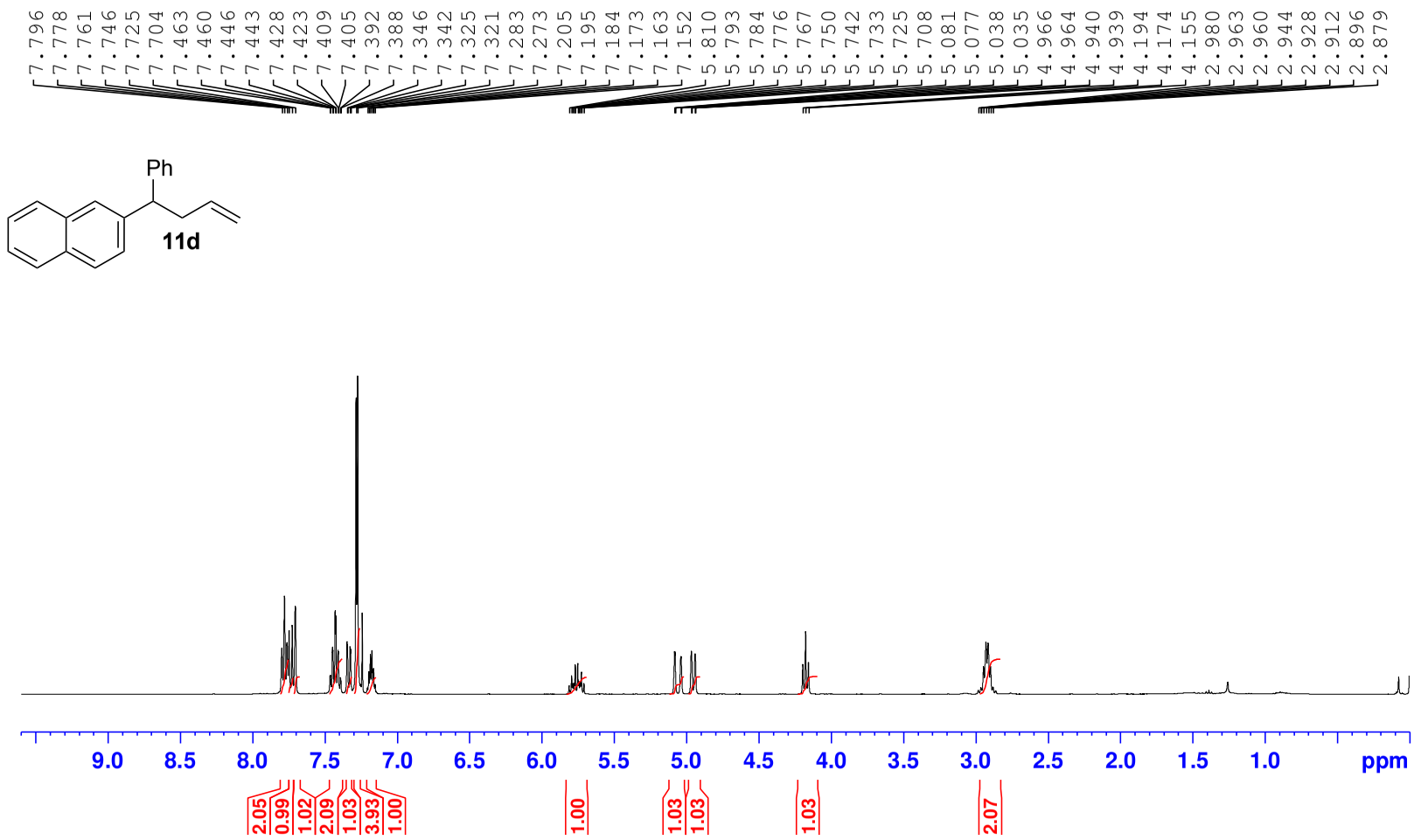


NSM-9-108finalC13

11c

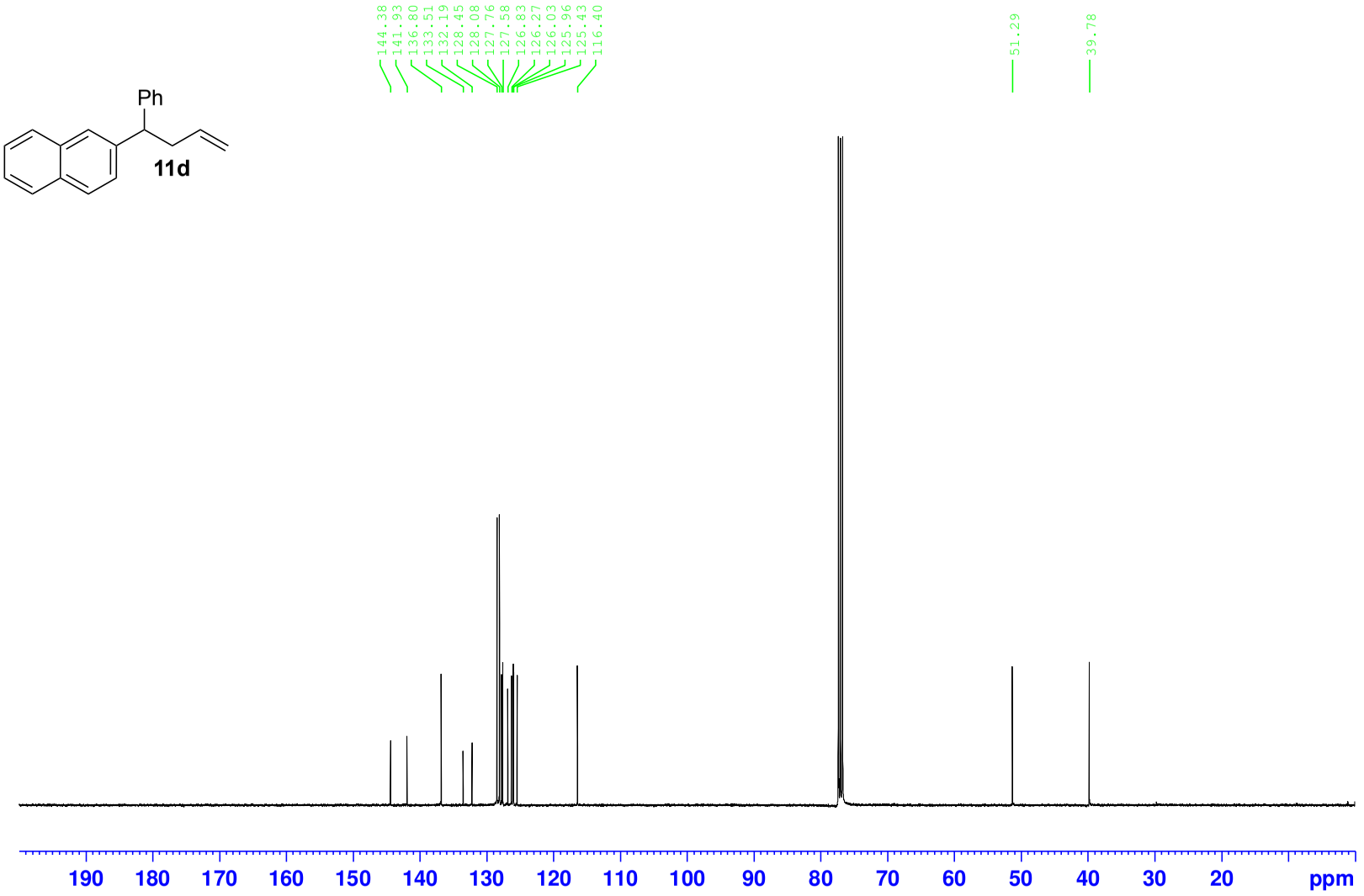


NSM-9-113Final

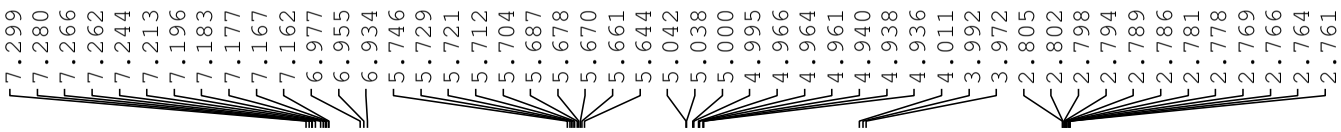


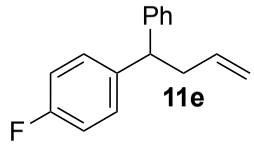
NSM-9-113Final

11d

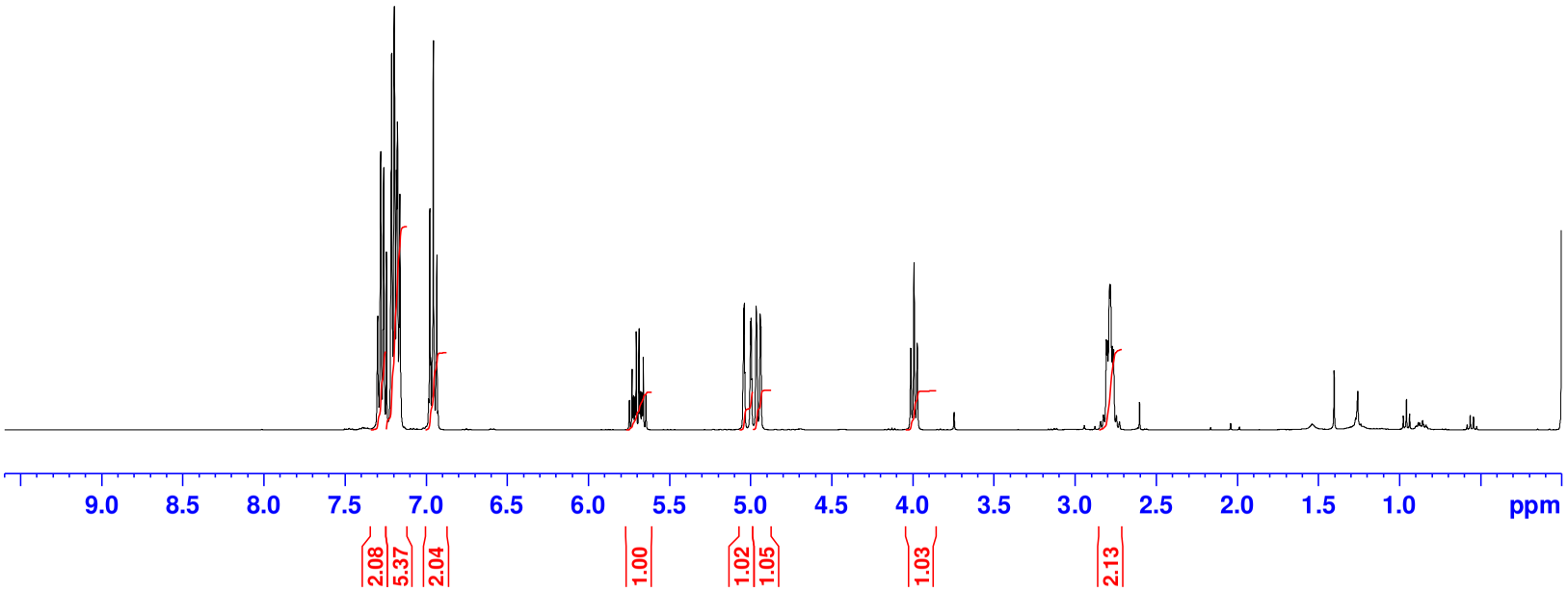


NSM-9-129Final.2

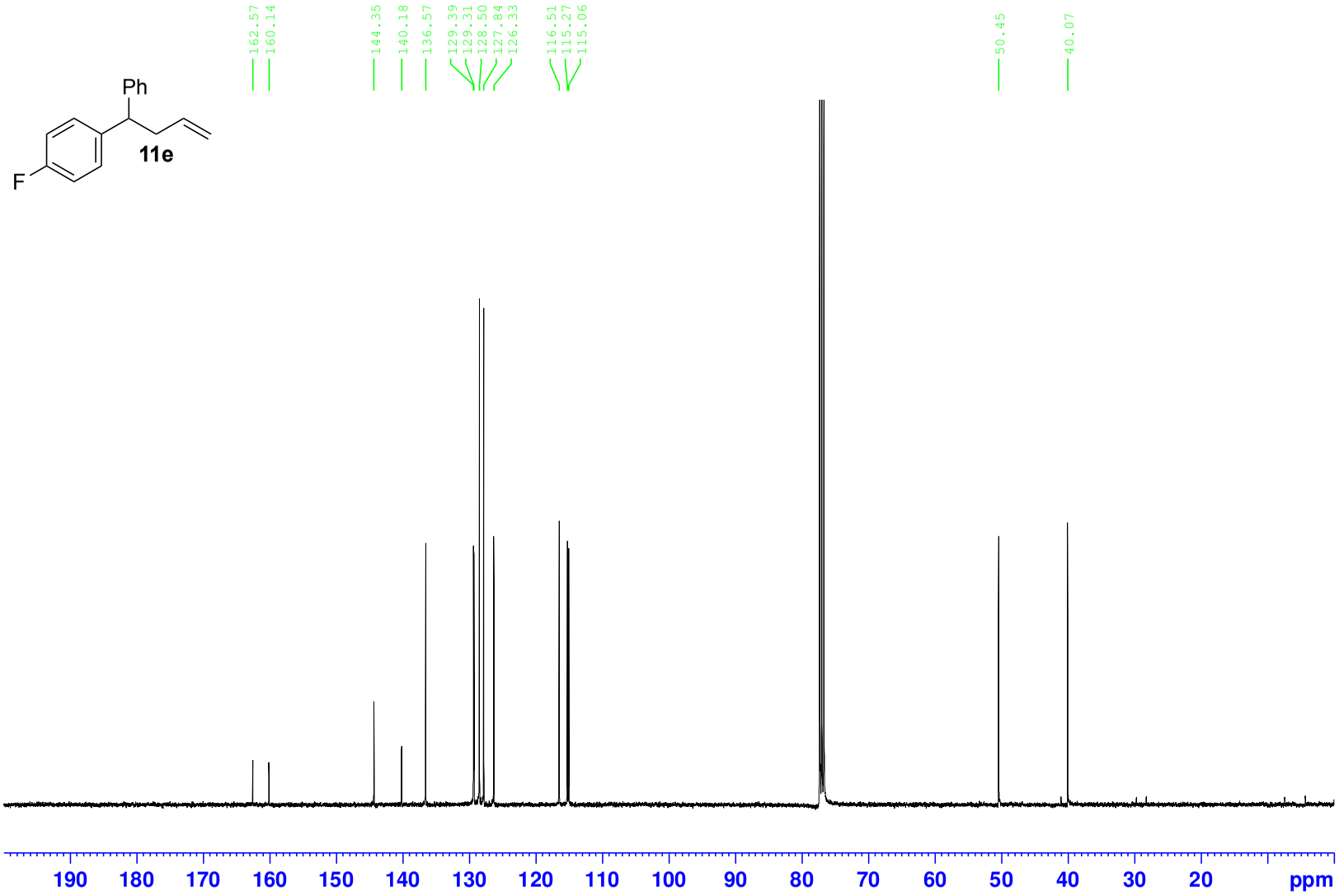




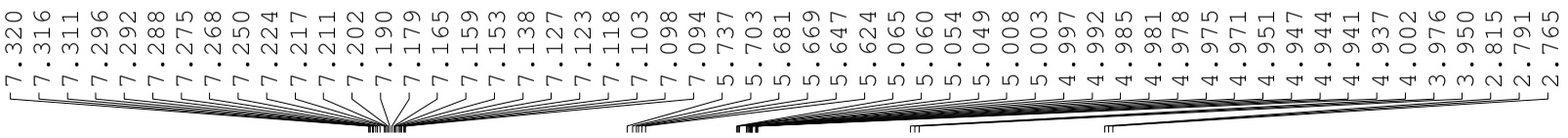
11e

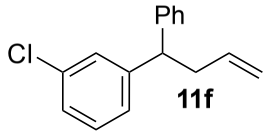


NSM-9-129FinalC13

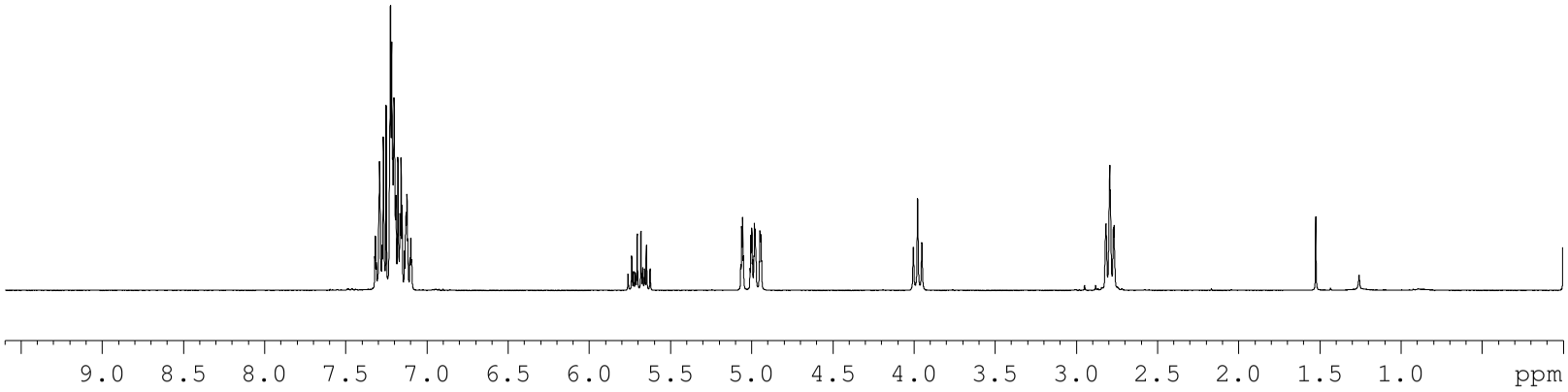


NSM-9-116Final.300

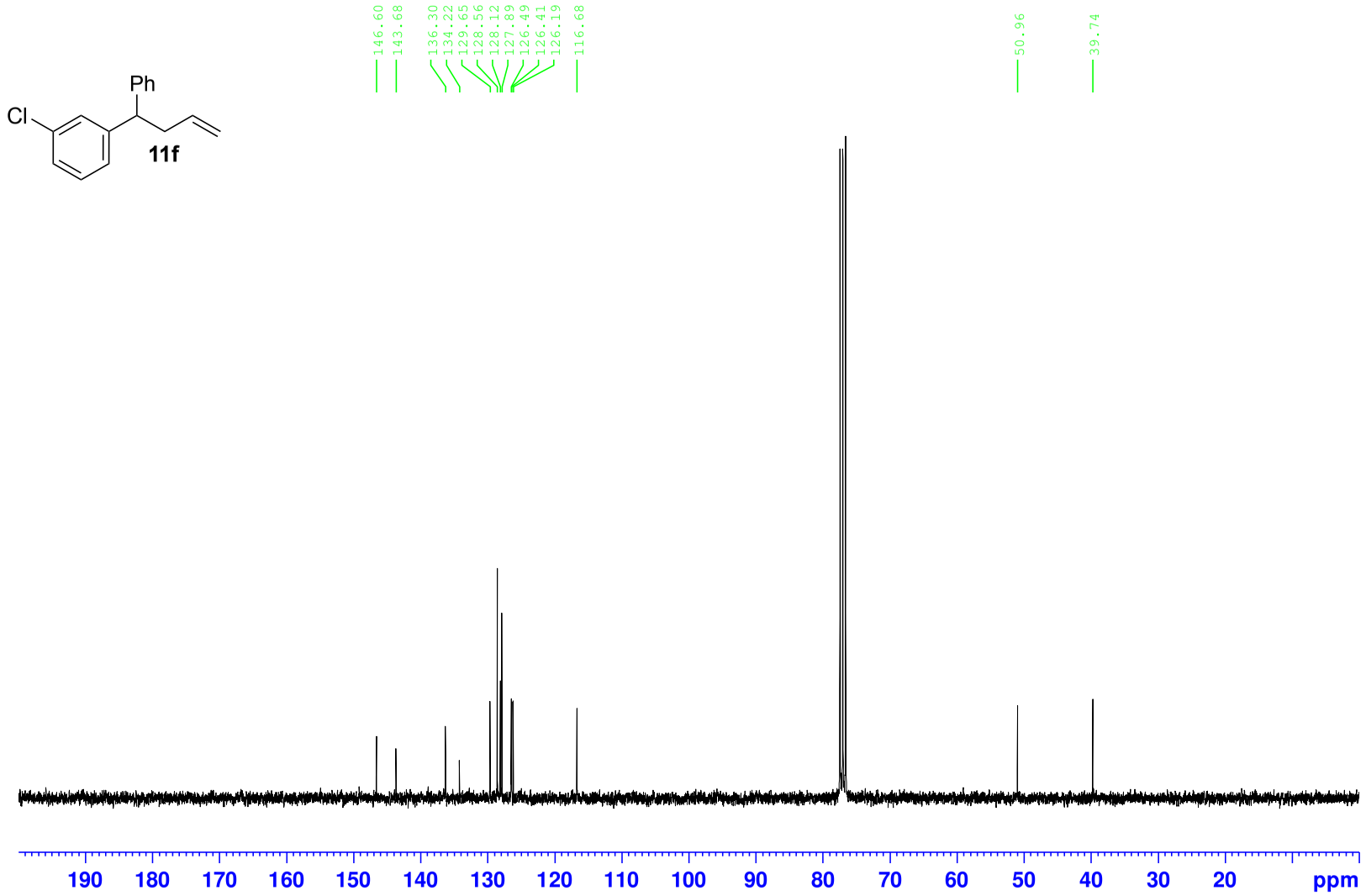




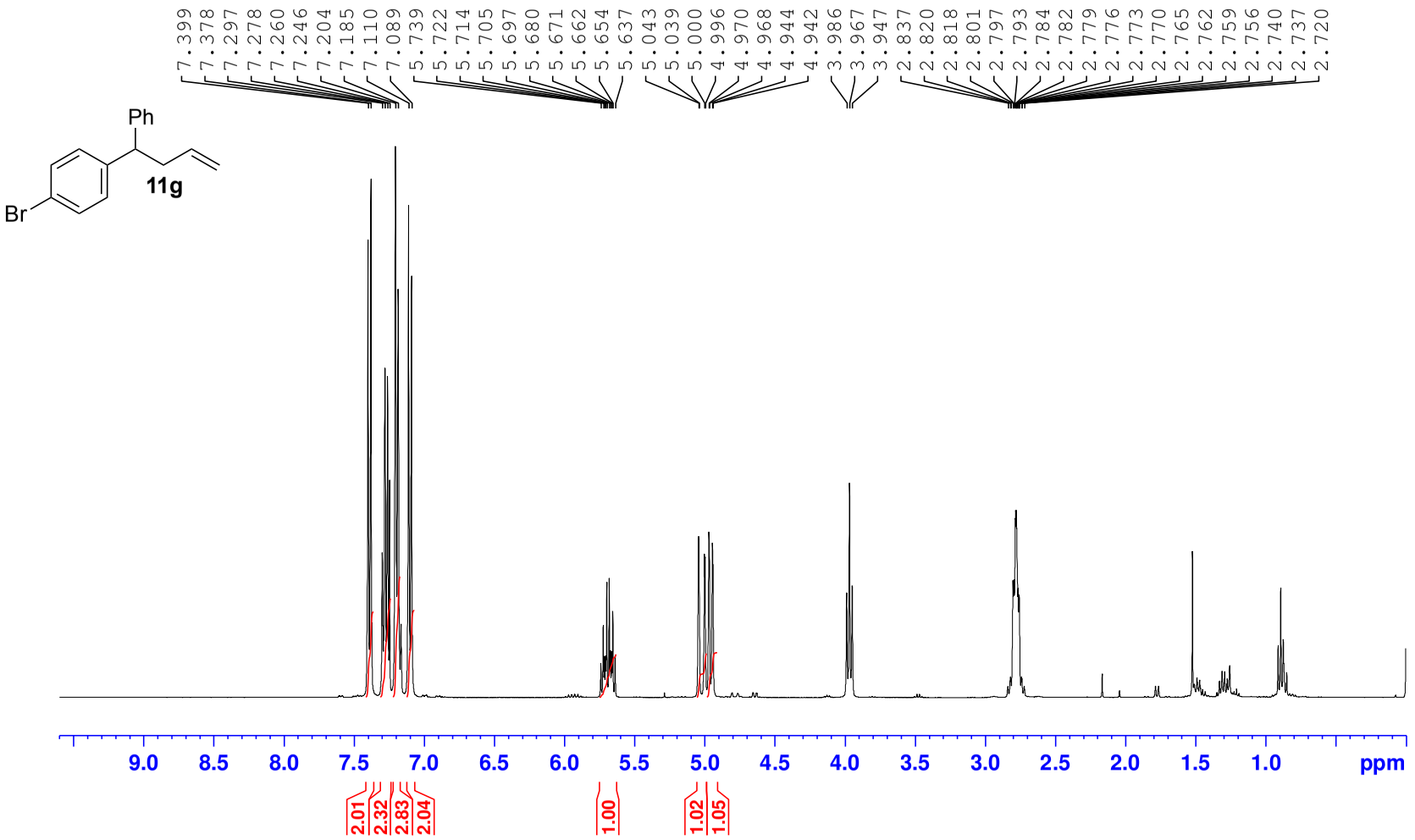
11f



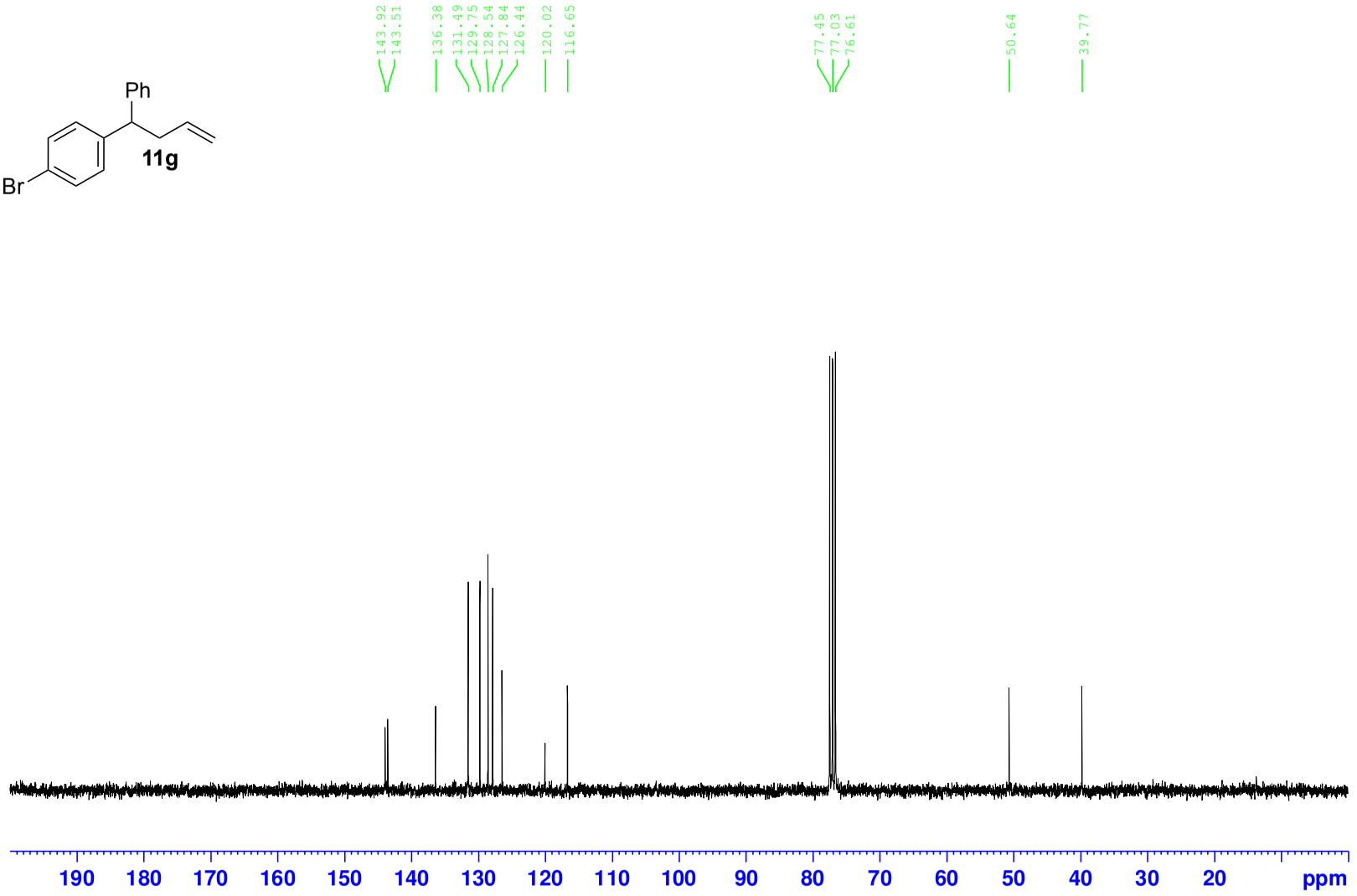
NSM-9-116FinalC13.300

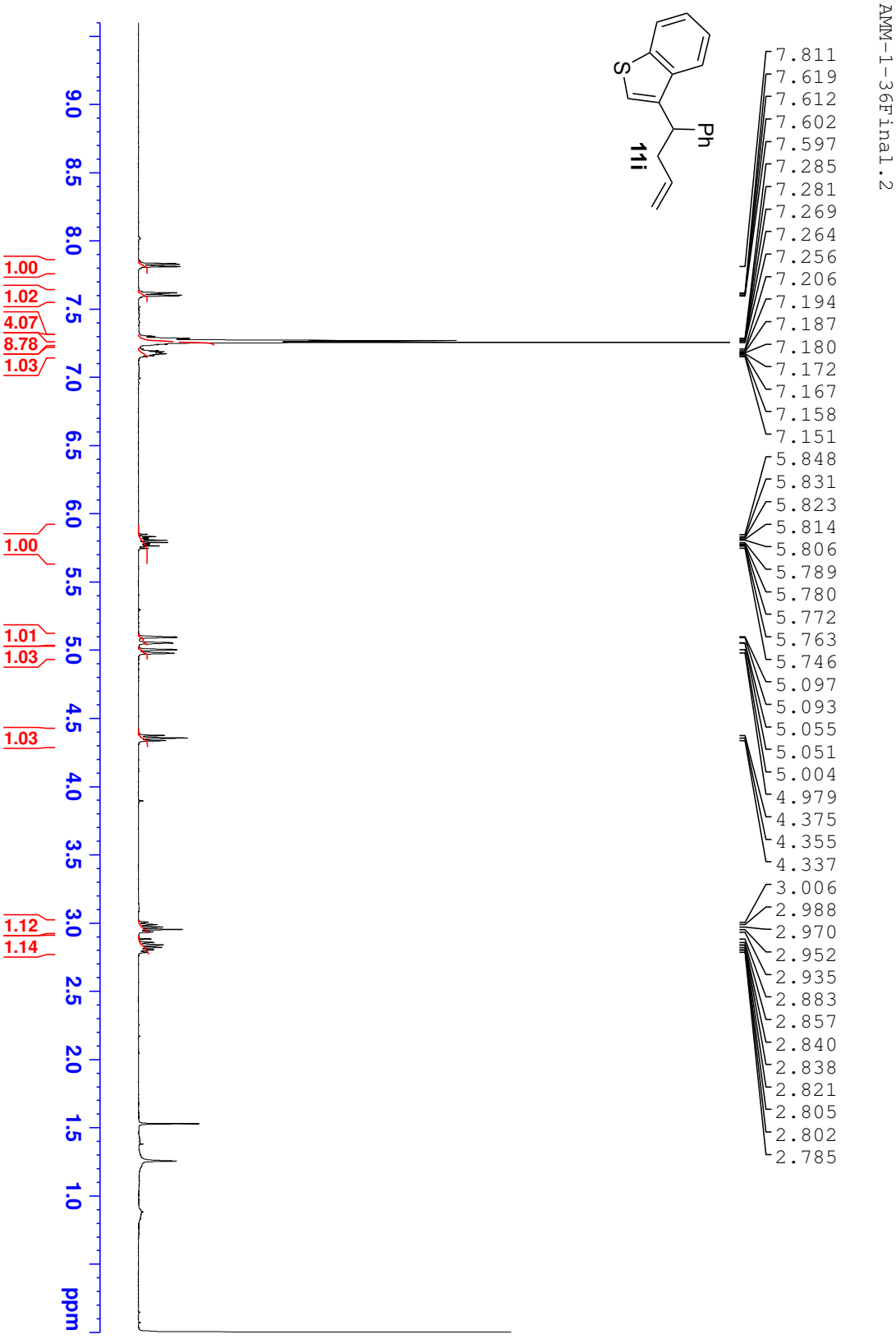


NSM-9-115Final

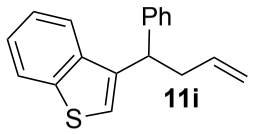


NSM-9-115FinalC13




AMM-1-36Final.2
7.811
7.619
7.612
7.602
7.597
7.285
7.281
7.269
7.264
7.256
7.206
7.194
7.187
7.180
7.172
7.167
7.158
7.151
5.848
5.831
5.823
5.814
5.806
5.789
5.780
5.772
5.763
5.746
5.097
5.093
5.055
5.051
5.004
4.979
4.375
4.355
4.337
3.006
2.988
2.970
2.952
2.935
2.883
2.857
2.840
2.838
2.821
2.805
2.802
2.785
S
Ph
11i
1.00
1.02
4.07
8.78
1.03
1.00
1.01
1.03
1.03
1.12
1.14
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

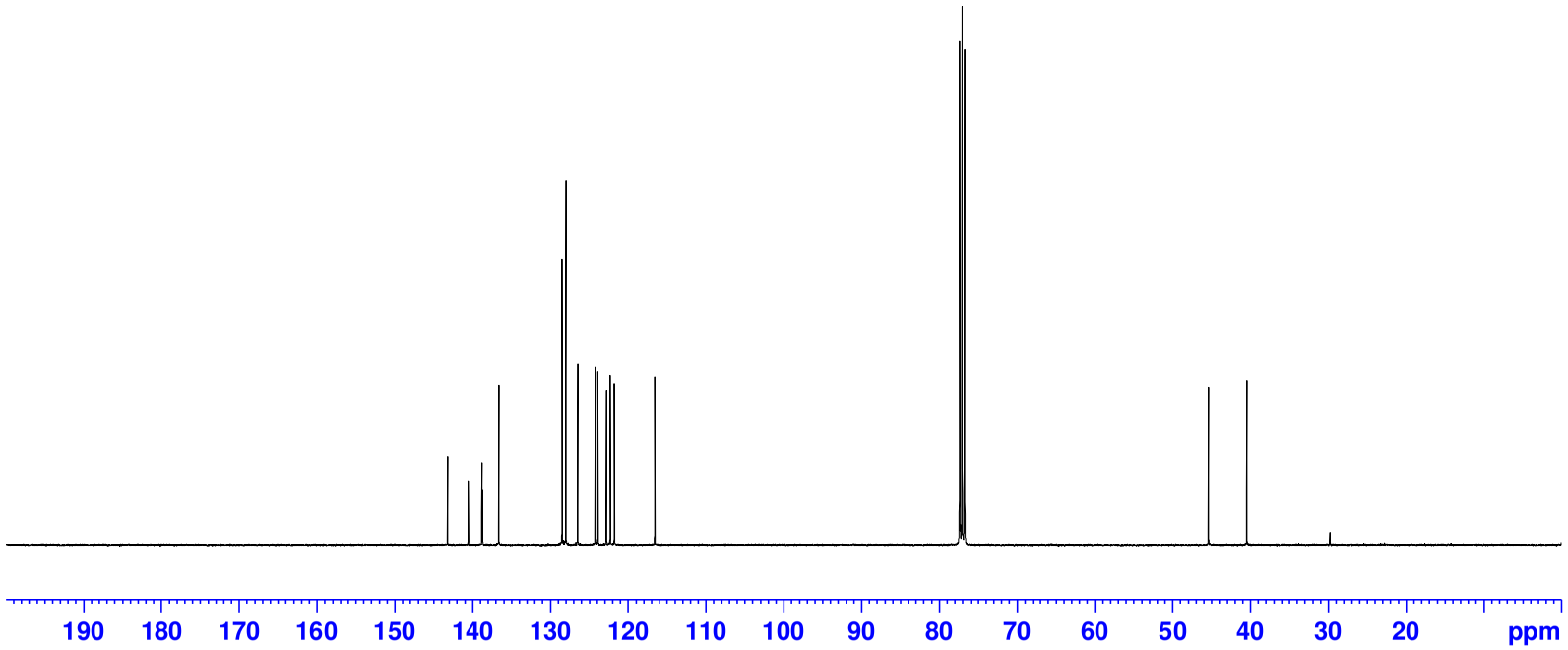
AMM-1-36FinalC13.400



11i

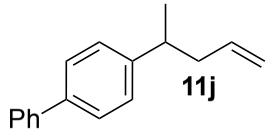
143.20
140.54
138.80
138.70
136.61
128.48
127.99
126.48
124.22
123.87
122.78
122.28
121.76
116.57

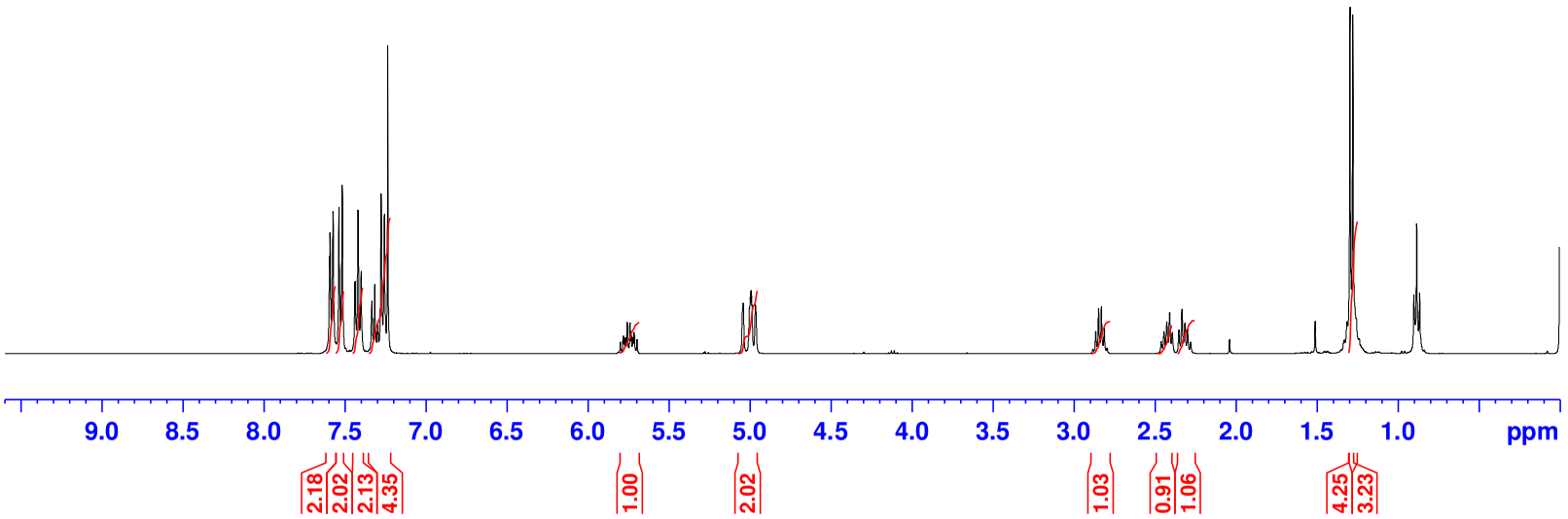
45.37
40.44



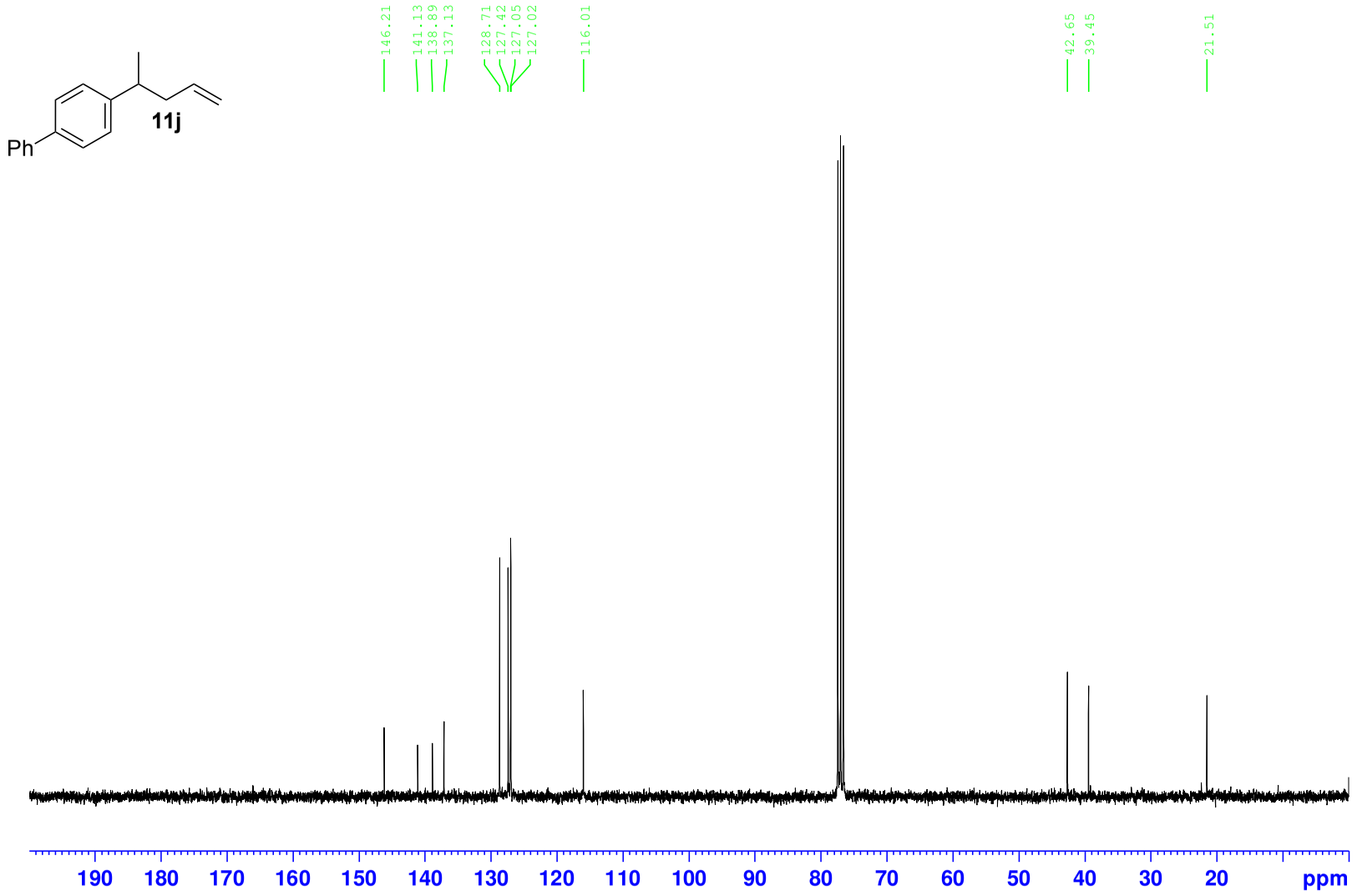
NSM-9-152.2Final 2

7.591 7.573 7.536 7.516 7.437 7.418 7.399 7.333 7.315 7.276 7.255 7.235 5.799 5.782 5.780 5.773 5.763 5.756 5.738 5.731 5.721 5.714 5.712 5.696 5.045 5.040 5.002 5.000 4.997 4.993 4.991 4.989 4.986 4.966 4.963 4.961 2.881 2.864 2.846 2.828 2.811 2.793 2.459 2.456 2.442 2.424 2.407 2.394 2.391 2.350 2.331 2.315 2.312 2.296 2.277 1.294 1.276

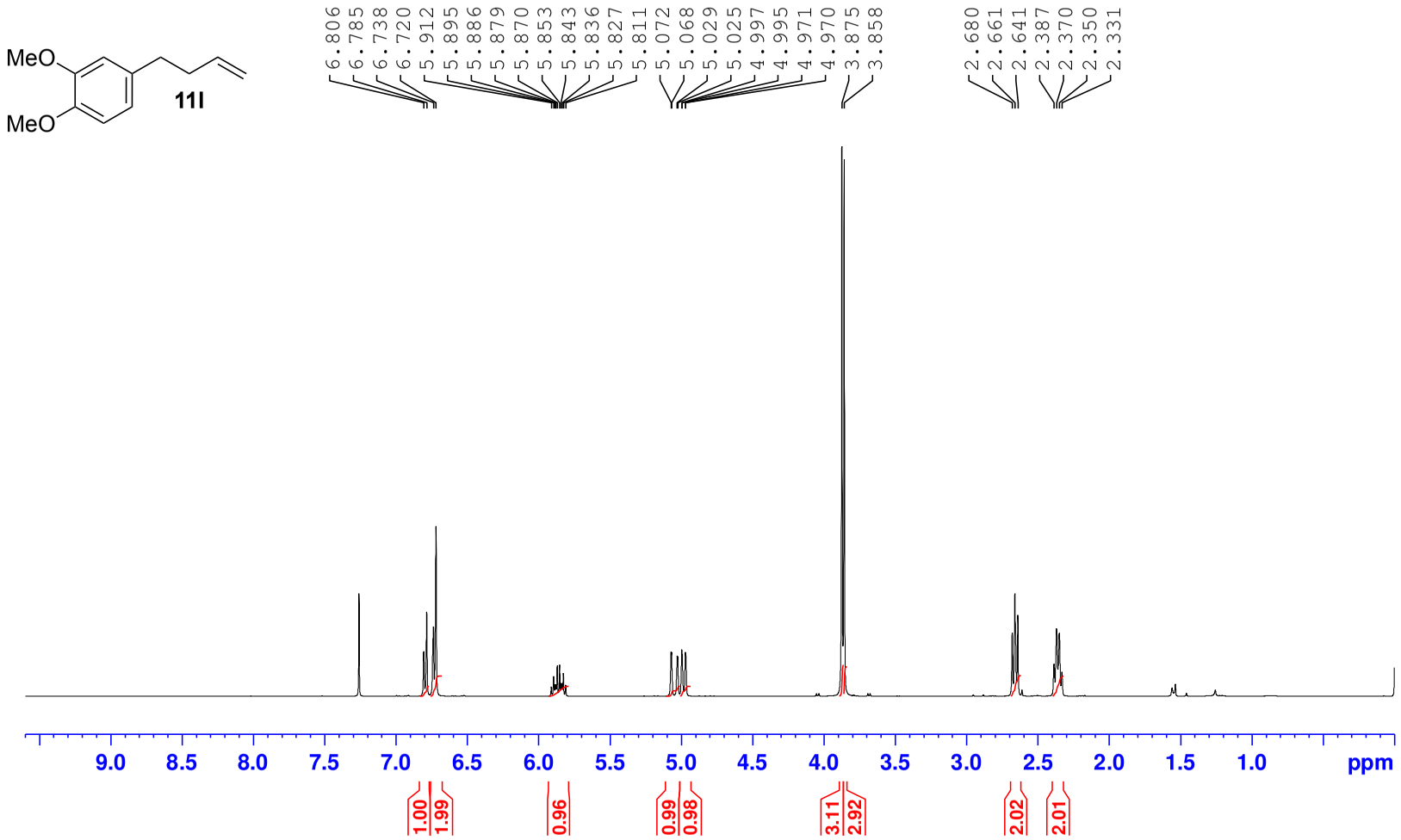




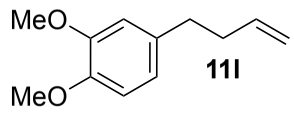
NSM-9-152.2final.C13



NSM-9-131.3

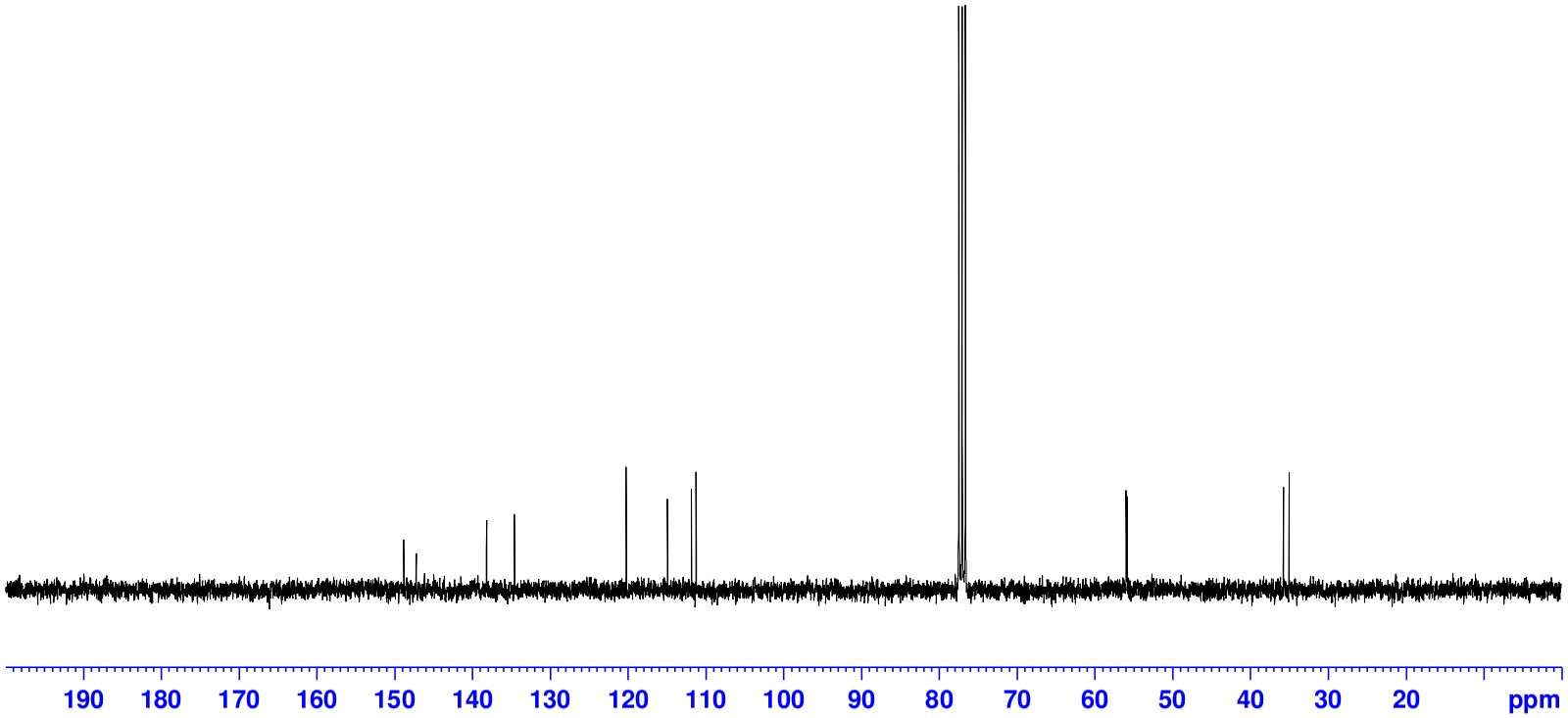

MeO
MeO
11l
6.806
6.785
6.738
6.720
5.912
5.895
5.886
5.879
5.870
5.853
5.843
5.836
5.827
5.811
5.072
5.068
5.029
5.025
4.997
4.995
4.971
4.970
3.875
3.858
2.680
2.661
2.641
2.387
2.370
2.350
2.331
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
1.00
1.99
0.96
0.99
0.98
3.11
2.92
2.02
2.01

SM-9-131FinalC13.300

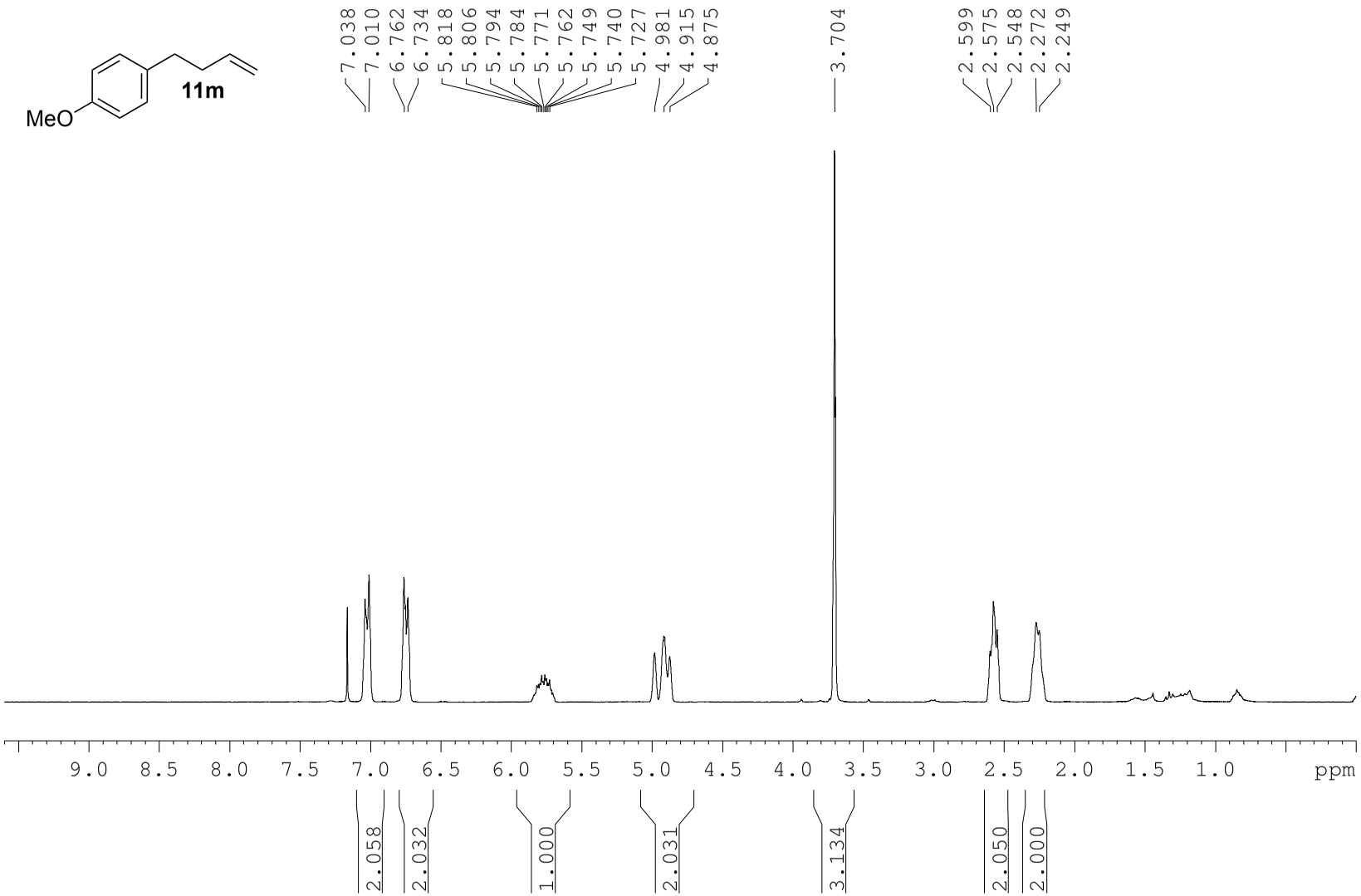


11l

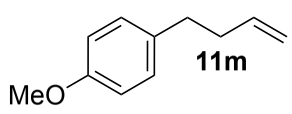
148.79
147.20
138.15
134.56
120.20
114.90
111.84
111.22
55.93
35.71
34.99



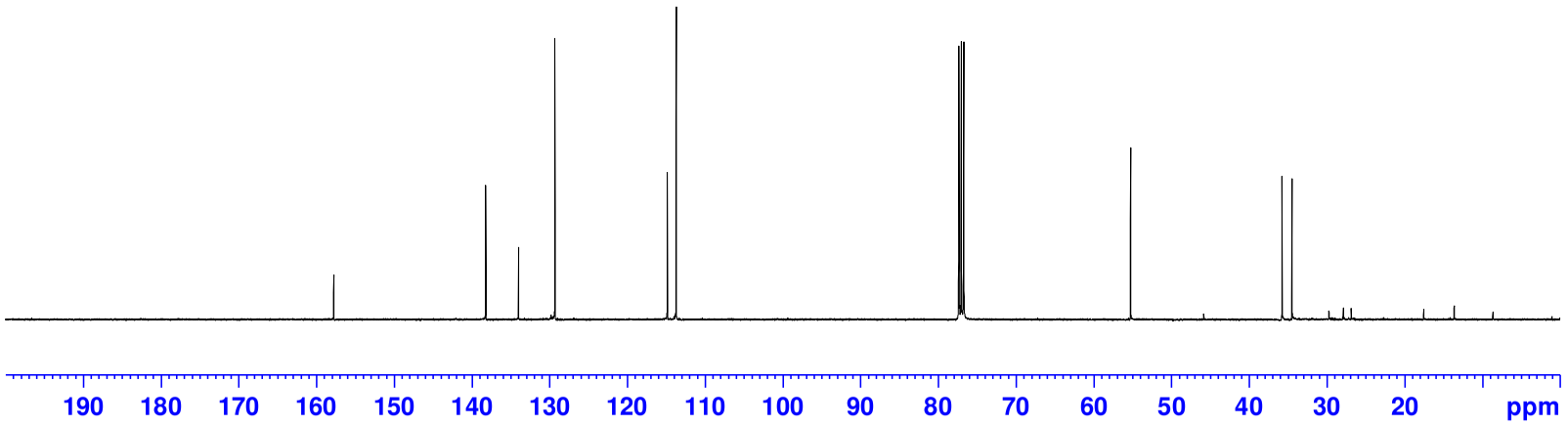
NSM-9-105Final



NSM-9-105FinalC13.400

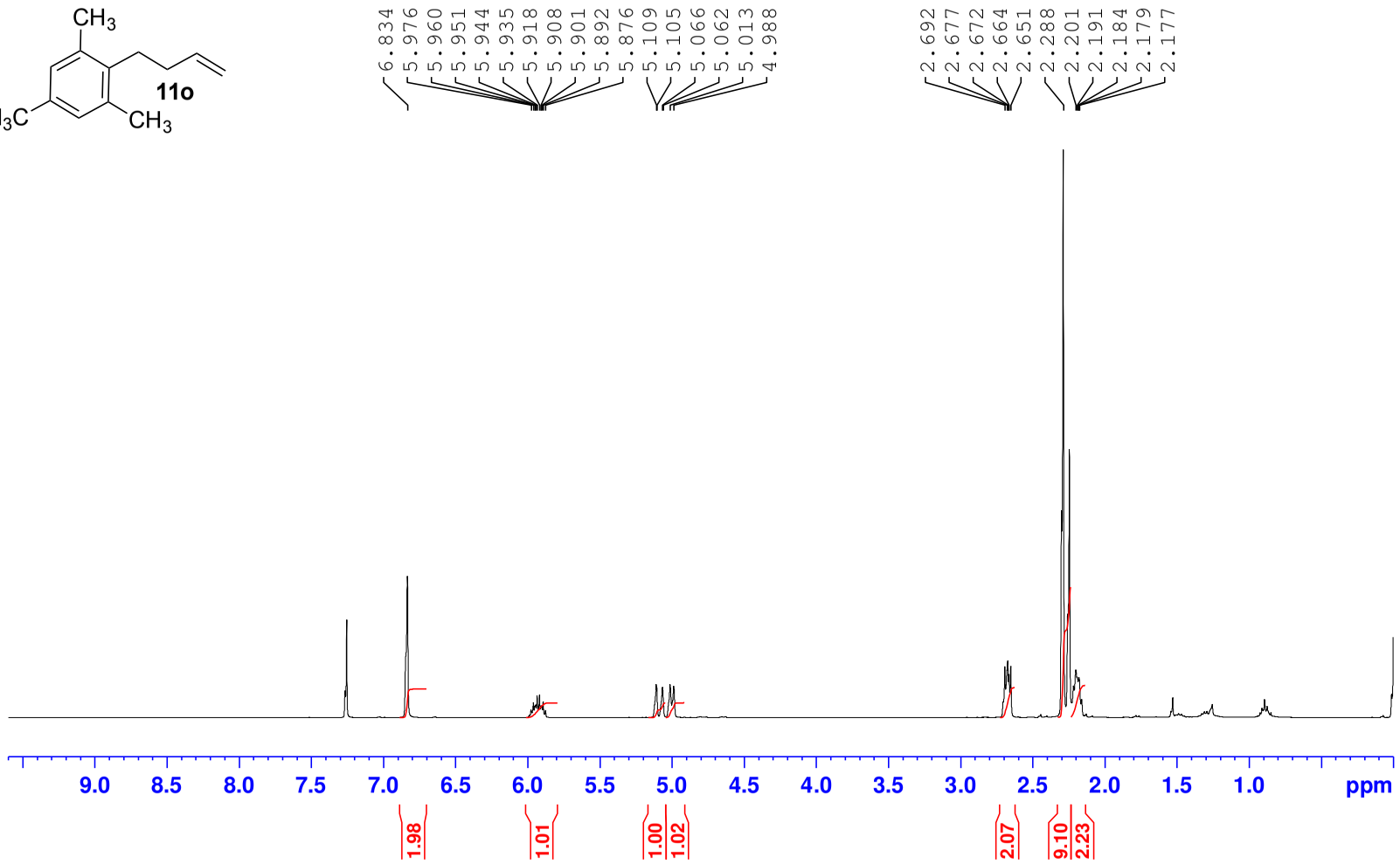


157.78
138.21
133.98
129.31
114.85
113.72
55.25
35.78
34.49

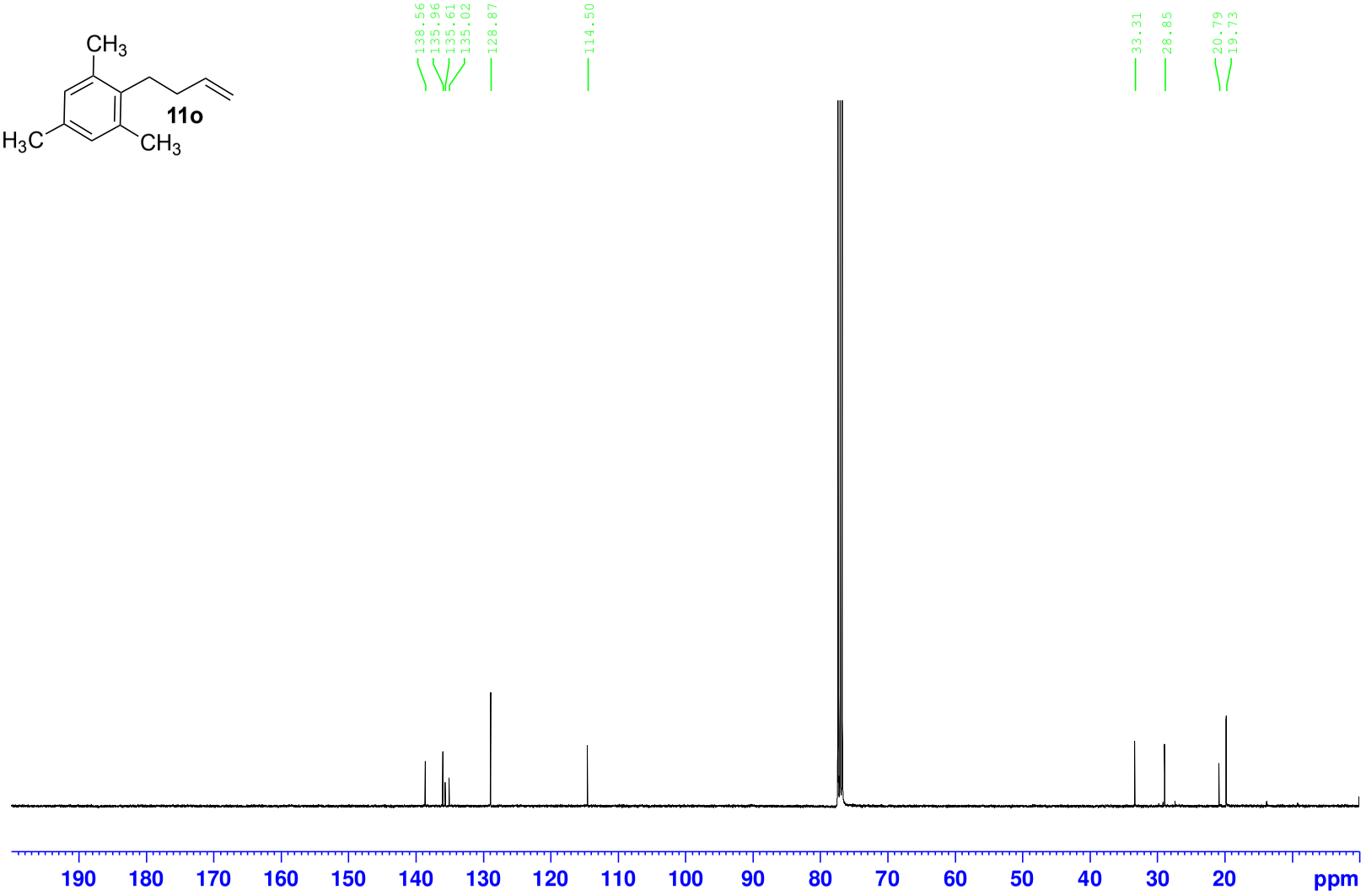


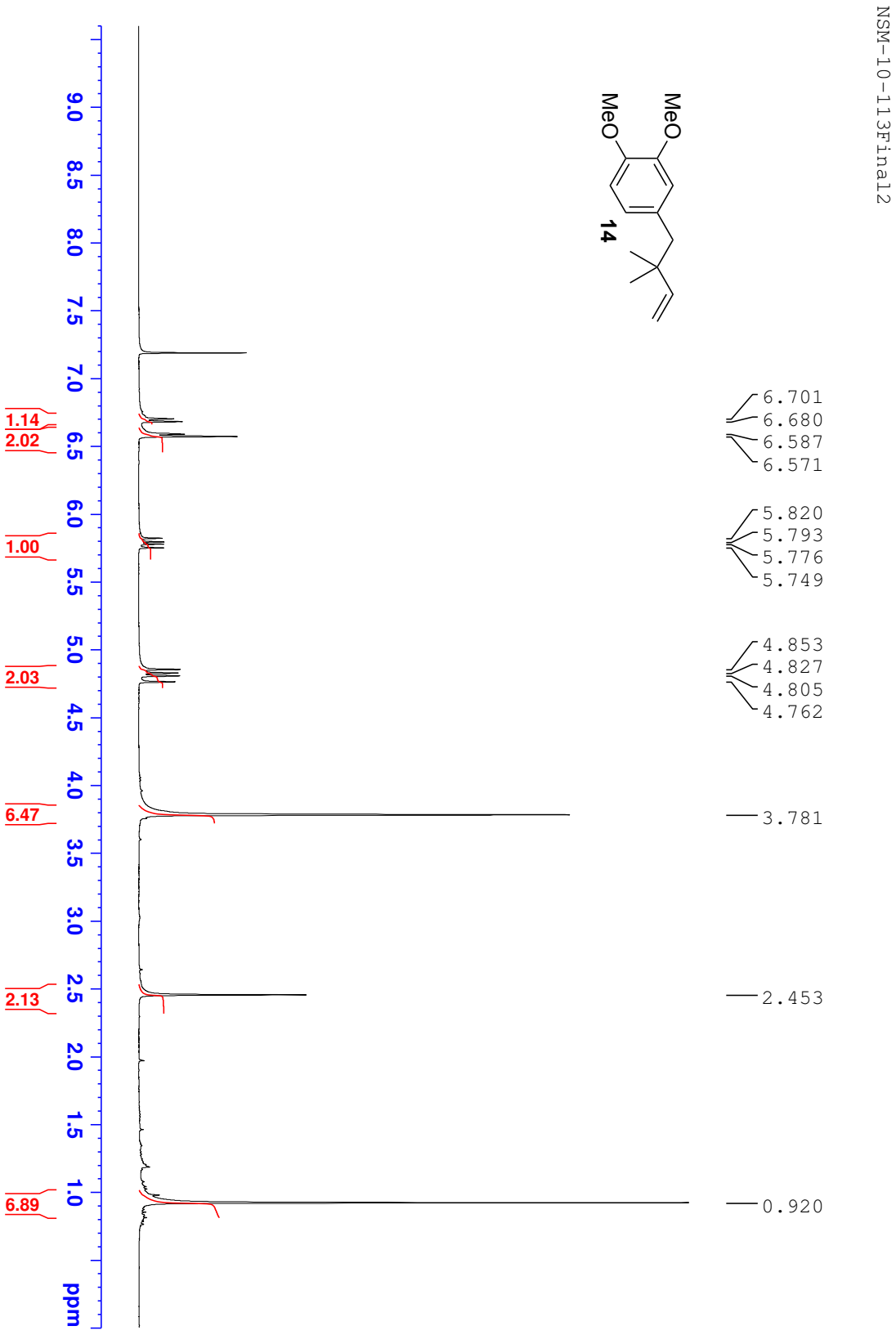
NSM-10-20C2

CH3
11o
H3C
CH3

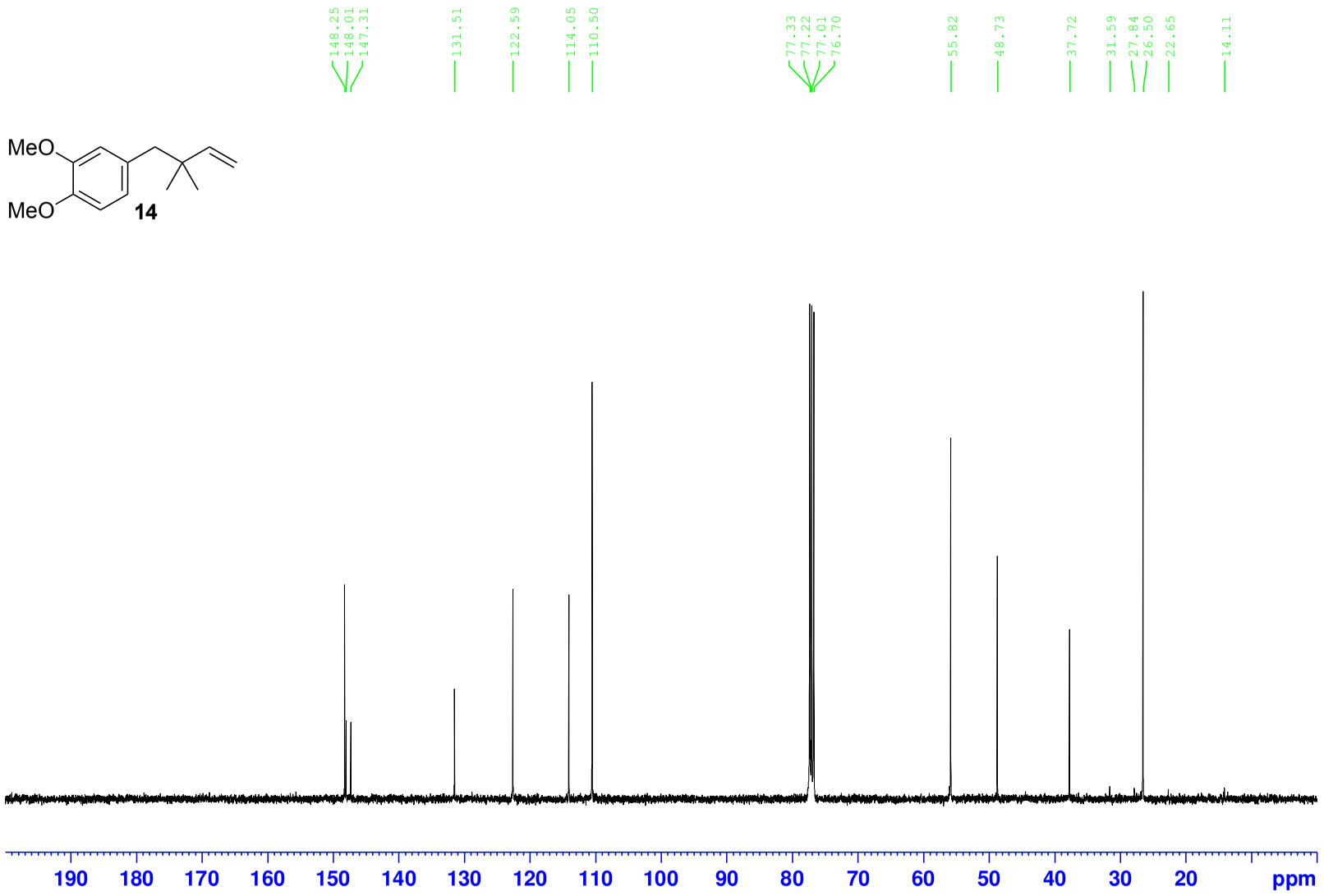

6.834
5.976
5.960
5.951
5.944
5.935
5.918
5.908
5.901
5.892
5.876
5.109
5.105
5.066
5.062
5.013
4.988
2.692
2.677
2.672
2.664
2.651
2.288
2.201
2.191
2.184
2.179
2.177
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
1.98
1.01
1.00
1.02
2.07
9.10
2.23

NSM-10-20C2FinalC13

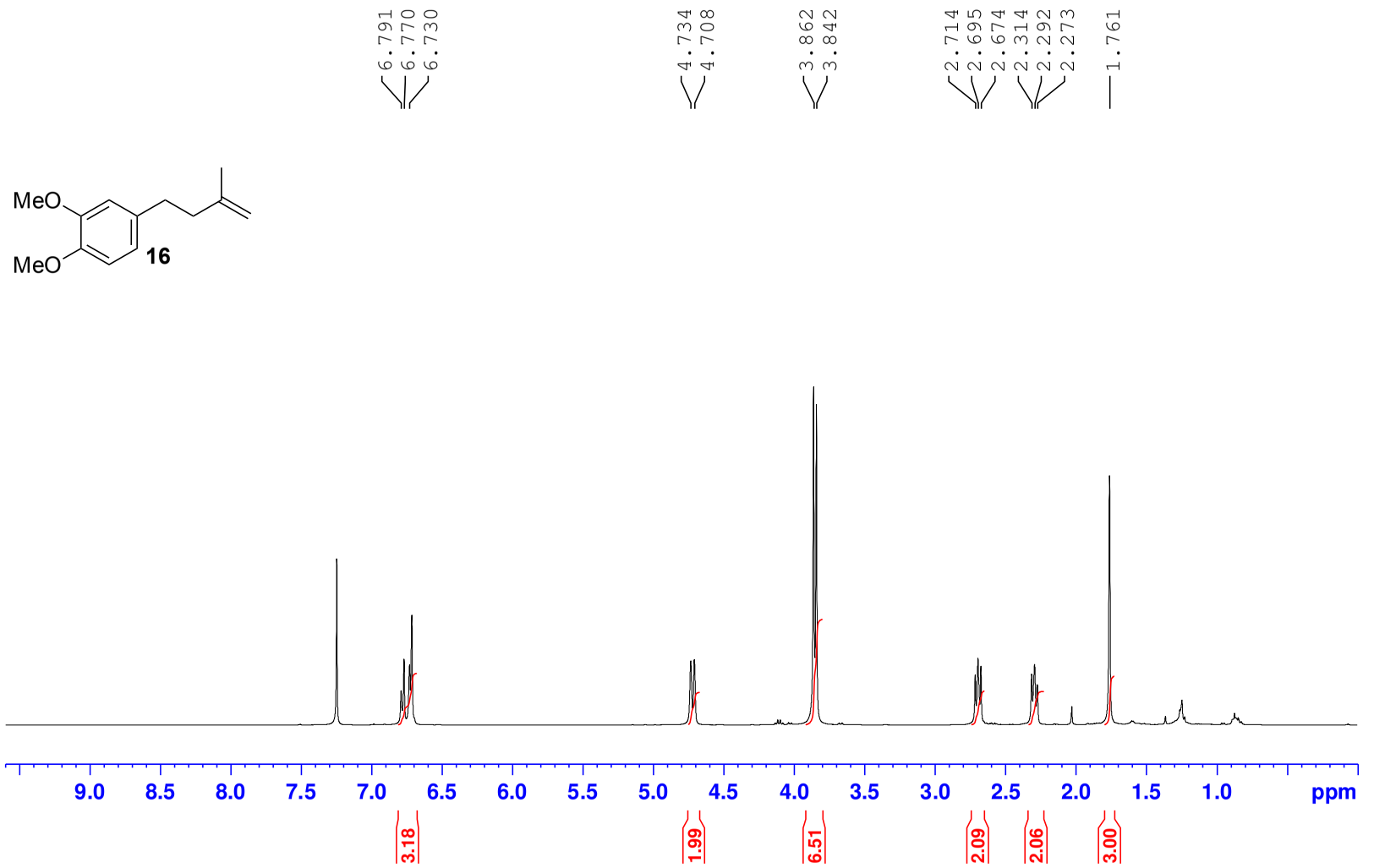



NSM-10-113Final2
MeO
MeO
14
6.701
6.680
6.587
6.571
5.820
5.793
5.776
5.749
4.853
4.827
4.805
4.762
3.781
2.453
0.920
1.14
2.02
1.00
2.03
6.47
2.13
6.89
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

NSM-10-113FinalC13

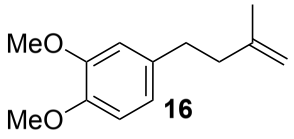


NSM-10-111Final2

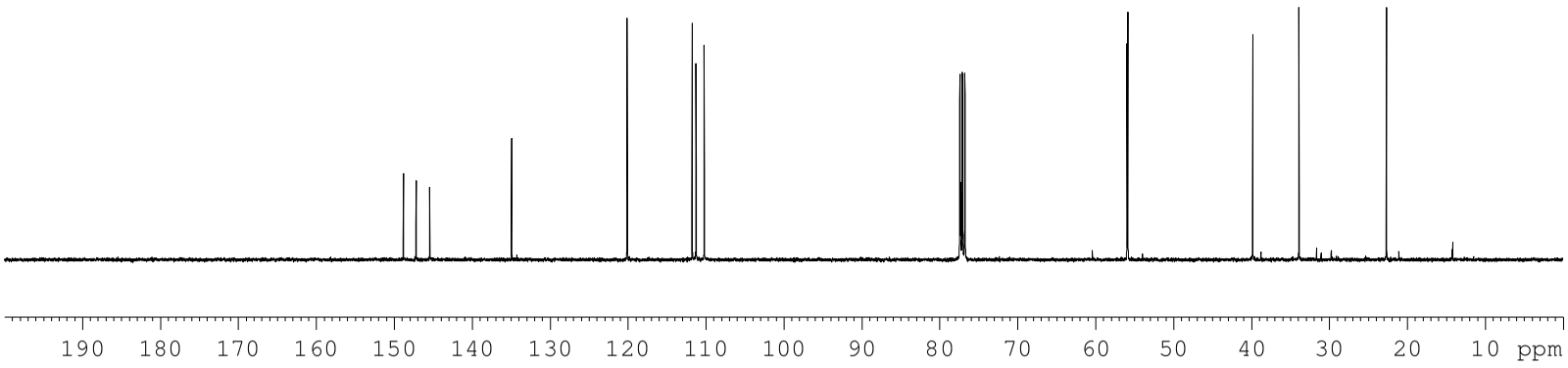


NSM-10-111FinalC13

148.792
147.166
145.421
134.902
120.079
111.738
111.231
110.190
55.920
55.813
39.785
33.852
22.617



16



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