Supporting information

Electrochemical (Radio)-Halodesilylation of Aromatic Silanes

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1. General

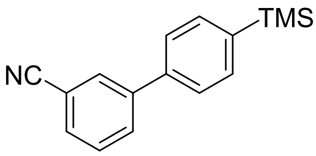
All reactions were carried out under ambient atmosphere without protection. Commercial reagents and solvents were obtained from Adamas-beta®, J&K Scientific®, Shanghai Bidepharm®, Jiangsu Sinocompound®, Shanghai Leyan® and used without further purification. Sodium [^{125}I] iodide was purchased from HTA Co., It is none carrier added [^{125}I] sodium iodide in 1 x 10^{-5} M NaOH. The products were purified using a commercial flash chromatography system or a regular glass column. TLC was developed on silica gel 60 F254 glass plates.

Electrolysis experiments were performed using an IKA ElectraSyn 2.0 purchased from IKA China agent or a DC power supply (Henghui PLD-7505). The platinum electrodes were purchased from Gaossunion, Tianjin. 2B pencil lead and carbon felt were used as the carbon electrodes. The CV experiments were performed using electrochemical analyzer Chenhua CHI760E.

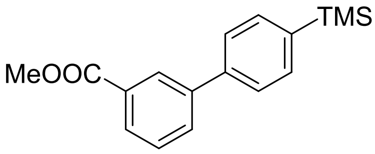
^{1}H NMR (400 MHz or 600 MHz) and ^{13}C NMR (101 MHz or 151 MHz) spectra were recorded on a Bruker NMR apparatus. The chemical shifts are reported in δ (ppm) values (^{1}H and ^{13}C NMR relative to $CHCl_3$, δ 7.26 ppm for ^{1}H NMR and δ 77.0 ppm for ^{13}C NMR). Or alternatively, ^{1}H NMR chemical shifts were referenced to tetramethylsilane signal (0 ppm). Multiplicities are recorded by s (singlet), d (doublet), t (triplet), q (quartet), p (pentet), h (hextet), m (multiplet) and br (broad). Coupling constants (*J*), are reported in Hertz (Hz). GC analyses were performed using a Shimadzu GC-2010 ultra gas chromatography–mass spectrometry instrument equipped with a Shimadzu AOC-20s autosampler. The crude radiolabelled product was separated by HPLC on UltiMate 3000 system (Thermo Fisher Scientific, USA) equipped with a Flow-Count radio-HPLC detector (Eckert & Ziegler) under the following conditions: Agilent Eclipse XDB- C18, 5 µm, 4.6 × 250 mm, the data were recorded and processed by Chromatography Data System (Thermo Fisher Scientific, USA) for determination of radiochemical conversion. The activities of [^{125}I] radiolabeled samples were determined using a radioisotope dose calibrator CRC-55tR (Capintec Inc.)

2. Synthesis of Aryl Trimethylsilanes Starting Material 1

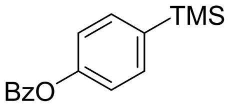
All the aryl trimethylsilanes used in the present study were prepared following a known procedure. All spectroscopic data were matched in those reported (**1a**,[1] **1b**,[1] **1c**,[2] **1d**,[2] **1e**,[3] **1h**, [1] **1i**,[4] **1k**,[5] **1m**,[1] **1n**,[6] **1o**[7]). The following paragraphs list the characterization data for the new compounds.



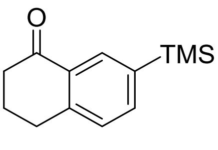
4'-(Trimethylsilyl)-[1,1'-biphenyl]-3-carbonitrile (**1f**). Yellow solid, 45% yield. purified by flash column chromatography (silica gel, hexane/EtOAc = 50:1). ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.87 (t, *J* = 1.5 Hz, 1H), 7.82 (dt, *J* = 7.8, 1.5 Hz, 1H), 7.66 – 7.61 (m, 3H), 7.57 – 7.52 (m, 3H), 0.31 (s, 9H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 142.5, 141.0, 139.2, 134.2, 131.5, 130.8, 130.7, 129.7, 126.4, 118.9, 113.0, -1.1. HRMS (EI$^+$): Calcd. for $C_{16}H_{17}NSi$ [M$^+$]: *m/z* 251.1125. Found: 251.1121.



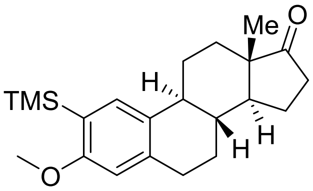
Methyl 4'-(trimethylsilyl)-[1,1'-biphenyl]-3-carboxylate (**1g**). Yellow solid, 38% yield. purified by flash column chromatography (silica gel, hexane/EtOAc = 100:1). ^{1}H NMR (400 MHz, CDCl$_3$) δ 8.29 (t, *J* = 1.6 Hz, 1H), 8.02 (dt, *J* = 7.7, 1.4 Hz, 1H), 7.79 (dt, *J* = 7.7, 1.8 Hz, 1H), 7.62 (s, 4H), 7.51 (t, *J* = 7.7 Hz, 1H), 3.95 (s, 3H), 0.31 (s, 9H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 167.1, 141.5, 140.5, 140.0, 134.0, 131.6, 130.8, 128.9, 128.5, 128.4, 126.5, 52.3, -1.0. HRMS (EI$^+$): Calcd. for $C_{17}H_{20}O_2Si$ [M$^+$]: *m/z* 284.1227. Found: 284.1231.



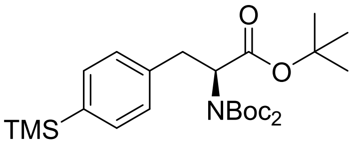
4-(Trimethylsilyl)phenyl benzoate (**1j**). White solid 62% yield. purified by flash column chromatography (silica gel, hexane/EtOAc = 100:1). ^{1}H NMR (400 MHz, CDCl$_3$) δ 8.21 (dd, *J* = 8.3, 1.2 Hz, 2H), 7.64 (t, *J* = 7.4 Hz, 1H), 7.58 (d, *J* = 8.4 Hz, 2H), 7.51 (t, *J* = 7.7 Hz, 2H), 7.20 (d, *J* = 8.4 Hz, 2H), 0.29 (s, 9H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 165.2, 151.6, 138.1, 134.7, 133.6, 130.2, 129.7, 128.6, 121.0, -1.0. HRMS (EI$^+$): Calcd. for $C_{16}H_{18}O_2Si$ [M$^+$]: *m/z* 270.1071. Found: 270.1074.



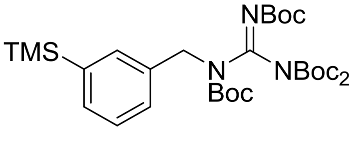
7-(Trimethylsilyl)-3,4-dihydronaphthalen-1(2H)-one (**1l**). Yellow oil, 69% yield. purified by flash column chromatography (silica gel, hexane/EtOAc = 50:1). ^{1}H NMR (400 MHz, CDCl$_3$) δ 8.20 (s, 1H), 7.62 (dd, *J* = 7.5, 1.3 Hz, 1H), 7.24 (d, *J* = 6.9 Hz, 1H), 2.96 (t, *J* = 6.1 Hz, 2H), 2.68 – 2.64 (m, 2H), 2.14 (q, *J* = 6.4 Hz, 2H), 0.28 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.8, 145.1, 139.0, 138.3, 132.3, 131.8, 128.3, 39.4, 29.8, 23.3, -1.1. HRMS (EI$^+$): Calcd. for $C_{13}H_{18}OSi$ [M$^+$]: *m/z* 218.1121. Found: 218.1119.



(8R,9S,13S,14S)-3-methoxy-13-methyl-2-(trimethylsilyl)-6,7,8,9,11,12,13,14,15,16-decahydro-17H-cyclopenta[a]phenanthren-17-one (**1ak**). Yellow solid, 27% yield. purified by flash column chromatography (silica gel, hexane/EtOAc = 20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (s, 1H), 6.63((s, 1H), 3.81(s, 3H), 3.12 – 2.87 (m, 2H), 2.66 - 1.96 (m, 7H), 1.77 – 1.38 (m, 6H), 0.98 (s, 3H), 0.32 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.3, 140.1, 132.9, 132.1, 125.9, 110.8, 55.9, 51.2, 48.9, 44.9, 39.4, 36.7, 32.5, 30.9, 27.4, 26.8, 22.4, 14.7, -0.0. HRMS (EI$^+$): Calcd. for $C_{22}H_{32}O_2Si$ [M$^+$]: *m/z* 356.2172. Found: 356.2174.



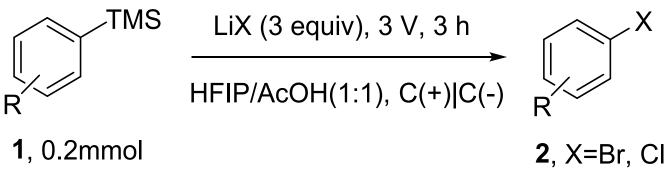
Tert-butyl (S)-2-(bis(tert-butoxycarbonyl)amino)-3-(4-(trimethylsilyl)phenyl)propanoate (**1al**). Yellow solid, 34% yield. purified by flash column chromatography (silica gel, hexane/EtOAc = 20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 (d, *J* = 7.6 Hz, 2H), 7.19 (d, *J* = 7.6 Hz, 2H), 5.05 (dd, *J* = 10.4, 4.8 Hz, 1H), 3.43 - 3.18 (m, 2H), 1.50(s, 9H), 1.41 (s, 18H), 0.25(s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.1, 156.2, 139.7, 138.1, 134.5, 130.1, 83.1, 80.7, 55.9, 39.7, 29.4, 29.0, -0.0. HRMS (EI$^+$): Calcd. for $C_{26}H_{43}NO_6Si$ [M$^+$]: *m/z* 493.2860. Found: 493.2865.



(**1am**). Yellow solid, 38% yield. purified by flash column chromatography (silica gel, hexane/EtOAc = 20:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.6 (s, 1H), 7.46 (d, *J* = 7.6 Hz, 1H), 7.40 (d, *J* = 7.2 Hz,1H), 7.29 (d, *J* = 7.2 Hz,1H), 5.05 (s, 2H), 1.51 (s, 9H), 1.44 (s, 27H), 0.27(s, 9H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 157.5, 151.4, 147.4, 144.3, 140.1, 136.5, 133.5, 132.2, 128.6, 127.6, 83.6, 83.6, 81.9, 50.1, 28.0, 27.9, 27.8, -1.1. HRMS (EI$^+$): Calcd. for $C_{31}H_{51}N_3O_8Si$ [M$^+$]: *m/z* 621.3445. Found: 621.3449.

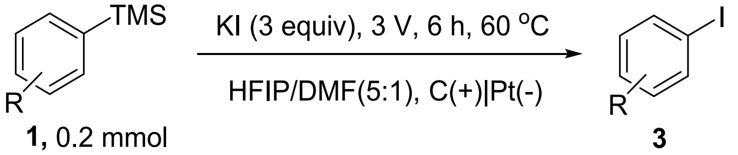
3. General Procedure for Halodesilylation of Aryl Trimethylsilanes

3.1 small-scale reactions for bromination and chlorination



In a 10 mL tube equipped with a stir bar, aryl trimethylsilanes **1** (0.2 mmol), lithium halides (0.6 mmol, 3 equiv) were dissolved in a mixed solvent of HFIP (1 mL) and AcOH (1 mL), following with two carbon felt electrodes (10 ×10 × 2 mm) were immerged. The reaction mixture was stirred at room temperature, with constant voltage (3 V) was continuous given for 3 h (**Fig S1**). The slightly modified conditions were given in each case. Upon completion, the reaction mixture was diluted with Et$_2$O and washed with water and saturated NaHCO$_3$ solution. The organic layer was dried over Na$_2$SO$_4$ and concentrated to dryness. The residue was purified by flash chromatography on silica gel (n-hexane/ethyl acetate).

3.2 Small-scale reactions for iodination



In a 10 mL tube equipped with a stir bar, aryl trimethylsilanes (**1**, 0.2 mmol), KI (0.6 mmol, 3 equiv) were dissolved in a mixed solvent of HFIP (1.67 mL) and DMF (0.33 mL), following with a carbon felt anode (10 ×10 × 2 mm) and a platinum cathode (10 ×10 × 0.1 mm) were immerged. The reaction mixture was stirred at 60 °C, with constant voltage (3 V) was continuous given for 6 h. The slightly modified conditions were given in each case. Upon completion, the reaction mixture was diluted with Et$_2$O and washed with water and saturated Na$_2$S$_2$O$_3$ solution. The organic layer was dried over Na$_2$SO$_4$ and concentrated to dryness. The residue was purified by flash chromatography on silica gel (n-hexane/ethyl acetate). The iodinated product **4k**,[8] **4l**,[9] and **4m**[10] are known compounds and were prepared in accordance with references. All spectroscopic data were matched in those reported.

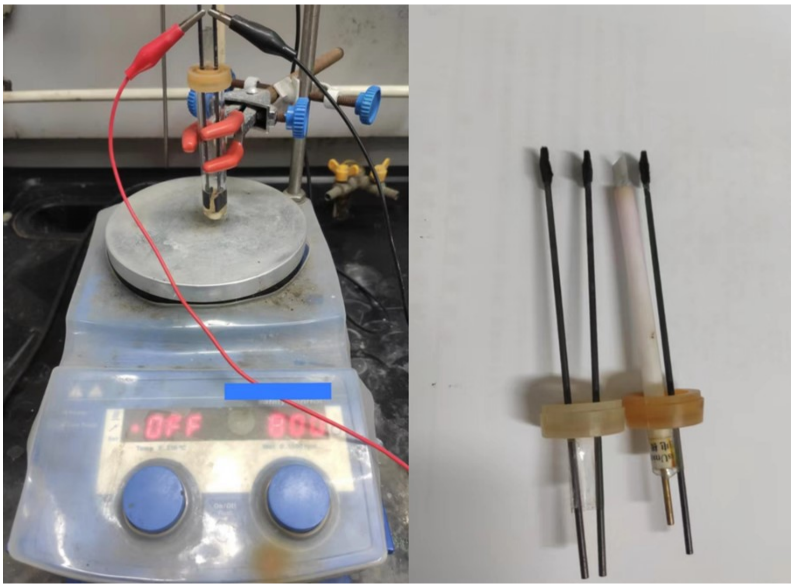
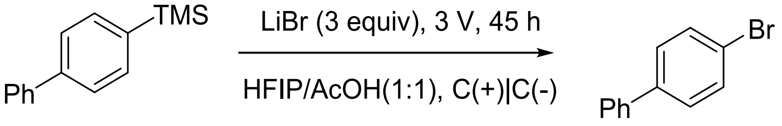


Fig S1. Setup for the small-scale reactions

3.3 Gram-scale reactions



1b, 5 mmol

2b

In a 50 mL three-necked flask equipped with a stir bar, (1,1'-biphenyl)-4-yl-trimethylsilane (**1b**, 5 mmol, 1.13 g), lithium bromide (15 mmol, 1.29 g) were dissolved in a mixed solvent of HFIP (15 mL) and AcOH (15 mL), following with two carbon felt electrodes (20 ×20 × 2 mm) were immersed. The reaction mixture was stirred at room temperature, with constant voltage (3 V) was continuously given for 45 h (**Fig S2**). Upon completion, the reaction mixture was diluted with Et_2O and washed with water, and saturated $NaHCO_3$ solution. The organic layer was dried over Na_2SO_4 and concentrated to dryness. The residue was purified by flash chromatography on silica gel (n-hexane/ethyl acetate) to yield product **2b** (1.07 g, 91.8%).

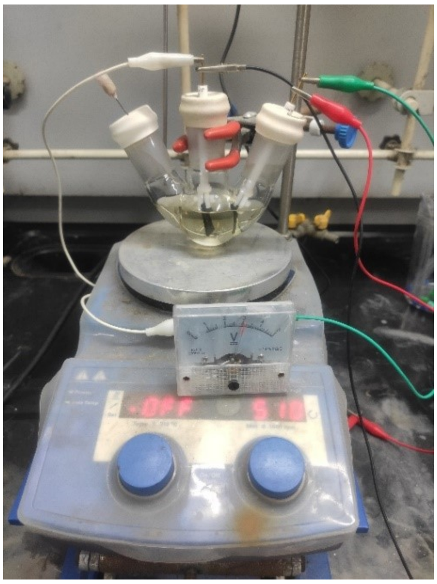
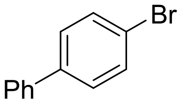
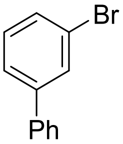


Fig S2. Setup for the gram-scale reactions

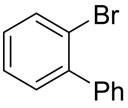
4. Analytic Data of Products



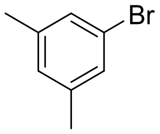
4-Bromo-1,1'-biphenyl (**2b**). White solid, 91% yield (42.6 mg), purified by flash column chromatography (silica gel, hexane/EtOAc = 30:1). ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.57 – 7.50 (m, 4H), 7.47 – 7.38 (m, 4H), 7.37 – 7.32 (m, 1H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 140.2, 140.0, 131.9, 128.9, 128.8, 127.7, 127.0, 121.6.



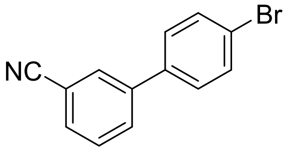
3-Bromo-1,1'-biphenyl (**2c**). Colorless oil, 93% yield (43.3 mg), purified by flash column chromatography (silica gel, hexane). ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.73 (t, *J* = 1.9 Hz, 1H), 7.59 – 7.53 (m, 2H), 7.52 – 7.42 (m, 4H), 7.40 – 7.33 (m, 1H), 7.30 (t, *J* = 7.8 Hz, 1H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 143.4, 139.7, 130.3, 130.2, 130.2, 128.9, 127.9, 127.2, 125.8, 122.9.



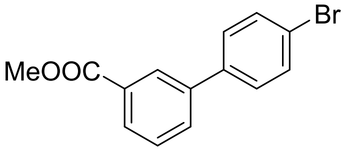
3-Bromo-1,1'-biphenyl (**2d**). Colorless oil, 94% yield (43.9 mg), purified by flash column chromatography (silica gel, hexane). ^{1}H NMR (600 MHz, CDCl$_3$) δ 7.72 (d, *J* = 8.2 Hz, 1H), 7.51 – 7.43 (m, 5H), 7.42 – 7.35 (m, 2H), 7.25 (td, *J* = 7.5, 2.2 Hz, 1H). ^{13}C NMR (151 MHz, CDCl$_3$) δ 142.6, 141.1, 133.1, 131.3, 129.4, 128.8, 128.0, 127.6, 127.4, 122.7.



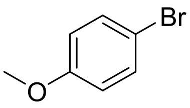
1-Bromo-3,5-dimethylbenzene (**2e**). Colorless oil, 70% yield (26.0 mg), purified by flash column chromatography (silica gel, hexane), 91% yield was determined by GC-MS (because of its high volatility, **2e** may not be easily isolated completely). ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.12 (s, 2H), 6.89 (s, 1H), 2.27 (s, 6H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 139.9, 129.1, 128.7, 122.1, 21.1.



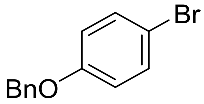
4'-Bromo-[1,1'-biphenyl]-3-carbonitrile (**2f**). White solid, 73% yield (37.4 mg), purified by flash column chromatography (silica gel, hexane/EtOAc = 50:1). ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.82 (t, *J* = 1.8 Hz, 1H), 7.77 (dt, *J* = 7.8, 1.6 Hz, 1H), 7.65 (dt, *J* = 7.7, 1.4 Hz, 1H), 7.62 – 7.58 (m, 2H), 7.55 (t, *J* = 7.7 Hz, 1H), 7.45 – 7.39 (m, 2H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 141.3, 137.8, 132.3, 131.3, 131.1, 130.5, 129.8, 128.7, 122.9, 118.6, 113.2.



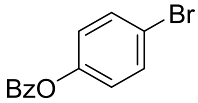
Methyl 4'-bromo-[1,1'-biphenyl]-3-carboxylate (**2g**). Yellow oil, 87% yield (50.8 mg), purified by flash column chromatography (silica gel, hexane/EtOAc = 100:1). ^{1}H NMR (400 MHz, CDCl$_3$) δ 8.23 (t, *J* = 1.9 Hz, 1H), 8.02 (dt, *J* = 7.8, 1.4 Hz, 1H), 7.72 (dd, *J* = 7.7, 1.9 Hz, 1H), 7.60 – 7.54 (m, 2H), 7.52 – 7.45 (m, 3H), 3.94 (s, 3H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 166.9, 140.2, 139.0, 132.0, 131.3, 130.8, 129.0, 128.7, 128.7, 128.0, 122.1, 52.3.



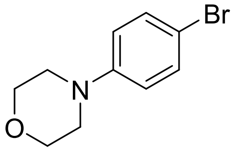
1-Bromo-4-methoxybenzene (**2h**). Colorless oil, 72% yield (26.8 mg), purified by flash column chromatography (silica gel, hexane/EtOAc = 100:1), 93% yield was determined by GC-MS (because of its high volatility, **2h** may not be easily isolated completely). ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.37 (d, *J* = 8.9 Hz, 2H), 6.77 (d, *J* = 8.9 Hz, 2H), 3.77 (s, 3H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 158.7, 132.2, 115.7, 112.8, 55.4.



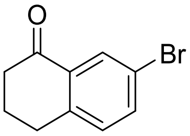
1-(Benzyloxy)-4-bromobenzene (**2i**). White solid, 91% yield (47.9 mg), purified by flash column chromatography (silica gel, hexane/EtOAc = 100:1). ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.45 – 7.28 (m, 7H), 6.86 – 6.81 (m, 2H), 5.01 (s, 2H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 157.9, 136.6, 132.3, 128.7, 128.1, 127.5, 116.7, 113.1, 70.2.



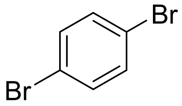
4-Bromophenyl benzoate (**2j**). White solid, 85% yield (45.1 mg), purified by flash column chromatography (silica gel, hexane/EtOAc = 100:1). ^{1}H NMR (400 MHz, CDCl$_3$) δ 8.18 (dd, *J* = 8.4, 1.4 Hz, 2H), 7.66 – 7.60 (m, 1H), 7.56 – 7.46 (m, 4H), 7.16 – 7.07 (m, 2H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 164.9, 150.0, 133.8, 132.6, 130.2, 129.2, 128.7, 123.6, 119.0.



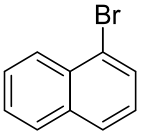
4-(4-Bromophenyl)morpholine (**2k**). Yellow solid, 79% yield (38.2 mg), purified by flash column chromatography (silica gel, hexane/EtOAc = 50:1). ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.43 – 7.30 (m, 2H), 6.81 – 6.74 (m, 2H), 3.90 – 3.81 (m, 4H), 3.15 – 3.08 (m, 4H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 150.3, 117.3, 112.2, 66.8, 49.2.



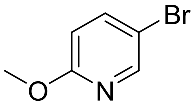
7-Bromo-3,4-dihydronaphthalen-1(2H)-one (**2l**). Yellow solid, 48% yield (21.6 mg), purified by flash column chromatography (silica gel, hexane/EtOAc = 50:1). Constant voltage: 5 V, temperature: 60 °C, reaction time: 9 h. ^{1}H NMR (400 MHz, CDCl$_3$) δ 8.15 (d, *J* = 2.2 Hz, 1H), 7.57 (dd, *J* = 8.2, 2.2 Hz, 1H), 7.14 (d, *J* = 8.2 Hz, 1H), 2.91 (t, *J* = 6.1 Hz, 2H), 2.68 – 2.62 (m, 2H), 2.13 (p, *J* = 6.4 Hz, 2H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 197.0, 143.1, 136.1, 134.1, 130.6, 130.0, 120.7, 38.8, 29.2, 23.0.



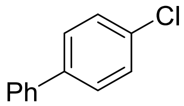
1,4-Dibromobenzene (**2m**). White solid, 73% yield (34.2 mg), purified by flash column chromatography (silica gel, hexane). Constant voltage: 5 V, temperature: 60 °C. ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.34 (s, 4H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 133.2, 121.1.



1-Bromonaphthalene (**2n**). Yellow oil, 89% yield (37.1 mg), purified by flash column chromatography (silica gel, hexane). ^{1}H NMR (400 MHz, CDCl$_3$) δ 8.23 (d, *J* = 8.5 Hz, 1H), 7.83 – 7.75 (m, 3H), 7.63 – 7.53 (m, 1H), 7.56 – 7.47 (m, 1H), 7.32 – 7.26 (m, 1H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 134.7, 132.0, 129.9, 128.4, 128.0, 127.3, 127.1, 126.7, 126.2, 122.9.

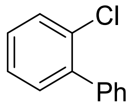


5-Bromo-2-methoxypyridine (**2o**). Colorless oil, 69% yield (25.8 mg), purified by flash column chromatography (silica gel, hexane/EtOAc = 50:1), 91% yield was determined by GC-MS (because of its high volatility, **2o** may not be easily isolated completely). Constant voltage: 5 V, temperature: 60 °C. ^{1}H NMR (400 MHz, CDCl$_3$) δ 8.20 (d, *J* = 2.5 Hz, 1H), 7.63 (dd, *J* = 8.8, 2.5 Hz, 1H), 6.66 (d, *J* = 8.8 Hz, 1H), 3.91 (s, 3H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 163.0, 147.5, 141.0, 112.6, 111.7, 53.7.

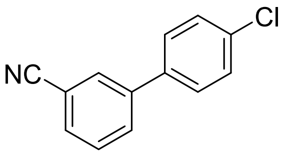


4-Chloro-1,1'-biphenyl (**2q**). White solid, 76% yield (28.7 mg), purified by flash column chromatography (silica gel, hexane). Reaction time: 6 h, LiCl (1.2 mmol, 6 equiv). ^{1}H NMR (400

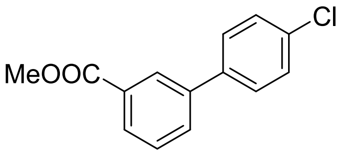
MHz, $CDCl_3$) δ 7.58 – 7.48 (m, 4H), 7.48 – 7.32 (m, 5H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 140.0, 139.7, 133.4, 128.93, 128.9, 128.4, 127.6, 127.0.



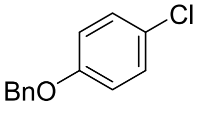
2-Chloro-1,1'-biphenyl (**2r**). White solid, 71% yield (26.7 mg), purified by flash column chromatography (silica gel, hexane). Reaction time: 6 h, LiCl (1.2 mmol, 6 equiv). ^1H NMR (400 MHz, $CDCl_3$) δ 7.46−7.43 (m, 1H), 7.45 – 7.42 (m, 3H), 7.42 – 7.21 (m, 5H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 140.6, 139.4, 132.5, 131.4, 130.0, 129.5, 128.5, 128.1, 127.6, 126.8.



4'-Chloro-[1,1'-biphenyl]-3-carbonitrile (**2s**). White solid, 82% yield (35.1 mg), purified by flash column chromatography (silica gel, hexane/EtOAc = 100:1). Reaction time: 6 h, LiCl (1.2 mmol, 6 equiv). ^1H NMR (600 MHz, $CDCl_3$) δ 7.84 (s, 1H), 7.80 (d, J = 7.9 Hz, 1H), 7.67 (d, J = 7.7 Hz, 1H), 7.58 (t, J = 7.8 Hz, 1H), 7.51 (d, J = 8.5 Hz, 2H), 7.47 (d, J = 8.6 Hz, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 141.2, 137.3, 134.7, 131.3, 131.0, 130.5, 129.8, 129.3, 128.3, 118.7, 113.1.

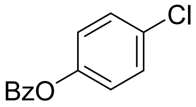


Methyl 4'-chloro-[1,1'-biphenyl]-3-carboxylate (**2t**). Yellow oil, 82% yield (41.5 mg), purified by flash column chromatography (silica gel, hexane/EtOAc = 100:1). Reaction time: 6 h, LiCl (1.2 mmol, 6 equiv). ^1H NMR (400 MHz, $CDCl_3$) δ 8.23 (s, 1H), 8.02 (d, J = 7.8 Hz, 1H), 7.73 (d, J = 7.8 Hz, 1H), 7.59 – 7.48 (m, 3H), 7.42 (d, J = 8.5 Hz, 2H), 3.95 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.9, 140.2, 138.5, 133.9, 131.3, 130.8, 129.1, 129.0, 128.7, 128.4, 128.1, 52.3.

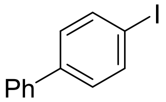


1-(Benzyloxy)-4-chlorobenzene (**2u**). White solid, 85% yield (37.2 mg), purified by flash column chromatography (silica gel, hexane/EtOAc = 100:1). Reaction time: 6 h, LiCl (1.2 mmol, 6 equiv). ^1H

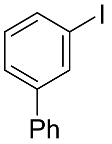
NMR (400 MHz, $CDCl_3$) δ 7.43 (d, *J* = 7.3 Hz, 2H), 7.38 (t, *J* = 7.3 Hz, 2H), 7.35 – 7.25 (m, 3H), 7.01 – 6.87 (m, 3H), 5.06 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 158.8, 137.1, 129.5, 128.6, 128.0, 127.5, 121.0, 114.9, 69.9.



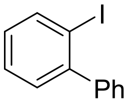
4-Chlorophenyl benzoate (**2v**). White solid, 79% yield (36.8 mg), purified by flash column chromatography (silica gel, hexane/EtOAc = 100:1). Reaction time: 6 h, LiCl (1.2 mmol, 6 equiv). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 (d, *J* = 7.0 Hz, 2H), 7.65 (t, *J* = 7.4 Hz, 1H), 7.52 (t, *J* = 7.7 Hz, 2H), 7.45 – 7.32 (m, 2H), 7.20 – 7.12 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.8, 150.8, 138.6, 133.8, 130.2, 129.2, 128.7, 124.0, 90.0.



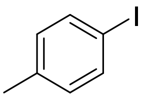
4-Iodo-1,1'-biphenyl (**3b**). White solid, 89% yield (49.6 mg), purified by flash column chromatography (silica gel, hexane). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 8.3 Hz, 2H), 7.52 (d, *J* = 7.2 Hz, 2H), 7.42 (t, *J* = 7.5 Hz, 2H), 7.35 (d, *J* = 7.1 Hz, 1H), 7.30 (d, *J* = 8.3 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 140.8, 140.1, 137.9, 129.1, 129.0, 127.7, 126.9, 93.1.



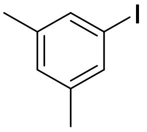
3-Iodo-1,1'-biphenyl (**3c**). Yellow oil, 93% yield (52.0 mg), purified by flash column chromatography (silica gel, hexane). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (t, *J* = 1.7 Hz, 1H), 7.66 (dd, *J* = 7.9, 1.7 1H), 7.55 – 7.49 (m, 3H), 7.46 – 7.39 (m, 2H), 7.38 – 7.31 (m, 1H), 7.15 (t, *J* = 7.8 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 143.5, 139.7, 136.2, 136.2, 130.4, 128.9, 127.9, 127.1, 126.4, 94.9.



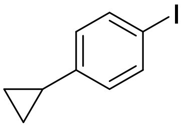
2-Iodo-1,1'-biphenyl (**3d**). Yellow solid, 91% yield (51.0 mg), purified by flash column chromatography (silica gel, hexane). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.44 – 7.35 (m, 4H), 7.36 – 7.25 (m, 4H), 7.01 (td, *J* = 7.6, 1.8 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 146.7, 144.2, 139.5, 130.1, 129.3, 128.8, 128.2, 128.0, 127.7, 98.7.



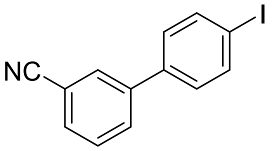
1-Iodo-4-methylbenzene (**3e**). Yellow solid, 62% yield (27.1 mg), purified by flash column chromatography (silica gel, hexane), 94% yield was determined by GC-MS (because of its high volatility, **3e** may not be easily isolated completely). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 (d, *J* = 8.2 Hz, 2H), 6.92 (d, *J* = 8.0 Hz, 2H), 2.29 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.5, 137.2, 131.2, 90.2, 21.0.



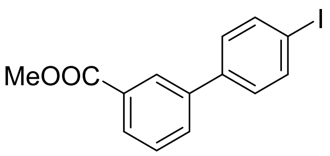
1-Iodo-3,5-dimethylbenzene (**3f**). Yellow solid, 83% yield (38.5 mg), purified by flash column chromatography (silica gel, hexane). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 (s, 2H), 6.94 (s, 1H), 2.25 (s, 6H).^{13}C NMR (101 MHz, $CDCl_3$) δ 140.0, 135.1, 129.4, 94.3, 20.9.



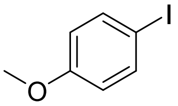
1-Cyclopropyl-4-iodobenzene (**3g**). Yellow solid, 86% yield (42.1 mg), purified by flash column chromatography (silica gel, hexane). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (d, *J* = 8.4 Hz, 2H), 6.82 (d, *J* = 8.4 Hz, 2H), 1.86 - 1.76 (m, 1H), 1.03 – 0.91 (m, 2H), 0.70 - 0.62 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 143.8, 137.2, 127.8, 89.8, 15.1, 9.4.



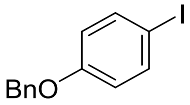
4'-Iodo-[1,1'-biphenyl]-3-carbonitrile (**3h**). Yellow solid, 89% yield (54.3 mg), purified by flash column chromatography (silica gel, hexane/EtOAc = 100:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 – 7.78 (m, 3H), 7.76 (dt, *J* = 7.8, 1.6 Hz, 1H), 7.64 (dt, *J* = 7.6, 1.4 Hz, 1H), 7.54 (t, *J* = 7.7 Hz, 1H), 7.33 – 7.26 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 141.3, 138.3, 138.3, 131.2, 131.1, 130.4, 129.8, 128.8, 118.6, 113.2, 94.51. HRMS (EI$^+$): Calcd. for $C_{13}H_8NI$ [M$^+$]: *m/z* 304.9696. Found: 304.9702.



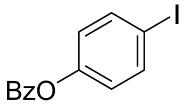
Methyl 4'-iodo-[1,1'-biphenyl]-3-carboxylate (**3i**). Yellow solid, 94% yield (63.4 mg), purified by flash column chromatography (silica gel, hexane/EtOAc = 100:1). ^{1}H NMR (400 MHz, CDCl$_3$) δ 8.22 (t, *J* = 1.8 Hz, 1H), 8.02 (dt, *J* = 7.9, 1.4 Hz, 1H), 7.79 – 7.74 (m, 2H), 7.71 (dd, *J* = 7.8, 2.0 Hz, 1H), 7.52 – 7.46 (m, 1H), 7.37 – 7.31 (m, 2H), 3.94 (s, 3H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 166.9, 140.3, 139.6, 138.0, 131.2, 130.9, 129.0, 129.0, 128.8, 128.0, 93.7, 52.3. HRMS (EI$^+$): Calcd. for $C_{14}H_{11}O_2I$ [M$^+$]: *m/z* 337.9798. Found: 337.9803.



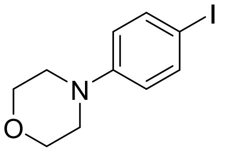
1-Iodo-4-methoxybenzene (**3j**). Yellow solid, 81% yield (37.9 mg), purified by flash column chromatography (silica gel, hexane/EtOAc = 100:1). ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.66 – 7.43 (m, 2H), 6.75 – 6.62 (m, 2H), 3.77 (s, 3H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 159.5, 138.2, 116.4, 82.7, 55.33.



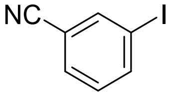
1-(Benzyloxy)-4-iodobenzene (**3k**). Yellow solid, 81% yield (50.2 mg), purified by flash column chromatography (silica gel, hexane/EtOAc = 100:1). ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.57 – 7.50 (m, 2H), 7.41 – 7.29 (m, 5H), 6.76 – 6.69 (m, 2H), 5.01 (s, 2H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 158.7, 138.2, 136.6, 128.7, 128.2, 127.5, 117.3, 83.1, 70.1.



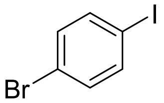
4-Iodophenyl benzoate (**3l**). White solid, 68% yield (44.2 mg), purified by flash column chromatography (silica gel, hexane/EtOAc = 100:1). ^{1}H NMR (400 MHz, CDCl$_3$) δ 8.18 (dd, *J* = 8.3, 1.4 Hz, 2H), 7.76 – 7.69 (m, 2H), 7.64 (t, *J* = 7.4 Hz, 1H), 7.50 (t, *J* = 7.7 Hz, 2H), 7.02 – 6.95 (m, 2H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 158.7, 138.3, 136.6, 128.7, 128.1, 127.5, 117.3, 83.1, 70.1.



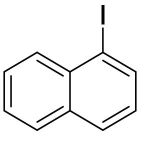
4-(4-Iodophenyl)morpholine (**3m**). Yellow solid, 66% yield (37.9 mg), purified by flash column chromatography (silica gel, hexane/EtOAc = 30:1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (d, *J* = 8.8 Hz, 2H), 6.66 (d, *J* = 8.8 Hz, 2H), 4.02 – 3.71 (m, 4H), 3.23 – 2.94 (m, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.9, 137.9, 117.7, 81.8, 66.7, 48.9.



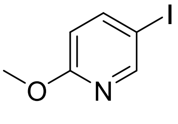
3-iodobenzonitrile (**3n**). Yellow solid, 41% yield (18.9 mg), purified by flash column chromatography (silica gel, hexane/ EtOAc = 100:1). Constant voltage: 6 V, additive: I_2 (0.1 mmol) and KF (0.2 mmol), reaction time: 32 h. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.00 (t, *J* = 1.5 Hz, 1H), 7.96 (dt, *J* = 8.0, 1.3 Hz, 1H), 7.65 (dt, *J* = 7.8, 1.2 Hz, 1H), 7.24 (t, *J* = 7.9 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 141.9, 140.5, 131.2, 130.6, 117.1, 114.2, 93.9.



1-Bromo-4-iodobenzene (**3o**). White solid, 78% yield (40.2 mg), purified by flash column chromatography (silica gel, hexane). Constant voltage: 5 V. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (d, *J* = 8.5 Hz, 2H), 7.25 (d, *J* = 8.5 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 140.2, 134.6, 123.4, 93.2.



1-Iodonaphthalene (**3p**). White solid, 86% yield (43.5 mg), purified by flash column chromatography (silica gel, hexane). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 – 7.99 (m, 2H), 7.81 (d, *J* = 8.3 Hz, 1H), 7.74 (d, *J* = 8.1 Hz, 1H), 7.60 – 7.51 (m, 1H), 7.54 – 7.45 (m, 1H), 7.19 – 7.10 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.5, 134.4, 134.2, 132.2, 129.0, 128.6, 127.7, 126.9, 126.8, 99.6.



5-Iodo-2-methoxypyridine (**3q**). Yellow oil, 85% yield (40.0 mg), purified by flash column chromatography (silica gel, hexane/EtOAc = 50:1). ^{1}H NMR (400 MHz, CDCl$_3$) δ 8.34 (d, J = 2.2 Hz, 1H), 7.77 (dd, J = 8.7, 2.4 Hz, 1H), 6.59 (d, J = 8.7 Hz, 1H), 3.90 (s, 3H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 163.5, 152.7, 146.3, 113.4, 82.1, 53.6.

5. General Procedure for Electrochemical ^{125}I Radiolabeling

5.1 Reactions of Radioiodination

$$\underset{\textbf{1},\ 0.05\ \text{mmol}}{\text{Ar-TMS}} \xrightarrow[\text{HFIP/DMF(5:1, 1.2 mL), 60 °C, Pt(+,-)}]{[^{125}\text{I}]\ \text{NaI}(\textit{ca.}\ 100\ \mu\text{Ci}),\ \text{KF(0.15 M), 4 V, 40 min}} \underset{\textbf{4}}{\text{Ar-}^{125}\text{I}}$$

In an 8 mL tube equipped with a stir bar, aryl trimethylsilanes (**1**, 0.05 mmol), Na^{125}I (*ca.* 100 μCi), KF (0.15 M, 0.18 mmol) were dissolved in a mixed solvent of HFIP (1 mL) and DMF (0.2 mL), following with two platinum electrodes (8 ×10 × 0.1 mm) were immersed. The reaction mixture was stirred at 60 °C, with constant voltage (4 V) was continuous given for 40 min. An aliquot of the mixture was then analyzed by radio-HPLC to confirm the identity of the radiolabeled compound, along with the radiochemical conversions (RCCs). The reaction mixture was eluted with acetonitrile and ammonium formate buffer (0.1 M, pH 4.5), the flow rate is 1.0 mL/min. Because of the tubing distance between the UV detector and the radiation detector, the retention time for UV peak is about 0.5 min earlier than that of the radioactive peak.

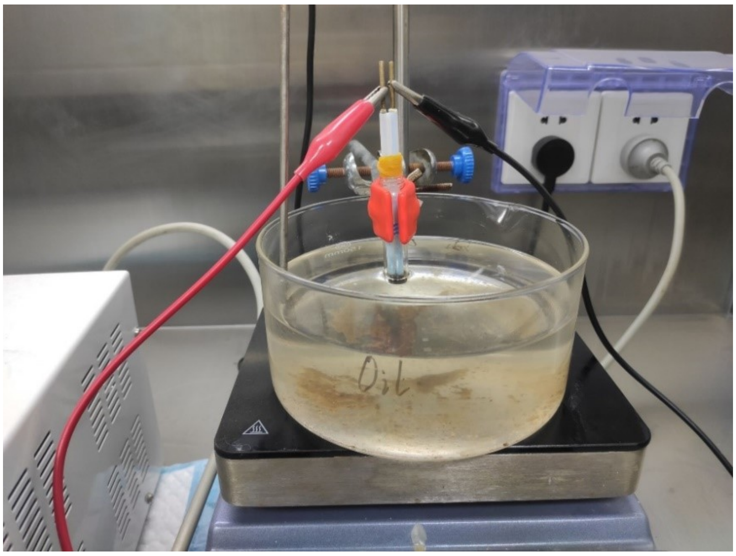


Fig S3. Setup for the radioiodination in hot cell

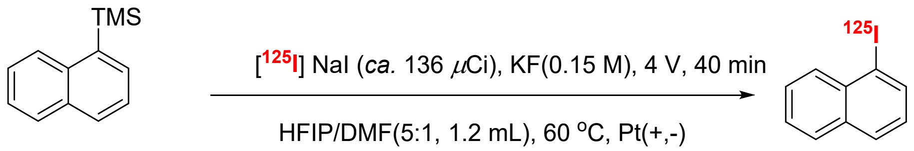
5.2 HPLC RCC

After the radiolabeling reaction, the reaction was cooled and 300 µL of the crude mixture was loaded into a HPLC column (Agilent Eclipse SB-C18, 5 µm, 4.6 × 250 mm). Purification was achieved with 1 mL/min flow eluting the column with acetonitrile and ammonium formate buffer (0.1 M, pH = 4.5). The final data was calculated by integration of each radio-peak and corresponding product.

5.2 Isolation yield

The reaction fraction was collected by an automatic fraction collector. The activity injected into HPLC was measured (this activity was denoted by A), and the time of injection was recorded. The fraction corresponding to the radiolabeled product was collected, and the activity was measured (this activity was denoted by B). The decay-corrected RCY was calculated by dividing the decay-corrected B by A. The activities of [^{125}I] radiolabeled samples were determined using a radioisotope dose calibrator CRC-55tR (Capintec Inc.).

5.3 Specific activity calculation

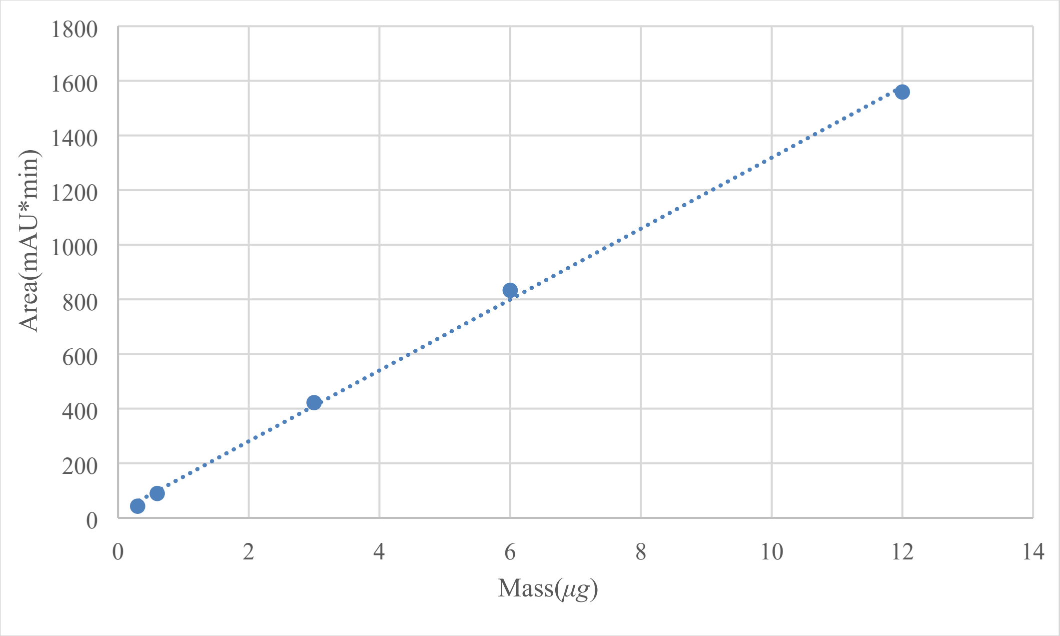


1, 0.05 mmol

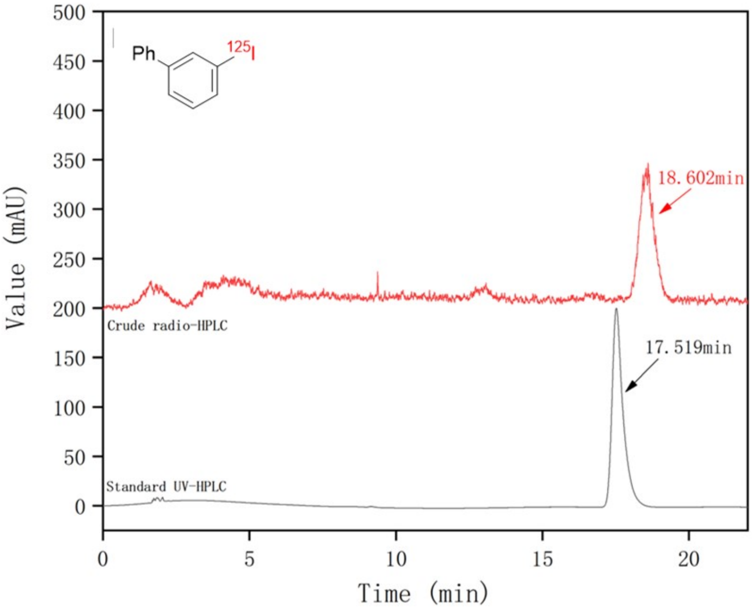
4h

In an 8 mL tube equipped with a stir bar, trimethyl(naphthalen-1-yl)silane (**1h**, 0.05 mmol), Na^{125}I (*ca.* 136 μCi = 5.03*10^{-3} GBq), KF (0.15 M, 0.18 mmol) were dissolved in a mixed solvent of HFIP (1 mL) and DMF (0.2 mL), following with two platinum electrodes (8 ×10 × 0.1 mm) were immerged. The reaction mixture was stirred at 60°C, with constant voltage (4 V) was continuous given for 40 min. Then the reaction mixture was cooled and loaded onto a HPLC column (Agilent Eclipse SB-C18, 5 µm, 9.4 × 250 mm). Purification was achieved with 2.5 mL/min flow eluting the column with 70% acetonitrile and 35% ammonium formate buffer (0.1 M, pH = 4.5) and [^{125}I]-1-iodonaphthalene **4h** (2.16*10^{-3} GBq) was collected. UV absorbance (at 220 nm) of 24.8 was measured, corresponding to 0.030 µg for a specific activity of 18.3 ± 7.1 GBq/µmol (n =3) at the end of synthesis (EOS)

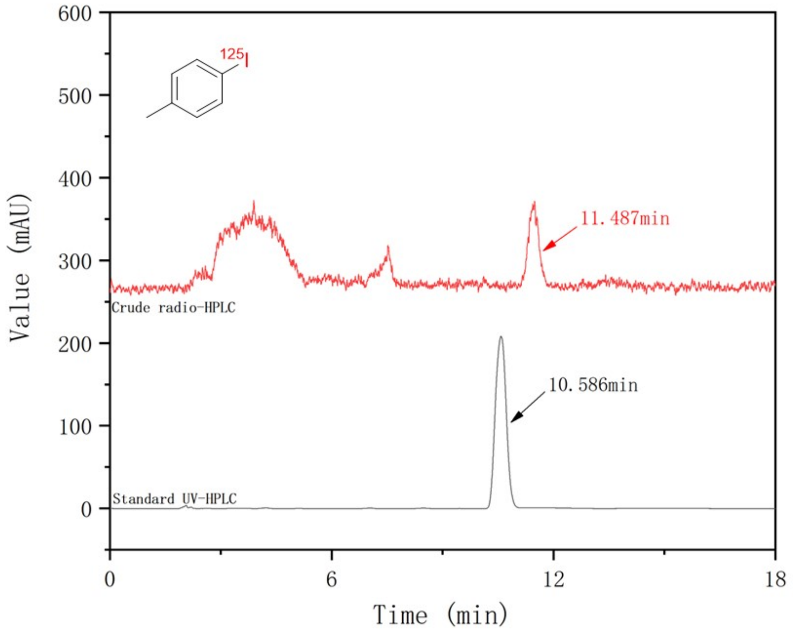
Calibration curve 1-iodonaphthalene



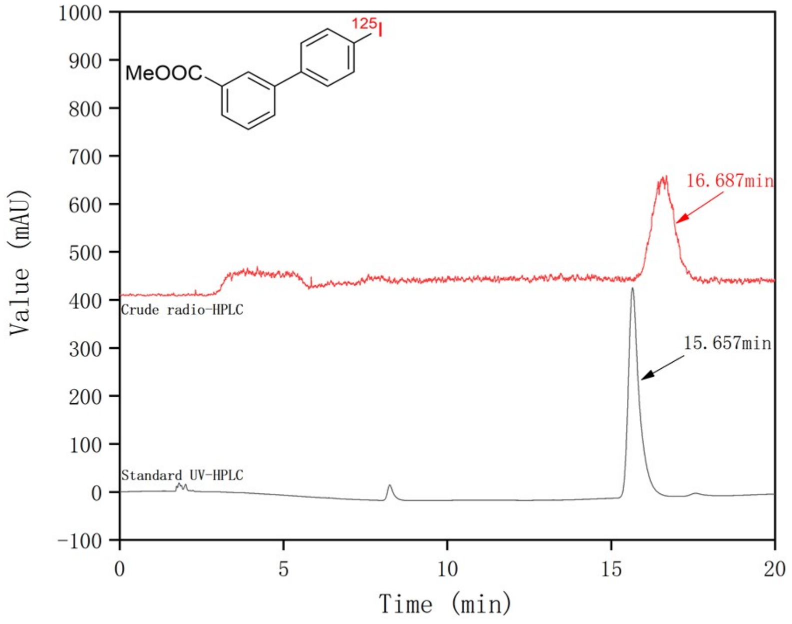
Radio-HPLC traces



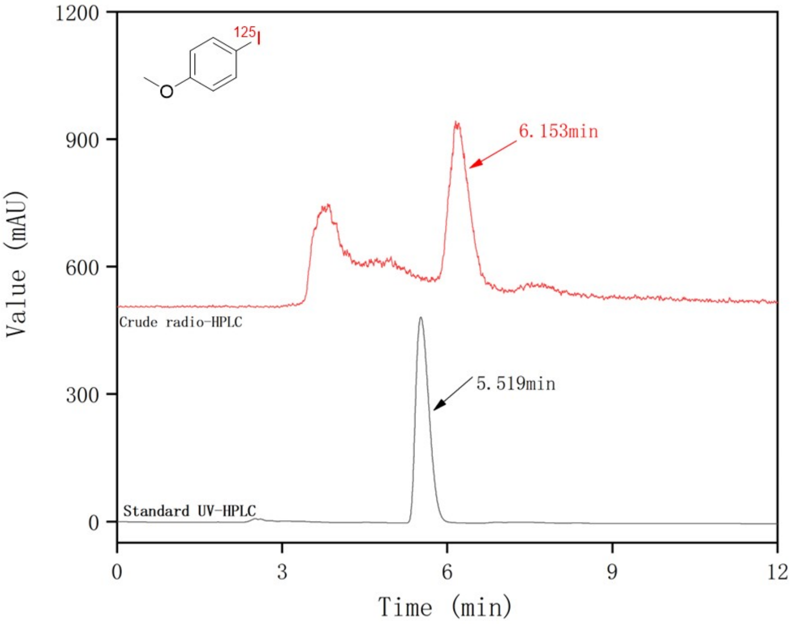
4a (n = 6)
70% MeCN
1.0 mL/min
RCC = 44 ± 3%



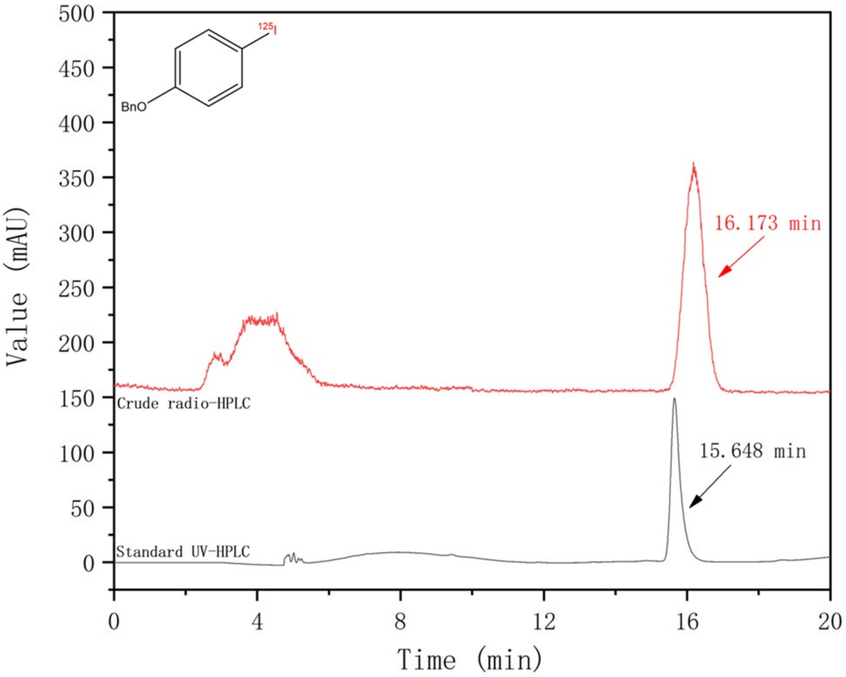
4b (n = 3)
70% MeCN
1.0 mL/min
RCC = 33 ± 3%



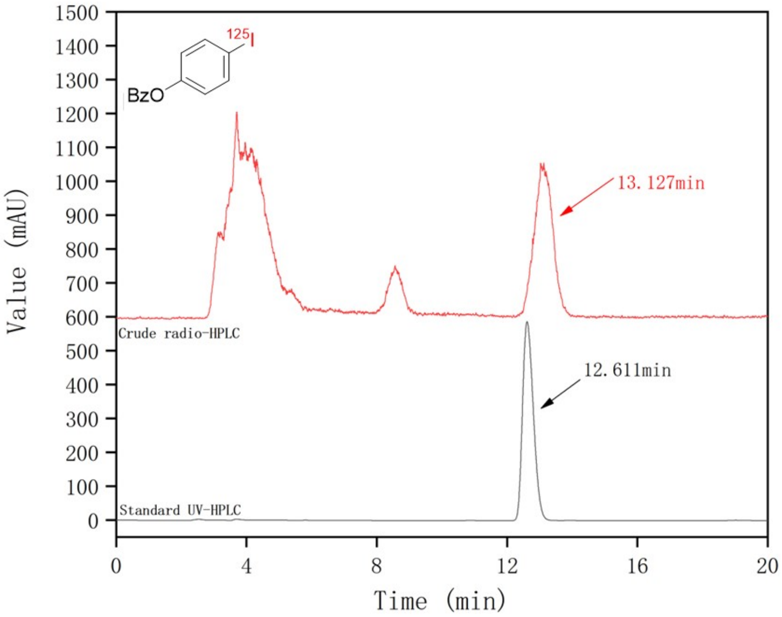
4c (n = 4)
70% MeCN
1.0 mL/min
RCC = 60 ± 2%



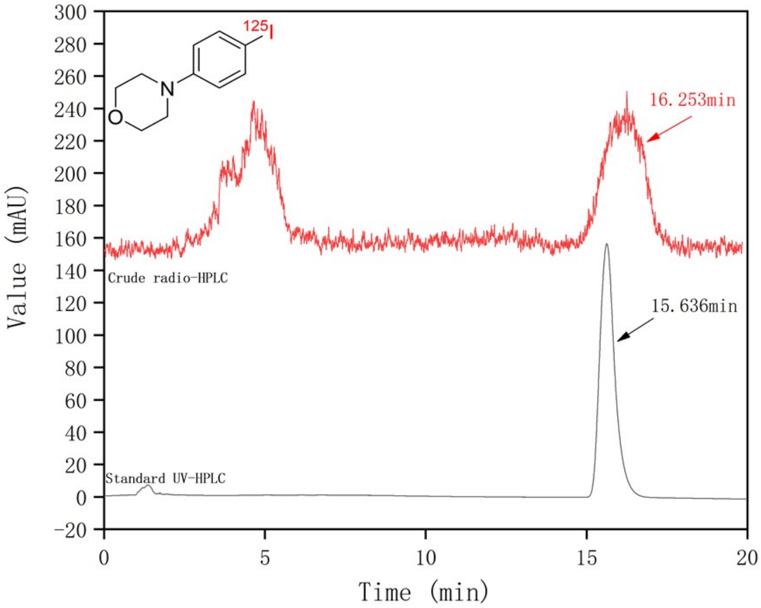
4d (n = 3)
80% MeCN
1.0 mL/min
RCC = 40 ± 4%



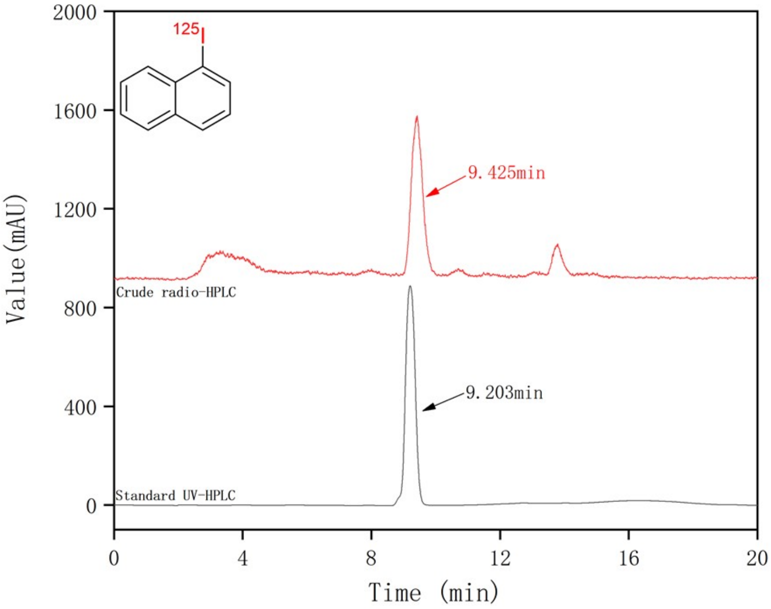
4e (n = 3)
70% MeCN
1.0 mL/min
RCC = 58 ± 9%



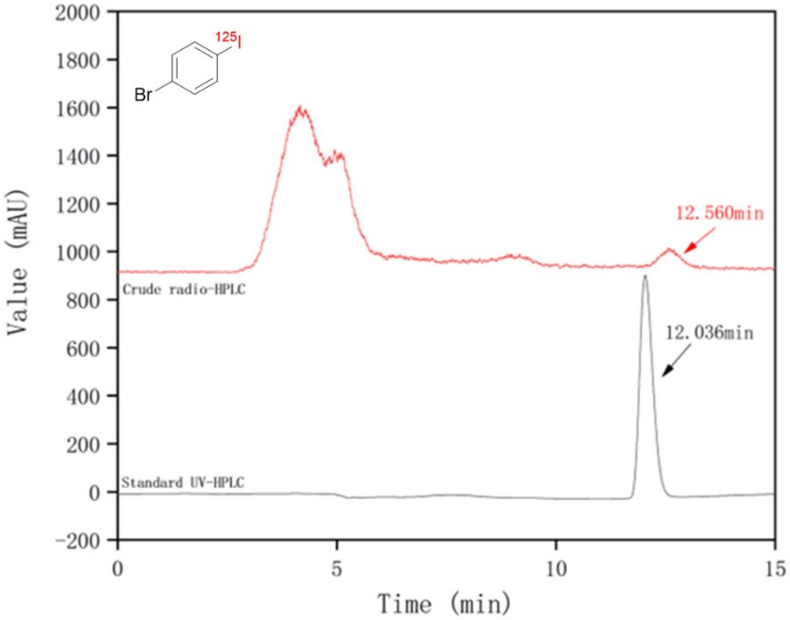
4f (n = 4)
70% MeCN
1.0 mL/min
RCC = 25 ± 2%



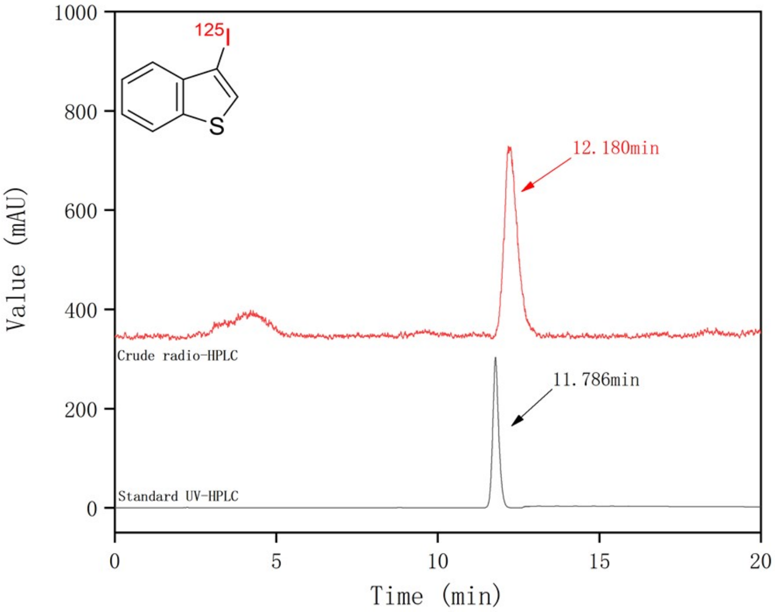
4g (n = 4)
50% MeCN
1.0 mL/min
RCC = 46 ± 4%



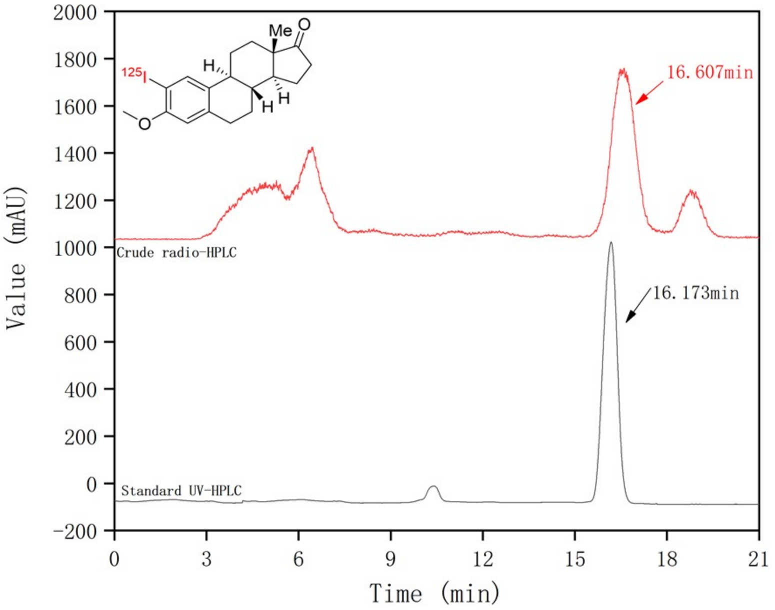
4h (n = 4)
70% MeCN
1.0 mL/min
RCC = 64% ± 6%



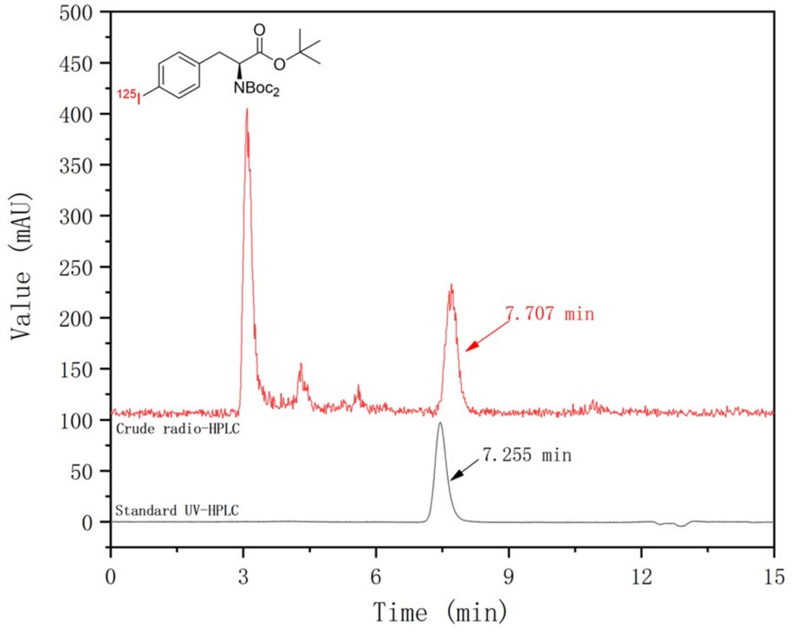
4i (n = 3)
70% MeCN
1.0 mL/min
RCC = 5 ± 2%



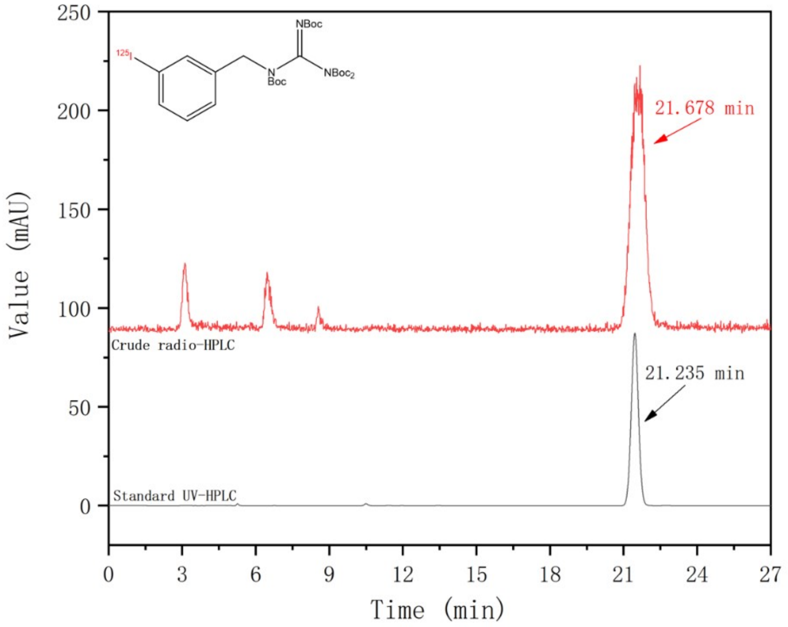
4j (n = 3)
70% MeCN
1.0 mL/min
RCC = 77 ± 5%



4k (n = 3)
70% MeCN
1.0 mL/min
RCC = 41 ± 4%

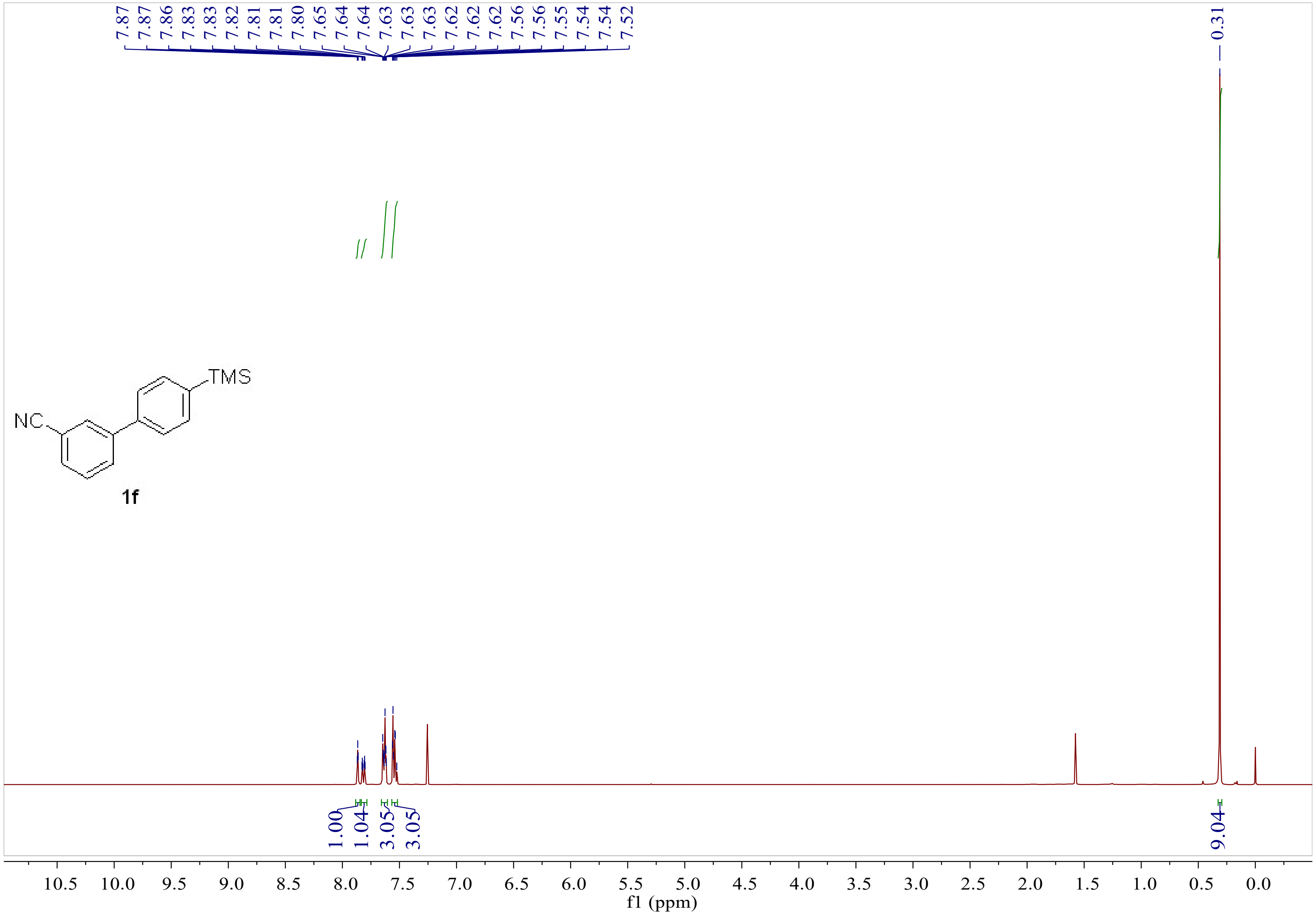


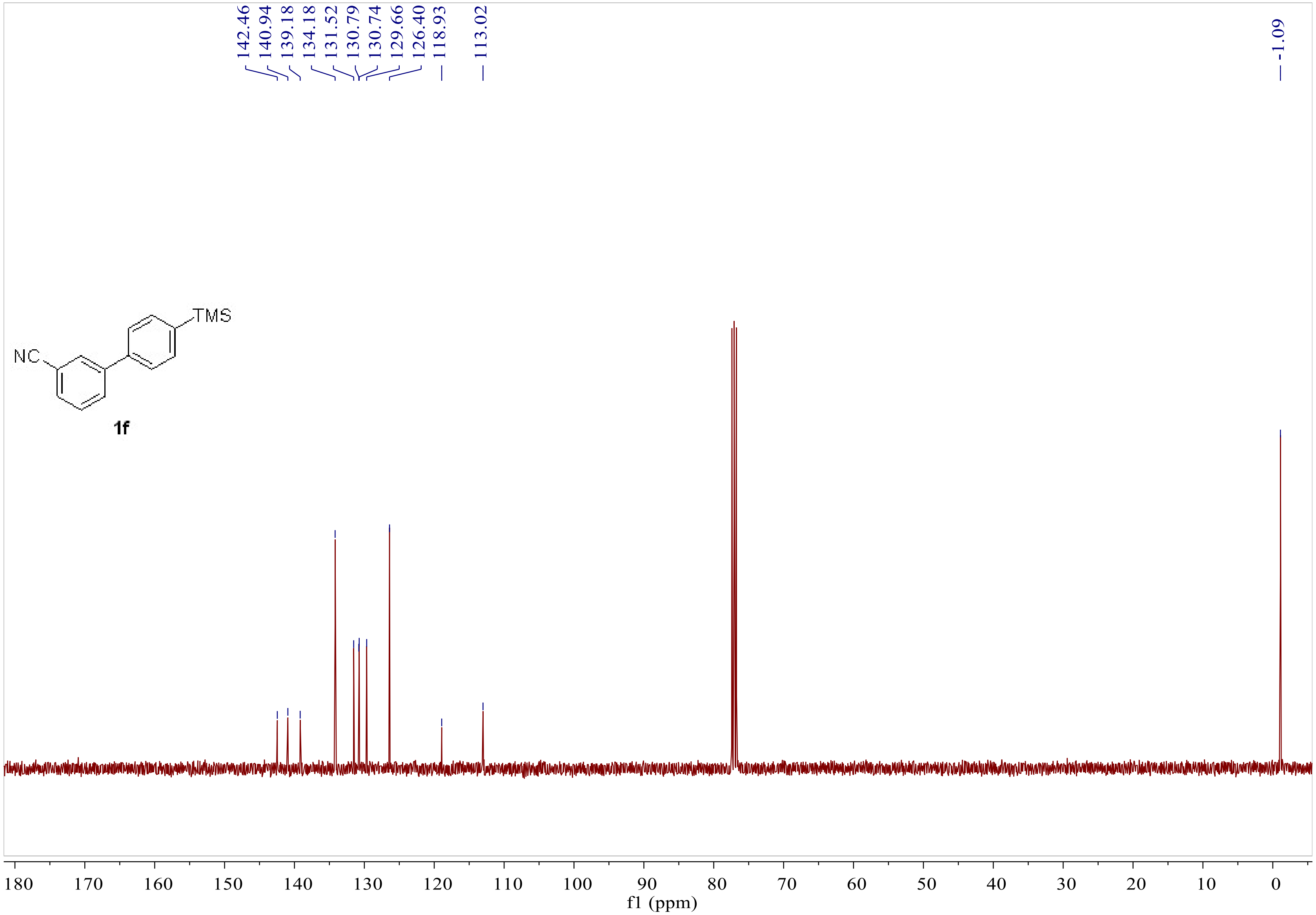
4l (n = 3)
70% MeCN
1.0 mL/min
RCC = 27 ± 8%



4m (n = 3)
50% MeCN
1.0 mL/min
RCC = 75 ± 6%

6. Copies of NMR Spectra.

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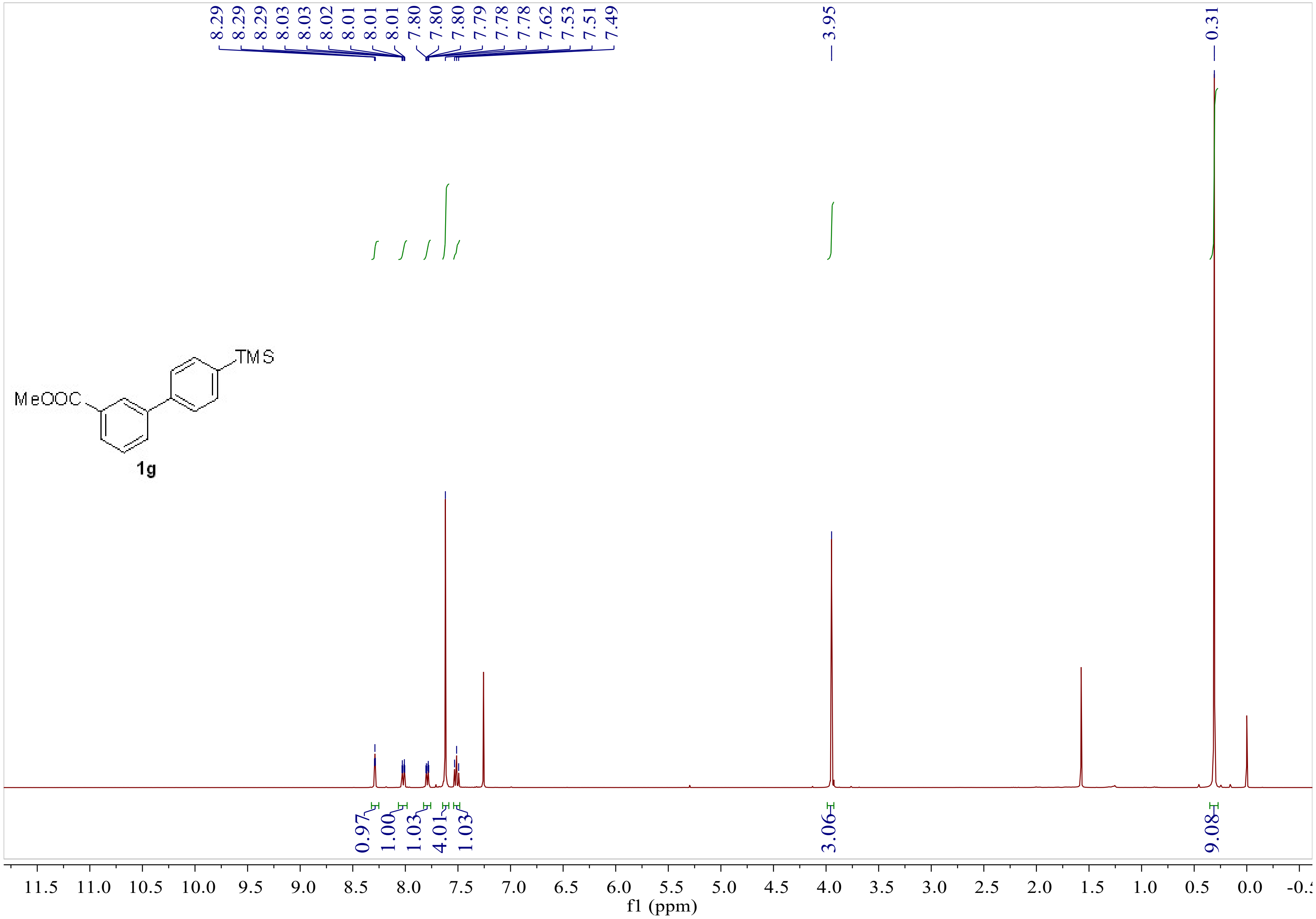
139.18
134.18
131.52
130.79
130.74
129.66
126.40
118.93
113.02
-1.09
NC
TMS
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

1f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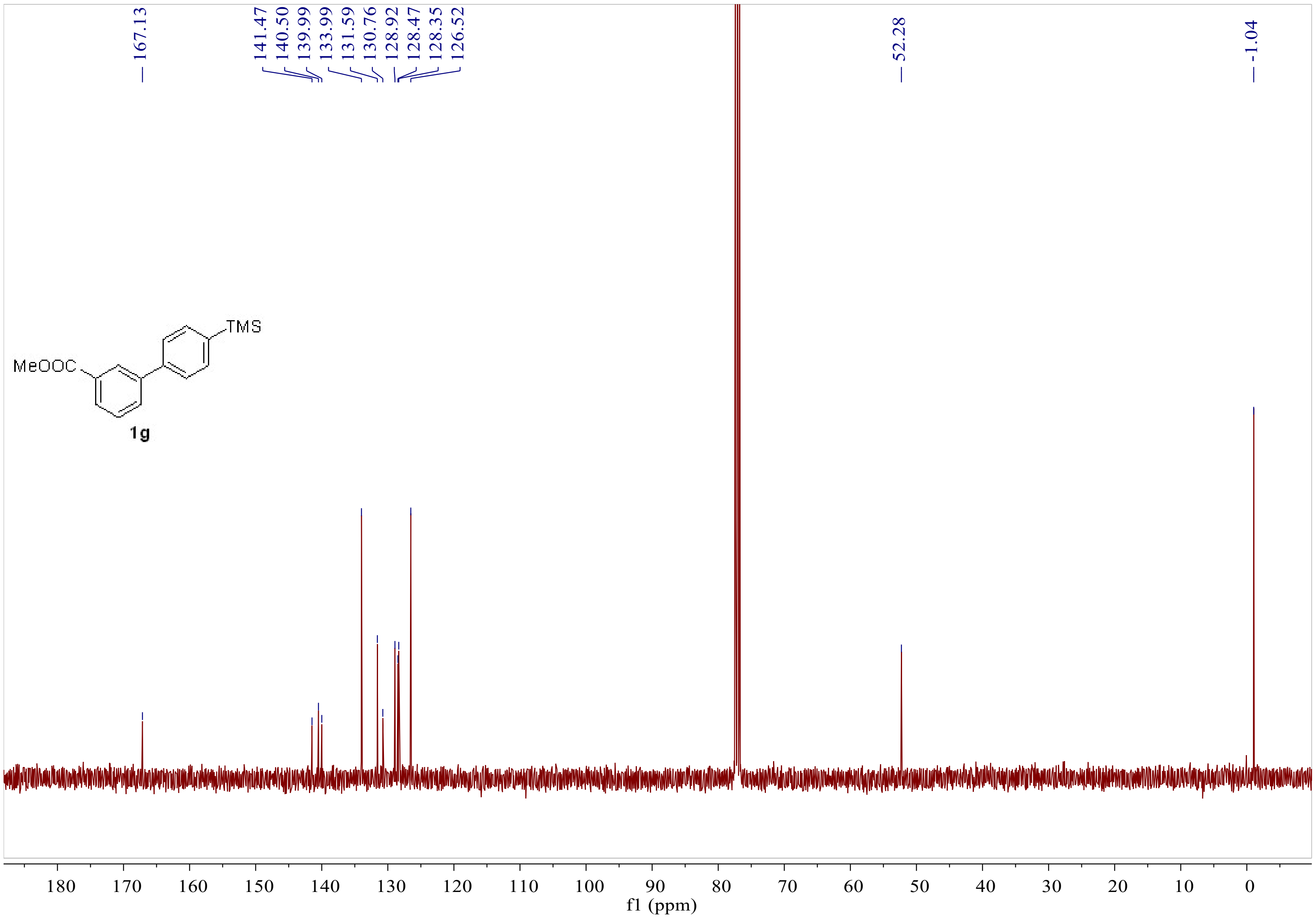
8.29
8.03
8.03
8.02
8.01
8.01
8.01
7.80
7.80
7.80
7.79
7.78
7.78
7.62
7.53
7.51
7.49
3.95
0.31
MeOOC
TMS
1g
0.97
1.00
1.03
4.01
1.03
3.06
9.08
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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139.99
133.99
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128.92
128.47
128.35
126.52
52.28
-1.04
MeOOC
TMS
1g
180
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160
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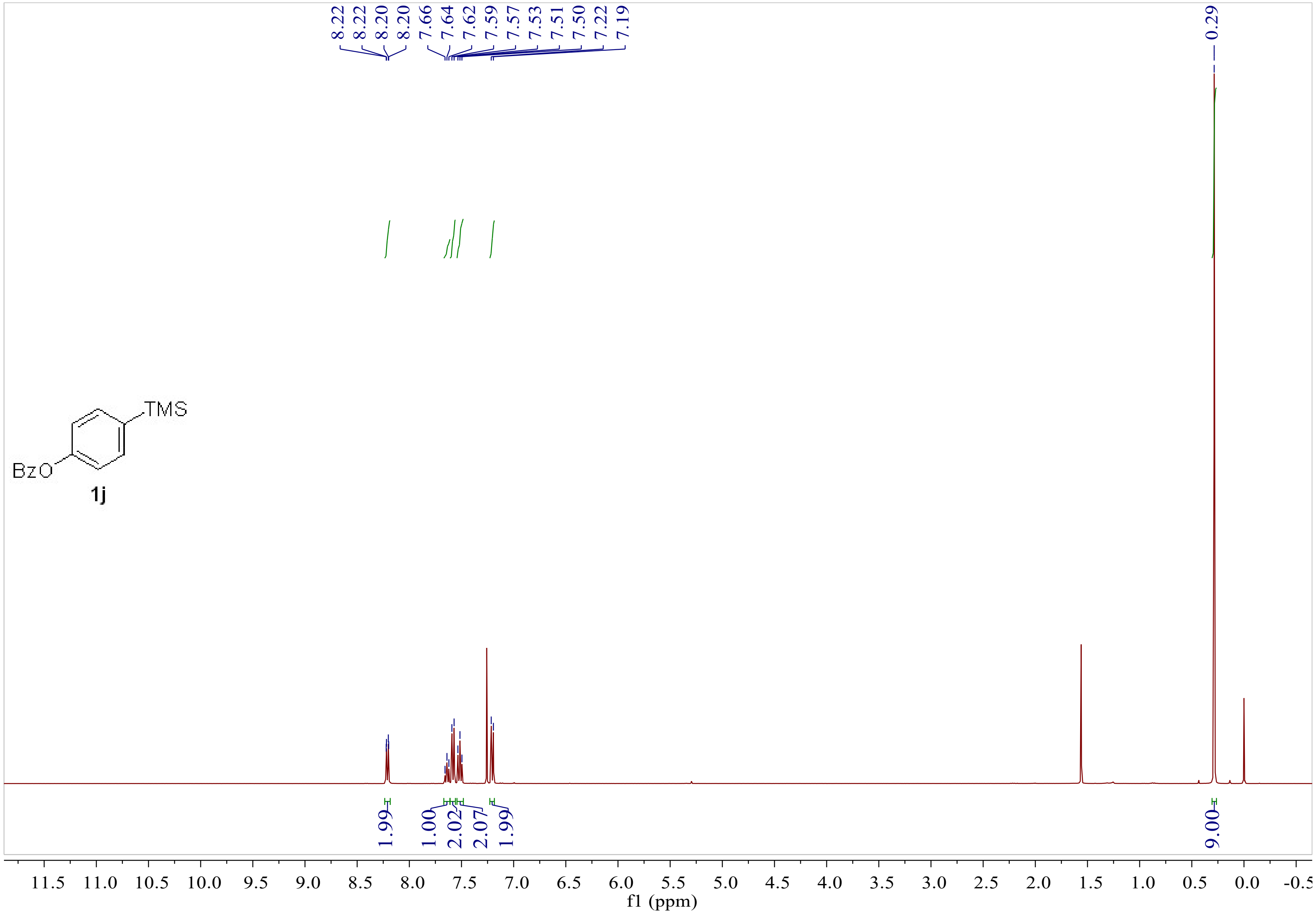
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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8.20
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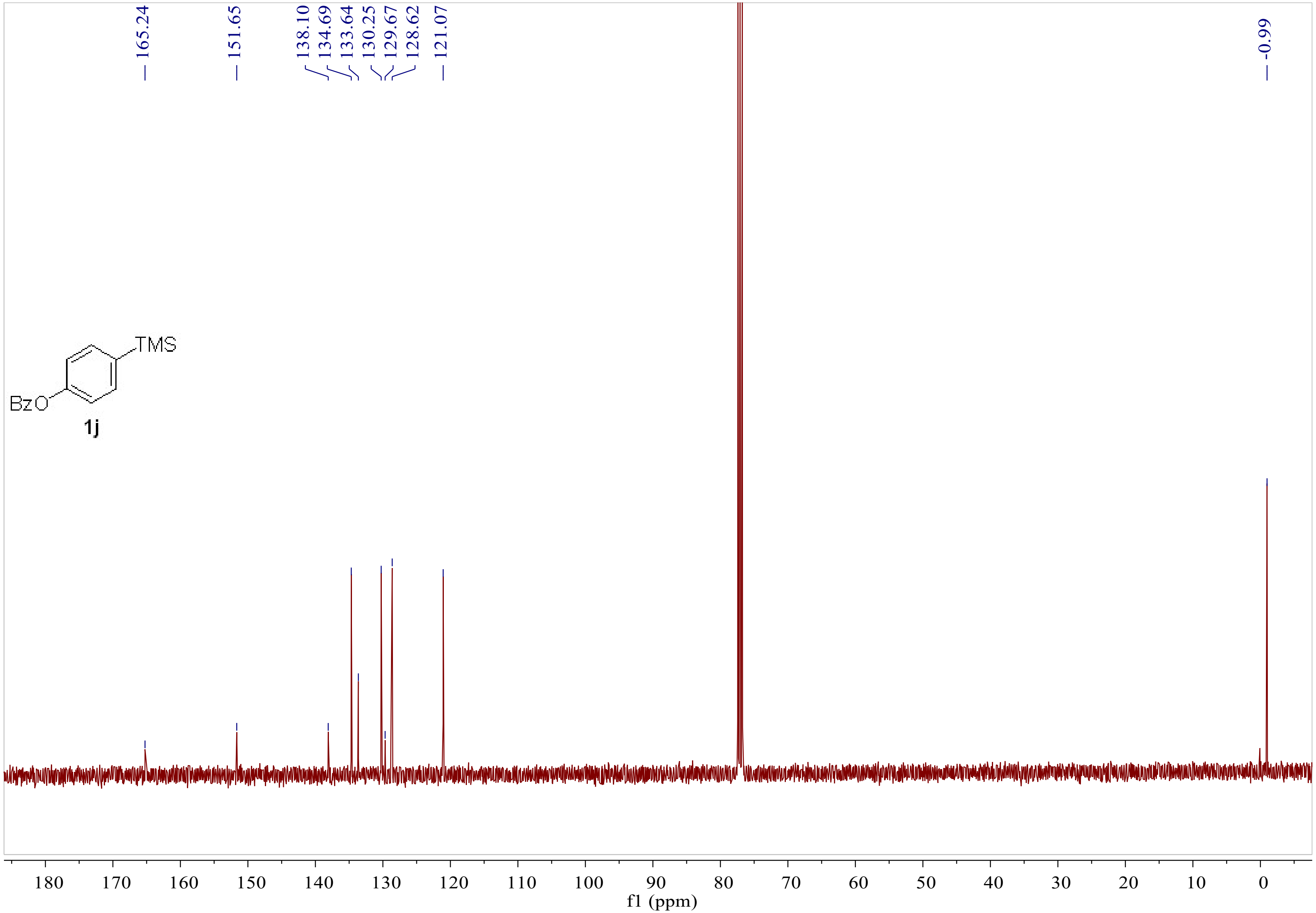
7.64
7.62
7.59
7.57
7.53
7.51
7.50
7.22
7.19
0.29
TMS
BzO
1j
1.99
1.00
2.02
2.07
1.99
9.00
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)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138.10
134.69
133.64
130.25
129.67
128.62
121.07
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BzO
1j
180
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150
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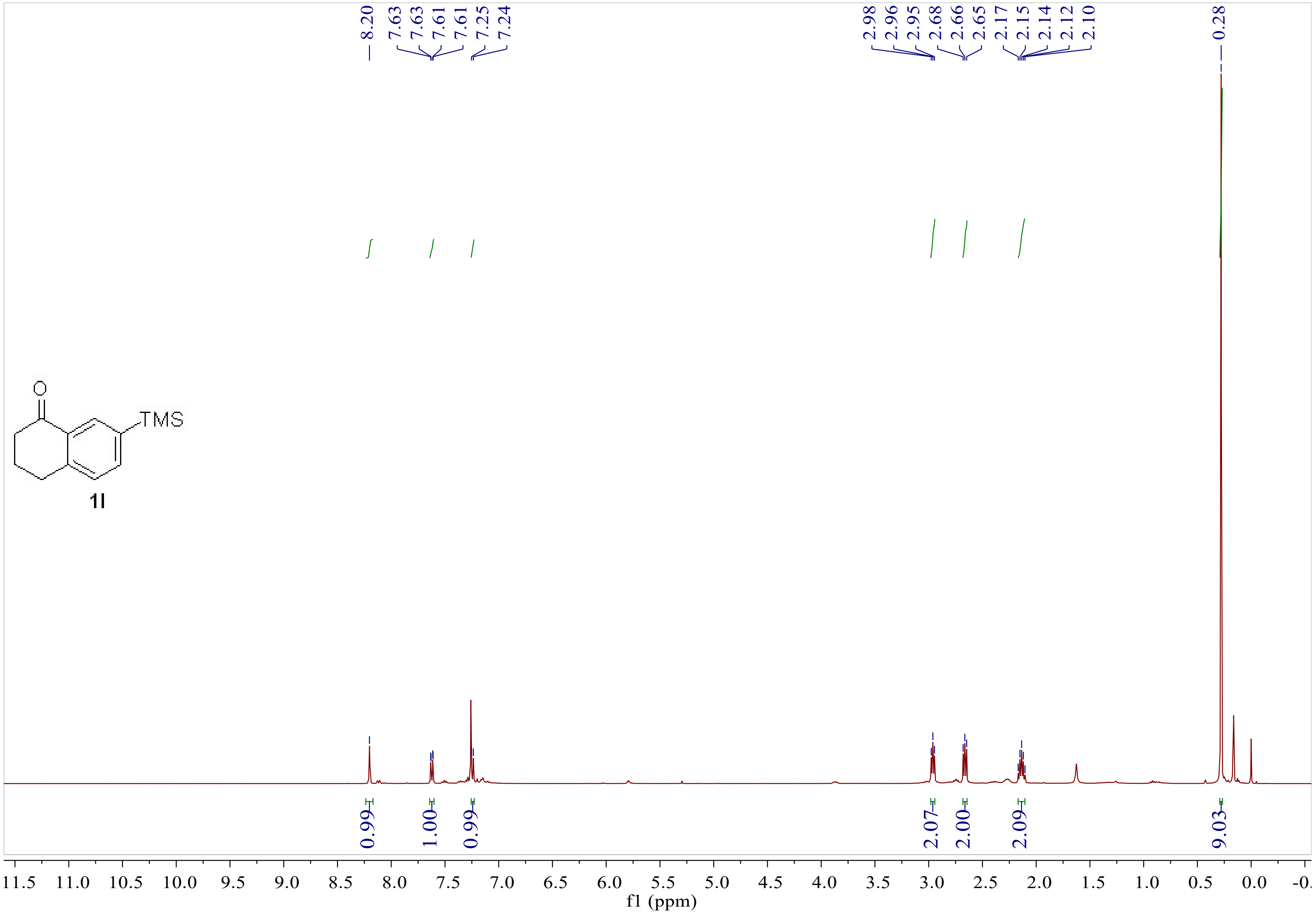
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.61
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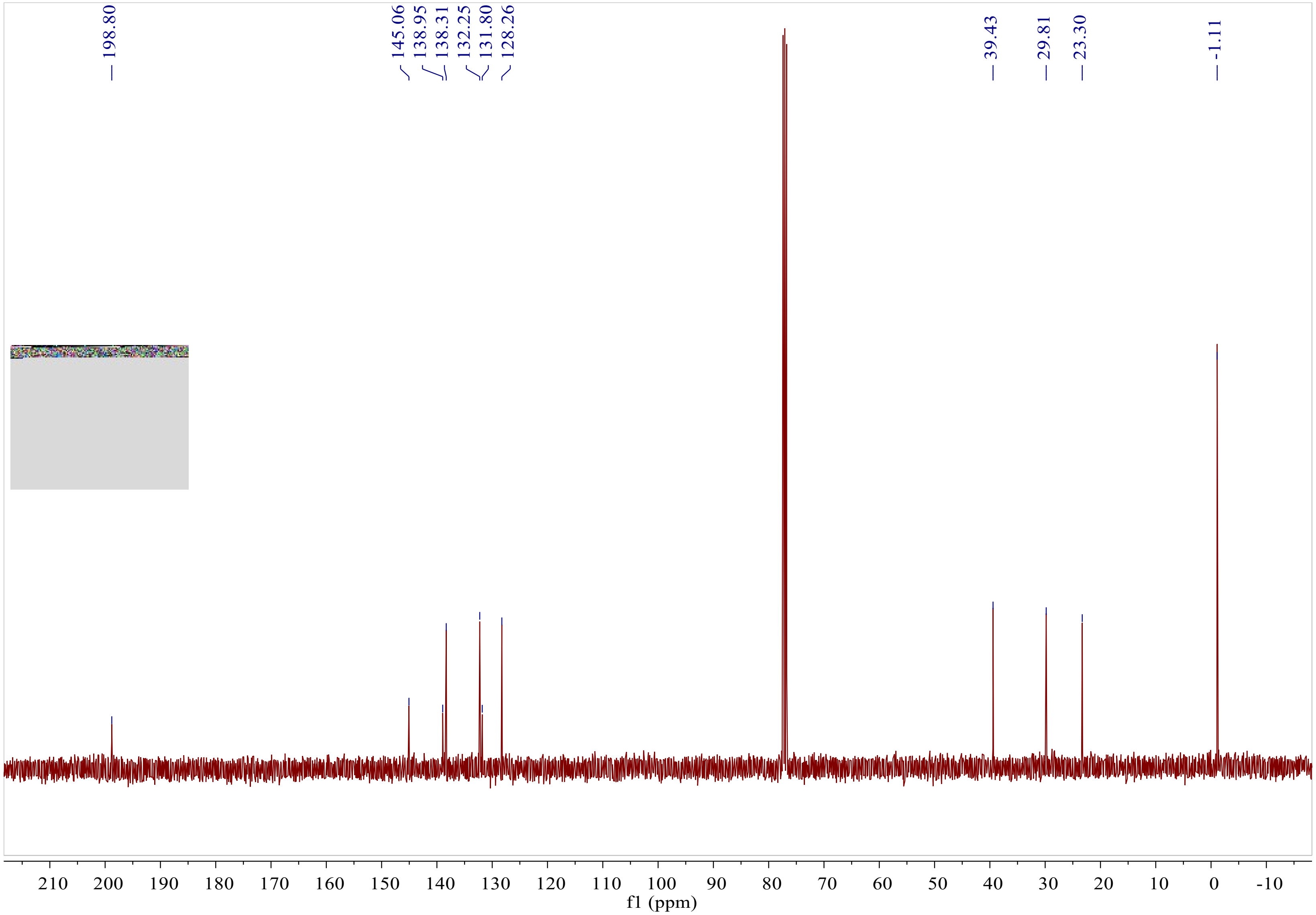
2.96
2.95
2.68
2.66
2.65
2.17
2.15
2.14
2.12
2.10
0.28
O
TMS
1l
0.99
1.00
0.99
2.07
2.00
2.09
9.03
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.
f1 (ppm)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128.26
39.43
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200
190
180
170
160
150
140
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120
110
100
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80
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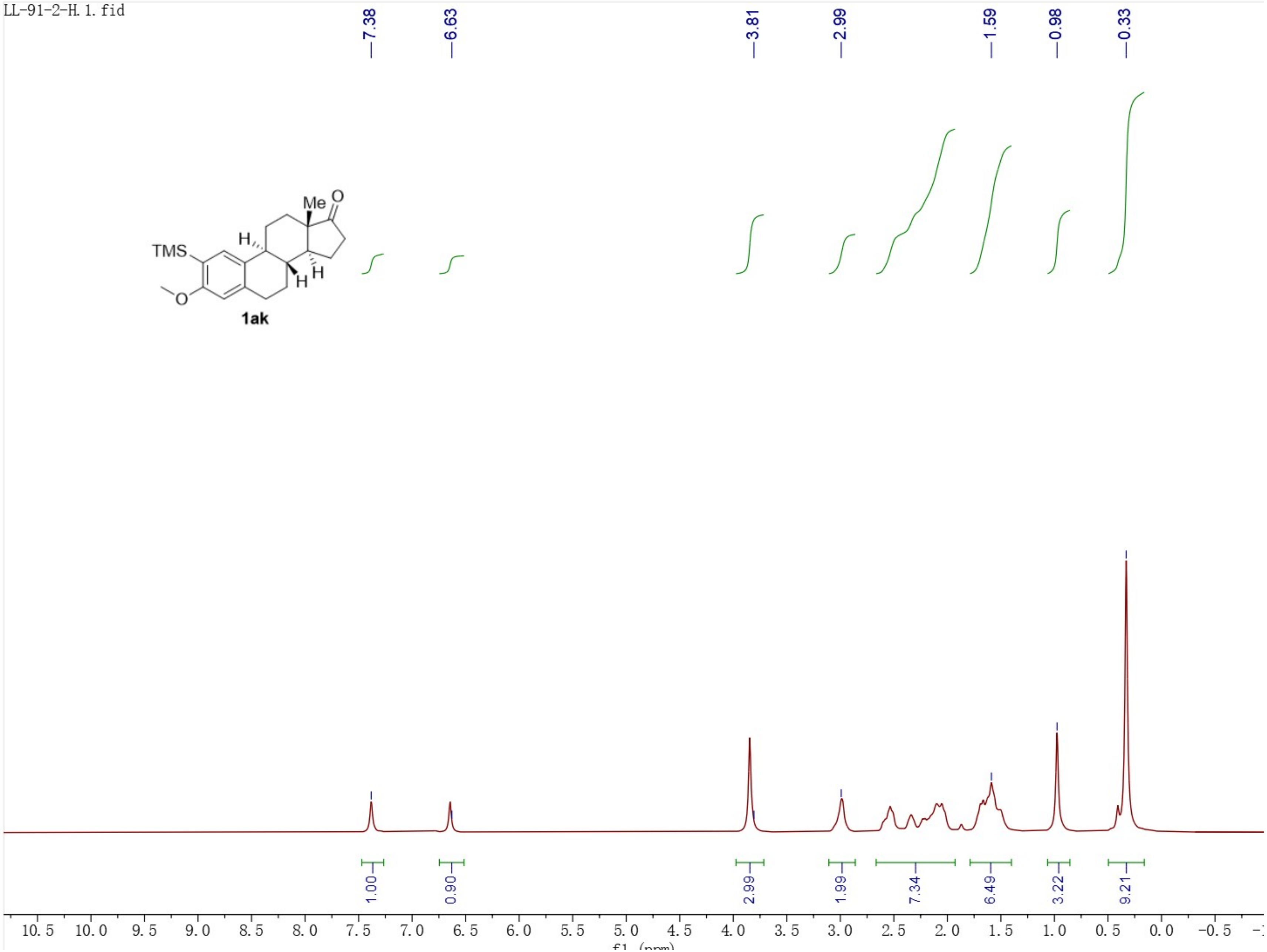
60
50
40
30
20
10
0
-10
f1 (ppm)


1ak

LL-91-2-H.1.fid
TMS
O
H
H
H
Me
O
7.38
6.63
3.81
2.99
1.59
0.98
0.33
1.00
0.90
2.99
1.99
7.34
6.49
3.22
9.21
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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3.0
2.5
2.0
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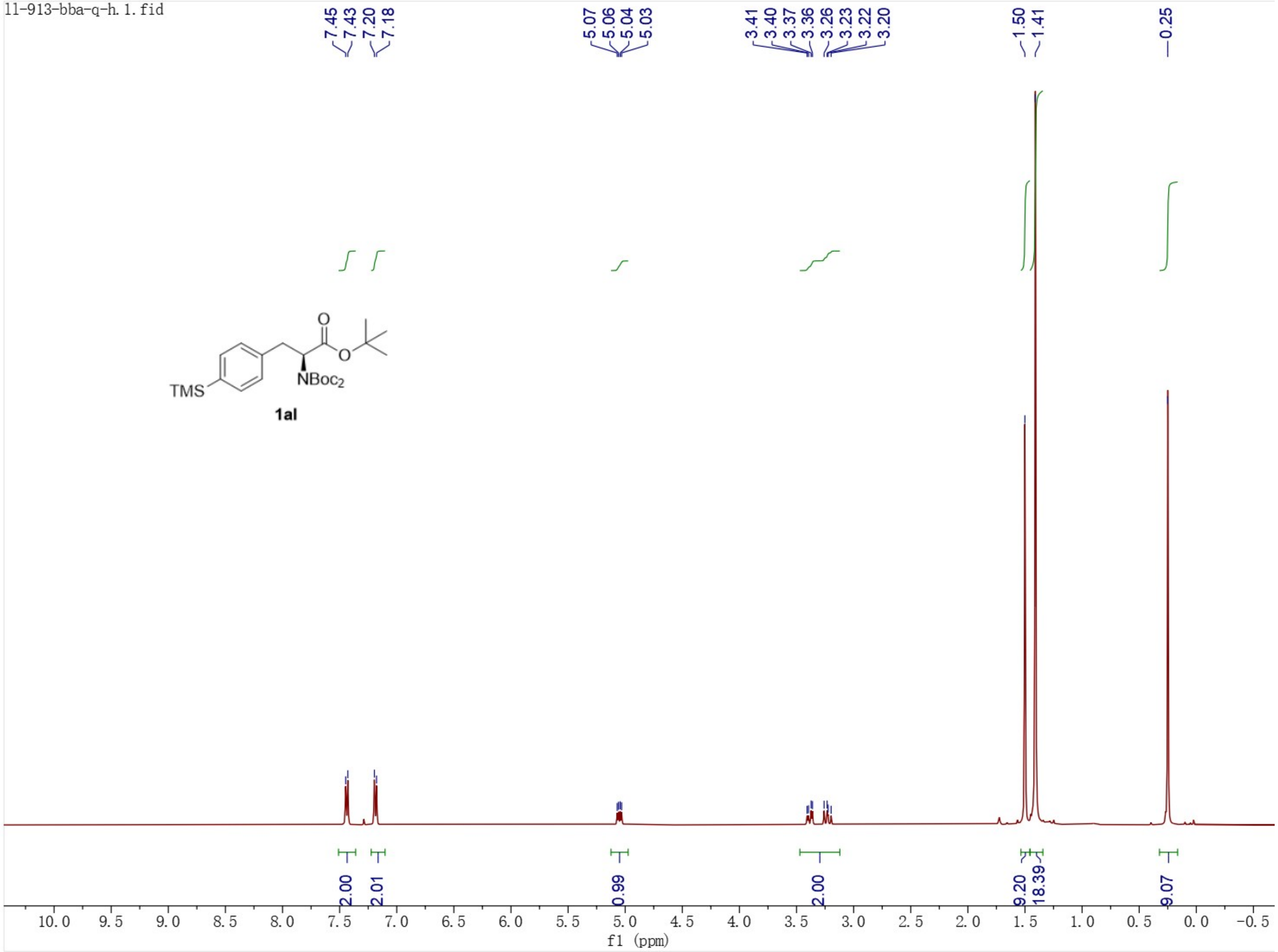
1.0
0.5
0.0
-0.5
f1 (ppm)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5.06
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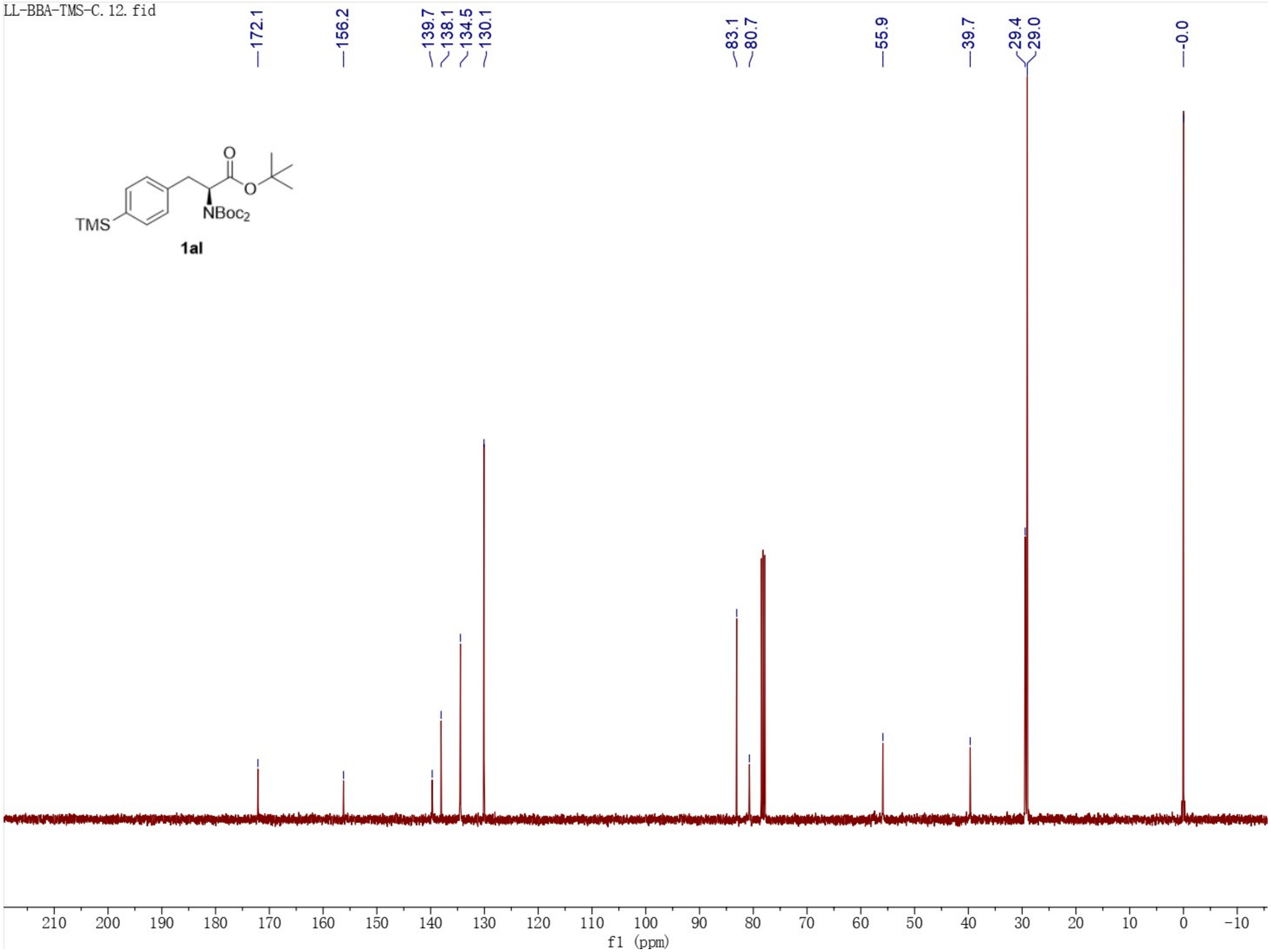
5.03
3.41
3.40
3.37
3.36
3.26
3.23
3.22
3.20
1.50
1.41
0.25
TMS
O
O
NBoc$_2$
1al
2.00
2.01
0.99
2.00
9.20
18.39
9.07
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)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156.2
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134.5
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83.1
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55.9
39.7
29.4
29.0
0.0
O
O
NBoc$_2$
TMS
1al
210
200
190
180
170
160
150
140
130
120
110
100
90
80
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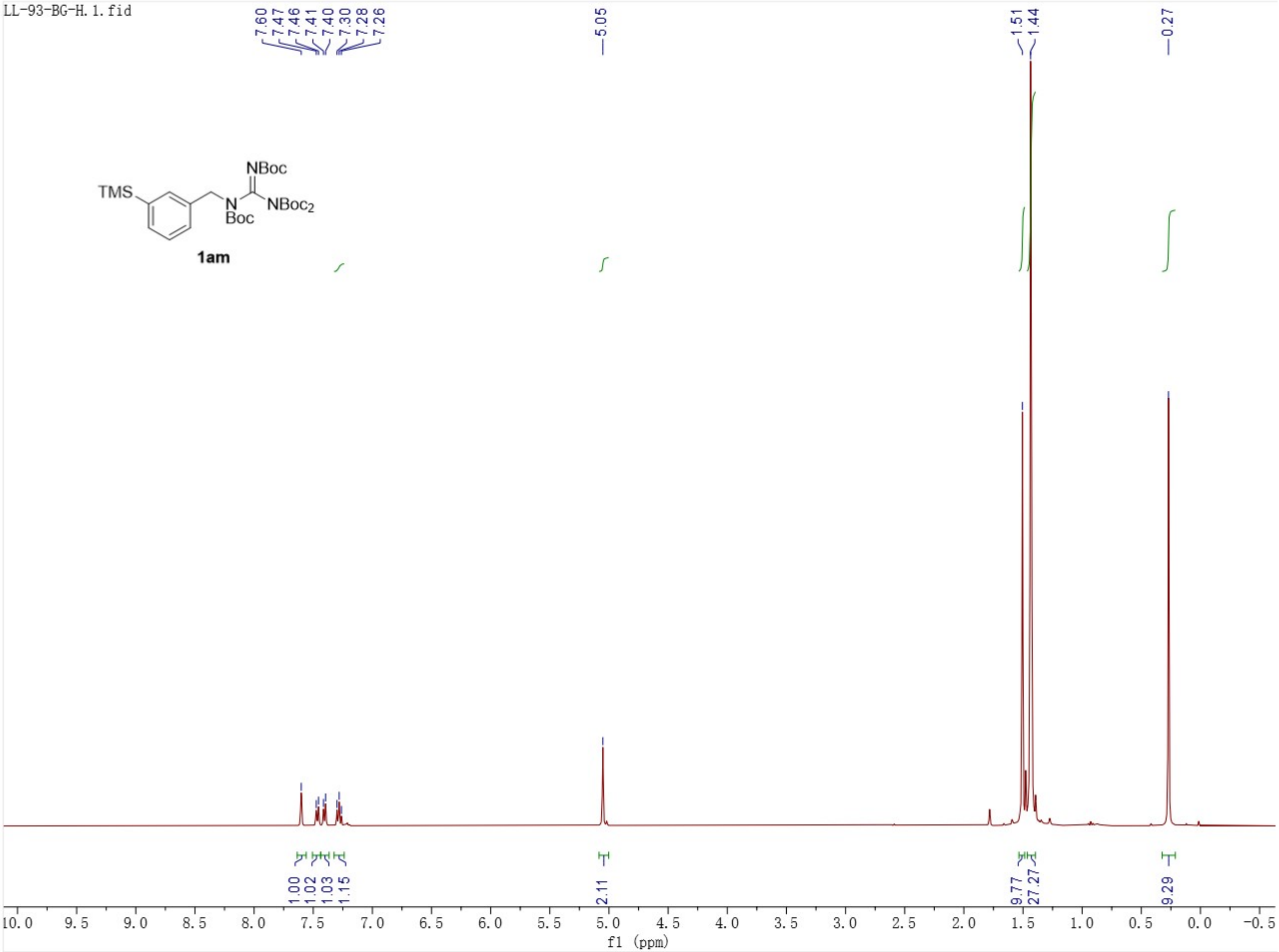
60
50
40
30
20
10
0
−10
f1 (ppm)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1.44
0.27
TMS
NBoc
N
Boc
NBoc2
1.00
1.02
1.03
1.15
2.11
9.77
27.27
9.29
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
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2.5
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1.5
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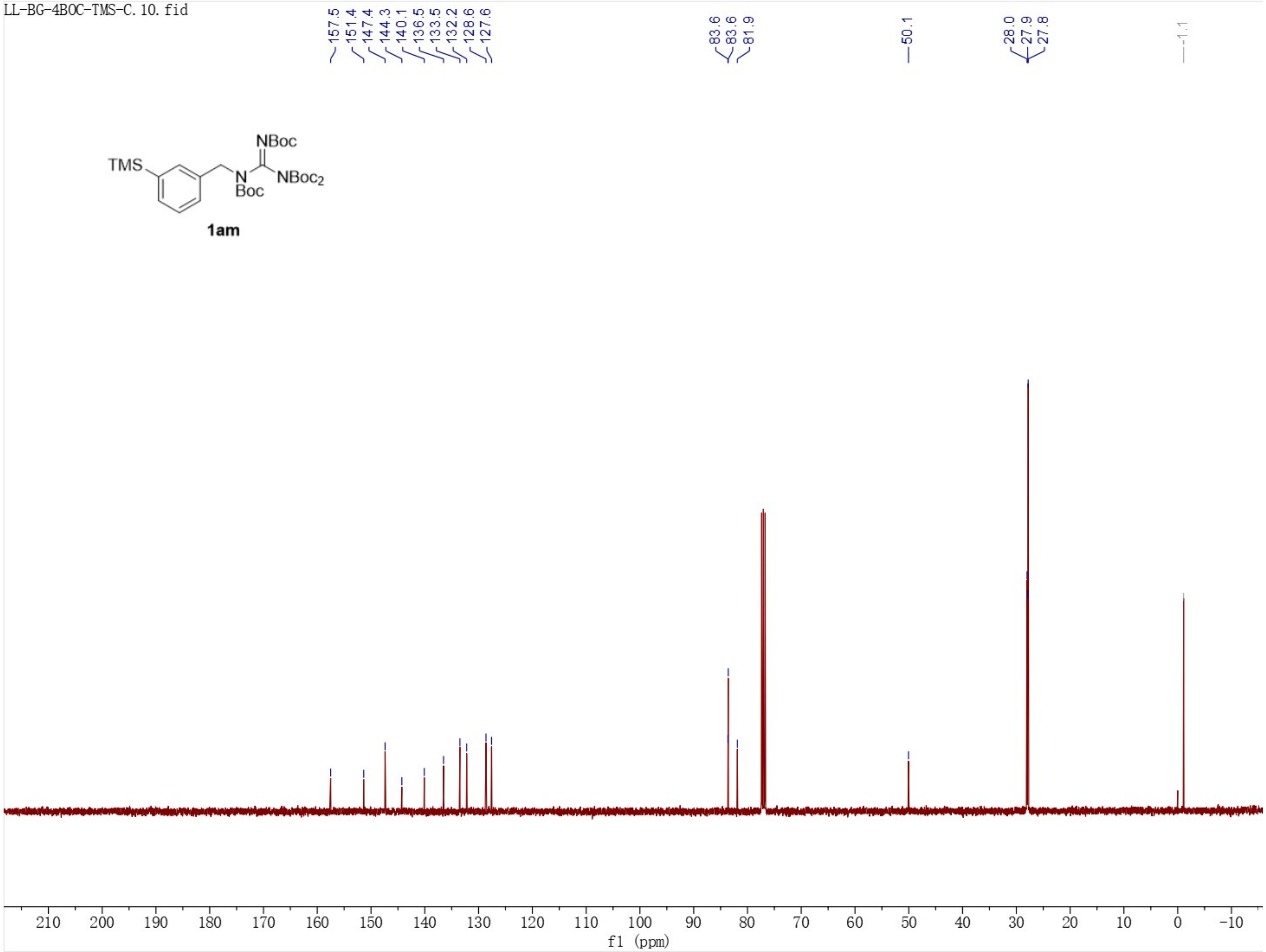
f1 (ppm)

1am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147.4
144.3
140.1
136.5
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127.6
83.6
83.6
81.9
50.1
28.0
27.9
27.8
-1.1
TMS
NBoc
N
NBoc2
Boc
1am
210
200
190
180
170
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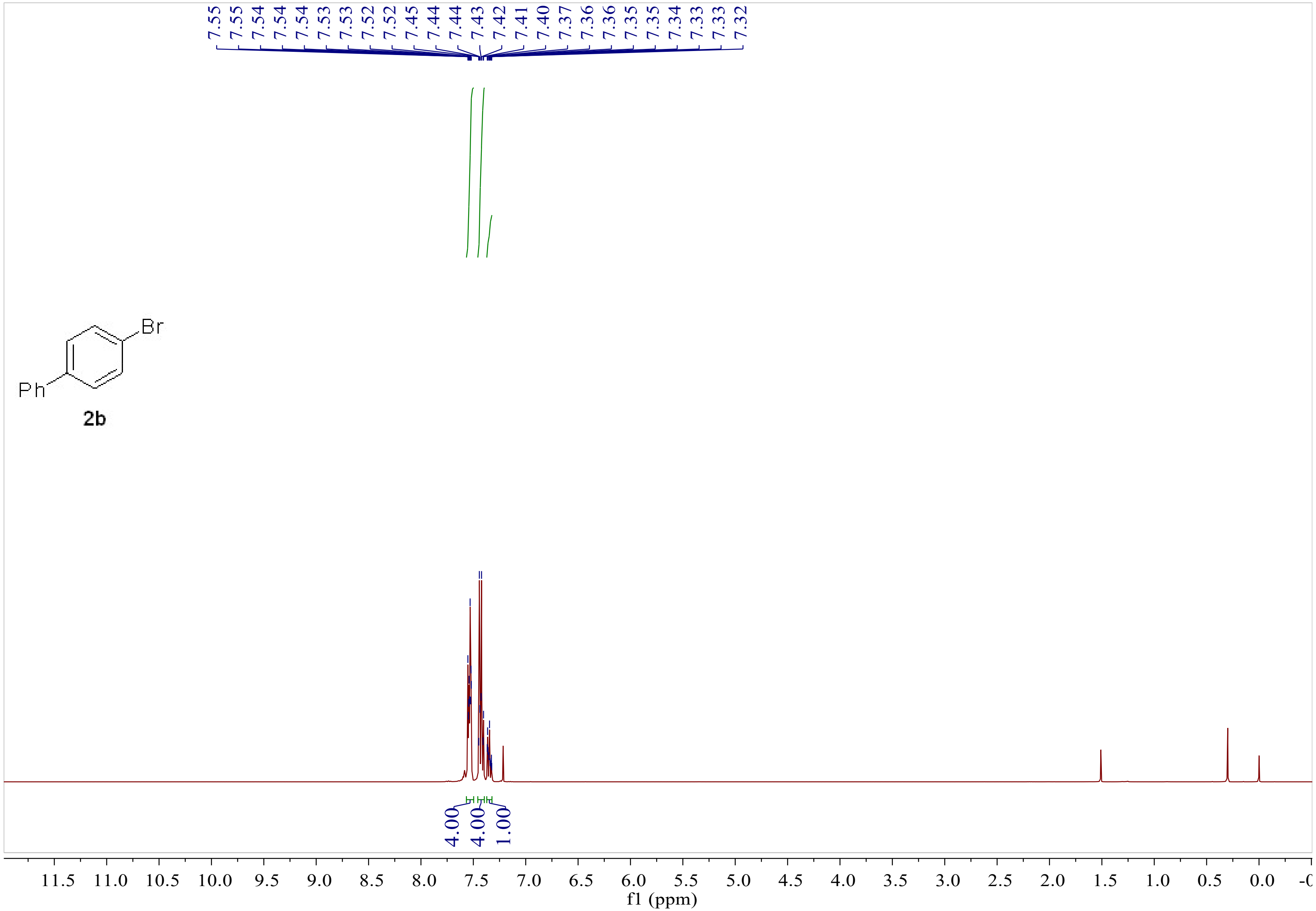
f1 (ppm)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7.42
7.41
7.40
7.37
7.36
7.36
7.35
7.35
7.34
7.33
7.33
7.32
Br
Ph
2b
4.00
4.00
1.00
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
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6.0
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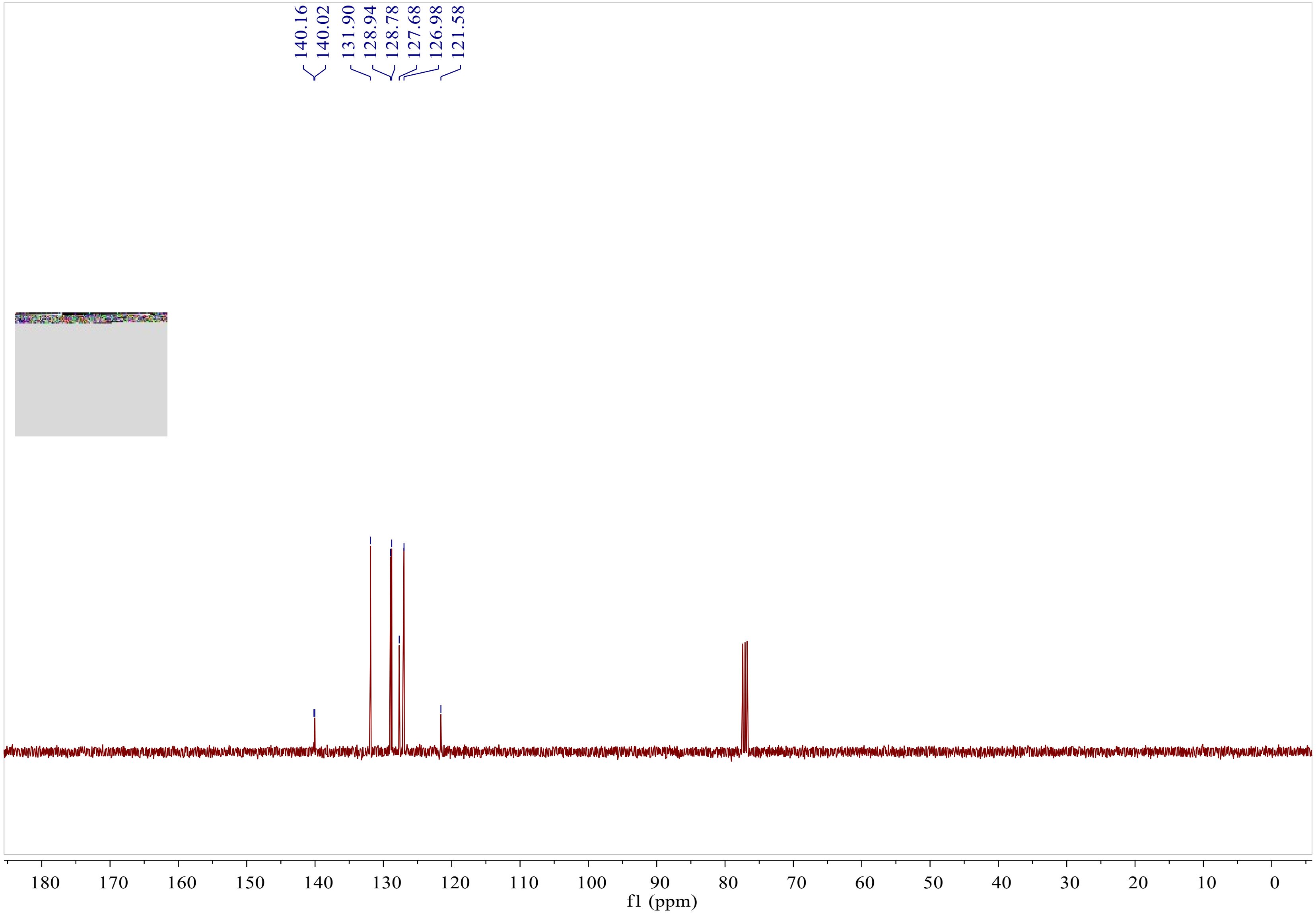
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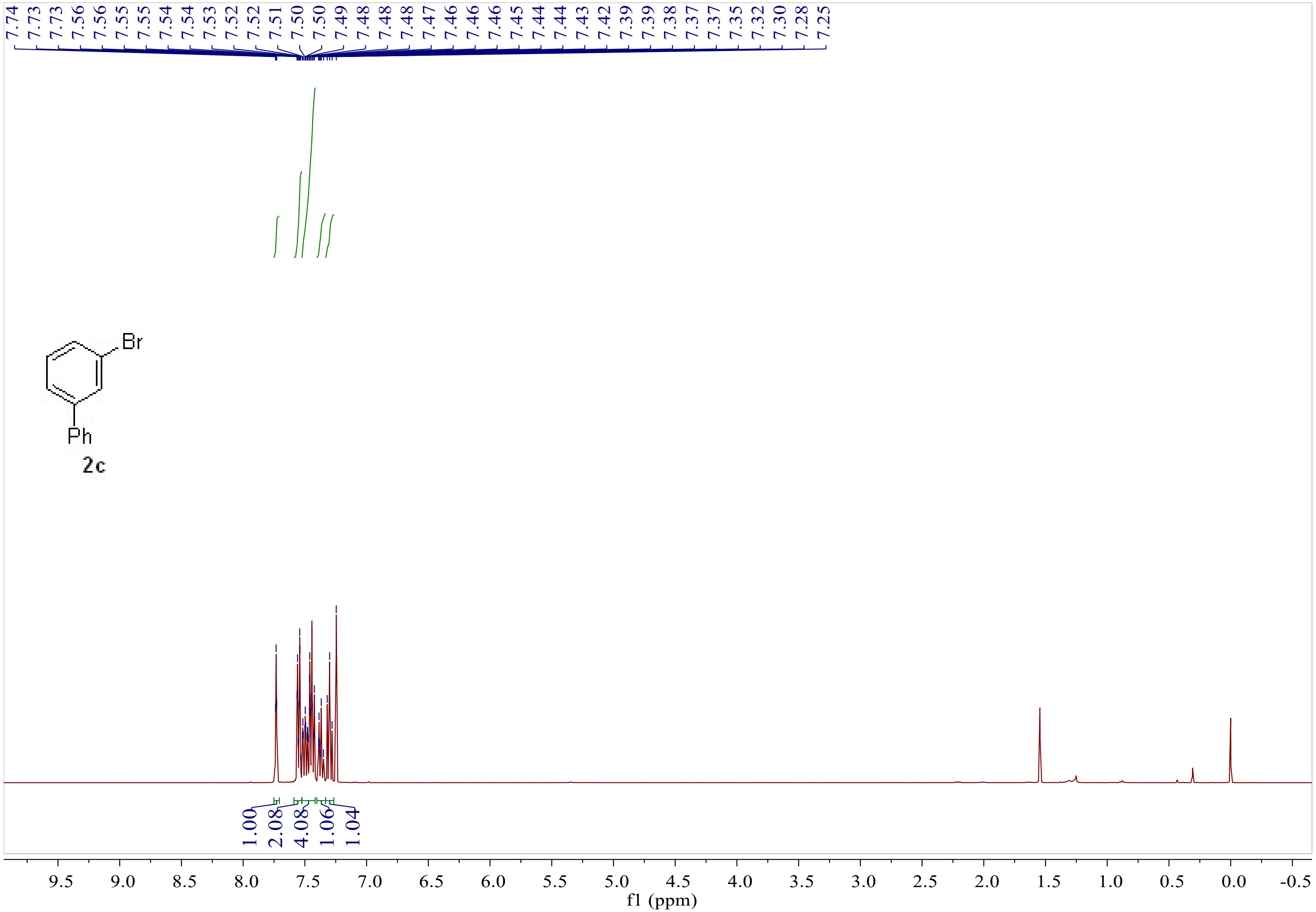
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7.44
7.43
7.42
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7.39
7.38
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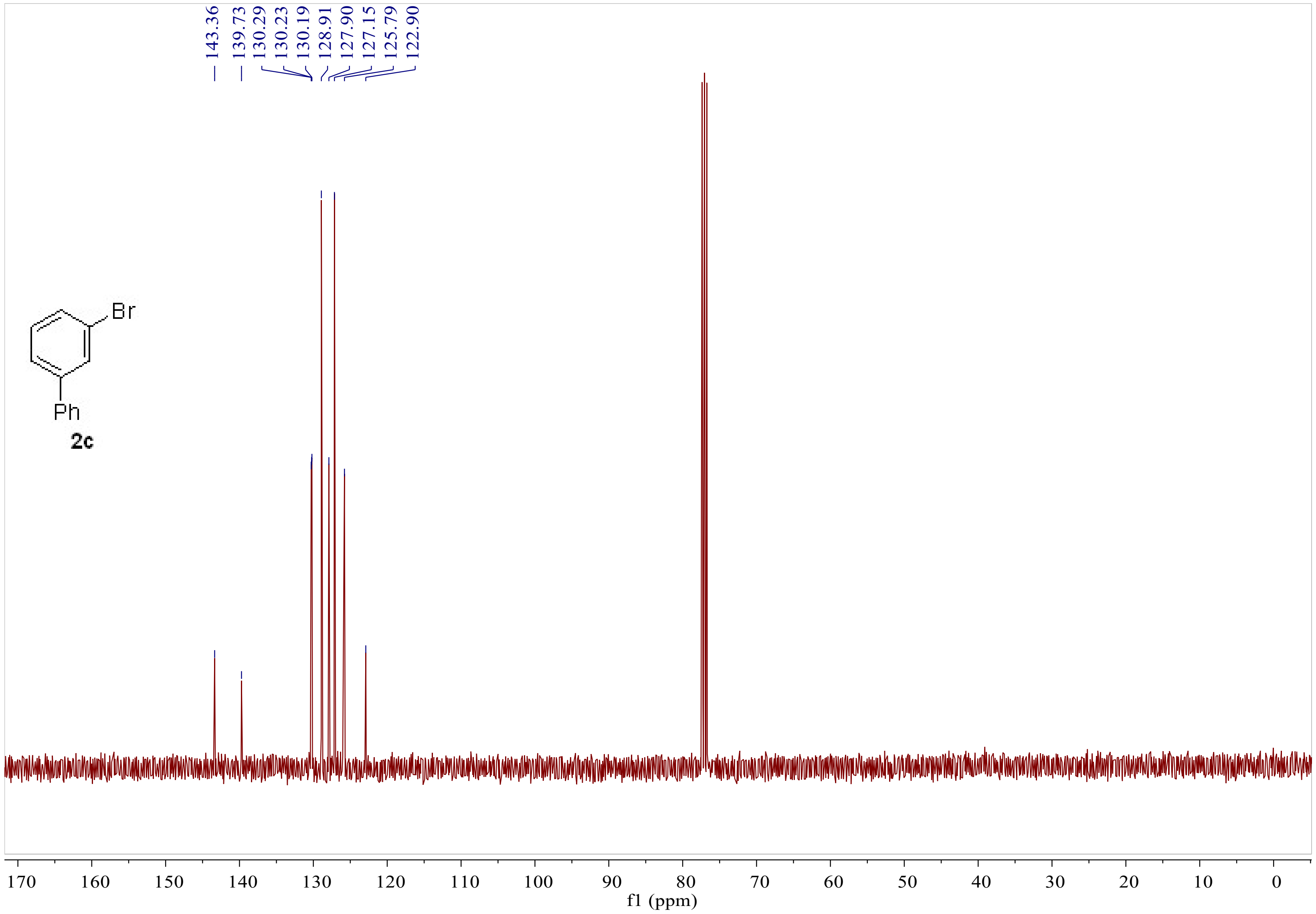
f1 (ppm)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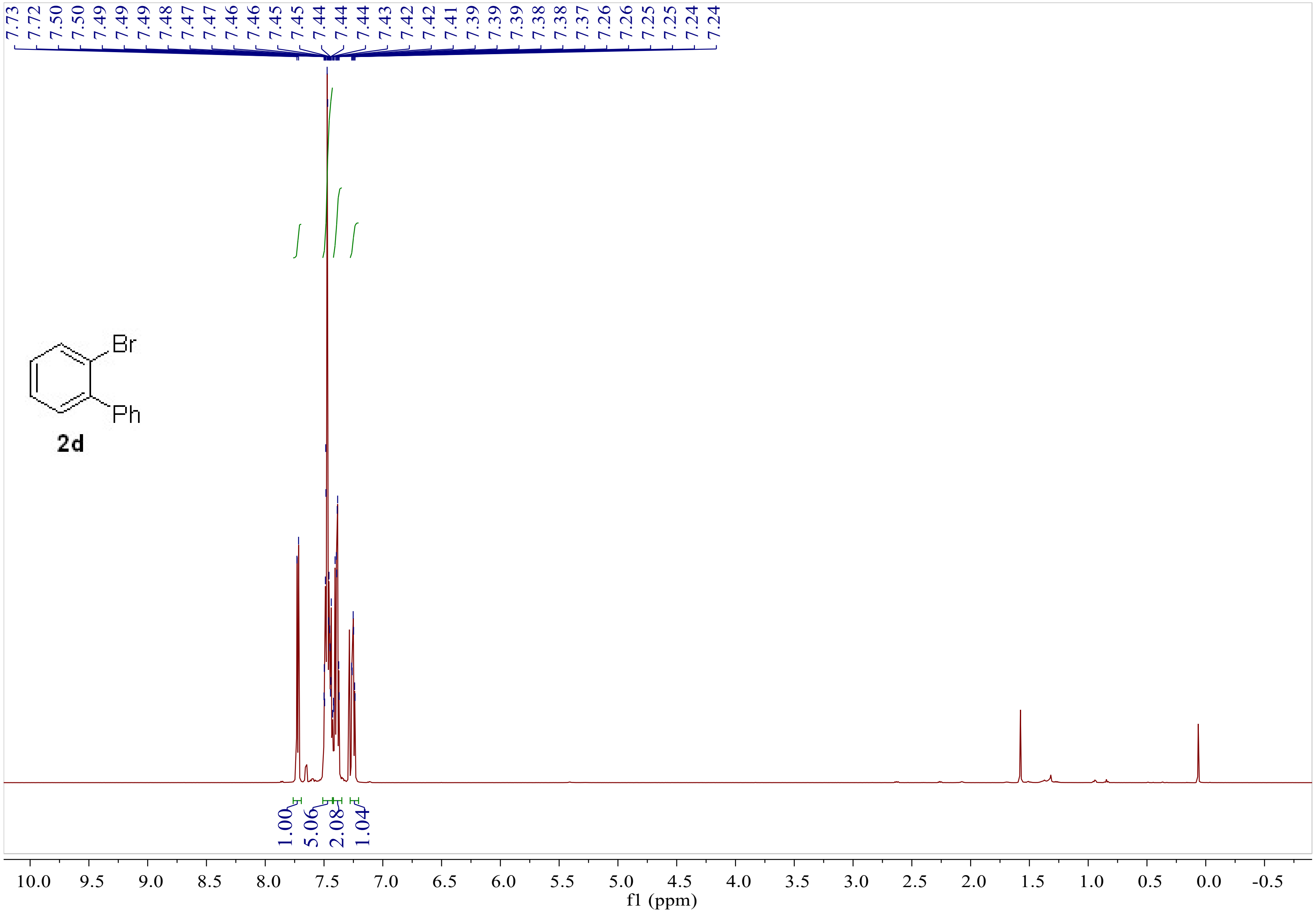
f1 (ppm)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7.44
7.44
7.44
7.44
7.43
7.42
7.42
7.41
7.39
7.39
7.39
7.38
7.38
7.37
7.26
7.26
7.25
7.25
7.24
7.24
Br
Ph
2d
1.00
5.06
2.08
1.04
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
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0.5
0.0
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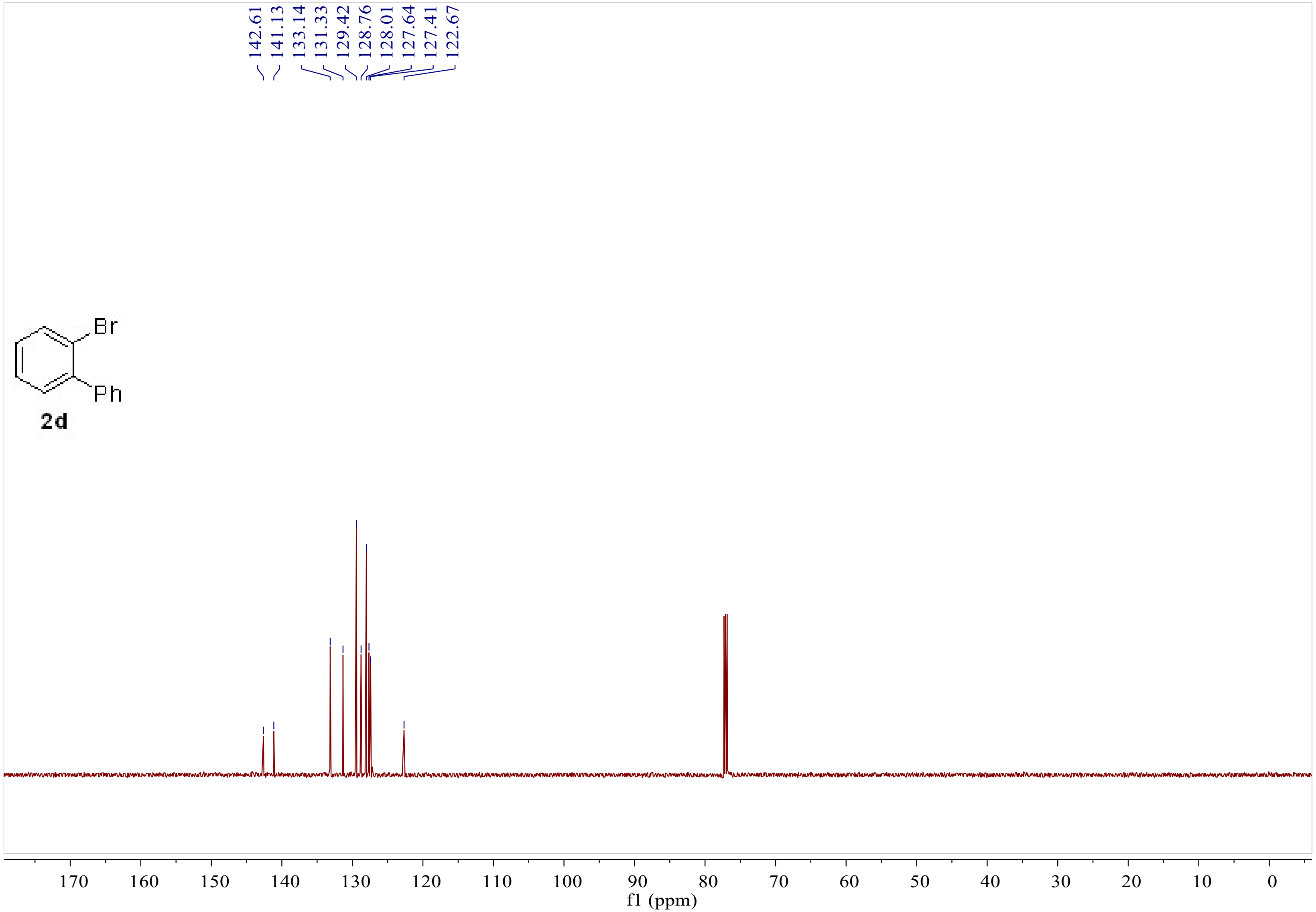
f1 (ppm)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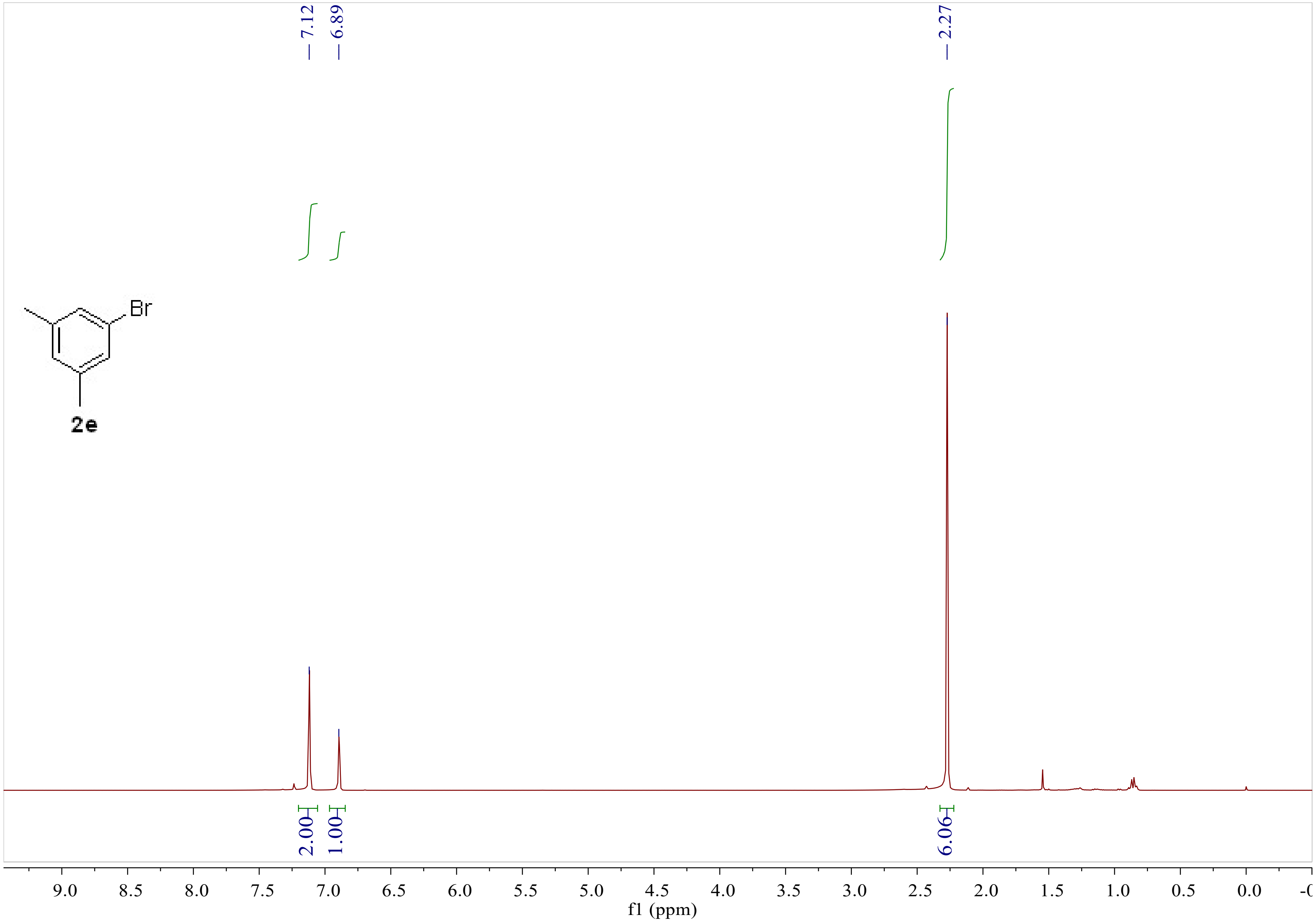
133.14
131.33
129.42
128.76
128.01
127.64
127.41
122.67
Br
Ph
2d
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



7.12
6.89
2.27
Br
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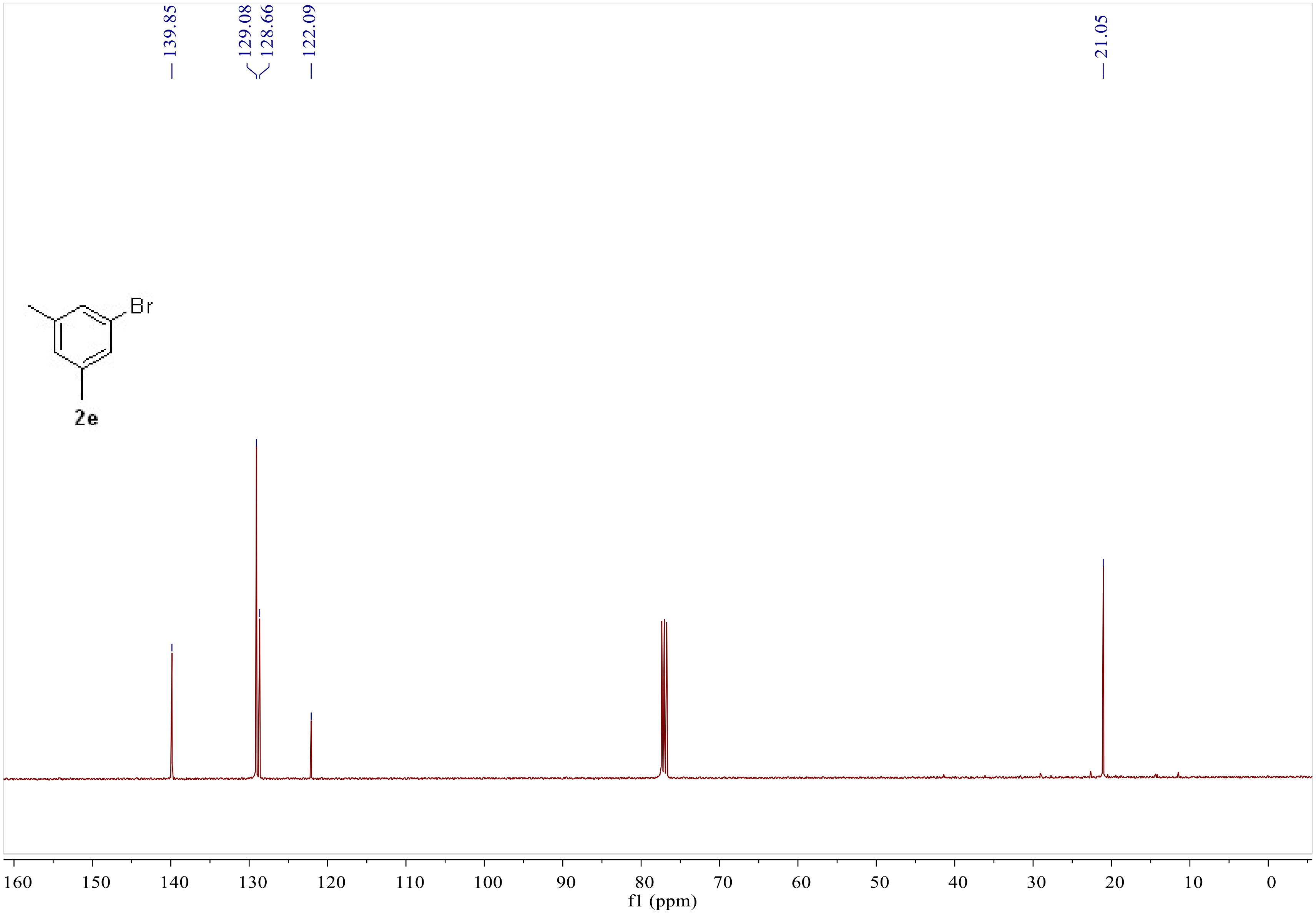
1.00
6.06
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

2e


139.85
129.08
128.66
122.09
21.05
Br
2e
160
150
140
130
120
110
100
90
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70
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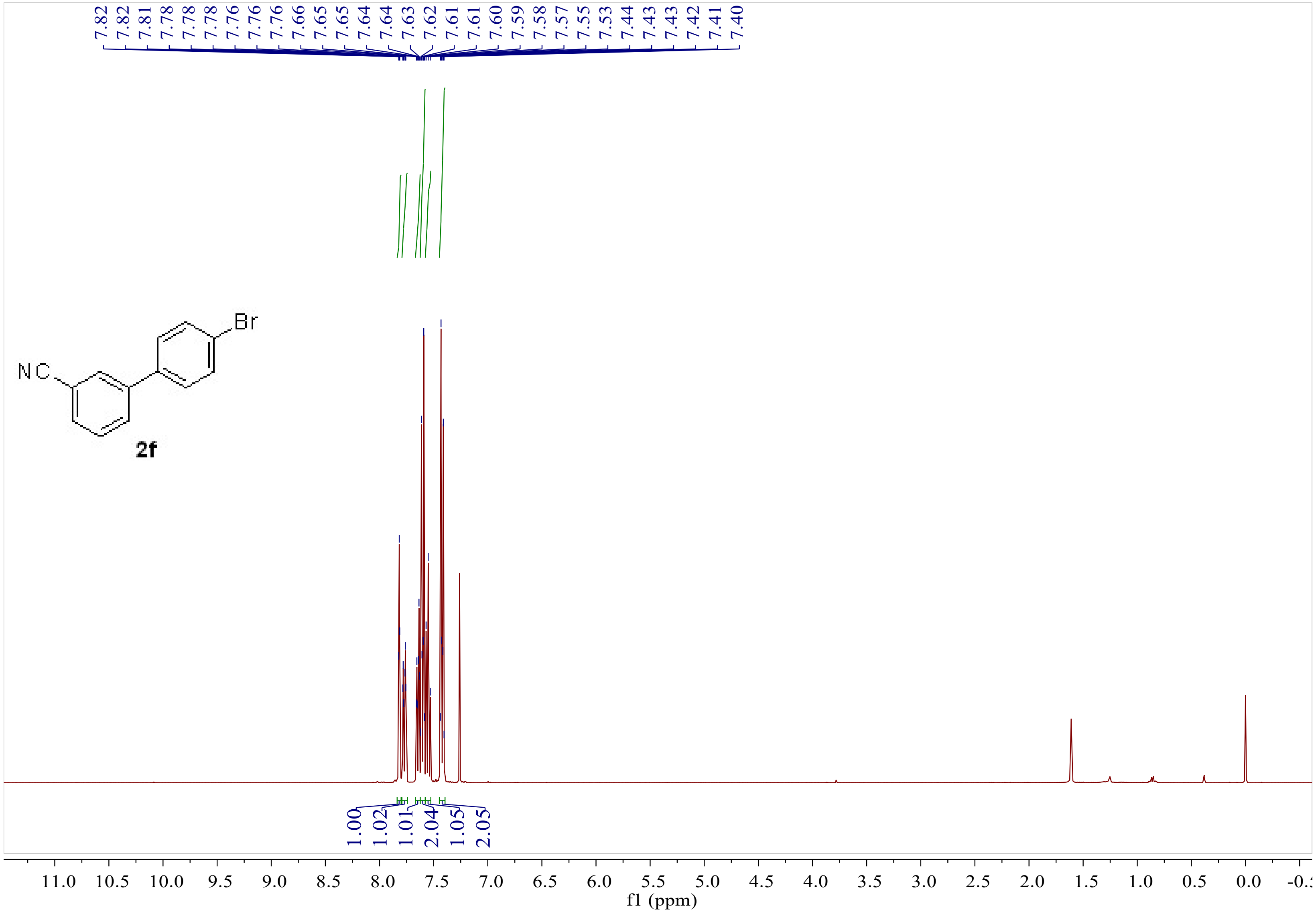
50
40
30
20
10
0
f1 (ppm)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7.78
7.78
7.76
7.76
7.76
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7.64
7.64
7.63
7.62
7.61
7.61
7.60
7.59
7.58
7.57
7.55
7.53
7.44
7.43
7.43
7.42
7.41
7.40
Br
NC
2f
1.00
1.02
1.01
2.04
1.05
2.05
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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3.0
2.5
2.0
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1.0
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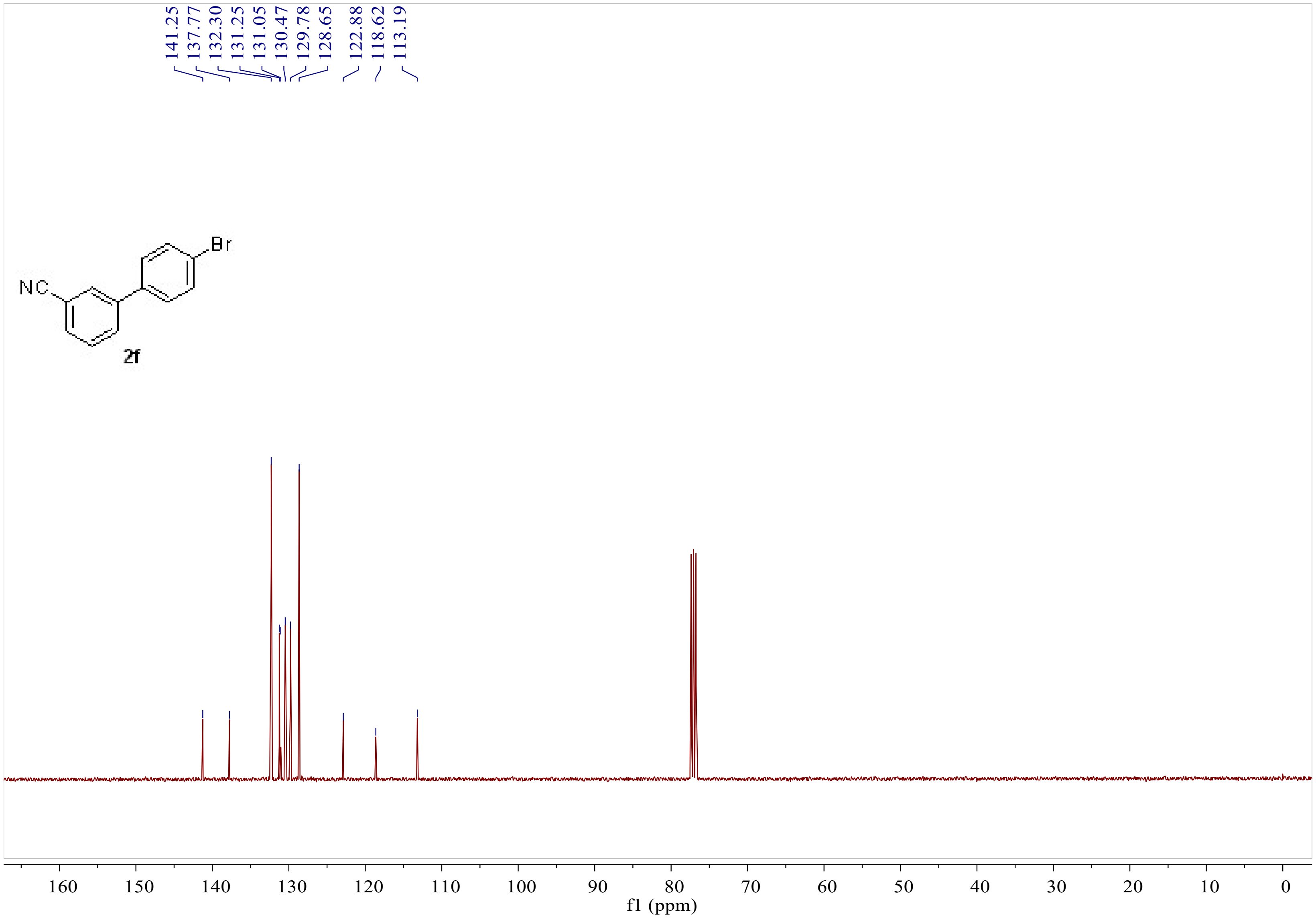
0.0
-0.
f1 (ppm)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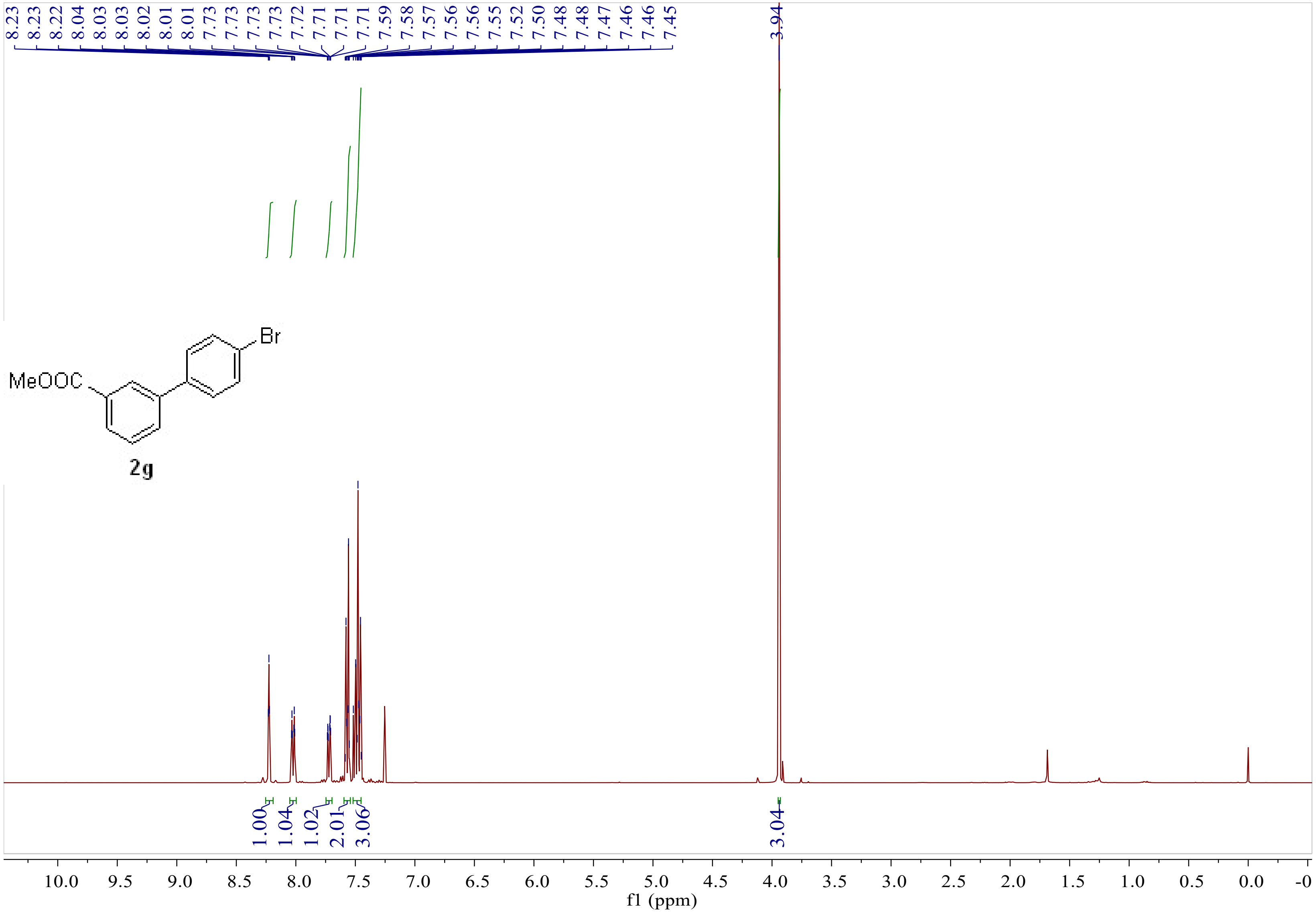
137.77
132.30
131.25
131.05
130.47
129.78
128.65
122.88
118.62
113.19
NC
Br
2f
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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8.22
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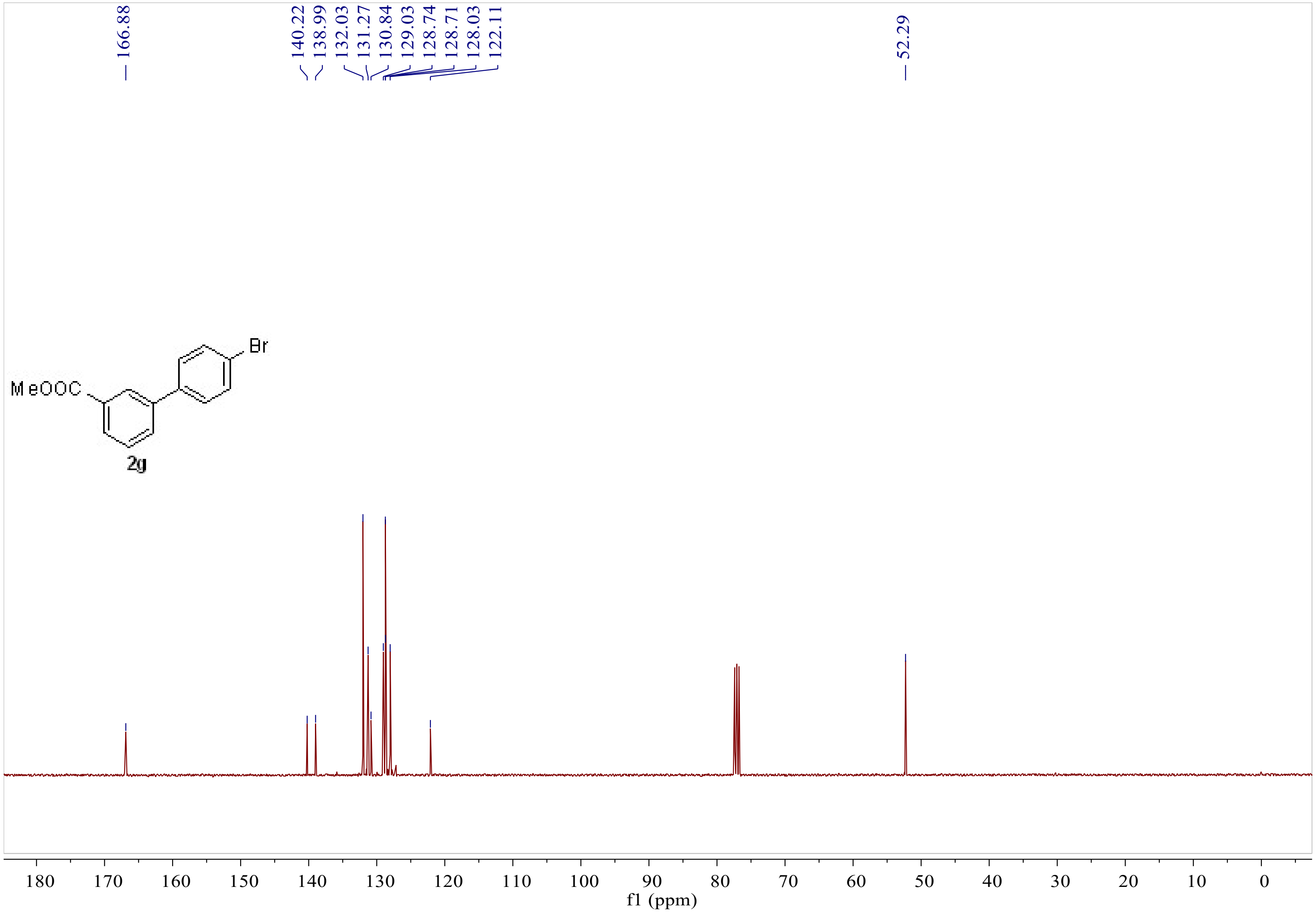
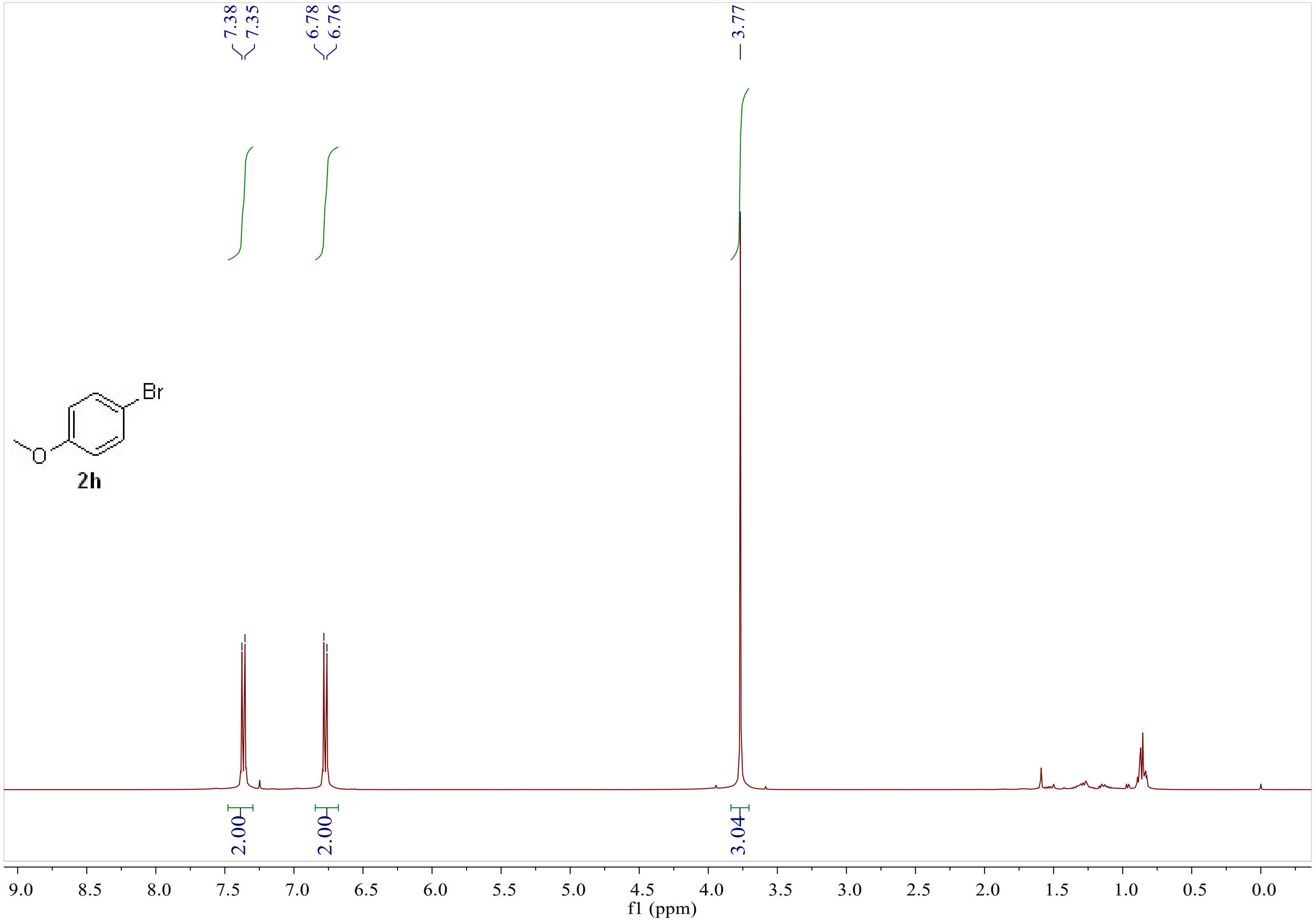
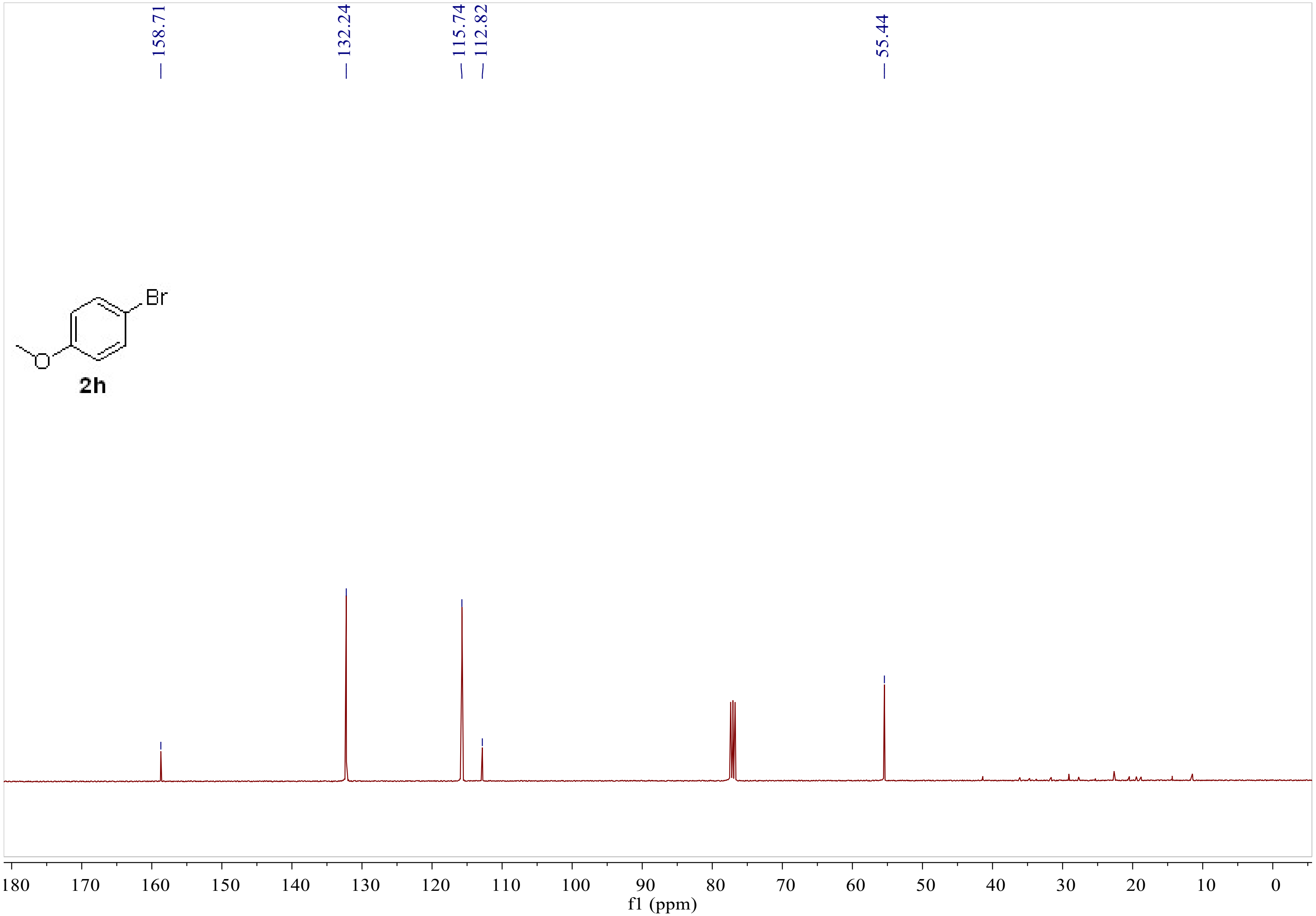
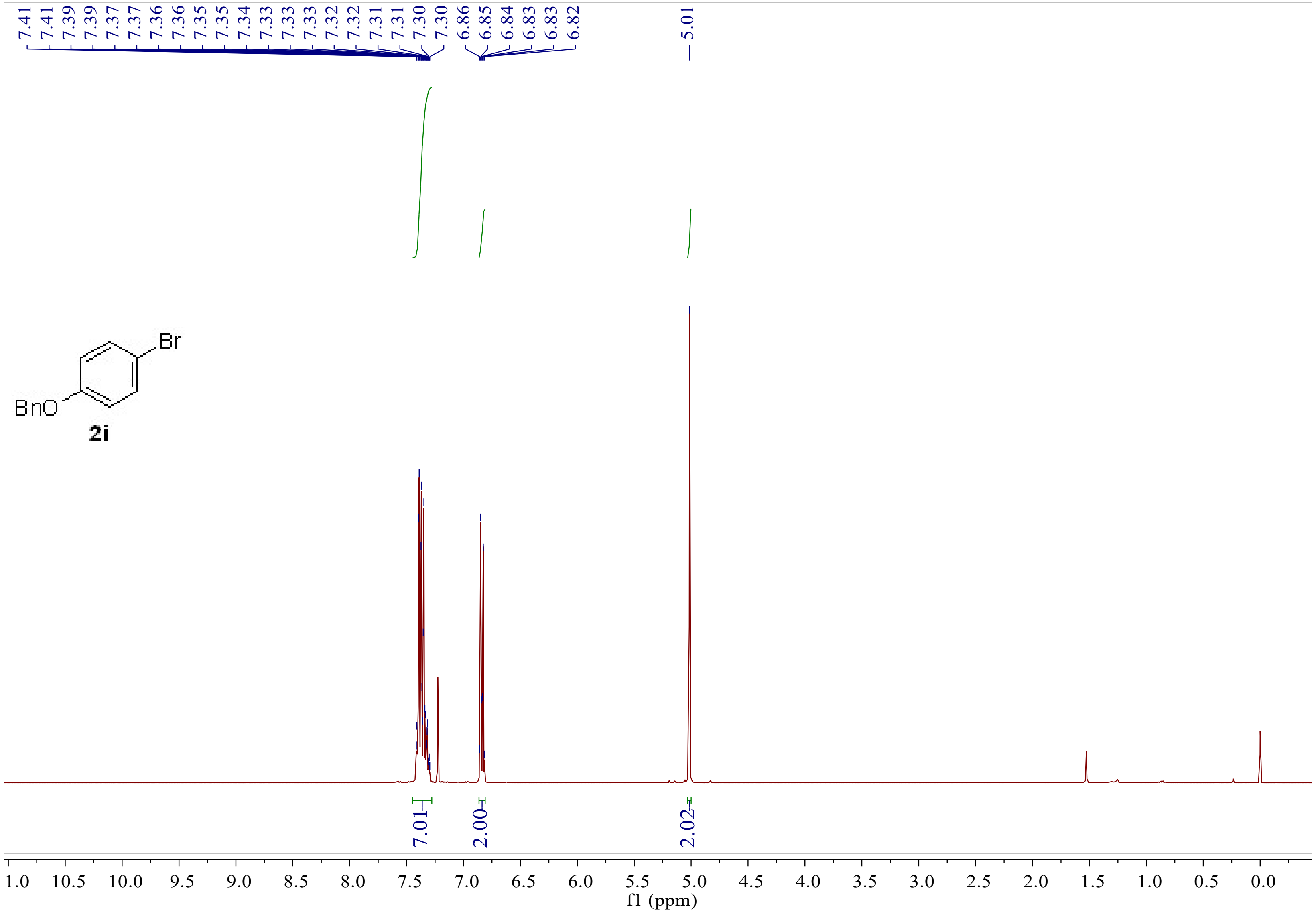
8.03
8.03
8.02
8.01
8.01
7.73
7.73
7.73
7.72
7.71
7.71
7.71
7.59
7.58
7.57
7.56
7.56
7.55
7.52
7.50
7.48
7.48
7.47
7.46
7.46
7.45
3.94
MeOOC
Br
1.00
1.04
1.02
2.01
3.06
3.04
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)

2g


166.88
140.22
138.99
132.03
131.27
130.84
129.03
128.74
128.71
128.03
122.11
52.29
MeOOC
Br
2g
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.38
7.35
6.78
6.76
3.77
Br
O
2h
2.00
2.00
3.04
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
0.5
0.0
f1 (ppm)


158.71
132.24
115.74
112.82
55.44
Br
O
2h
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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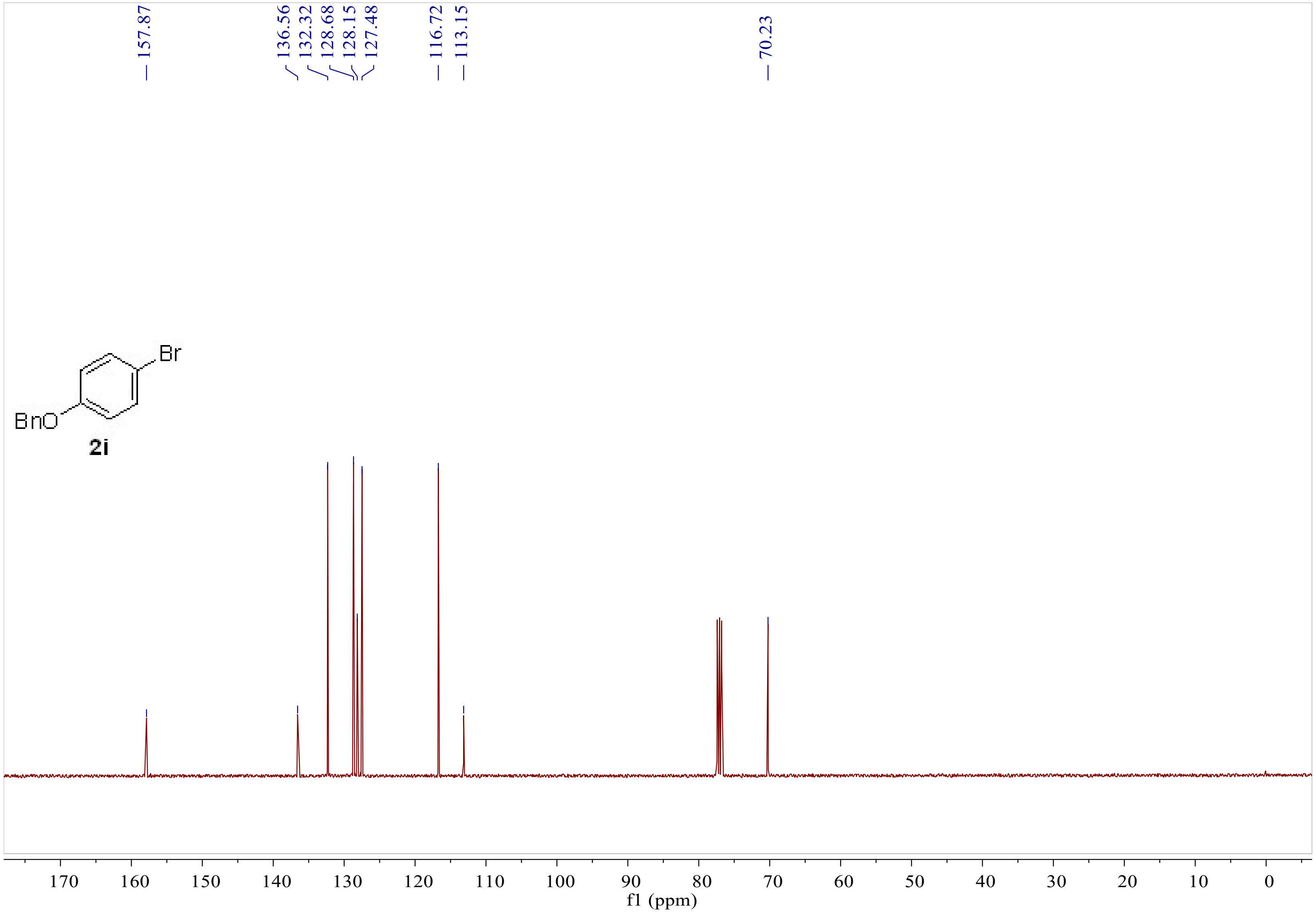
7.39
7.39
7.37
7.37
7.36
7.36
7.35
7.35
7.34
7.33
7.33
7.33
7.32
7.32
7.31
7.31
7.30
7.30
6.86
6.85
6.84
6.83
6.83
6.82
5.01
Br
BnO
2i
7.01
2.00
2.02
1.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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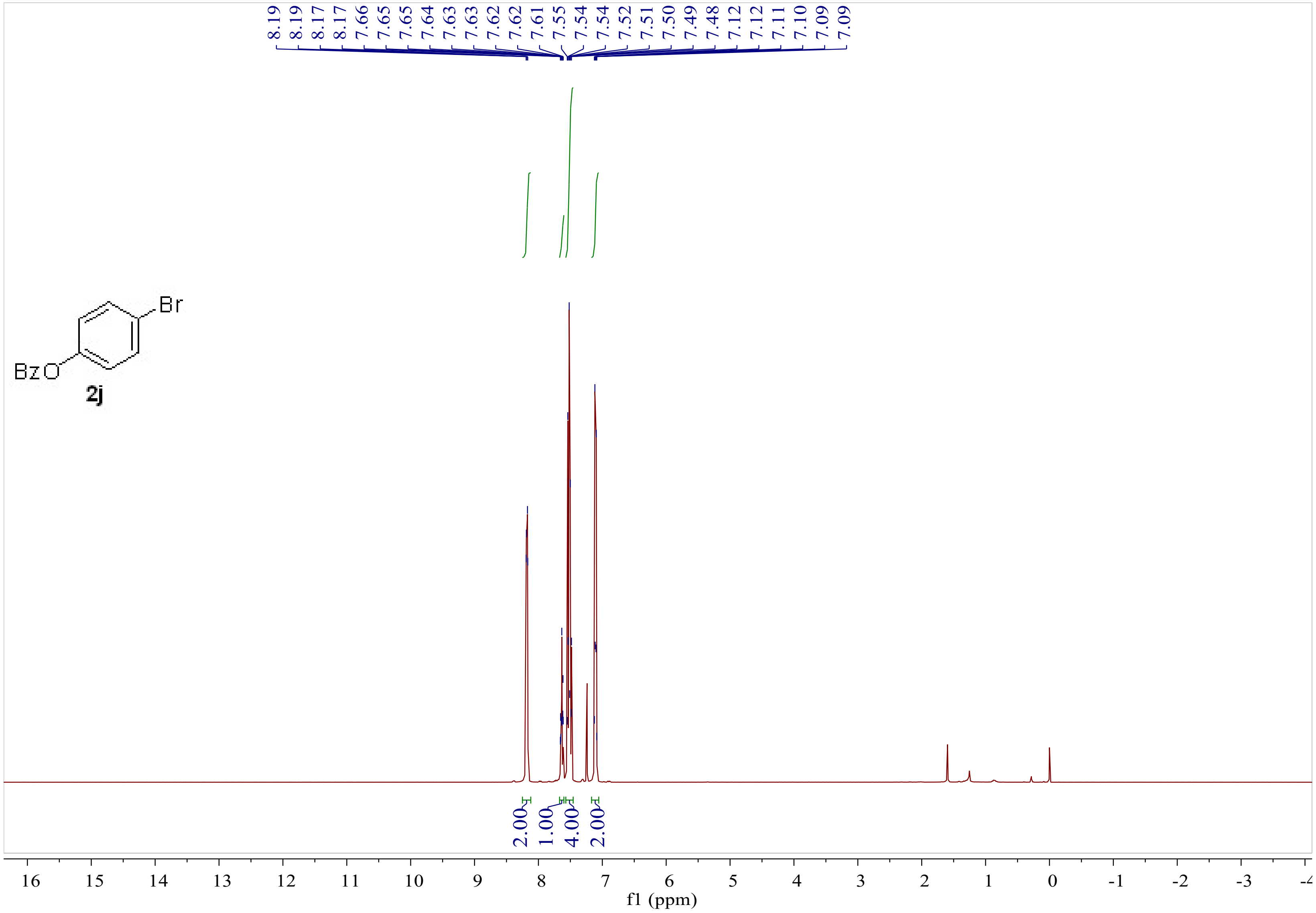
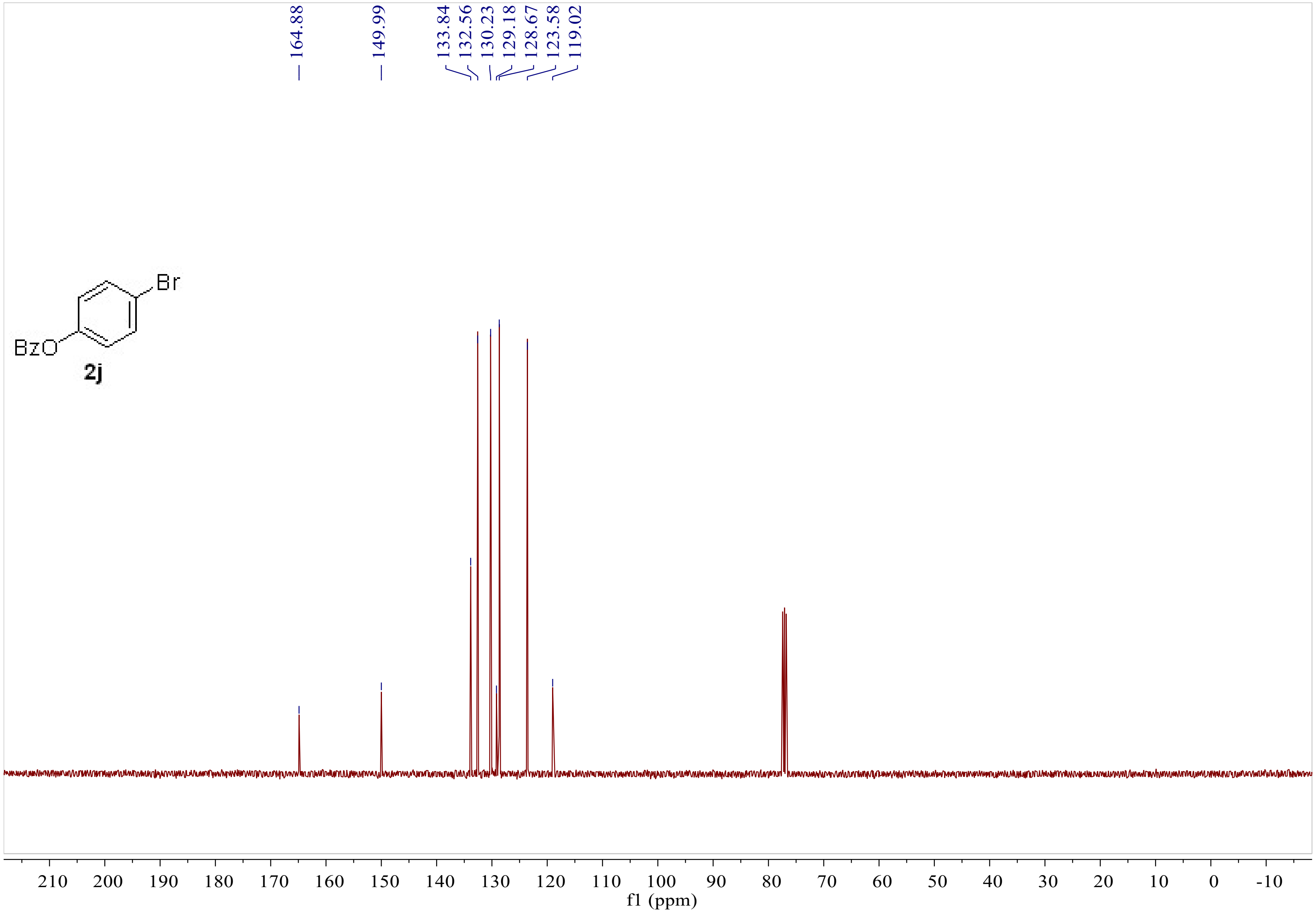
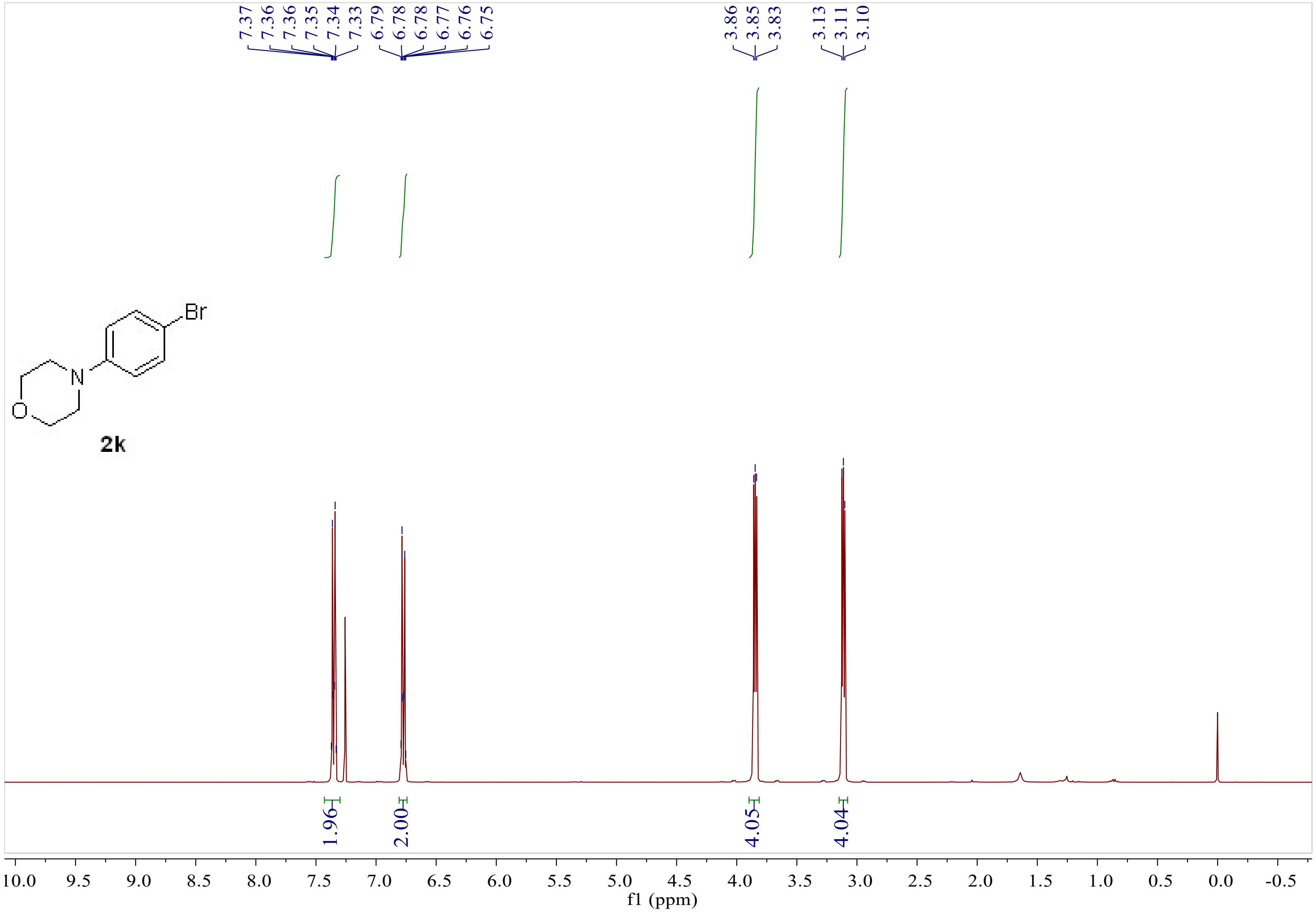
128.68
128.15
127.48
116.72
113.15
70.23
Br
BnO
2i
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


2j

8.19
8.19
8.17
8.17
7.66
7.65
7.65
7.64
7.63
7.63
7.62
7.62
7.61
7.55
7.54
7.54
7.52
7.51
7.50
7.49
7.48
7.12
7.12
7.11
7.10
7.09
7.09
Br
BzO
2.00
1.00
4.00
2.00
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)


164.88
149.99
133.84
132.56
130.23
129.18
128.67
123.58
119.02
Br
BzO
2j
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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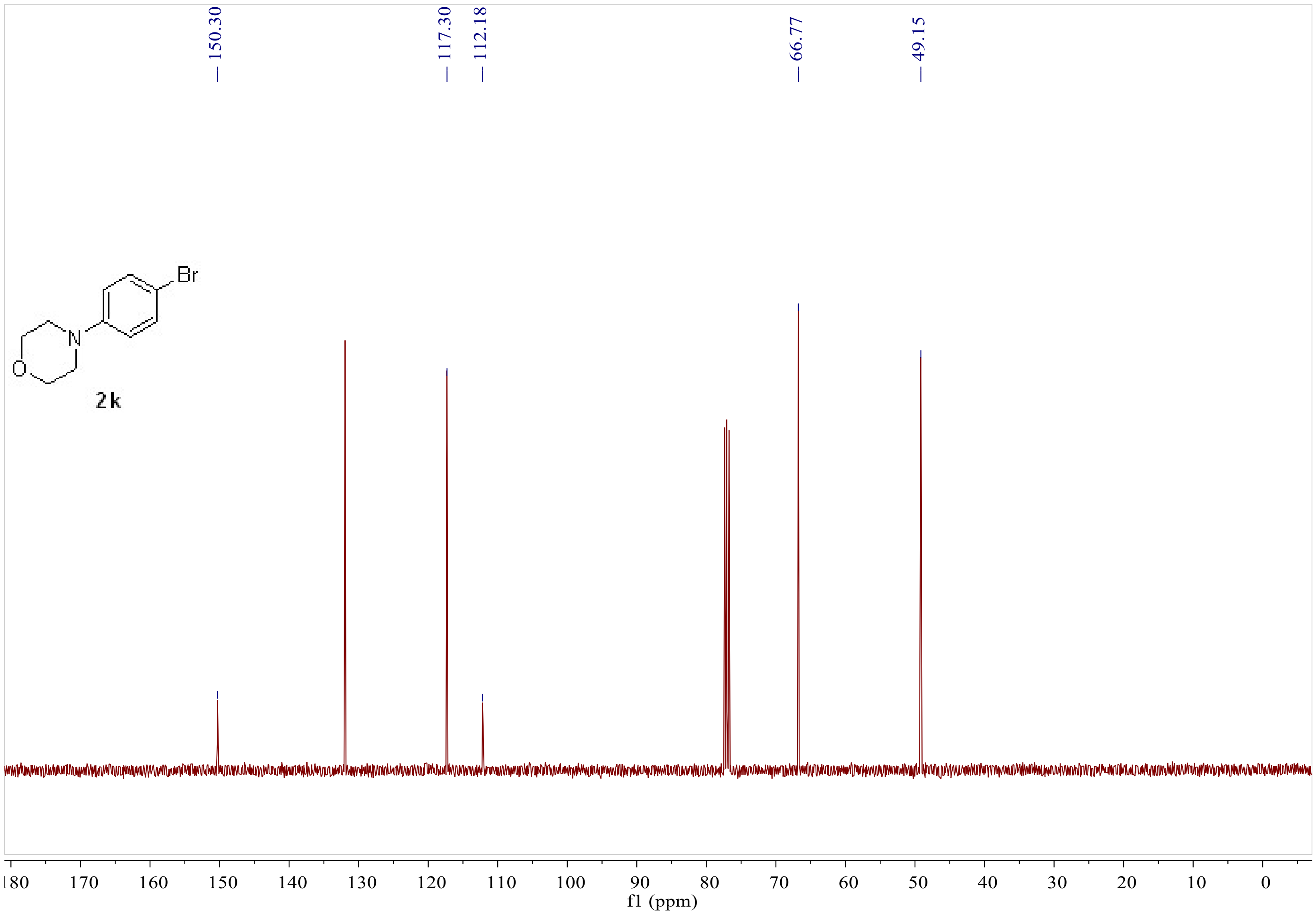
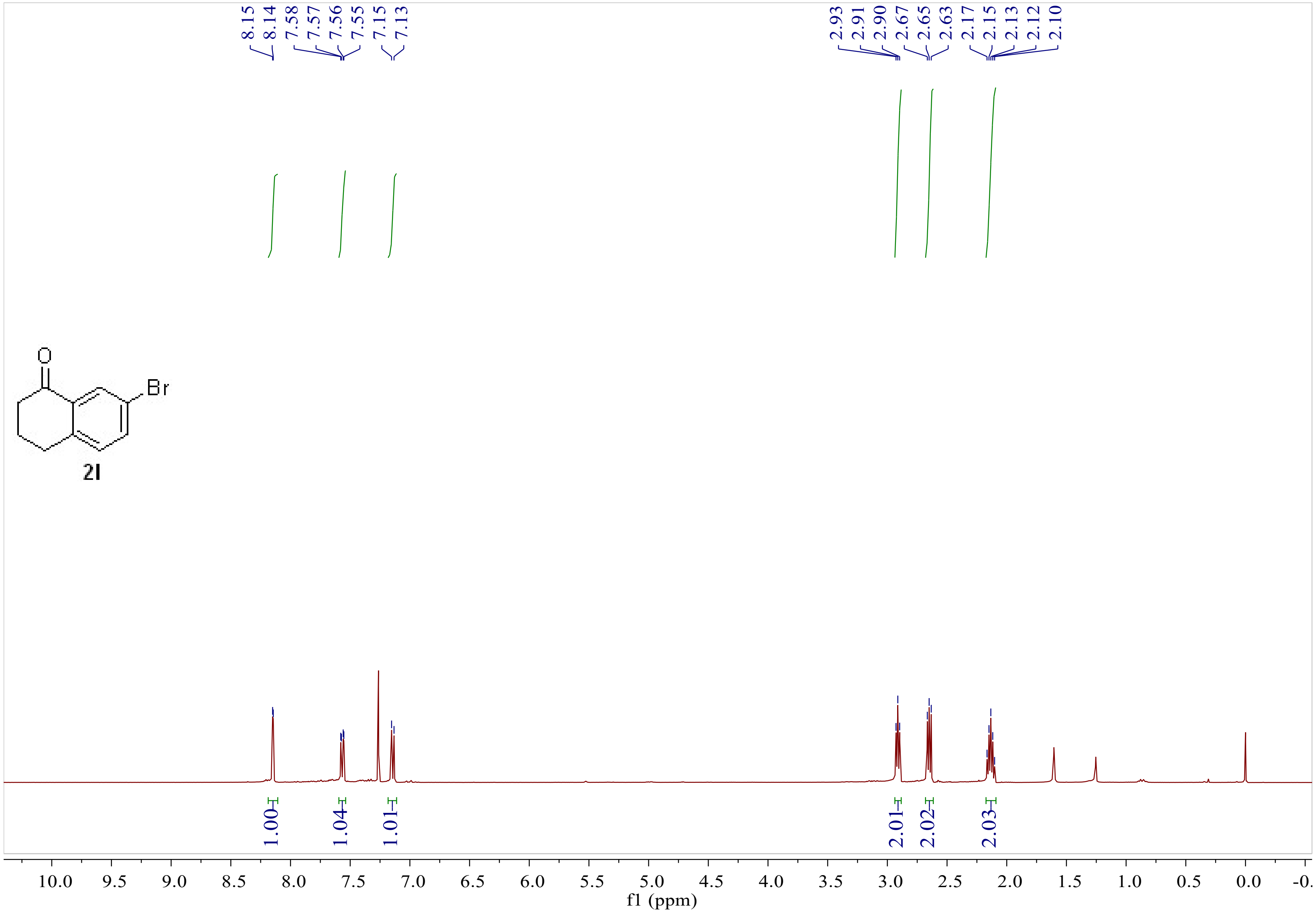
7.36
7.36
7.35
7.34
7.33
6.79
6.78
6.78
6.77
6.76
6.75
3.86
3.85
3.83
3.13
3.11
3.10
Br
N
O
2k
1.96
2.00
4.05
4.04
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


150.30
117.30
112.18
66.77
49.15
Br
N
O
2k
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.15
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7.58
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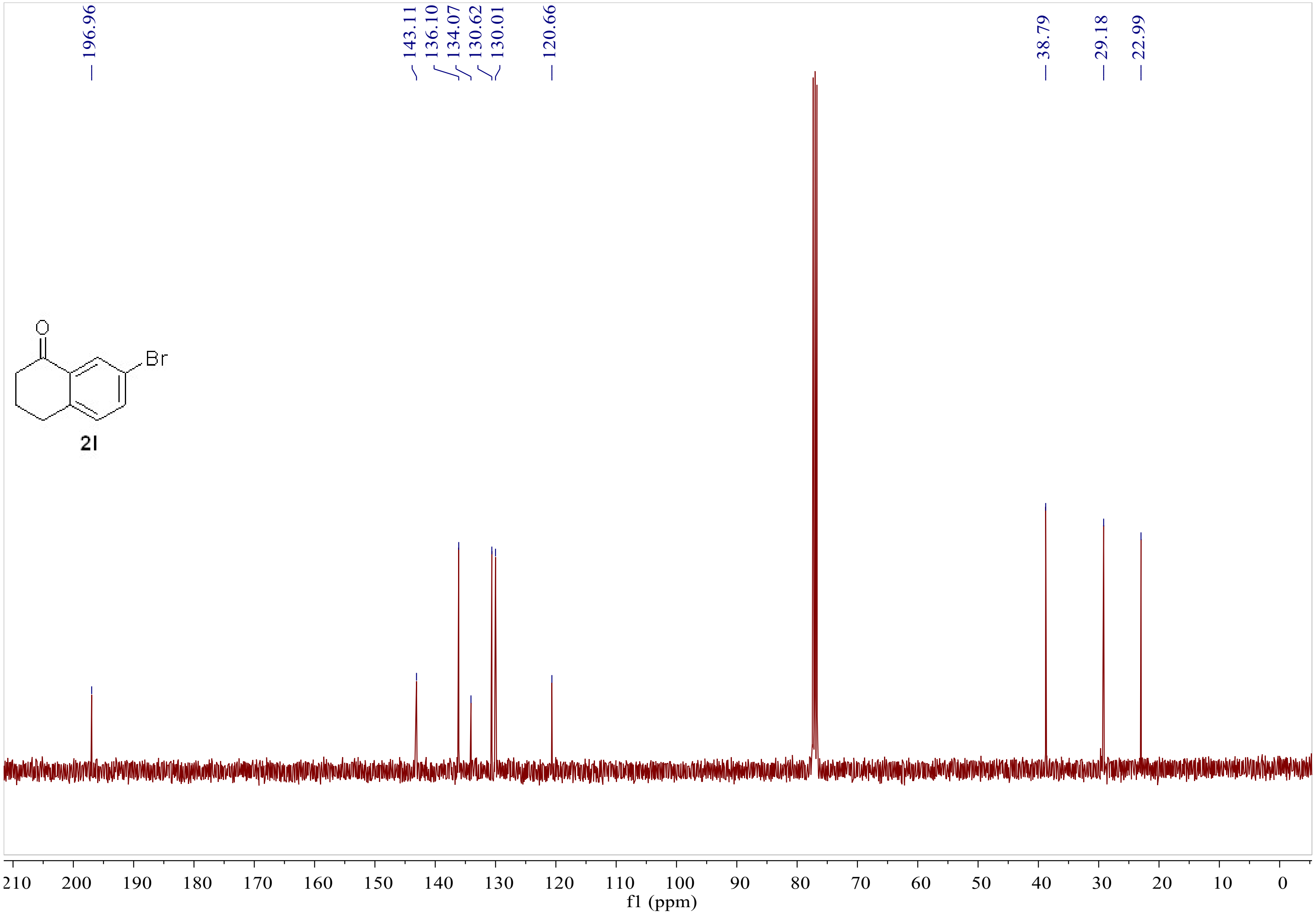
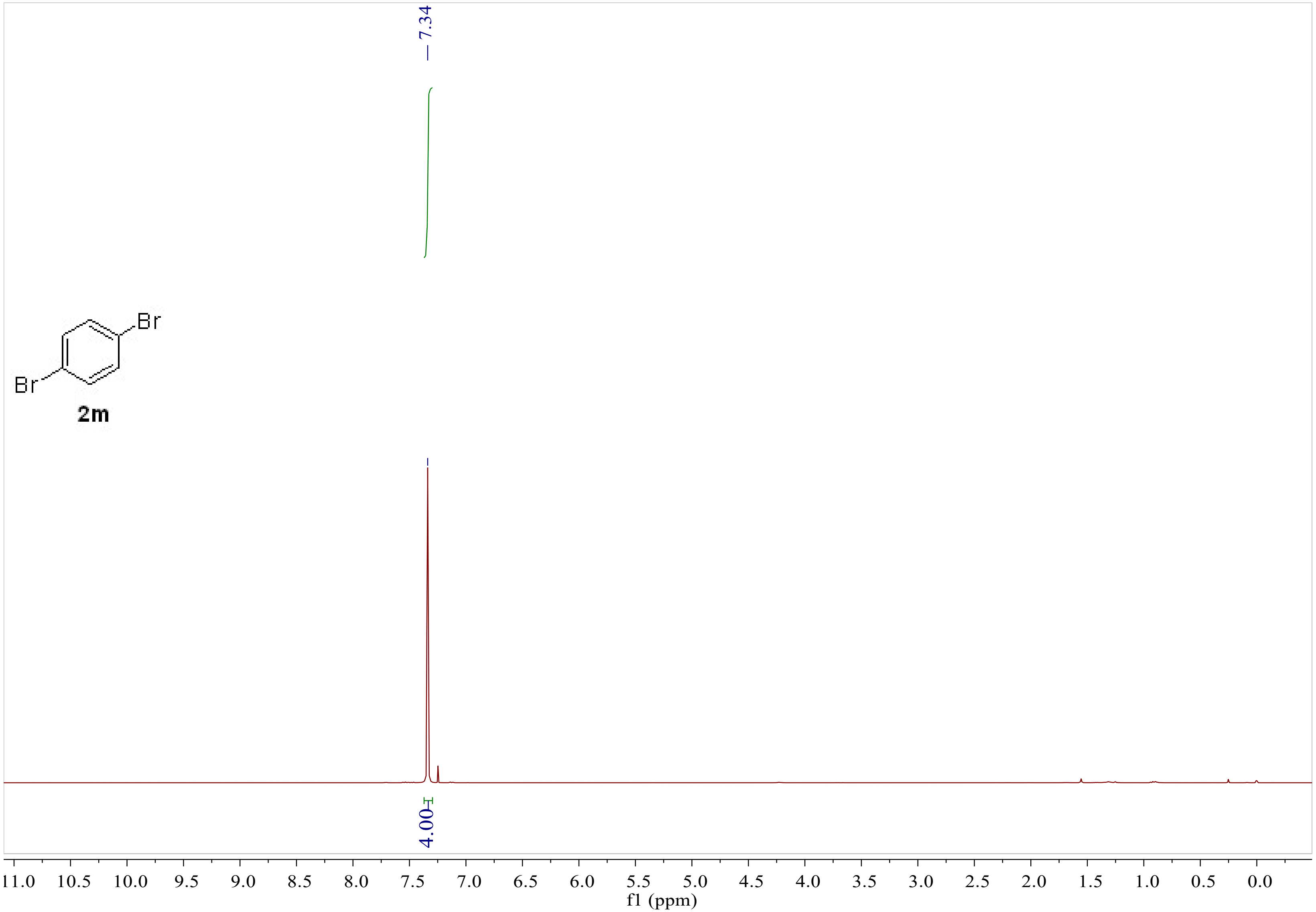
7.56
7.55
7.15
7.13
2.93
2.91
2.90
2.67
2.65
2.63
2.17
2.15
2.13
2.12
2.10
O
Br
2l
1.00
1.04
1.01
2.01
2.02
2.03
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.
f1 (ppm)


196.96
143.11
136.10
134.07
130.62
130.01
120.66
38.79
29.18
22.99
O
Br
2l
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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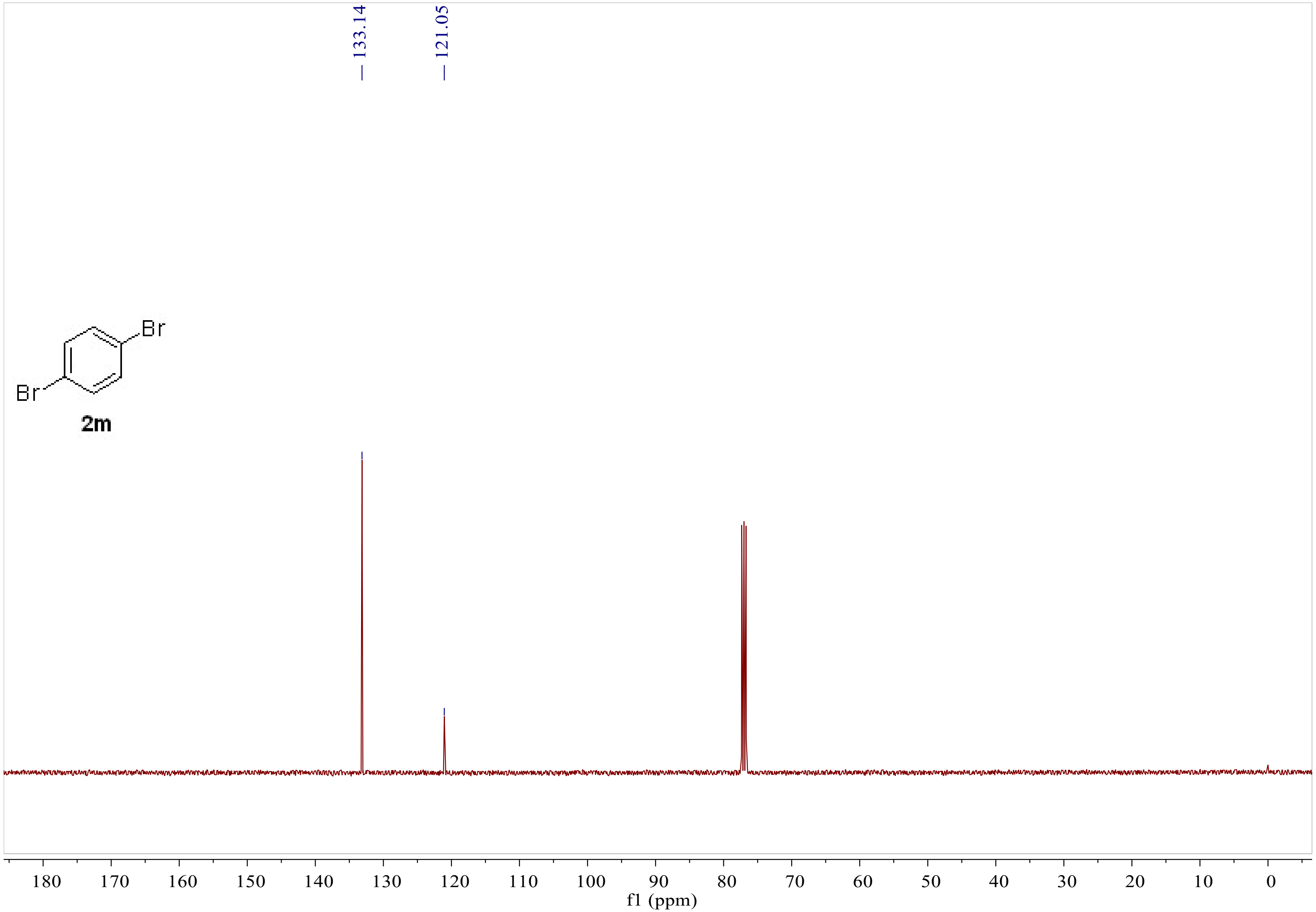
Br
Br
2m
4.00
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

Br
Br

2m


133.14
121.05
180
170
160
150
140
130
120
110
100
90
80
70
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50
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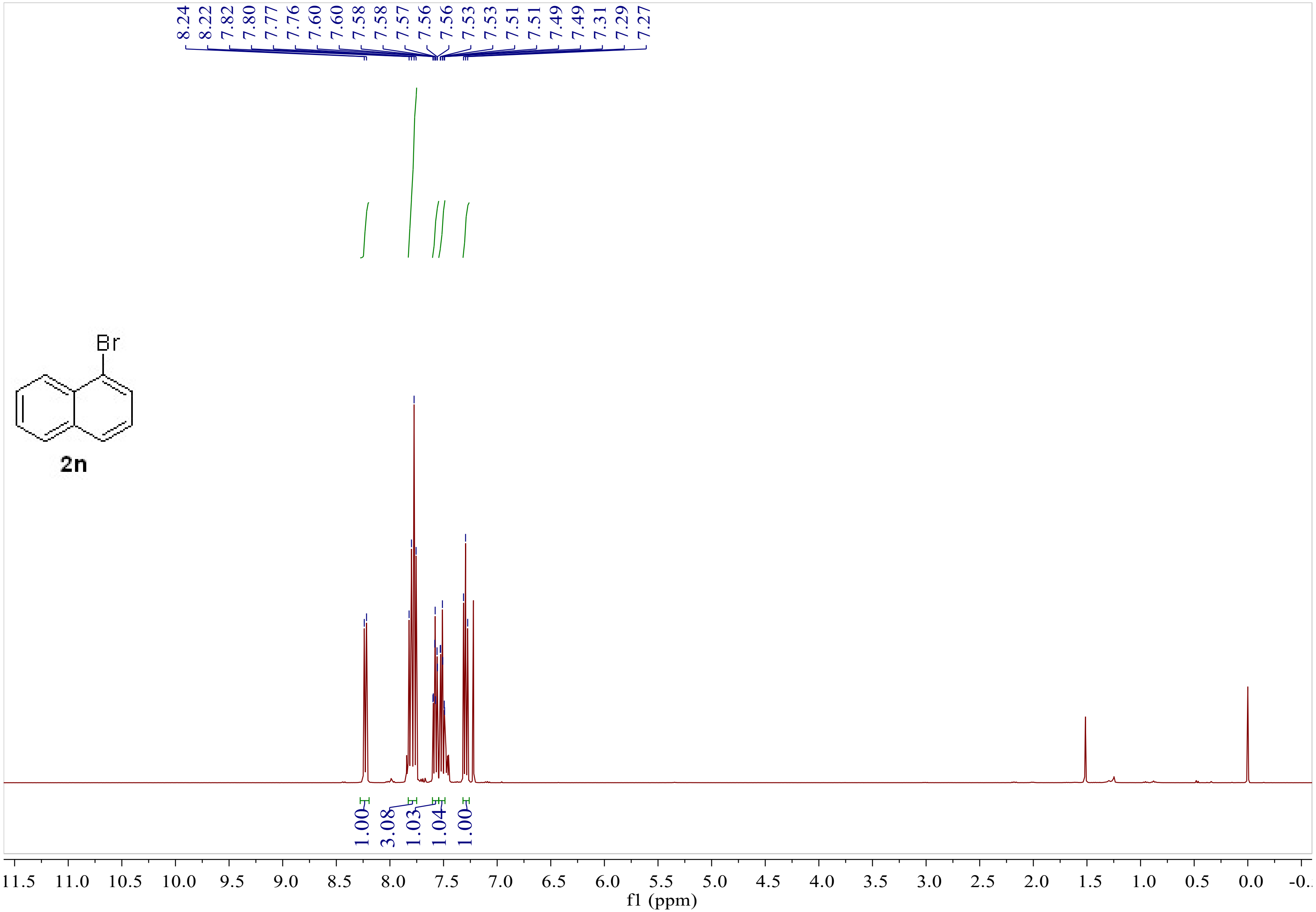
30
20
10
0
f1 (ppm)


8.24
8.22
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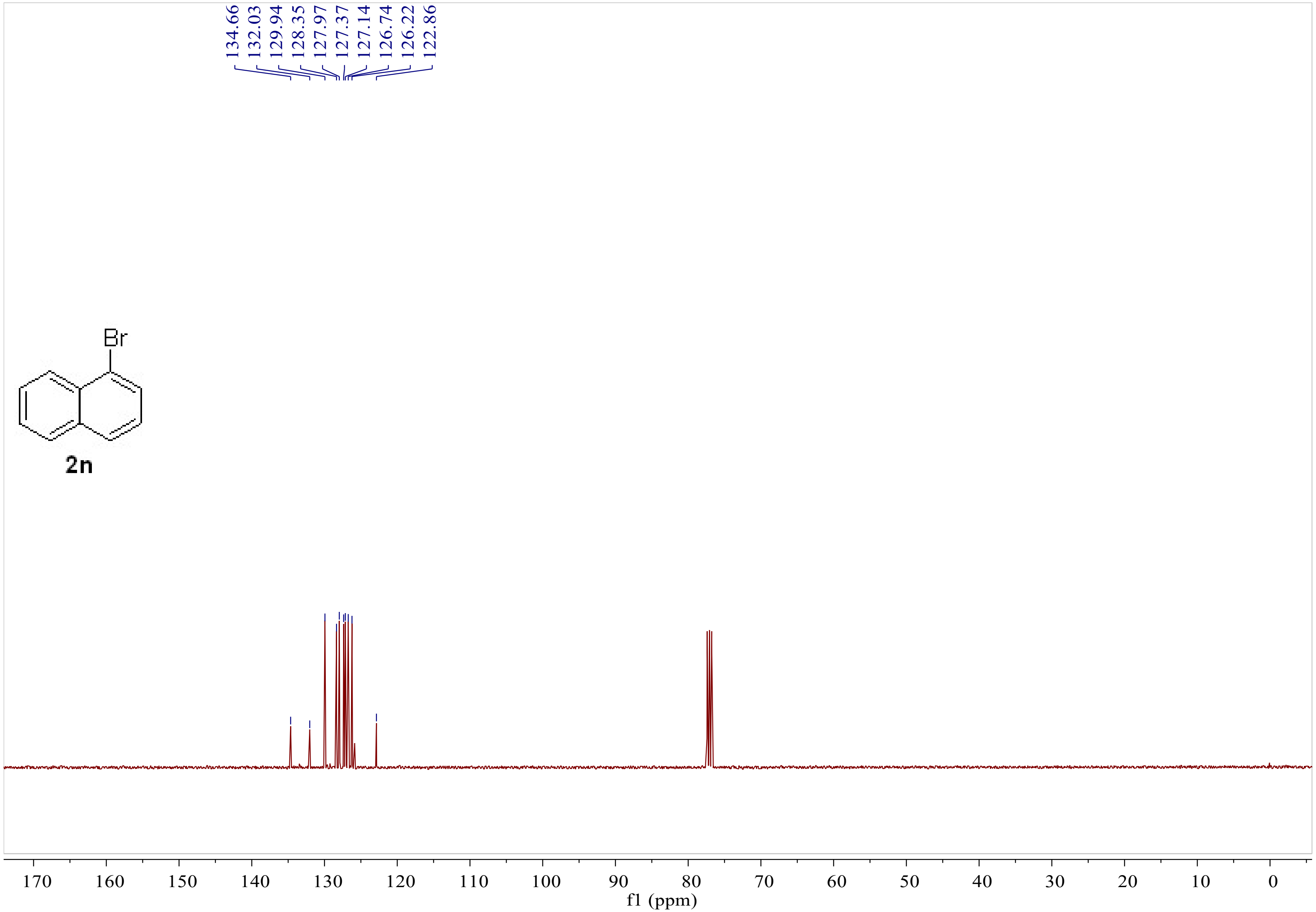
7.80
7.77
7.76
7.60
7.60
7.58
7.58
7.57
7.56
7.56
7.53
7.53
7.51
7.51
7.49
7.49
7.31
7.29
7.27
Br
1.00
3.08
1.03
1.04
1.00
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.
f1 (ppm)

2n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132.03
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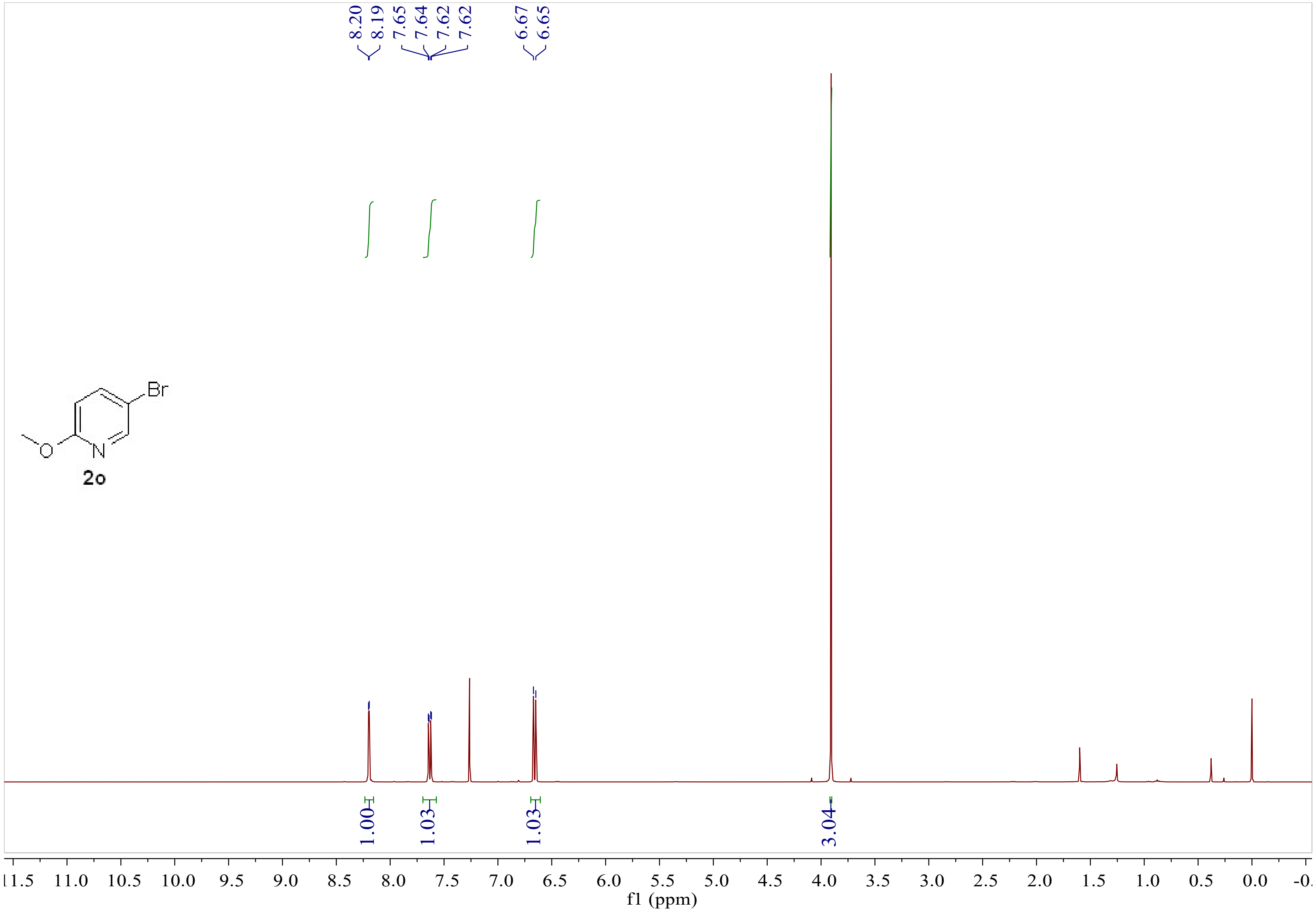
128.35
127.97
127.37
127.14
126.74
126.22
122.86
Br
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

2n


8.20
8.19
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7.64
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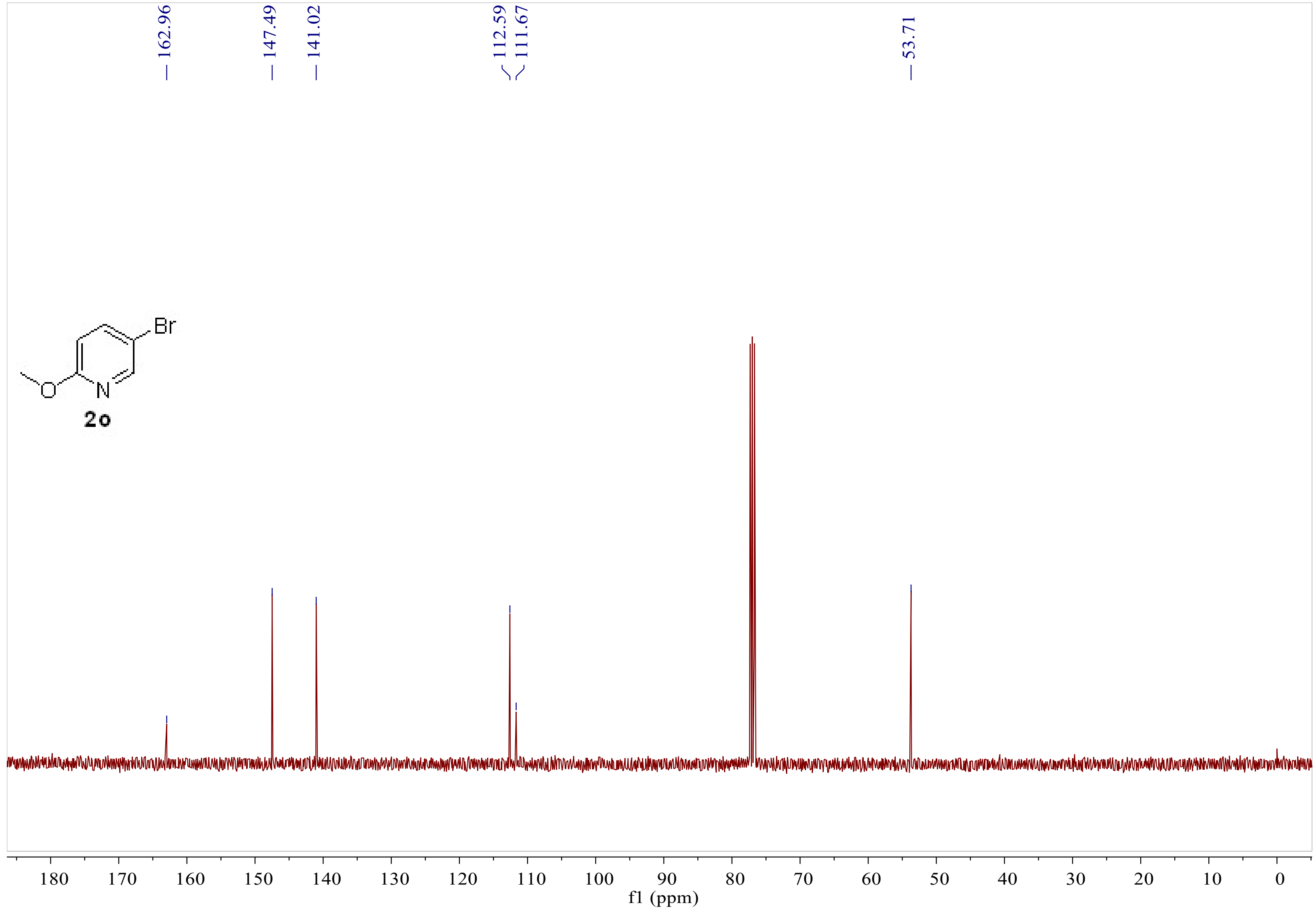
7.62
6.67
6.65
Br
O
N
2o
1.00
1.03
1.03
3.04
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.
f1 (ppm)


162.96
147.49
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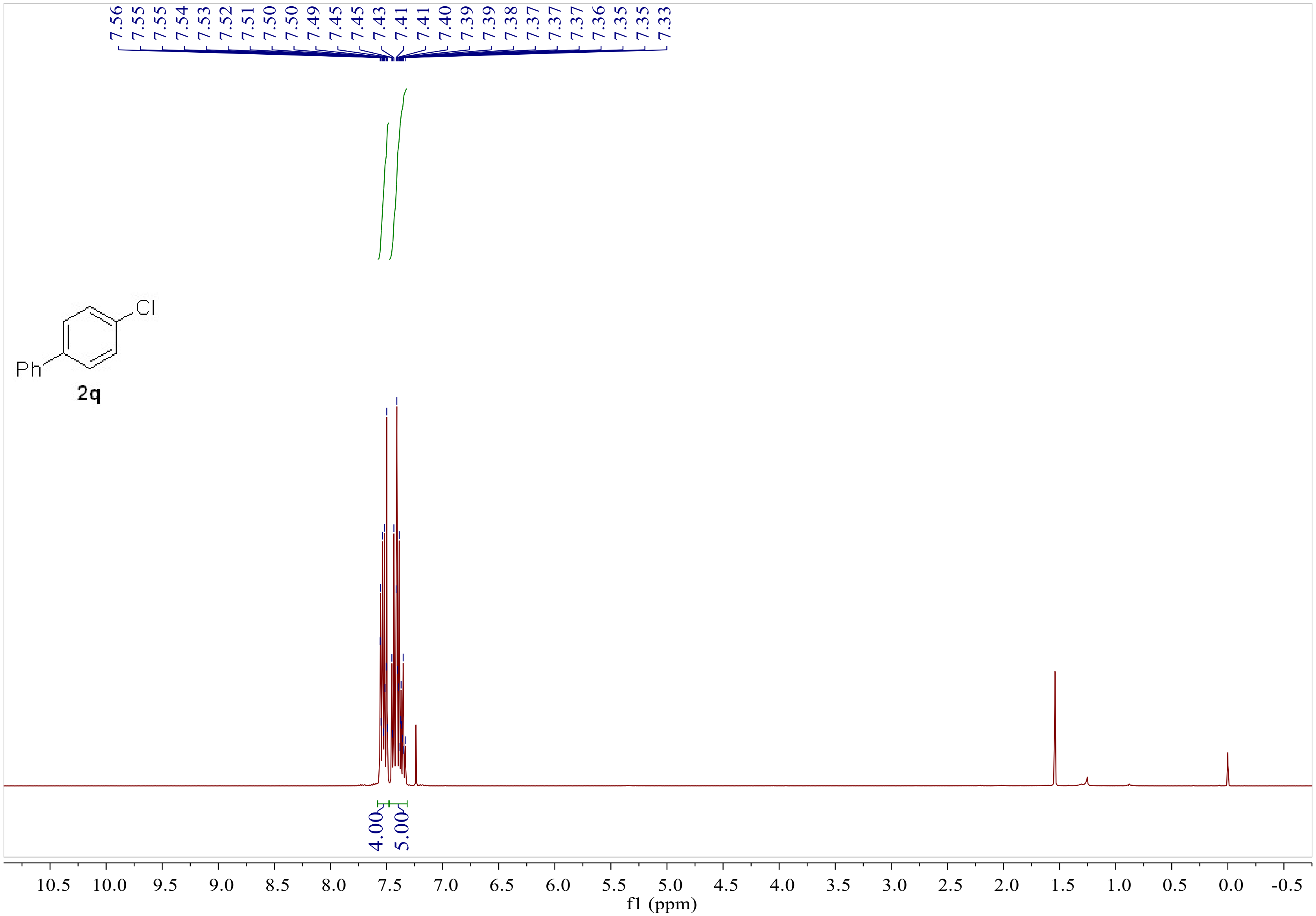
53.71
Br
O
N
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

2o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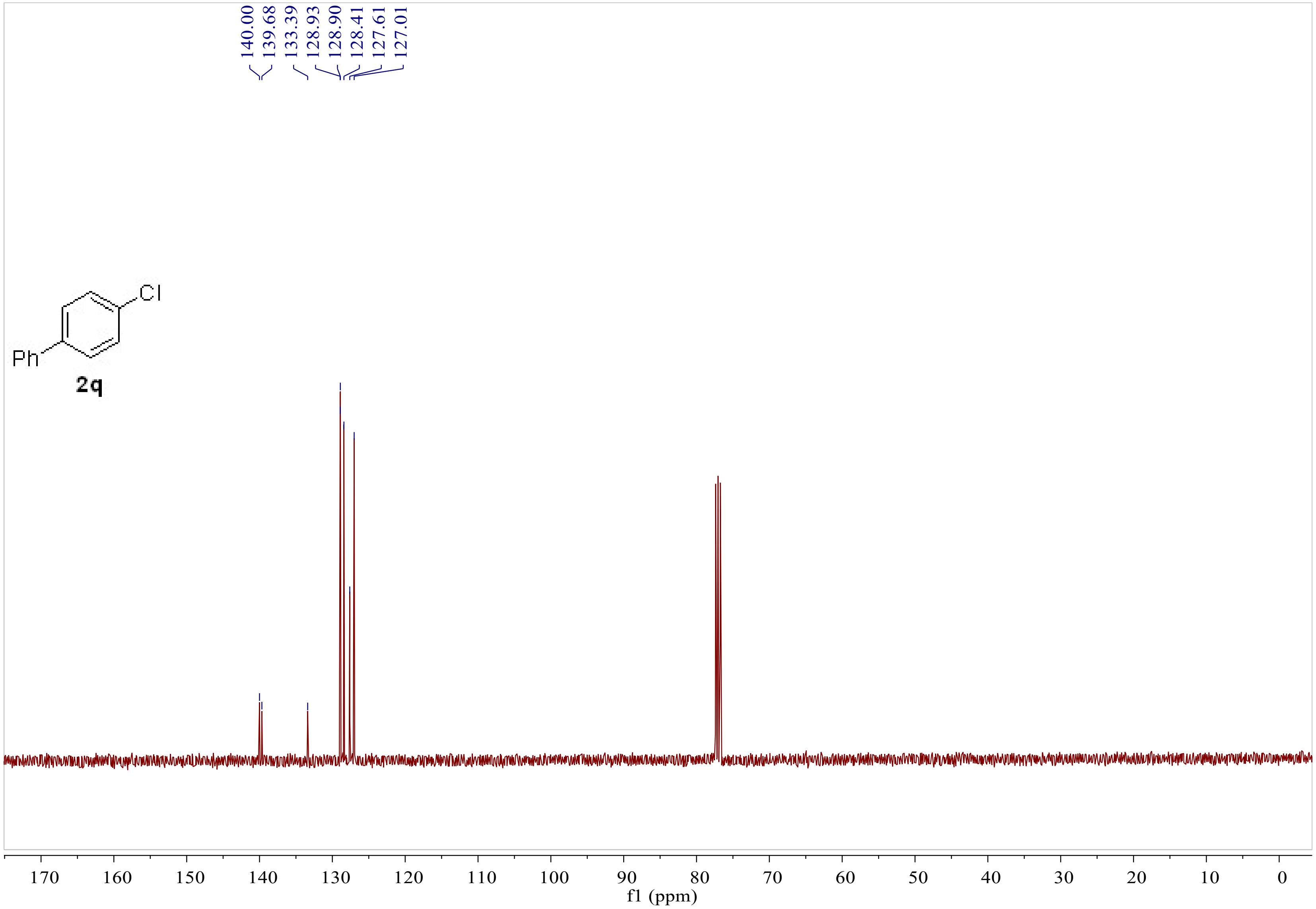
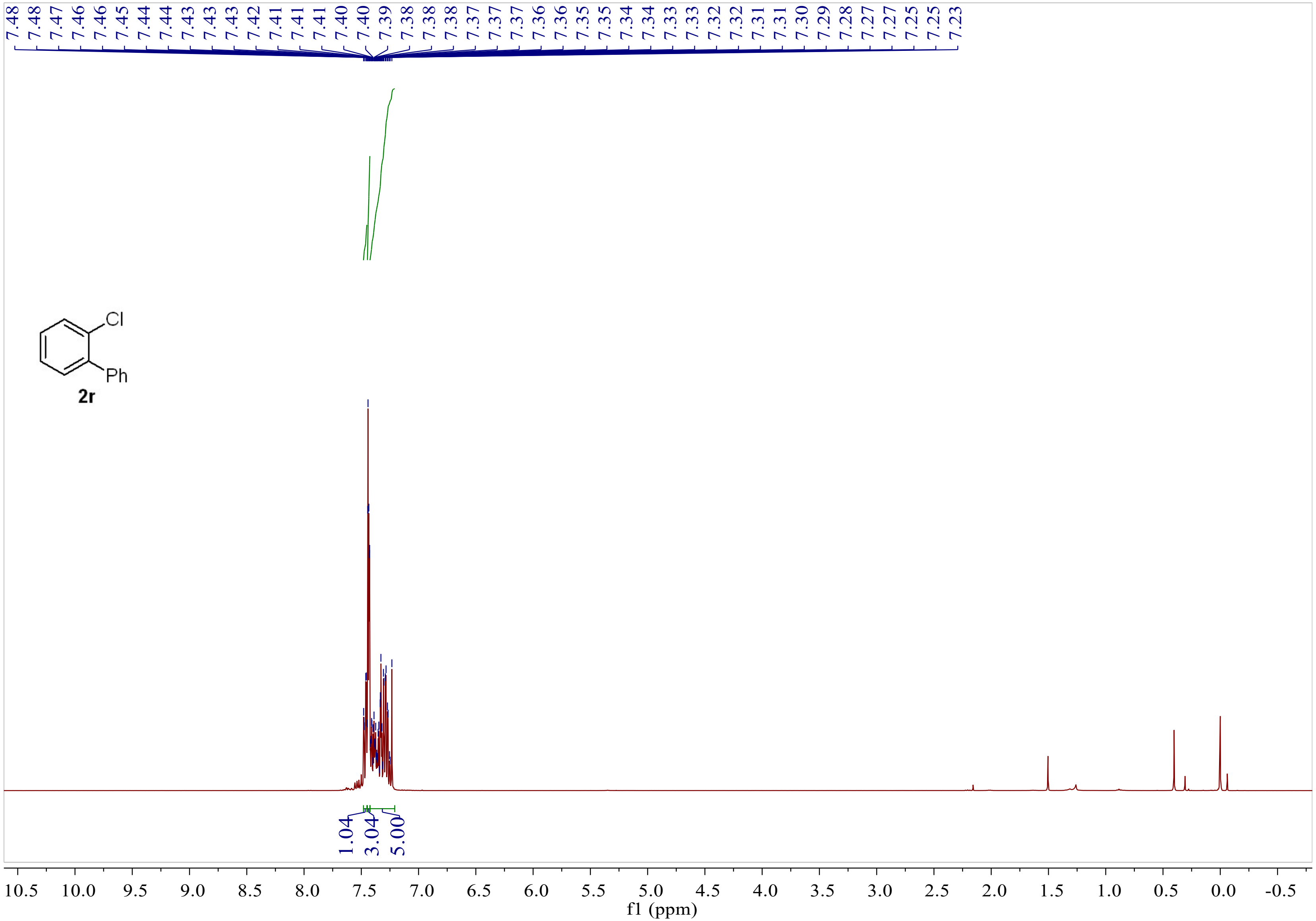
7.43
7.41
7.41
7.40
7.39
7.39
7.38
7.37
7.37
7.37
7.36
7.35
7.35
7.33
Cl
Ph
2q
4.00
5.00
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


140.00
139.68
133.39
128.93
128.90
128.41
127.61
127.01
Cl
Ph
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

2q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7.47
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7.44
7.44
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7.43
7.42
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7.41
7.41
7.40
7.40
7.39
7.38
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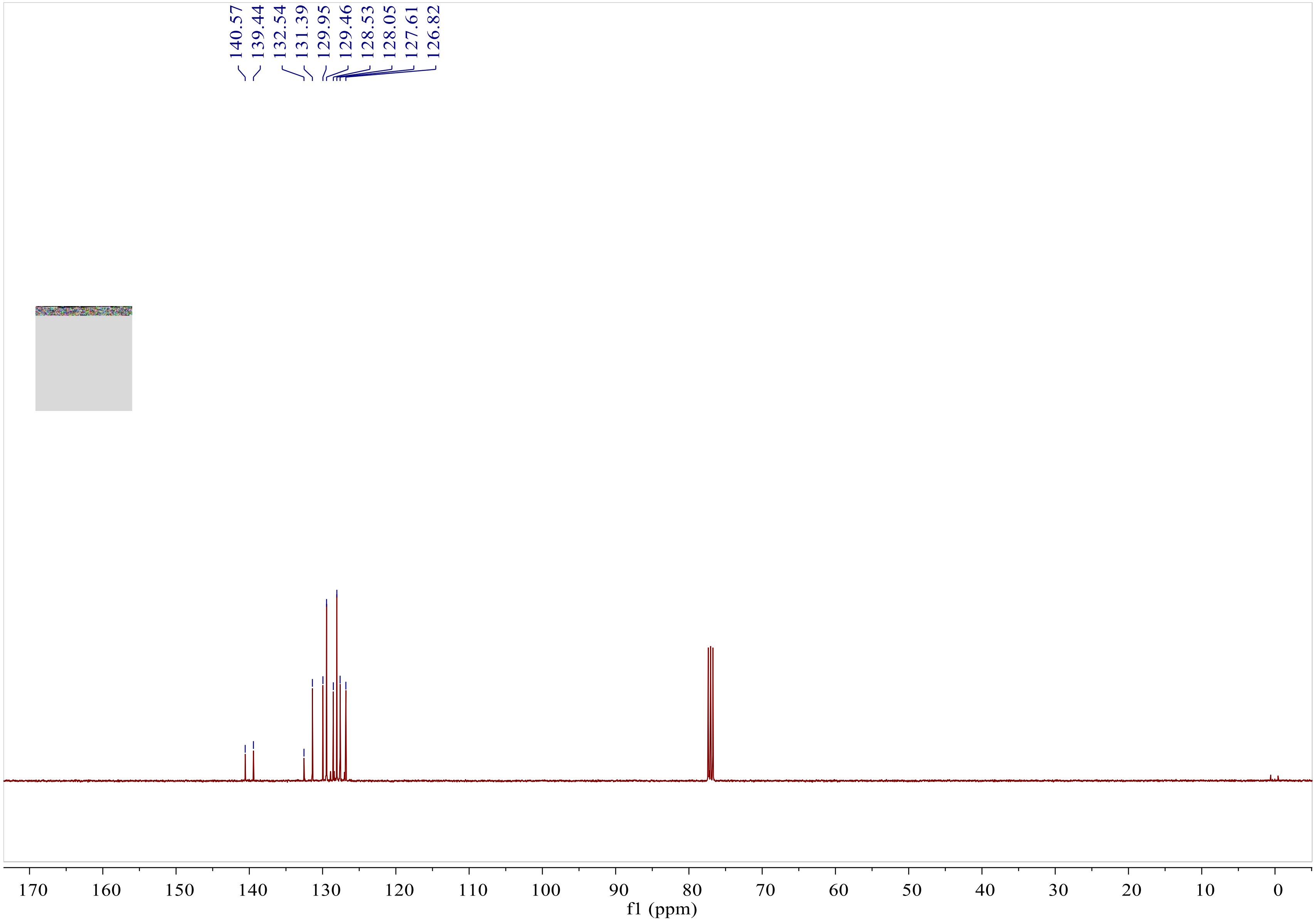
7.33
7.32
7.32
7.31
7.31
7.30
7.29
7.28
7.27
7.27
7.25
7.25
7.23
Cl
Ph
1.04
3.04
5.00
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

2r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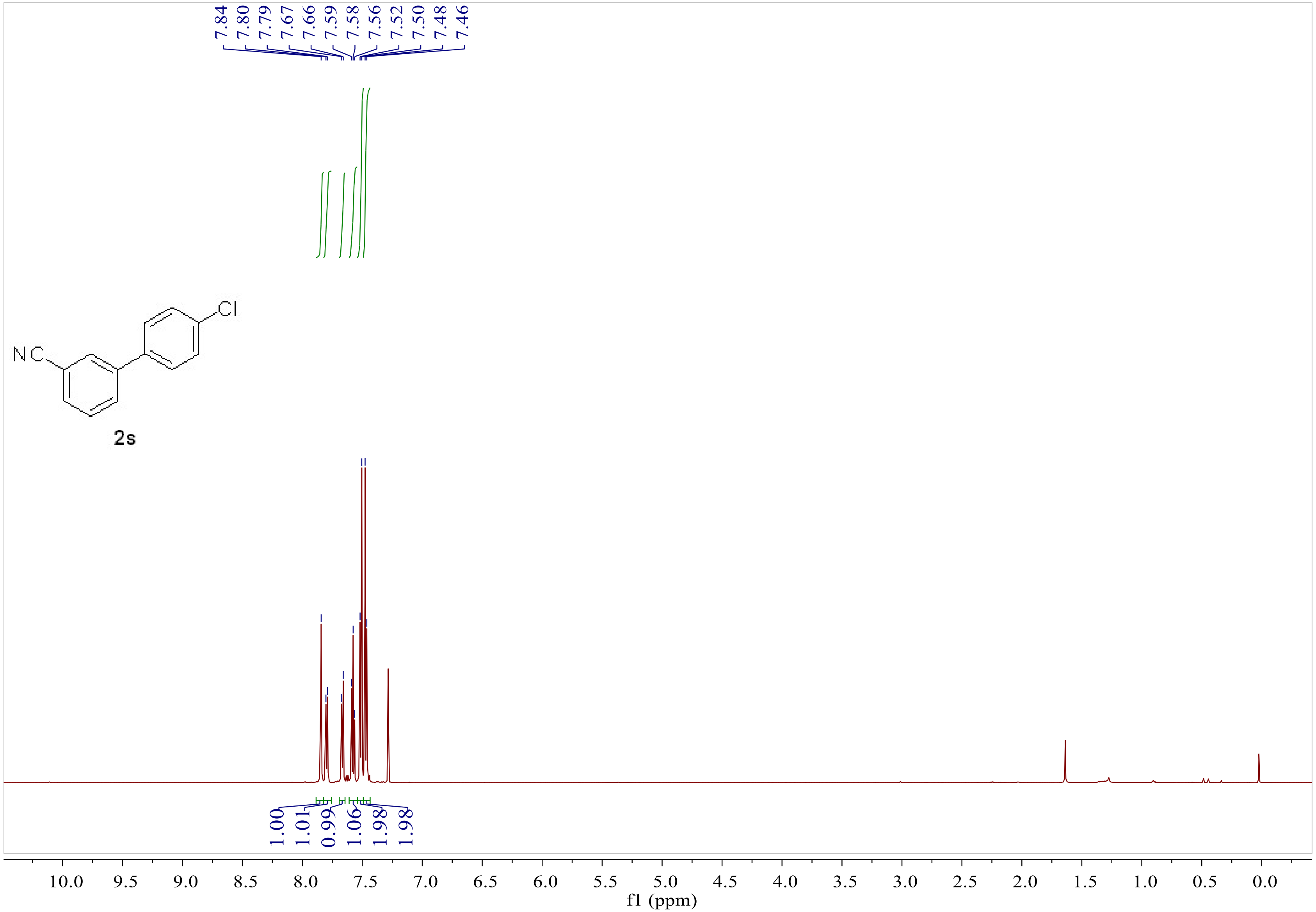
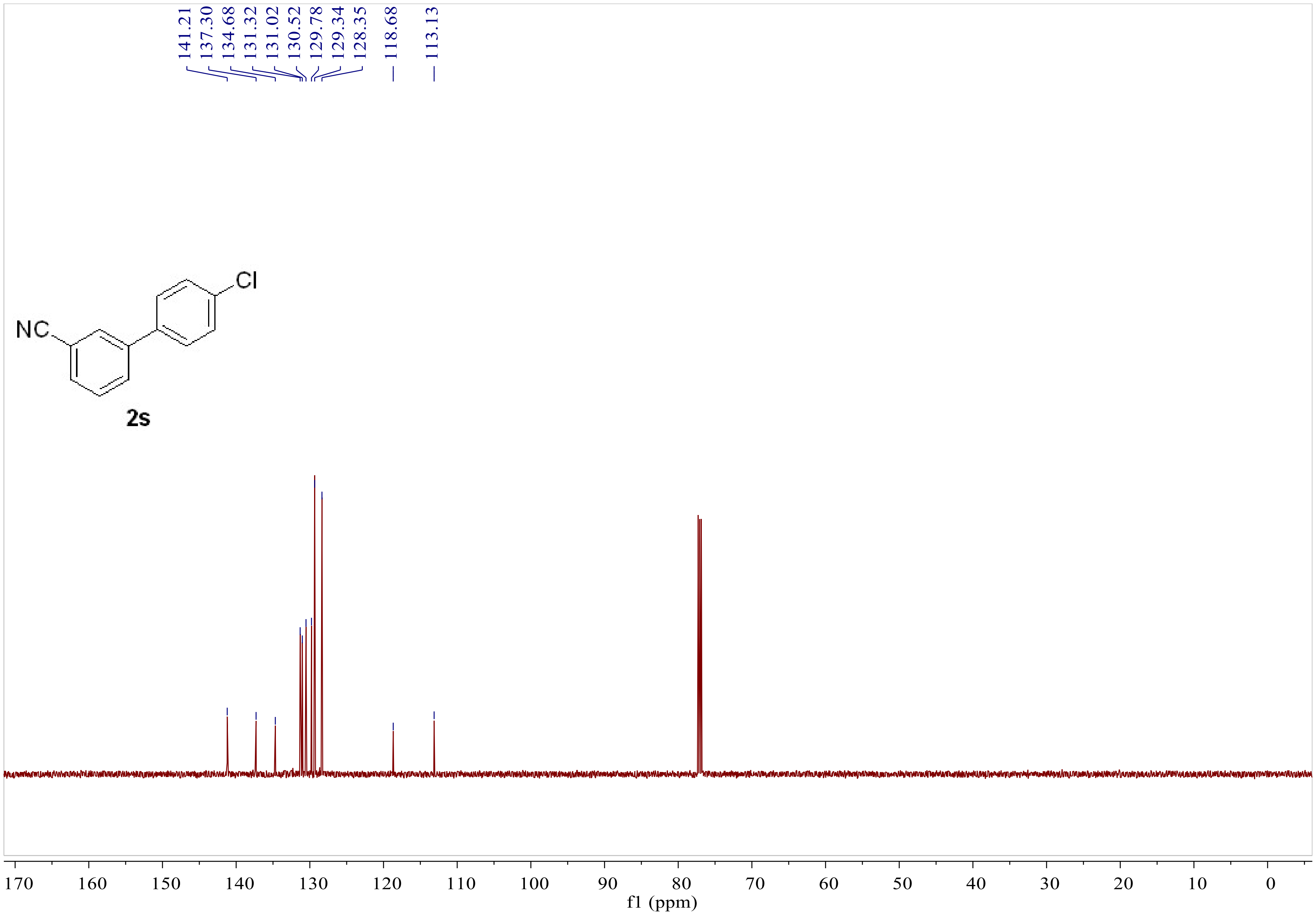
131.39
129.95
129.46
128.53
128.05
127.61
126.82
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.84
7.80
7.79
7.67
7.66
7.59
7.58
7.56
7.52
7.50
7.48
7.46
NC
Cl
2s
1.00
1.01
0.99
1.06
1.98
1.98
10.0
9.5
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
0.5
0.0
f1 (ppm)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113.13
NC
Cl
2s
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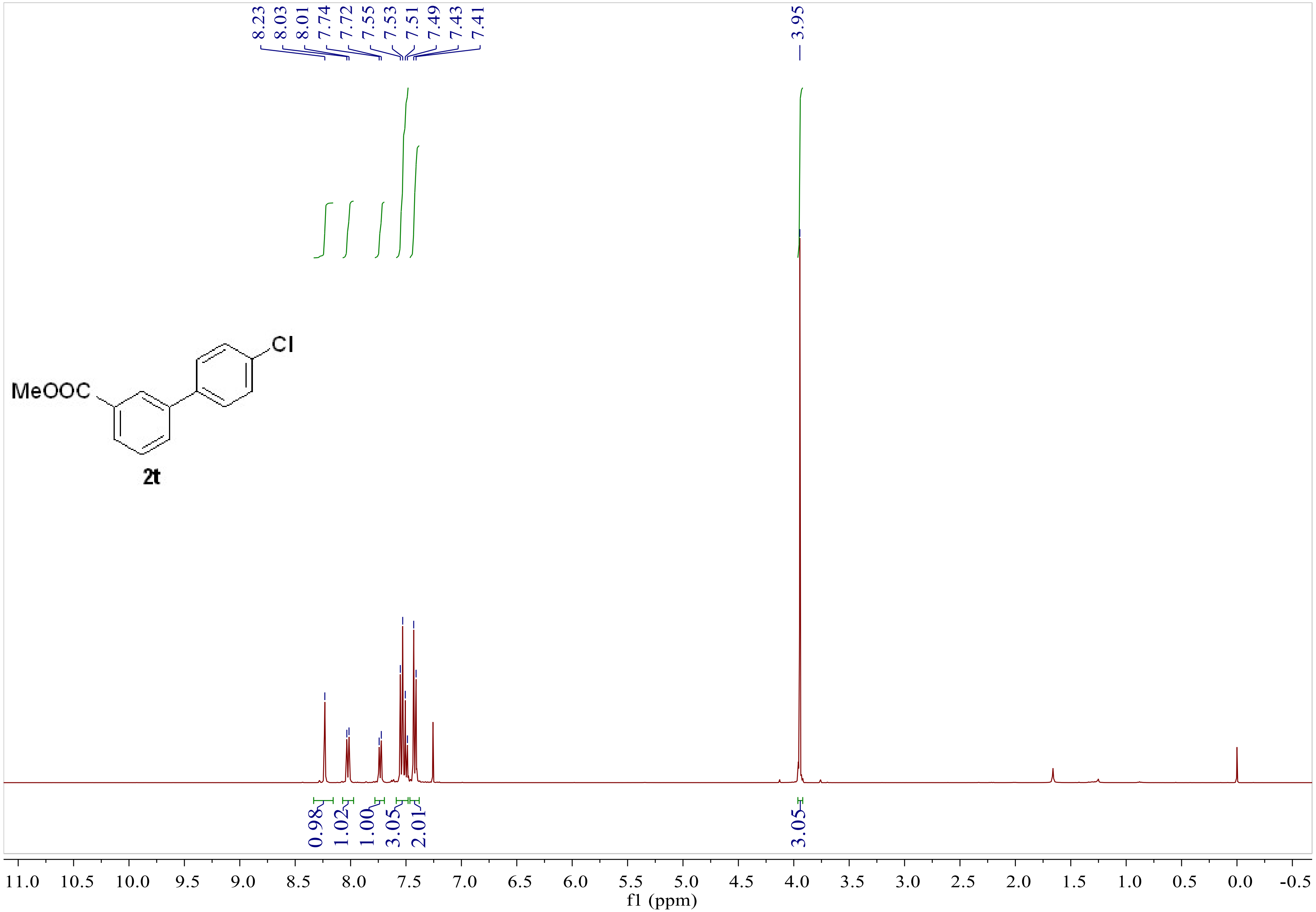
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Cl
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1.02
1.00
3.05
2.01
3.05
11.0
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10.0
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9.0
8.5
8.0
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6.5
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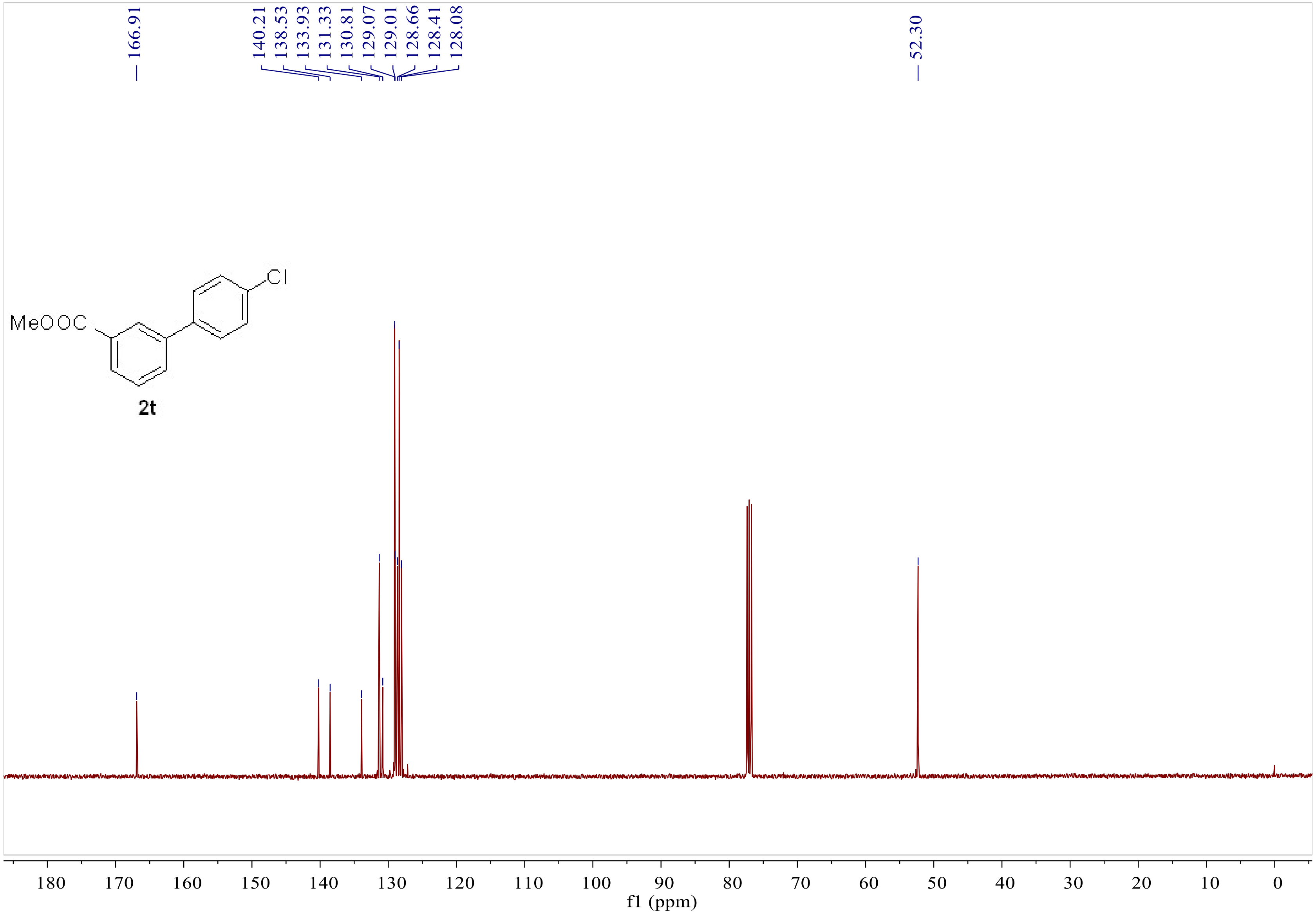
f1 (ppm)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Cl
2t
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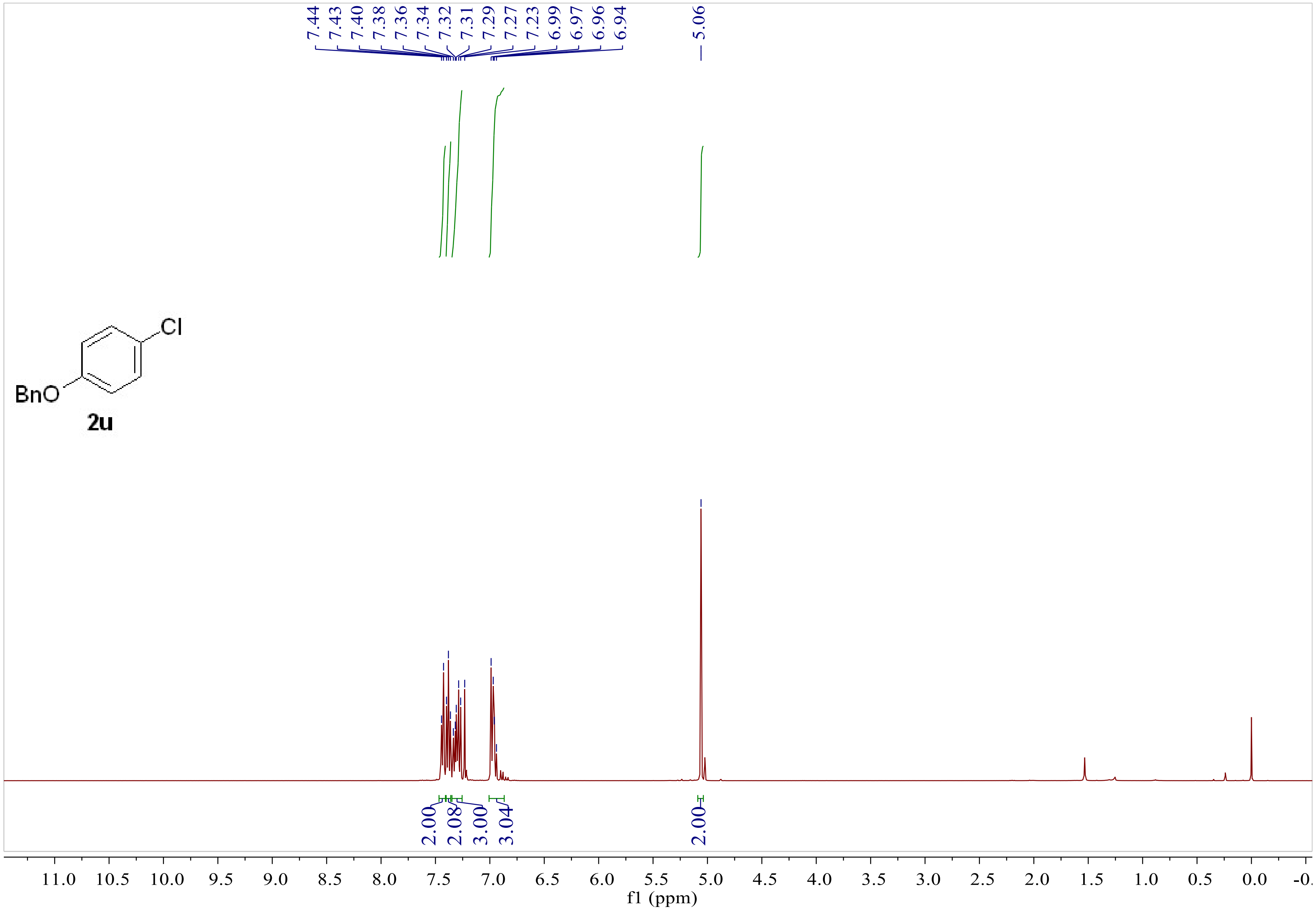7.44
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6.94
5.06
Cl
BnO
2u
2.00
2.08
3.00
3.04
2.00
11.0
10.5
10.0
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9.0
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6.0
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5.0
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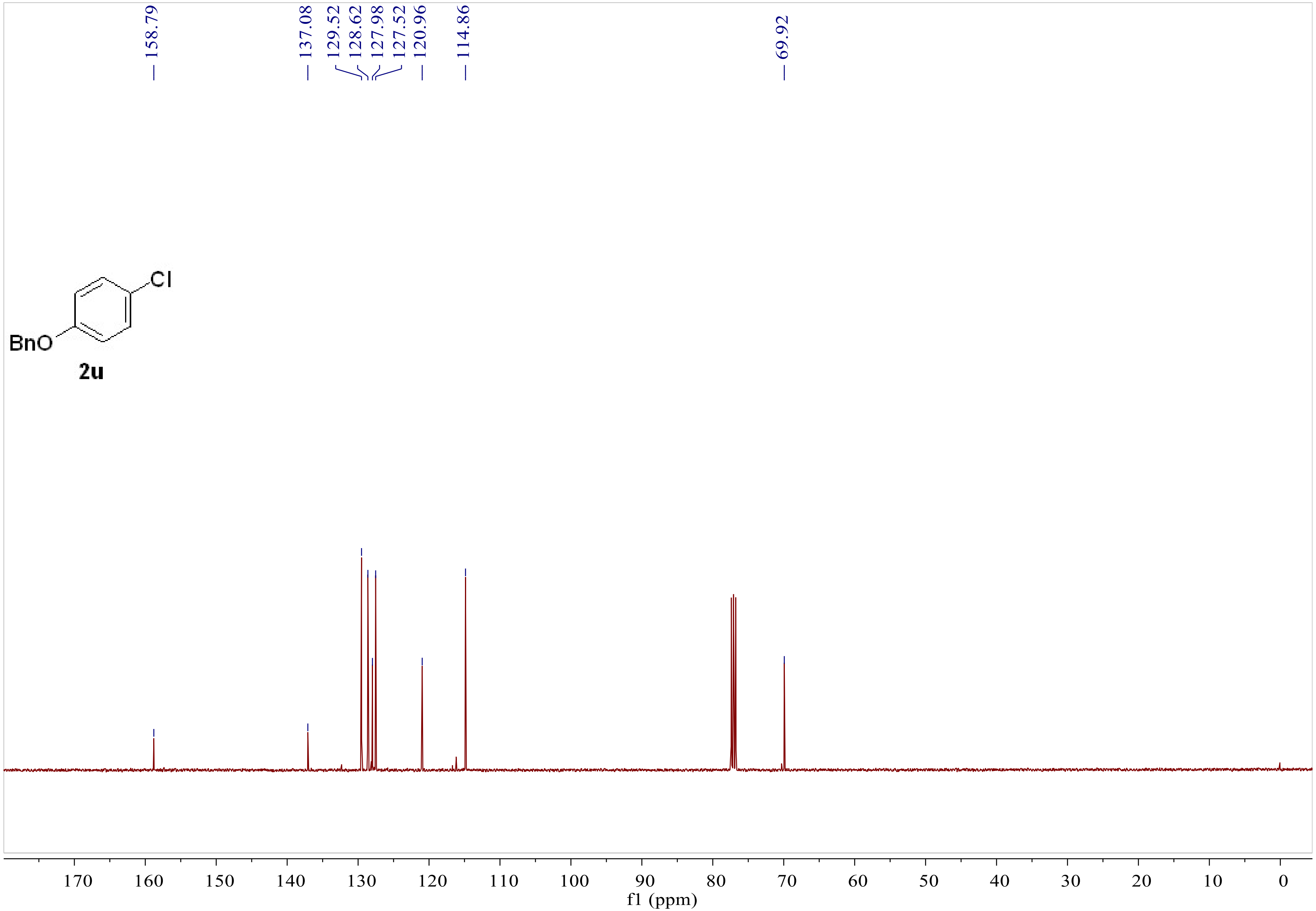
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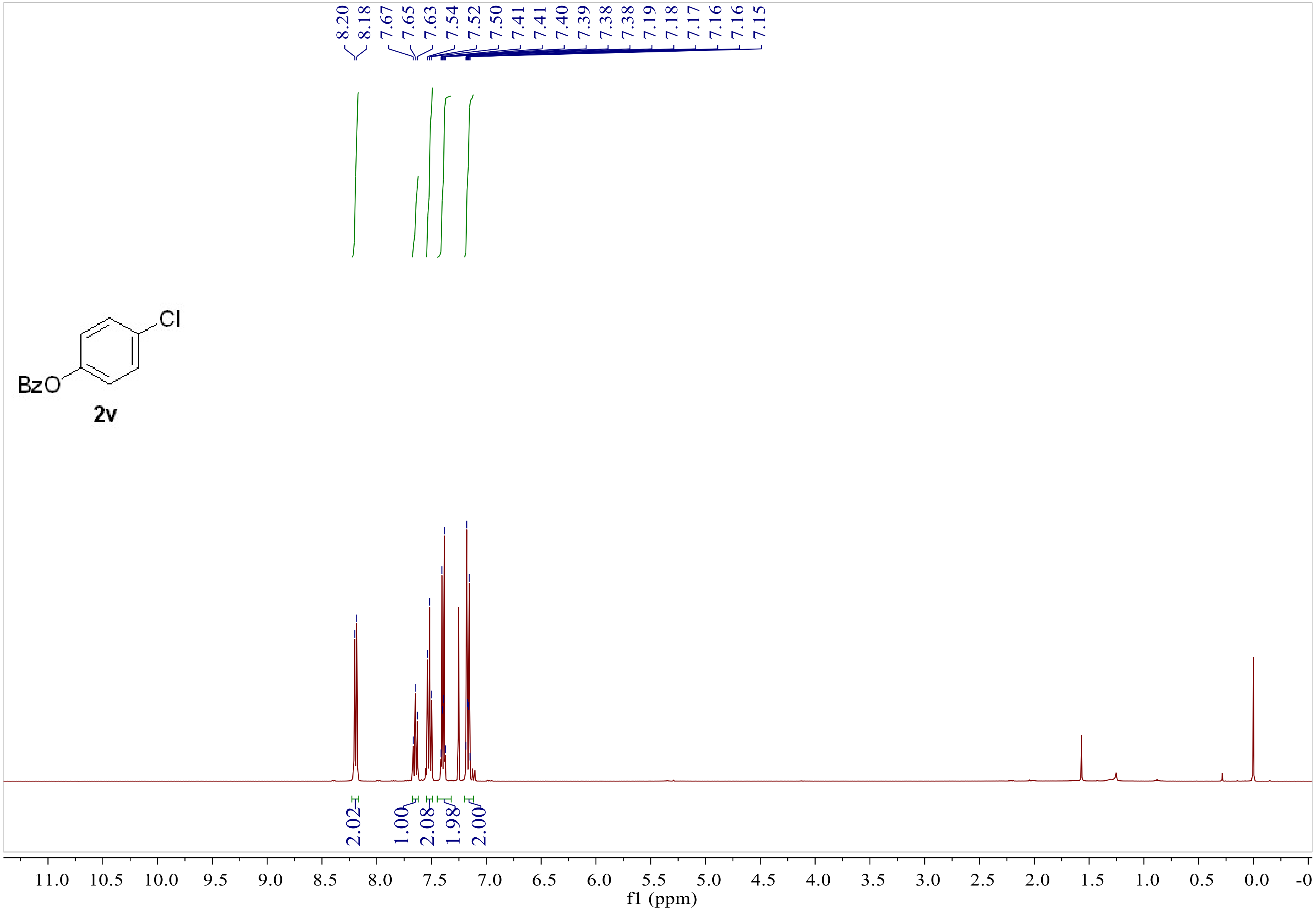
f1 (ppm)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7.18
7.17
7.16
7.16
7.15
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BzO
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1.00
2.08
1.98
2.00
11.0
10.5
10.0
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9.0
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8.0
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6.0
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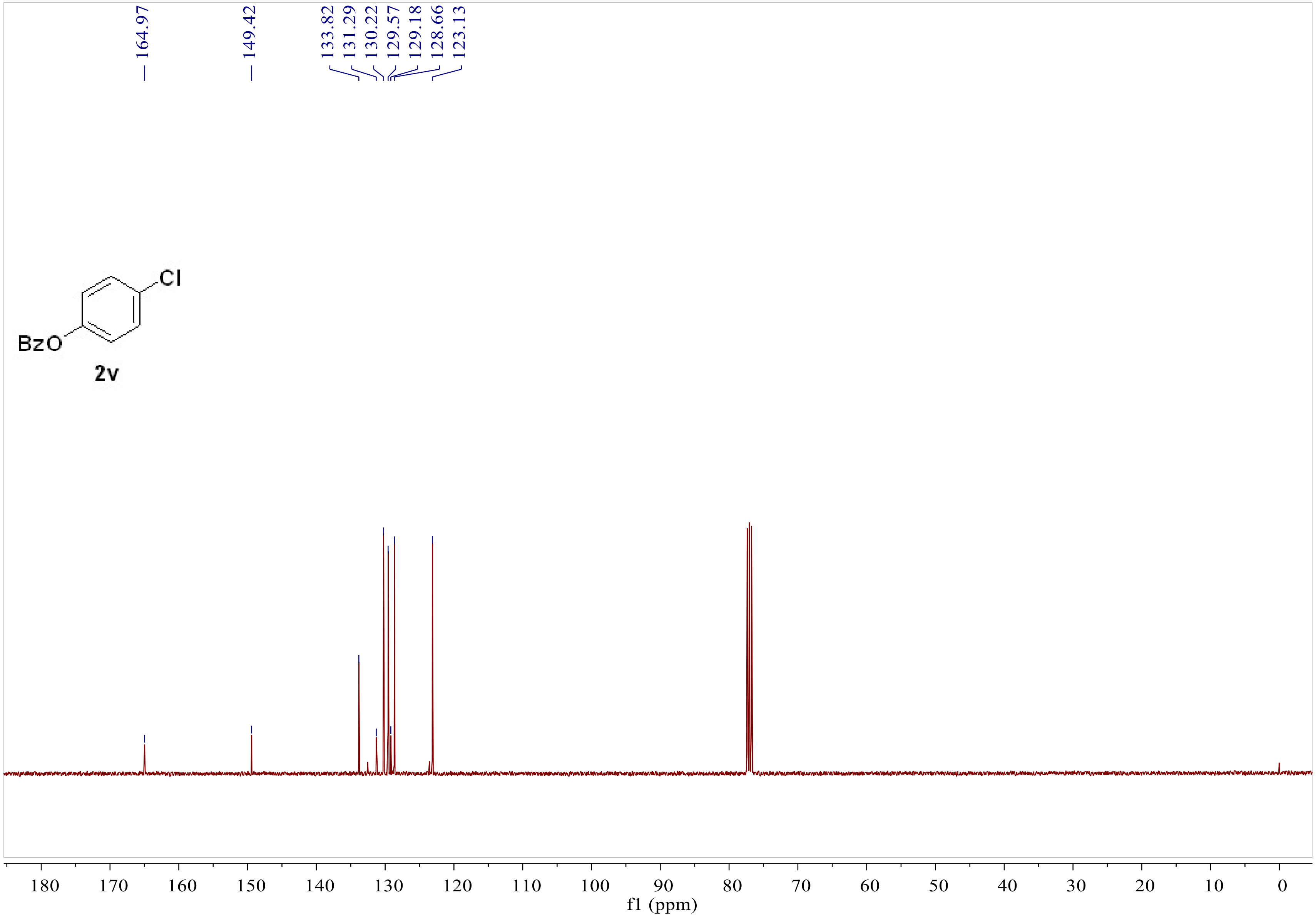
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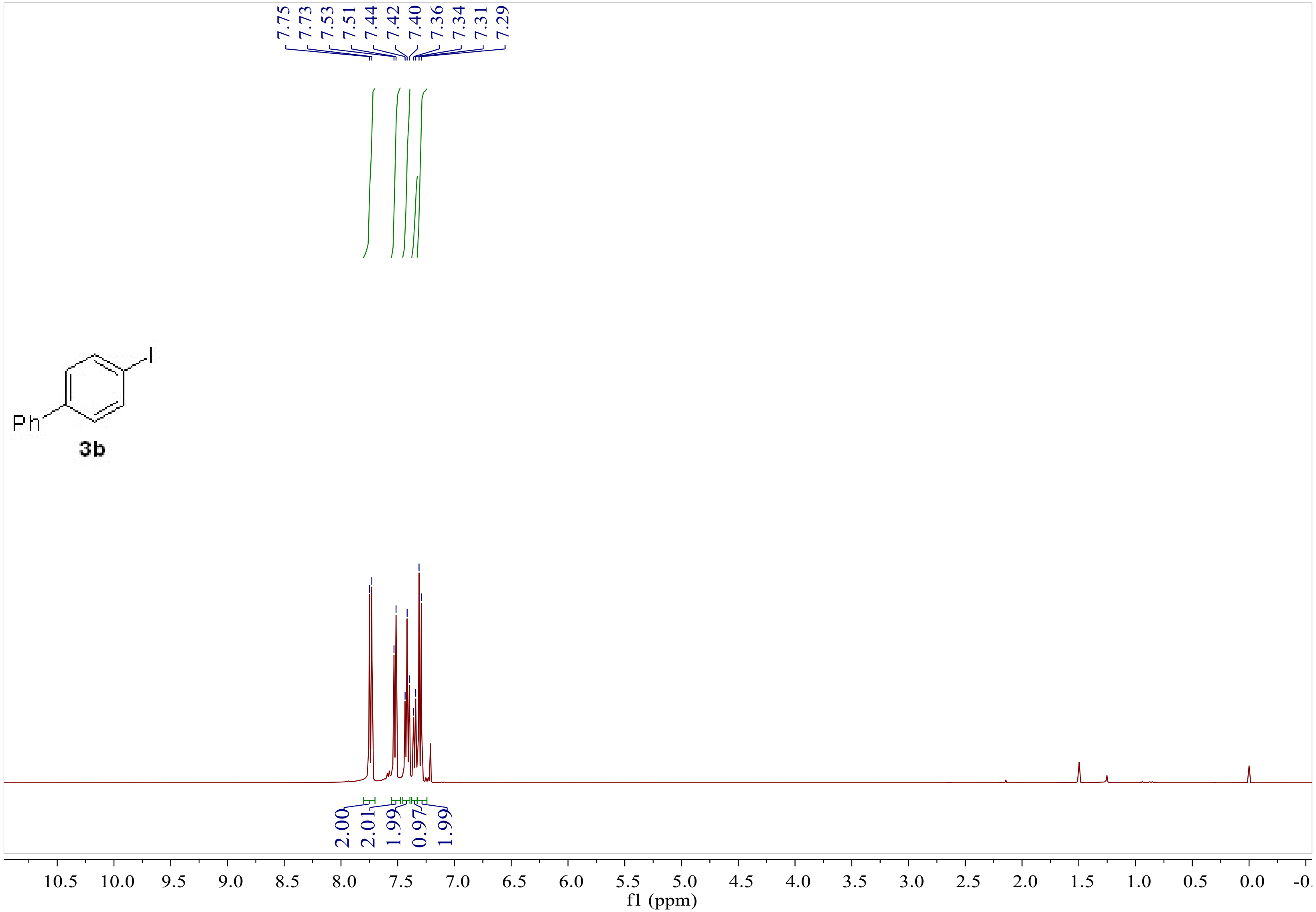
f1 (ppm)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7.29
I
Ph
3b
2.00
2.01
1.99
0.97
1.99
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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5.0
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2.0
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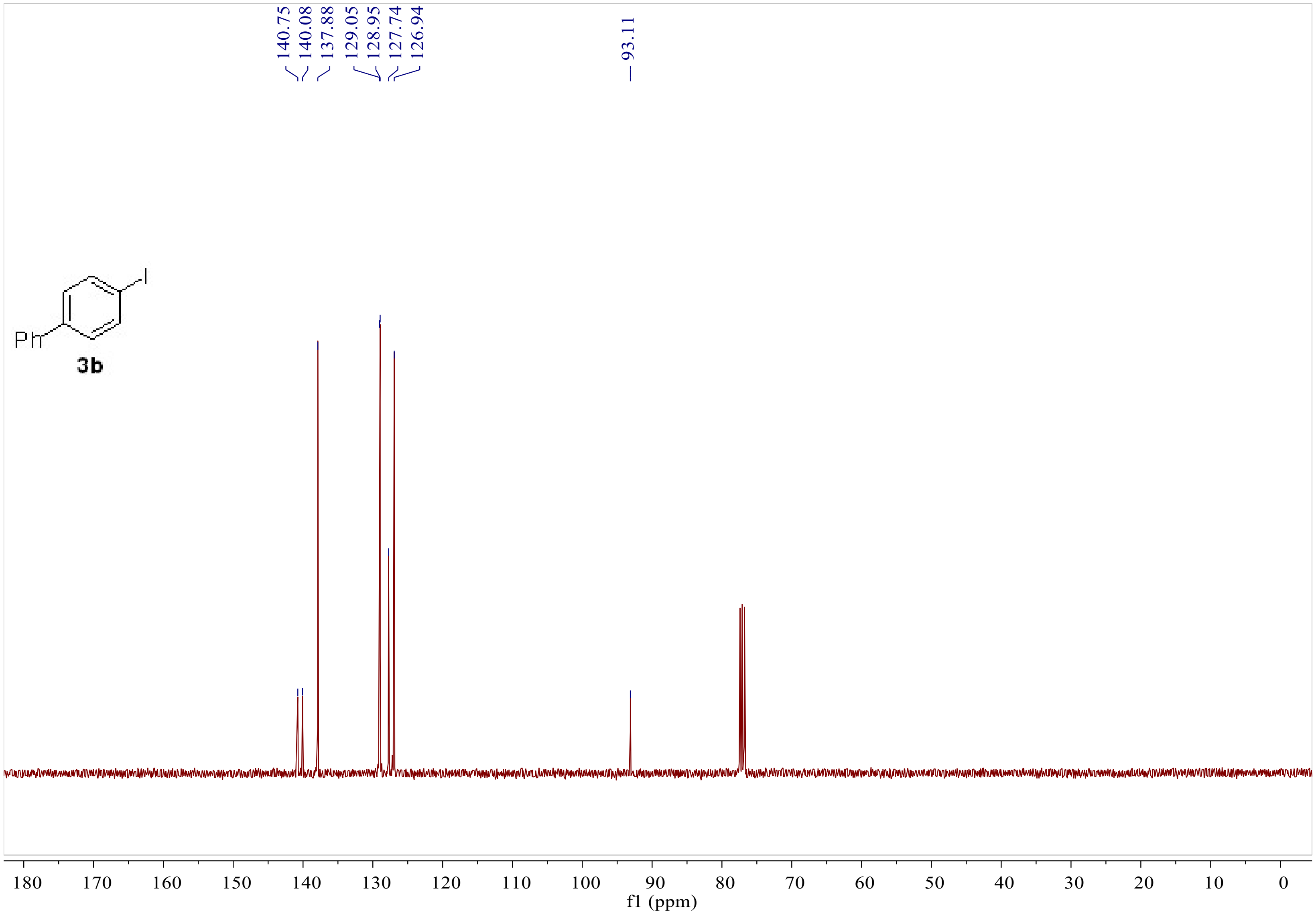
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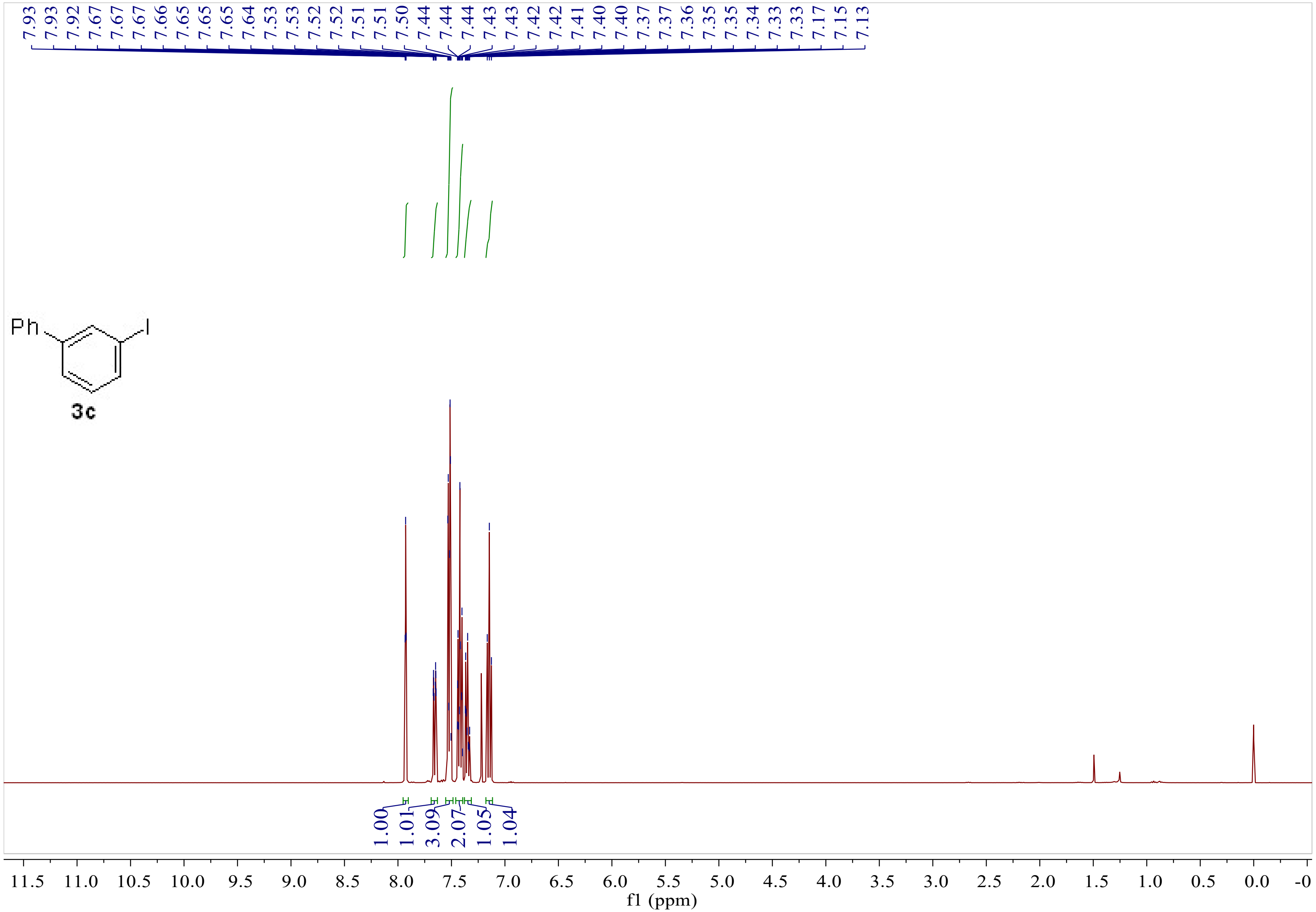
f1 (ppm)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7.52
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7.43
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7.33
7.17
7.15
7.13
Ph
I
3c
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1.01
3.09
2.07
1.05
1.04
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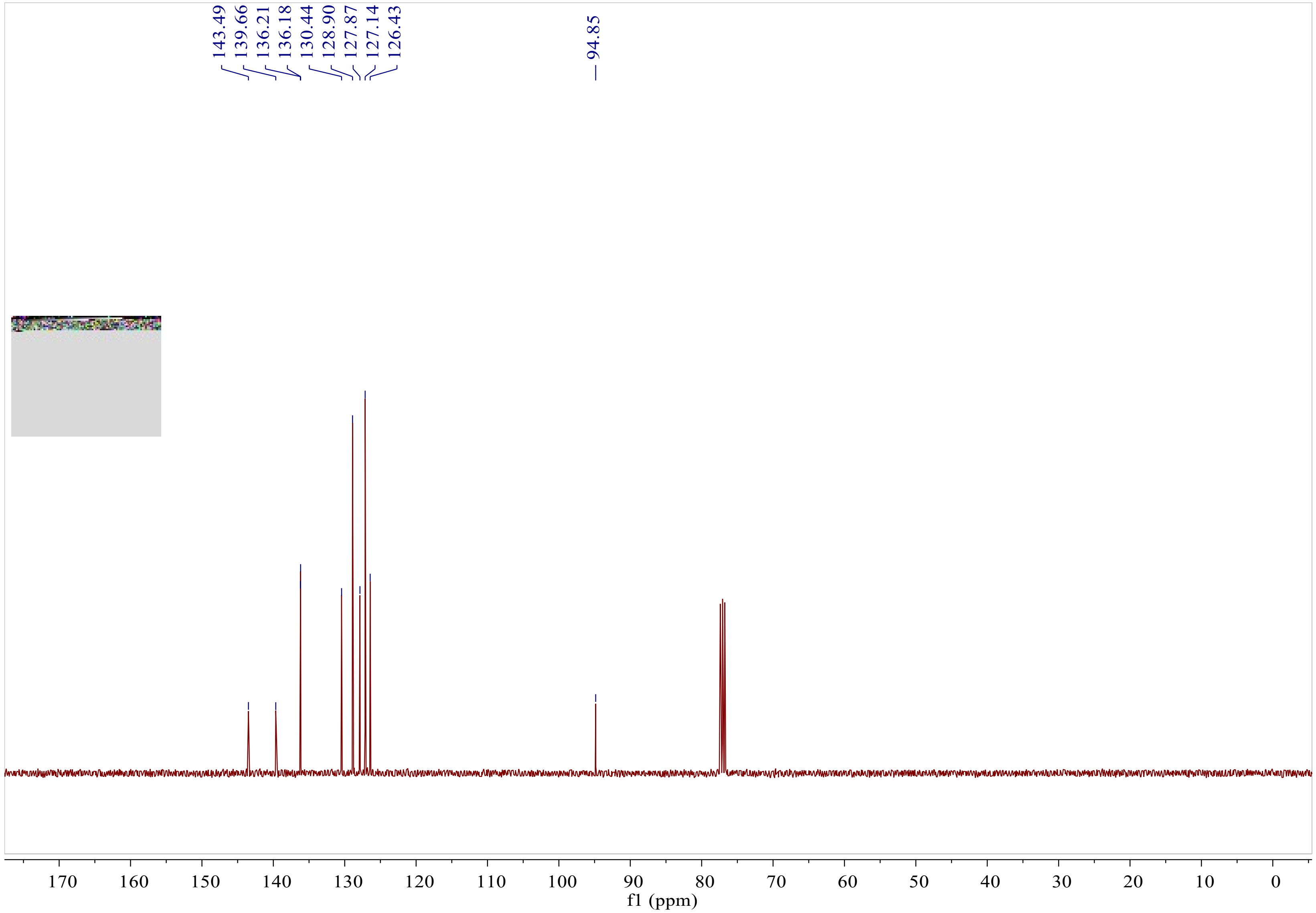
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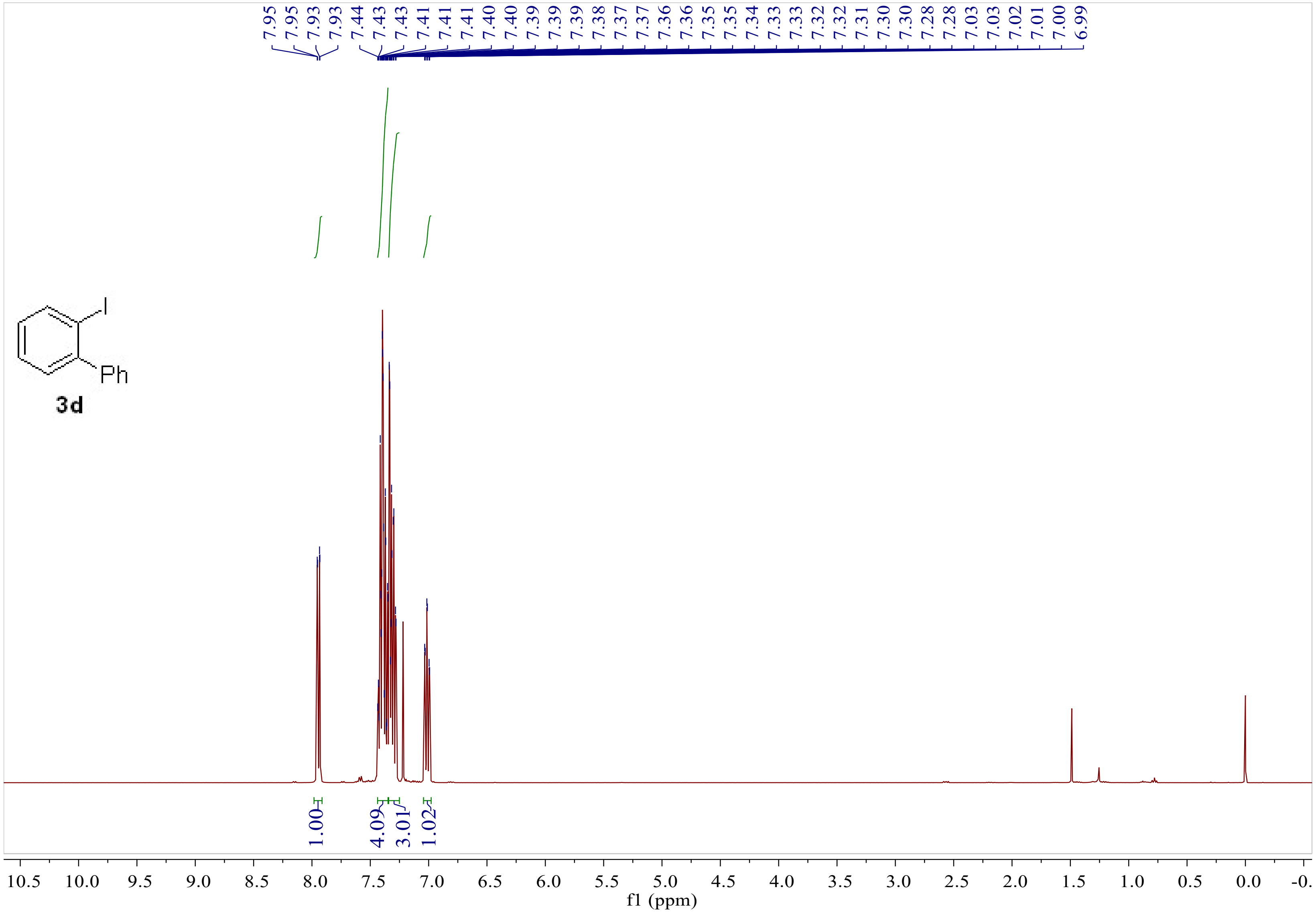
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7.00
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Ph
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3.01
1.02
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9.0
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7.5
7.0
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5.0
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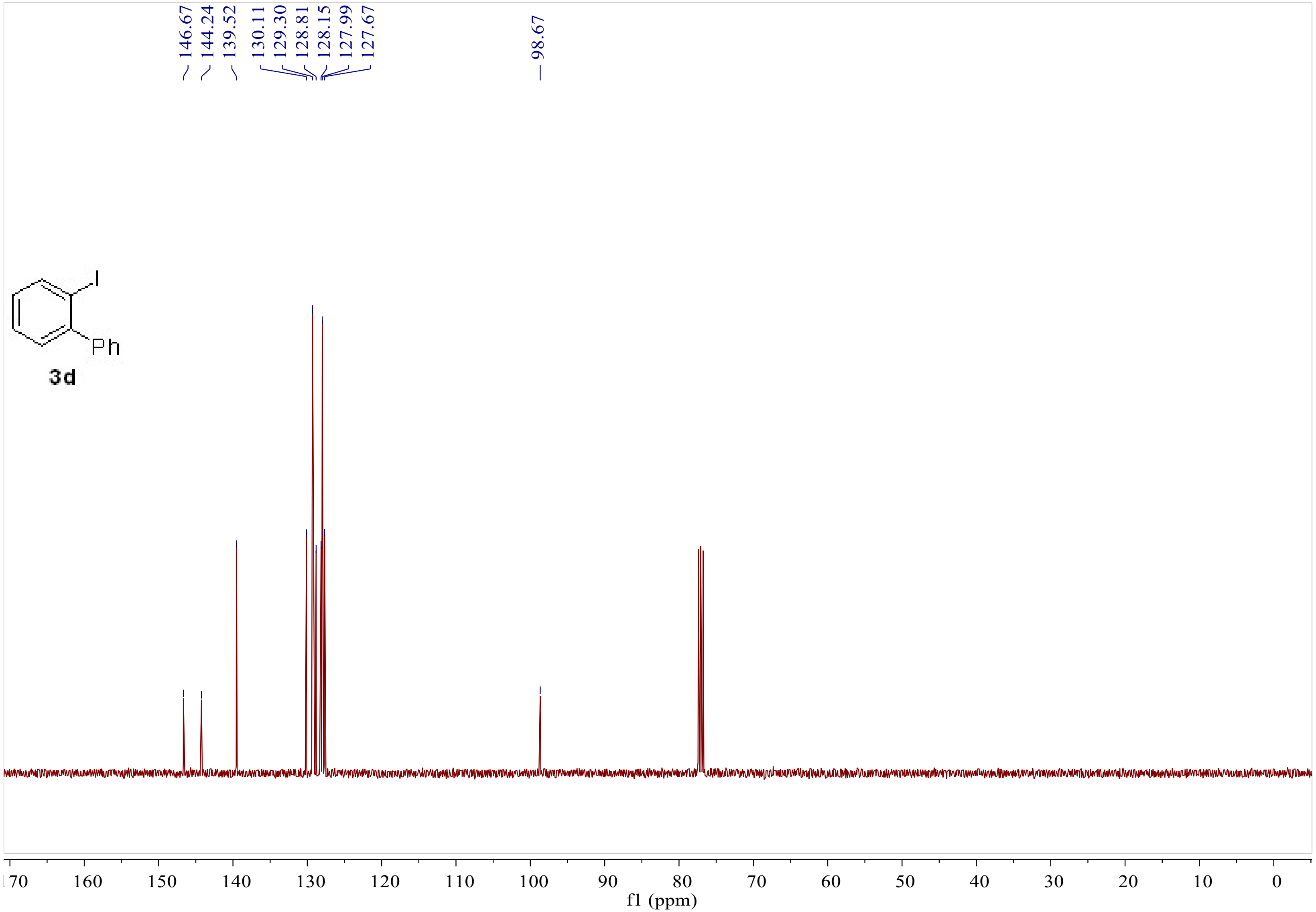
f1 (ppm)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98.67
I
Ph
3d
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120
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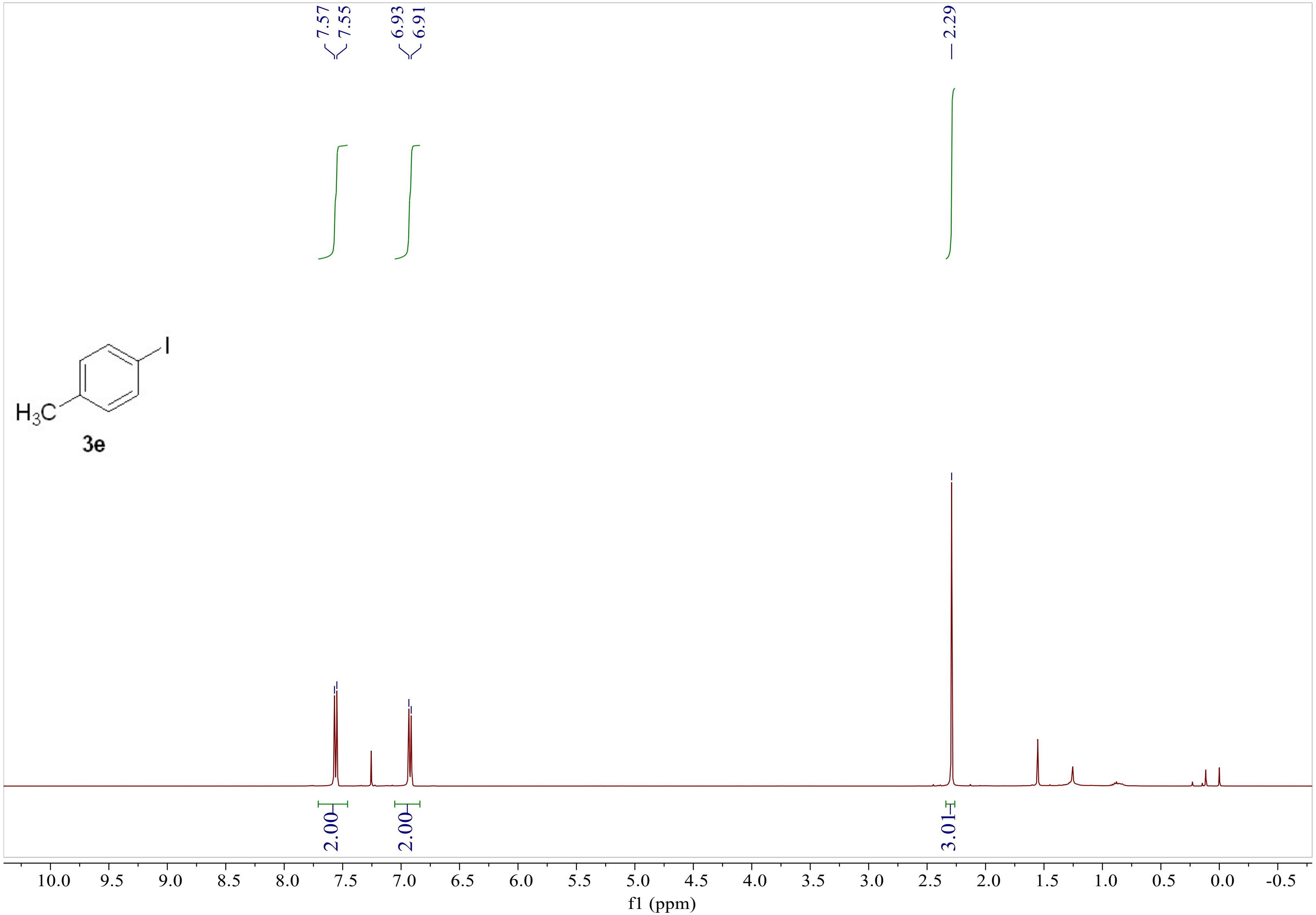
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I
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7.0
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6.0
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5.0
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2.0
1.5
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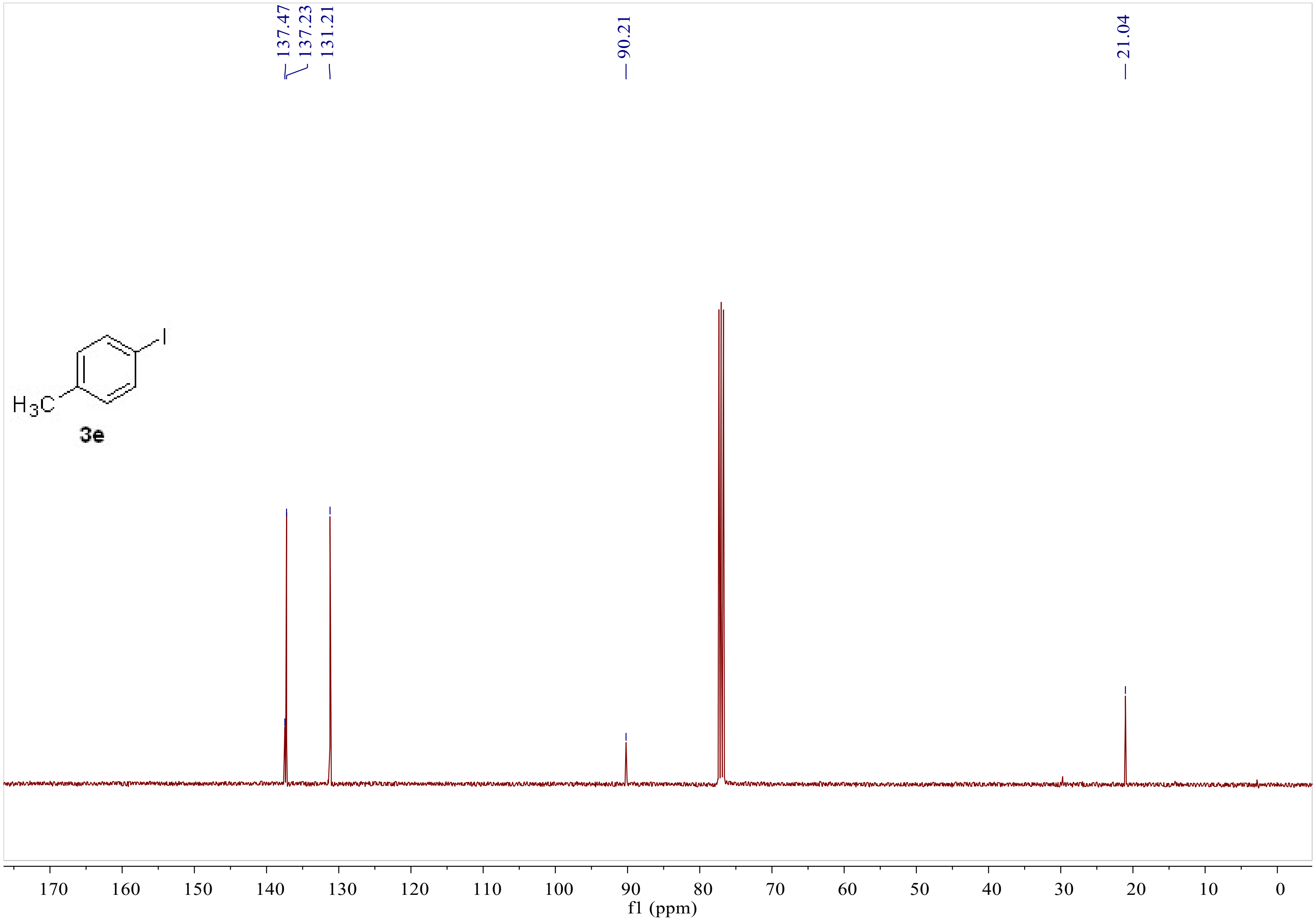
f1 (ppm)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90
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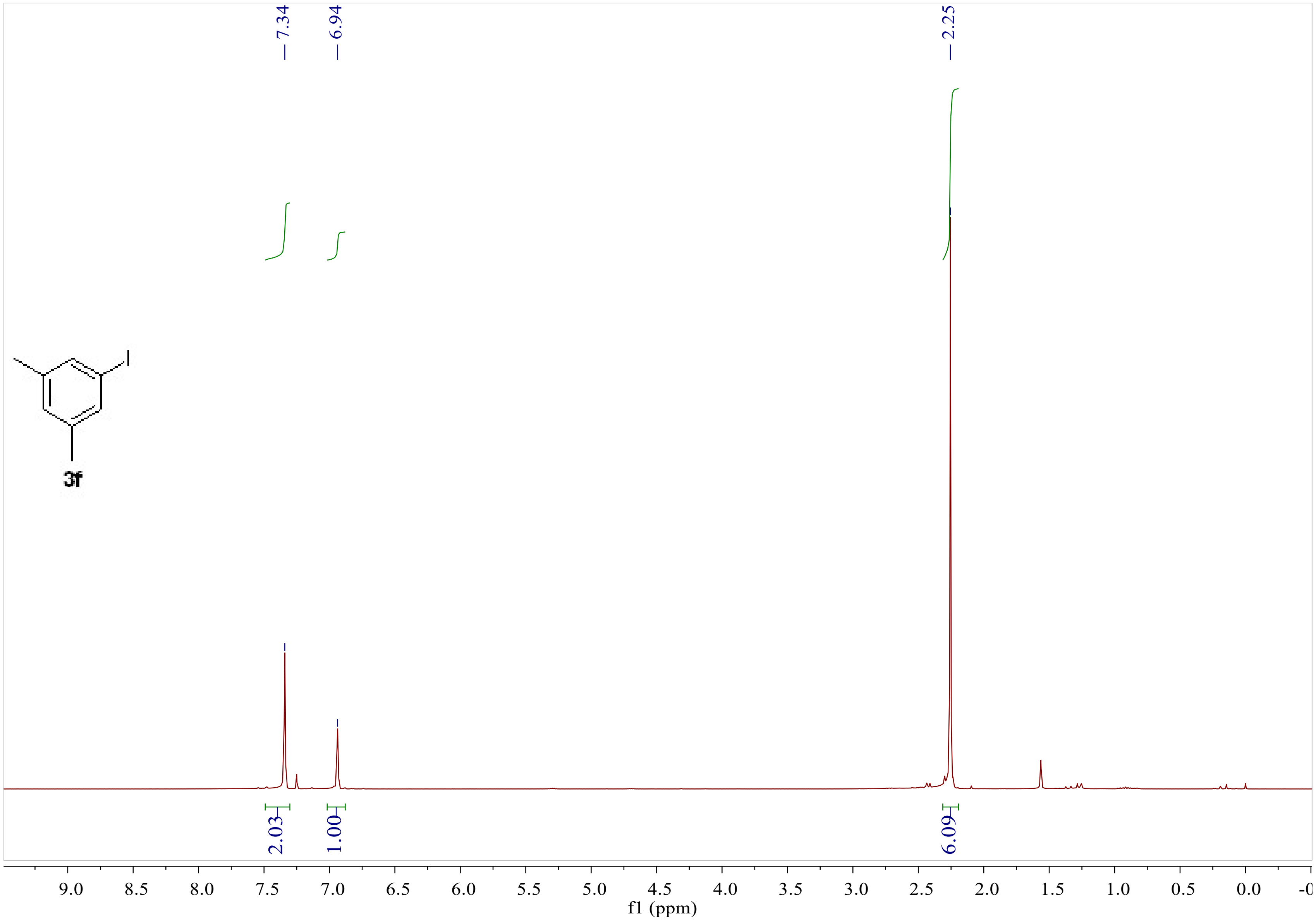
f1 (ppm)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6.0
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5.0
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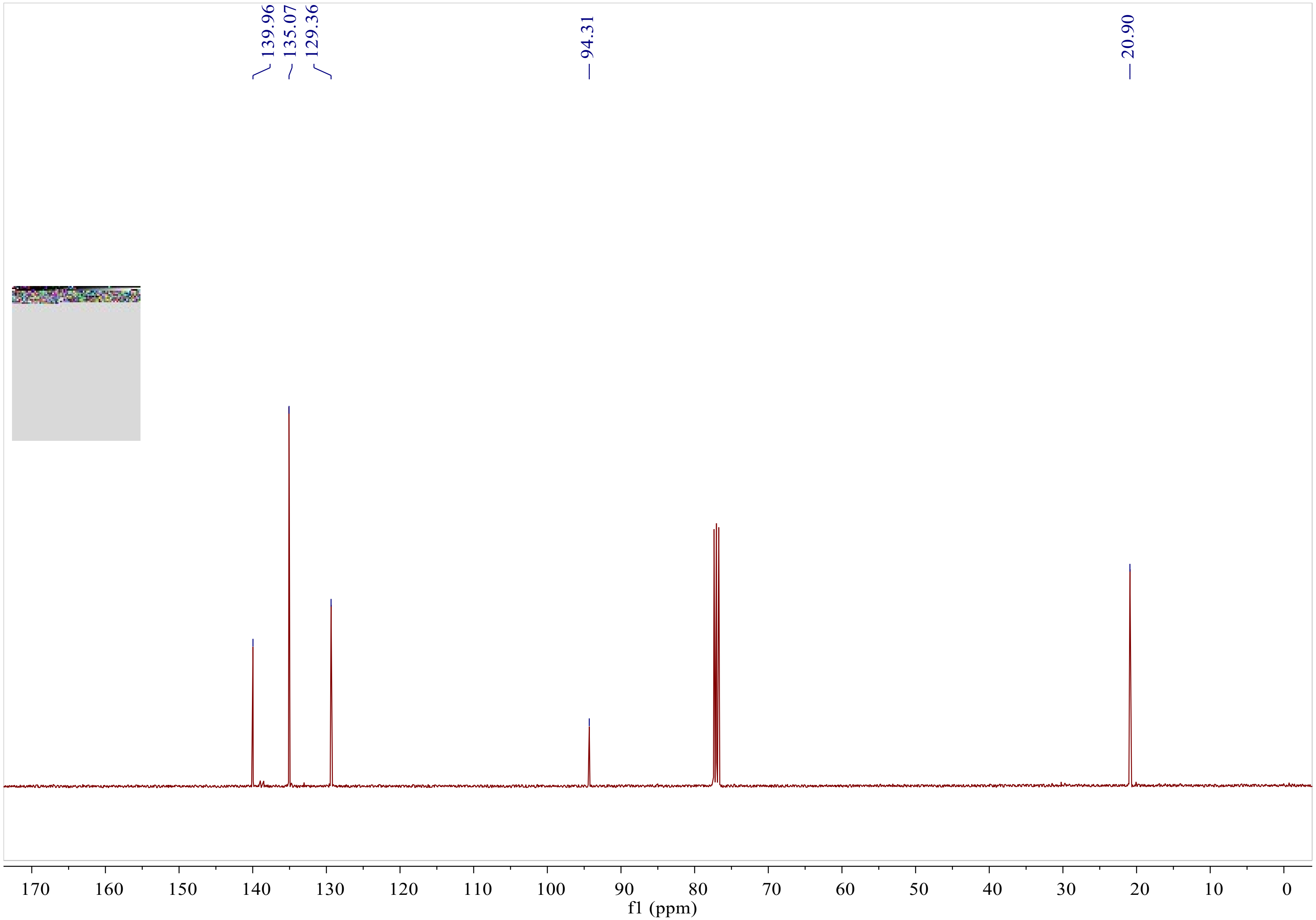
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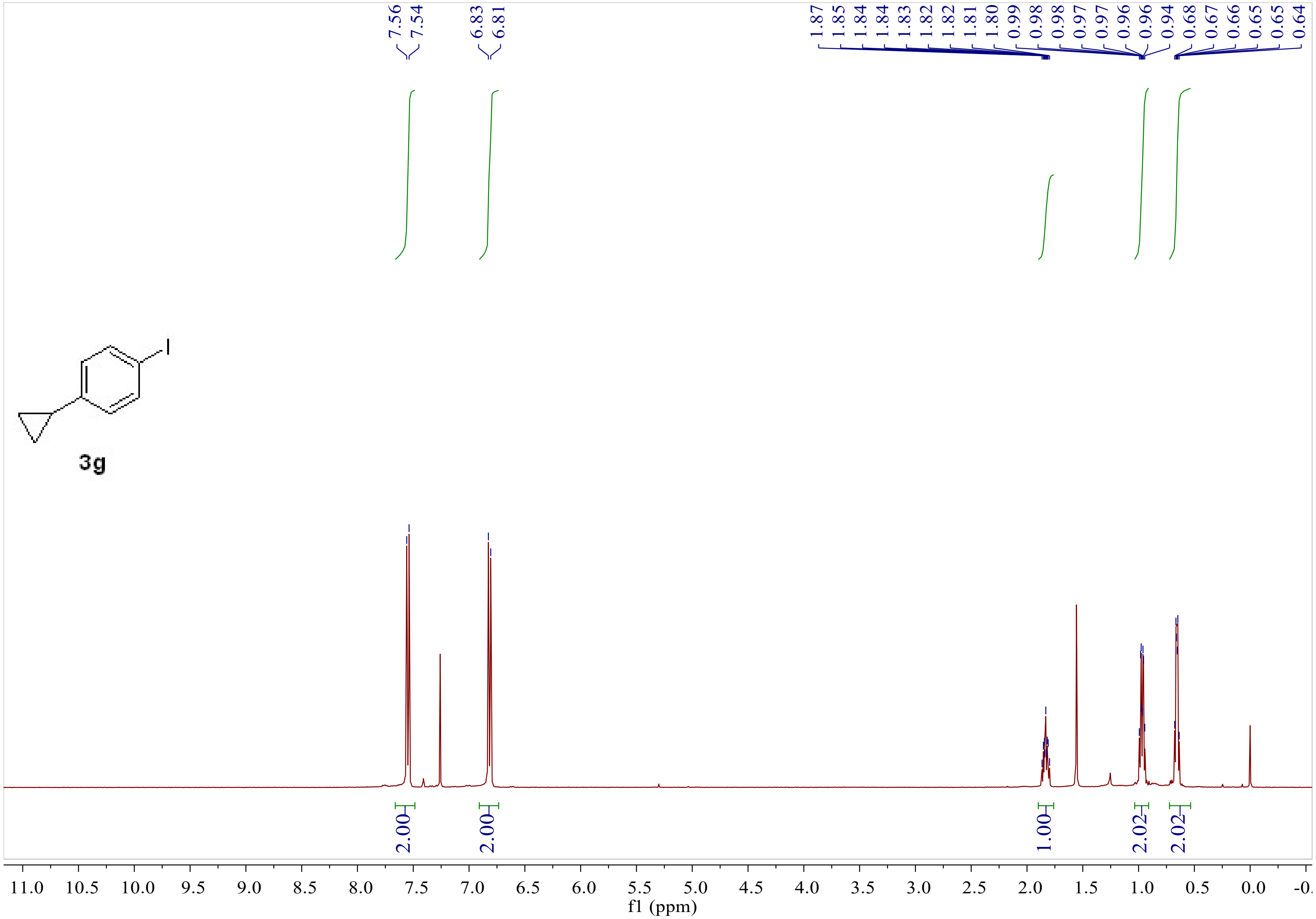
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0.98
0.98
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0.97
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0.96
0.94
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0.65
0.65
0.64
I
3g
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1.00
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2.02
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9.0
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6.0
5.5
5.0
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2.0
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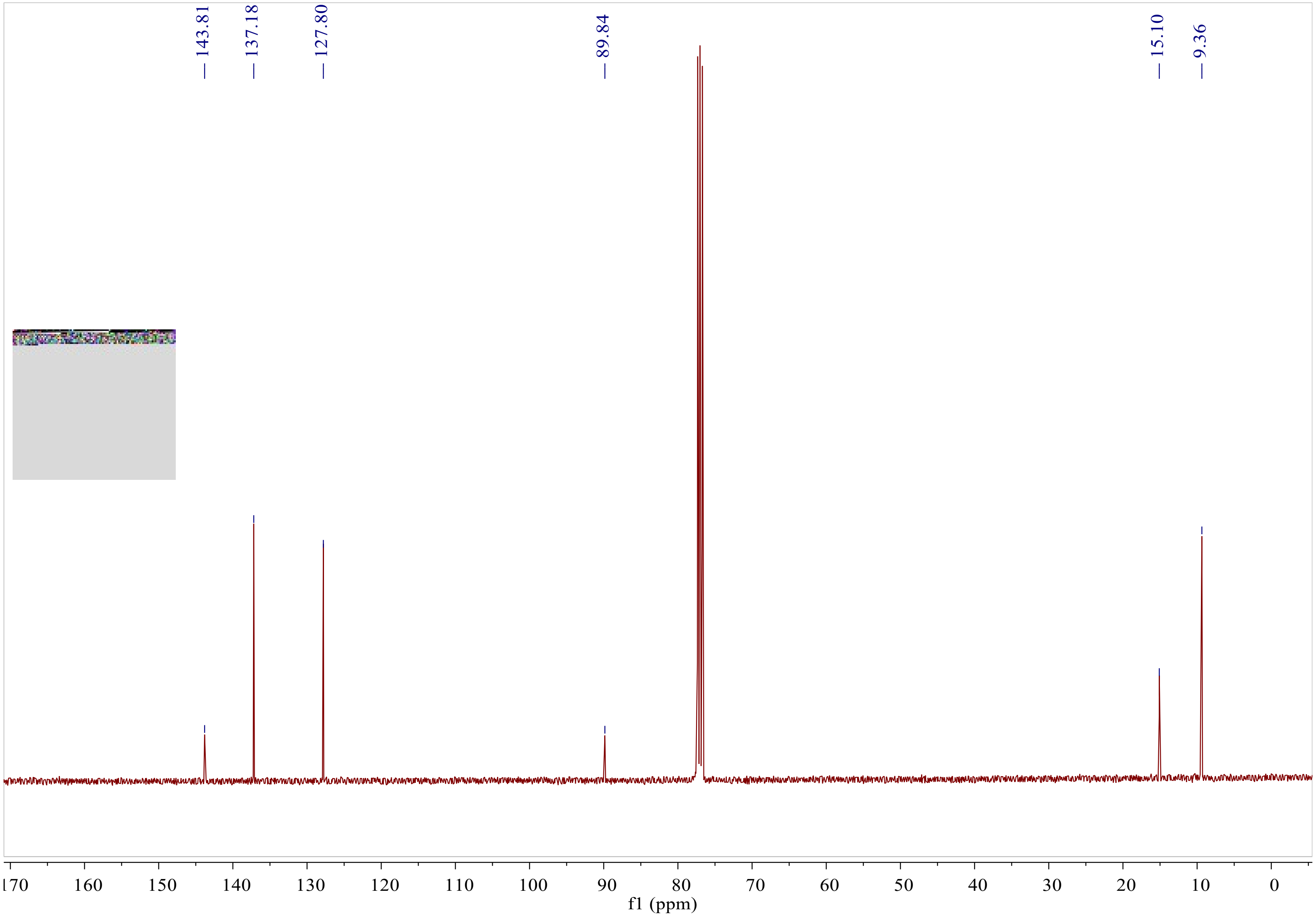
f1 (ppm)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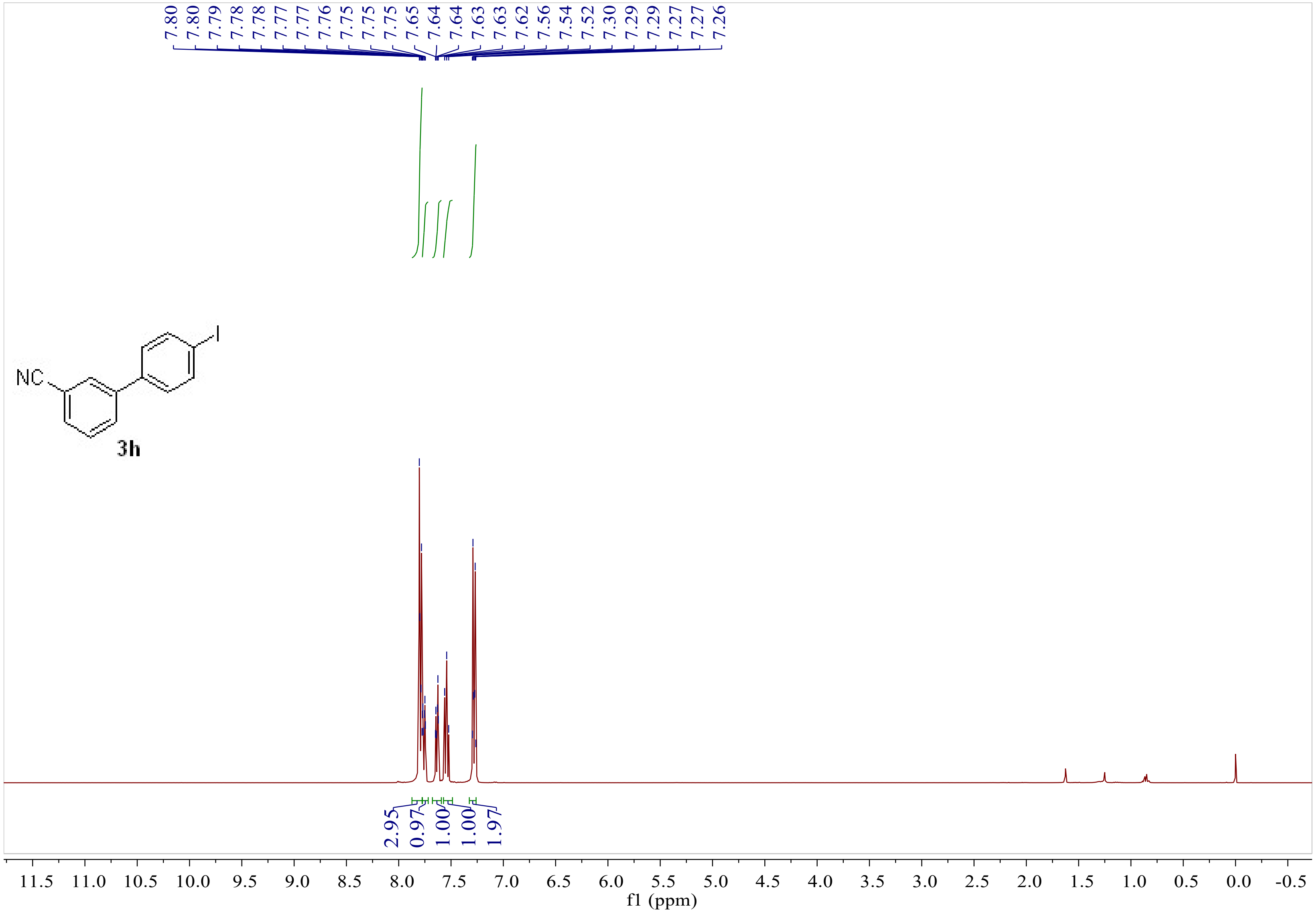
f1 (ppm)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7.63
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7.27
7.26
NC
I
3h
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1.00
1.00
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11.5
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10.0
9.5
9.0
8.5
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7.5
7.0
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5.5
5.0
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2.0
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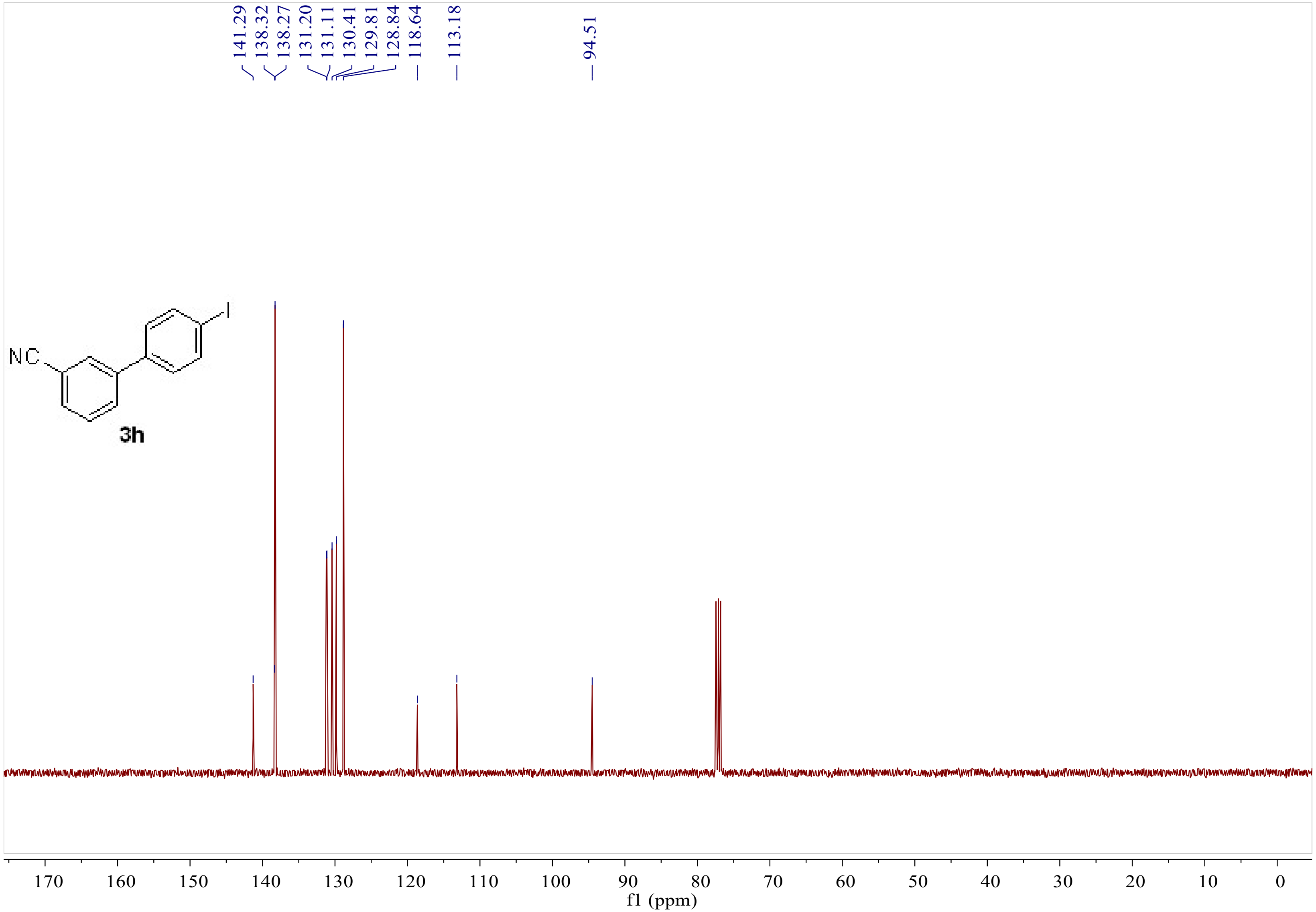
f1 (ppm)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NC
I
170
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100
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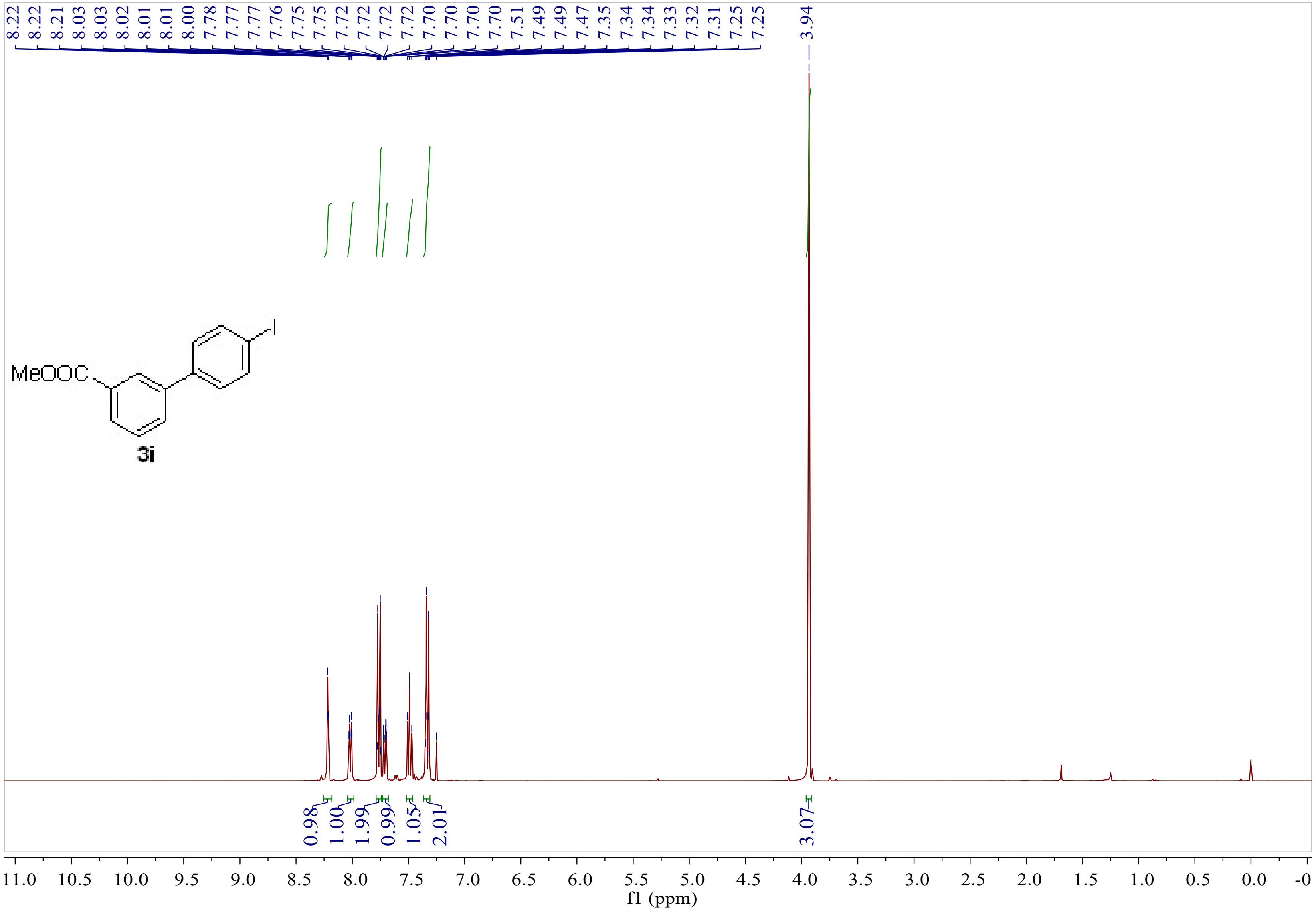
30
20
10
0
f1 (ppm)

3h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7.75
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7.70
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7.49
7.49
7.47
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7.33
7.32
7.31
7.25
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3.94
MeOOC
I
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1.99
0.99
1.05
2.01
3.07
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10.5
10.0
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9.0
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8.0
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5.0
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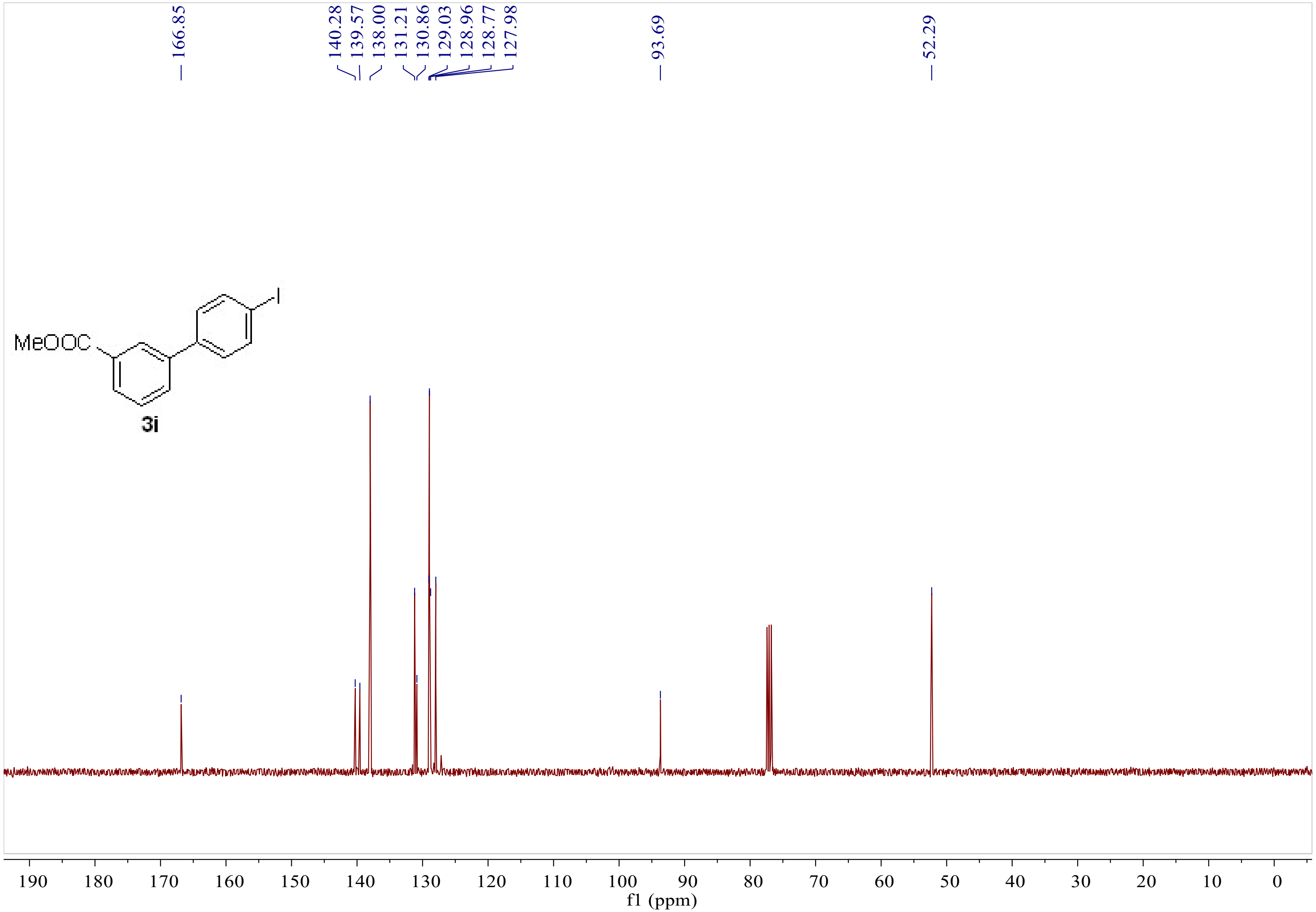
f1 (ppm)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127.98
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MeOOC
I
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180
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100
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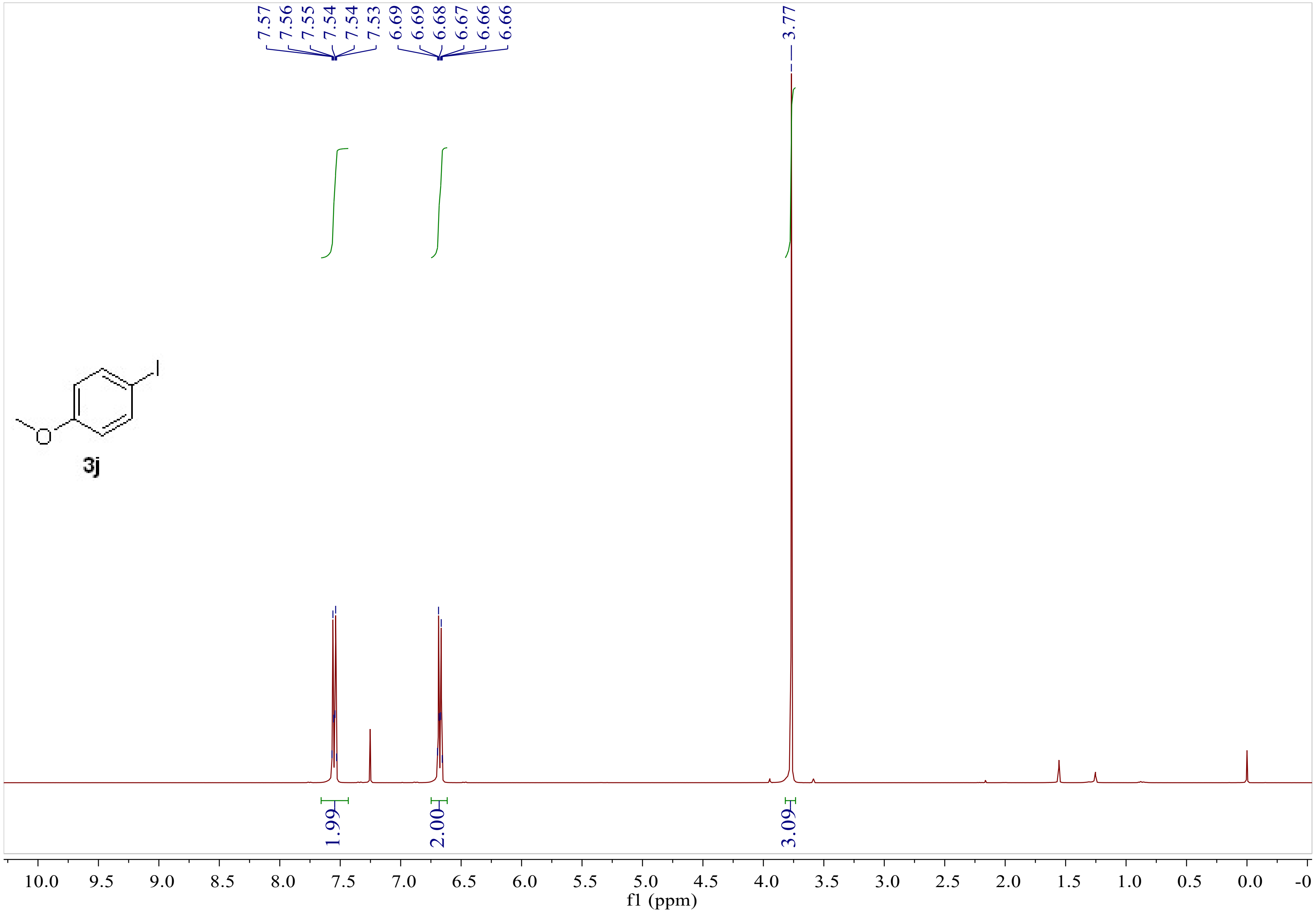
0
f1 (ppm)

3i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6.66
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I
O
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9.0
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7.5
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6.0
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3.0
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2.0
1.5
1.0
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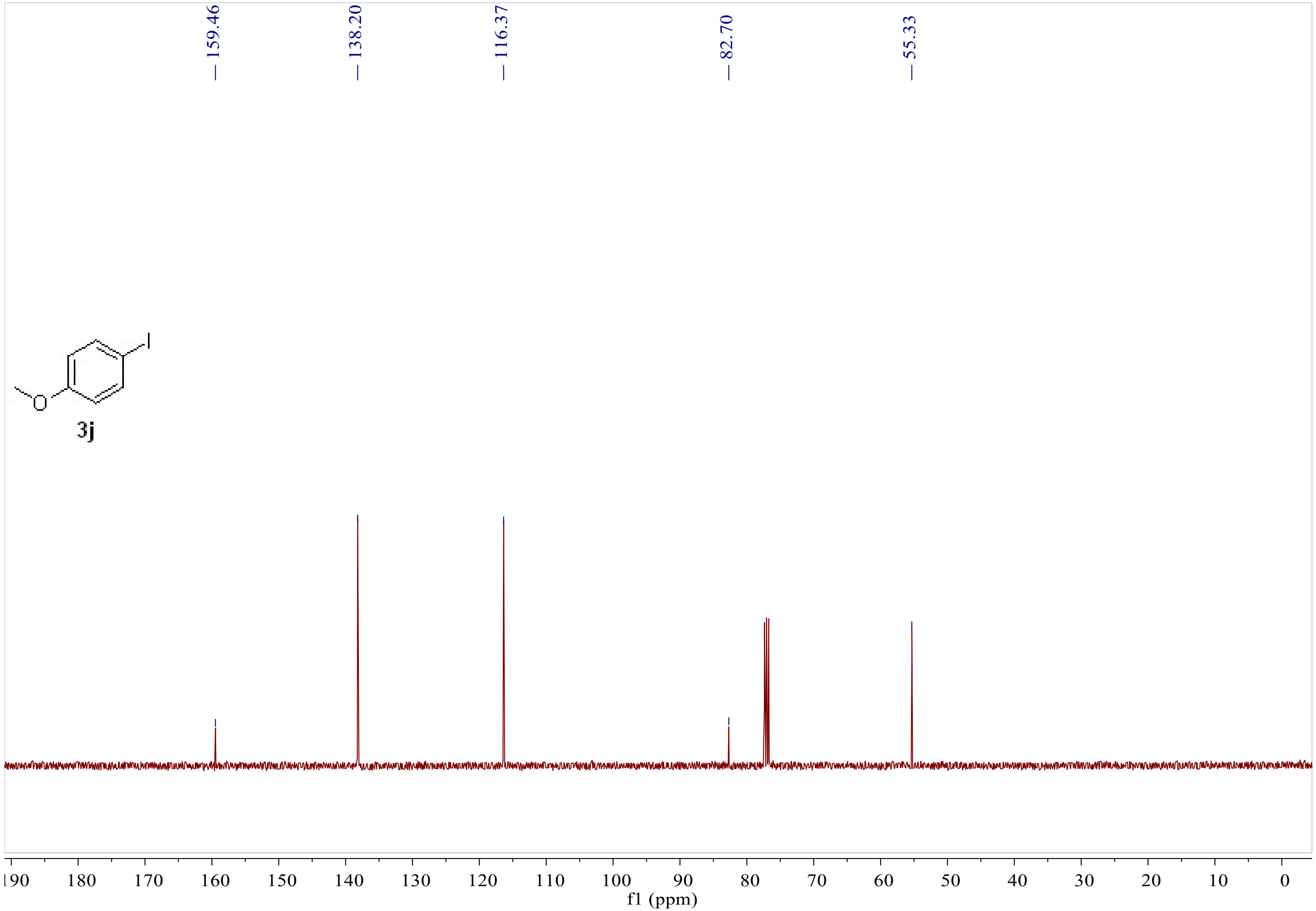
-0
f1 (ppm)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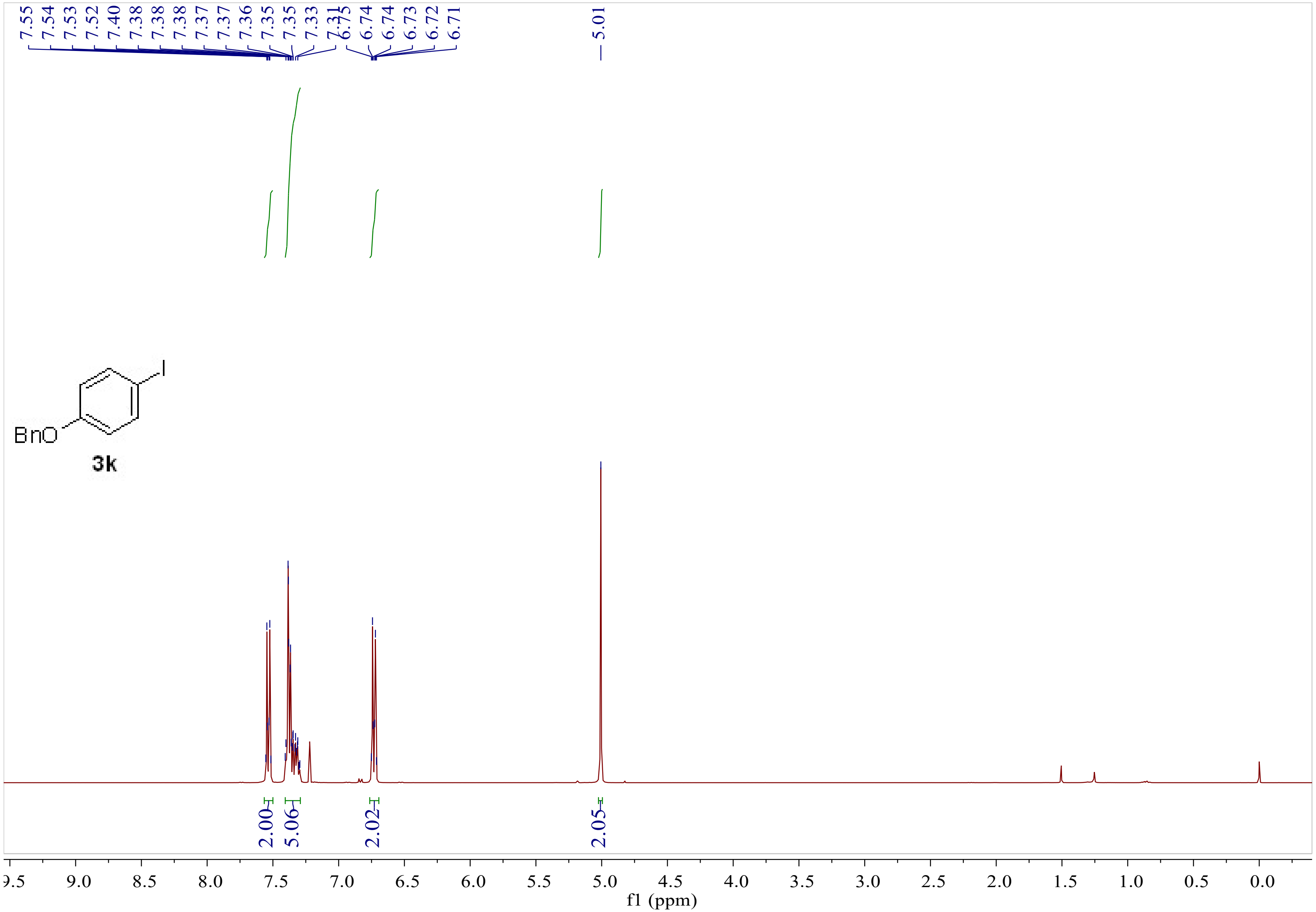
f1 (ppm)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7.35
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6.72
6.71
5.01
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BnO
3k
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5.06
2.02
2.05
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9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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2.5
2.0
1.5
1.0
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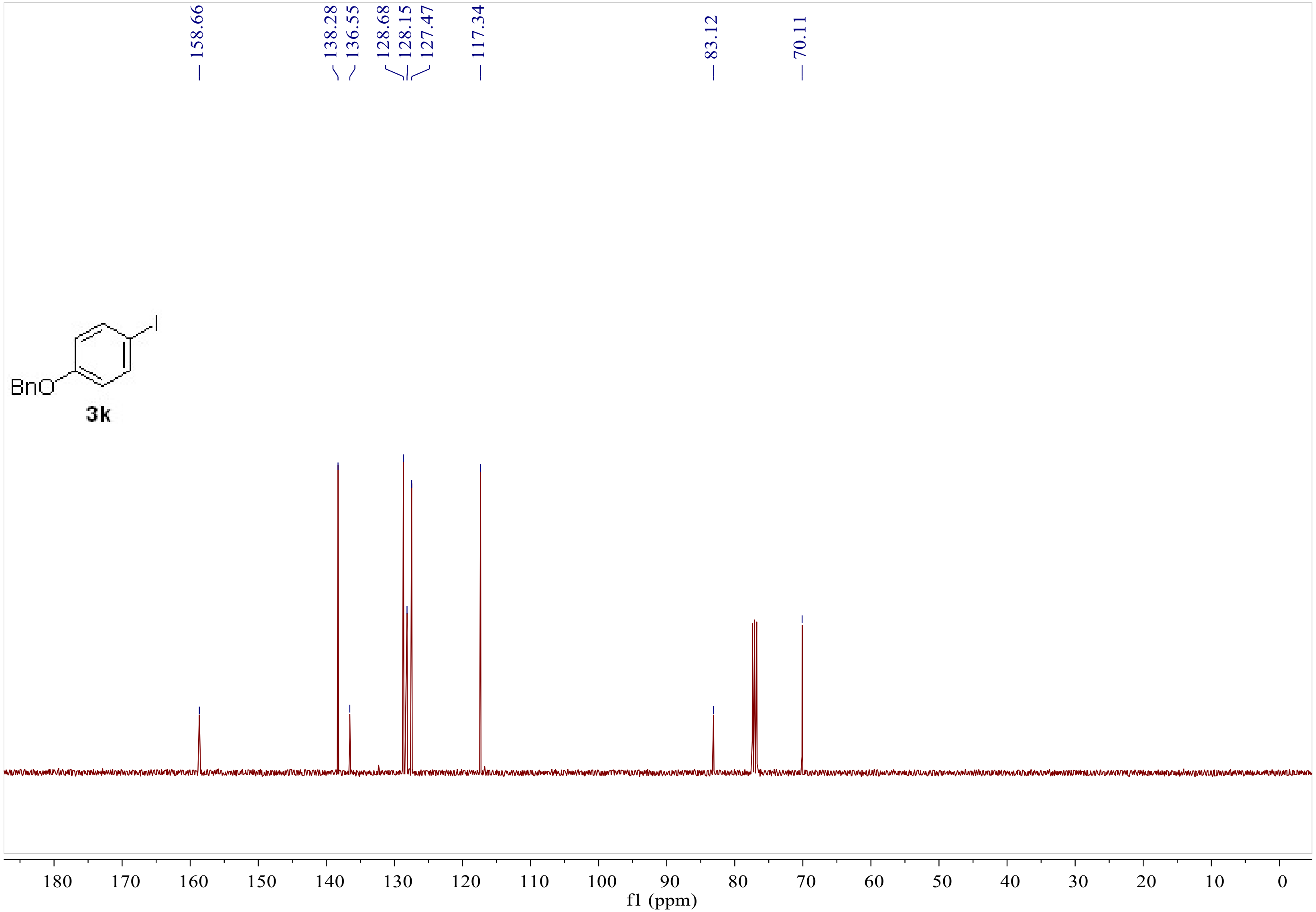
0.0
f1 (ppm)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BnO
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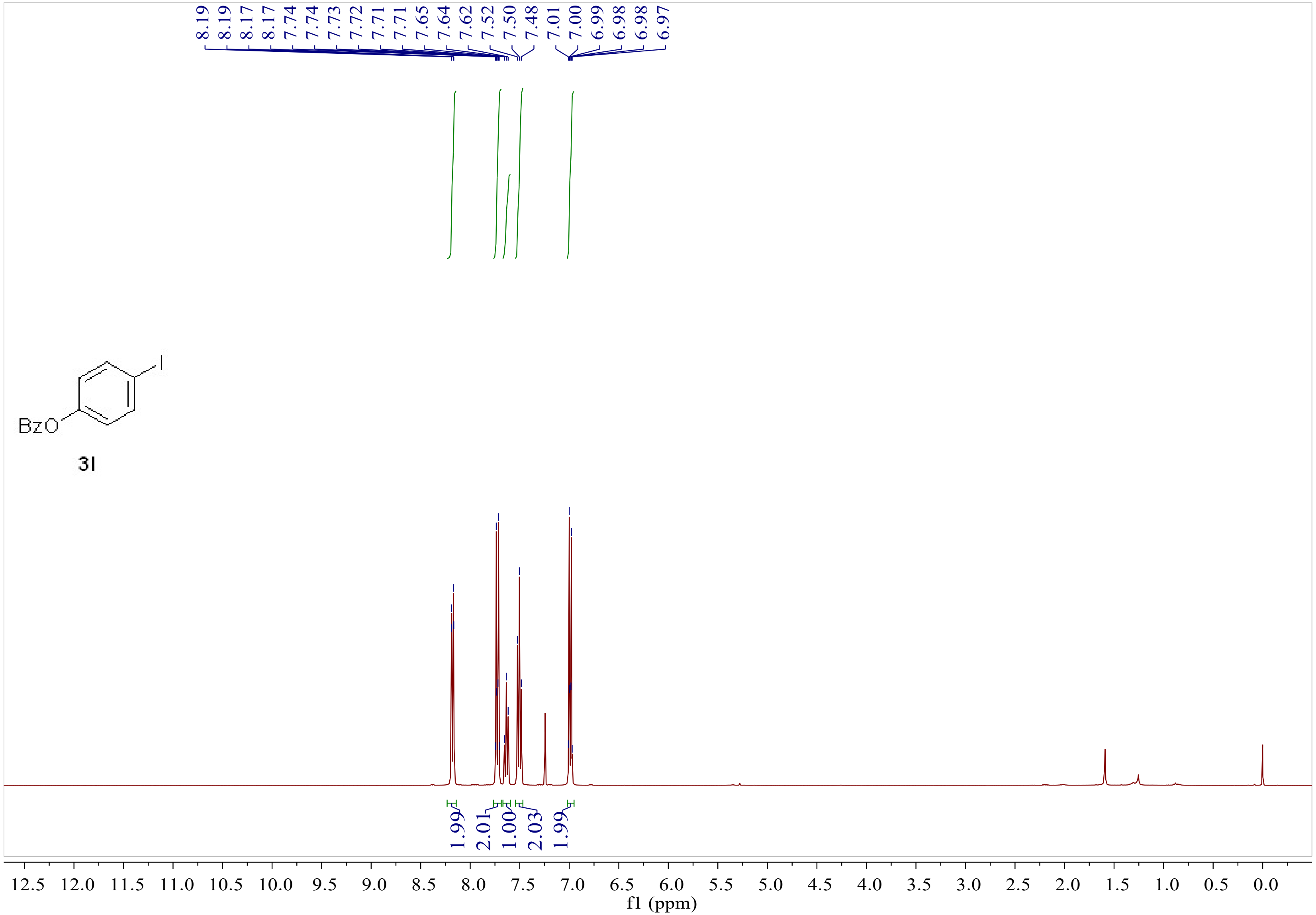
0
f1 (ppm)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6.98
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I
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1.00
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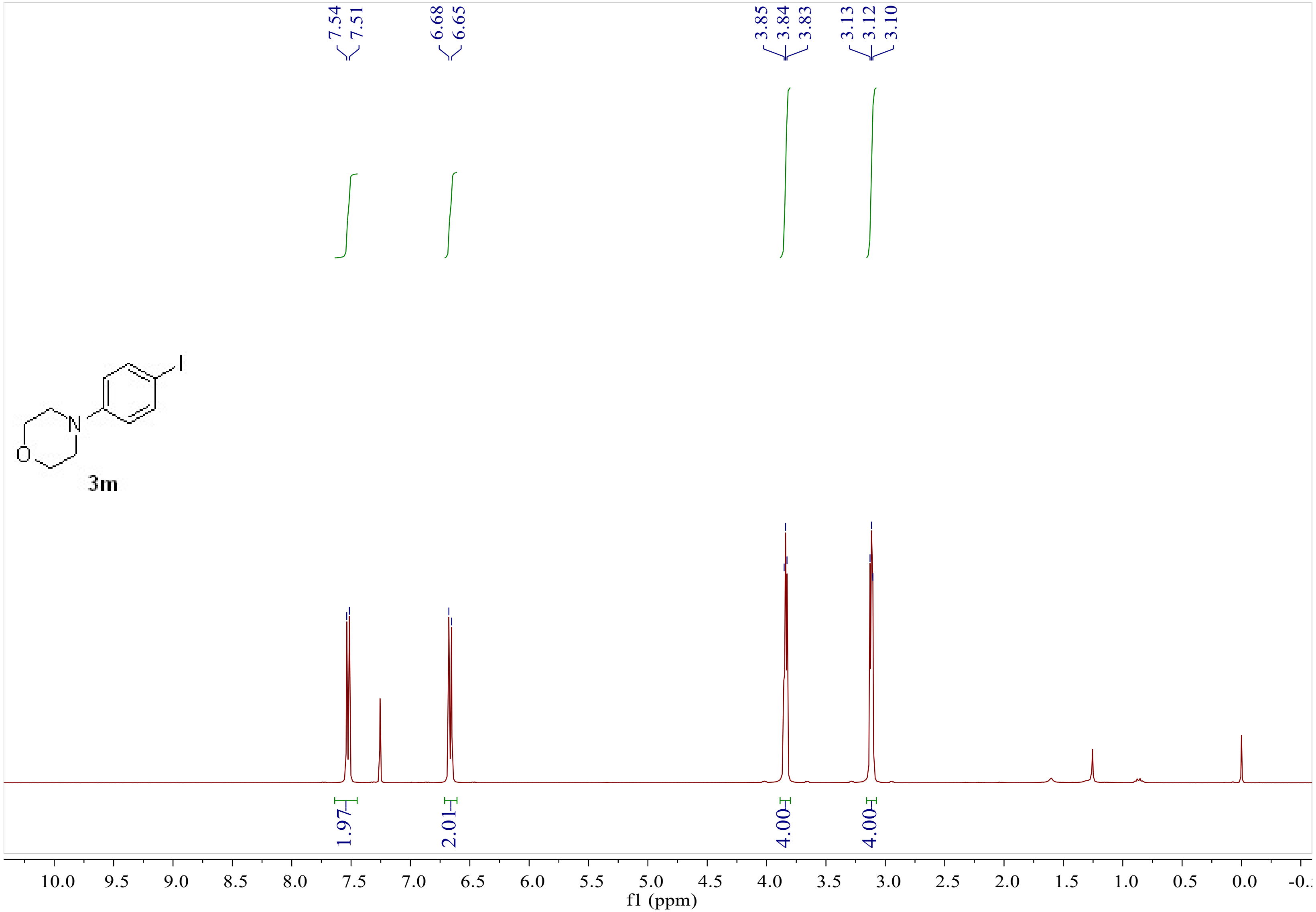
1.5
1.0
0.5
0.0
f1 (ppm)


7.54
7.51
6.68
6.65
3.85
3.84
3.83
3.13
3.12
3.10
O
N
I
3m
1.97
2.01
4.00
4.00
10.0
9.5
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
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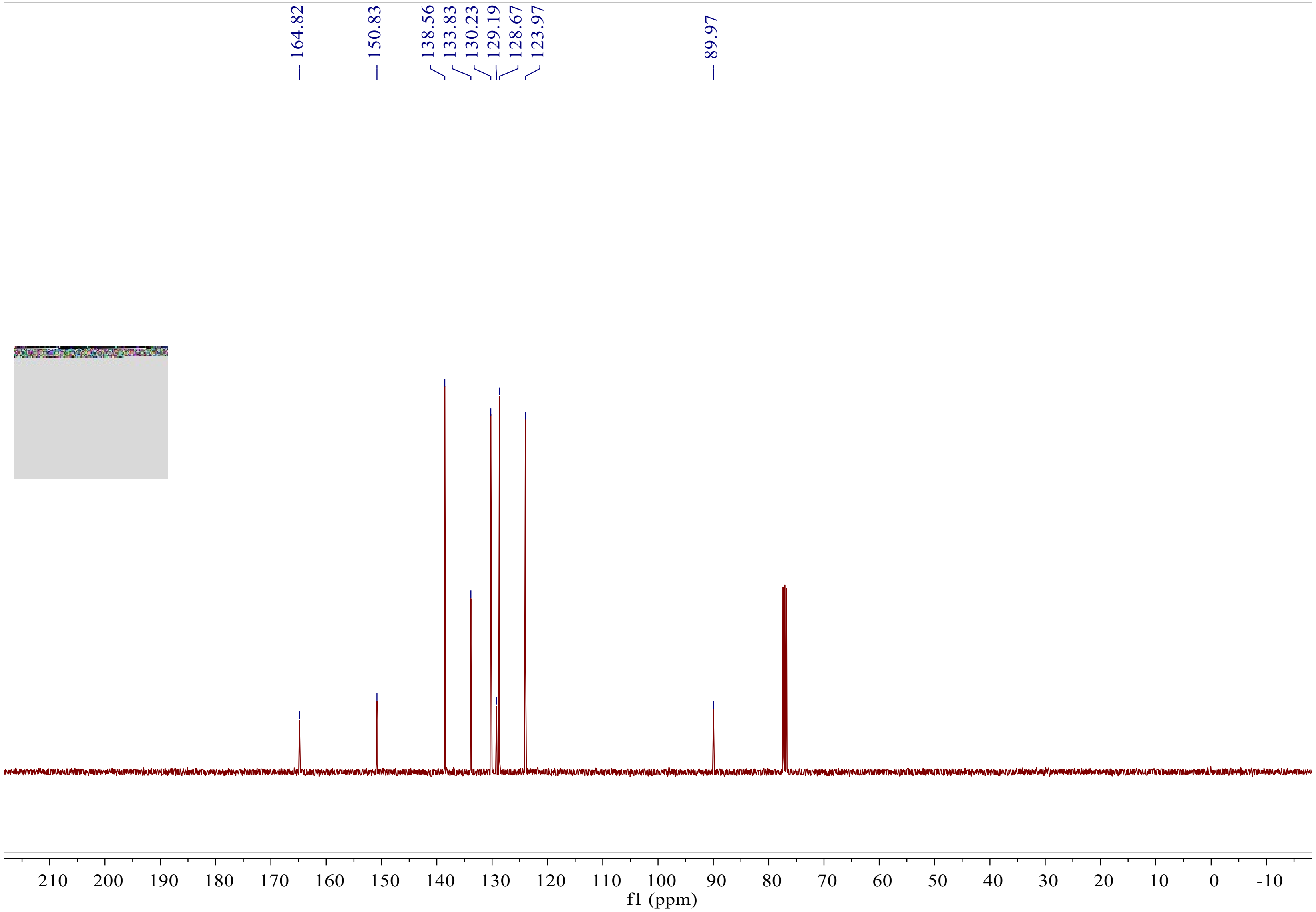
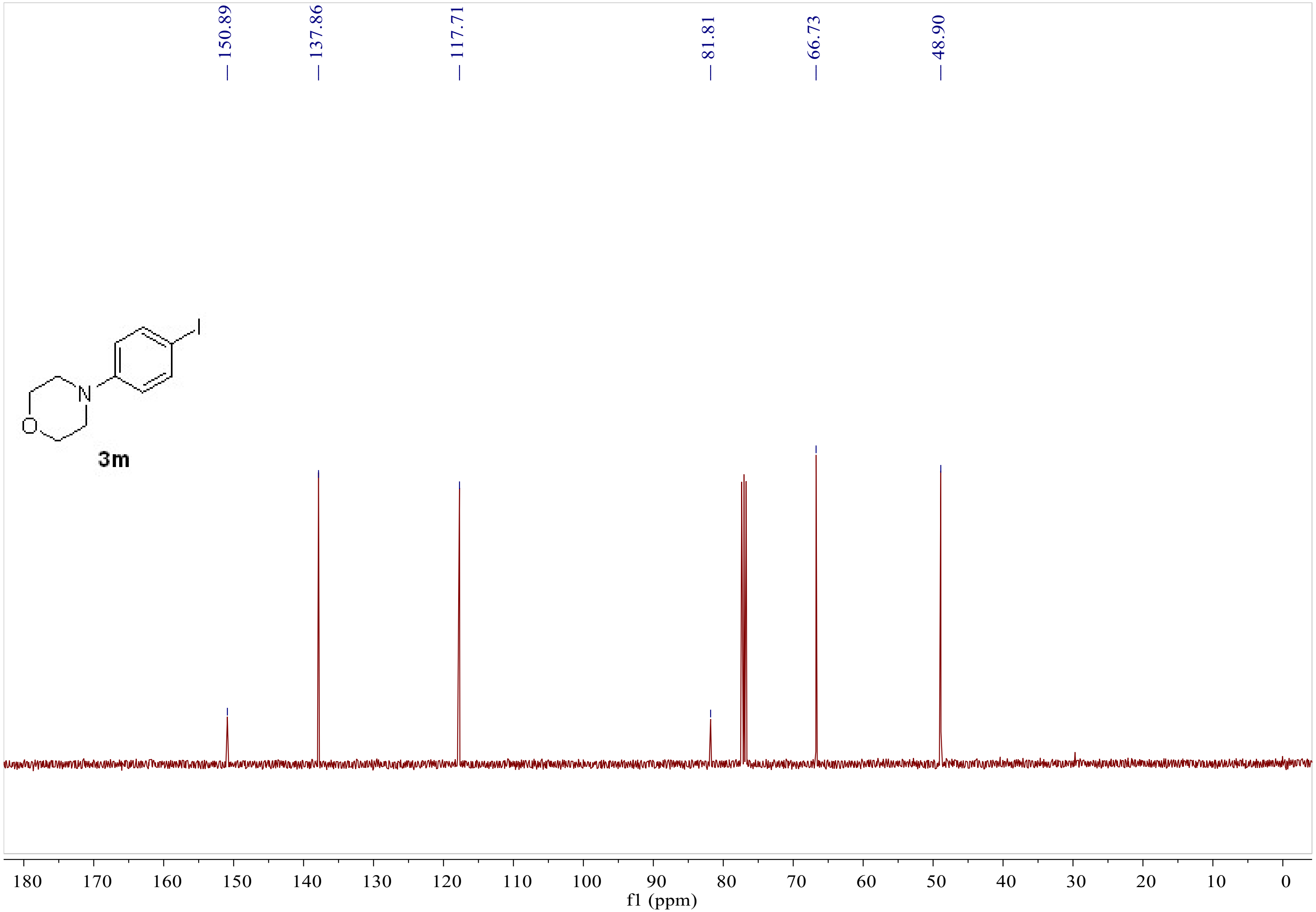
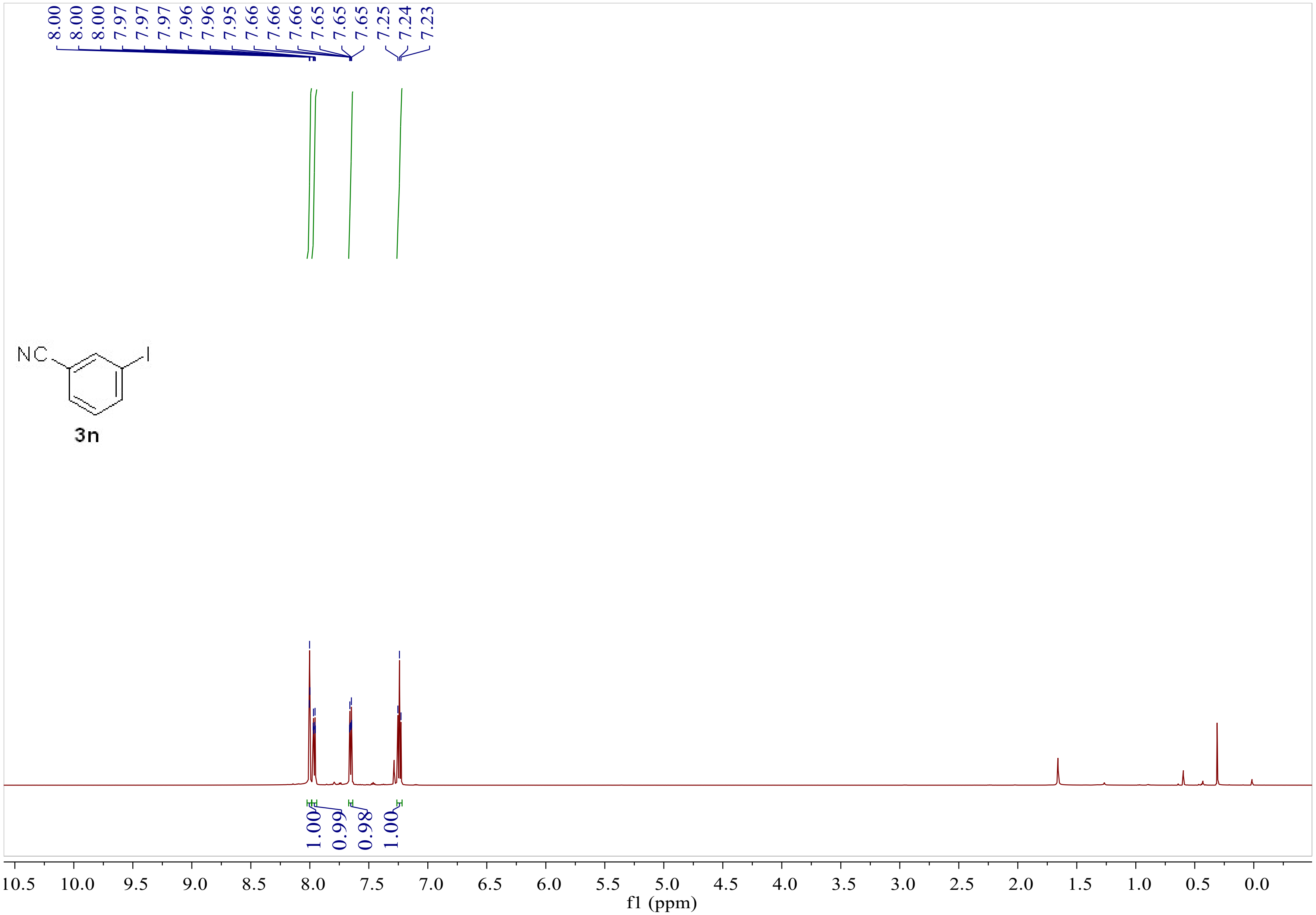
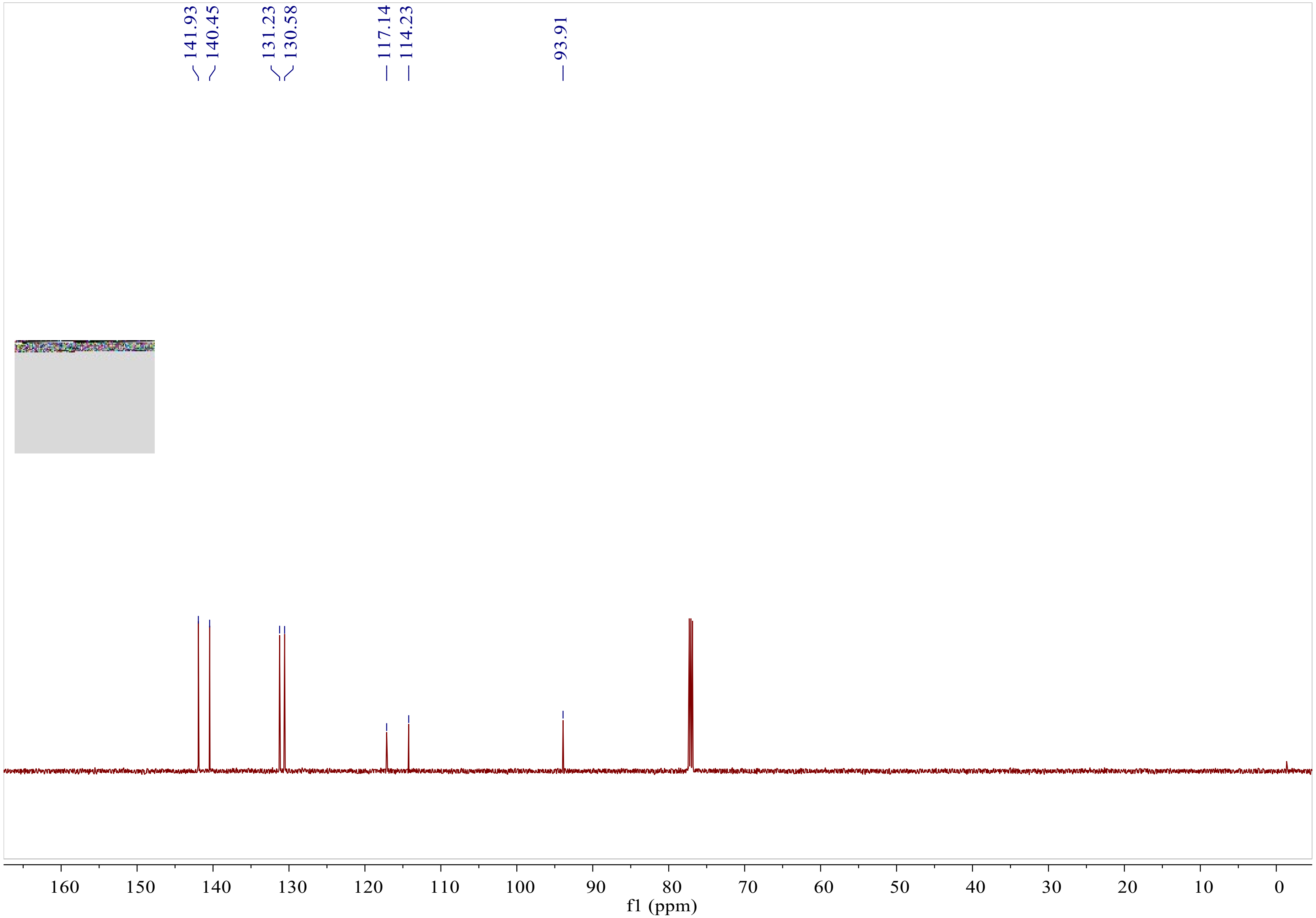
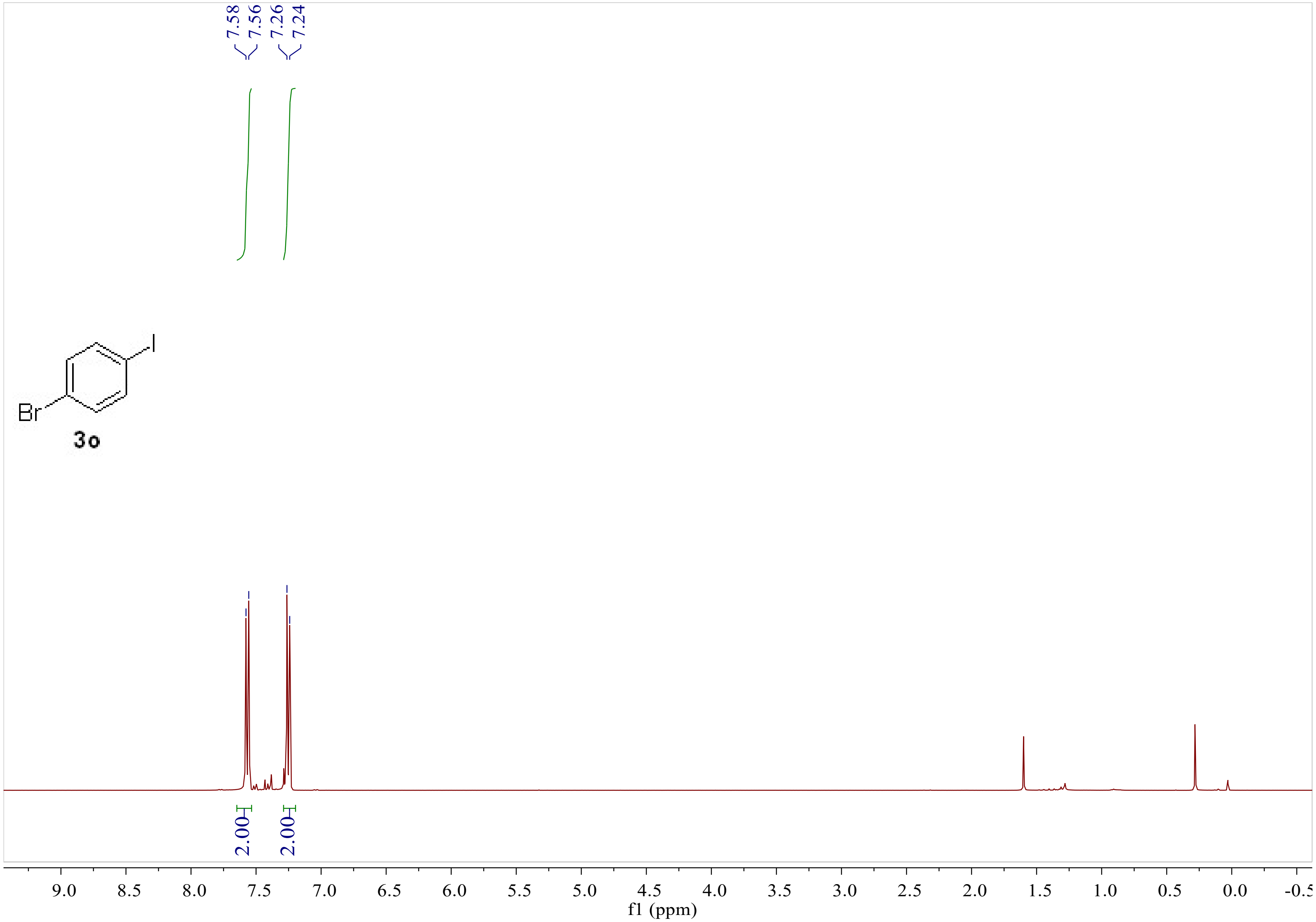
0.0
-0.
f1 (ppm)


164.82
150.83
138.56
133.83
130.23
129.19
128.67
123.97
89.97
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

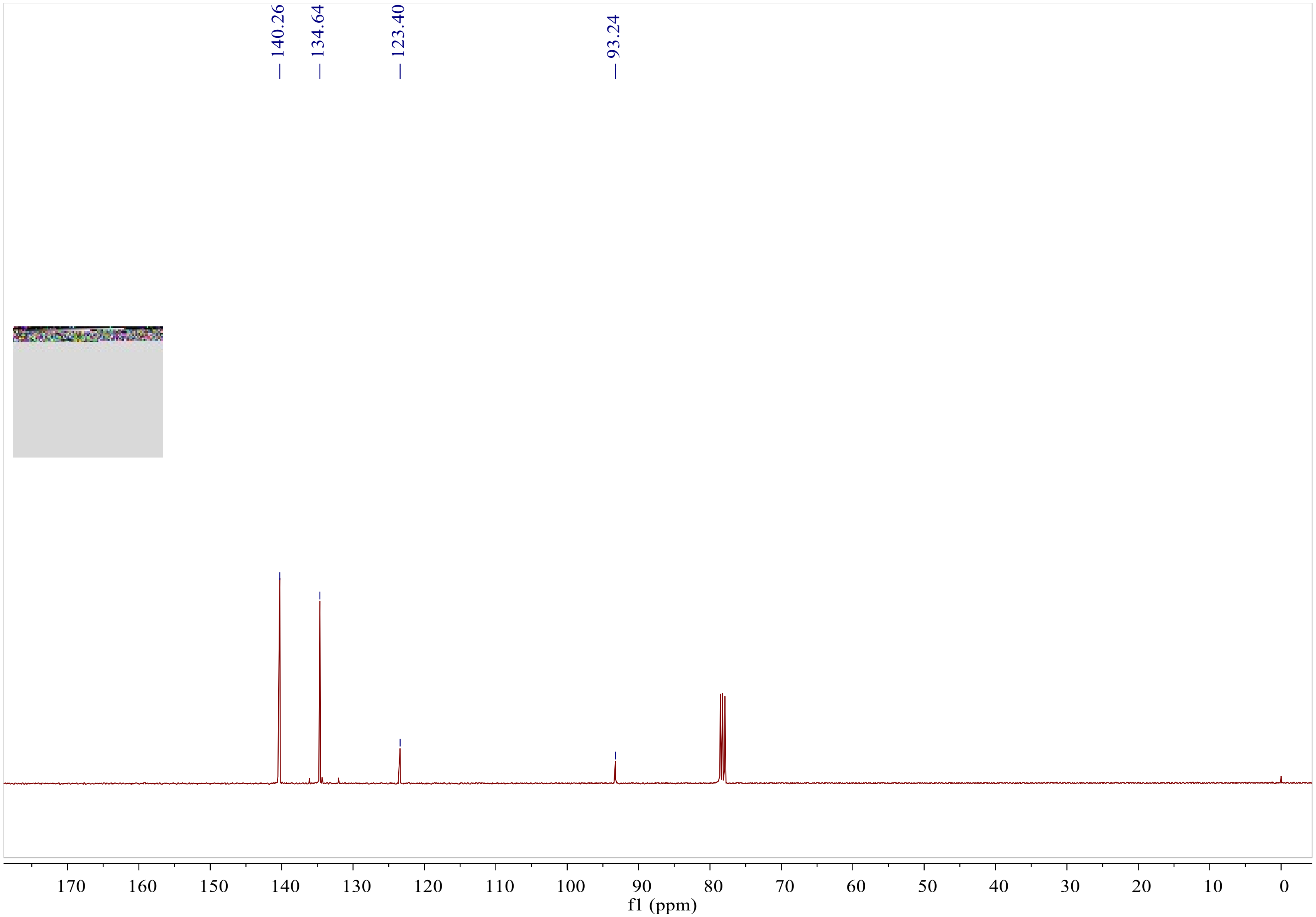

150.89
137.86
117.71
81.81
66.73
48.90
I
N
O
3m
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

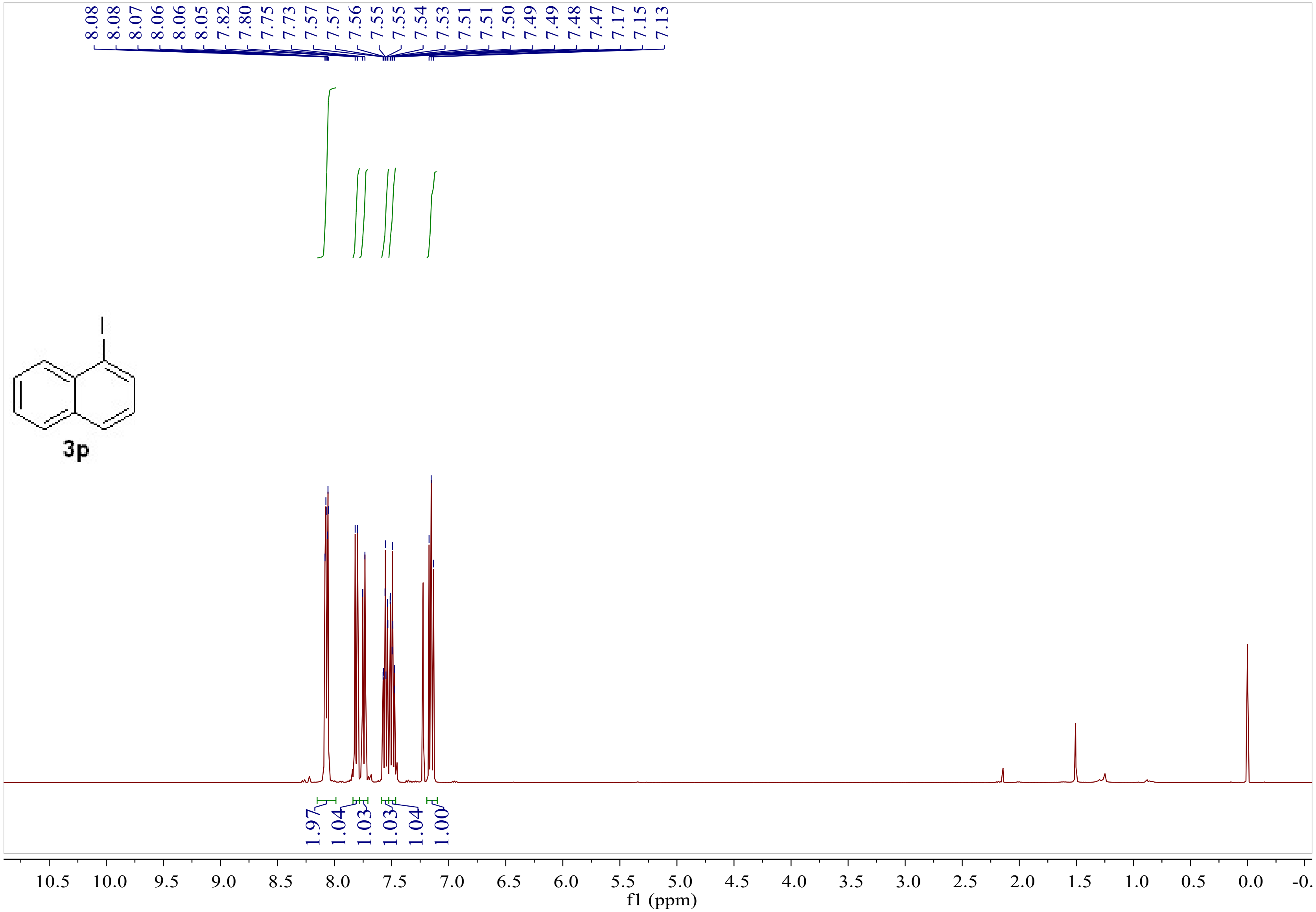

8.00
8.00
8.00
7.97
7.97
7.97
7.96
7.96
7.95
7.66
7.66
7.66
7.65
7.65
7.65
7.25
7.24
7.23
NC
I
3n
1.00
0.99
0.98
1.00
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

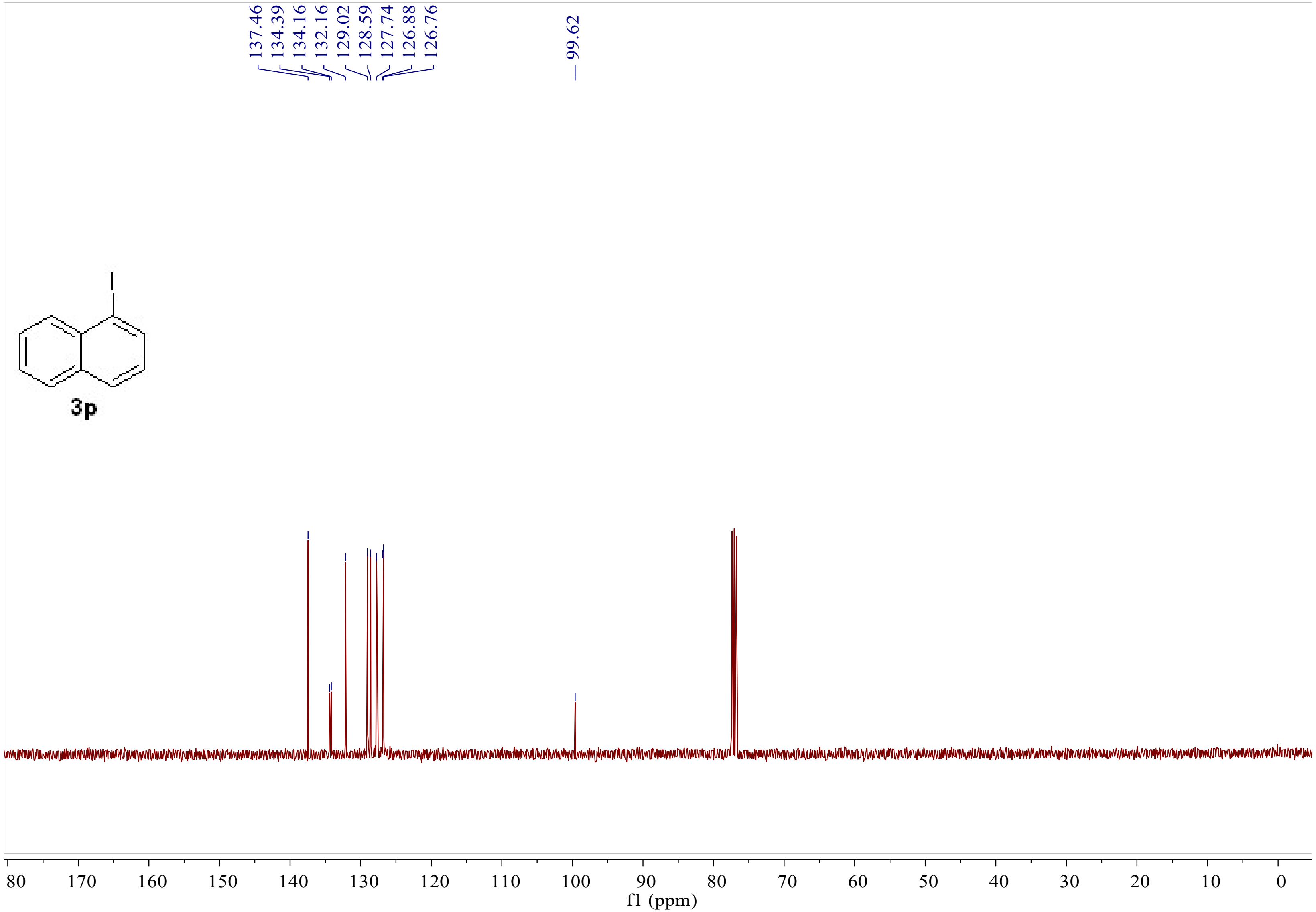

141.93
140.45
131.23
130.58
117.14
114.23
93.91
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.58
7.56
7.26
7.24
Br
I
2.00
2.00
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
0.5
0.0
-0.5
f1 (ppm)

3o

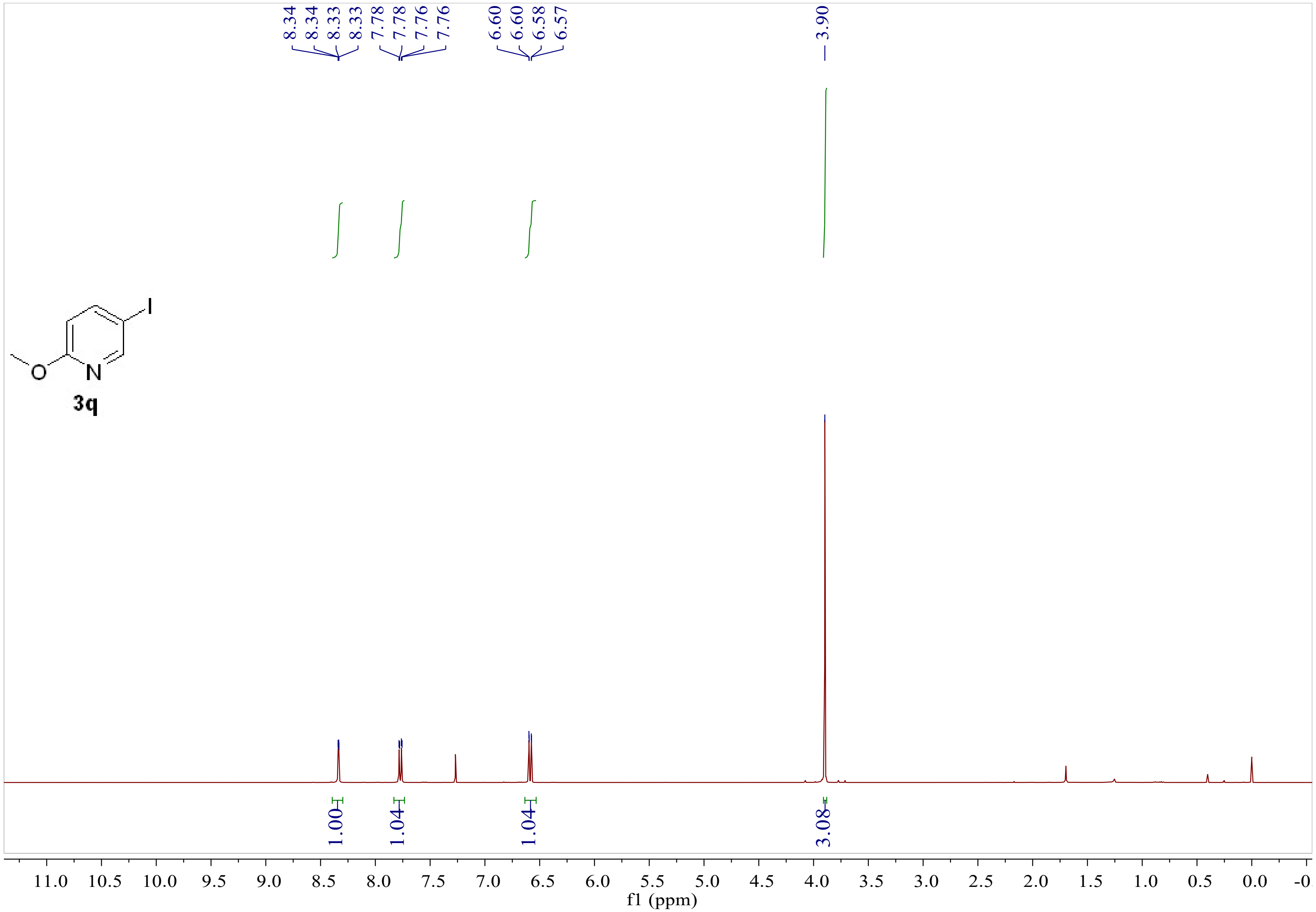

140.26
134.64
123.40
93.24
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.08
8.08
8.07
8.06
8.06
8.05
7.82
7.80
7.75
7.73
7.57
7.57
7.56
7.55
7.55
7.54
7.53
7.51
7.51
7.50
7.49
7.49
7.48
7.47
7.17
7.15
7.13
I
3p
1.97
1.04
1.03
1.03
1.04
1.00
10.5
10.0
9.5
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
0.5
0.0
-0.
f1 (ppm)

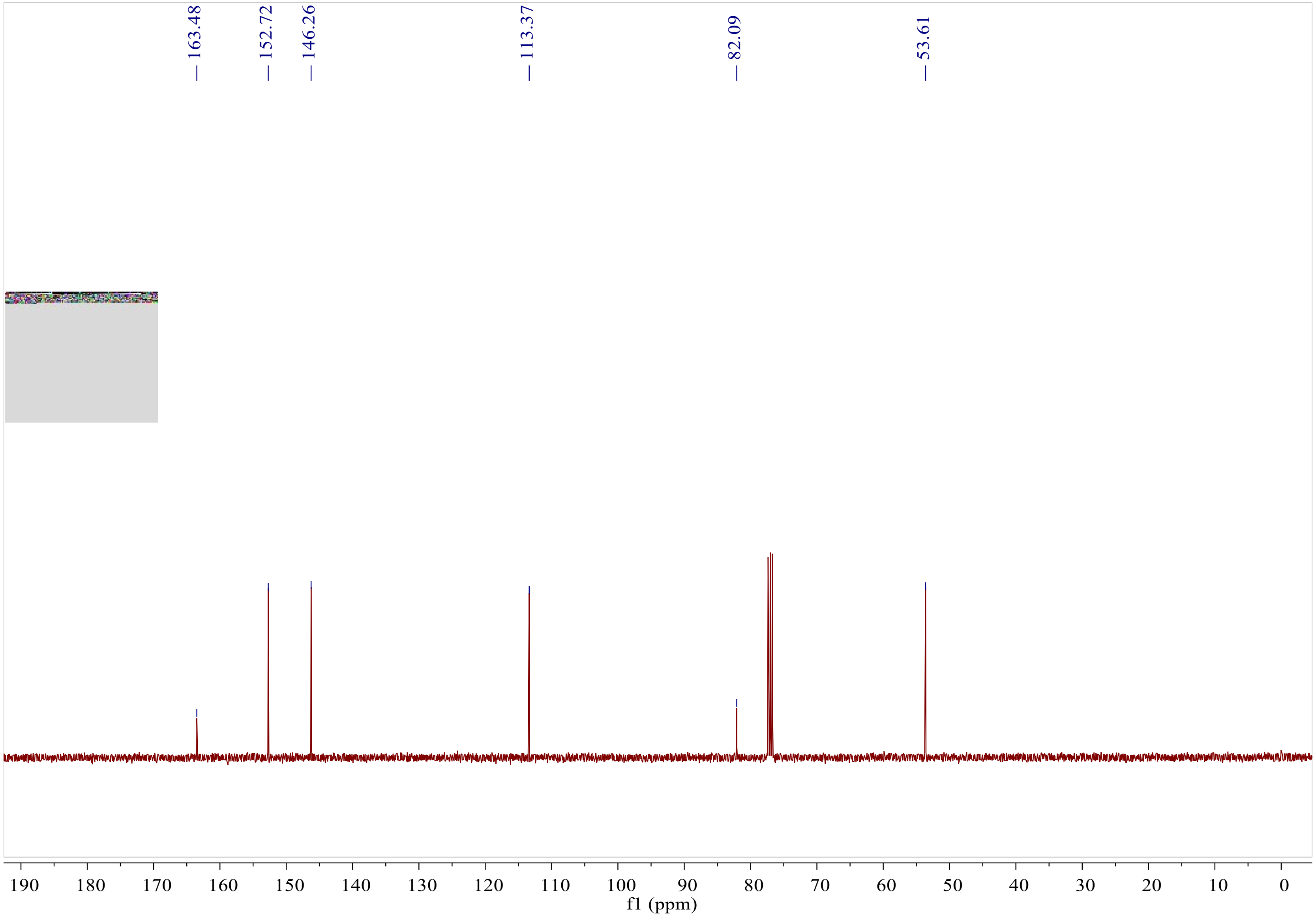


3p

137.46
134.39
134.16
132.16
129.02
128.59
127.74
126.88
126.76
99.62
80
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.34
8.34
8.33
8.33
7.78
7.78
7.76
7.76
6.60
6.60
6.58
6.57
3.90
I
O
N
1.00
1.04
1.04
3.08
11.0
10.5
10.0
9.5
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
0.5
0.0
-0
f1 (ppm)

3q


163.48
152.72
146.26
113.37
82.09
53.61
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

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