Ammonia Synthons for the Multicomponent Assembly of Complex g-Lactams

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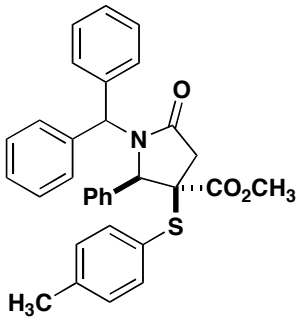
Supporting Information

Materials: Unless otherwise specified, all commercially available reagents were used as received. All reactions using dried solvents were carried out under an atmosphere of argon in flame-dried glassware with magnetic stirring. Dry solvent was dispensed from a solvent purification system that passes solvent through two columns of dry neutral alumina.

Instrumentation: ^{1}H NMR spectra and proton-decoupled ^{13}C NMR spectra were obtained on a 300, 400 or 600 MHz Varian NMR spectrometer. Chemical shifts (δ) are reported in parts per million (ppm) relative to $CHCl_3$ (s, δ 7.26) or TMS (s, δ 0). Multiplicities are given as: s (singlet), d (doublet), t (triplet), dd (doublet of doublets), m (multiplet), br m (broad multiplet), brs (broad singlet). ^{13}C NMR chemical shifts are reported relative to $CDCl_3$ (t, δ 77.0) unless otherwise noted. High resonance mass spectra were recorded on positive ESI mode in methanol or acetonitrile. Melting points were taken on an EZ-melting apparatus and were uncorrected. Infrared spectra were taken on a Bruker Tensor 27 spectrometer. Silica gel chromatographic purifications were performed by flash chromatography with silica gel (Silicycle, 40–63 μm) packed in glass columns. The eluting solvent for each purification was determined by thin layer chromatography (TLC) on glass plates coated with EMD silica gel 50 F254 and visualized by ultraviolet light. The following abbreviations are used throughout: ceric ammonium nitrate (CAN), trifluoroacetic acid (TFA), room temperature (rt), ethyl acetate (EtOAc), and dichloromethane (DCM).

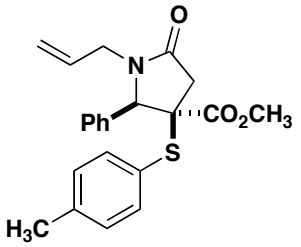
Experimental Procedures

General procedure A for the preparation of 20a-i (Table 2). A mixture of amine (1 equiv), aldehyde (1 equiv), thiol (1 equiv), and maleic anhydride (1 equiv) in toluene (0.2 M) were heated to 145 ^{o}C with a Dean Stark trap under a reflux condenser for 16 h. After cooling to rt, the reaction mixture was concentrated under reduced pressure and was dissolved in acetone (0.05 M). K_2CO_3 (4 equiv) and methyl iodide (4 equiv) were then added and the reaction was stirred for 16 h. The solvent was removed under reduced pressure and the residue was taken up in DCM and water and the layers were separated. The aqueous layer was extracted twice more with DCM. The combined organics was washed with brine, dried over Na_2SO_4, filtered, and concentrated. Purification by column chromatography afforded the desired lactams.



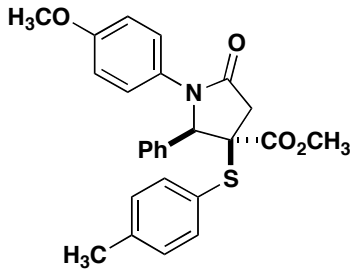
Lactam 20a. The compound was prepared according to general procedure A using maleic anhydride (0.300 g, 3.1 mmol), *p*-thiocresol (15.297 mL, 3.1 mmol, 0.2 M in toluene), diphenylmethylamine (0.527 mL, 3.1 mmol), and benzaldehyde (0.311 mL, 3.1 mmol), followed by purification using 20% EtOAc:hexanes gave the product as an oil (1.09 g, 70%): mp 144-147 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.29- 7.19 (m, 4H), 7.16 (t, J = 7.5, 2H), 7.10- 7.00 (m, 7H), 7.00-6.91 (m, 6H), 6.20 (s, 1H), 5.25 (s, 1H), 3.67 (s, 3H), 3.30 (d, J = 16.9, 1H), 3.01 (d, J = 16.9, 1H), 2.30 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 172.6, 172.4, 140.3, 139.8, 137.3, 136.3, 135.8, 130.3, 130.0, 128.6, 128.5, 128.2, 128.1, 127.8, 127.5, 127.4, 126.3, 68.4, 61.4, 60.1, 53.3, 41.3, 21.5; IR (neat) 1730, 1698 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{32}H_{29}NO_3S$ $(M + H)^+$ 508.1935, found 508.1939.

Deprotection of 20a. TFA (1.33 mL) was added to **20a** (0.200 g, 0.39 mmol) and the mixture was stirred for 48 h at reflux. The excess TFA was removed under reduced pressure and the residue was purified by column chromatography (50 to 70% EtOAc:hexanes) to afford **19** (0.08 g, 62%).



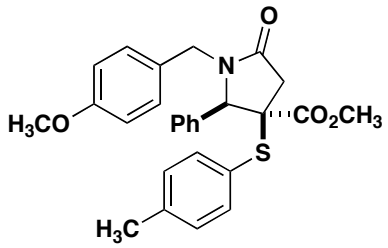
Lactam 20c. The compound was prepared according to general procedure A using maleic anhydride (0.196 g, 2 mmol), *p*-thiocresol (2 mL, 2 mmol, 1 M in toluene), allylamine (0.150 mL, 2 mmol), and benzaldehyde (0.203 mL, 2 mmol), followed by purification using 20% EtOAc:hexanes gave the product as an oil (0.398 g, 52%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.47-7.39 (m, 3H), 7.33-7.27 (m, 2H), 7.13-7.02 (m, 4H), 5.59 (dddd, J = 4.5, 7.6, 10.2, 17.7, 1H), 5.25 (s, 1H), 5.14-5.08 (m, 1H), 4.99-4.90 (m, 1H), 4.51-4.42 (m, 1H), 3.66 (s, 3H), 3.22 (d, J = 17.1, 1H), 3.08 (dd, J = 7.6, 15.5, 1H), 2.90 (d, J = 17.1, 1H), 2.31 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 172.2, 171.9, 140.3, 136.4, 134.4, 131.7, 130.0, 129.4, 128.8, 126.4, 118.6, 67.4, 58.8, 53.3, 43.6, 41.1, 21.5; IR (neat) 1733, 1702 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{22}H_{23}NO_3S$ $(M + H)^+$ 382.1466, found 382.1479.

Deprotection of 20c. To a solution of **20c** (0.050 g, 0.13 mmol) in ethanol (1 mL), rhodium trichloride trihydrate (0.004 g, 0.01 mmol) was added and the mixture was refluxed for 2 h. After the ethanol was removed by evaporation *in vacuo*, acetic acid and water (1:1, 1 mL) were added to the residue and the mixture was refluxed for 22 h. The reaction was concentrated under reduced pressure and then purified by column chromatography (50 to 70% EtOAc:hexanes) to afford **19** (0.029 g, 65%).



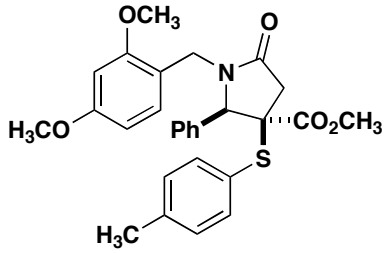
Lactam 20e. The compound was prepared according to general procedure A using maleic anhydride (0.098 g, 1 mmol), *p*-thiocresol (0.124 g, 1 mmol), *p*-methoxyphenylamine (0.123 mL, 1 mmol), and benzaldehyde (0.098 g, 1 mmol), followed by purification using 20% EtOAc:hexanes gave the product as an oil (0.29 g, 66%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.32-6.96 (m, 11H), 6.76 (d, J = 9.0, 2H), 5.74 (s, 1H), 3.72 (s, 3H), 3.67 (s, 3H), 3.30 (d, J = 17.1, 1H), 3.04 (d, J = 17.0, 1H), 2.33 (s, 3H). ^{1}H NMR corresponds to published data.[1]

Deprotection of 20e. 20e (0.244 g, 0.55 mmol) was dissolved in acetonitrile (16 mL) and the solution was cooled to 0 ^{o}C under argon. A solution of CAN (0.956 g, 1.74 mmol) in deionized water (8 mL) was added dropwise. After the addition was complete, the reaction mixture was stirred for 30 min at 0 ^{o}C. 10% $NaHSO_3$ solution and saturated $NaHCO_3$ solution were added and the mixture was stirred for 30 min at rt. Ethyl acetate was added and the mixture was filtered through Celite and concentrated. The crude product was dissolved in DCM, dried over Na_2SO_4, and reduced *in vacuo*. Purification by column chromatography (50 to 70% EtOAc:hexanes) produced **19** (0.055 g, 30%).



Lactam 20f. The compound was prepared according to procedure A using maleic anhydride (0.098 g, 1 mmol), *p*-thiocresol (0.124 g, 1 mmol), *p*-methoxybenzylamine (0.130 mL, 1 mmol), and benzaldehyde (0.101 g, 1 mmol), followed by purification using 20% EtOAc:hexanes gave the product as a pale yellow solid (0.298 g, 65%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.51-7.40 (m, 3H), 7.35-7.21 (m, 2H), 7.12-6.98 (m, 4H), 6.91 (d, J = 8.6, 2H), 6.78 (d, J = 8.7, 2H), 5.10 (d, J = 14.6, 1H), 4.91 (s, 1H), 3.78 (s, 3H), 3.54 (s, 3H), 3.45 (d, J = 14.5, 1H), 3.20 (d, J = 17.0, 1H), 2.89 (d, J = 16.0, 1H), 2.30 (s, 3H). ^{1}H NMR corresponds to published data.[1]

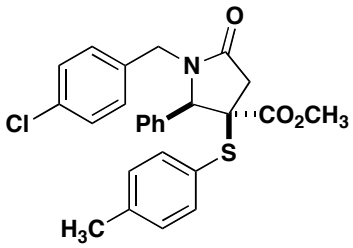
Deprotection of 20f. 20f (0.261 g, 0.57 mmol) was dissolved in DCM:H_2O (25 mL, 9:1). CAN was added and the reaction mixture was stirred at room temperature for 16 h. Water was added and the aqueous phase 10% $NaHSO_3$ solution and saturated $NaHCO_3$ solution were added and the mixture was stirred for 30 min at rt. Ethyl acetate was added and the mixture was filtered through Celite and concentrated. The crude product was dissolved in DCM, dried over Na_2SO_4, and reduced *in vacuo*. Purification by column chromatography (50 to 70% EtOAc:hexanes) produced **19** (0.055 g, 30%).



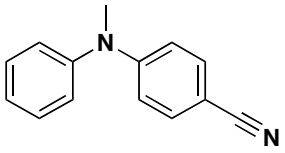
Lactam 20g. The compound was prepared according to general procedure A using maleic anhydride (0.090 g, 0.92 mmol), *p*-thiocresol (4.59 mL, 0.92 mmol, 0.2 M in toluene), 2,4-dimethoxybenzylamine (0.138 mL, 0.92 mmol), and benzaldehyde (0.093 mL, 0.92 mmol), followed by purification using 20% EtOAc:hexanes gave the product (0.27 g, 66%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.44-7.35 (m, 3H), 7.30-7.22 (m, 2H), 7.06 (d, J = 8.1, 2H), 7.01 (d, J = 8.1, 2H), 6.88-6.82 (m, 1H), 6.37-6.29 (m, 2H), 5.01 (s, 1H), 4.90 (d, J = 14.6, 1H), 3.73 (s, 3H), 3.69 (d, J = 14.5, 1H), 3.60 (s, 3H), 3.51 (s, 3H), 3.18 (d, J = 16.9, 1H), 2.88 (d, J = 16.9, 1H), 2.26 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 172.0, 171.8, 160.9, 158.8, 140.2 136.3, 134.9, 131.1, 123.0, 129.1, 128.6, 126.6, 116.3, 104.1, 98.3, 67.5, 59.0, 55.5, 55.3, 53.0, 41.1, 40.0, 21.4; IR (neat) 1734, 1696 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{28}H_{29}NO_5S$ $(M + H)^+$ 492.1833, found 492.1839.

Deprotection of 20g. 20g (0.090 g, 0.2 mmol) was dissolved in acetonitrile and water (9:1, 2 mL) at rt then CAN (0.331 g, 0.6 mmol) was added. After 16 h, water was added and the aqueous phase was extracted twice with ethyl acetate. The combined organic phases were washed with saturated $NaHCO_3$ solution and brine, dried over Na_2SO_4,

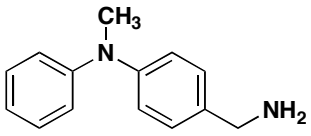
and concentrated *in vacuo*. The oil was purified by column chromatography (50 to 70% EtOAc:hexanes) to produce **19** (0.020 g, 29%).



Lactam 20h. The compound was prepared according to general procedure A using maleic anhydride (0.300 g, 3.1 mmol), *p*-thiocresol (15.297 mL, 3.1 mmol, 0.2 M in toluene), 4-chlorobenzylamine (0.374 g, 3.1 mmol), and benzaldehyde (0.311 mL, 3.1 mmol), followed by purification using 20% EtOAc:hexanes gave the product (0.883 g, 62%): mp 121-124 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.47-7.39 (m, 3H), 7.30-7.24 (m, 2H), 7.24-7.20 (m, 2H), 7.09-7.00 (m, 4H), 6.96-6.89 (m, 2H), 5.10 (d, J = 14.9, 1H), 4.92 (s, 1H), 3.55 (s, 3H), 3.50 (d, J = 14.9, 1H), 3.22 (d, J = 17.1, 1H), 2.90 (dd, J = 0.8, 17.1, 1H), 2.29 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 172.2, 171.9, 140.4, 136.4, 134.4, 134.0, 133.8, 130.0, 129.7, 129.5, 129.0, 128.9, 126.3, 67.2, 58.9, 53.2, 44.2, 40.9, 21.5; IR (neat) 1731, 1699 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{26}H_{24}ClNO_3S$ (M + H)$^{+}$ 466.1233, found 466.1239.

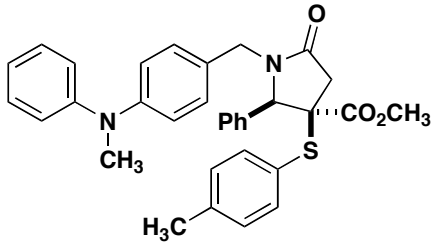


Precursor of amine 29. To a stirred mixture of 2-dicyclohexylphosphino-2'-(*N,N*-dimethylamino)biphenyl (0.179 g, 0.46 mmol) in toluene (19 mL) was added $Pd_2(dba)_3$ (0.174 g, 0.19 mmol). The mixture was stirred at room temperature for 30 min. To this solution was added 4-iodobenzonitrile (0.869 g, 3.79 mmol), N-methylaniline (0.49 mL, 4.55 mmol), and sodium tert-butoxide (0.518 g, 5.39), in that order. The orange solution was heated in the microwave for 1 h at 100 ^{o}C. The reaction was then filtered through Celite, washing with EtOAc, concentrated to a brown oil and purified by column chromatography (10% EtOAc:hexanes) to afford the desired compound (0.79 g, 99%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.46-7.38 (m, 4H), 7.30-7.23 (m, 1H), 7.20 (d, J = 7.4, 2H), 6.75-6.68 (m, 2H), 3.34 (s, 3H). ^{1}H NMR corresponds to published data.[1]



Amine 29. To a solution of the above nitrile (0.79 g, 3.79 mmol) in THF (9.5 mL) was added borane-tetrahydrofuran complex solution (12 mL, 11.38 mmol, 1 M in THF). The reaction mixture was refluxed for 16 h. After cooling to room temperature, DCM and 1 M HCl aqueous solution were added. The aqueous layer was extracted twice with DCM. The aqueous layer was basified with NaOH and then extracted with DCM. The combined organics was dried over Na_2SO_4, filtered and concentrated under reduced

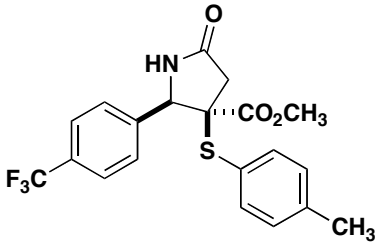
pressure. Purification by column chromatography (5% MeOH:DCM with 2% TEA) afforded the desired compound (0.28 g, 34%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.46-7.19 (m, 4H), 7.14-6.88 (m, 5H), 3.84 (s, 2H), 3.33 (s, 3H).; ^{13}C NMR (75 MHz, $CDCl_3$) δ 149.3, 148.1, 136.7, 129.4, 128.4, 121.5, 121.1, 120.0, 46.2, 40.6; IR (neat) 3405, 1596, 1500 cm^{-1}.



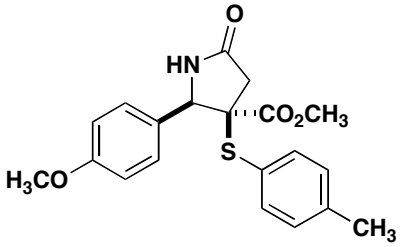
Lactam 20i. The compound was prepared according to general procedure A using maleic anhydride (0.098 g, 1 mmol), *p*-thiocresol (1 mL, 1 mmol, 1 M in toluene), amine **29** (0.212 g, 1 mmol), and benzaldehyde (0.101 mL, 1 mmol), followed by purification using 20% EtOAc:hexanes gave the product (0.357 g, 67%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.48-7.39 (m, 3H), 7.31-7.24 (m, 4H), 7.08 (d, J = 8.1, 2H), 7.02 (m, 4H), 6.98-6.94 (m, 1H), 6.87 (s, 4H), 5.09 (d, J = 14.7, 1H), 4.98 (s, 1H), 3.56 (s, 3H), 3.47 (d, J = 14.7, 1H), 3.27 (s, 3H), 3.22 (d, J = 17.0, 1H), 2.90 (d, J = 16.7, 1H), 2.28 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 172.1, 172.0, 149.0, 148.7, 140.4, 136.4, 134.3, 130.0, 129.5, 129.4, 129.3, 128.8, 127.9, 126.4, 122.1, 121.4, 119.9, 67.1, 59.0, 53.2, 44.4, 41.1, 40.5, 21.6, 21.5; IR (neat) 1738, 1697 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{33}H_{32}N_2O_3S$ $(M + H)^+$ 537.2201, found 537.2207.

Deprotection of 20i. 20i (0.038 g, 0.07 mmol) was dissolved in acetonitrile and water (9:1, 2 mL) at rt then CAN (0.194 g, 0.35 mmol) was added. After 16 h, water was added and the aqueous phase was extracted twice with ethyl acetate. The combined organic phases were washed with saturated $NaHCO_3$ solution and brine, dried over Na_2SO_4, and concentrated *in vacuo*. The resulting oil was purified by column chromatography (50%-70% EtOAc:hexanes) to produce **19** (0.007 g, 29%).

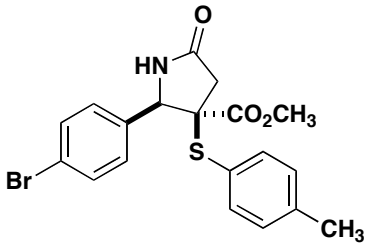
General procedure B for the preparation of 30a-f (Figure 3). In a microwave vial were combined maleic anhydride (0.098 g, 1.0 mmol), thiol (1.0 mmol) ammonium acetate (0.077 g, 1.0 mmol), and aldehyde (1.0 mmol) in toluene (1.0 M). The reaction mixture was stirred at rt for 5 min then heated in the microwave at 150 ^{o}C for 1 h. After cooling to rt, the solvent was evaporated under reduced pressure and the reaction mixture was dissolved in acetone (20 mL). Potassium carbonate (0.55 g, 4.0 mmol) and methyl iodide (0.25 mL, 4.0 mmol) were then added and the reaction was stirred for 16 h. The solvent was removed under reduced pressure and the residue was partitioned with DCM and water. The aqueous layer was extracted twice with DCM. The combined organics was washed with brine, dried over Na_2SO_4, filtered, and concentrated. Purification was accomplished using 20-80% EtOAc:hexanes, which gave the desired lactams.



Lactam 30a. The compound was prepared according to general procedure B using maleic anhydride (0.098 g, 1.0 mmol), *p*-thiocresol (1.0 mL, 1.0 mmol, 1.0 M in toluene), ammonium acetate (0.077 g, 1.0 mmol), and 4-(trifluoromethyl)benzaldehyde (0.14 mL, 1.0 mmol) which gave the product (0.099 g, 24%): mp 182-184 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.74-7.63 (m, 4H), 7.23 (s, 1H), 7.10 (d, J = 8.2, 2H), 7.07 (d, J = 8.0, 2H), 5.42 (s, 1H), 3.74 (s, 3H), 3.03 (d, J = 17.7, 1H), 2.70 (d, J = 17.7, 1H), 2.32 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 174.8, 171.9, 140.8, 139.7, 137.0, 131.7, 131.5, 131.3, 131.0, 130.1, 129.2, 126.9, 125.8, 125.4, 125.4, 125.4, 125.3, 125.1, 123.3, 121.5, 63.1, 60.0, 53.2, 40.5, 21.5; IR (neat) 1728, 1702 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{20}H_{18}F_3NO_3S$ $(M + H)^+$ 410.1027, found 410.1032.

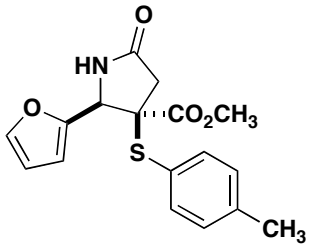


Lactam 30b. The compound was prepared according to general procedure B using maleic anhydride (0.098 g, 1.0 mmol), *p*-thiocresol (1.0 mL, 1.0 mmol, 1.0 M in toluene), ammonium acetate (0.077 g, 1.0 mmol), and 4-methoxybenzaldehyde (0.12 mL, 1.0 mmol) which gave the product (0.22 g, 59%): mp 185-187 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.43 (d, J = 8.8, 2H), 7.15 (d, J = 8.1, 2H), 7.08 (d, J = 7.9, 2H), 6.93 (d, J = 8.7, 2H), 6.76 (s, 1H), 5.28 (s, 1H), 3.82 (s, 3H), 3.71 (s, 3H), 3.03 (d, J = 17.6, 1H), 2.72 (d, J = 17.6, 1H), 2.32 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 174.8, 172.2, 160.4, 140.4, 136.9, 130.0, 129.8, 127.5, 126.5, 113.8, 63.1, 61.0, 55.5, 53.1, 40.5, 21.5; IR (neat) 1728, 1694 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{20}H_{21}NO_4S$ $(M + H)^+$ 372.1259, found 372.1265.

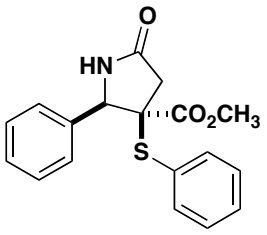


Lactam 30c. The compound was prepared according to general procedure B using maleic anhydride (0.098 g, 1.0 mmol), *p*-thiocresol (1.0 mmol, 1.0 M in toluene), ammonium acetate (0.077 g, 1.0 mmol), and 4-bromobenzaldehyde (0.185 g, 1.0 mmol) which gave the product (0.19 g, 45%): mp 190-192 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$) δ

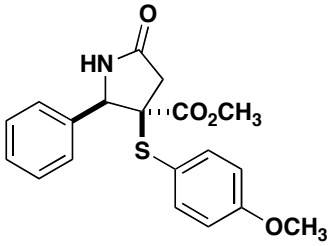
7.54 (d, J = 8.5, 2H), 7.41 (d, J = 8.4, 2H), 7.13 (d, J = 8.1, 2H), 7.09 (d, J = 8.0, 2H), 6.57-6.48 (m, 1H), 5.31 (s, 1H), 3.72 (s, 3H), 3.02 (d, J = 17.7, 1H), 2.70 (d, J = 17.7, 1H), 2.33 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 174.6, 172.0, 140.7, 137.0, 134.6, 131.6, 130.3, 130.1, 126.0, 123.5, 62.9, 60.2, 53.1, 40.4, 21.5; IR (neat) 3089, 3059, 2952, 1724, 1694 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{19}H_{18}BrNO_3S$ $(M + H)^+$ 420.0258, found 420.0263.



Lactam 30d. The compound was prepared according to general procedure B using maleic anhydride (0.098 g, 1.0 mmol), *p*-thiocresol (1.0 mmol, 1.0 M in toluene), ammonium acetate (0.077 g, 1.0 mmol), and aldehyde (1.0 mmol) which gave the product (0.022 g, 56%): mp 187-189 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.53-7.44 (m, 1H), 7.21 (d, J = 8.0, 2H), 7.08 (d, J = 8.0, 2H), 6.46 (d, J = 3.3, 1H), 6.42-6.39 (m, 1H), 5.82 (s, 1H), 5.34 (s, 1H), 3.66 (s, 3H), 3.03 (d, J = 17.1, 1H), 2.86 (d, J = 17.0, 1H), 2.33 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 174.2, 171.3, 150.2, 143.6, 140.6, 136.6, 130.0, 126.2, 110.8, 110.6, 61.2, 57.9, 53.3, 39.8, 21.5; IR (neat) 3211, 1734, 1693, 1658 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{17}H_{17}NO_4S$ $(M + H)^+$ 332.0948, found 332.0953.

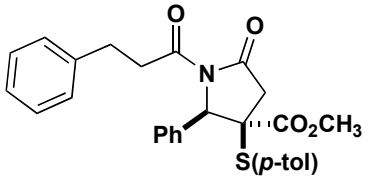


Lactam 30e. The compound was prepared in toluene according to general procedure B using maleic anhydride (0.098 g, 1.0 mmol), benzenethiol (0.102 mL, 1.0 mmol), ammonium acetate (0.077 g, 1.0 mmol), and benzaldehyde (0.1 mL, 1.0 mmol) which gave the product as an oil (0.110 g, 34%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51-7.49 (m, 2H), 7.41-7.40 (m, 3H), 7.34-7.31 (m, 1H), 7.37-7.23 (m, 4H), 7.09 (s, 1H), 5.33 (s, 1H), 3.68 (s, 3H), 3.05 (d, J = 17.5, 1H), 2.72 (d, J = 17.5,1H); ^{13}C NMR (151 MHZ, $CDCl_3$) δ 174.9, 172.1, 136.8, 135.7, 130.1,130.0, 129.3, 129.2, 128.6, 128.5, 63.9, 60.8, 53.4, 40.5; IR (neat) 2954, 2930, 1730, 1710 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{18}H_{17}NO_3S$ $(M + H)^+$ 328.1002, found 328.1005.

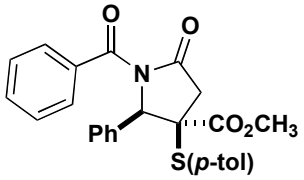


Lactam 30f. The compound was prepared in toluene according to general procedure B using maleic anhydride (0.098 g, 1.0 mmol), 4-methoxybenzenethiol (0.123 mL, 1.0 mmol), ammonium acetate (0.077 g, 1.0 mmol), and benzaldehyde (0.1 mL, 1.0 mmol) which gave the product as an oil (0.156 g, 44%): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53-7.51 (m, 2H), 7.43-7.41 (m, 3H), 7.15 (d, J = 8.9, 2H), 6.78 (d, J = 8.9, 2H), 6.15 (s, 1H), 5.34 (s, 1H), 3.78 (s, 3H), 3.71 (s, 3H), 3.03 (d, J = 17.6, 1H), 2.72 (d, J = 17.6, 1H); ^{13}C NMR (151 MHZ, $CDCl_3$) δ 174.4, 172.1, 161.5, 138.7, 135.7, 129.4, 128.6, 128.5, 120.4, 114.7, 63.3, 60.4, 55.6, 53.0, 40.3; IR (neat) 2958, 2930, 1726, 1695 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{19}H_{19}NO_4S$ $(M + H)^+$ 358.1108, found 358.1111.

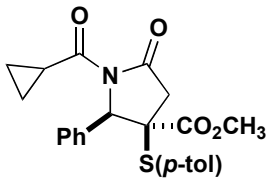
General procedure C for the preparation of 31a-h (Figure 4). To a flame dried flask was added lactam **19** (1 equiv) dissolved in 2 mL of dry THF. The solution was cooled to -78 °C and allowed to stir for 10 min. At this time, *n*-BuLi (1 equiv) was added and the solution was stirred at -78 °C for 2 h. Acylating reagent was then added and the solution was stirred at -78 °C for an additional 1 h. Next, the reaction was warmed to 0 °C, stirred for 20 min, and then warmed to room temperature and stirred from 1-16 hr, until reaction completion as determined by TLC. Finally, the reaction was quenched with 5 mL sat'd $NaHCO_3$ solution and allowed to stir for 20 min. After quenching, THF was removed *in vacuo* and the remaining mixture of water and oily solid was partitioned between 5 mL of water and 10 mL of DCM. The layers were separated and the aqueous layer was extracted with 3 x 5 mL of DCM. The combined organic layers were washed with 3 x 5 mL of brine, dried over $NaSO_4$, filtered, and concentrated to give the crude reaction material. Purification by flash chromatography (20 to 80% EtOAc:hexanes) afforded the title compounds as solids or oils.



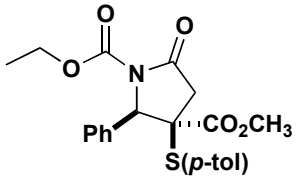
Lactam 31a. According to general experimental C, the compound was prepared using lactam **19** (0.053 g, 0.15 mmol), *n*-BuLi (0.1 mL, 0.15 mmol, 1.58 M in hexanes), and 3-phenylpropionyl chloride (0.040 g, 0.225 mmol) which produced **31a** (0.046 g, 63% yield) as a yellow solid: mp 111-115 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42–7.41 (m, 3H), 7.33-7.31 (m, 2H), 7.26-7.22 (m, 2H), 7.17-7.15 (m, 3H), 7.10-7.05 (m, 4H), 5.92 (s, 1H), 3.70 (s, 3H), 3.33 (d, J = 17.5, 1H) 3.23 (t, J = 7.8, 2H), 3.08 (d, J = 17.5, 1H), 2.90-2.85 (m, 2H), 2.31 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 172.3, 172.1, 171.7, 140.7, 140.7, 135.5, 130.2, 129.3, 128.9, 128.7, 128.7, 127.4, 126.4, 66.2, 57.7, 53.7, 43.1, 39.3, 30.2, 21.6; IR (neat) 1745, 1731, 1687 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{28}H_{27}NO_4S$ $(M + H)^+$ 474.1734, found 474.1733.



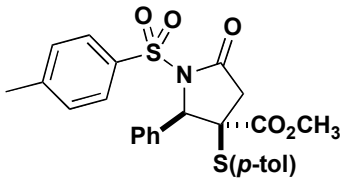
Lactam 31b. According to general experimental C, the compound was prepared using lactam **19** (0.053 g, 0.15 mmol), *n*-BuLi (0.1 mL, 0.15 mmol, 1.58 M in hexanes), and benzenecarbonyl chloride (0.032 g, 0.225 mmol) which produced **31b** (0.040 g, 57% yield) as a yellow solid: mp 127-132 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.62-7.60 (m, 2H), 7.54-7.51 (m, 1H), 7.48-7.47 (m, 2H), 7.45-7.40 (m, 5H) 7.14 (d, J = 7.9, 2H), 7.7 (d, J = 7.7, 2H), 5.98 (s, 1H), 3.77 (s, 3H), 3.32 (d, J = 17.8, 1H), 3.05 (d, J = 17.8, 1H), 2.33 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 168.2, 167.9, 166.0, 137.0, 133.0, 131.5, 130.5, 129.1, 126.6, 125.7, 125.7, 125.1, 124.6, 124.1, 122.3, 66.4, 54.4, 50.0, 39.0, 17.9; IR (neat) 1737, 1675, 1450 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{26}H_{23}NO_4S$ $(M + H)^+$ 446.1421, found 446.1423.



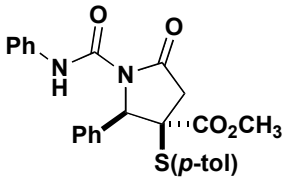
Lactam 31c. According to general experimental C, the compound was prepared using lactam **19** (0.090 g, 0.26 mmol), *n*BuLi (0.17 mL, 0.26 mmol, 1.58 M in hexanes), and cyclopropanecarbonyl chloride (0.041 g, 0.390 mmol) which produced **31c** (0.058 g, 53% yield) as a yellow oil: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43-7.39 (m, 3H), 7.36-7.34 (m, 2H), 7.11-7.06 (m, 4H) 5.92 (s, 1H), 3.72 (s, 3H), 3.38 (d, J = 17.5, 1H), 3.13 (d, J = 17.4, 1H), 2.32 (s, 3H), 1.09-1.06 (m, 1H), 0.97-0.93 (m, 2H); ^{13}C NMR (600 MHz, $CDCl_3$) δ 174.2, 172.5, 171.7, 140.6, 136.3, 135.7, 130.1, 129.2, 128.8, 127.3, 126.0, 66.6, 57.5, 53.7, 43.4, 21.5, 14.3, 11.4, 10.9; IR (neat) 2956, 1737, 1690 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{23}H_{23}NO_4S$ $(M + H)^+$ 410.1421, found 410.1421.



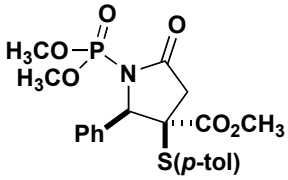
Lactam 31e. According to general experimental C, the compound was prepared using lactam **19** (0.055 g, 0.16 mmol), *n*-BuLi (0.10 mL, 0.16 mmol, 1.58 M in hexanes), and ethylchloroformate (0.025 g, 0.23 mmol) which produced **31e** (0.039 g, 58% yield) as a colorless solid: mp 132-140 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44-7.43 (m, 3H), 7.38-7.36 (m, 2H), 7.11-7.05 (m, 4H), 5.81 (s, 1H), 4.20-4.10 (m, 2H), 3.68 (s, 3H), 3.29 (d, J = 17.5, 1H), 3.03 (d, J = 17.5, 1H), 2.31 (s, 3H), 1.13 (t, J = 7.1, 3H); ^{13}C NMR (600 MHz, $CDCl_3$) δ 171.5, 170.5, 150.6, 140.7, 136.4, 135.6, 130.1, 129.3, 128.8, 127.5, 125.9, 67.2, 63.3, 57.8, 53.6, 42.2, 21.5, 14.1; IR (neat) 2977, 1790, 1729 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{22}H_{23}NO_5S$ $(M + H)^+$ 414.1370, found 414.1371.



Lactam 31f. According to general experimental C, the compound was prepared using lactam **19** (0.064 g, 0.18 mmol), *n*-BuLi (0.11 mL, 0.18 mmol, 1.58 M in hexanes), and *p*-toluenesulfonyl chloride (0.051 g, 0.27 mmol) which produced **31f** (0.023 g, 25% yield) as a yellow oil: ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.54-7.53 (m, 2H), 7.40 (s, 1H), 7.33 (s, 2H), 7.20-7.15 (m, 4H), 7.01 (s, 4H), 5.97 (s, 1H), 3.61 (s, 3H), 3.22 (d, J = 17.5, 1H), 2.91 (d, J = 17.5, 1H), 2.83 (s, 3H), 2.29 (s, 3H); ^{13}CNMR (151 MHz, $CDCl_3$) δ 171.0, 169.9, 145.4, 140.7, 134.3, 130.1, 129.5, 129.4, 128.9, 128.7, 128.4, 125.6, 68.7, 59.2 , 53.7, 41.7, 21.9, 21.5; IR (neat) 2961, 1734, 1602 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{26}H_{25}NO_5S_2$ $(M + H)^+$ 496.1242, found 496.1244.

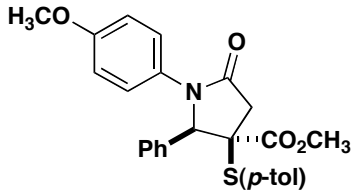


Lactam 31g. According to general experimental C, the compound was prepared using lactam **19** (0.041 g, 0.12 mmol), *n*-BuLi (0.08 mL, 0.12 mmol, 1.58 M in hexanes), and phenyl isocyanate (0.021 g, 0.18 mmol) which produced **31g** (0.016 g, 30% yield) as a brown solid: mp 167-170 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 10.44 (s, 1H), 7.45-7.42 (m, 4H), 7.39-7.38 (m, 2H), 7.27-7.24 (m, 3H), 7.11-7.06 (m, 5H), 6.03 (s, 1H), 3.74 (s, 3H), 3.43 (d, J = 17.5, 1H), 3.18 (d, J = 17.5, 1H), 2.32 (s, 3H); ^{13}C NMR (600 MHz, $CDCl_3$) δ 174.32, 171.57, 148.77, 140.71, 137.25, 136.32, 135.27, 130.20, 129.41, 129.16, 128.97, 127.32, 125.82, 124.47, 120.12, 66.61, 57.63, 53.80, 43.10, 21.50; IR (neat) 3305, 1733, 1698, 1535 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{26}H_{24}N_2O_4S$ $(M + H)^+$ 461.1524, found 461.1529.

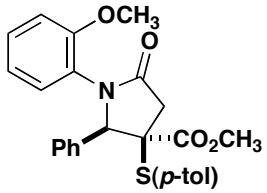


Lactam 31h. According to general experimental C, the compound was prepared using lactam **19** (0.060 g, 0.175 mmol), *n*-BuLi (0.11 mL, 0.175 mmol, 1.58 M in hexanes), and chlorodimethylphosphate (0.038 g, 0.263 mmol) which produced **31h** (0.034 g, 43% yield) as a yellow oil: ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.46-7.40 (m, 5H), 7.07-7.04 (m, 4H), 5.68 (s, 1H), 3.70 (s, 3H), 3.65 (d, J = 11.7, 3H), 3.61 (d, J = 11.7, 3H), 3.24 (d, J = 17.4, 1H), 2.97 (d, 17.4), 2.30 (s, 3H); ^{13}C NMR (600 MHz, $CDCl_3$) δ 174.5, 171.9, 140.5, 136.2, 135.9, 130.1, 129.5, 128.8, 128.1, 126.0, 68.7, 68.6, 60.3, 60.3, 54.9, 54.9, 54.4, 54.3, 53.6, 41.8, 41.8, 21.5; IR (neat) 2960, 1733 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{21}H_{24}NO_6PS$ $(M + H)^+$ 450.1129, found 450.1134.

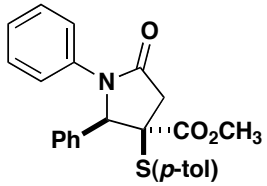
General procedure D for arylated lactams 32a-p (Figure 5). A flask with 3 Å molecular sieves and a stir bar was flame dried under vacuum and allowed to cool to rt under argon. Lactam **19** (1 equiv), boronic acid (4 equiv), and $Cu(OAc)_2$ (2 equiv) were weighed in air combined in the flask. Dry acetonitrile (0.3 M) and triethylamine (4 equiv) were added through the septa. The reaction mixture was stirred at rt for 48 h then filtered through a pad of Celite. The solvent was removed under reduced pressure and the resulting oil was purified by column chromatography (20-50% EtOAc:hexanes) to afford the desired product.



Lactam 32b. The compound was prepared according to general procedure D using lactam **19** (0.015 g, 0.04 mmol), 4-methoxyphenylboronic acid (0.027 g, 0.18 mmol), $Cu(OAc)_2$ (0.016 g, 0.09 mmol), and triethylamine (0.024 mL, 0.18 mmol) which gave the product as an oil (0.016 g, 81%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.32-6.96 (m, 11H), 6.76 (d, J = 9.0, 2H), 5.74 (s, 1H), 3.72 (s, 3H), 3.67 (s, 3H), 3.30 (d, J = 17.1, 1H), 3.04 (d, J = 17.0, 1H), 2.33 (s, 3H). ^{1}H NMR corresponds to published data.[1]

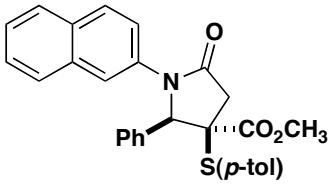


Lactam 32c. The compound was prepared according to general procedure D using lactam **19** (0.030 g, 0.09 mmol), 2-methoxyphenylboronic acid (0.053 g, 0.35 mmol), $Cu(OAc)_2$ (0.032 g, 0.18 mmol), and triethylamine (0.049 mL, 0.35 mmol) which gave the product as an oil (0.022 g, 56%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.44 (d, J = 7.3, 2H), 7.41-7.31 (m, 3H), 7.20-7.11 (m, 4H), 7.08 (d, J = 8.1, 2H), 6.90-6.80 (m, 2H), 5.75 (s, 1H), 3.77 (s, 3H), 3.72 (s, 3H), 3.32 (d, J = 17.0, 1H), 3.06 (d, J = 17.0, 1H), 2.32 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 172.2, 172.0, 154.9, 140.3, 136.5, 135.3, 130.0, 129.7, 129.2, 129.1, 129.0, 128.4, 126.5, 125.3, 120.9, 112.1, 70.2, 59.7, 55.8, 53.2, 40.9, 21.5; IR (neat) 1733, 1712 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{26}H_{25}NO_4S$ $(M + H)^+$ 448.1583, found 448.1574.

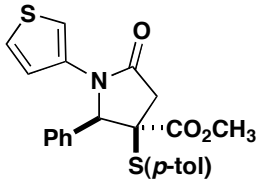


Lactam 32d. The compound was prepared according to general procedure D using lactam **19** (0.030 g, 0.09 mmol), phenylboronic acid (0.043 g, 0.35 mmol), $Cu(OAc)_2$ (0.032 g, 0.18 mmol), and triethylamine (0.049 mL, 0.35 mmol) which gave the product

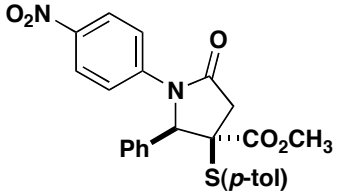
as an oil (0.018 g, 49%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.51-7.36 (m, 7H), 7.30-7.20 (m, 2H), 7.17-7.04 (m, 5H), 5.82 (s, 1H), 3.66 (s, 3H), 3.32 (d, J = 17.1, 1H), 3.06 (d, J = 17.1, 1H), 2.33 (s, 3H). ^{1}H NMR corresponds to published data.[1]



Lactam 32e. The compound was prepared according to general procedure D using lactam **19** (0.030 g, 0.09 mmol), 2-naphthaleneboronic acid (0.060 g, 0.35 mmol), $Cu(OAc)_2$ (0.032 g, 0.18 mmol), and triethylamine (0.049 mL, 0.35 mmol) which gave the product as an oil (0.018 g, 44%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.81 (d, J = 2.1, 1H), 7.73-7.66 (m, 3H), 7.62 (dd, J = 2.2, 9.0, 1H), 7.46 (d, J = 7.0, 2H), 7.43-7.31 (m, 5H), 7.15 (d, J = 8.1, 2H), 7.09 (d, J = 7.9, 2H), 5.96 (s, 1H), 3.68 (s, 3H), 3.36 (d, J = 17.1, 1H), 3.11 (d, J = 17.1, 1H), 2.33 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 172.1, 171.4, 140.5, 136.6, 135.4, 135.0, 133.5, 131.2, 130.1, 129.4, 128.9, 128.8, 128.4, 128.1, 127.7, 126.6, 126.2, 125.9, 121.2, 120.2, 70.1, 59.0, 53.5, 41.9, 21.5; IR (neat) 1728, 1697 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{29}H_{25}NO_3S$ (M + H)$^+$ 468.1622, found 468.1628.

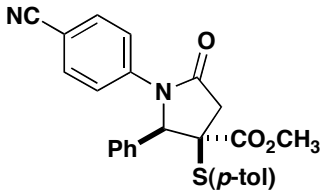


Lactam 32f. The compound was prepared according to general procedure D using lactam **19** (0.030 g, 0.09 mmol), 3-thiopheneboronic acid (0.045 g, 0.35 mmol), $Cu(OAc)_2$ (0.032 g, 0.18 mmol), and triethylamine (0.049 mL, 0.35 mmol) which gave the product as an oil (0.028 g, 74%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.50-7.31 (m, 5H), 7.23-7.15 (m, 2H), 7.15-6.99 (m, 5H), 5.76 (s, 1H), 3.64 (s, 3H), 3.31 (d, J = 17.0, 1H), 3.04 (d, J = 17.0, 1H), 2.32 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 171.6, 169.9, 140.3, 136.2, 136.0, 134.6, 129.8, 129.2, 128.8, 127.8, 125.9, 124.4, 121.1, 111.5, 69.7, 58.8, 53.3, 41.3, 29.7, 21.3; IR (neat) 1733, 1702 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{23}H_{21}NO_3S_2$ (M + H)$^+$ 424.1036, found 42.1035.

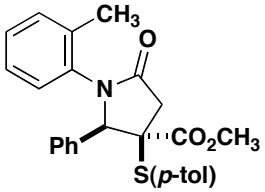


Lactam 32g. The compound was prepared according to general procedure D using lactam **19** (0.159 g, 0.47 mmol), 4-nitrophenylboronic acid (0.311 g, 1.86 mmol), $Cu(OAc)_2$ (0.17 g, 0.93 mmol), and triethylamine (0.26 mL, 1.86 mmol) which gave the product as an oil (0.042 g, 20%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.12-8.06 (m, 2H), 7.71-7.65 (m, 2H), 7.47-7.35 (m, 5H), 7.15-7.04 (m, 4H), 5.88 (s, 1H), 3.67 (s, 3H), 3.35 (d, J

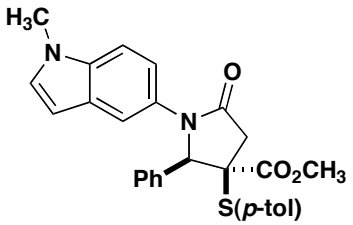
= 17.3, 1H), 3.07 (d, J = 17.3, 1H), 2.33 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 171.8, 171.6, 144.0, 143.5, 140.8, 136.6, 134.0, 130.2, 129.8, 129.3, 128.0, 125.7, 124.7, 120.8, 69.3, 58.5, 53.7, 41.8, 21.5; IR (neat) 1729, 1694 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{25}H_{22}N_2O_5S$ (M + H)$^+$ 463.1317, found 463.1319.



Lactam 32h. The compound was prepared according to general procedure D using lactam **19** (0.030 g, 0.09 mmol), 4-cyanophenylboronic acid (0.052 g, 0.35 mmol), $Cu(OAc)_2$ (0.032 g, 0.18 mmol), and triethylamine (0.049 mL, 0.35 mmol) which gave the product (0.017 g, 43%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.65-7.60 (m, 2H), 7.54-7.49 (m, 2H), 7.47-7.41 (m, 3H), 7.41-7.35 (m, 2H), 7.16-7.05 (m, 4H), 5.83 (s, 1H), 3.66 (s, 3H), 3.33 (d, J = 17.3, 1H), 3.05 (d, J = 17.3, 1H), 2.33 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 171.7, 171.7, 141.8, 140.8, 136.6, 134.1, 133.1, 130.1, 129.8, 129.2, 128.0, 125.8, 121.1, 118.8, 108.2, 69.2, 58.6, 53.6, 41.8, 21.5; IR (neat) 2226, 1727, 1688 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{26}H_{22}N_2O_3S$ (M + H)$^+$ 443.1418, found 443.1423.

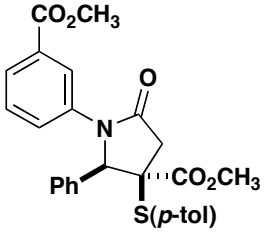


Lactam 32j. The compound was prepared according to general procedure D using lactam **19** (0.040 g, 0.12 mmol), 2-methylphenylboronic acid (0.071 g, 0.47 mmol), $Cu(OAc)_2$ (0.043 g, 0.23 mmol), and triethylamine (0.065 mL, 0.47 mmol) which gave the product as an oil (0.014 g, 28%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.51-7.33 (m, 5H), 7.20-7.05 (m, 6H), 6.99 (t, J = 7.2, 1H), 6.82 (d, J = 7.9, 1H), 5.52 (s, 1H), 3.69 (s, 3H), 3.39 (d, J = 17.2, 1H), 3.16 (d, J = 17.2, 1H), 2.33 (s, 3H), 2.15 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 172.1, 171.1, 140.2, 136.2, 135.8, 135.6, 135.2, 131.2, 129.8, 129.2, 128.9, 128.4, 128.1, 127.1, 126.5, 126.1, 71.2, 59.7, 53.1, 40.9, 21.3, 18.0; IR (neat) 2364, 2343, 1733, 1705 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{26}H_{25}NO_3S$ (M + H)$^+$ 432.1622, found 432.1626.

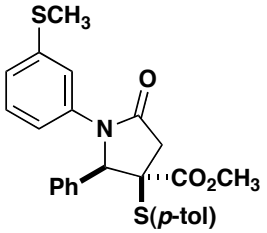


Lactam 32k. The compound was prepared according to general procedure D using lactam **19** (0.030 g, 0.09 mmol), 1-methyl-5-indolylboronic acid (0.062 g, 0.35 mmol), $Cu(OAc)_2$ (0.032 g, 0.18 mmol), and triethylamine (0.049 mL, 0.35 mmol) which gave

the product as an oil (0.032 g, 77%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.53-7.47 (m, 1H), 7.47-7.41 (m, 2H), 7.41-7.31 (m, 3H), 7.19-7.11 (m, 4H), 7.08 (d, J = 8.3, 2H), 6.98 (d, J = 3.1, 1H), 6.36 (d, J = 3.1, 1H), 5.83 (s, 1H), 3.69 (s, 3H), 3.68 (s, 3H), 3.34 (d, J = 16.9, 1H), 3.11 (d, J = 17.0, 1H), 2.32 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 172.2, 171.2, 140.2, 136.3, 135.3, 135.0, 129.9, 129.8, 129.7, 129.1, 128.6, 128.5, 128.4, 126.4, 118.2, 116.4, 109.5, 101.4, 77.4, 77.2, 77.0, 71.3, 59.2, 53.3, 41.6, 33.0, 21.4; IR (neat) 1724, 1693 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{28}H_{26}N_2O_3S$ $(M + H)^+$ 471.1732, found 471.1736.



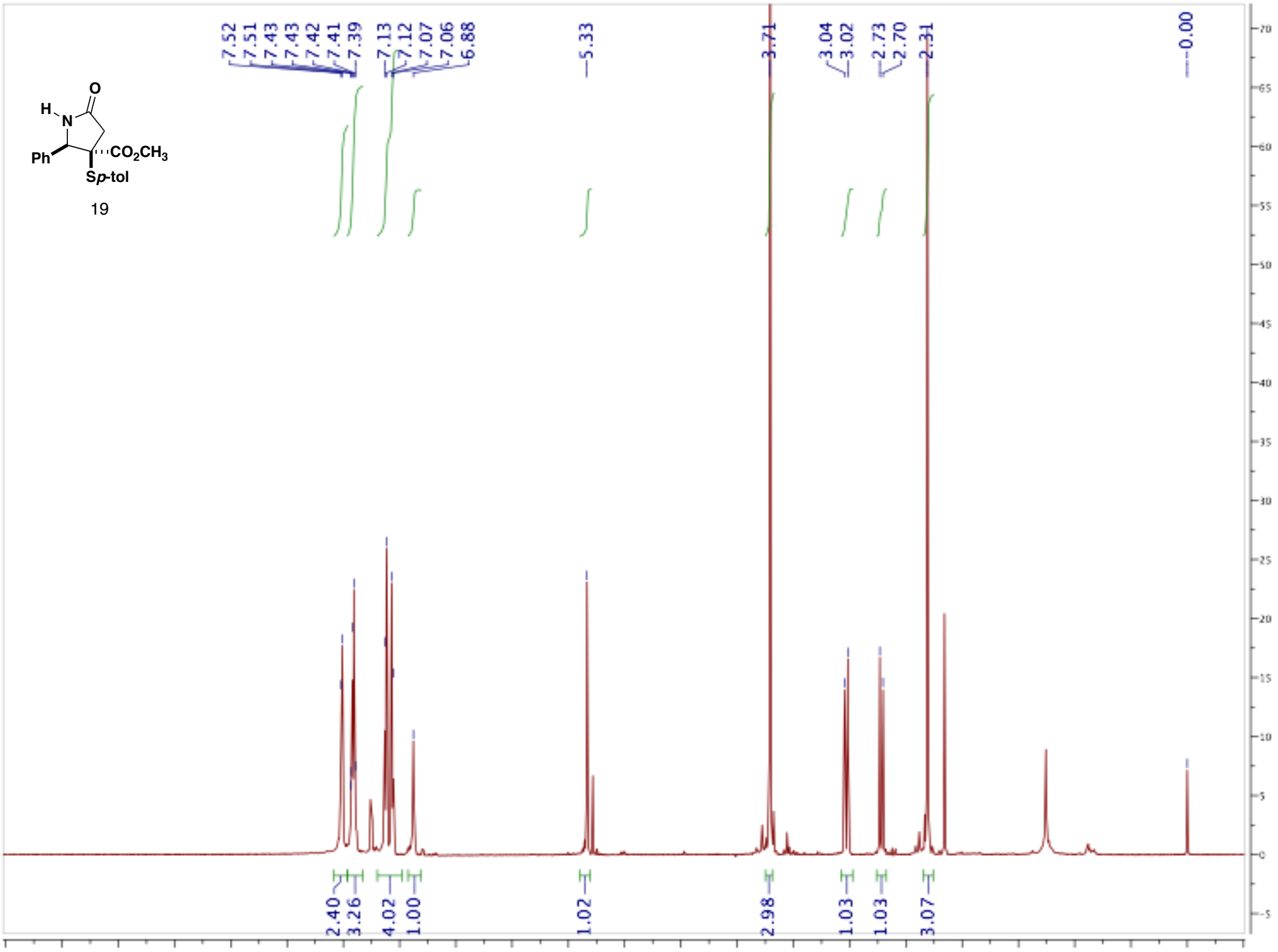
Lactam 32o. The compound was prepared according to general procedure D using lactam **19** (0.102 g, 0.3 mmol), 3-methoxycarbonylphenylboronic acid (0.215 g, 1.2 mmol), $Cu(OAc)_2$ (0.109 g, 1.2 mmol), and triethylamine (0.167 mL, 1.2 mmol) which gave the product as an oil (0.057 g, 40%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.11-8.03 (m, 1H), 7.79-7.72 (m, 1H), 7.72-7.66 (m, 1H), 7.44-7.37 (m, 5H), 7.31 (t, J = 8.0, 1H), 7.18-7.11 (m, 2H), 7.08 (d, J = 8.4, 2H), 5.88 (s, 1H), 3.85 (s, 3H), 3.68 (s, 3H), 3.33 (d, J = 17.2, 1H), 3.08 (d, J = 17.2, 1H), 2.32 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 171.9, 171.3, 166.6, 140.6, 138.0, 136.6, 134.6, 131.0, 130.1, 129.5, 129.1, 129.0, 128.4, 126.6, 126.4, 126.1, 123.0, 69.6, 58.8, 53.5, 52.5, 41.8, 21.5; IR (neat) 1720, 1605, 1587 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{27}H_{25}NO_5S$ $(M + H)^+$ 476.1520, found 476.1526.



Lactam 32p. The compound was prepared according to general procedure D using lactam **19** (0.030 g, 0.09 mmol), 3-(methylthio)phenylboronic acid (0.059 g, 0.35 mmol), $Cu(OAc)_2$ (0.032 g, 0.18 mmol), and triethylamine (0.049 mL, 0.35 mmol) which gave the product as an oil (0.013 g, 32%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.44-7.36 (m, 6H), 7.16-7.07 (m, 6H), 7.00-6.92 (m, 1H), 5. 78 (s, 1H), 3.66 (s, 3H), 3.31 (d, J = 17.1, 1H), 3.05 (d, J = 17.1, 1H), 2.34 (s, 3H), 2.33 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 171.9, 171.3, 140.5, 139.6, 138.5, 136.5, 135.0, 130.1, 129.4, 129.2, 129.0, 128.2, 126.1, 123.5, 119.6, 118.5, 69.8, 58.9, 53.5, 41.8, 21.5, 15.8; IR (neat) 1730, 1707 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{26}H_{25}NO_3S_2$ $(M + H)^+$ 464.1343, found 464.1350.

Reference

[1] Wei JQ & Shaw JT (2007) Diastereoselective synthesis of gamma-lactams by a one-pot, four-component reaction. *Org. Lett.* 9(20):4077-4080.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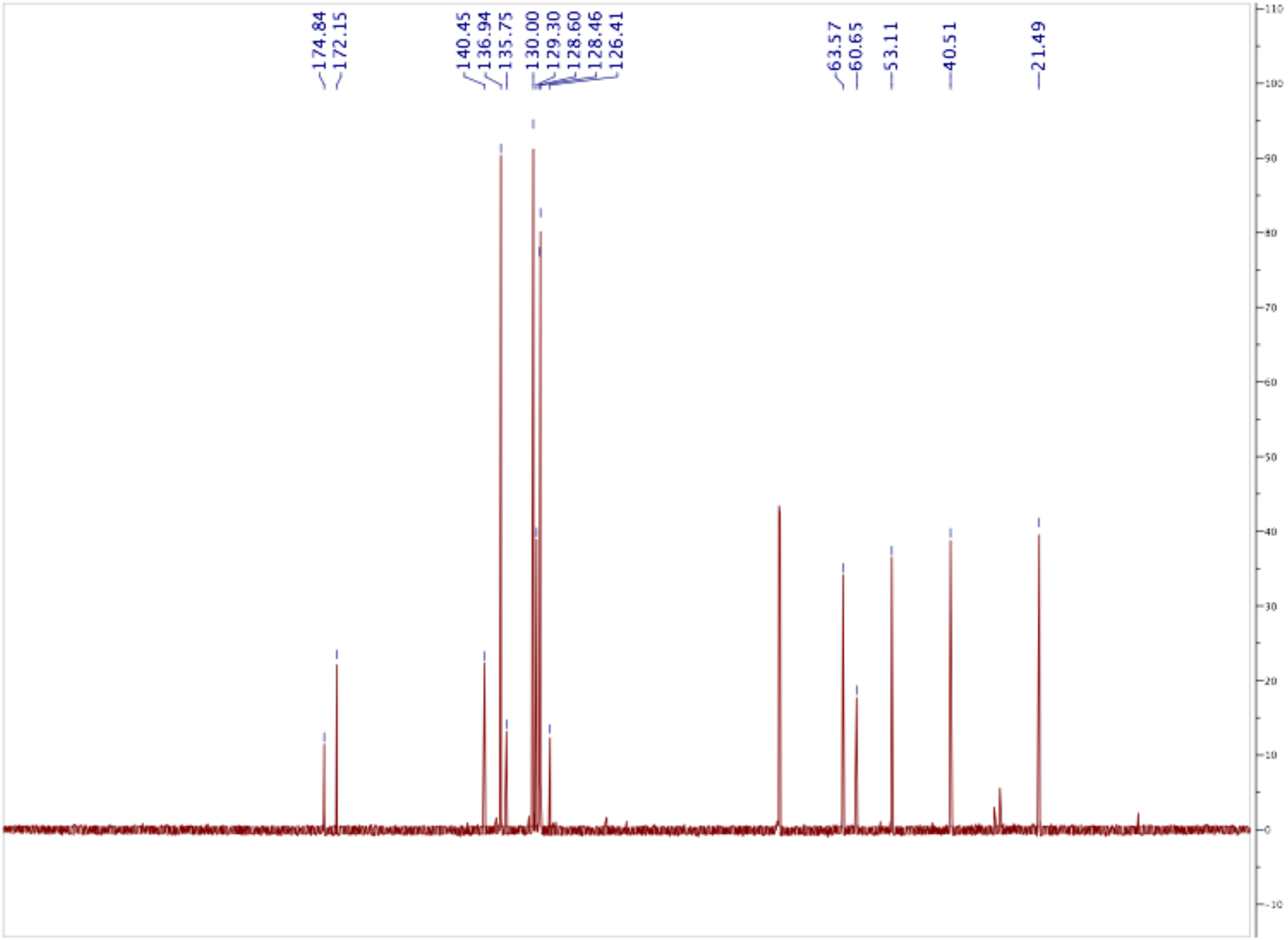
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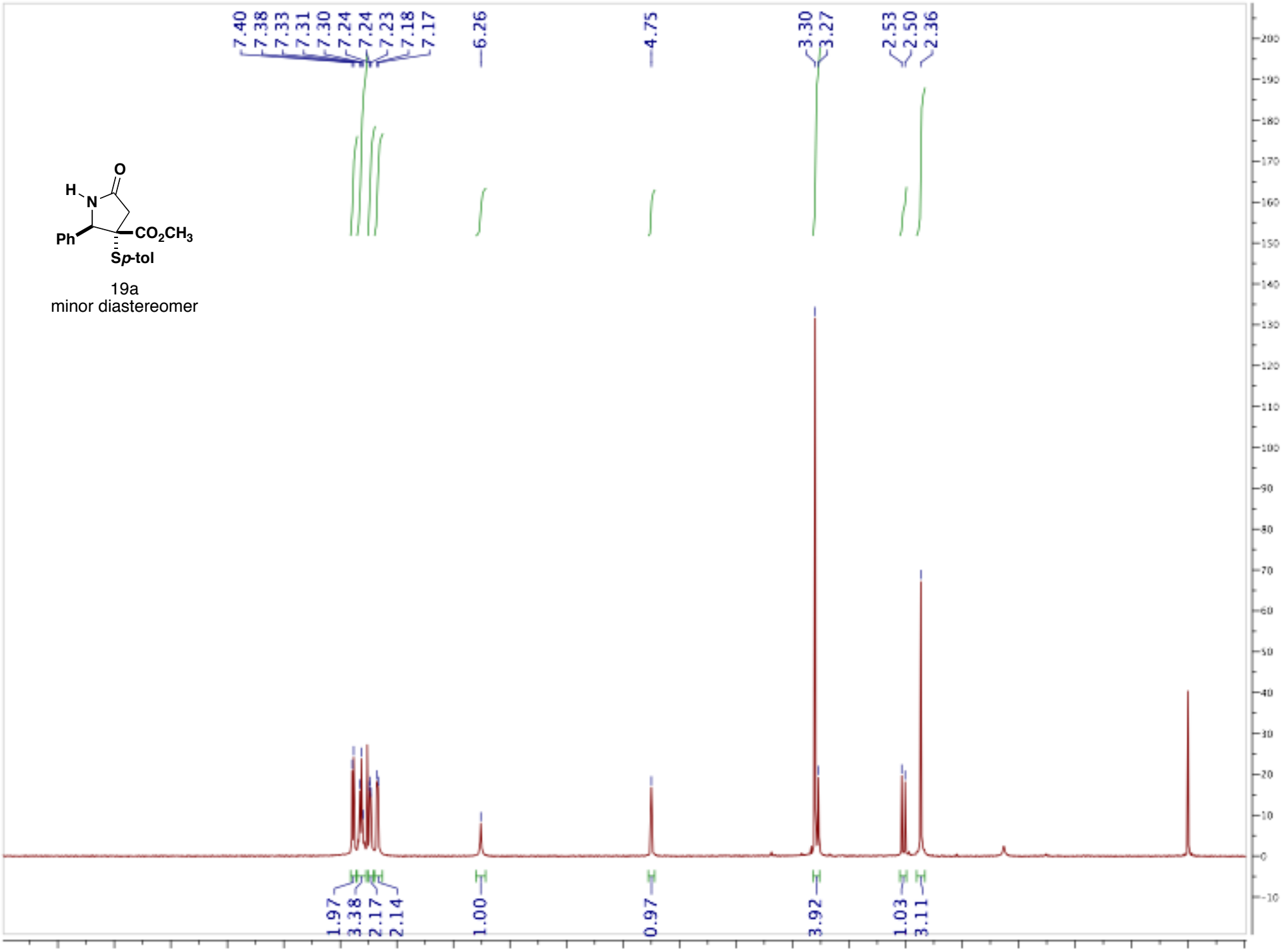
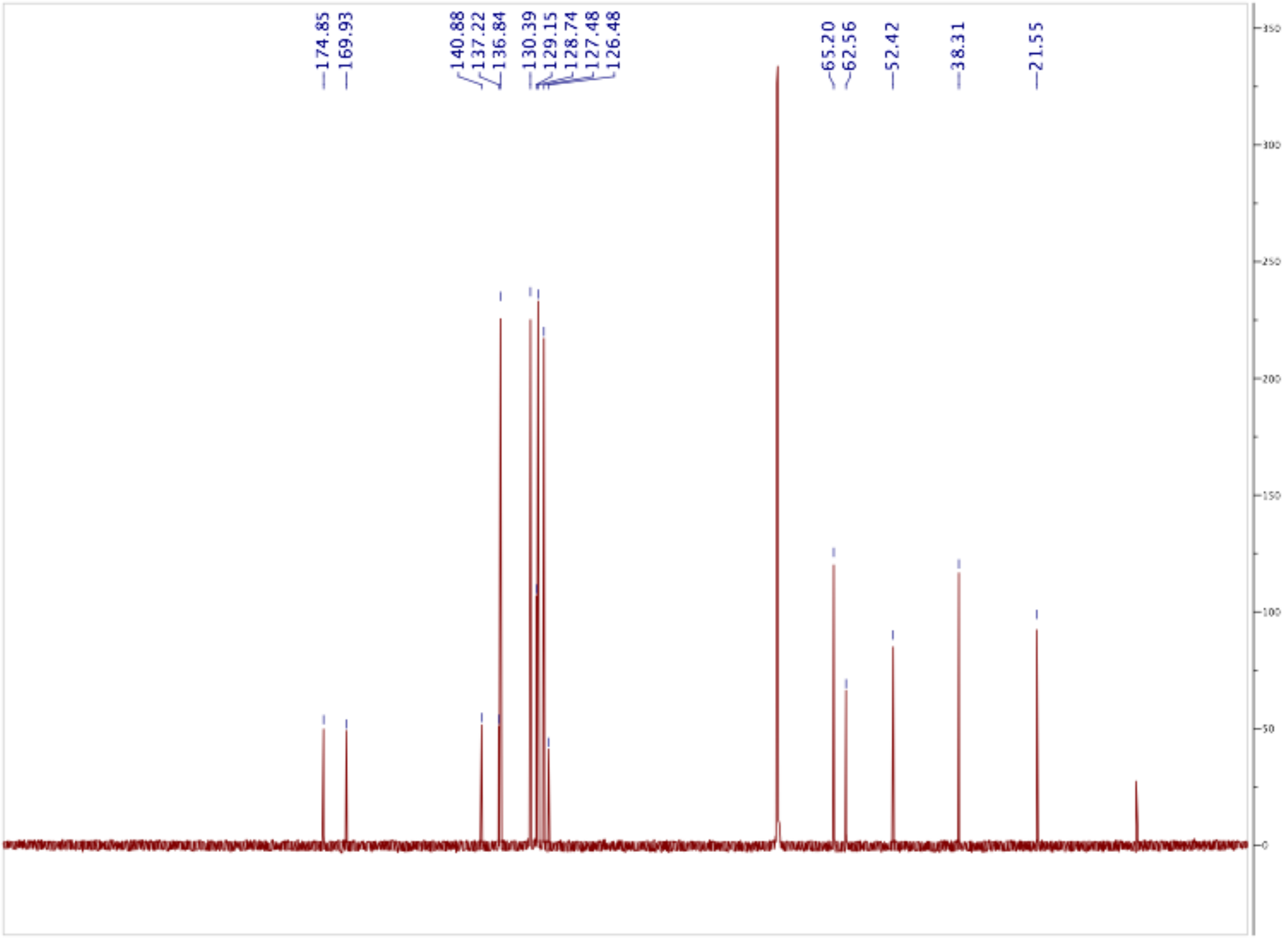


19a
minor diastereomer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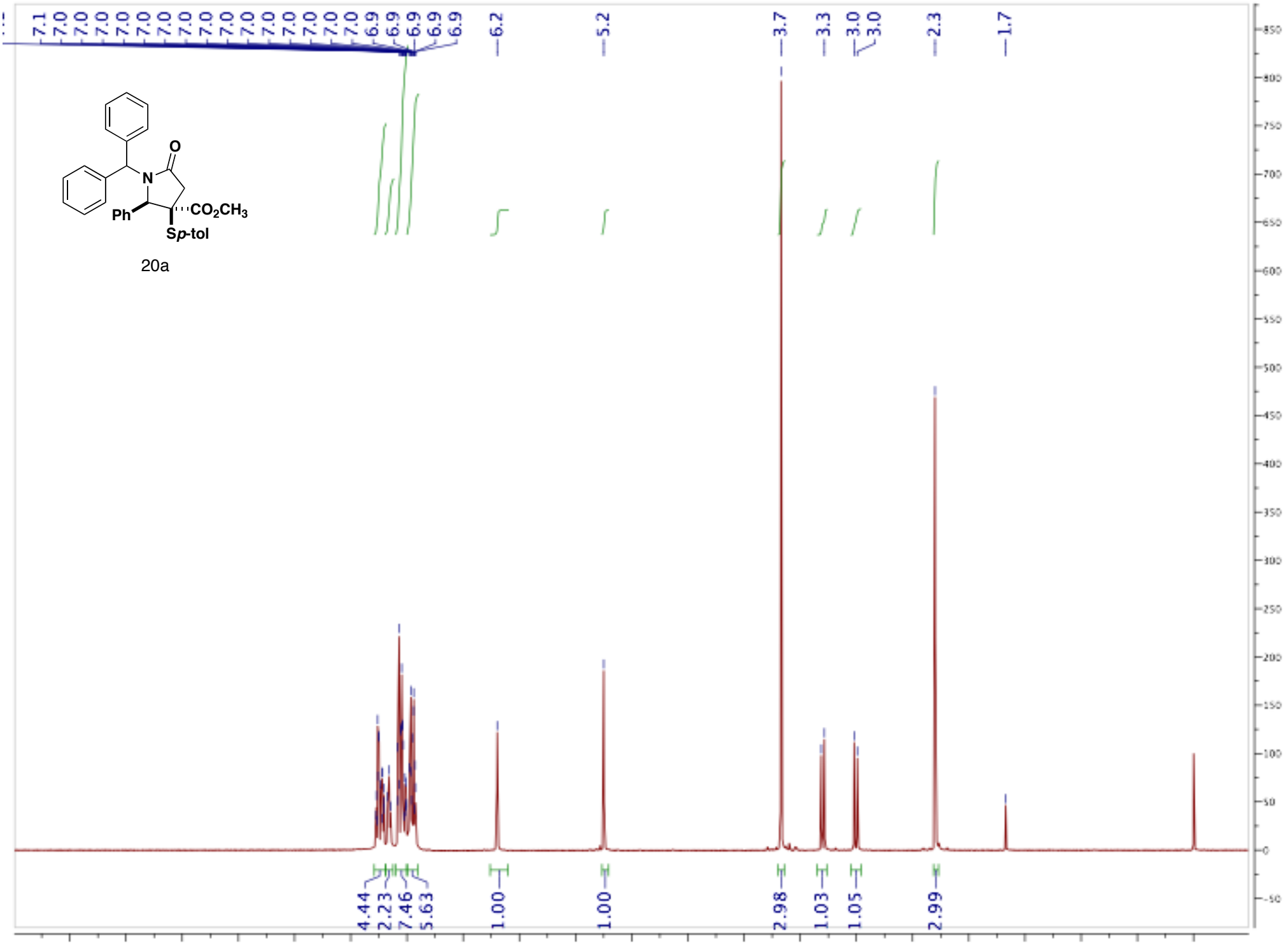
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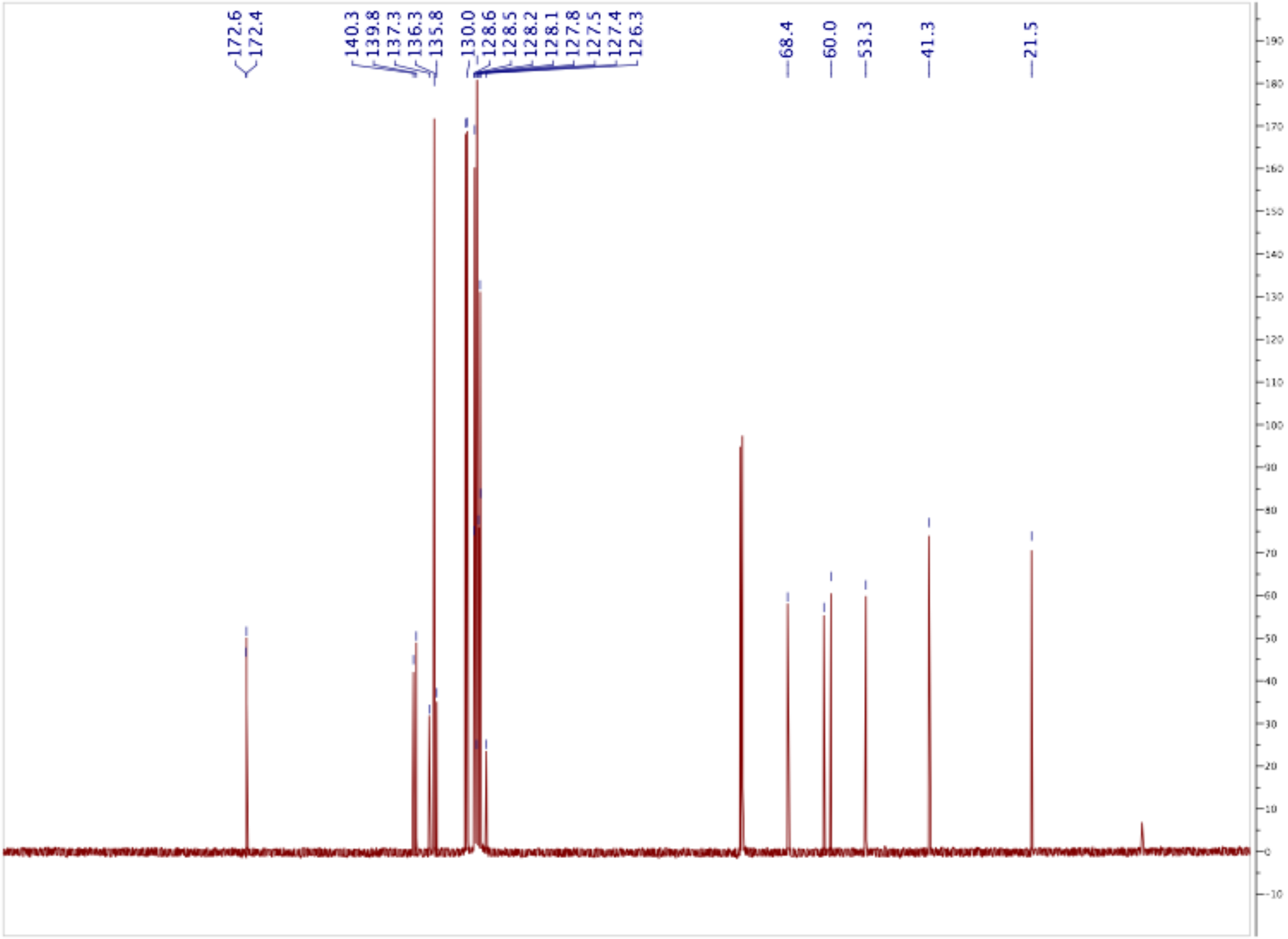
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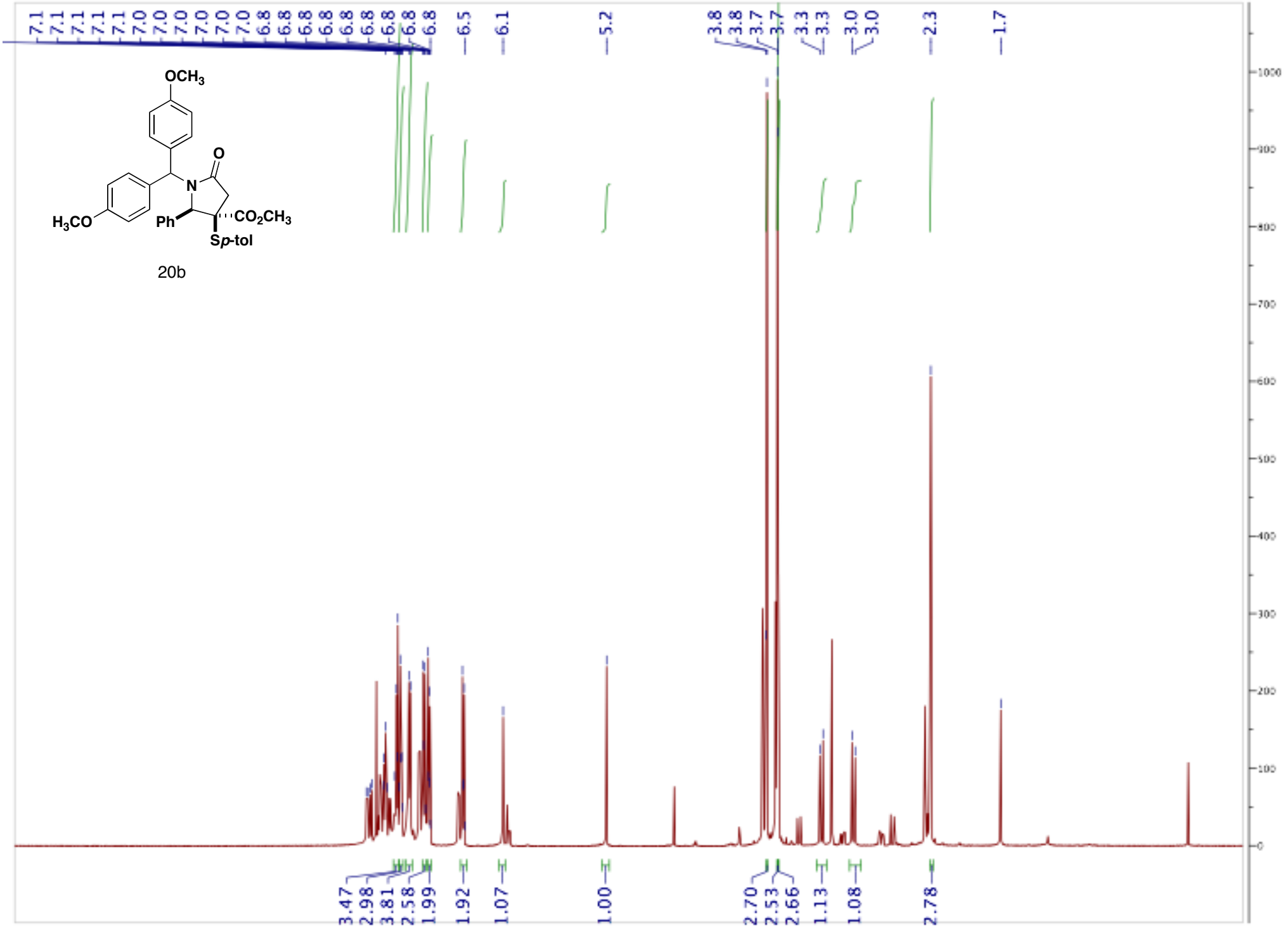
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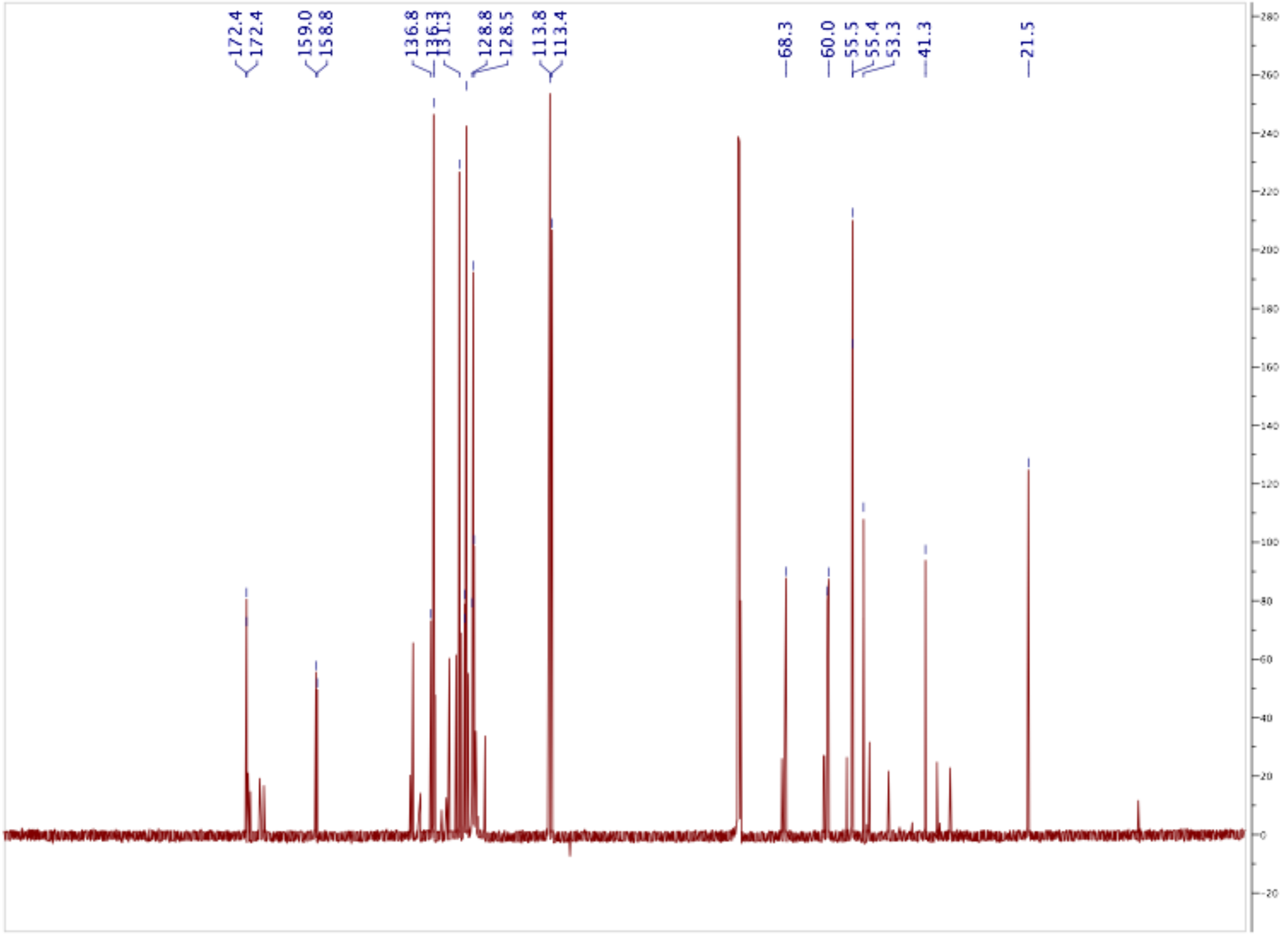
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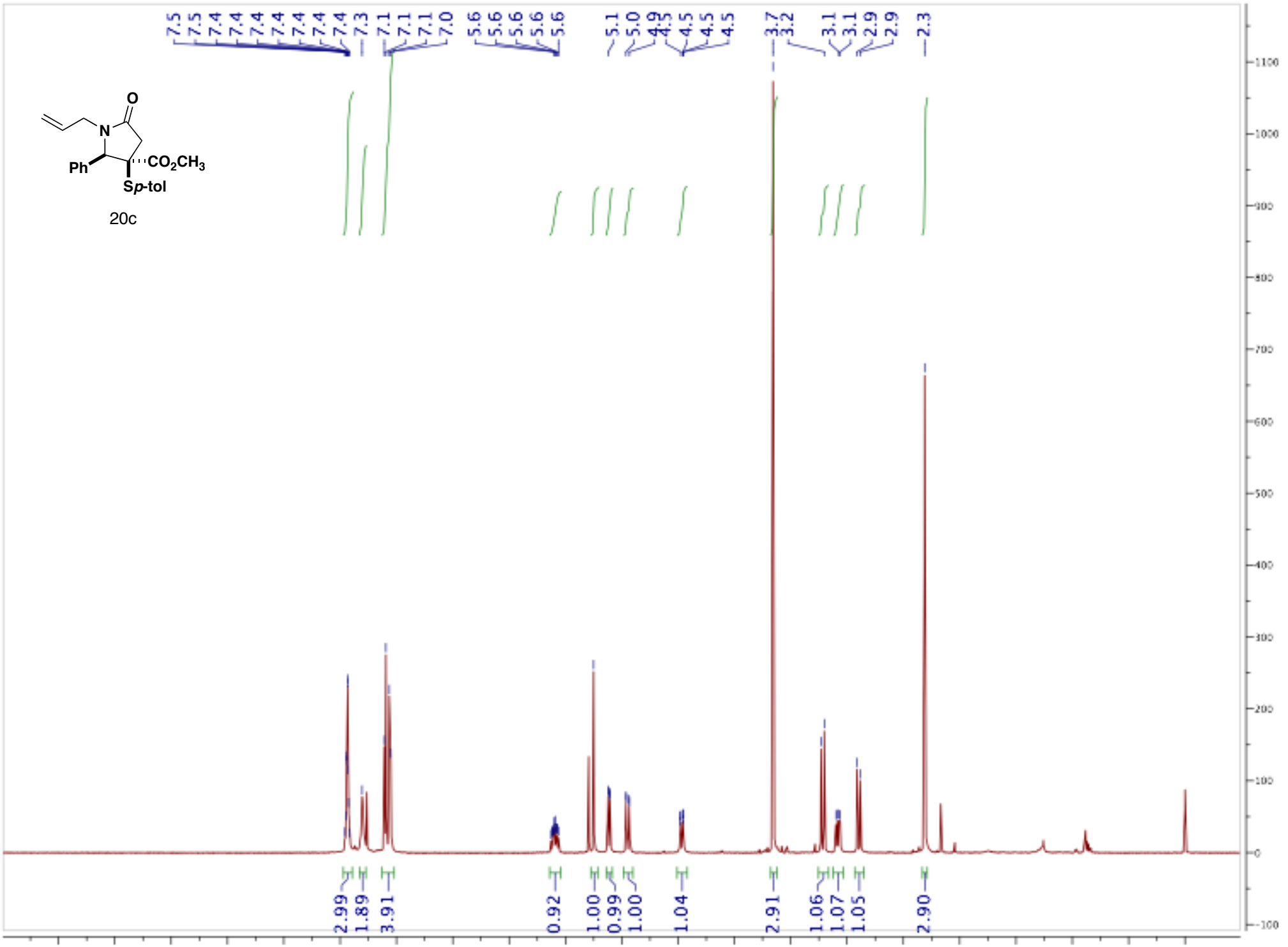
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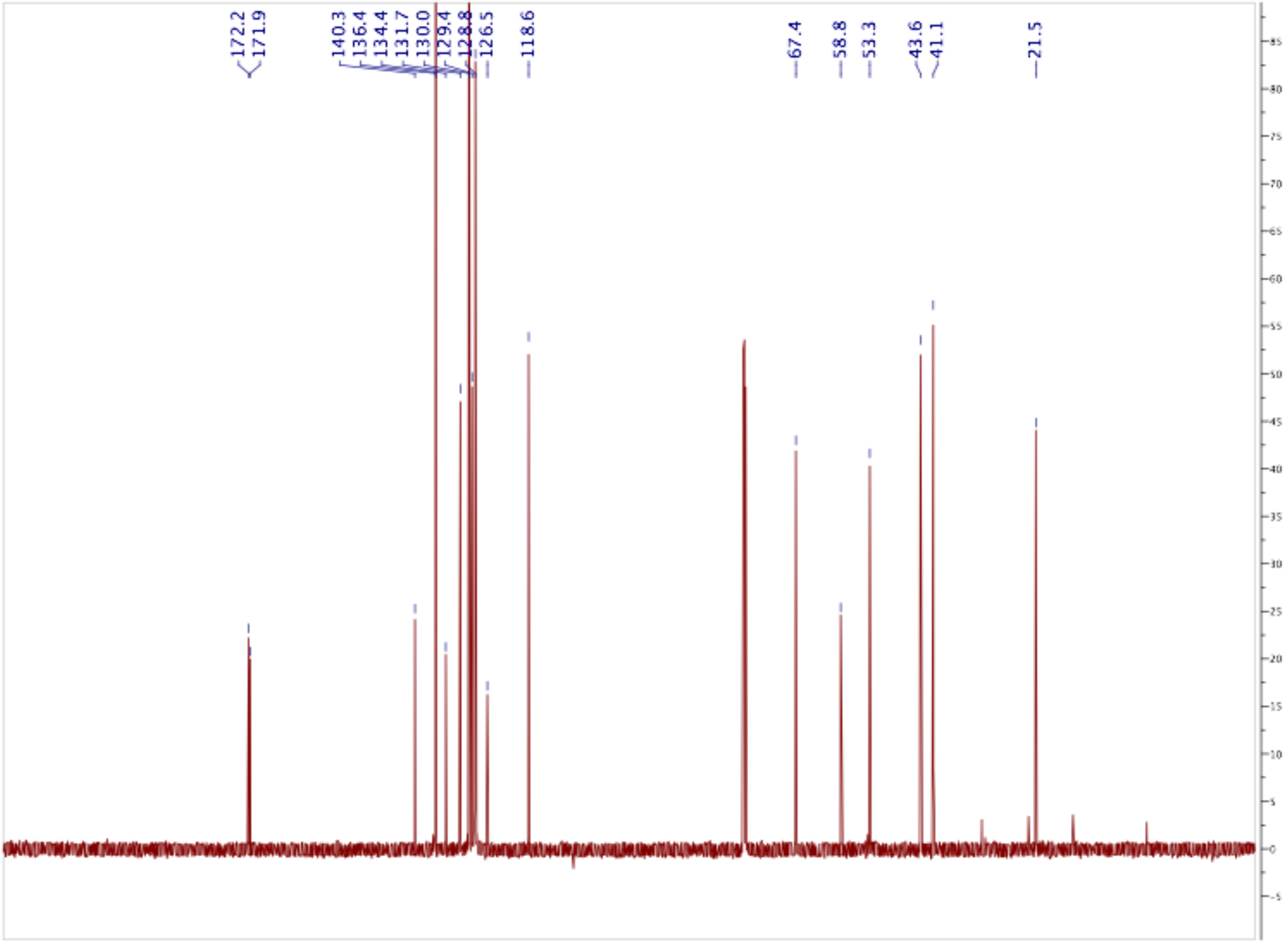
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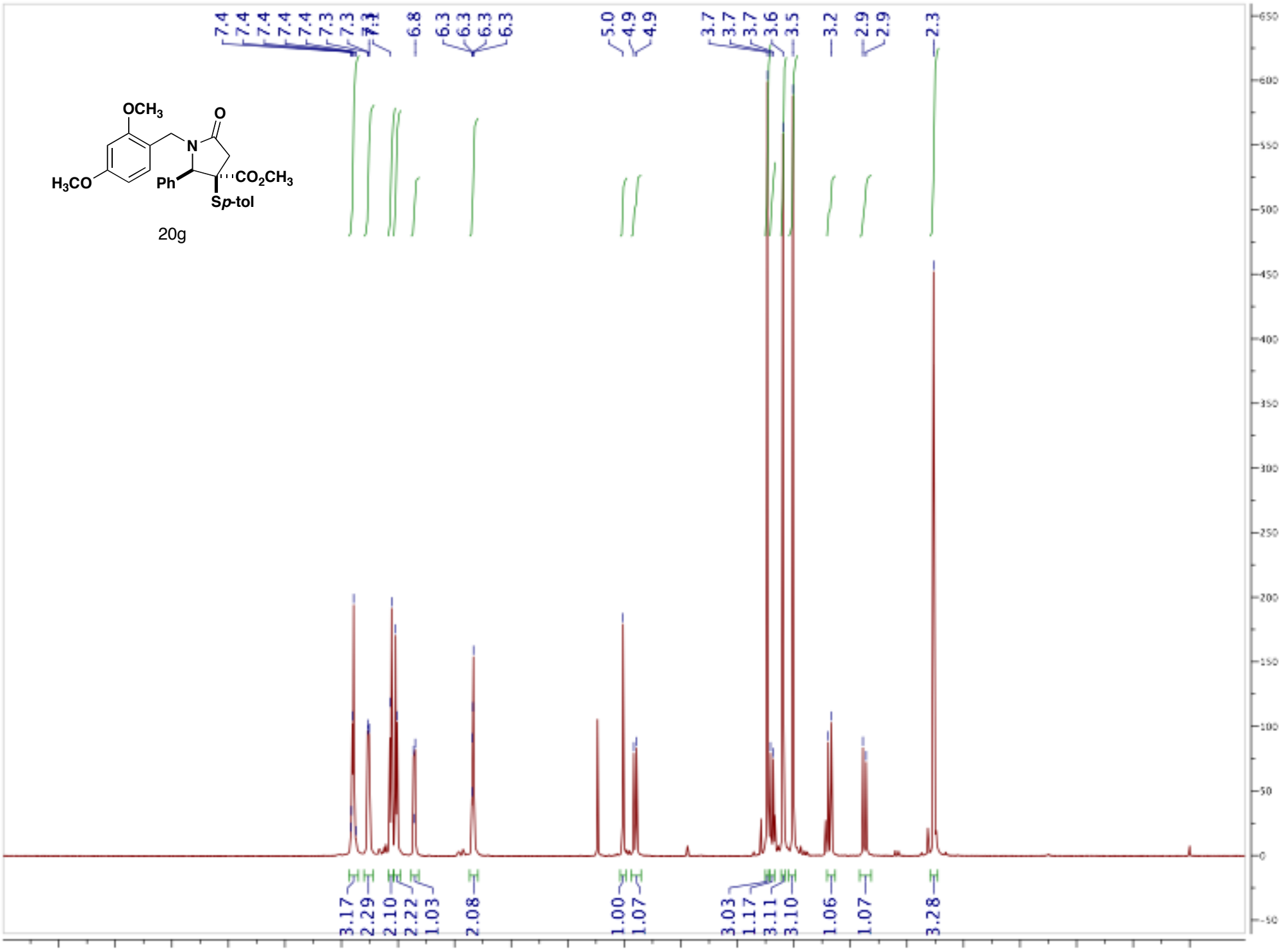
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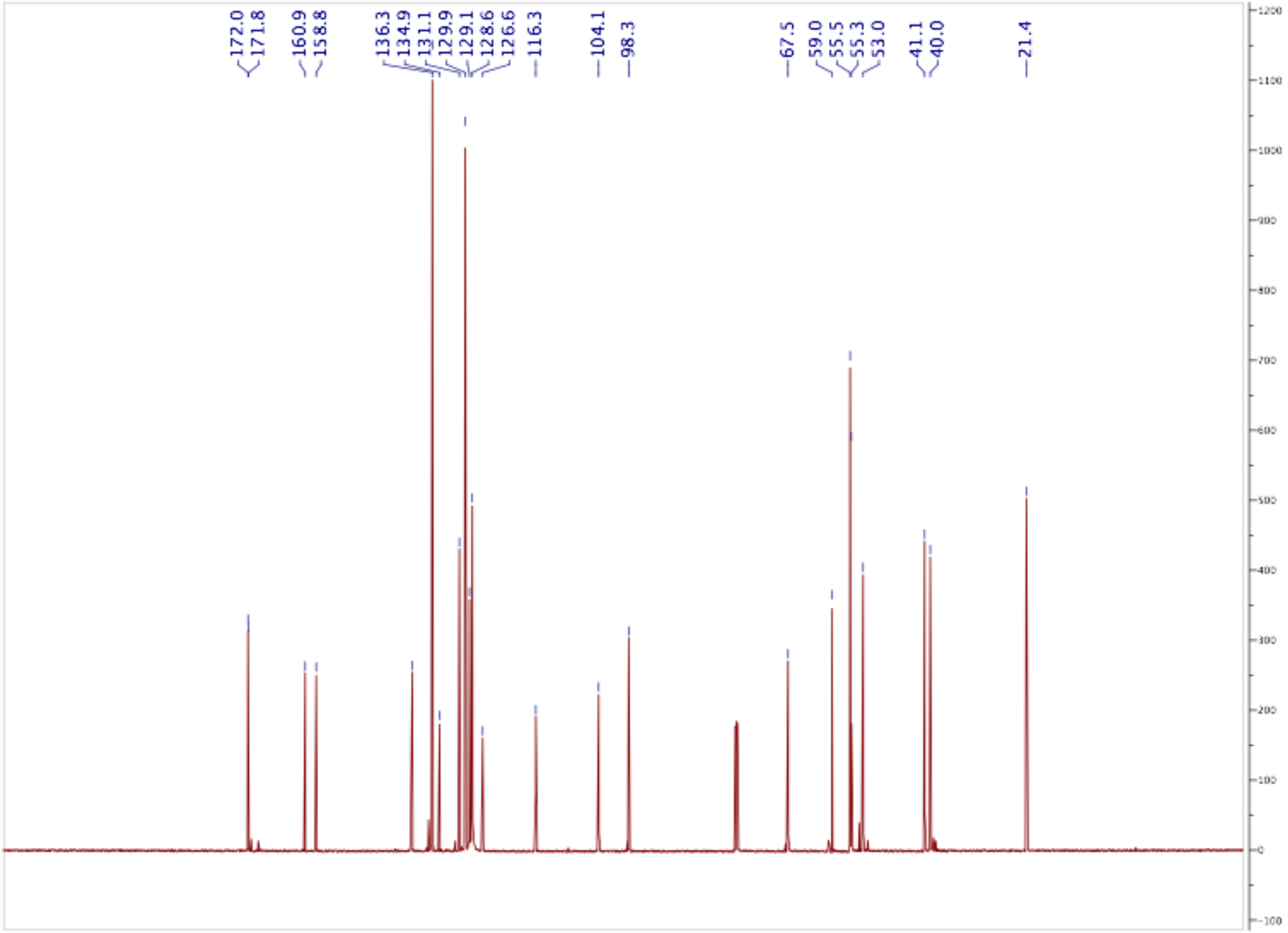
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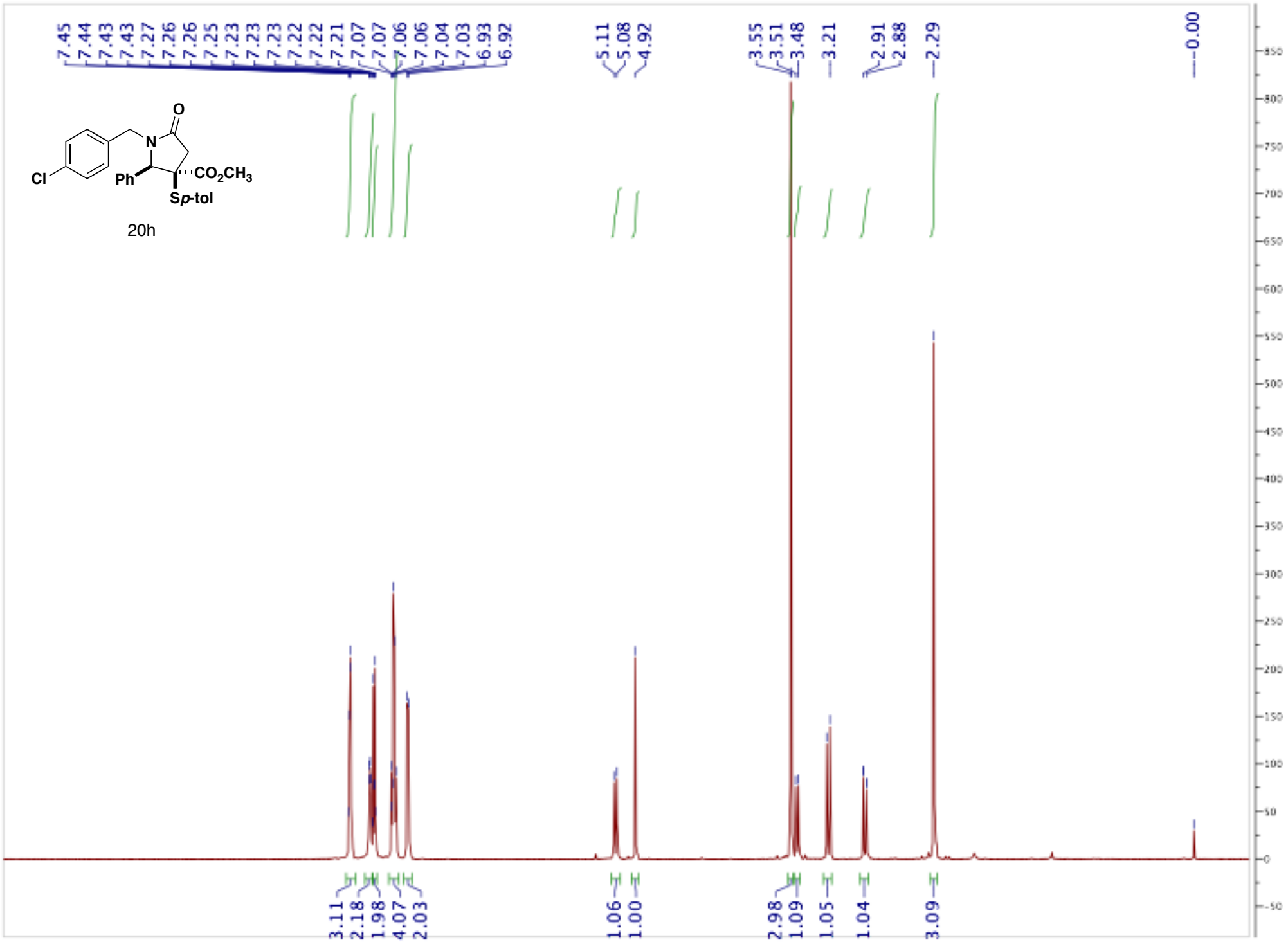
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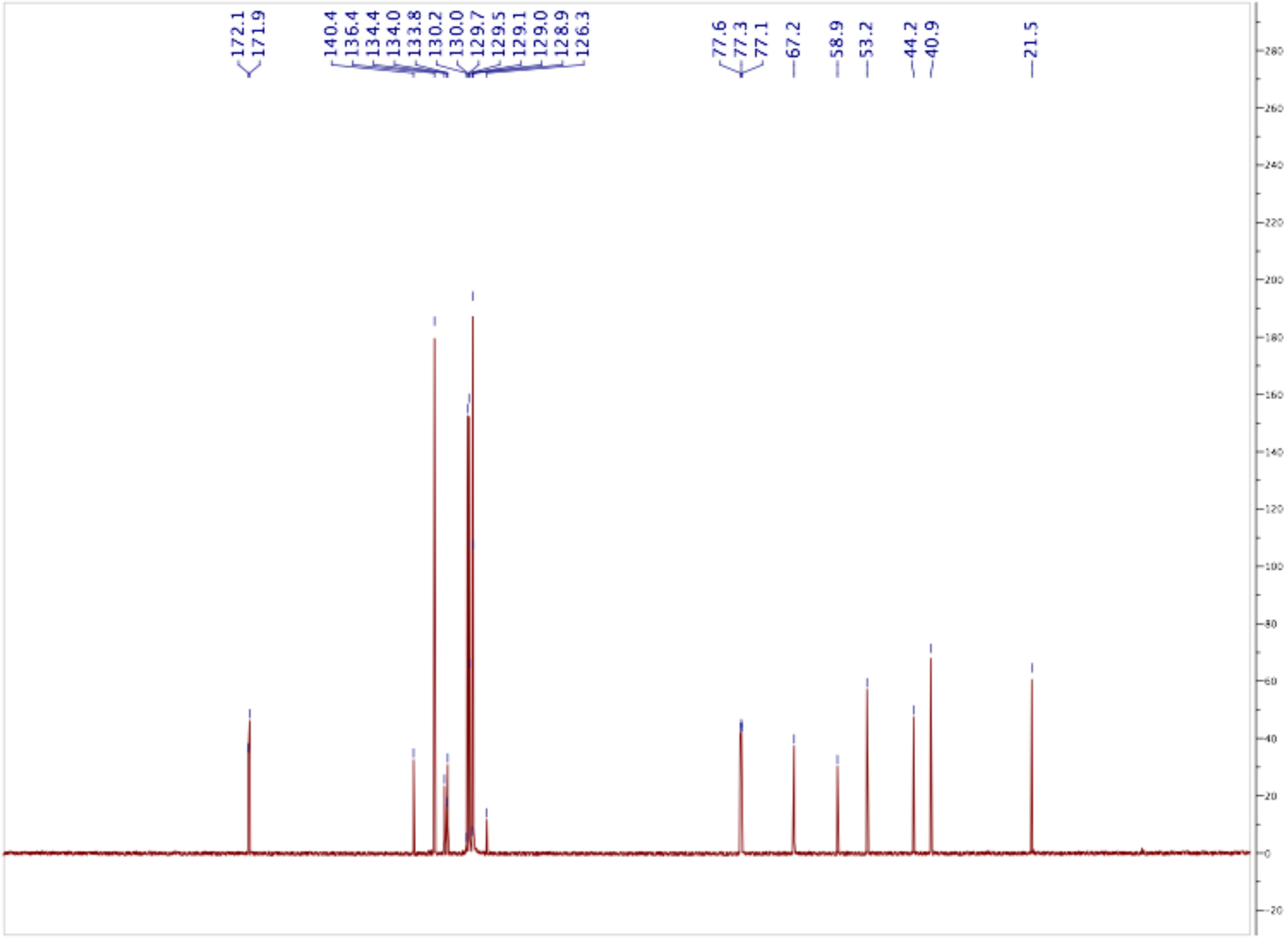
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129.5
129.1
129.0
128.9
126.3
77.6
77.3
77.1
67.2
58.9
53.2
44.2
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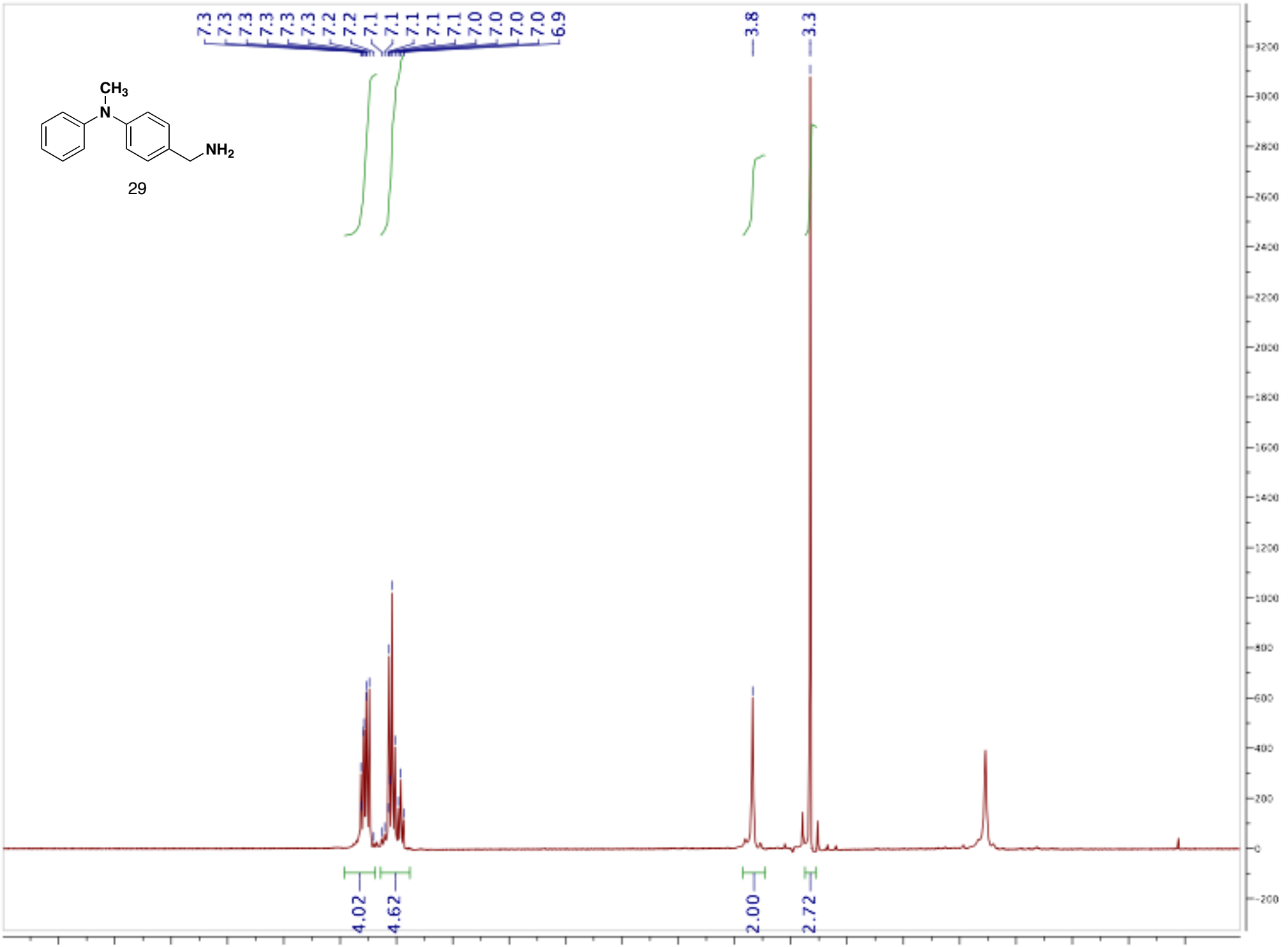
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7.3
7.3
7.3
7.3
7.2
7.2
7.1
7.1
7.1
7.1
7.0
7.0
7.0
7.0
6.9
3.8
3.3
CH3
N
NH2
29
4.02
4.62
2.00
2.72
3200
3000
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2600
2400
2200
2000
1800
1600
1400
1200
1000
800
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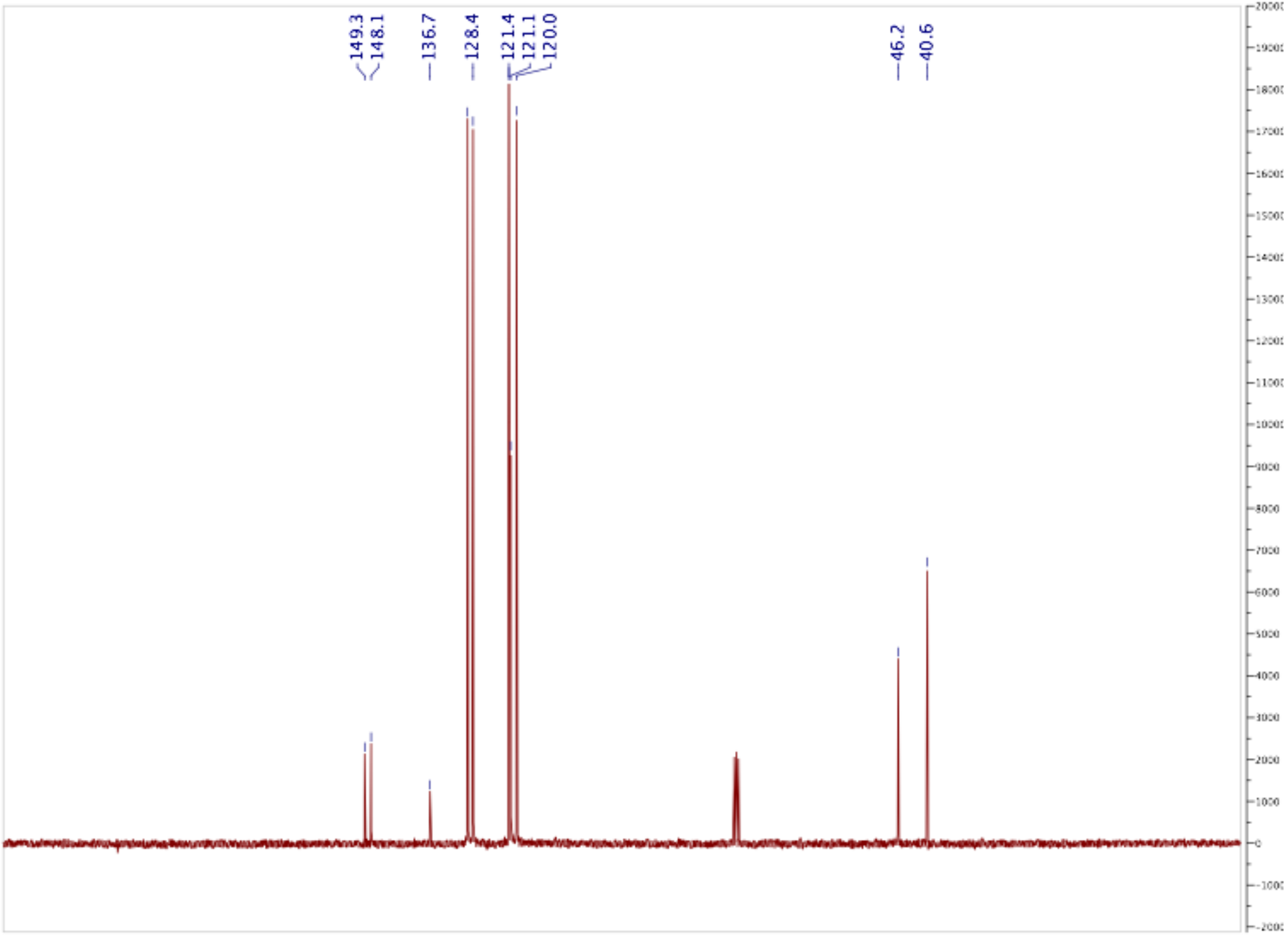
200
0
-200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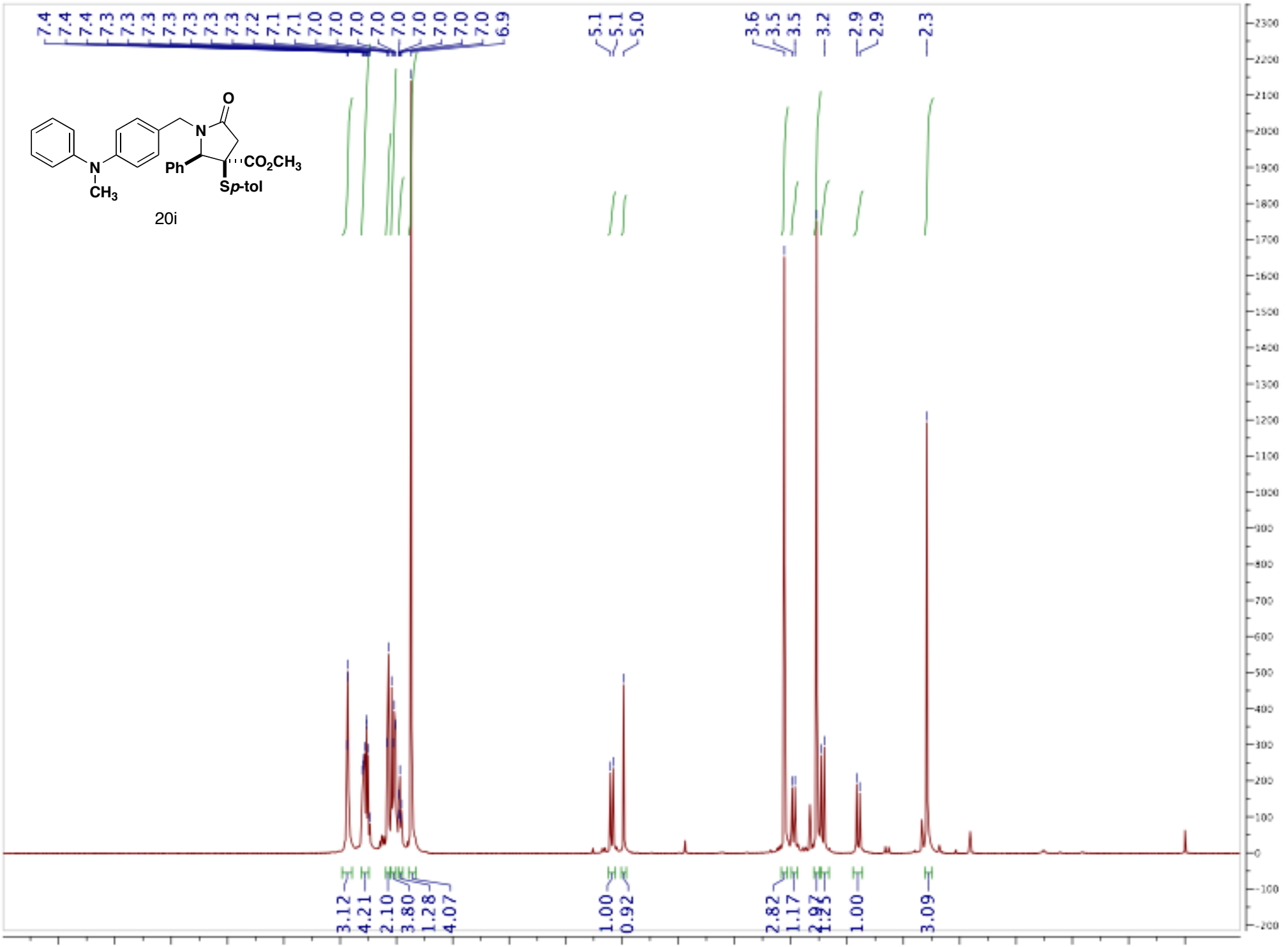
46.2
40.6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5.1 5.1 5.0
3.6 3.5 3.5 3.2
2.9 2.9
2.3
O
N
Ph
CO2CH3
Sp-tol
N
CH3
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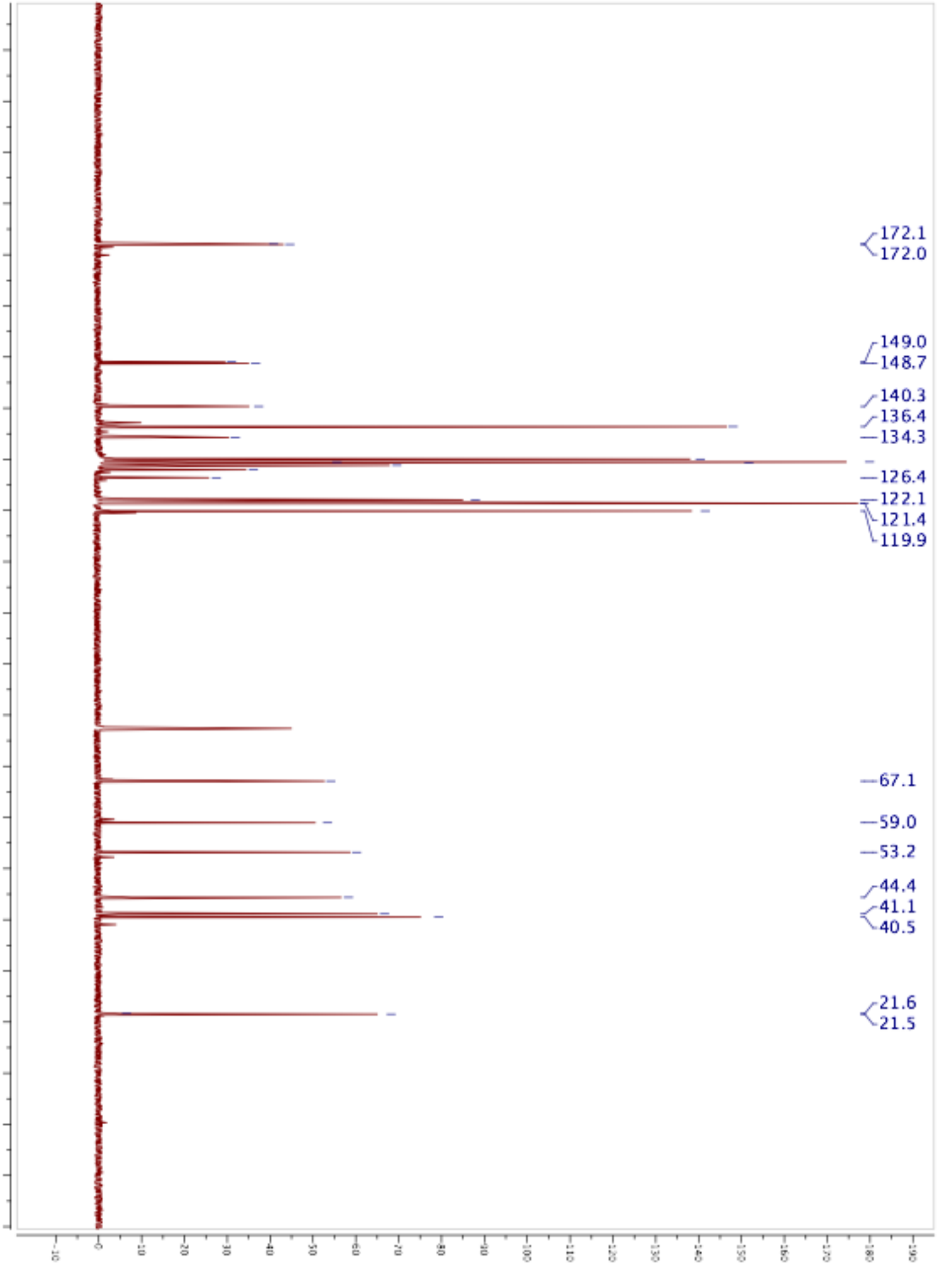
1.00 0.92
2.82 1.17 2.97 1.25 1.00 3.09

20i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134.3
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121.4
119.9
67.1
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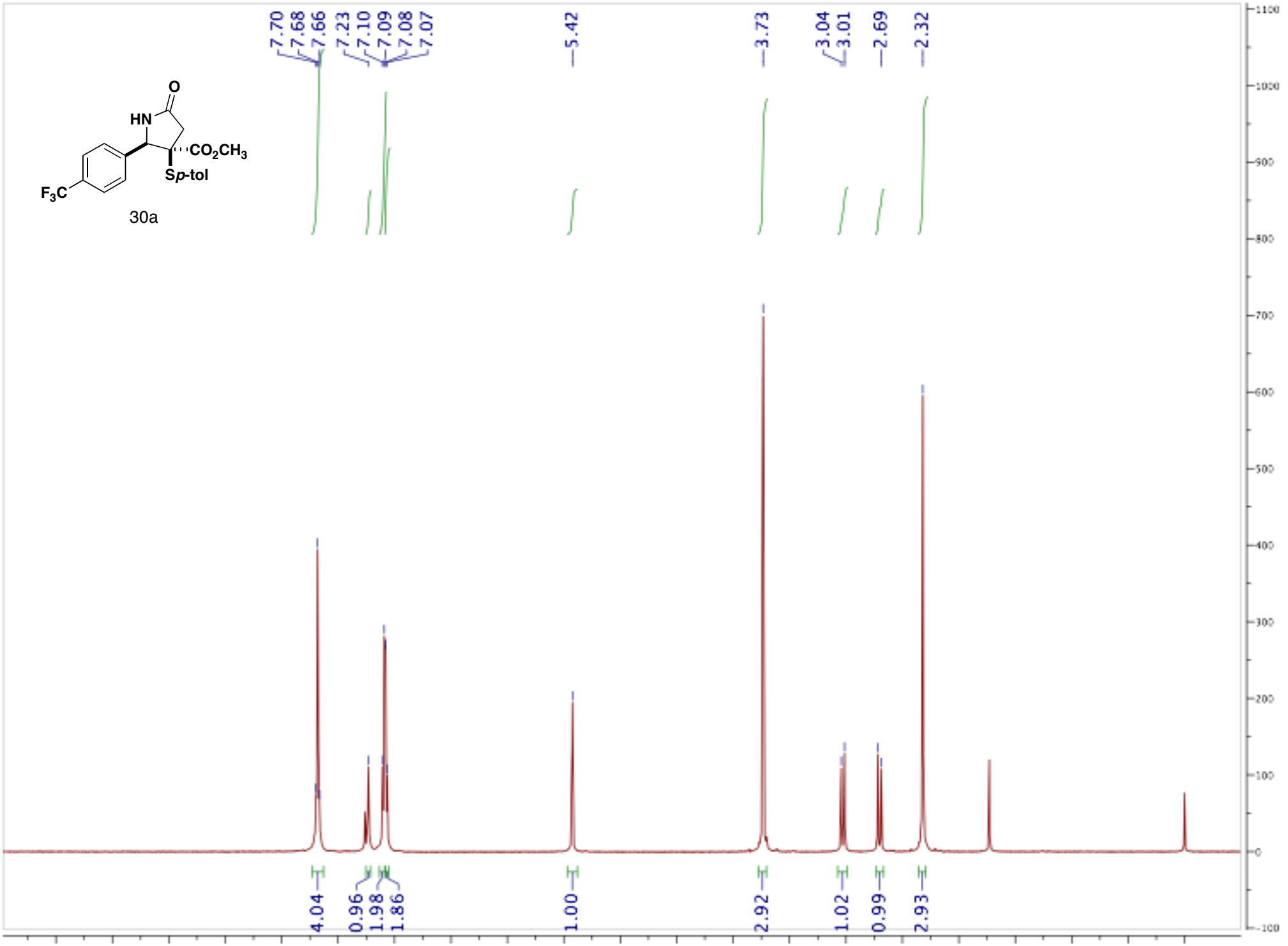
40.5
21.6
21.5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3.04
3.01
2.69
2.32
4.04
0.96
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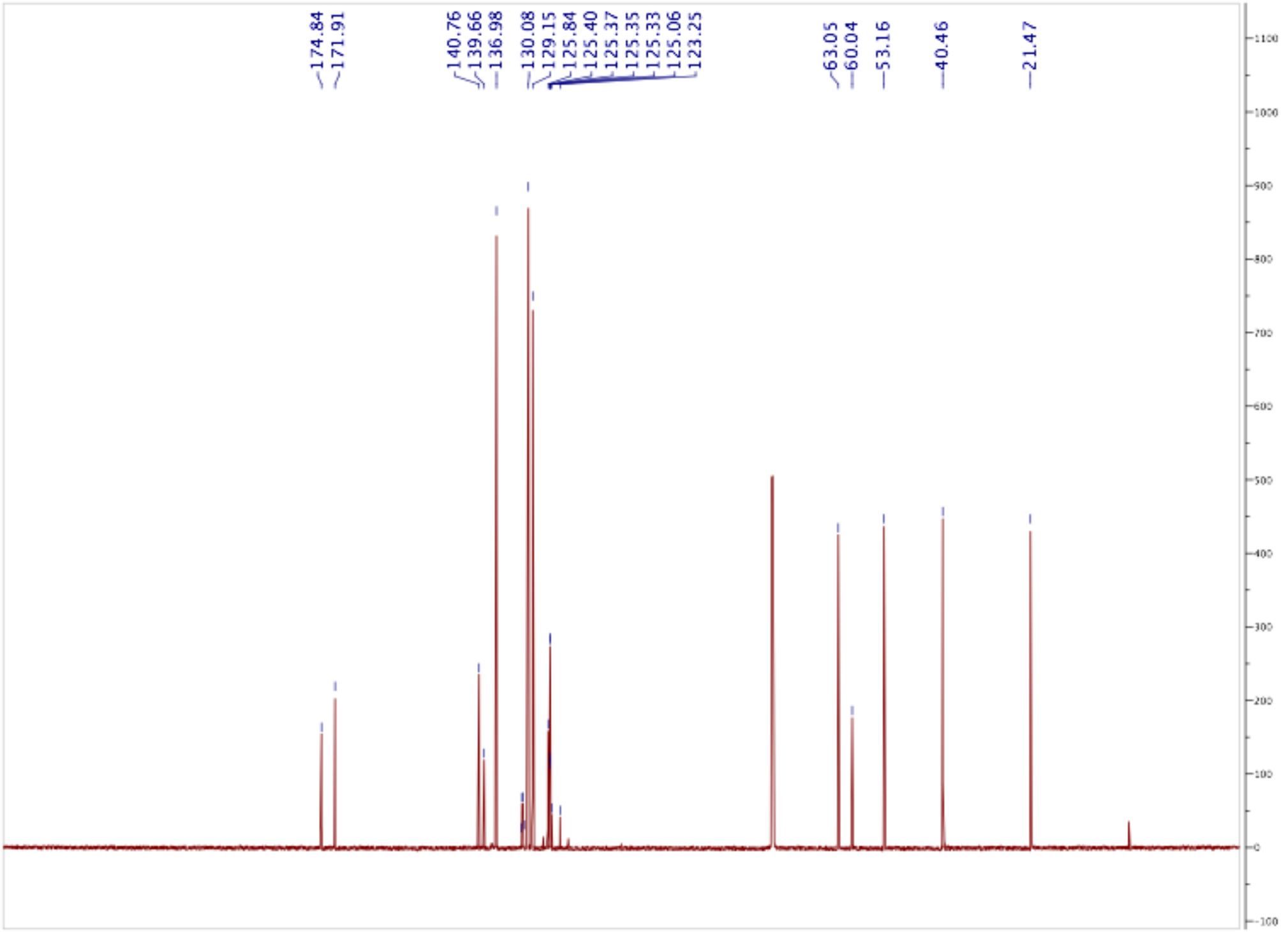
0.99
2.93
HN
O
CO2CH3
Sp-tol
F3C

30a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125.37
125.35
125.33
125.06
123.25
63.05
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21.47
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1000
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800
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500
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0
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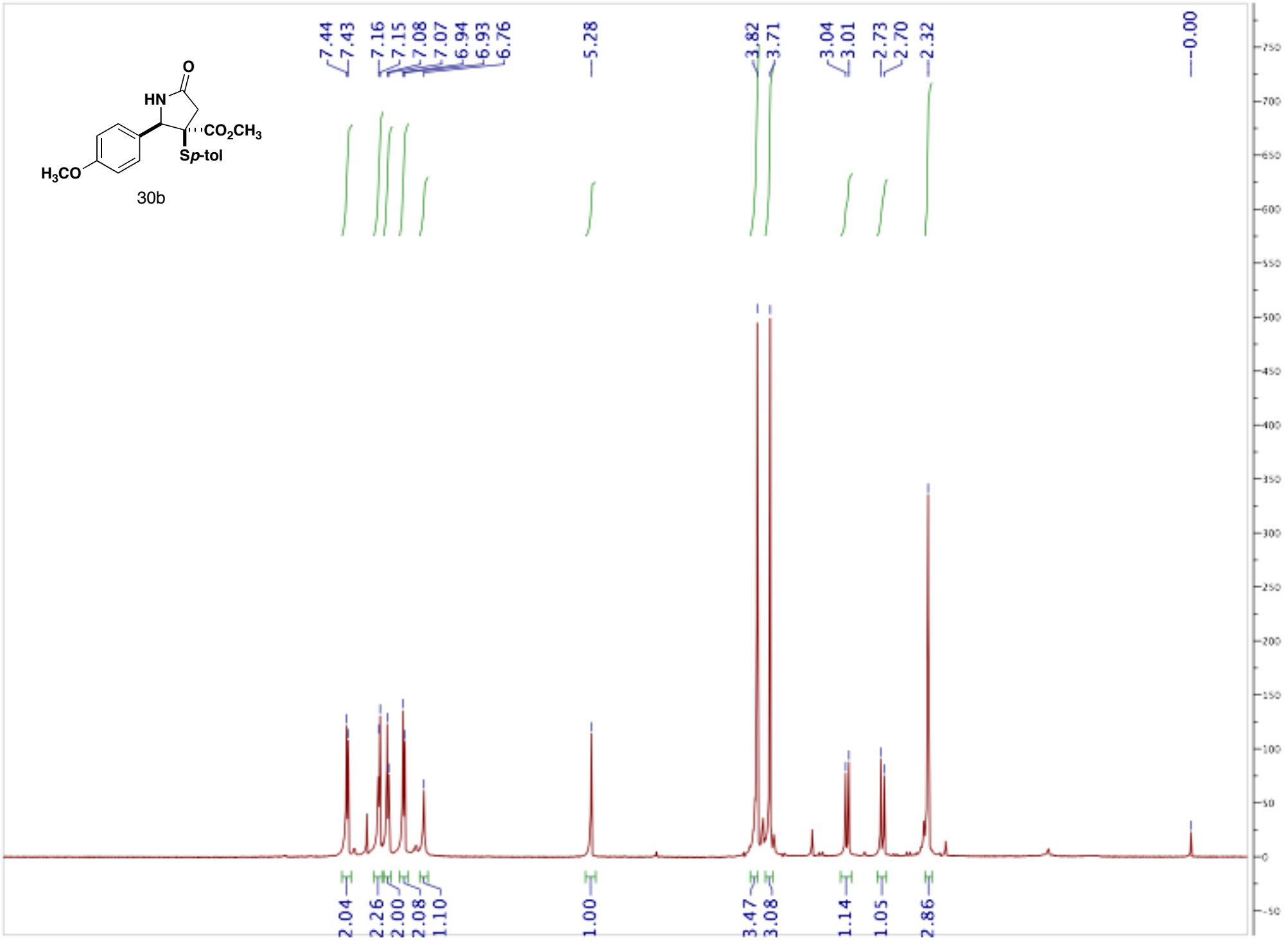
O
HN
CO2CH3
Sp-tol
H3CO
30b
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7.08
7.07
6.94
6.93
6.76
5.28
3.82
3.71
3.04
3.01
2.73
2.70
2.32
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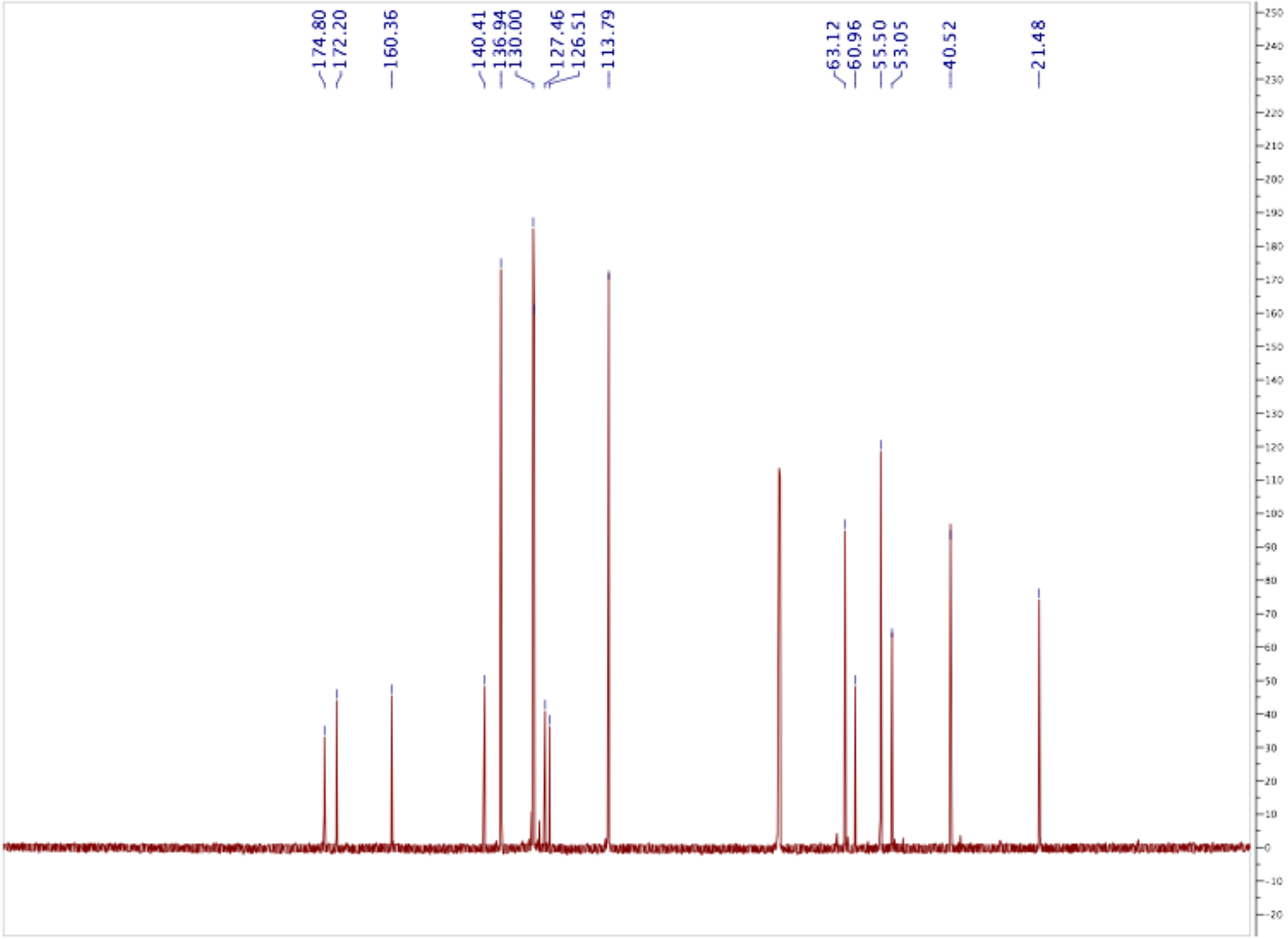
2.26
2.00
2.08
1.10
1.00
3.47
3.08
1.14
1.05
2.86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60.96
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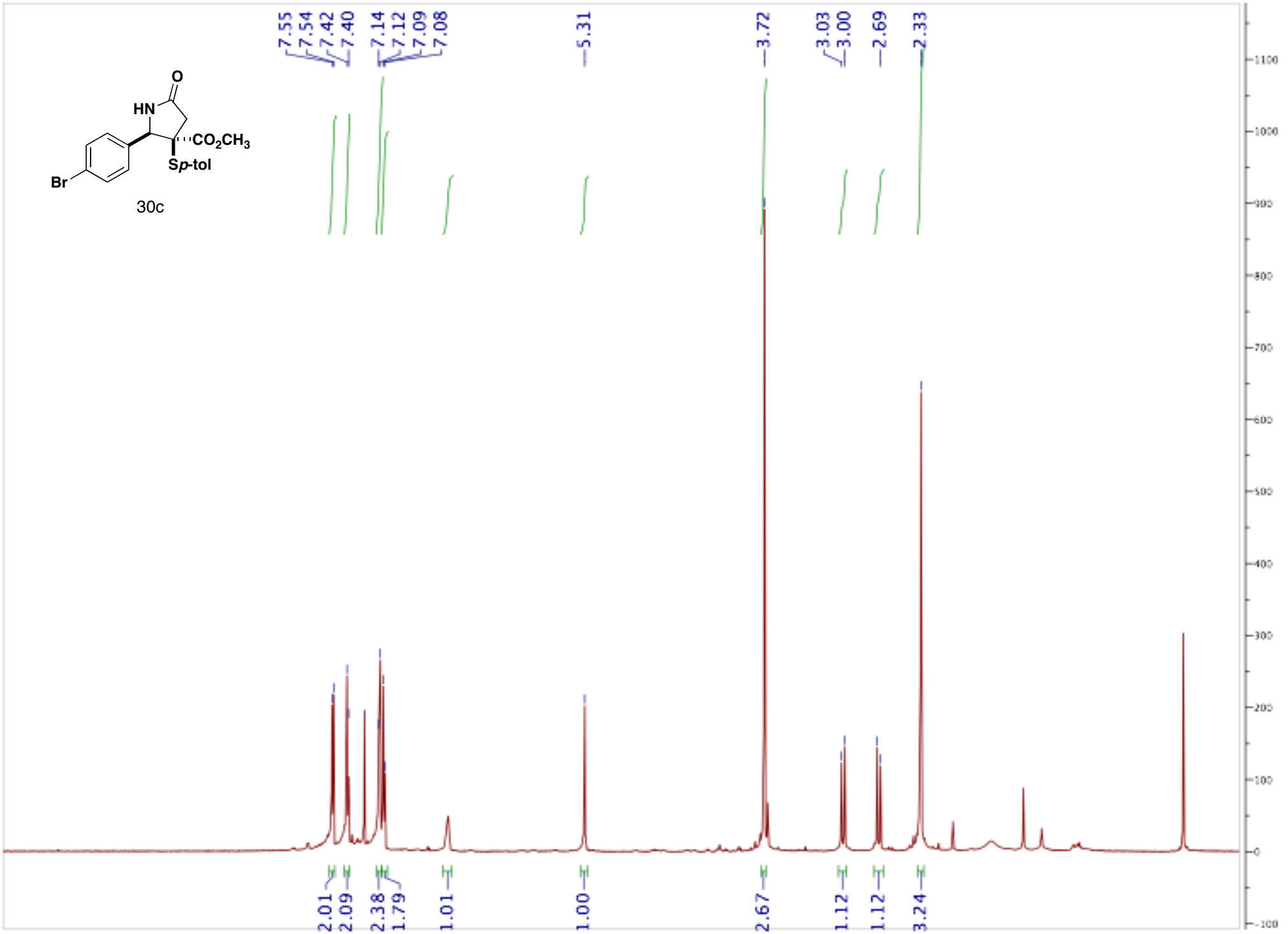
53.05
40.52
21.48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HN
CO2CH3
Sp-tol
Br
30c
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7.54
7.42
7.40
7.14
7.12
7.09
7.08
5.31
3.72
3.03
3.00
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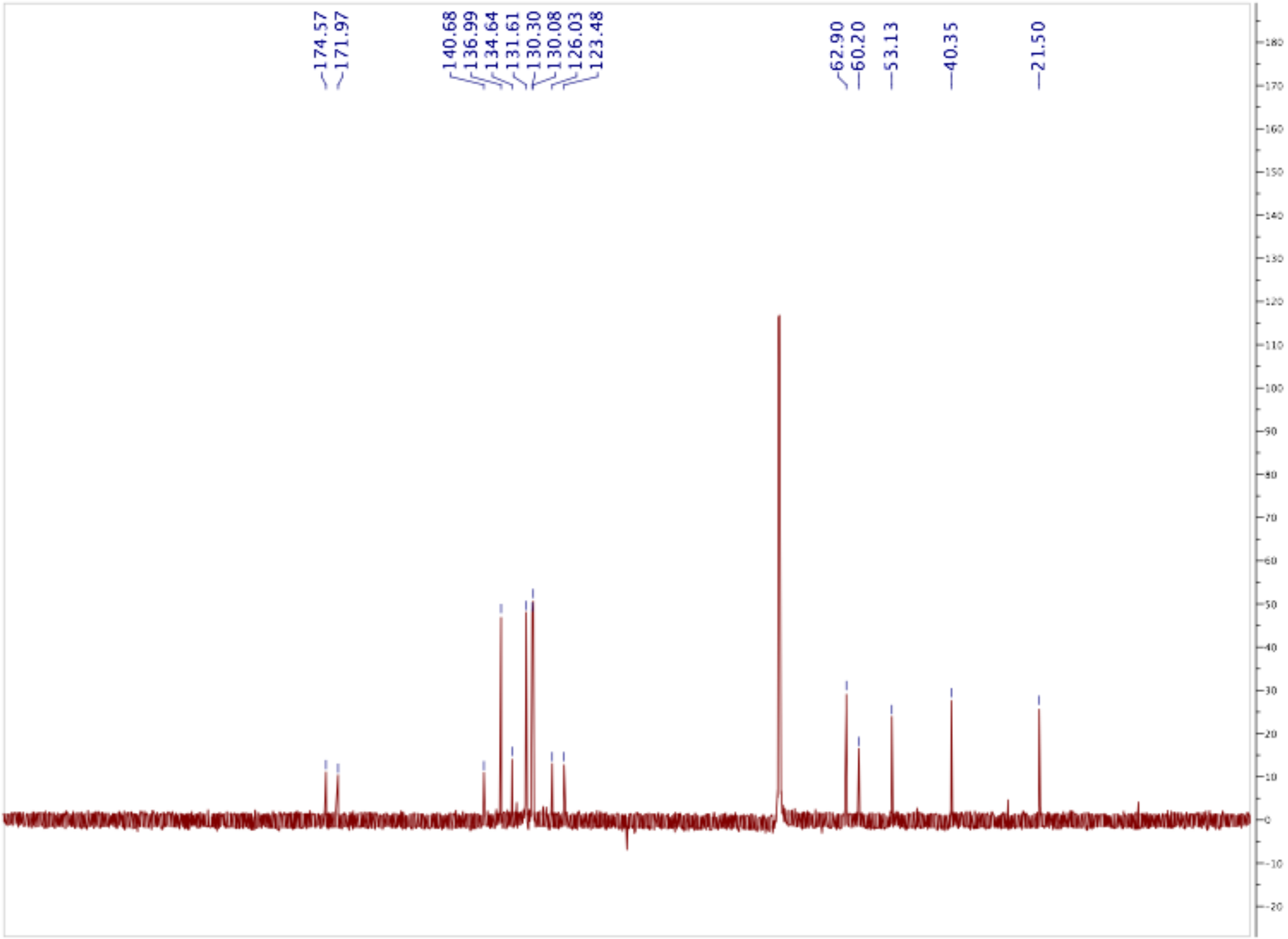
2.01
2.09
2.38
1.79
1.01
1.00
2.67
1.12
1.12
3.24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130.30
130.08
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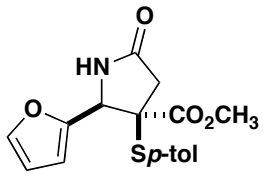
123.48
62.90
60.20
53.13
40.35
21.50


O
HN
O
CO2CH3
Sp-tol

30d

7.48
7.48
7.48
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7.09
7.07
6.41
6.41
6.40
5.82
5.34
3.63
3.06
3.01
2.89
2.83
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2.11
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1.00
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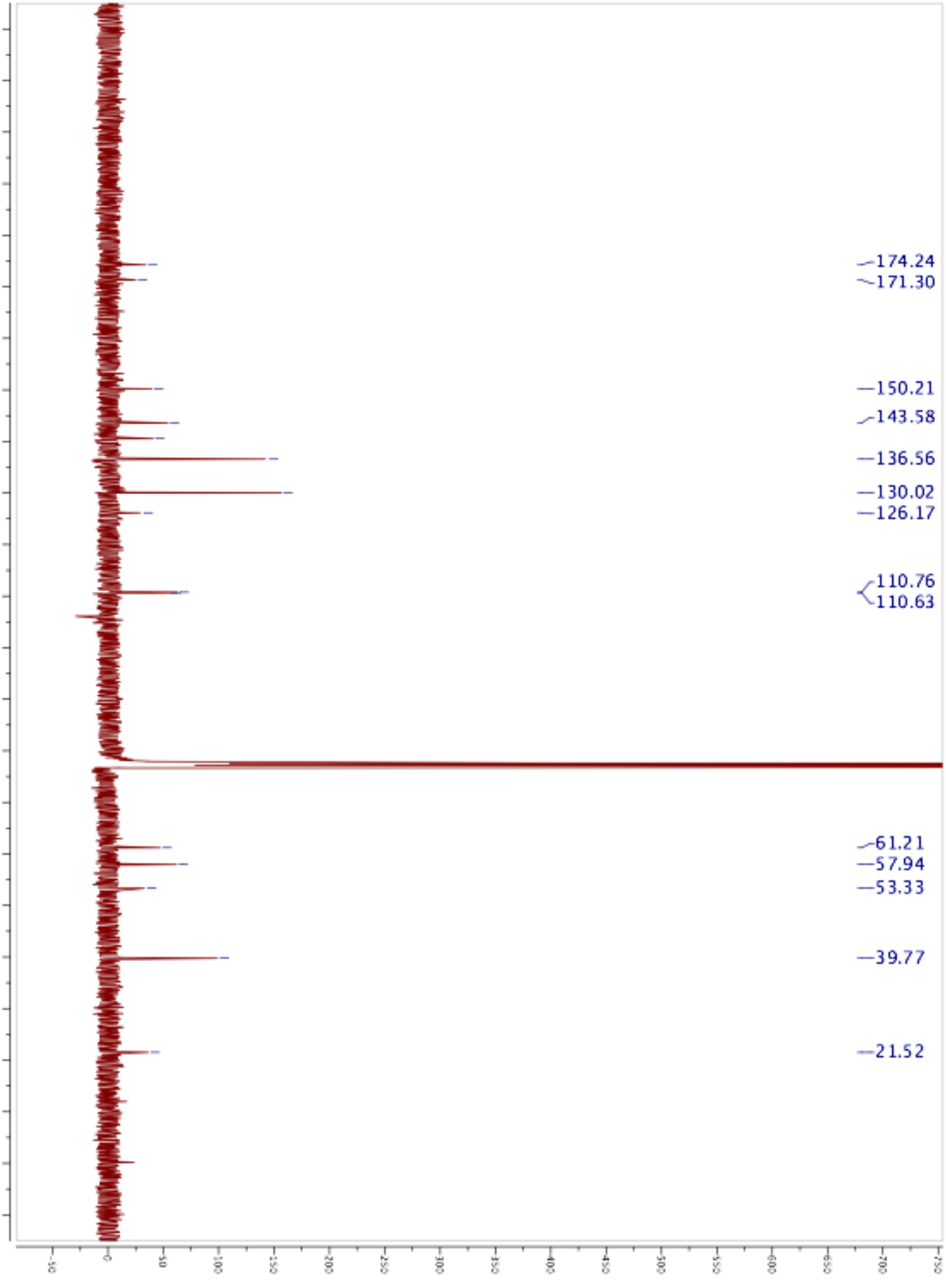
1.27
1.22
3.29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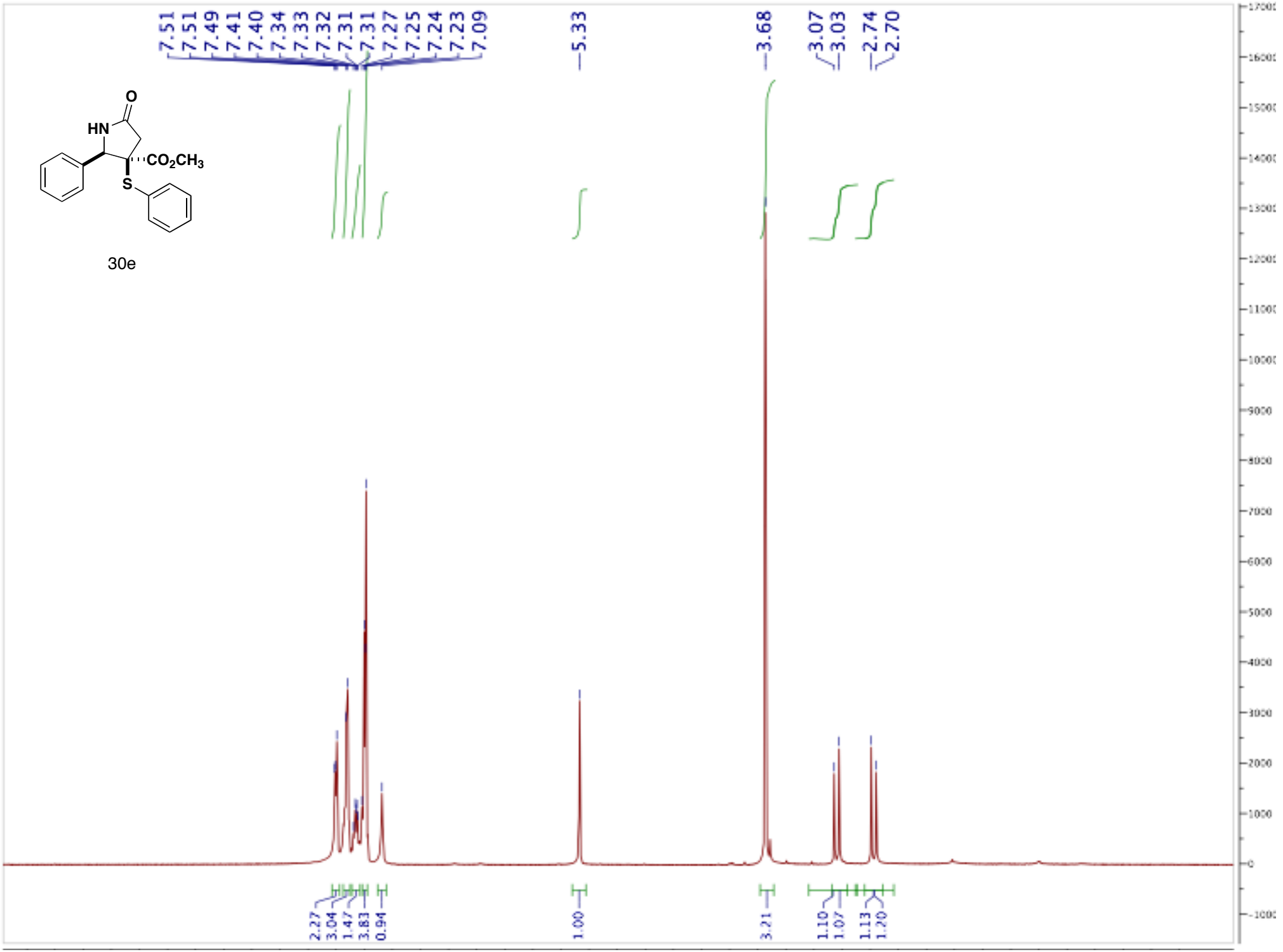
61.21
57.94
53.33
39.77
21.52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7.27
7.25
7.24
7.23
7.09
5.33
3.68
3.07
3.03
2.74
2.70
O
HN
CO2CH3
S
30e
2.27
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1.47
3.83
0.94
1.00
3.21
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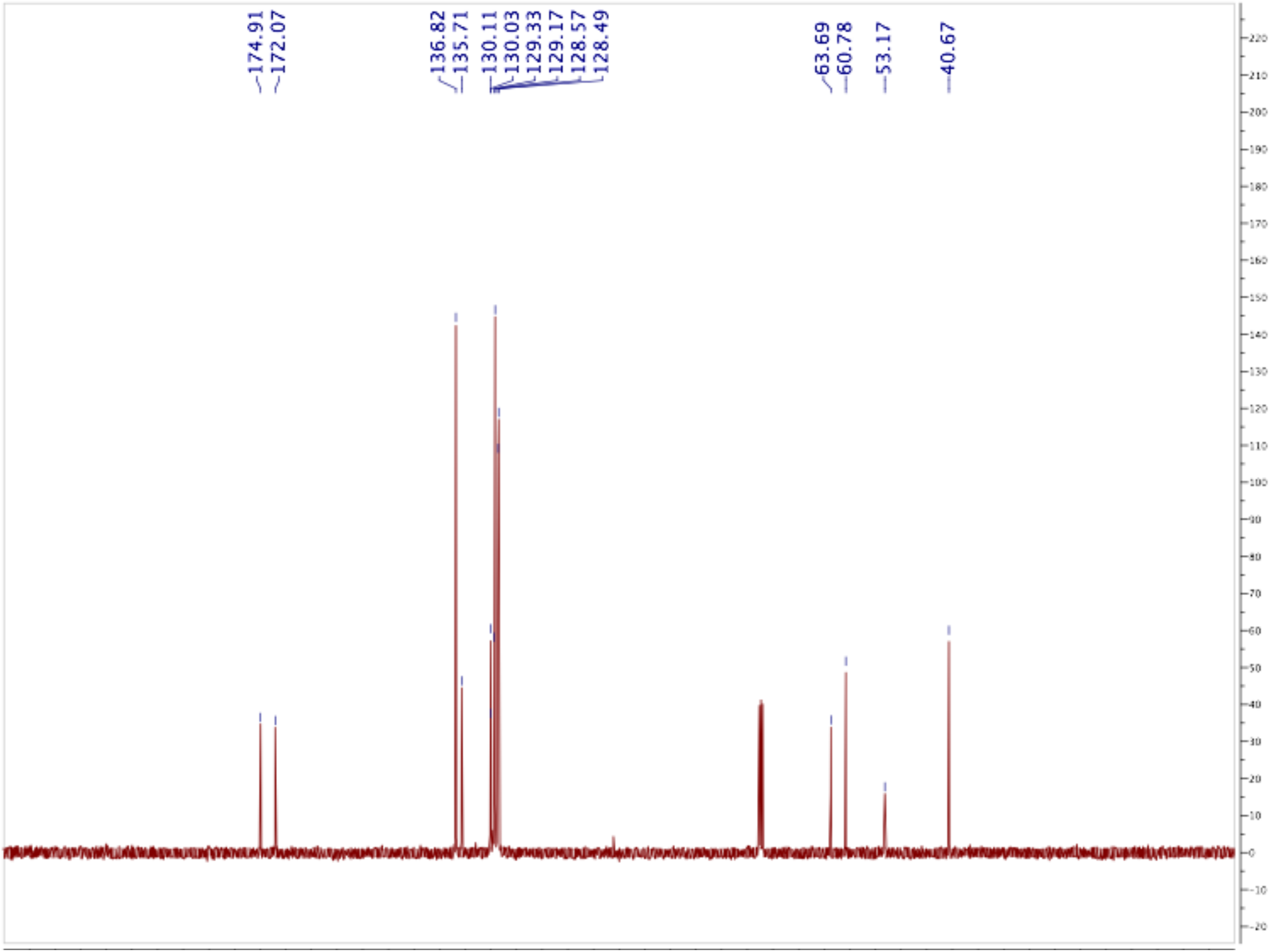
1.07
1.13
1.20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129.17
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53.17
40.67
220
210
200
190
180
170
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120
110
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90
80
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50
40
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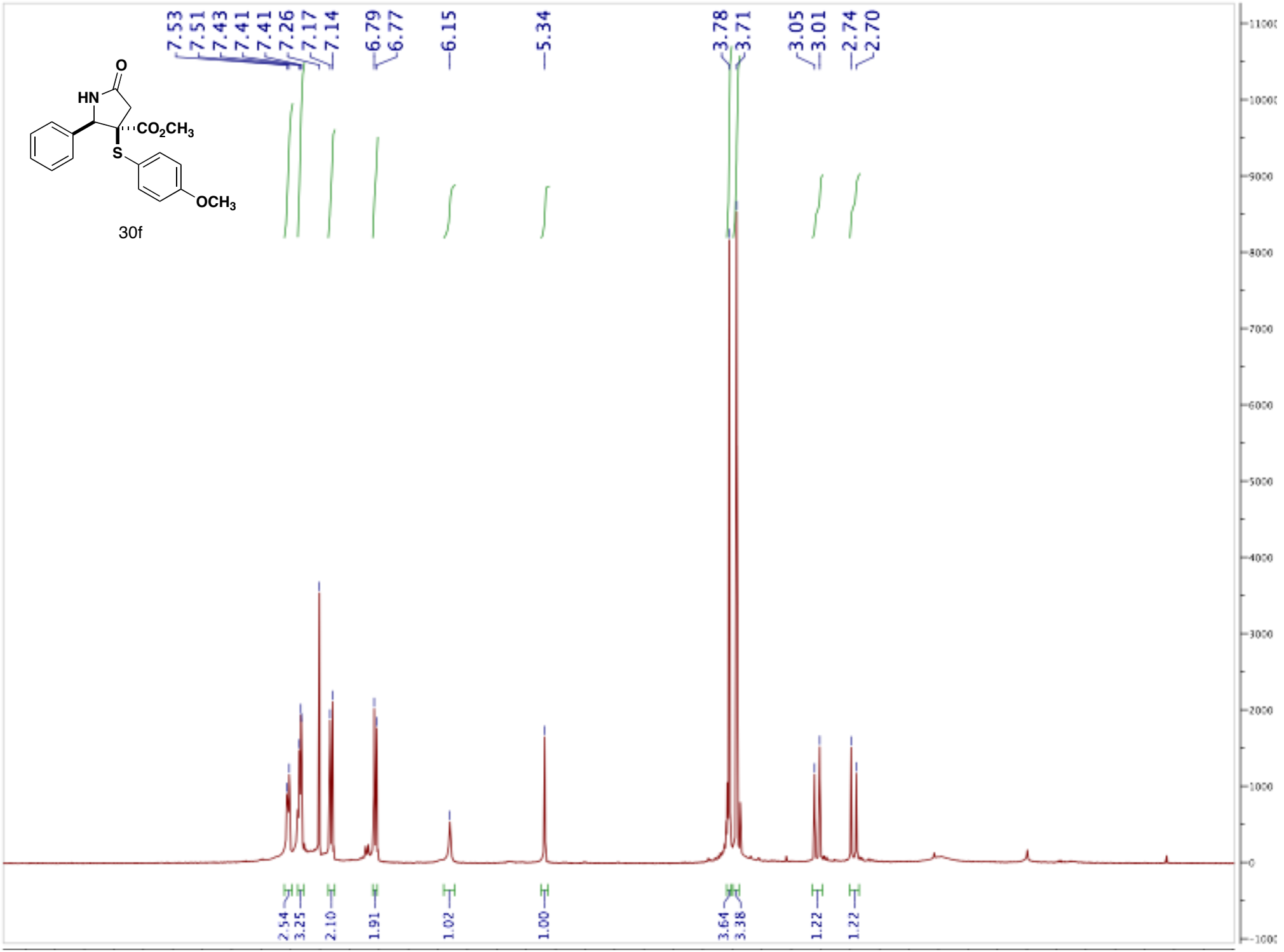
10
0
-10
-20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7.14
6.79
6.77
6.15
5.34
3.78
3.71
3.05
3.01
2.74
2.70
HN
O
CO2CH3
S
OCH3
30f
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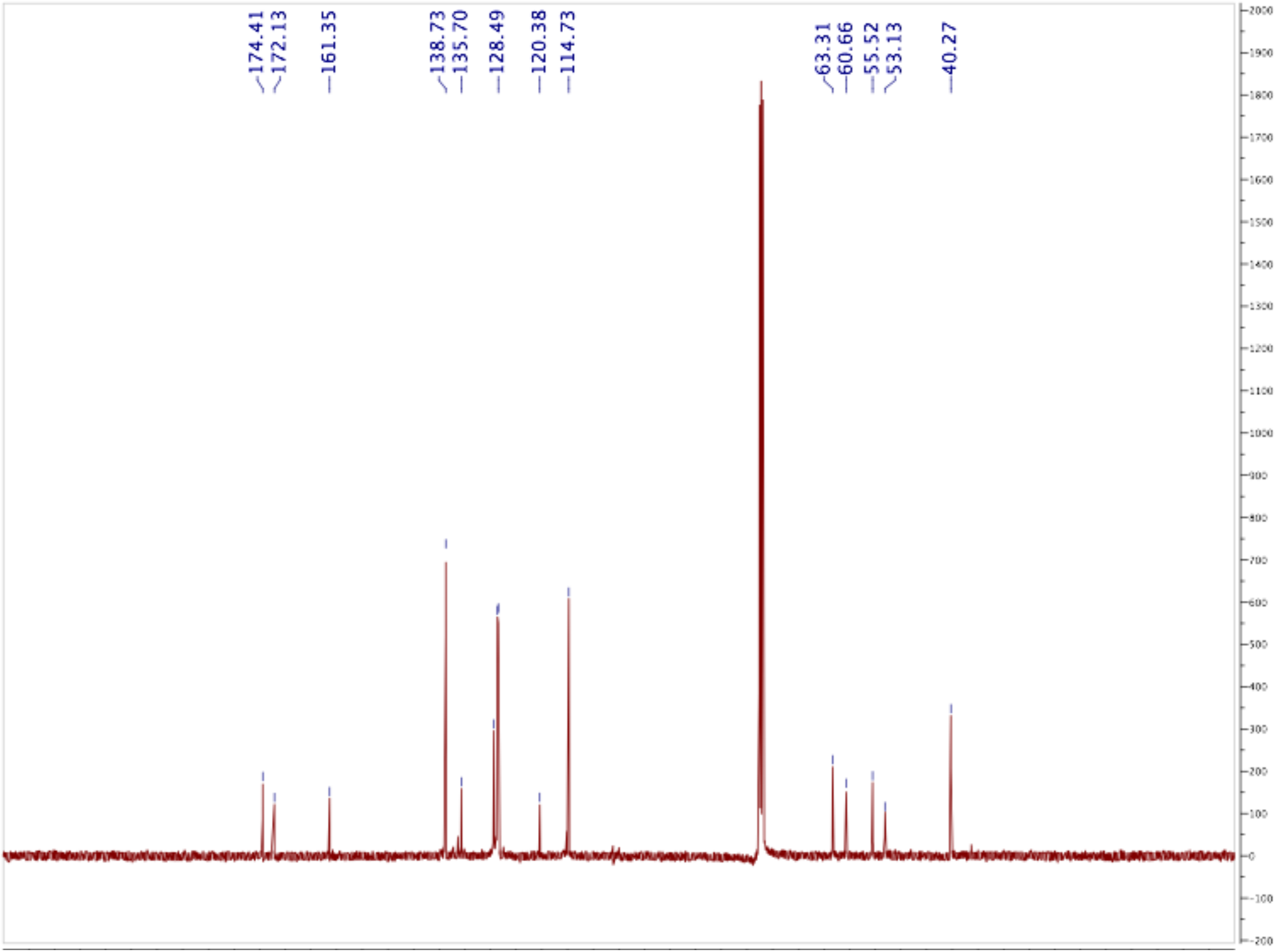
2.10
1.91
1.02
1.00
3.64
3.38
1.22
1.22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60.66
55.52
53.13
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200
190
180
170
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150
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120
110
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90
80
70
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40
30
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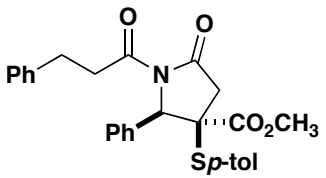
10
0
-10
-20


O
O
Ph
N
Ph
CO2CH3
Sp-tol

31a

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3.42
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3.31
1.26
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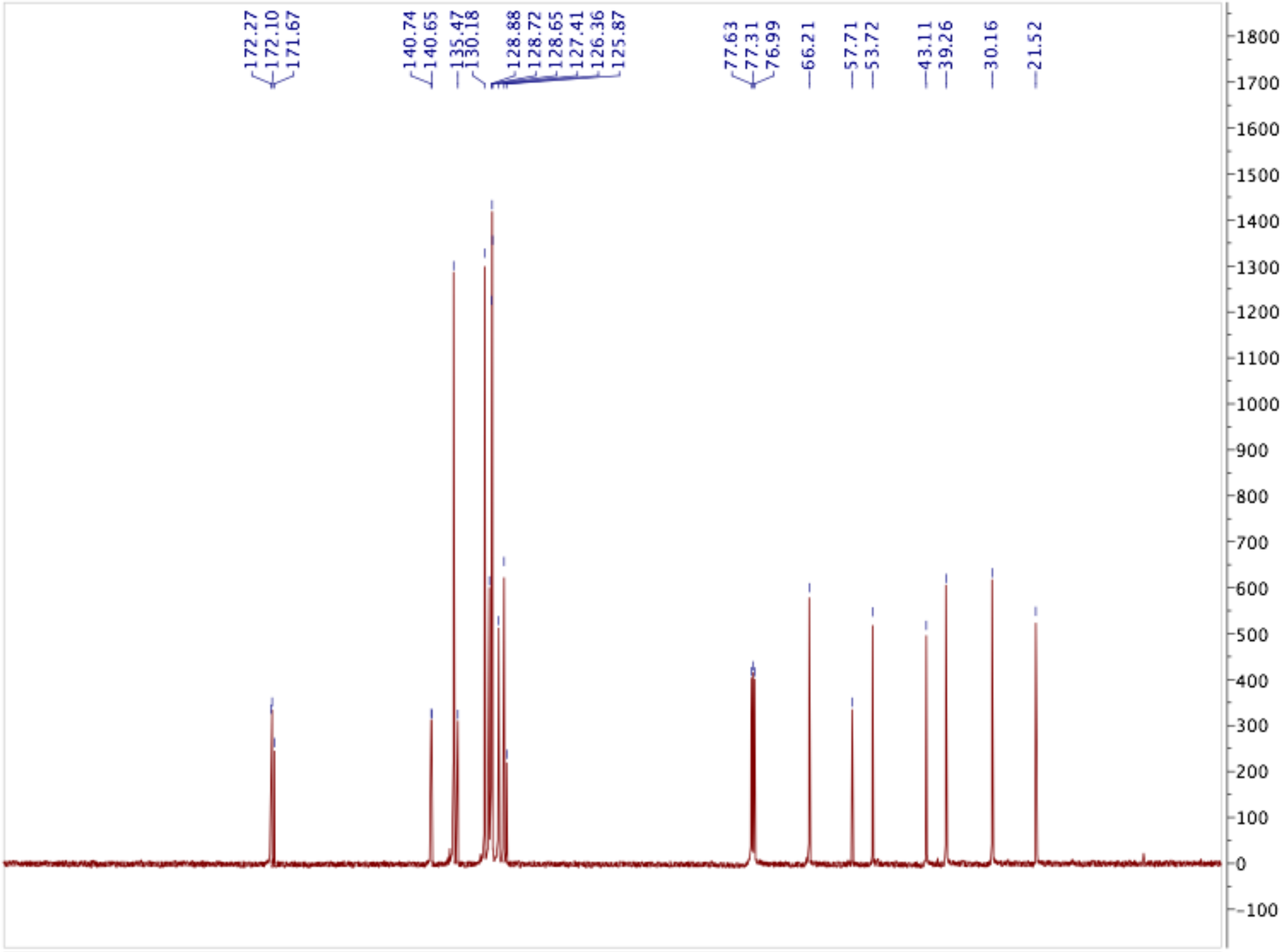
1.12
2.45
3.47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125.87
77.63
77.31
76.99
66.21
57.71
53.72
43.11
39.26
30.16
21.52
1800
1700
1600
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1300
1200
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1000
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0
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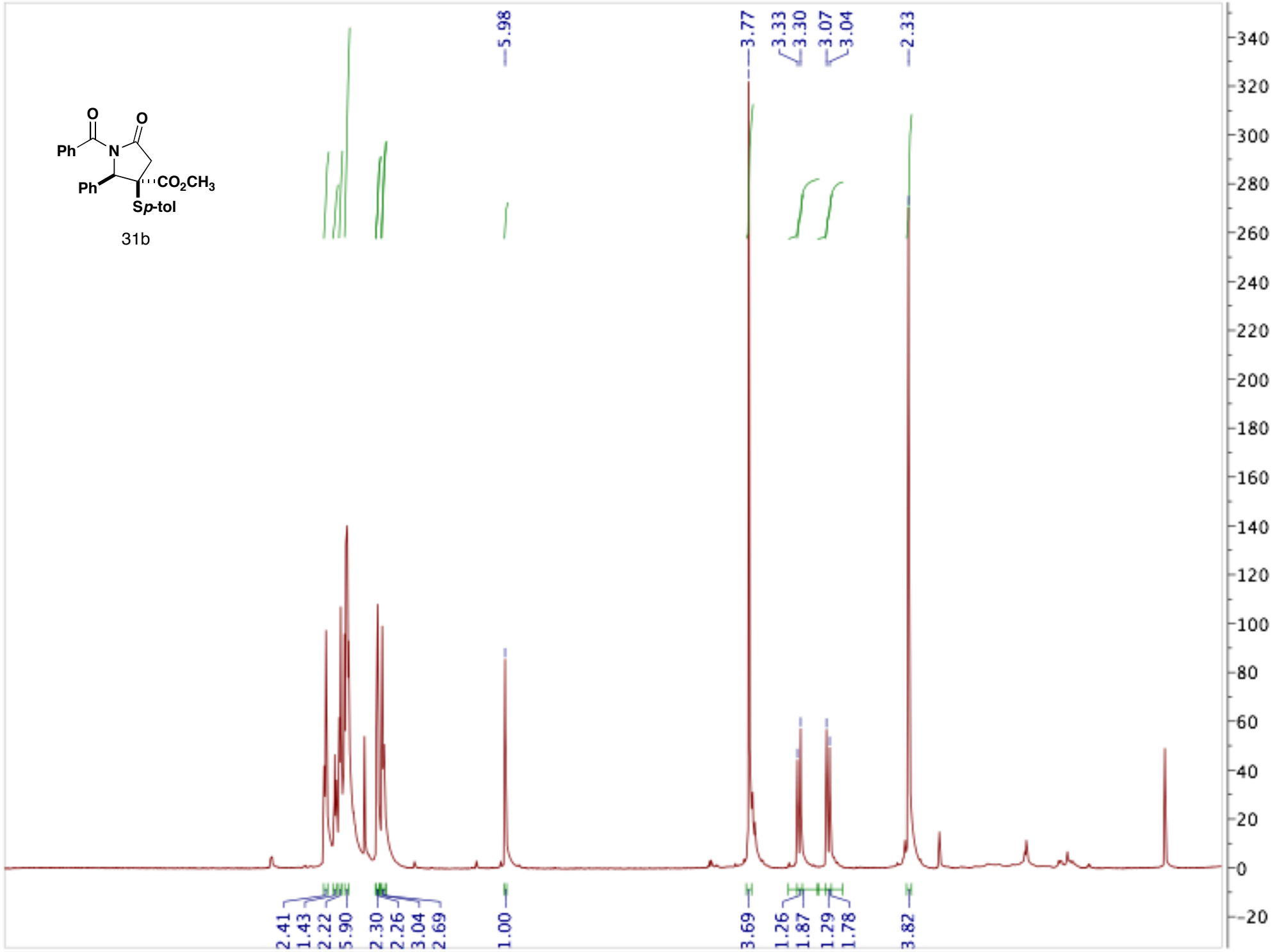
O
O
Ph
N
Ph
CO2CH3
Sp-tol
31b
5.98
3.77
3.33
3.30
3.07
3.04
2.33
2.41
1.43
2.22
5.90
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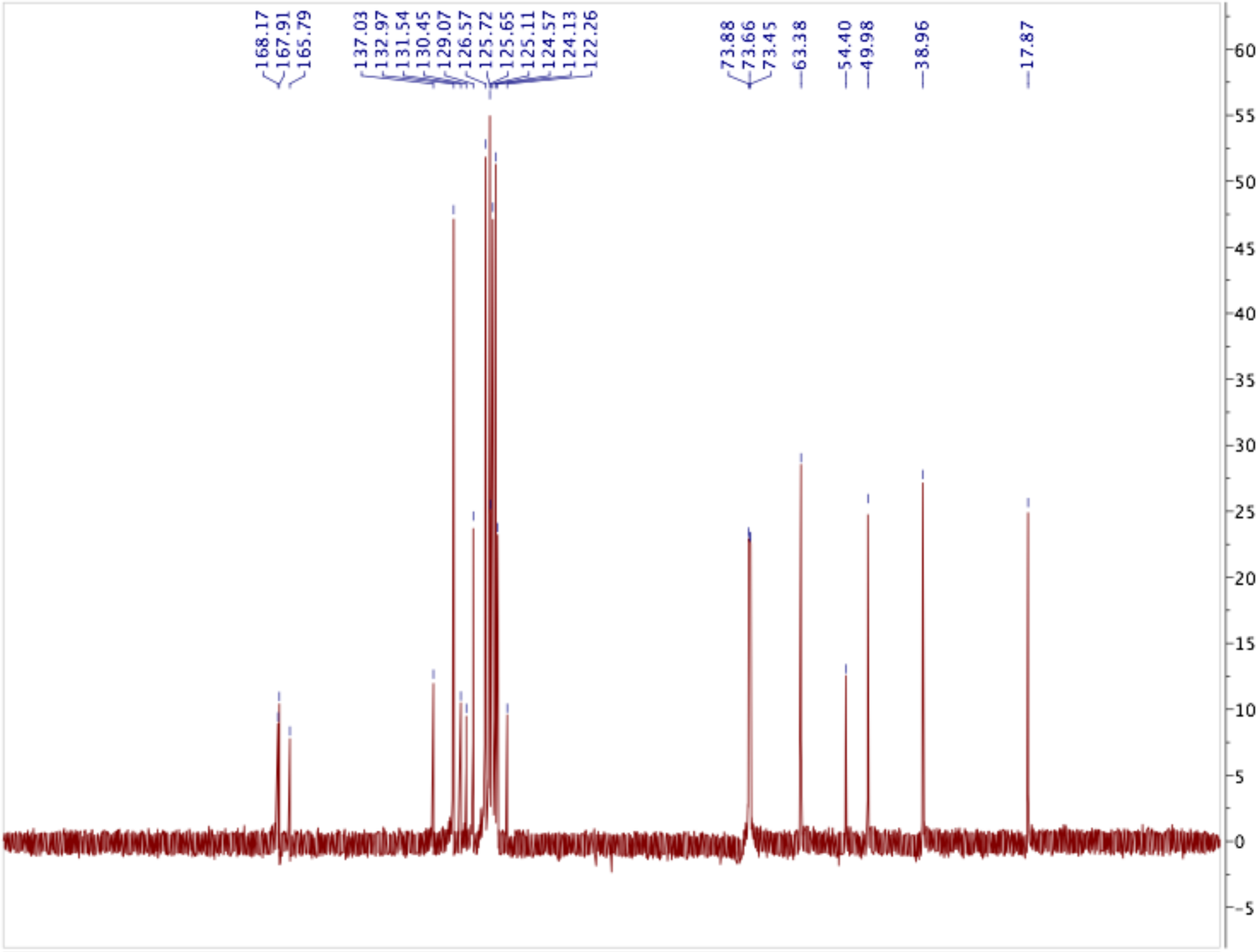
2.26
3.04
2.69
1.00
3.69
1.26
1.87
1.29
1.78
3.82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129.07
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125.11
124.57
124.13
122.26
73.88
73.66
73.45
63.38
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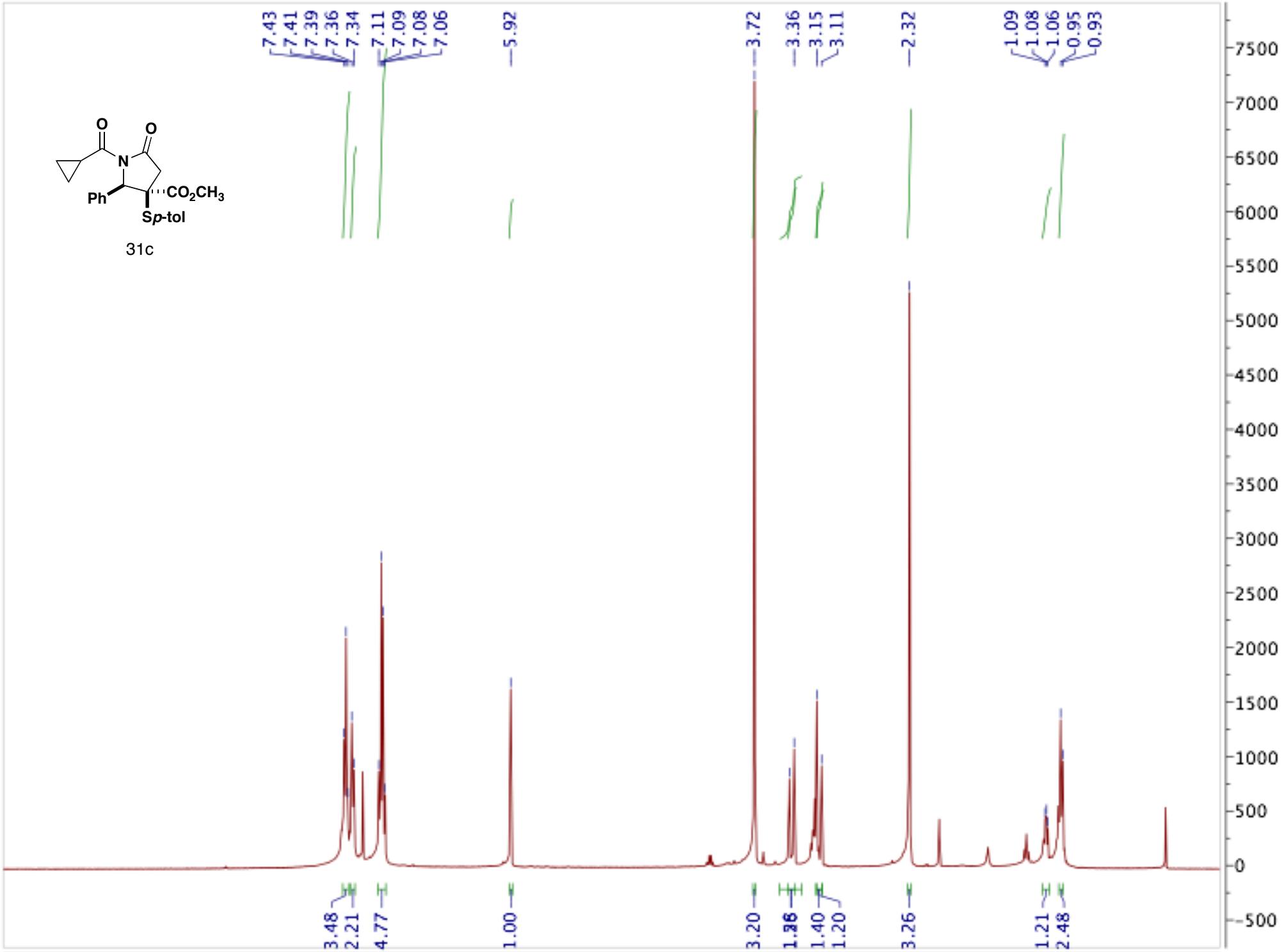
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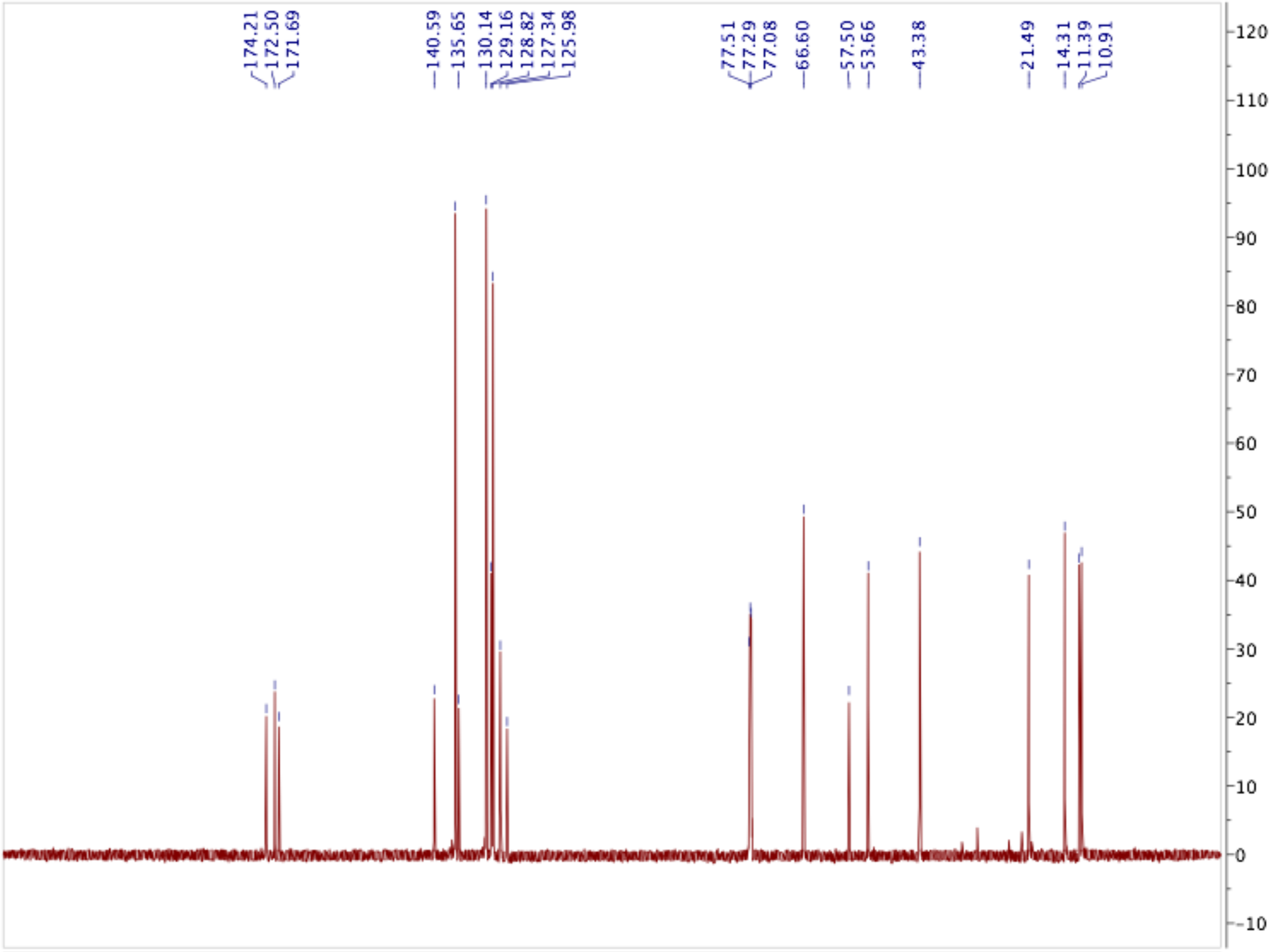
7.08
7.06
5.92
3.72
3.36
3.15
3.11
2.32
1.09
1.08
1.06
0.95
0.93
7500
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
3.48
2.21
4.77
1.00
3.20
1.40
1.20
3.26
1.21
2.48
O
O
N
Ph
CO2CH3
Sp-tol

31c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125.98
77.51
77.29
77.08
66.60
57.50
53.66
43.38
21.49
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100
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80
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60
50
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20
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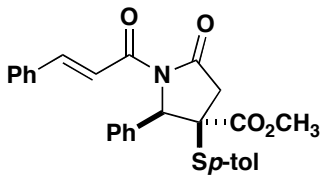
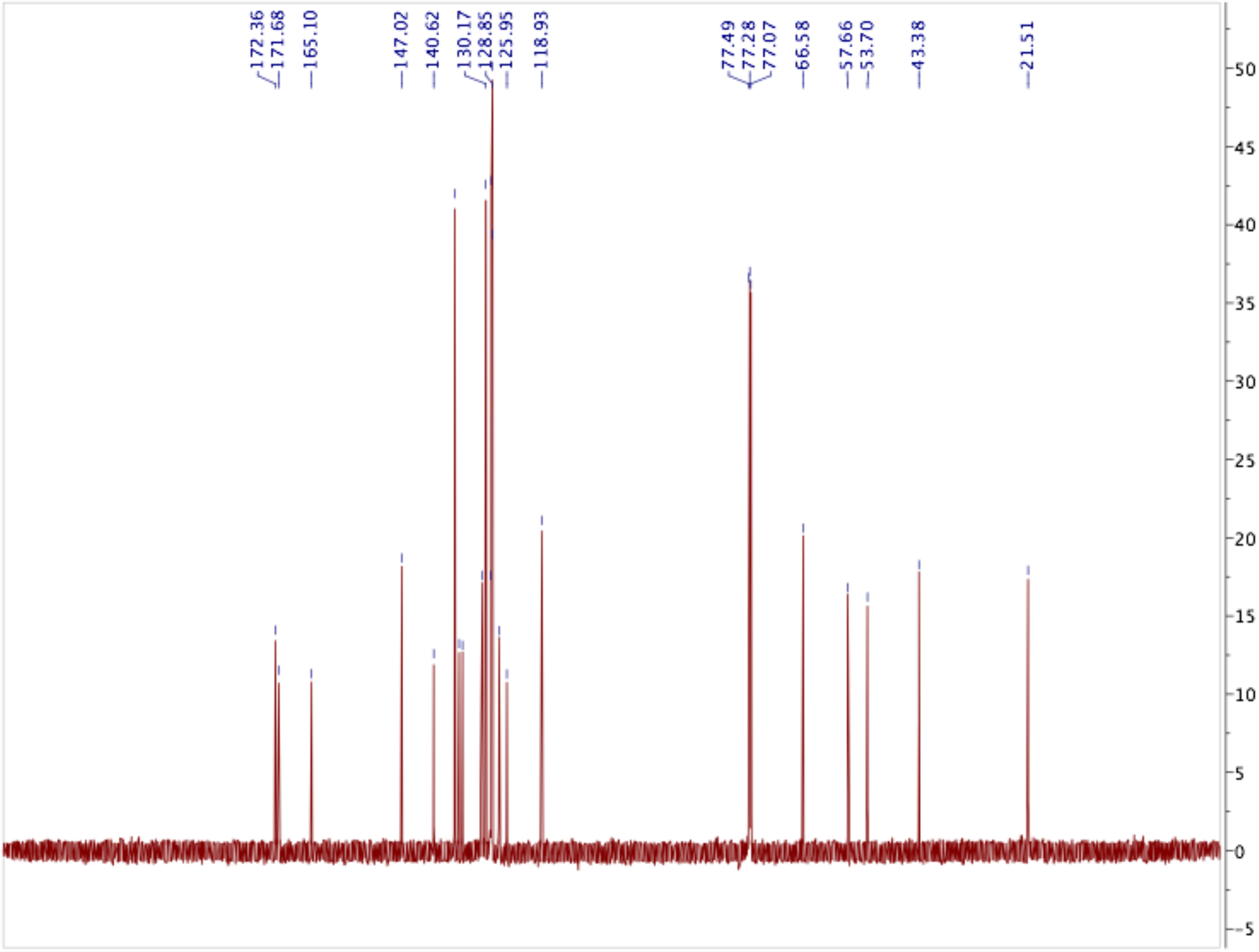

O
O
Ph
N
Ph
CO2CH3
Sp-tol

31d

550
500
450
400
350
300
250
200
150
100
50
0
-50
1.27
1.28
1.29
1.20
2.55
8.96
4.42
1.00
3.02
1.50
1.27
3.82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77.49
77.28
77.07
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30
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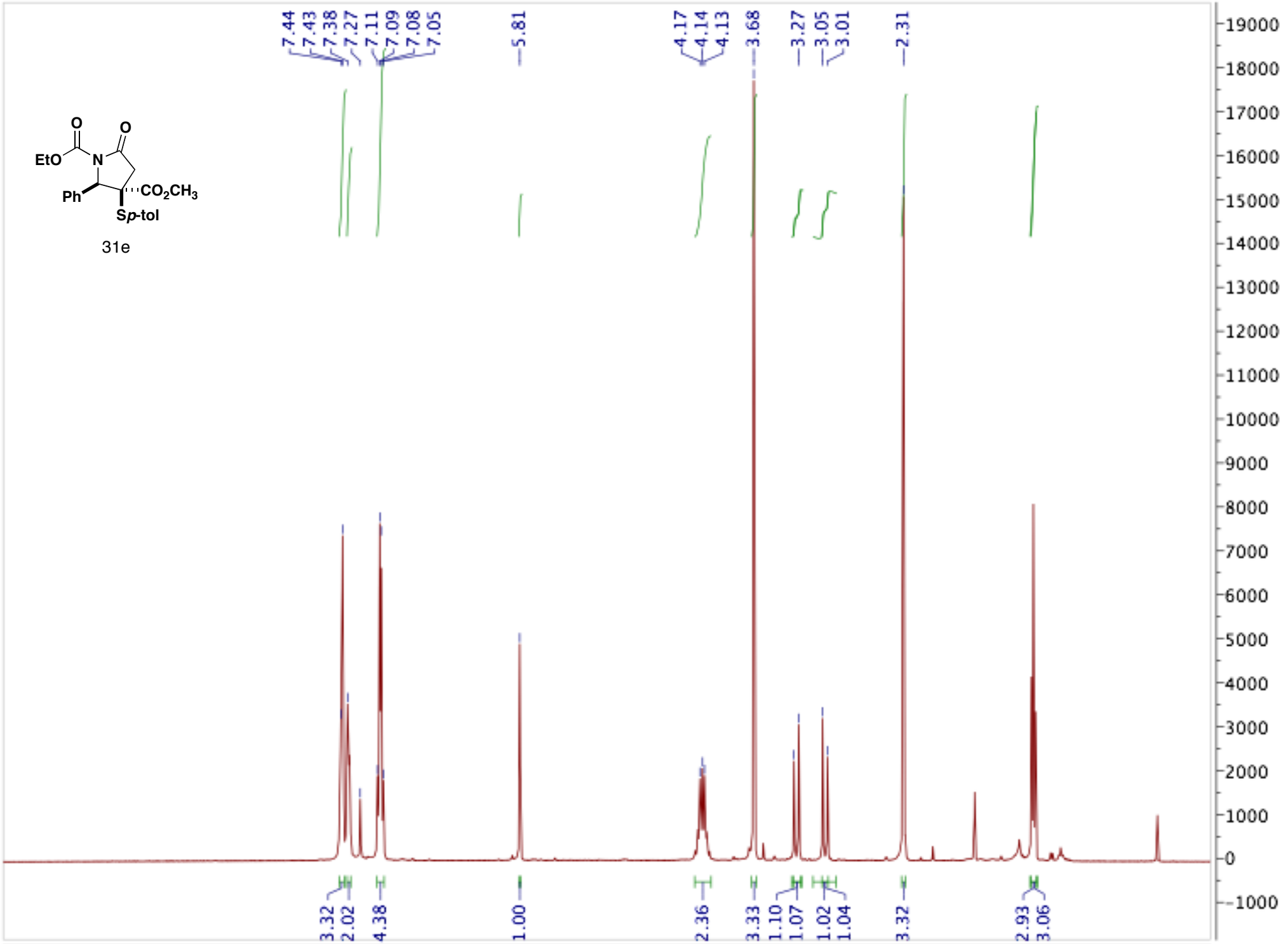
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7.05
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4.13
3.68
3.27
3.05
3.01
2.31
3.32
2.02
4.38
1.00
2.36
3.33
1.10
1.07
1.02
1.04
3.32
2.93
3.06
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
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6000
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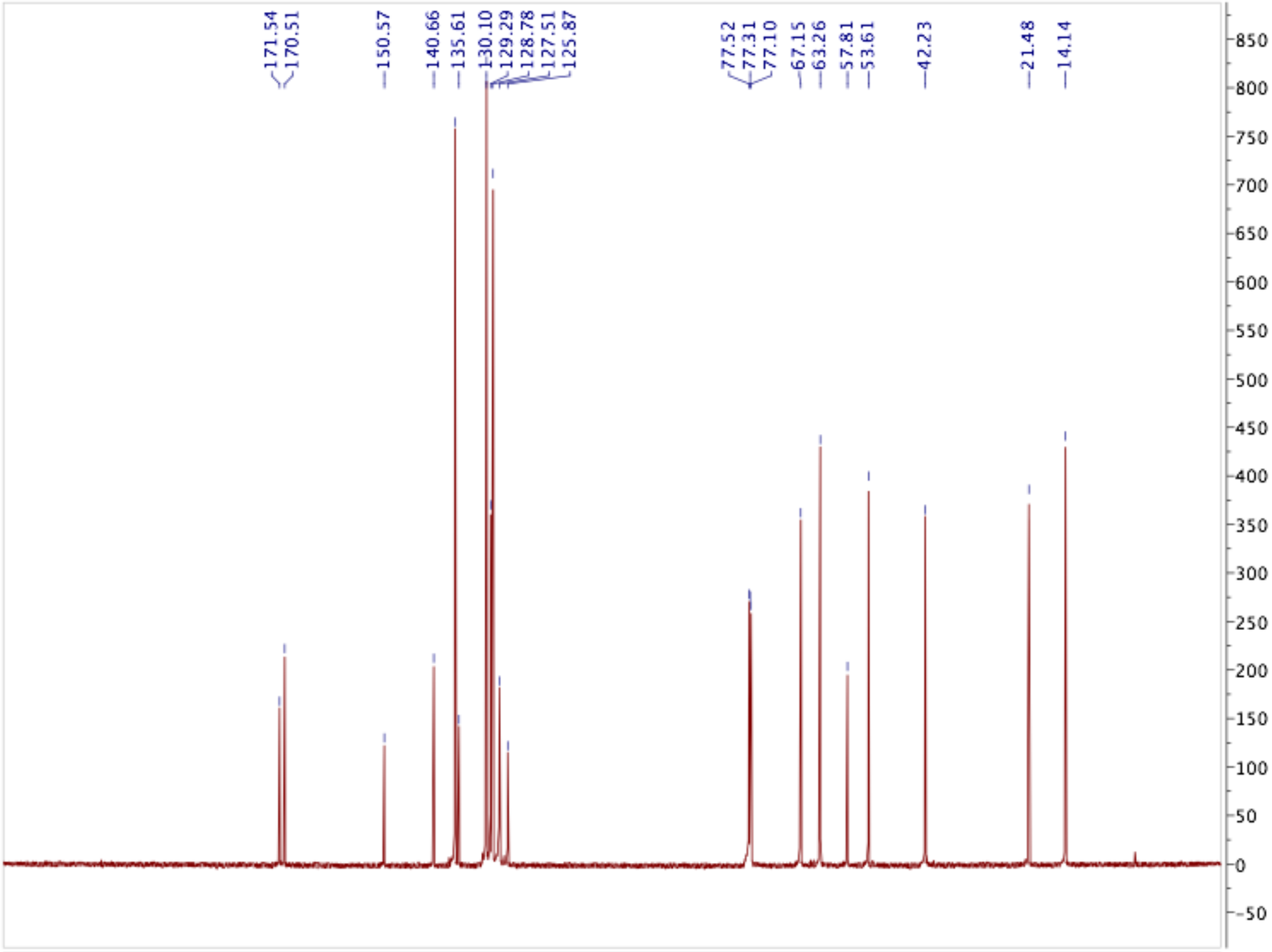
4000
3000
2000
1000
0
-1000

O
O
EtO
N
Ph
CO2CH3
Sp-tol

31e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77.52
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77.10
67.15
63.26
57.81
53.61
42.23
21.48
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700
650
600
550
500
450
400
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300
250
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0
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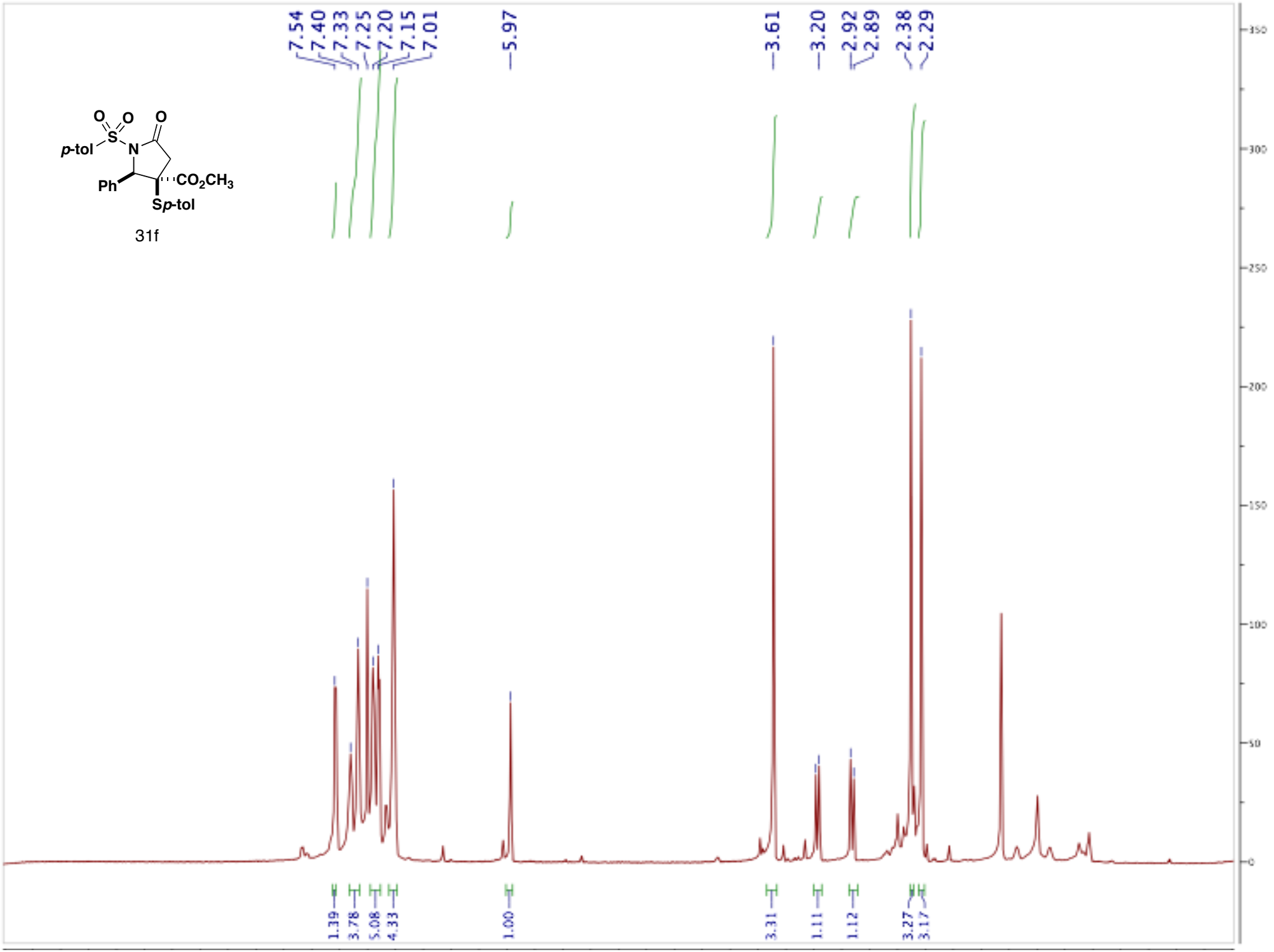
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7.15
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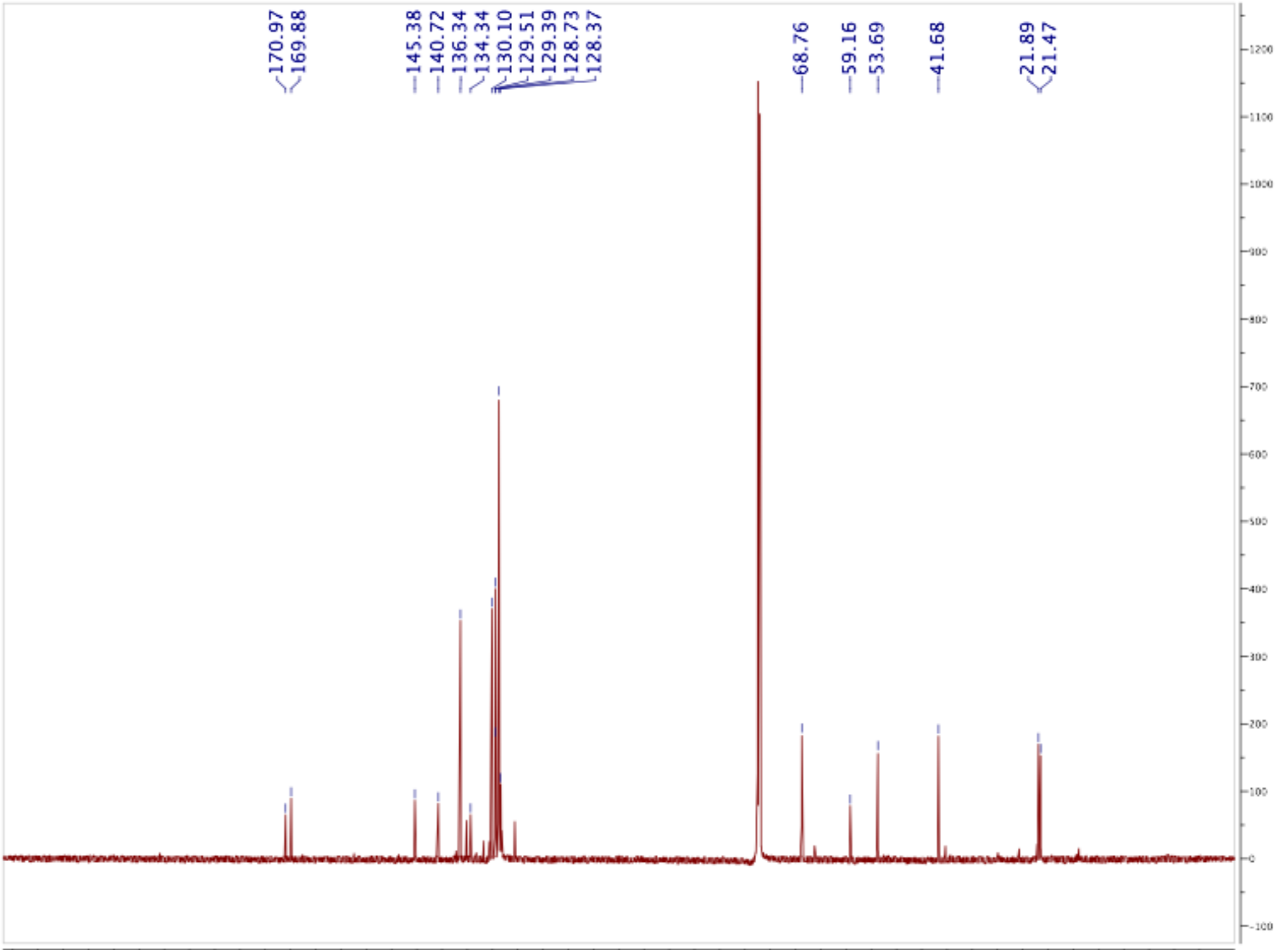
5.97
3.61
3.20
2.92
2.89
2.38
2.29
1.39
3.78
5.08
4.33
1.00
3.31
1.11
1.12
3.27
3.17
350
300
250
200
150
100
50
0
p-tol
Ph
CO2CH3
Sp-tol

31f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134.34
130.10
129.51
129.39
128.73
128.37
68.76
59.16
53.69
41.68
21.89
21.47
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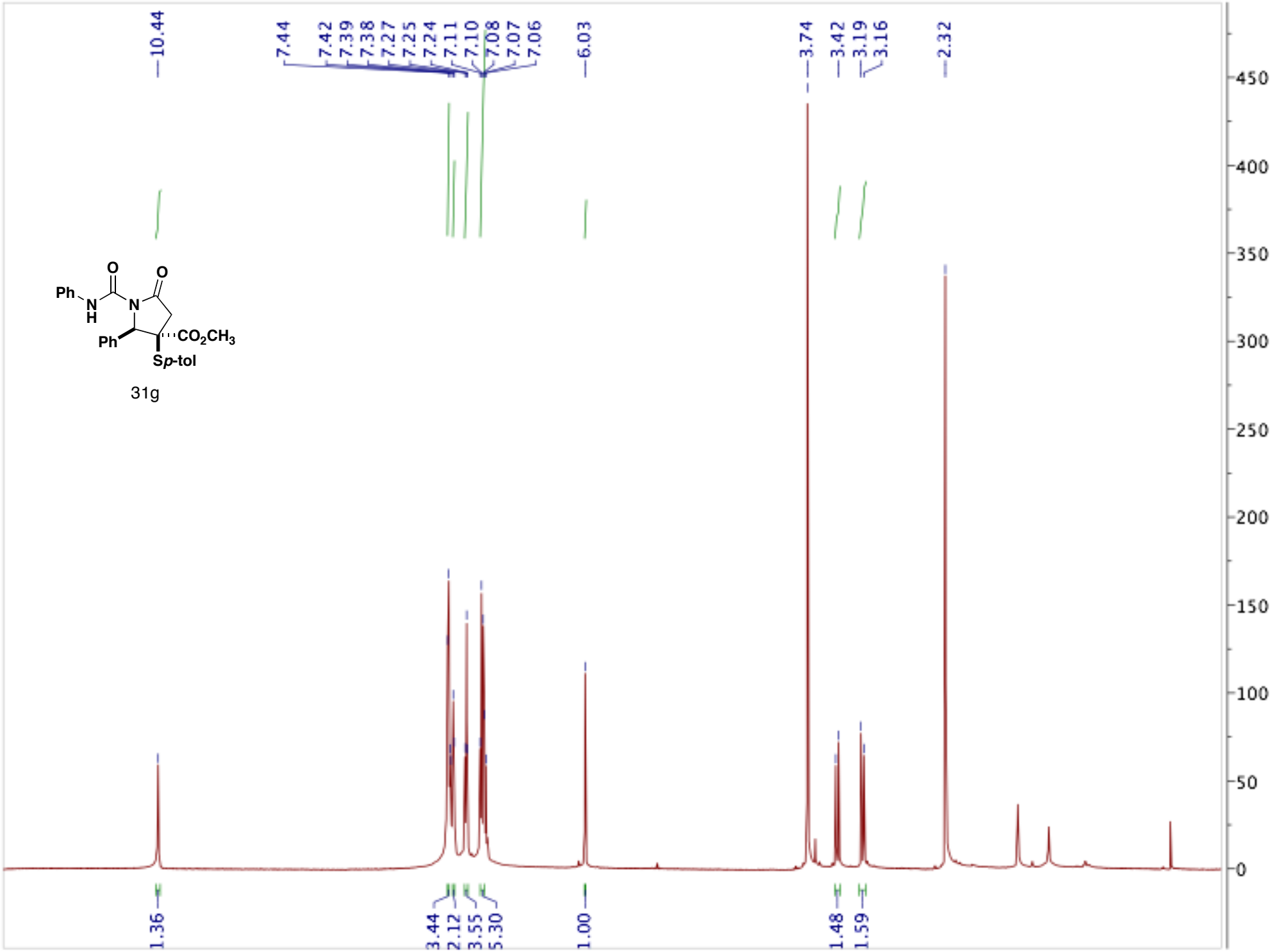
1100
1000
900
800
700
600
500
400
300
200
100
0
-100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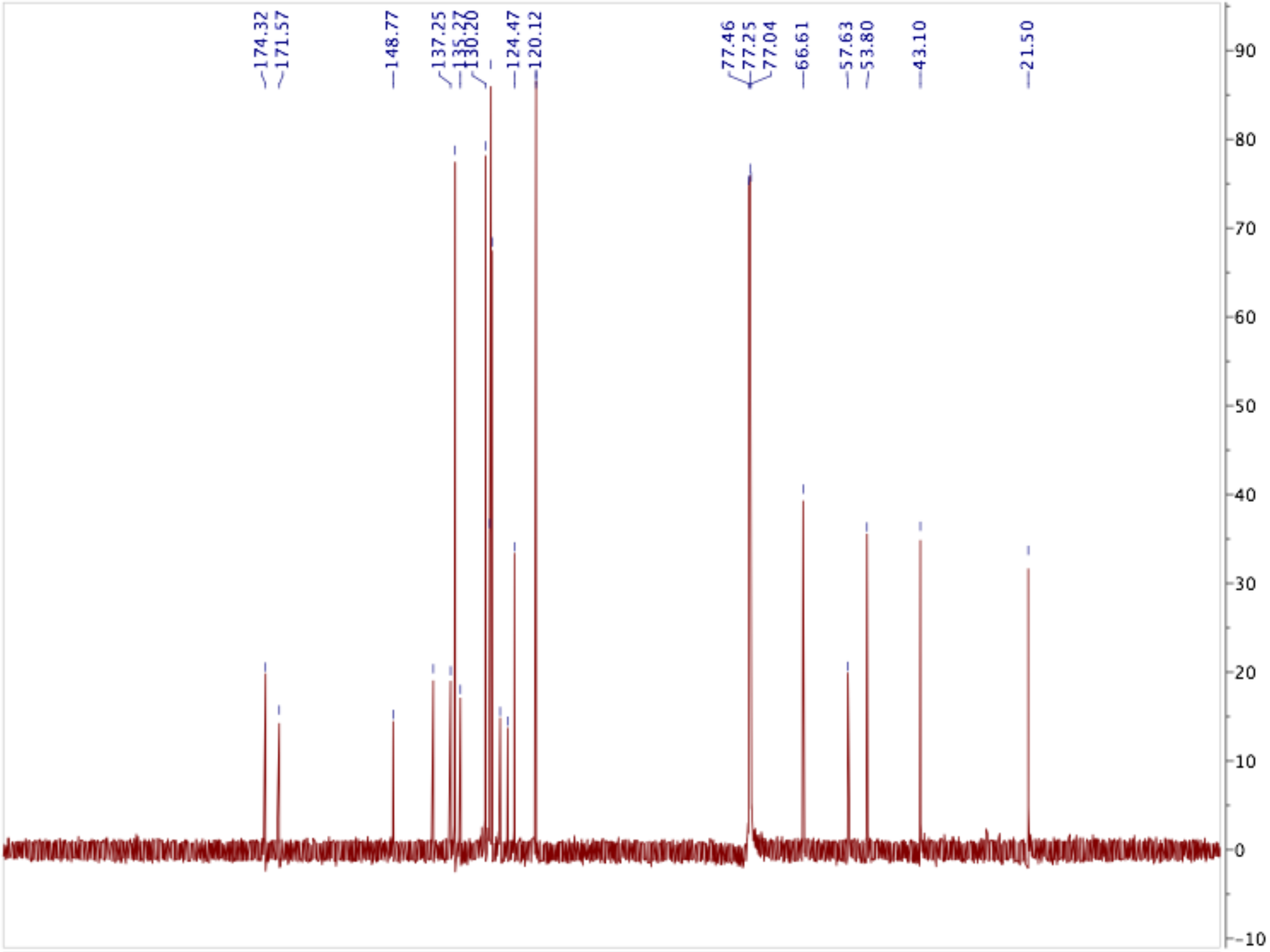
7.24
7.11
7.10
7.08
7.07
7.06
6.03
3.74
3.42
3.19
3.16
2.32
450
400
350
300
250
200
150
100
50
0
1.36
3.44
2.12
3.55
5.30
1.00
1.48
1.59

Ph
N
H
O
N
O
Ph
CO2CH3
Sp-tol

31g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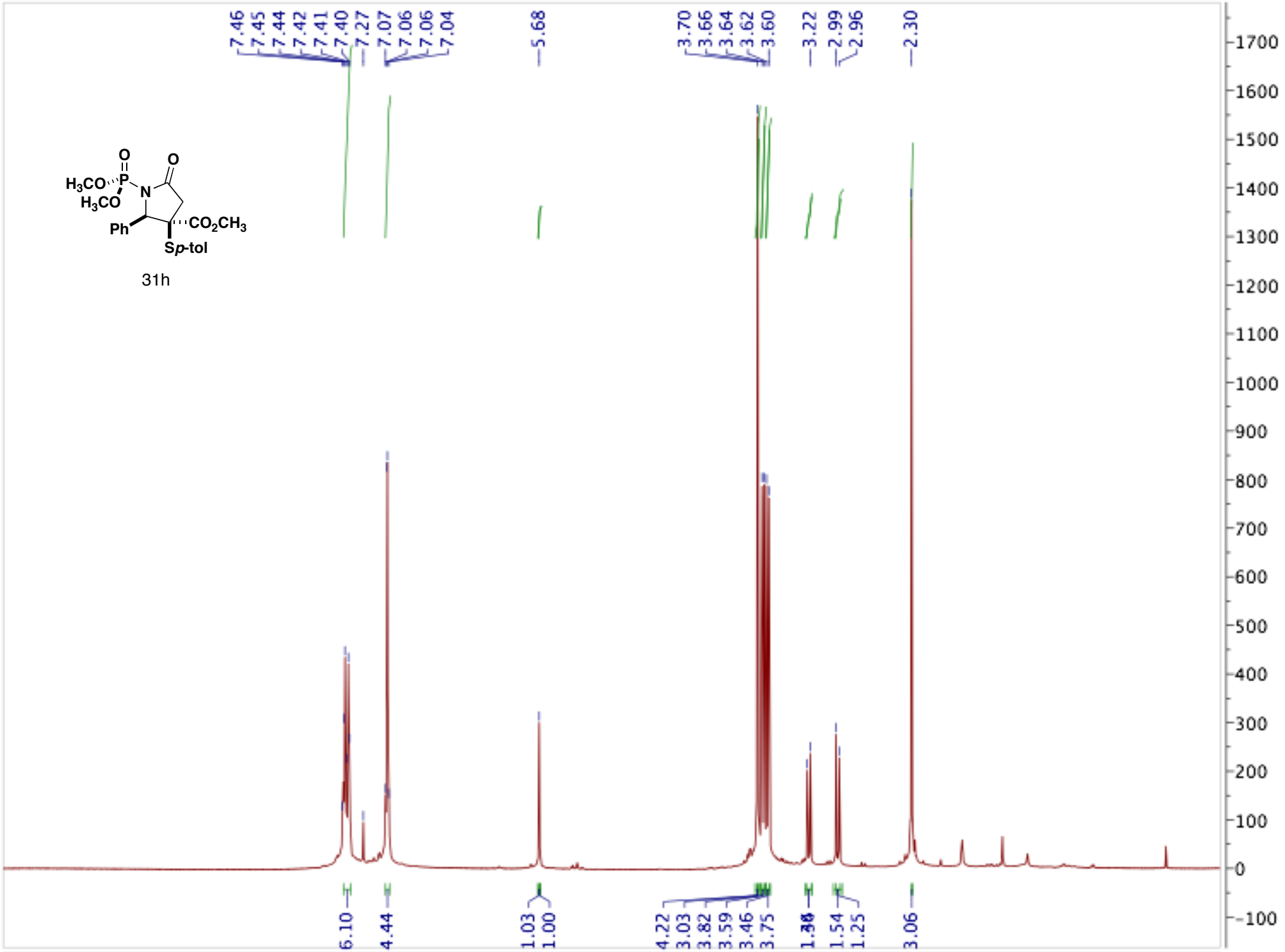
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3.75
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1.25
3.06
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1600
1500
1400
1300
1200
1100
1000
900
800
700
600
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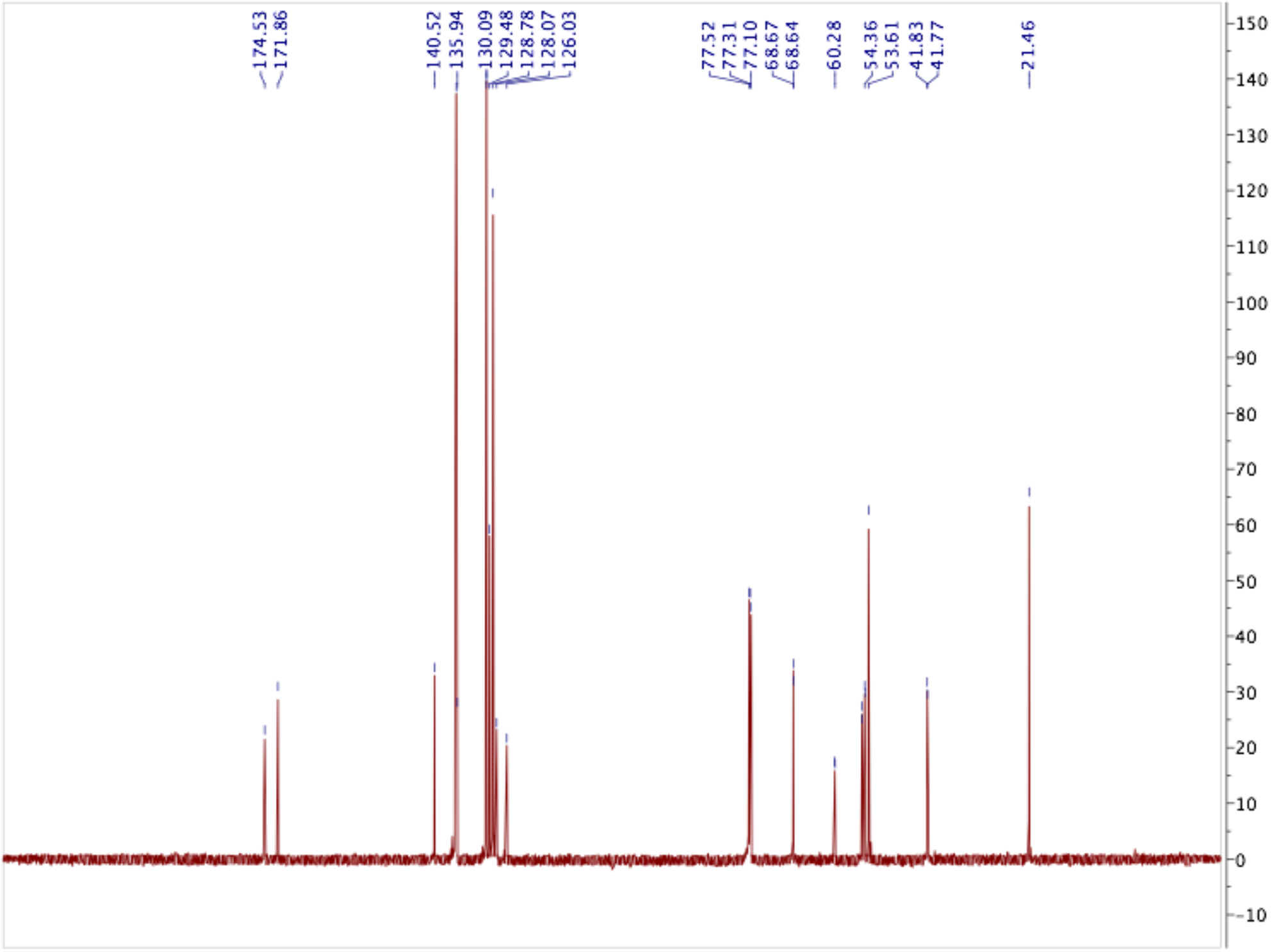
H3CO
Ph
CO2CH3
Sp-tol

31h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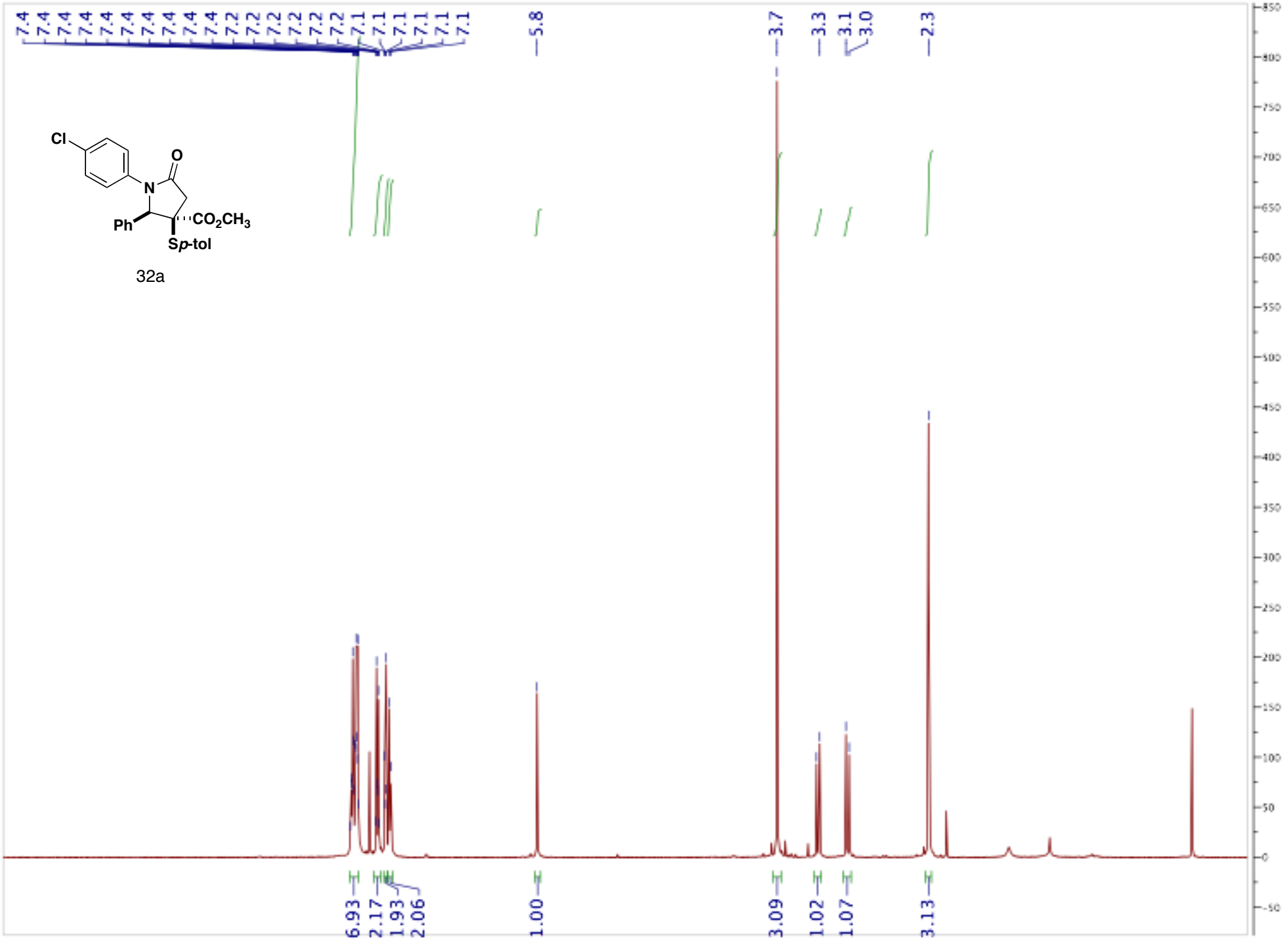
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3.7
3.3
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3.0
2.3
Cl
O
N
Ph
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Sp-tol
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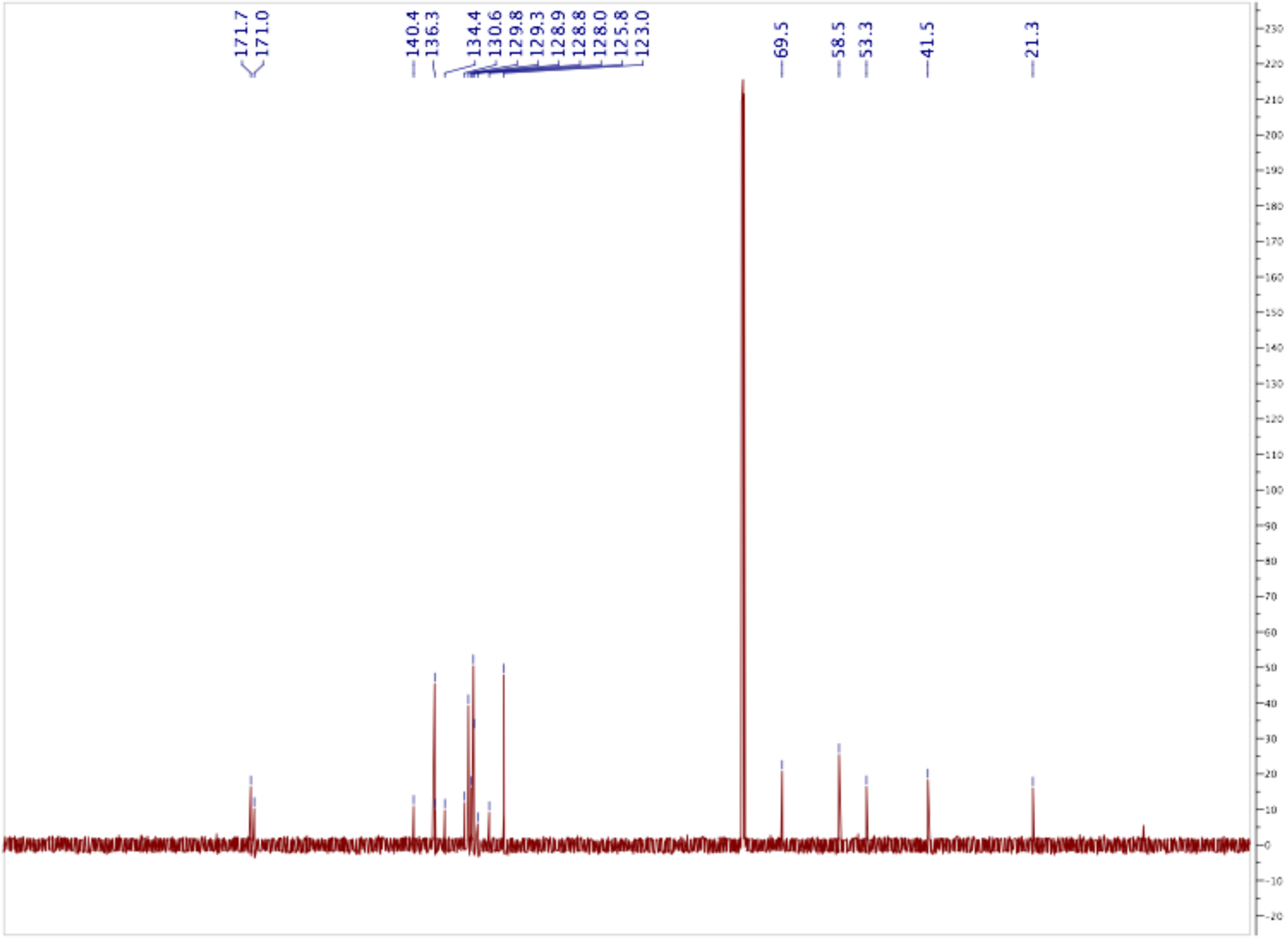
3.09
1.02
1.07
3.13

32a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53.3
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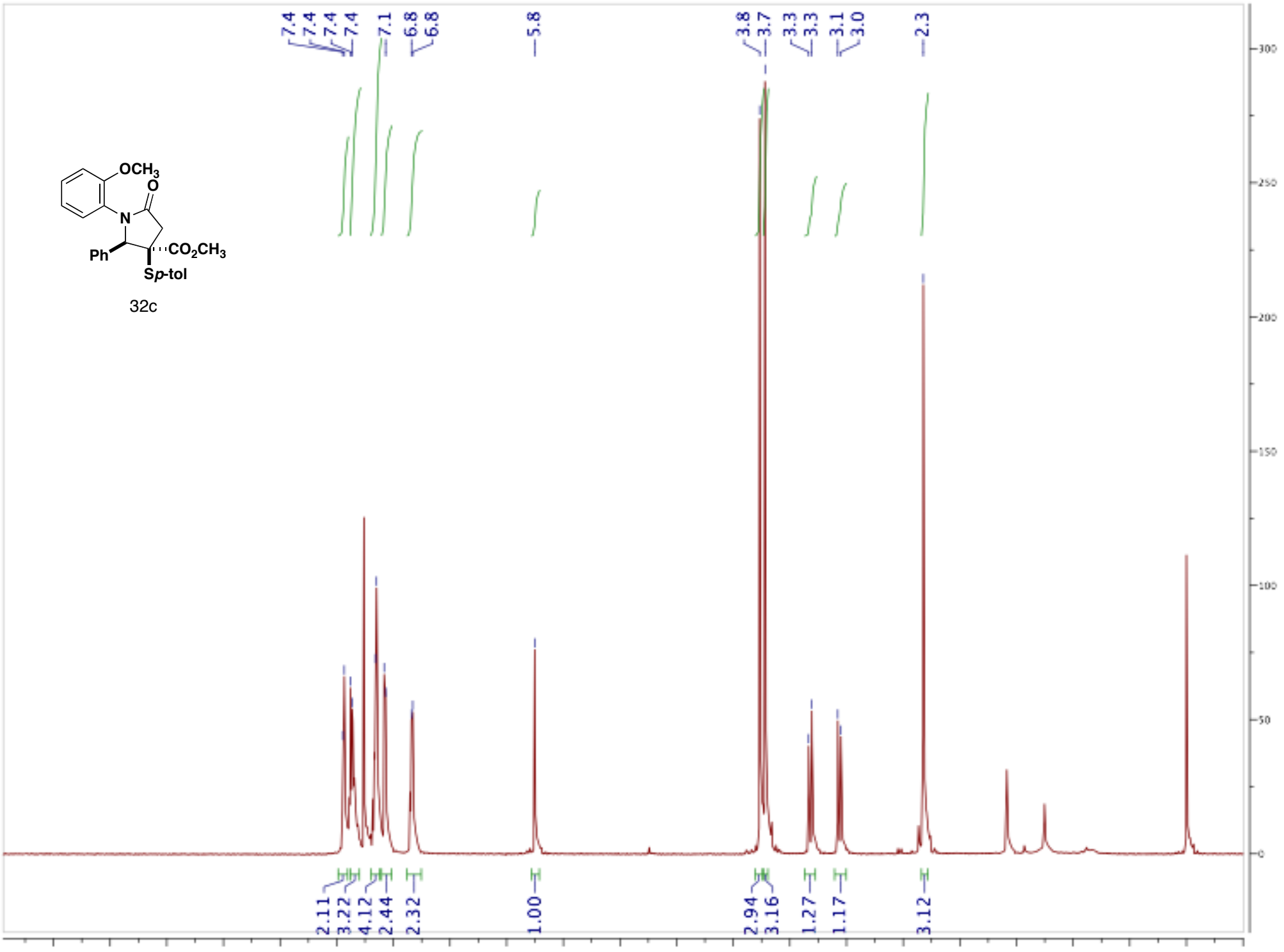
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N
Ph
CO2CH3
Sp-tol
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5.8
3.8
3.7
3.3
3.3
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3.22
4.12
2.44
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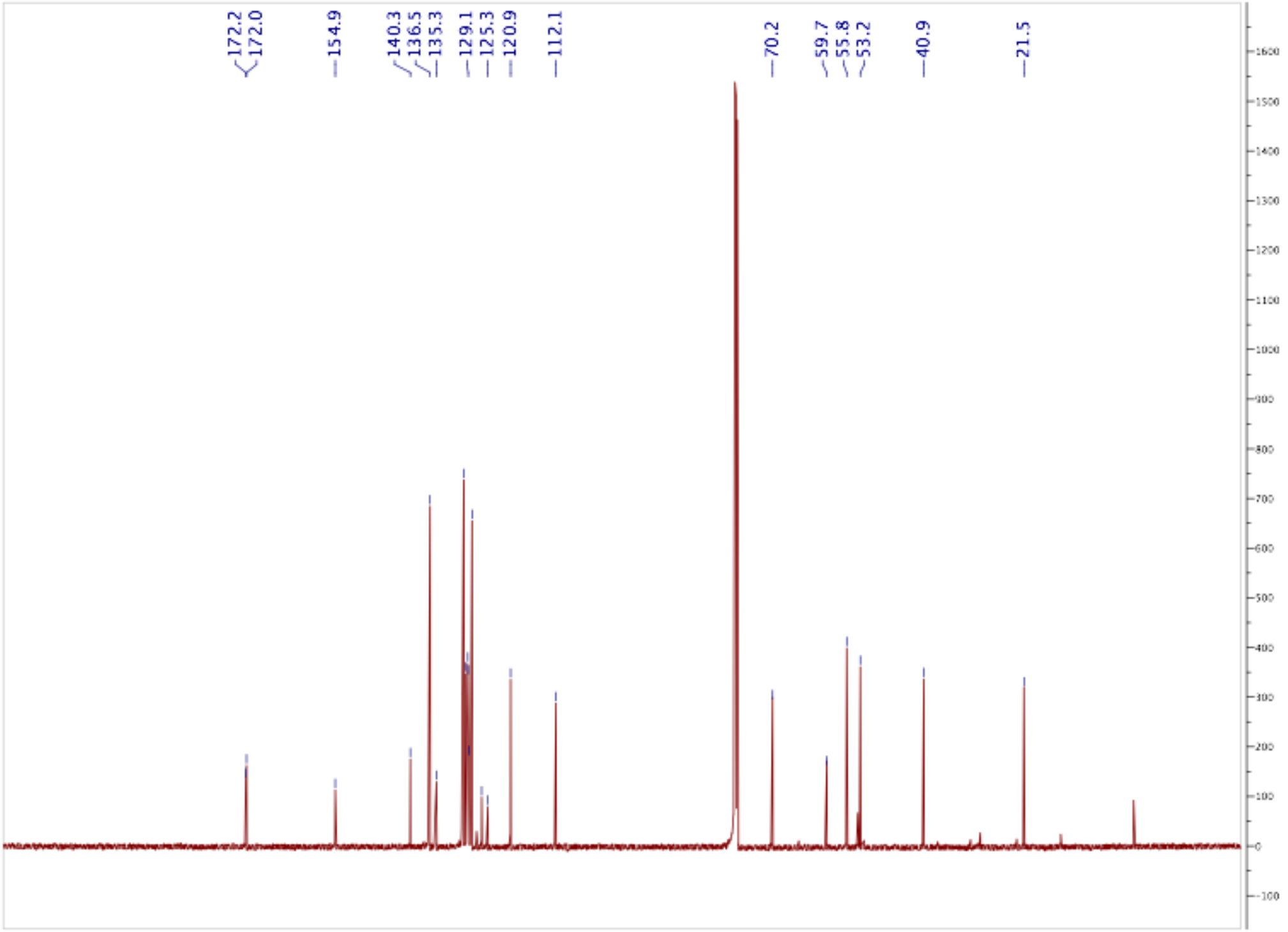
32c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40.9
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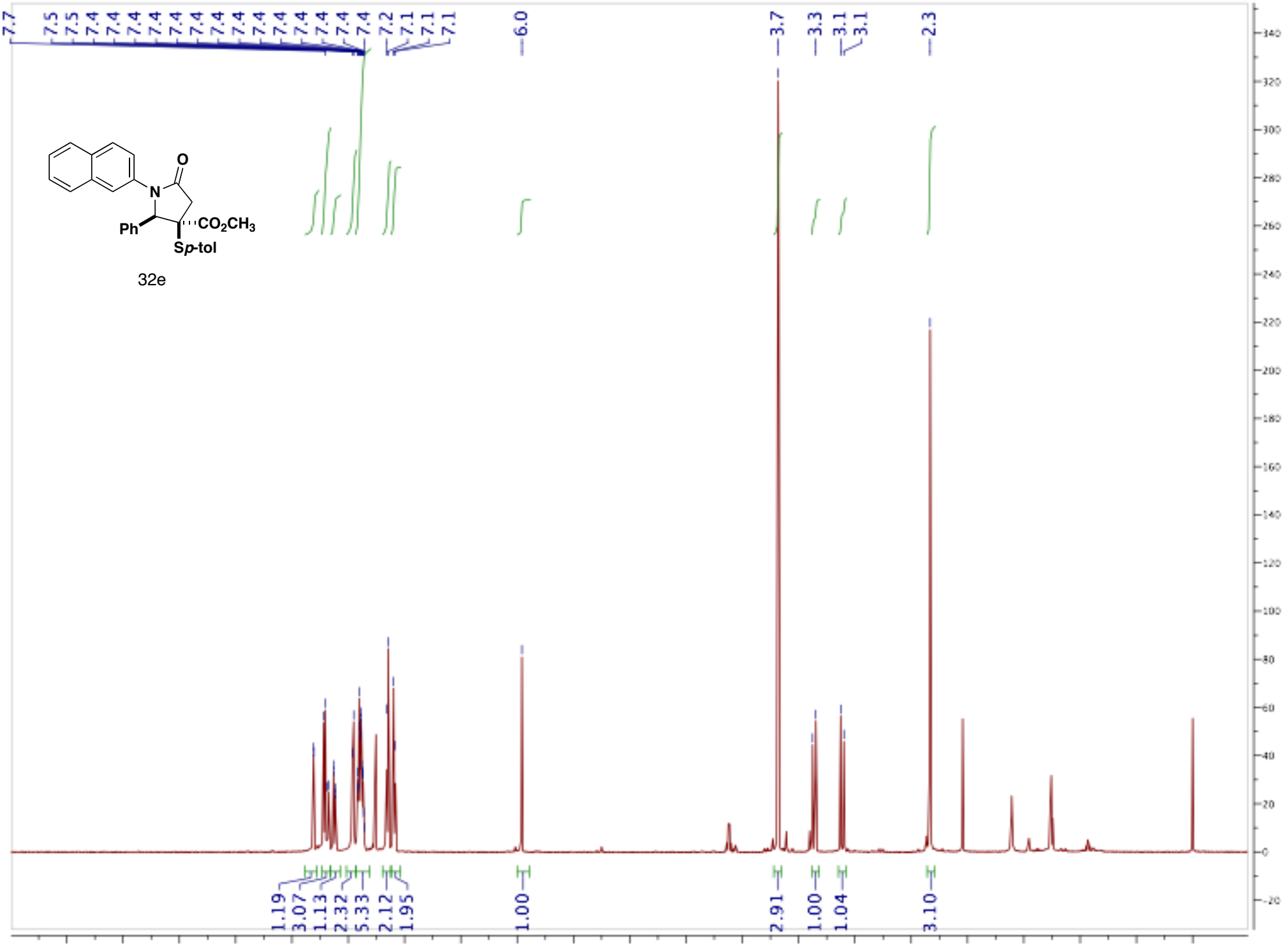
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3.7
3.3
3.1
3.1
2.3
O
N
Ph
CO2CH3
Sp-tol
1.19
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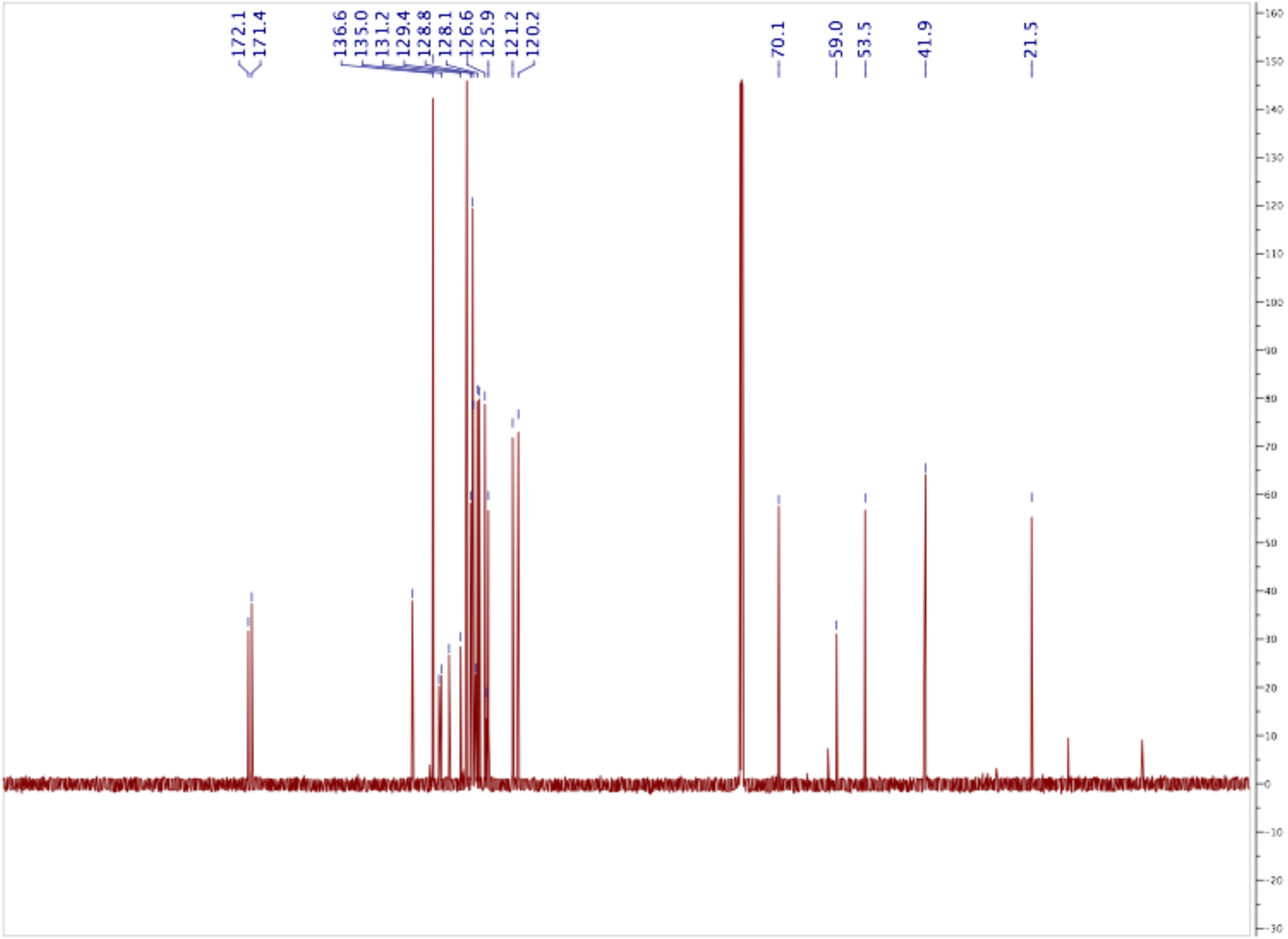
3.10

32e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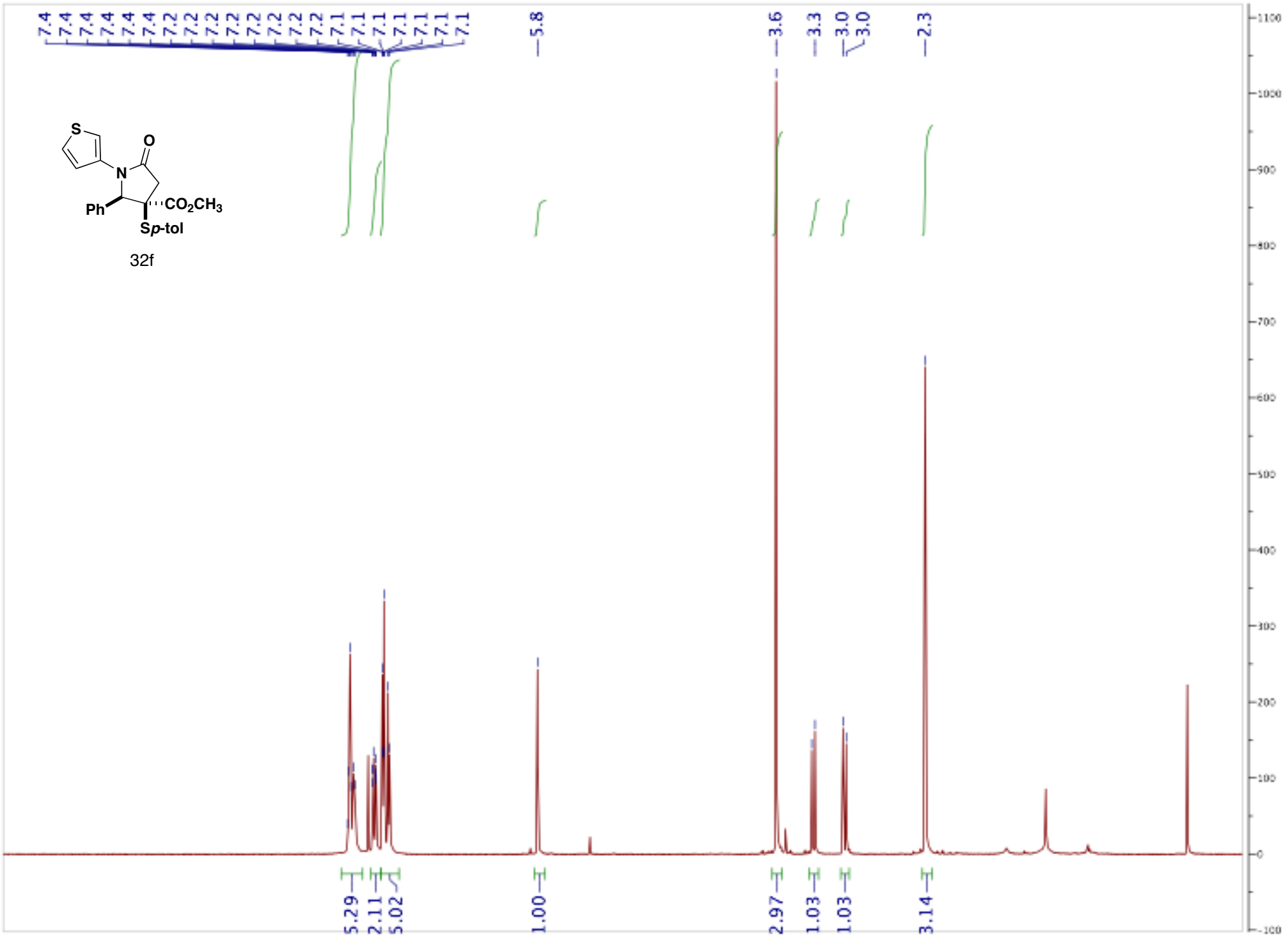
53.5
41.9
21.5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3.0
2.3
S
O
N
Ph
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Sp-tol
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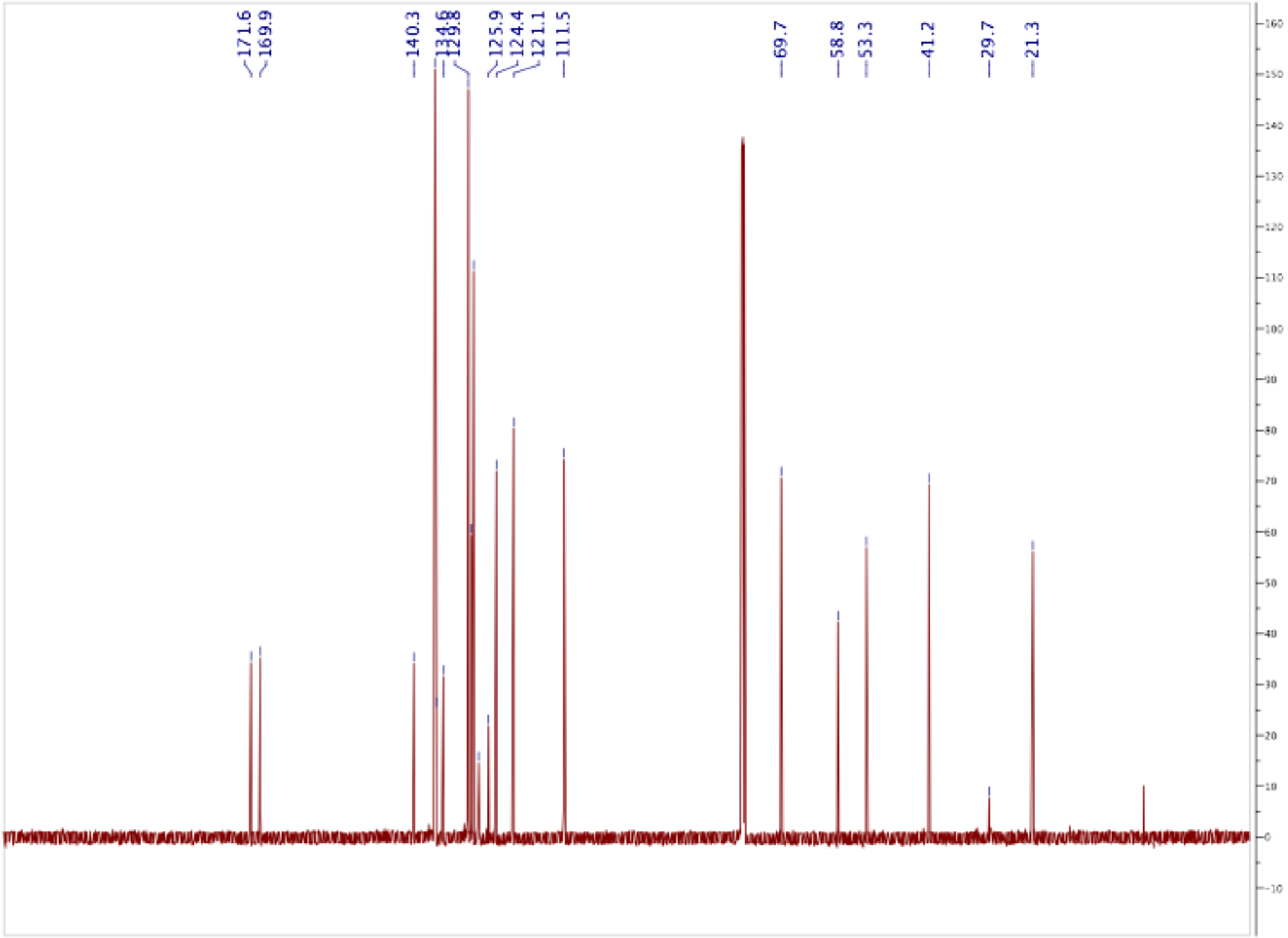
32f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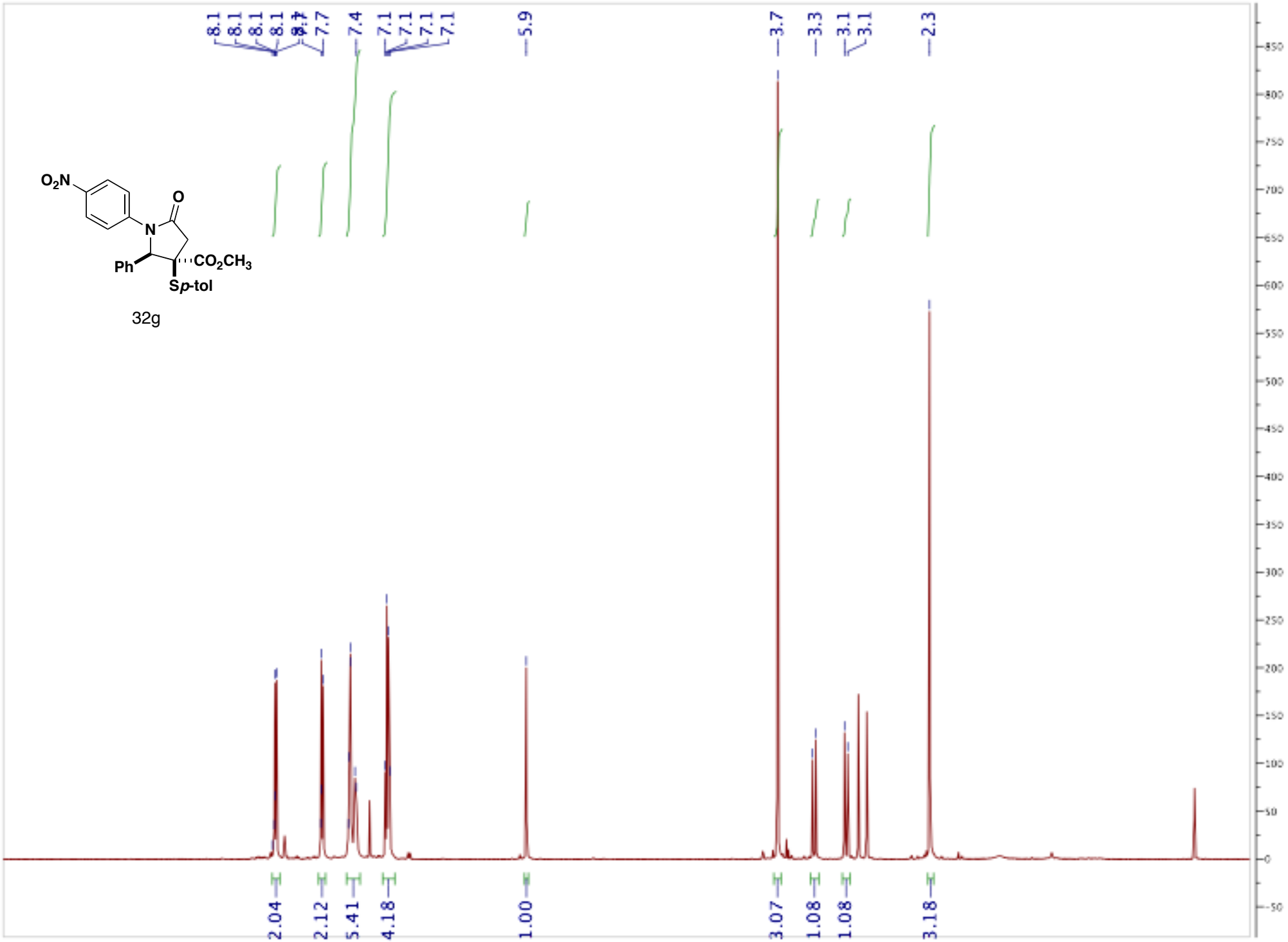
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3.3
3.1
3.1
2.3
O2N
O
N
Ph
CO2CH3
Sp-tol
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2.12
5.41
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3.07
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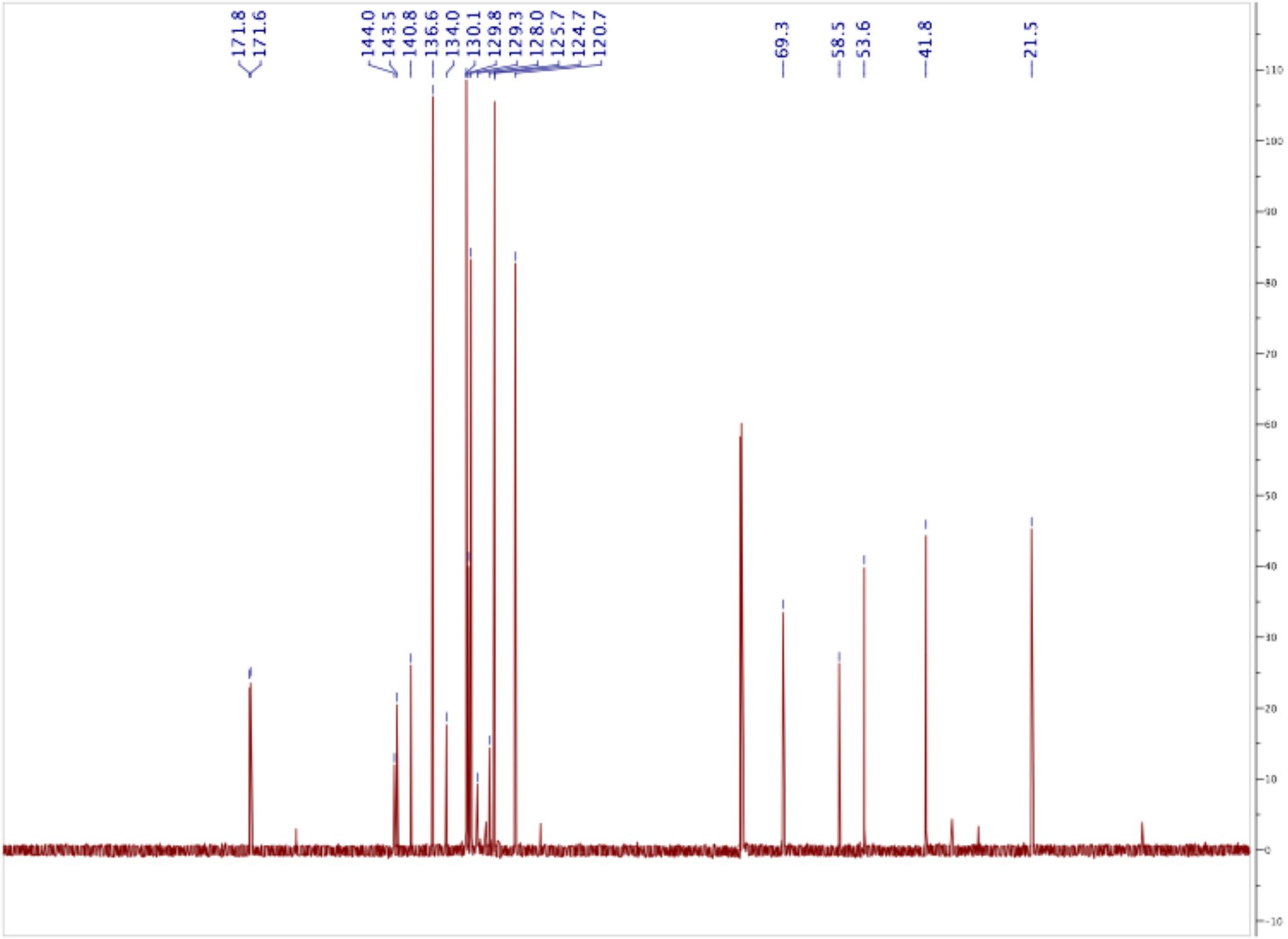
1.08
3.18

32g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60
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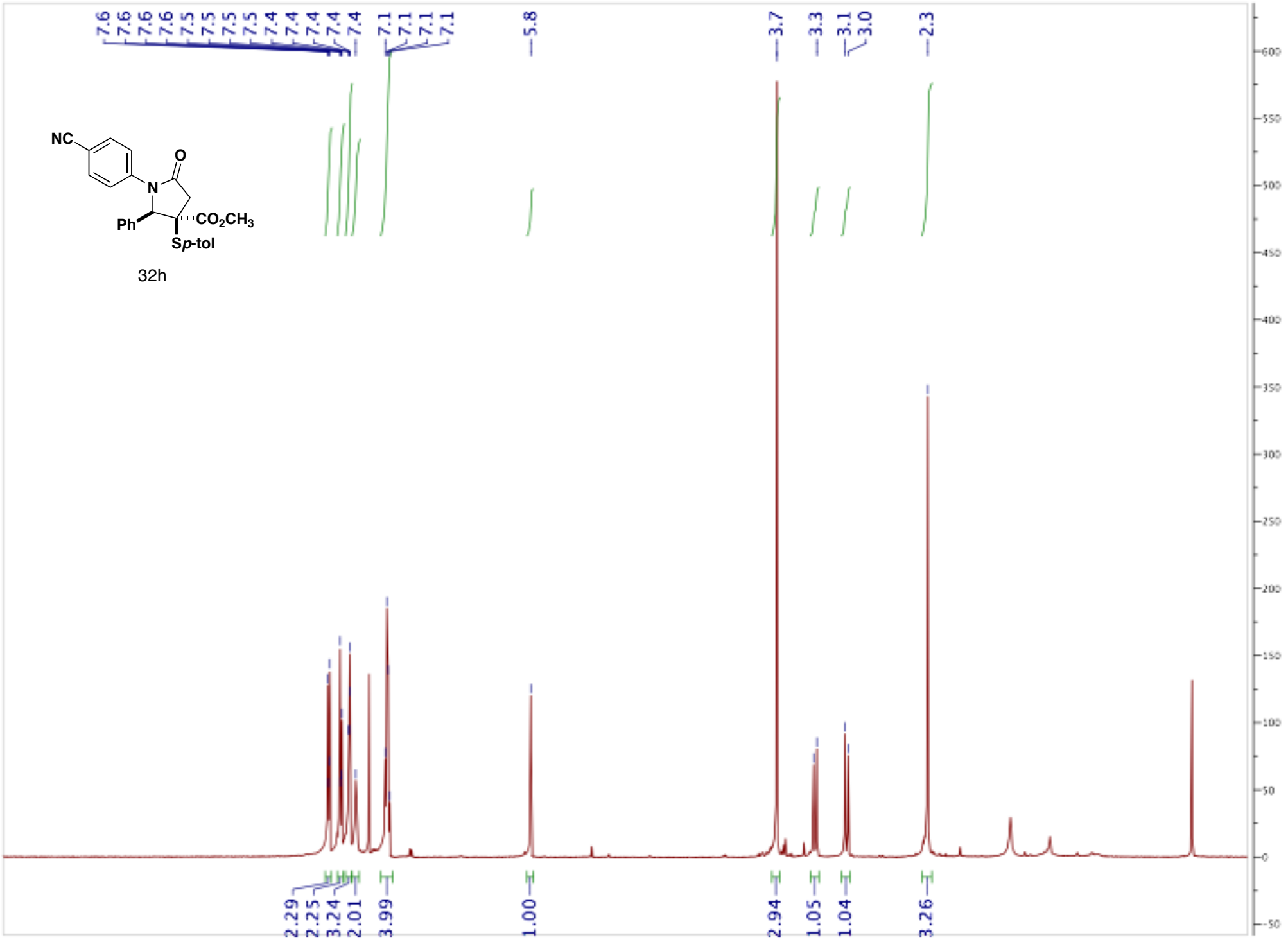
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7.1
7.1
7.1
5.8
3.7
3.3
3.1
3.0
2.3
NC
O
N
Ph
CO2CH3
Sp-tol
32h
2.29
2.25
3.24
2.01
3.99
1.00
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3.26
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50
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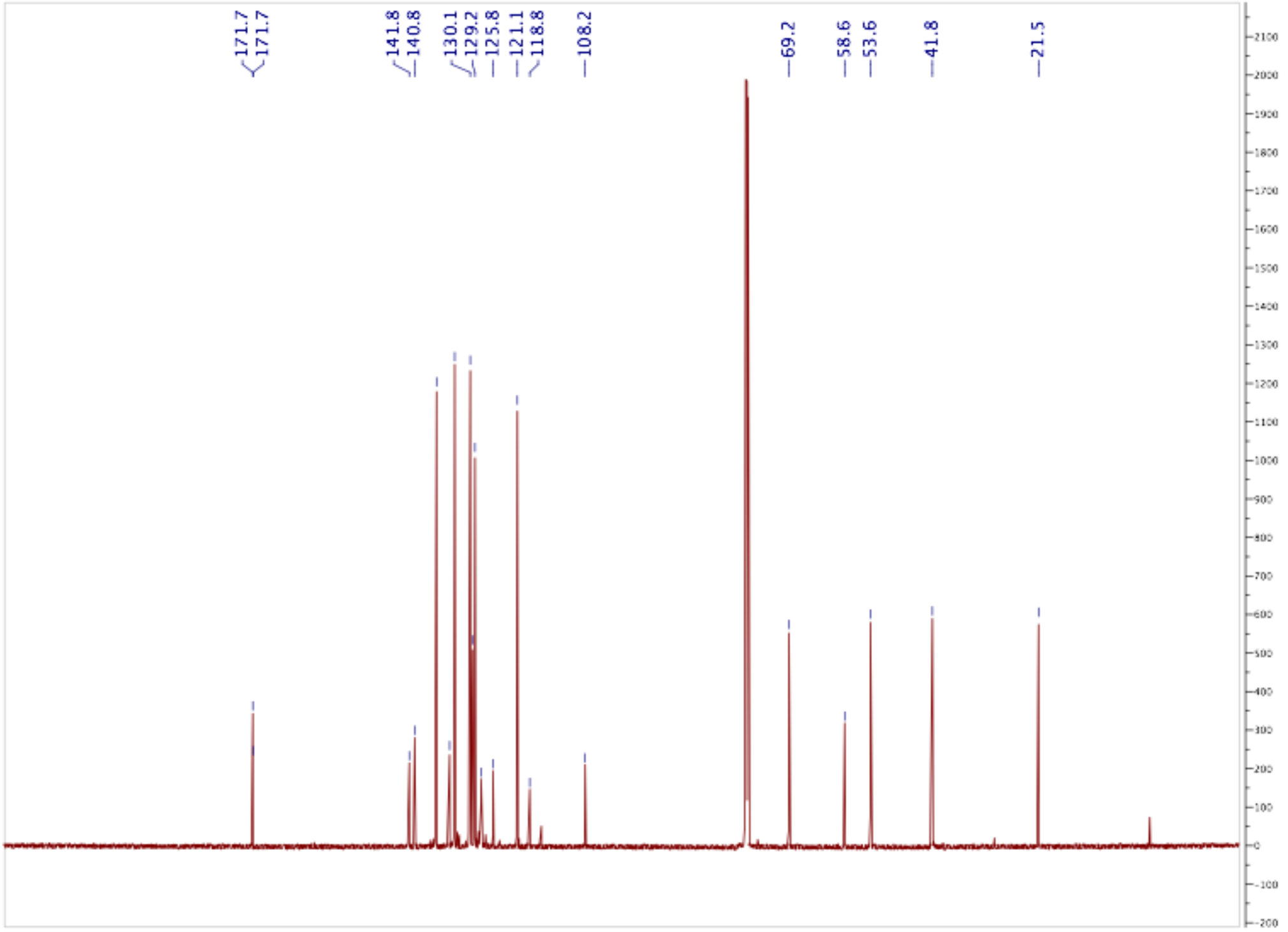
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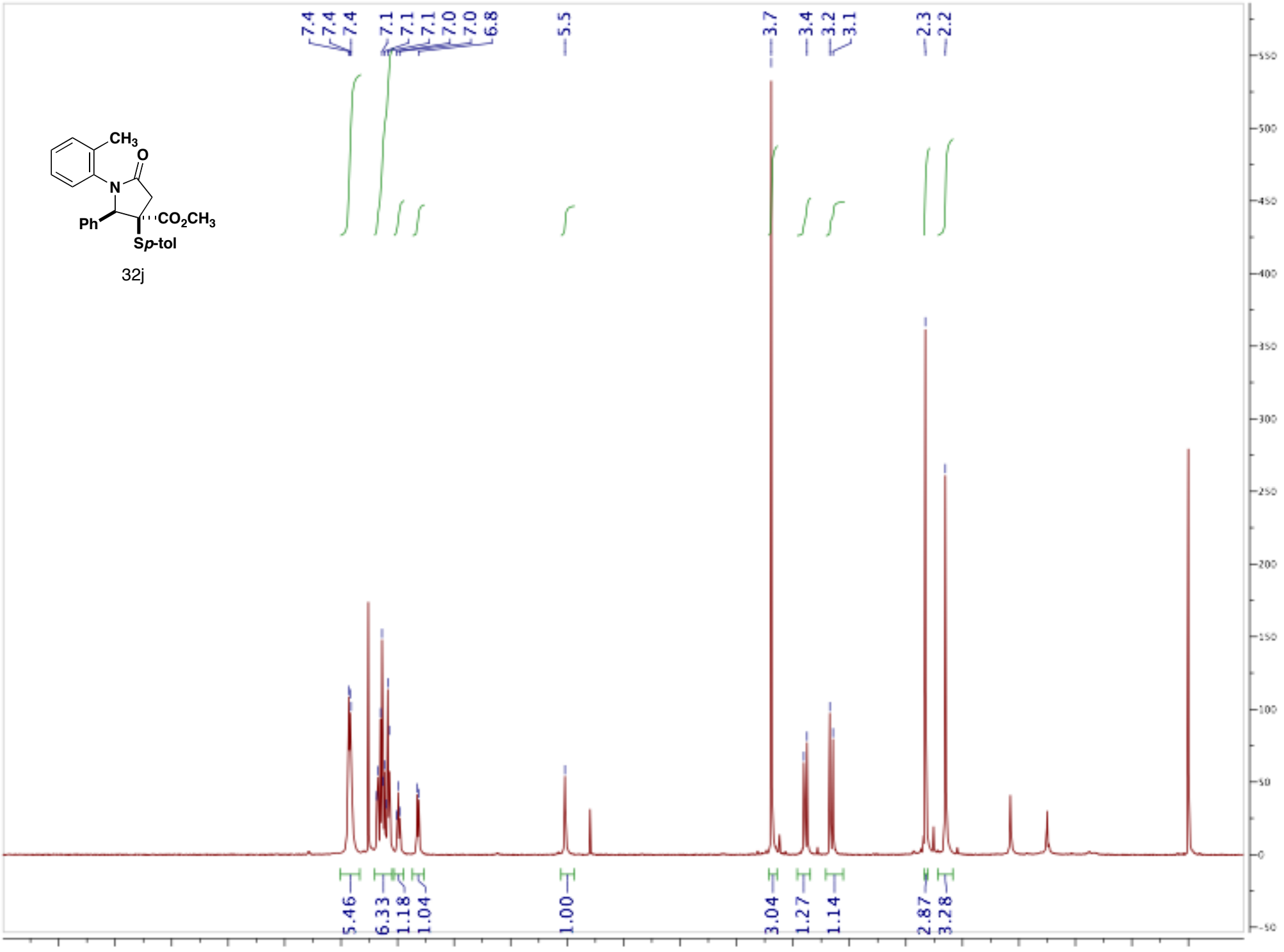
21.5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CH3
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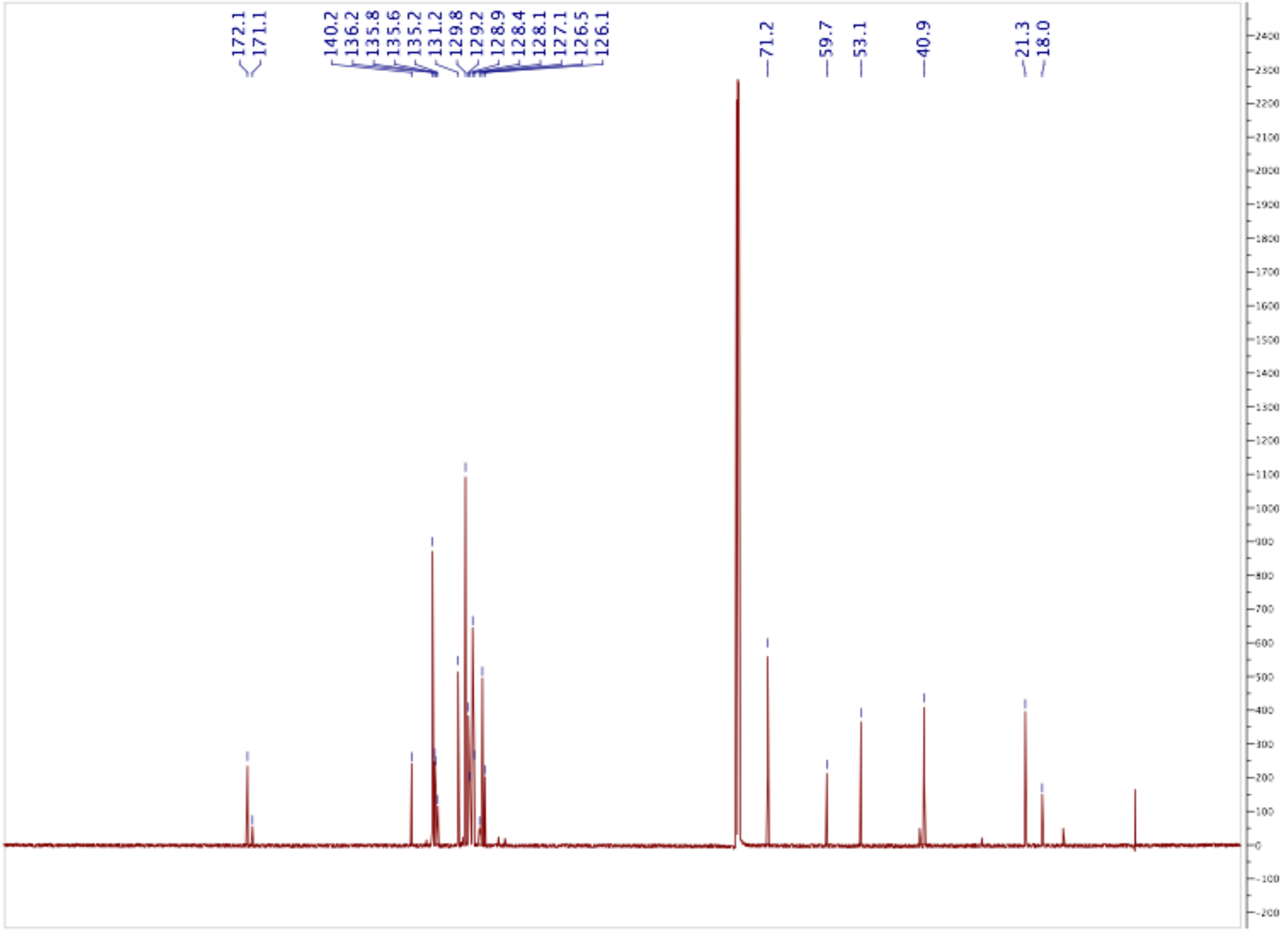
Ph
CO2CH3
Sp-tol

32j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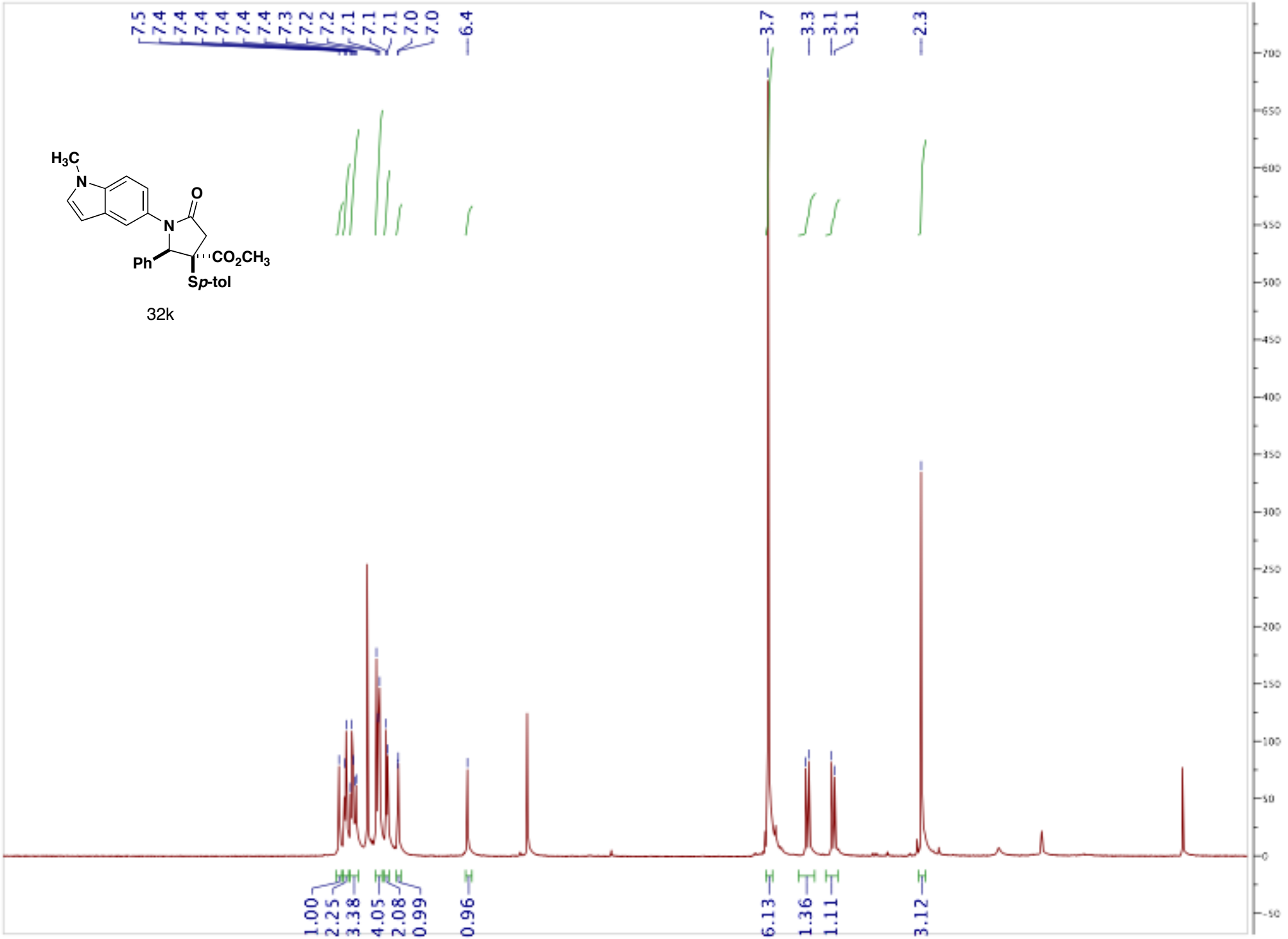
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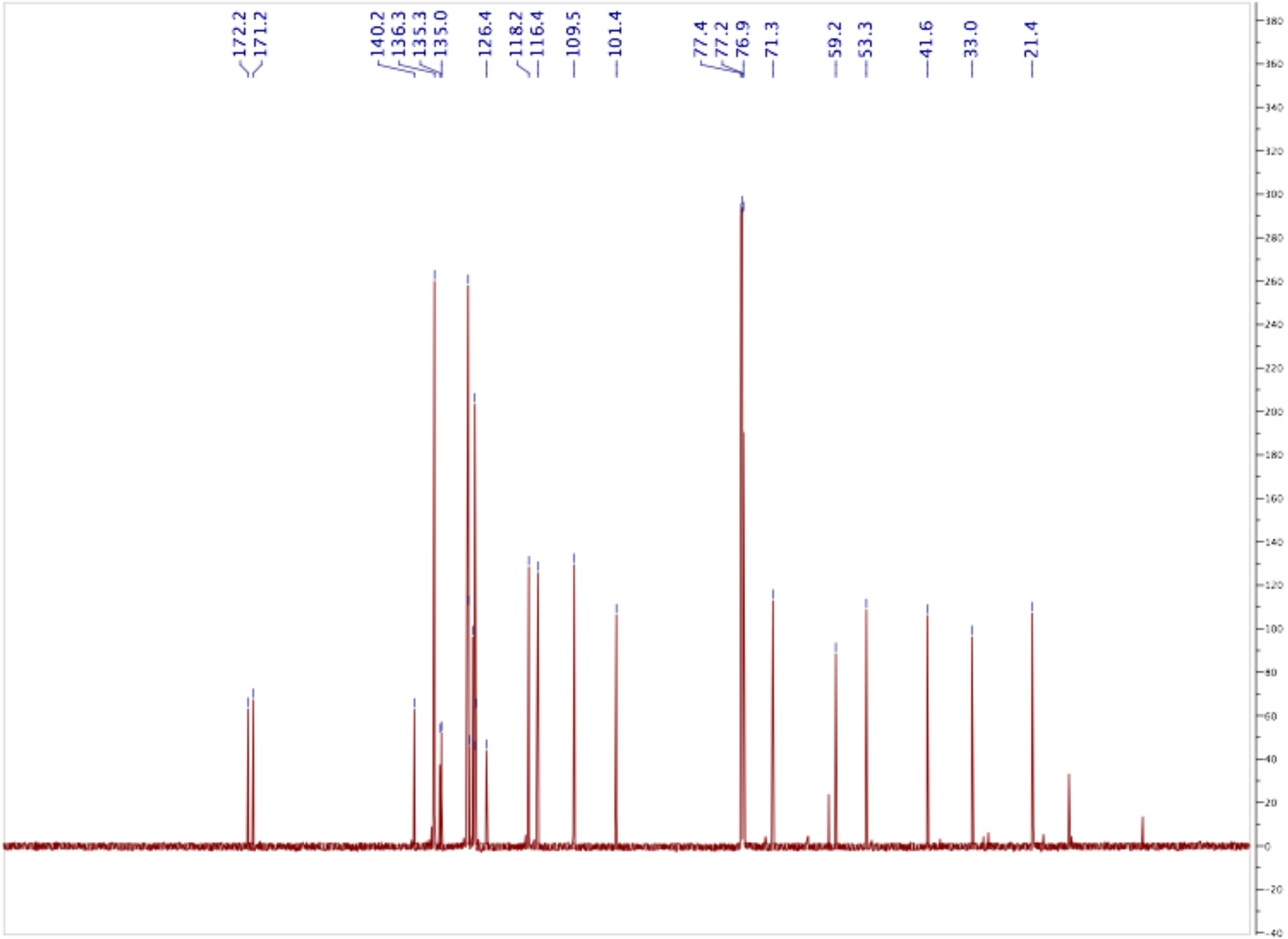
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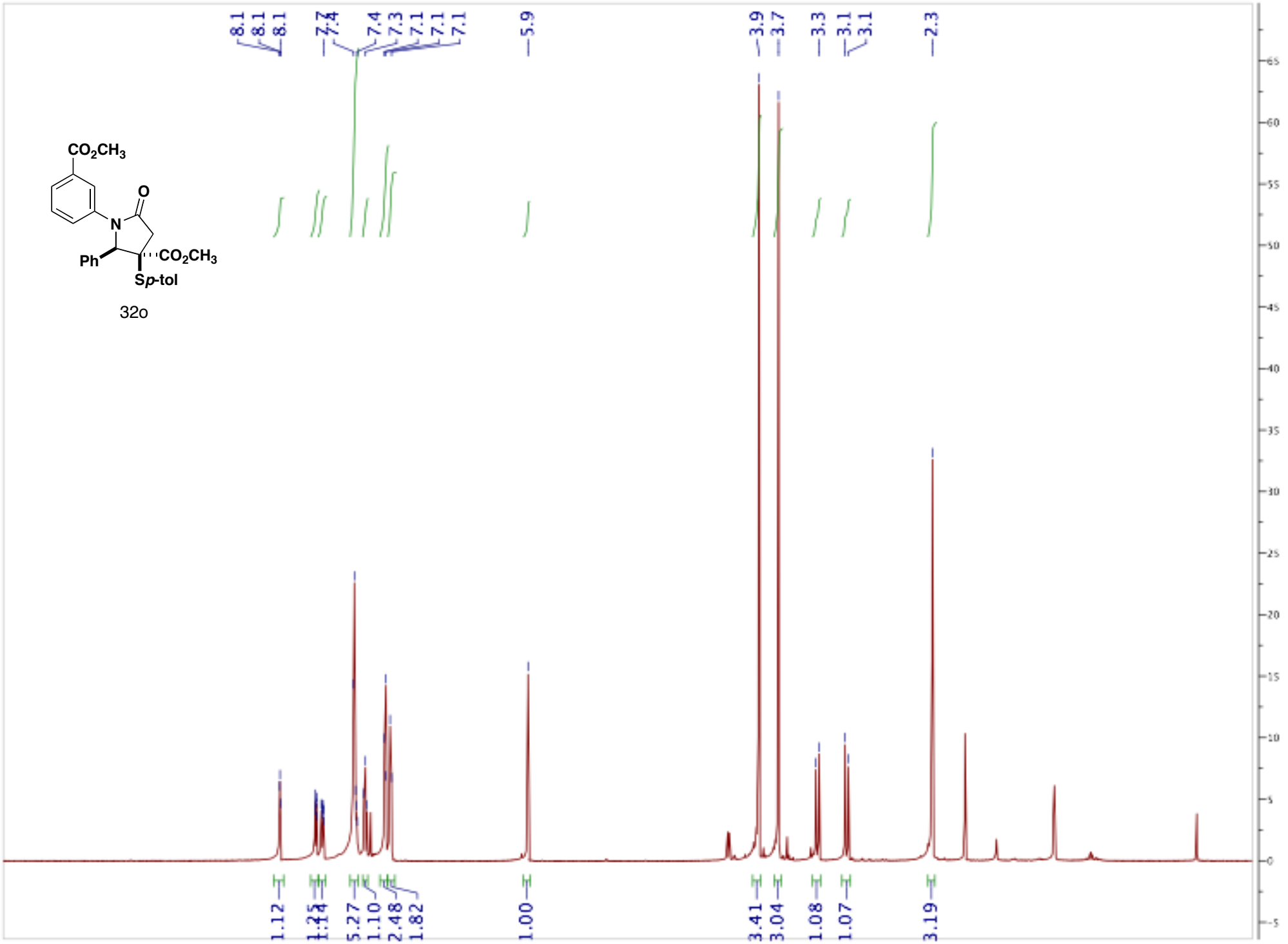
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3.3
3.1
3.1
2.3
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5.27
1.10
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1.00
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1.07
3.19
65
60
55
50
45
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10
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CO2CH3
O
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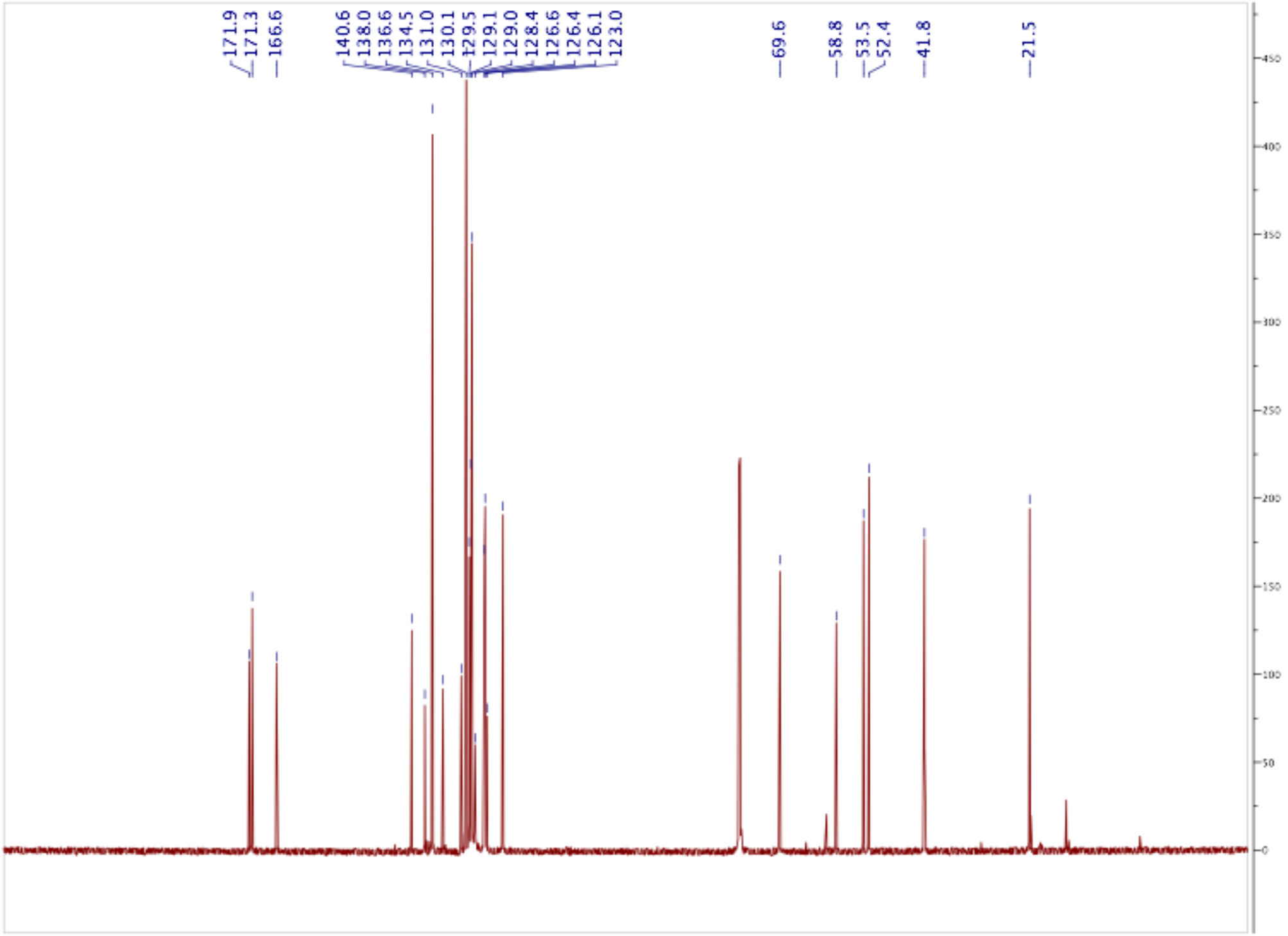
Ph
CO2CH3
Sp-tol

32o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123.0
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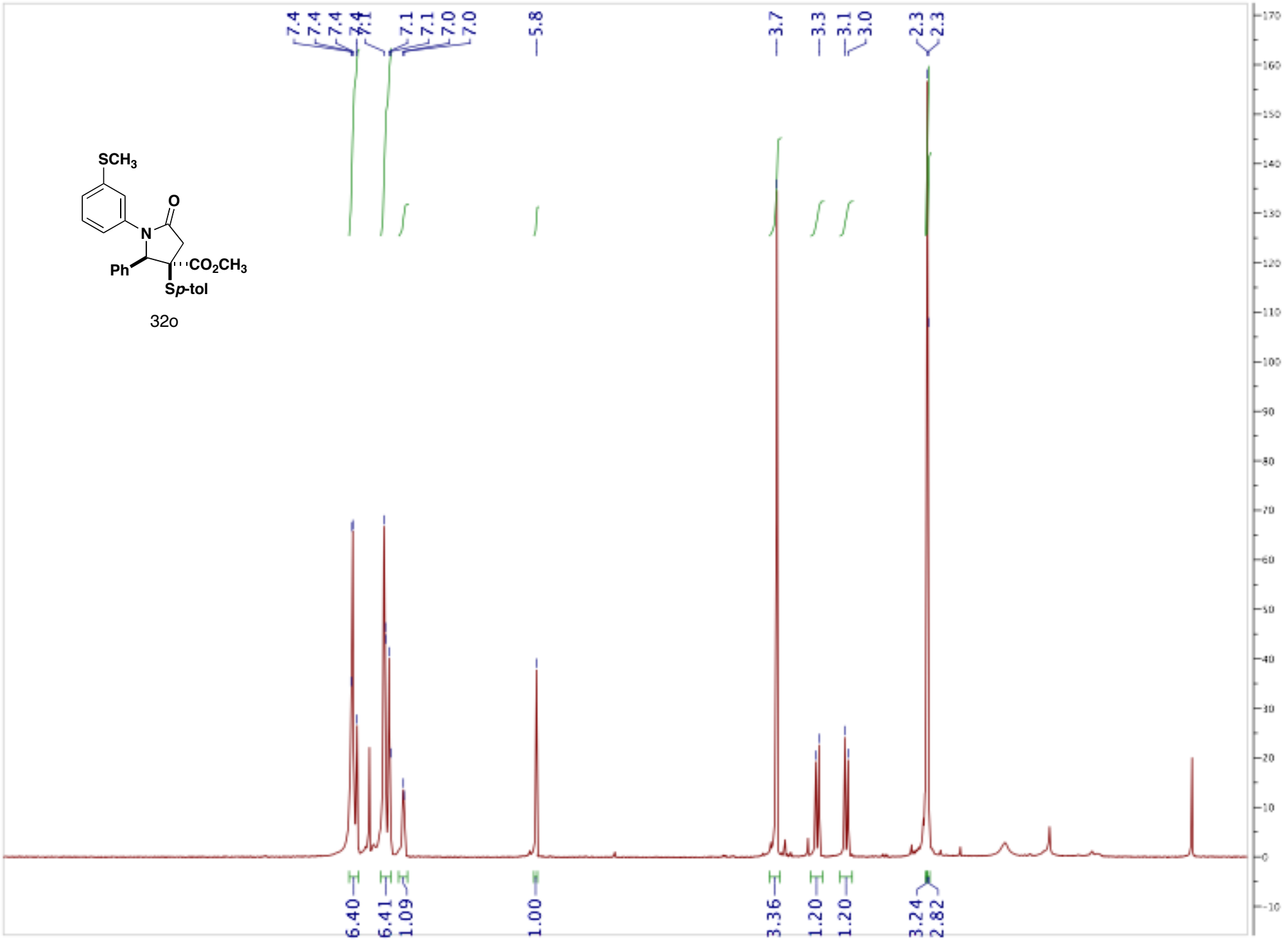
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Ph
CO2CH3
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3.0
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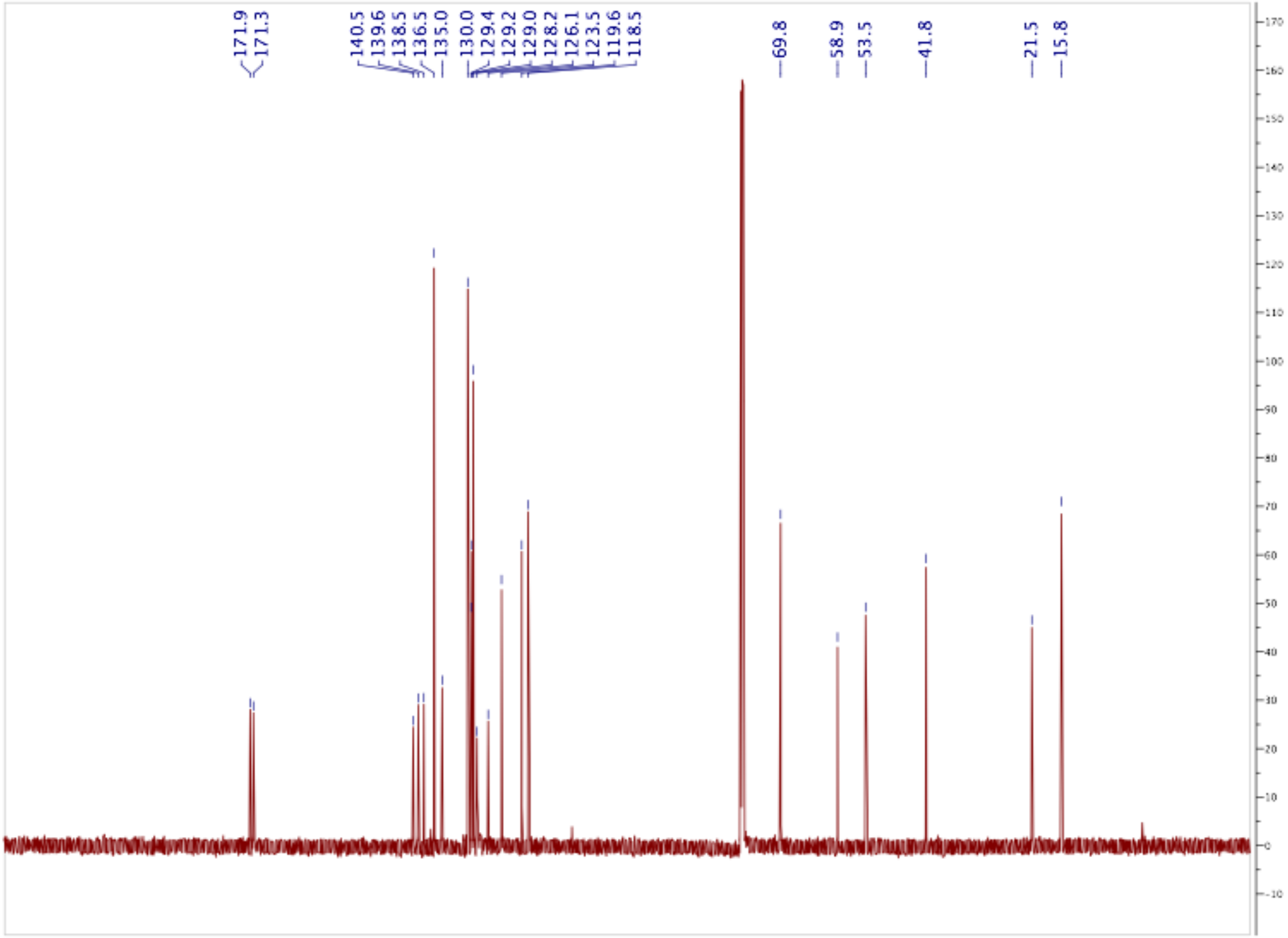
3.24
2.82

32o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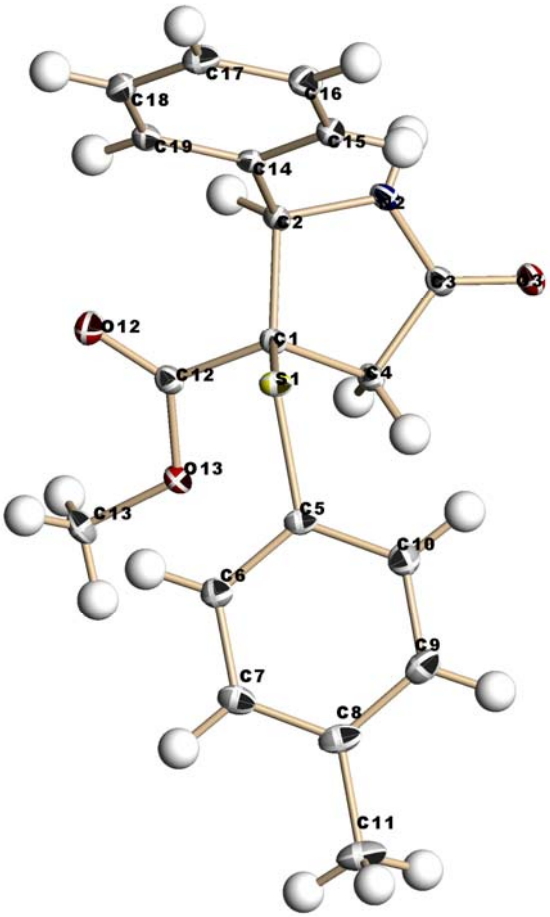
69.8
58.9
53.5
41.8
21.5
15.8

26 October, 2010

JF1965

A colorless block with approximate orthogonal dimensions 0.44 x 0.41 x 0.38mm^3 was placed and optically centered on the Bruker SMART 1000[1] CCD system at 90(2)K. The initial unit cell was indexed using a least-squares analysis of a random set of reflections collected from three series of 0.3° wide ω-scans, 5 seconds per frame, and 30 frames per series that were well distributed in reciprocal space. Five ω-scan data frame series were collected [MoKα] with 0.3° wide scans, 15 seconds per frame and 606 frames were collected, at varying phi angles (phi=0°, 72°, 144°, 216°, 288°), for each series, respectively. The crystal to detector distance was 4.36cm, thus providing a complete sphere of data with processing to $2\theta_{max}$=55.10°.



Structural determination and Refinement:

All crystallographic calculations were performed on an iMac with an Intel Core i7 2.80GHz processor and 8GB of extended memory at 1067MHz DDR3. A total of 13173 reflections were collected and corrected for Lorentz and polarization effects and absorption using crystal faces and Blessing's method as incorporated into the program SADABS[2,3,4] with 3818 unique. The SHELXTL[5] program package was implemented to determine the probable space group and set up the initial files. System symmetry, lack of systematic absences and intensity statistics indicated the centrosymmetric triclinic space group P-1 (no. 2). The structure was determined by direct methods with the successful location of the entire molecule using the program XS[6]. The structure was refined with XL[6]. The data collected were merged, based upon identical indices, to 7586 [R(int)=0.0141] and then truncated to $2\theta_{max}$=55.0° resulting in 7582 data that were further merged in least-squares refinement to 3815 unique data [R(int)=0.0100]. All non-hydrogen atoms were refined anisotropically. All hydrogen atoms were located directly from a difference-Fourier map and allowed to refine freely throughout the refinement process (xyzU). The final structure was refined to convergence with R(F)=3.10%, wR(F^2)=7.73%, GOF=1.029 for all 3815 unique reflections [R(F)=2.88%, wR(F^2)=7.55% for those 3576 data with Fo > 4σ(Fo)]. The final difference-Fourier map was featureless indicating that the structure is both correct and complete. An empirical correction for extinction was also attempted but found to be only 2 sigma and therefore not applied.

References:

1. Bruker (2004) SMART (Version 5.054) and SAINT 2009 (Version 7.68a). Bruker AXS Inc., Madison, Wisconsin, USA.

2. An Empirical Correction for Absorption Anisotropy, Blessing, R. H. (1995). Acta Cryst., A51, 33-38.

3. Sheldrick, G.M., SADABS (2008) Version 2008/2, 'Siemens Area Detector Absorption Correction' Universität Göttingen: Göttingen, Germany.

4. A short history of SHELX, George M. Sheldrick, Acta Cryst. (2008). A64, 112-122

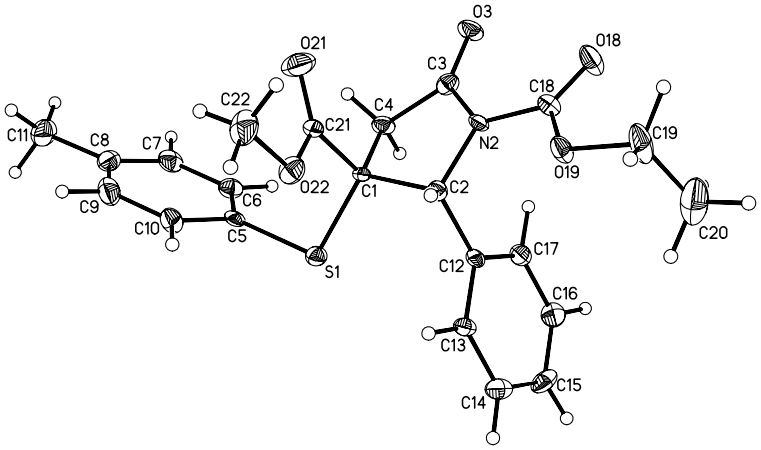
5. Sheldrick, G.M., (2002). SHELXTL. Version 6.1. Bruker AXS Inc., Madison, Wisconsin, USA.

6. Sheldrick, G. M., (1997). SHELXS97 and SHELXL97. Universität Göttingen: Göttingen, Germany.

29 October, 2010

JF1968

A colorless block with approximate orthogonal dimensions 0.48 x 0.25 x 0.19mm^3 was placed and optically centered on the Bruker SMART 1000[1] CCD system at 90(2)K. The initial unit cell was indexed using a least-squares analysis of a random set of reflections collected from three series of 0.3° wide ω-scans, 10 seconds per frame, and 30 frames per series that were well distributed in reciprocal space. Five ω-scan data frame series were collected [MoKα] with 0.3° wide scans, 20 seconds per frame and 606 frames were collected, at varying phi angles (phi=0°, 72°, 144°, 216°, 288°), for each series, respectively. The crystal to detector distance was 4.353cm, thus providing a complete sphere of data with processing to $2\theta_{max}$=55.05°.



Structural determination and Refinement:

All crystallographic calculations were performed on an iMac with an Intel Core i7 2.80GHz processor and 8GB of extended memory at 1067MHz DDR3. A total of 16596 reflections were collected and corrected for Lorentz and polarization effects and absorption using crystal faces and Blessing's method as incorporated into the program SADABS[2,3,4] with 4791 unique. The SHELXTL[5] program package was implemented to determine the probable space group and set up the initial files. System symmetry, lack of systematic absences and intensity statistics indicated the centrosymmetric triclinic space group P-1 (no. 2). The structure was determined by direct methods with the successful location of the molecule using the program XS[6]. The structure was refined with XL[6]. The data collected were and merged, based upon identical indices, to 9526 [R(int)=0.0159] and then further merged in least-squares refinement to 4791 unique data [R(int)=0.0113]. All non-hydrogen atoms were refined anisotropically. All hydrogen atoms were initially idealized and then allowed to refine freely throughout the final refinement process. The final structure was refined to convergence with R(F)=3.41%, wR(F^2)=8.11%, GOF=1.044 for all 4791 unique reflections [R(F)=3.03%, wR(F^2)=7.71% for those 4358 data with Fo > 4σ(Fo)]. The final difference-Fourier map was featureless indicating that the structure is both correct and complete. An empirical correction for extinction was also attempted but found to be negative and therefore not applied.

References:

1. Bruker (2004) SMART (Version 5.054) and SAINT 2009 (Version 7.68a). Bruker AXS Inc., Madison, Wisconsin, USA.

2. An Empirical Correction for Absorption Anisotropy, Blessing, R. H. (1995). Acta Cryst., A51, 33-38.

3. Sheldrick, G.M., SADABS (2008) Version 2008/2, 'Siemens Area Detector Absorption Correction' Universität Göttingen: Göttingen, Germany.

4. A short history of SHELX, George M. Sheldrick, Acta Cryst. (2008). A64, 112-122

5. Sheldrick, G.M., (2002). SHELXTL. Version 6.1. Bruker AXS Inc., Madison, Wisconsin, USA.

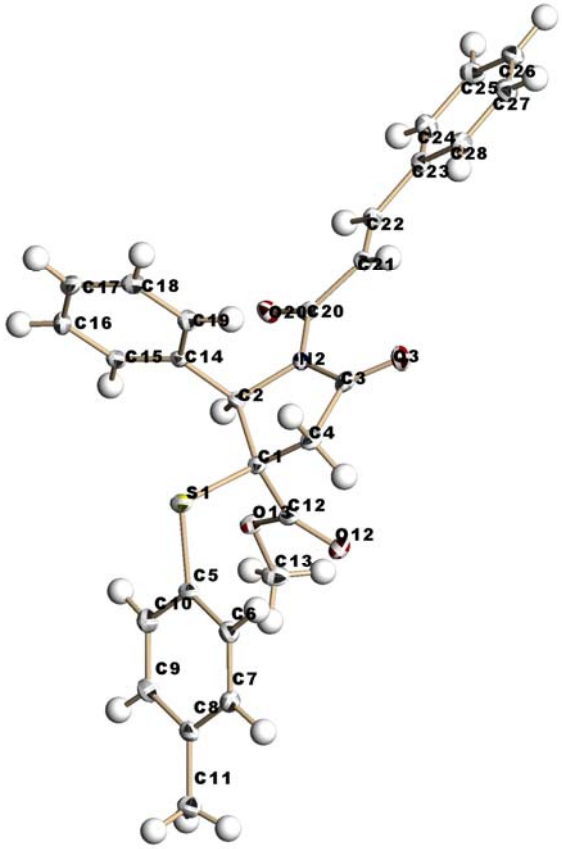
6. Sheldrick, G. M., (1997). SHELXS97 and SHELXL97. Universität Göttingen: Göttingen, Germany.

5 November, 2010

JF1973 (SADABS TwinRotMat TWIN)

A colorless plate with approximate orthogonal dimensions 0.32 x 0.15 x 0.08mm^3 was placed and optically centered on the Bruker SMART1000[1] CCD diffractometer at –183°C. The initial unit cell was indexed using a least-squares analysis of a random set of reflections collected from three series of 0.3° wide ω-scans, 10 seconds per frame, and 30 frames per series that were well distributed in reciprocal space. Four ω-scan data frame series were collected [MoKα] with 0.3° wide scans, 55 seconds per frame and 606 frames collected per series at varying φ angles (φ=0°, 90°, 180°, 270°).

Structural determination and Refinement:



The crystal to detector distance was 4.36cm, thus providing a complete sphere of data to $2\theta_{max}$=55.30°. A total of 28929 reflections were collected and corrected for Lorentz and polarization effects and absorption using Blessing's method as incorporated into the program SADABS[2,3,6] with 5671 unique. All crystallographic calculations were performed on a Personal computer (PC) with a Pentium 3.20GHz processor and 4GB of extended memory. The SHELXTL[4] program package was implemented to determine the probable space group and set up the initial files. System symmetry, systematic absences and intensity statistics indicated the centrosymmetric monoclinic space group $P2_1/c$ (no. 14). The structure was determined by direct methods with the successful location of a majority of the molecule within the asymmetric unit using the program XS[5]. The structure was refined with XL[5]. The 28929 data collected were merged based upon identical indices yielding 20630 data [R(int)=0.0247] that were truncated to $2\theta_{max}$=55.0° and further merged during least-squares refinement to 5272 unique data [R(int)=0.000]. A single least-squares difference-Fourier cycle was required to locate the remaining non-hydrogen atoms. All non-hydrogen atoms were refined anisotropically. All hydrogen atoms were initially idealized but then allowed to refine freely but for the methyl group C(11) that had its hydrogen atoms partially idealized. Rotax[7] suggested minor twinning of 180.0 degree rotation about the 1. 0. 0. reciprocal lattice direction with a twin matrix of: [1 0 0.126 0 -1 0 0 0 -1]. This matrix was also found and used in Platon's TwinRotMat[8] so an HKLF5 type file was generated and the resulting BASF parameter was determined to be 0.048. The final structure was refined to convergence

with R(F)=6.10%, wR(F^2)=12.17%, GOF=1.199 for all 5272 unique reflections [R(F)=5.02%, wR(F^2)=11.89% for those 4482 data with Fo > 4σ(Fo)]. The final difference-Fourier map was featureless indicating that the structure is both correct and complete.

References:

1. Bruker (2002) SMART (Version 5.054) and (2009) SAINT (Version 7.68A). Bruker AXS Inc., Madison, Wisconsin, USA.

2. An Empirical Correction for Absorption Anisotropy, Blessing, R. H. (1995). Acta Cryst., A51, 33-38.

3. Sheldrick, G.M., SADABS (2008/2), 'Siemens Area Detector Absorption Correction' Universität Göttingen: Göttingen, Germany.

4. Sheldrick, G.M., (2002). SHELXTL. Version 6.1. Bruker AXS Inc., Madison, Wisconsin, USA.

5. Sheldrick, G. M., (1997). SHELXS97 and SHELXL97. Universität Göttingen: Göttingen, Germany.

6. A short history of SHELX, George M. Sheldrick, Acta Cryst. (2008). A64, 112-122.

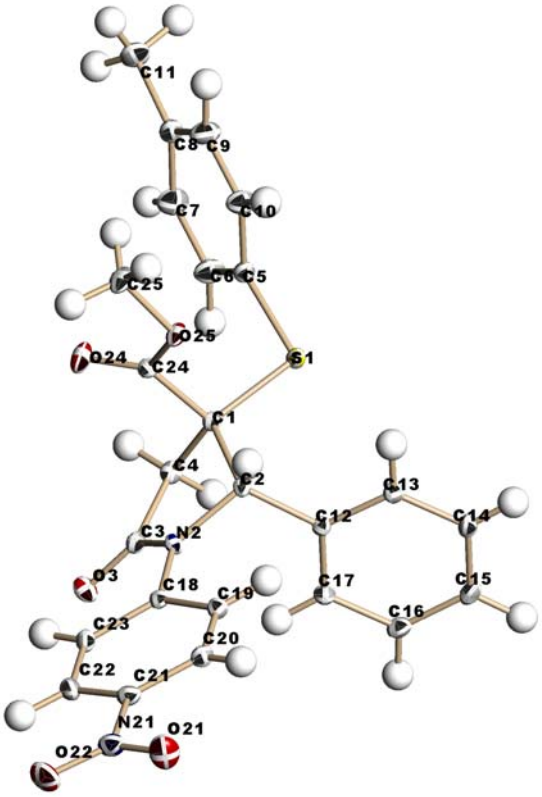
7. ROTAX: The derivation of non-merohedral twin laws during refinement by analysis of poorly fitting intensity data and the refinement of non-merohedrally twinned crystal structures in the program CRYSTALS. R. I. Cooper, R. O. Gould, S. Parsons and D. J. Watkin, J.Appl.Cryst. 2002, 35, 168-174.

8. Spek, A.L., (2009), Acta Cryst. D65, 148-155. PLATON/TwinRotMat

16 December, 2010

JF1980 (Twinabs)

A colorless plate with approximate orthogonal dimensions 0.47 x 0.35 x 0.06mm^3 was placed and optically centered on the Bruker APEX DUO[1] CCD system at –183°C(90K). Indexing of the unit cell was attempted using a random set of reflections collected from three series of 0.3° wide ω-scans, 10 seconds per frame, and 30 frames per series that were well distributed in reciprocal space. Data were collected [MoKα] with 0.3° wide scans, 30 seconds per frame and at varying φ and omega angles such that all unique reflections were collected at least once; overall 100% were collected. The crystal to detector distance was 4.96cm, thus providing a complete sphere of data to $2\theta_{max}$=61.11°.



Structural determination and Refinement:

All crystallographic calculations were performed on a Personal computer (PC) with a Pentium 3.20GHz processor and 4GB of extended memory. Data collected were corrected for Lorentz and polarization effects and absorption using Blessing's method and merged as incorporated into the program Twinabs[2,6]. The SHELXTL[3] program package was now implemented to determine, based upon intensity statistics and systematic absences, the centrosymmetric monoclinic space group C2/c (no.15). The structure was determined by direct methods with all of the non-hydrogen atoms being located directly using the program XS[4]. Refinement of the structure was achieved using the program XL[4]. Refinement converged to approximately R_F=13% for those data observed with an incredibly large weighting scheme. It was evident from the outset that the structure possessed more than a single domain when examining the individual frames so reflections were thresholded in APEX[1] and these reflections were input into Cellnow[5] that determined the twin relationship between the two components and generated the orientation matrices for the components and output a useable multiple matrice input file for the integration program SAINT[1]. Saint was run six times using the output optimized merged matrix file from the previous run. Data collected were now corrected absorption using Blessing's method and merged as incorporated into the program TWINABS[2,6], generating both HKLF4 and HKLF5 files. Convergence of the structure proceeded quickly using the HKLF5 file and all of the non-hydrogen atoms were refined anisotropically with the two domains being present as follows: major component 62.5% and minor 37.5%. Data were truncated to $2\theta_{max}$=60.00°. All of the hydrogen atoms on carbon atoms were initially placed in calculated positions, then allowed to refine freely

throughout the final convergence cycles. The final structure was refined to convergence with R(F)=3.60, wR(F^2)=8.70%, GOF=1.057 for all 6512 reflections [R(F)=3.35%, wR(F^2)=8.58% for those 6138 data with Fo > 4σ(Fo)]. A final difference-Fourier map was featureless indicating that the structure is therefore both correct and complete.

References:

1. Bruker (2010) SMART APEX (2010.9-1) and (2009) SAINT (Version 7.68a). Bruker AXS Inc., Madison, Wisconsin, USA.

2. An Empirical Correction for Absorption Anisotropy, Blessing, R. H. (1995). Acta Cryst., A51, 33-38.

3. Sheldrick, G.M., (2002). SHELXTL. Version 6.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

4. Sheldrick, G. M., (1997). SHELXS97 and SHELXL97. Universität Göttingen: Göttingen, Germany.

5. Sheldrick, G.M., CELLNOW, Twin matrix determination program, Universität Göttingen: Göttingen, Germany, Version 2008/3.

6. Sheldrick, G.M., TWINABS Version 2008/4 'An Empirical Correction for Absorption Anisotropy applied to Twinned crystals'. Universität Göttingen: Göttingen, Germany, 2003.