Electrochemical Oxidative Cyclization of *N*-Allylcarboxamides: Efficient Synthesis of Halogenated Oxazolines

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1. General comments

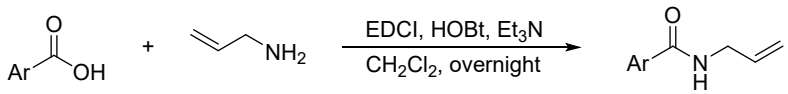
Chemicals were purchased from Adamas, Bidepharm., TCI, or Aladdin and used as such unless stated otherwise. Acetonitrile was purchased from Aladdin (AR, >99% (GC)). The instrument for electrolysis is a domestic dual display DC stabilized power supply (HY3005B). Cyclic voltammograms were obtained on a CHI660E potentiostat. NMR spectra were recorded on Bruker AV 400 spectrometer. Chemical shifts (ppm) are given relative to TMS (0.00 ppm) for ^{1}H and $CDCl_3$ (77.0 ppm) for ^{13}C solvent. Multiplets were assigned as s (singlet), d (doublet), t (triplet), q (quartet), p (pentet), dd (doublet of doublet), m (multiplet) and br.s (broad singlet). All measurements were carried out at room temperature unless otherwise stated. High-resolution mass spectra HRMS spectra were recorded on a Thermo Scientific Exactive Orbitrap Mass Spectrometer under Electron Spray Ionization conditions preparing sample solution in methanol. The data are given as mass units per charge (m/z). GC yields were calculated using hexadecane as an internal standard. Gas chromatography analysis was performed on an Agilent 6820 instrument with an FID detector and HP-5 capillary column (polydimethylsiloxane with 5% phenyl groups, 30 m, 0.32 mm i.d. 0.25 μm film thickness) using nitrogen as carrier gas. The products were isolated from the reaction mixture by column chromatography on silica gel., 54-74 μm, 200-300 mesh (Yucheng Chemical CO., LTD, Shanghai).

2. General procedures

2.1 General procedures for starting materials

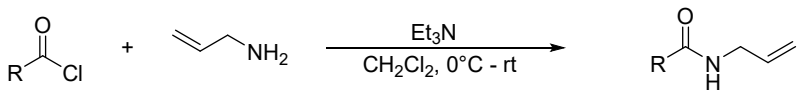
2.11 *N*-alkenyl amides were synthesized according to the literature.[1]

Starting from carboxylic acid for the synthesis of **1a-1v :**



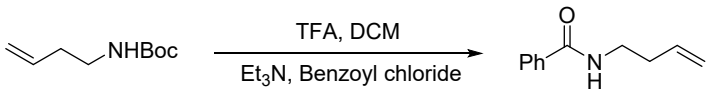
To a solution of the carboxylic acid derivative (1 equiv.) 1-ethyl-3-(3-dimethyl-aminopropyl)-carbodiimide (1 equiv.), NEt_3 (1 equiv.) and hydroxybenzotriazole (1 equiv.) in dried dichloromethane were added allylamine (1 equiv.). The reaction mixture was stirred overnight at ambient temperature. The reaction solution was washed with water (50 mL) and brine (50 mL). The organic layer was separated and dried over Na_2SO_4. For further workup, the crude product was purified by column chromatography after the removal of the solvent.

2.12 Starting from acyl chloride for the synthesis of **1p, 1q, 1s:**



To a solution of the NEt_3 (1 equiv.) and allylamine (1 equiv.) in dried dichloromethane was added the carboxylic acid chloride derivative (1 equiv.) at 0 ℃, then the reaction mixture was stirred overnight at ambient temperature. The reaction solution was washed with water (50 mL) and brine (50 mL). The organic layer was separated and dried over Na_2SO_4. For further workup, the crude product was purified by column chromatography after the removal of the solvent.

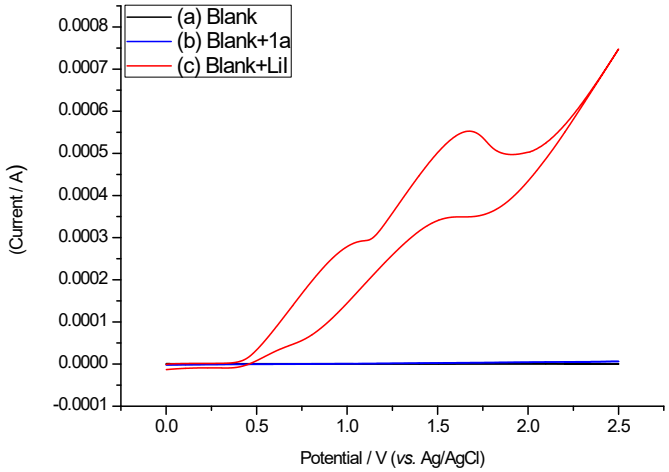
2.13 Preparation of *N*-(but-3-en-1-yl)benzamide **1r**[2]**:**



To a 250 mL round bottle equipped with a Teflon-coated magnetic stir bar were added tert-butyl but-3-en-1-ylcarbamate (342 mg, 2 mmol, 1.0 equiv.) and DCM (10 mL). TFA (1.82 g, 16 mmol, 8.0 equiv.) was then added dropwise. The resulting mixture was stirred at room temperature for 8 h until the starting material was consumed. To the resulting mixture were added NEt_3 (2.02 g, 20 mmol, 10.0 equiv.), DMAP (24.4 mg, 0.2 mmol, 0.1 equiv.) and benzoyl chloride (337 mg, 2.4mmol, 1.2 equiv.). The resulting mixture was stirred at room temperature overnight. After the reaction was completed, the reaction mixture was washed with brine, dried over Na_2SO_4. For further workup, the crude product was purified by column chromatography after the removal of the solvent.

2.2 Cyclic voltammetry measurements.

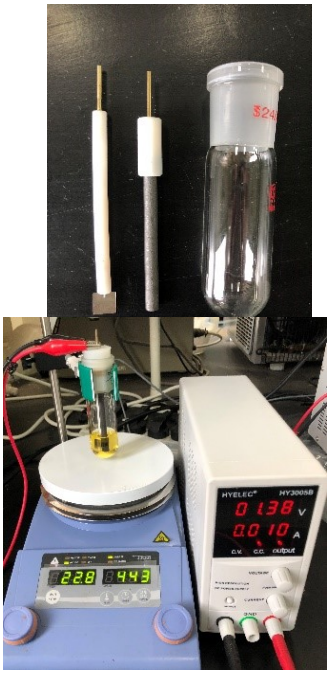
Cyclic voltammetry was obtained using a glassy carbon working electrode, a platinum counter electrode. The reference was an Ag/AgCl electrode submerged in saturated aqueous KCl solution (3.5 M) and separated from the reaction by a salt bridge. The scan rate was 0.10 V/s, ranging from 0.0 V to 2.5 V. (a) (black line) blank; 1.6 mL H_2O and 16 mL CH_3CN was poured into the electrochemical cell in cyclic voltammetry experiments. (b) (blue line)0.2 mmol **1a** *N*-allylbenzamide in 1.6 mL H_2O and 16 mL CH_3CN were poured into the electrochemical cell in cyclic voltammetry experiments. (c) (red line) 0.02 M LiI solution in 1.6 mL H_2O and 16 mL CH_3CN were poured into the electrochemical cell in cyclic voltammetry experiments.



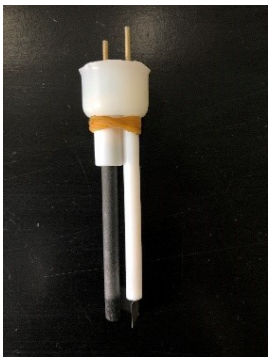
2.3 General procedures for the electrochemical oxidative cyclization of *N*-Allylcarboxamides .

To an oven-dried undivided tube (10 mL) equipped with a stirring bar was added **1a** (0.4 mmol, 95.6 mg), LiI (0.8 mmol, 107.2 mg), and CH_3CN/H_2O (6/0.6 mL). The reaction tube was equipped with platinum electrodes (1.0 cm×1.0 cm×0.1 mm) as the cathode and graphite rod (0.6 × 6 cm) as the anode. Then the reaction mixture was stirred and electrolyzed at a constant current of 20 mA under room temperature. After TLC indicated complete conversion of the starting material, the reaction mixture was diluted with sat. $Na_2S_2O_3$ and extracted with EtOAc (3 x 3 mL). The combined organic phase was washed with brine twice and concentrated under reduced pressure.

The crude products were purified by a short flash column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 10:1) to give the pure product **2a** as colorless oil in 90% yield (103.3 mg).



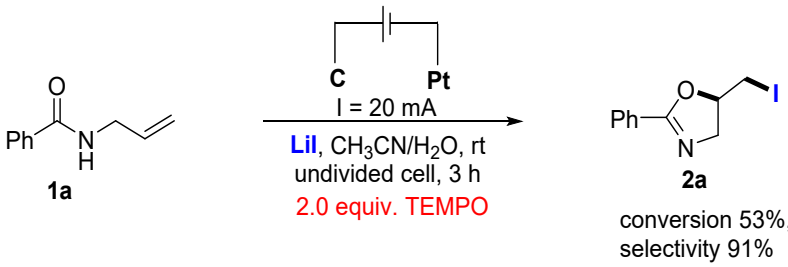
1. Reaction apparatuses



2. Power supply

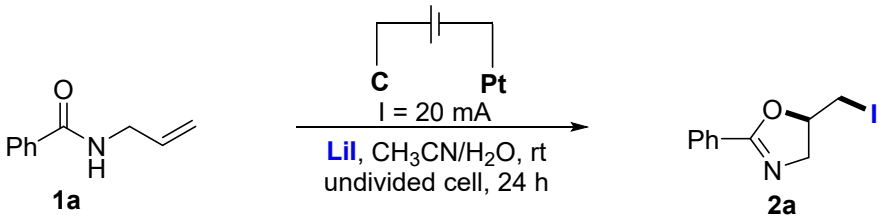
3. Ongoing reaction

2.4 Control experiment.



To an oven-dried undivided tube (10 mL) equipped with a stirring bar was added **1a** (0.4 mmol), LiI (0.8 mmol), TEMPO (0.8 mmol, 124.8 mg), and CH_3CN/H_2O (8/0.8 mL). The reaction tube was equipped with platinum electrodes (1.0 cm×1.0 cm×0.1 mm) as the cathode and graphite rod (0.6 × 6 cm) as the anode. Then the reaction mixture was stirred and electrolyzed at a constant current of 20 mA under room temperature for 3 hours. Only part of the starting material **1a** was converted to the desired product **2a** (conversion 53%, selectivity 91% detected by GC-FID).

2.5 Large-Scale Synthesis.



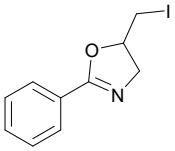
5 mmol, 0.805 g

78%,1.119g

To an oven-dried three-necked round-bottomed flask (100 mL) equipped with a stirring bar was added **1a** (5 mmol, 0.805 g), LiI (10 mmol, 1.34 g), and CH_3CN/H_2O (60/6 mL). The reaction tube was equipped with platinum electrodes (1.0 cm×1.0 cm×0.1 mm) as the cathode and graphite rod (0.6 × 6 cm) as the anode. Then the reaction mixture was stirred and electrolyzed at a constant current of 20 mA under room temperature for 24 hours. After TLC indicated complete conversion of the starting material, the reaction mixture was diluted with sat. $Na_2S_2O_3$ and extracted with EtOAc (3 x 3 mL). The combined organic phase was washed with brine twice and concentrated under reduced pressure. The crude products were purified by a short flash column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 10:1) to give the pure product **2a** as colorless oil in 90% yield (1.119 g).

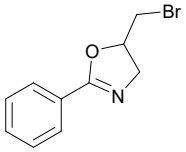
3. Spectroscopic Data of Products

5-(Iodomethyl)-2-phenyl-4,5-dihydrooxazole[3]



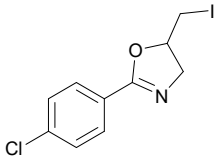
103.3 mg, colorless oil, yield: 90%
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (m, 2H), 7.53 – 7.44 (m, 1H), 7.42 (m, 2H), 4.87 – 4.74 (m, 1H), 4.18 (dd, *J* = 15.1, 9.5 Hz, 1H), 3.81 (dd, *J* = 15.1, 6.6 Hz, 1H), 3.38 (dd, *J* = 10.3, 4.9 Hz, 1H), 3.32 (dd, *J* = 10.3, 7.1 Hz, 1H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 163.58, 131.55, 128.36, 128.19, 127.30, 78.32, 60.60, 7.57.
The analytical data are consistent with those previously reported literature.[3]

5-(Bromomethyl)-2-phenyl-4,5-dihydrooxazole[4]



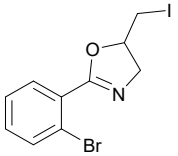
75.5 mg, colorless oil, yield: 79%
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 – 7.88 (m, 2H), 7.52 – 7.44 (m, 1H), 7.40 (m, 2H), 5.01 – 4.86 (m, 1H), 4.17 (dd, *J* = 15.2, 9.7 Hz, 1H), 3.90 (dd, *J* = 15.1, 6.7 Hz, 1H), 3.59 – 3.46 (m, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 163.58, 131.55, 128.36, 128.19, 127.30, 78.32, 60.60, 7.57.
The analytical data are consistent with those previously reported literature.[4]

2-(4-Chlorophenyl)-5-(iodomethyl)-4,5-dihydrooxazole[3]



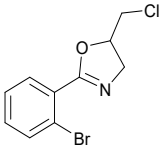
111.7 mg, colorless oil, yield: 87%
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 – 7.83 (m, 2H), 7.43 – 7.35 (m, 2H), 4.86 – 4.74 (m, 1H), 4.17 (dd, *J* = 15.3, 9.6 Hz, 1H), 3.79 (dd, *J* = 15.2, 6.7 Hz, 1H), 3.43 – 3.28 (m, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 162.60, 137.68, 129.49, 128.64, 125.81, 78.41, 60.70, 7.49.
The analytical data are consistent with those previously reported literature.[3]

2-(2-Bromophenyl)-5-(iodomethyl)-4,5-dihydrooxazole[3]



115.3 mg, colorless oil, yield: 79%
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (dd, *J* = 7.6, 1.9 Hz, 1H), 7.62 (dd, *J* = 7.9, 1.3 Hz, 1H), 7.33 (td, *J* = 7.5, 1.4 Hz, 1H), 7.27 (td, *J* = 7.7, 1.9 Hz, 1H), 4.86 – 4.73 (m, 1H), 4.19 (dd, *J* = 15.2, 9.6 Hz, 1H), 3.84 (dd, *J* = 15.2, 6.5 Hz, 1H), 3.43 – 3.27 (m, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 162.58, 133.84, 131.74, 131.33, 129.12, 127.05, 121.72, 78.39, 60.87, 7.47.
The analytical data are consistent with those previously reported literature.[3]

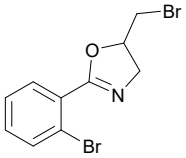
2-(2-Bromophenyl)-5-(chloromethyl)-4,5-dihydrooxazole[3]



42.6 mg, colorless oil, yield: 39%
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.74 – 7.61 (m, 2H), 7.39 – 7.25 (m, 2H), 5.00 – 4.89 (m, 1H), 4.22 (dd, *J* = 15.1, 9.7 Hz, 1H), 4.00 (dd, *J* = 15.2, 6.6 Hz, 1H), 3.71 (dd, *J* = 5.3, 1.2 Hz, 2H).

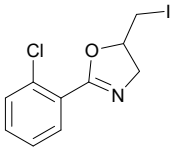
^{13}C NMR (101 MHz, $CDCl_3$) δ 162.91, 133.91, 131.81, 131.38, 129.12, 127.12, 121.79, 78.24, 58.53, 45.22.
The analytical data are consistent with those previously reported literature.[3]

5-(Bromomethyl)-2-(2-bromophenyl)-4,5-dihydrooxazole[3]



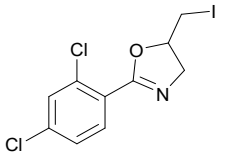
90.0 mg, colorless oil, yield: 71%
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.71 (dd, *J* = 7.6, 1.9 Hz, 1H), 7.65 (dd, *J* = 7.9, 1.3 Hz, 1H), 7.35 (td, *J* = 7.5, 1.4 Hz, 1H), 7.29 (td, *J* = 7.7, 1.9 Hz, 1H), 5.01 – 4.90 (m, 1H), 4.23 (dd, *J* = 15.2, 9.7 Hz, 1H), 3.97 (dd, *J* = 15.2, 6.5 Hz, 1H), 3.64 – 3.52 (m, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 162.62, 133.81, 131.74, 131.28, 129.00, 127.04, 121.68, 77.85, 59.44, 33.44.
The analytical data are consistent with those previously reported literature.[3]

2-(2-Chlorophenyl)-5-(iodomethyl)-4,5-dihydrooxazole[3]



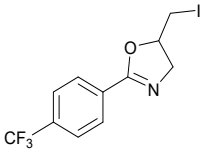
91.2 mg, colorless oil, yield: 71%
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (dd, *J* = 7.7, 1.8 Hz, 1H), 7.44 (dd, *J* = 8.0, 1.4 Hz, 1H), 7.36 (td, *J* = 7.7, 1.8 Hz, 1H), 7.29 (td, *J* = 7.5, 1.4 Hz, 1H), 4.87 – 4.73 (m, 1H), 4.21 (dd, *J* = 15.3, 9.6 Hz, 1H), 3.85 (dd, *J* = 15.3, 6.6 Hz, 1H), 3.45 – 3.27 (m, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 161.90, 133.39, 131.69, 131.28, 130.70, 126.91, 126.51, 78.15, 60.97, 7.47.
The analytical data are consistent with those previously reported literature.[3]

2-(2,4-Dichlorophenyl)-5-(iodomethyl)-4,5-dihydrooxazole[5]



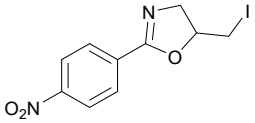
93.7 mg, colorless oil, yield: 66%
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 8.4 Hz, 1H), 7.46 (d, *J* = 2.1 Hz, 1H), 7.28 (dd, *J* = 8.4, 2.1 Hz, 1H), 4.84 – 4.72 (m, 1H), 4.20 (dd, *J* = 15.4, 9.6 Hz, 1H), 3.84 (dd, *J* = 15.4, 6.6 Hz, 1H), 3.42 – 3.28 (m, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 161.03, 137.21, 134.35, 132.18, 130.65, 126.93, 125.32, 78.13, 61.03, 7.38.
The analytical data are consistent with those previously reported literature.[5]

5-(Iodomethyl)-2-(4-(trifluoromethyl)phenyl)-4,5-dihydrooxazole



116.4 mg, white solid, yield: 82%
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 8.1 Hz, 2H), 7.68 (d, *J* = 8.1 Hz, 2H), 4.88 – 4.78 (m, 1H), 4.21 (dd, *J* = 15.4, 9.6 Hz, 1H), 3.83 (dd, *J* = 15.4, 6.7 Hz, 1H), 3.44 – 3.30 (m, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 162.40, 133.16 (d, *J* = 33.3 Hz), 130.78, 128.61, 125.40, (q, *J* = 4.0 Hz), 123.76 (d, *J* = 77.8 Hz), 78.57, 60.91, 7.48.
HRMS (ESI) Calc. for $C_{11}H_9F_3INO$, 355.9754, found 355.9763

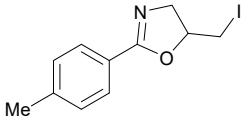
5-(Iodomethyl)-2-(4-nitrophenyl)-4,5-dihydrooxazole[5]



70.4 mg, white solid, yield: 53%

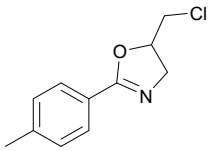
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.27 (d, *J* = 8.6 Hz, 2H), 8.11 (d, *J* = 8.6 Hz, 2H), 4.92 – 4.78 (m, 1H), 4.23 (dd, *J* = 15.6, 9.5 Hz, 1H), 3.85 (dd, *J* = 15.6, 6.7 Hz, 1H), 3.44 – 3.31 (m, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 161.80, 149.60, 133.10, 129.27, 123.58, 78.84, 61.05, 7.32.
The analytical data are consistent with those previously reported literature.[5]

5-(Iodomethyl)-2-(p-tolyl)-4,5-dihydrooxazole[3]



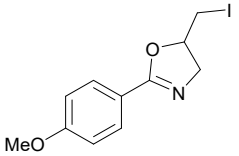
98.7 mg, white solid, yield: 82%
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (d, *J* = 8.0 Hz, 2H), 7.21 (d, *J* = 7.9 Hz, 2H), 4.85 – 4.71 (m, 1H), 4.15 (dd, *J* = 15.1, 9.5 Hz, 1H), 3.78 (dd, *J* = 15.1, 6.6 Hz, 1H), 3.42 – 3.24 (m, 2H), 2.38 (s, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 163.61, 141.93, 129.06, 128.12, 124.54, 78.18, 60.58, 21.54, 7.66.
The analytical data are consistent with those previously reported literature.[3]

5-(Chloromethyl)-2-(p-tolyl)-4,5-dihydrooxazole[3]



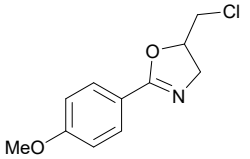
33.4 mg, colorless oil, yield: 40%
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (d, *J* = 8.2 Hz, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 5.01 – 4.85 (m, 1H), 4.19 (dd, *J* = 15.0, 9.7 Hz, 1H), 3.95 (dd, *J* = 15.0, 6.7 Hz, 1H), 3.70 (m, 2H), 2.41 (s, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 163.83, 141.93, 129.07, 128.13, 124.41, 78.02, 58.22, 45.42, 21.54.
The analytical data are consistent with those previously reported literature.[3]

5-(Iodomethyl)-2-(4-methoxyphenyl)-4,5-dihydrooxazole[3]



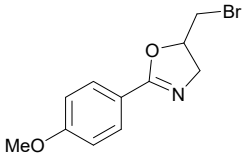
91.3 mg, colorless oil, yield: 72%
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 – 7.83 (m, 2H), 6.98 – 6.86 (m, 2H), 4.84 – 4.74 (m, 1H), 4.15 (dd, *J* = 14.9, 9.5 Hz, 1H), 3.84 (s, 3H), 3.77 (dd, *J* = 14.9, 6.6 Hz, 1H), 3.42 – 3.26 (m, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 163.32, 162.18, 129.91, 119.74, 113.69, 78.19, 60.49, 55.31, 7.70.
The analytical data are consistent with those previously reported literature.[3]

5-(Chloromethyl)-2-(4-methoxyphenyl)-4,5-dihydrooxazole[3]



31.5 mg, colorless oil, yield: 35%
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.87 (d, *J* = 8.8 Hz, 2H), 6.90 (d, *J* = 8.8 Hz, 2H), 4.94 – 4.82 (m, 1H), 4.14 (dd, *J* = 14.9, 9.6 Hz, 1H), 3.91 (d, *J* = 6.7 Hz, 1H), 3.82 (s, 3H), 3.72 – 3.61 (m, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 163.53, 162.16, 129.89, 119.62, 113.68, 78.01, 58.10, 55.29, 45.41.
The analytical data are consistent with those previously reported literature.[3]

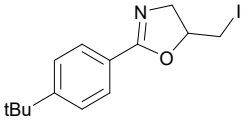
5-(Bromomethyl)-2-(4-methoxyphenyl)-4,5-dihydrooxazole[3]



69.9 mg, white solid, yield: 65%

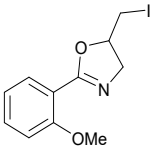
^{1}H NMR (400 MHz, Chloroform-d) δ 7.87 (d, J = 8.8 Hz, 2H), 6.91 (d, J = 8.8 Hz, 2H), 4.96 – 4.84 (m, 1H), 4.15 (dd, J = 14.9, 9.6 Hz, 1H), 3.95 – 3.78 (m, 4H), 3.60 – 3.41 (m, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 163.36, 162.16, 129.89, 119.76, 113.70, 77.76, 59.24, 55.32, 33.66.
The analytical data are consistent with those previously reported literature.[3]

2-(4-(tert-Butyl)phenyl)-5-(iodomethyl)-4,5-dihydrooxazole[3]



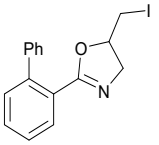
105.6 mg, colorless oil, yield: 77%
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.87 (d, *J* = 8.5 Hz, 2H), 7.44 (d, *J* = 8.5 Hz, 2H), 4.86 – 4.76 (m, 1H), 4.17 (dd, *J* = 15.0, 9.5 Hz, 1H), 3.80 (dd, *J* = 15.0, 6.6 Hz, 1H), 3.42 – 3.26 (m, 2H), 1.33 (s, 9H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 163.76, 155.22, 128.12, 125.44, 124.42, 78.29, 60.51, 35.02, 31.18, 7.73.
The analytical data are consistent with those previously reported literature.[3]

5-(Iodomethyl)-2-(2-methoxyphenyl)-4,5-dihydrooxazole



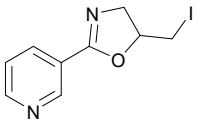
98.9 mg, colorless oil, yield: 78%
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (dd, *J* = 8.0, 1.8 Hz, 1H), 7.48 – 7.35 (m, 1H), 7.02 – 6.91 (m, 2H), 4.79 – 4.66 (m, 1H), 4.20 (dd, *J* = 15.2, 9.5 Hz, 1H), 3.91 (s, 3H), 3.84 (dd, *J* = 15.2, 6.4 Hz, 1H), 3.36 (dd, *J* = 10.2, 5.0 Hz, 1H), 3.29 (dd, *J* = 10.1, 7.2 Hz, 1H).
^{13}C NMR (101 MHz, cdcl$_3$) δ 161.82, 158.55, 132.53, 131.16, 120.16, 116.31, 111.56, 77.14, 61.15, 55.94, 7.69.
HRMS (ESI) Calc. for $C_{11}H_{12}INO_2$, 317.9985, found 317.9994.

2-([1,1'-Biphenyl]-2-yl)-5-(iodomethyl)-4,5-dihydrooxazole



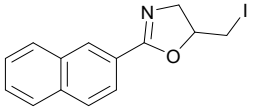
122.0 mg, colorless oil, yield: 84%
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (dd, *J* = 7.6, 1.4 Hz, 1H), 7.53 – 7.46 (m, 1H), 7.44 – 7.30 (m, 7H), 4.61 – 4.49 (m, 1H), 4.02 (dd, *J* = 15.0, 9.6 Hz, 1H), 3.68 (dd, *J* = 15.1, 6.6 Hz, 1H), 3.00 – 2.89 (m, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 164.78, 141.87, 141.03, 130.54, 130.30, 130.01, 128.47, 127.92, 127.23, 127.14, 127.06, 78.56, 60.58, 60.56, 6.92.
HRMS (ESI) Calc. for $C_{16}H_{14}INO$, 364.0193, found 364.0201

5-(Iodomethyl)-2-(pyridin-3-yl)-4,5-dihydrooxazole[3]



79.5 mg, colorless oil, yield: 69%
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.12 (dd, *J* = 2.2, 0.9 Hz, 1H), 8.70 (dd, *J* = 4.9, 1.7 Hz, 1H), 8.19 (dt, *J* = 8.0, 2.0 Hz, 1H), 7.35 (ddd, *J* = 7.9, 4.9, 0.9 Hz, 1H), 4.88 – 4.76 (m, 1H), 4.19 (dd, *J* = 15.4, 9.5 Hz, 1H), 3.81 (dd, *J* = 15.4, 6.7 Hz, 1H), 3.46 – 3.26 (m, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 161.61, 152.20, 149.44, 135.59, 123.66, 123.28, 78.46, 60.85, 7.46.
The analytical data are consistent with those previously reported literature.[3]

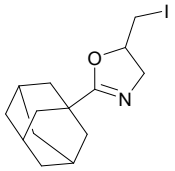
5-(Iodomethyl)-2-(naphthalen-2-yl)-4,5-dihydrooxazole [3]



101.1 mg, white solid, yield: 75%

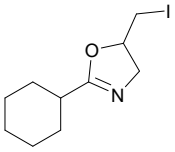
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.44 (d, *J* = 1.7 Hz, 1H), 8.02 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.95 – 7.88 (m, 1H), 7.86 (dd, *J* = 8.4, 5.8 Hz, 2H), 7.60 – 7.45 (m, 2H), 4.90 – 4.75 (m, 1H), 4.22 (dd, *J* = 15.2, 9.5 Hz, 1H), 3.85 (dd, *J* = 15.2, 6.7 Hz, 1H), 3.46 – 3.27 (m, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 163.70, 134.81, 132.66, 128.97, 128.84, 128.24, 127.82, 127.68, 126.65, 124.74, 124.70, 78.43, 60.90, 7.83.
The analytical data are consistent with those previously reported literature.[3]

2-(Adamantan-1-yl)-5-(iodomethyl)-4,5-dihydrooxazole[3]



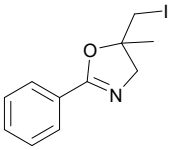
55.2 mg, colorless oil, yield: 40%
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.55 – 4.42 (m, 1H), 3.89 (dd, *J* = 14.5, 9.5 Hz, 1H), 3.53 (dd, *J* = 14.6, 6.3 Hz, 1H), 3.22 (d, *J* = 5.5 Hz, 2H), 2.03 – 1.96 (m, 3H), 1.88 (m, 6H), 1.76 – 1.64 (m, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 173.39, 77.08, 59.82, 39.46, 36.54, 35.39, 27.88, 8.60.
The analytical data are consistent with those previously reported literature.[3]

2-Cyclohexyl-5-(iodomethyl)-4,5-dihydrooxazole[3]



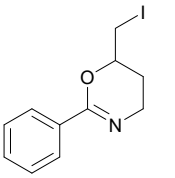
77.4 mg, colorless oil, yield: 66%
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.53 – 4.40 (m, 1H), 3.83 (dd, *J* = 14.5, 9.6 Hz, 1H), 3.46 (dd, *J* = 14.5, 6.4 Hz, 1H), 3.16 (d, *J* = 5.6 Hz, 2H), 2.30 – 2.13 (m, 1H), 1.92 – 1.79 (m, 2H), 1.74 – 1.62 (m, 2H), 1.61 – 1.52 (m, 1H), 1.42 – 1.29 (m, 2H), 1.25 – 1.12 (m, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 170.64, 77.18, 59.97, 37.33, 29.74, 29.61, 25.74, 25.54, 25.50, 8.31.
The analytical data are consistent with those previously reported literature.[3]

5-(Iodomethyl)-5-methyl-2-phenyl-4,5-dihydrooxazole[3]



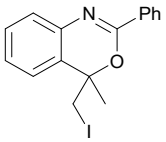
99.9 mg, colorless oil, yield: 83%
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 – 7.87 (m, 2H), 7.51 – 7.42 (m, 1H), 7.44 – 7.35 (m, 2H), 4.01 (d, *J* = 15.0 Hz, 1H), 3.83 (d, *J* = 15.1 Hz, 1H), 3.48 – 3.34 (m, 2H), 1.66 (s, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 162.82, 131.37, 128.26, 128.09, 127.61, 83.70, 65.41, 25.24, 13.96.
The analytical data are consistent with those previously reported literature.[3]

6-(Iodomethyl)-2-phenyl-5,6-dihydro-4H-1,3-oxazine[3]



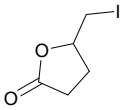
74.6 mg, colorless oil, yield: 62%
1 H NMR (400 MHz, $CDCl_3$) δ = 7.93−7.92 (m, 2H), 7.39−7.26 (m, 3H), 4.30−4.17 (m, 1H), 3.71−3.55 (m, 2H), 3.38−3.25 (m, 2H), 2.13−2.11 (m, 1H), 1.83−1.67 (m, 1H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 155.82, 133.01, 130.68, 128.07, 127.07, 73.79, 42.49, 27.18, 7.38.
The analytical data are consistent with those previously reported literature.[3]

4-(Iodomethyl)-4-methyl-2-phenyl-4H-benzo[d][1,3]oxazine[6]



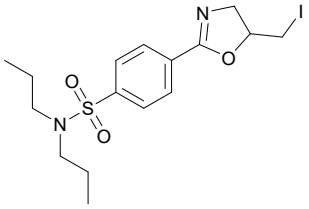
108.9 mg, white solid, yield: 75%
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.36 – 8.25 (m, 2H), 7.61 – 7.46 (m, 3H), 7.44 – 7.37 (m, 2H), 7.31 – 7.17 (m, 2H), 3.70 (d, *J* = 11.0 Hz, 1H), 3.51 (d, *J* = 11.0 Hz, 1H), 1.96 (s, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 156.02, 138.72, 132.14, 131.54, 129.43, 128.31, 128.20, 126.95, 126.76, 125.49, 122.99, 77.45, 26.55, 15.71.
The analytical data are consistent with those previously reported literature.[6]

5-(Iodomethyl)dihydrofuran-2(3H)-one[7]



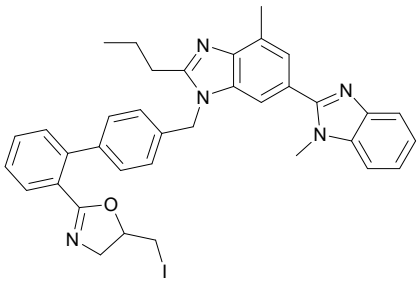
80.5 mg, colorless oil, yield: 89%
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.51 – 4.35 (m, 1H), 3.30 (dd, *J* = 10.5, 4.5 Hz, 1H), 3.23 (dd, *J* = 10.5, 6.7 Hz, 1H), 2.60 – 2.43 (m, 2H), 2.44 – 2.31 (m, 1H), 1.98 – 1.79 (m, 1H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 176.08, 78.23, 28.66, 27.87, 7.64.
The analytical data are consistent with those previously reported literature.[7]

4-(5-(Iodomethyl)-4,5-dihydrooxazol-2-yl)-N,N-dipropylbenzenesulfonamide



145.8 mg, white solid, yield: 81%
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, *J* = 8.5 Hz, 2H), 7.82 (d, *J* = 8.5 Hz, 2H), 4.87 – 4.75 (m, 1H), 4.19 (dd, *J* = 15.5, 9.6 Hz, 1H), 3.81 (dd, *J* = 15.5, 6.8 Hz, 1H), 3.42 – 3.27 (m, 2H), 3.19 – 2.97 (m, 4H), 1.52 (m, 4H), 0.84 (t, *J* = 7.4 Hz, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 162.21, 142.76, 130.82, 128.75, 126.94, 78.57, 60.80, 49.83, 21.84, 11.09, 7.45.
HRMS (ESI) Calc. for $C_{16}H_{23}IN_2O_3S$ $[M+H]^+$: 451.0547; found: 451.0539.

2-(4'-((1,7'-Dimethyl-2'-propyl-1H,3'H-[2,5'-bibenzo[d]imidazol]-3'-yl)methyl)-[1,1'-biphenyl]-2-yl)-5-(iodomethyl)-4,5-dihydrooxazole

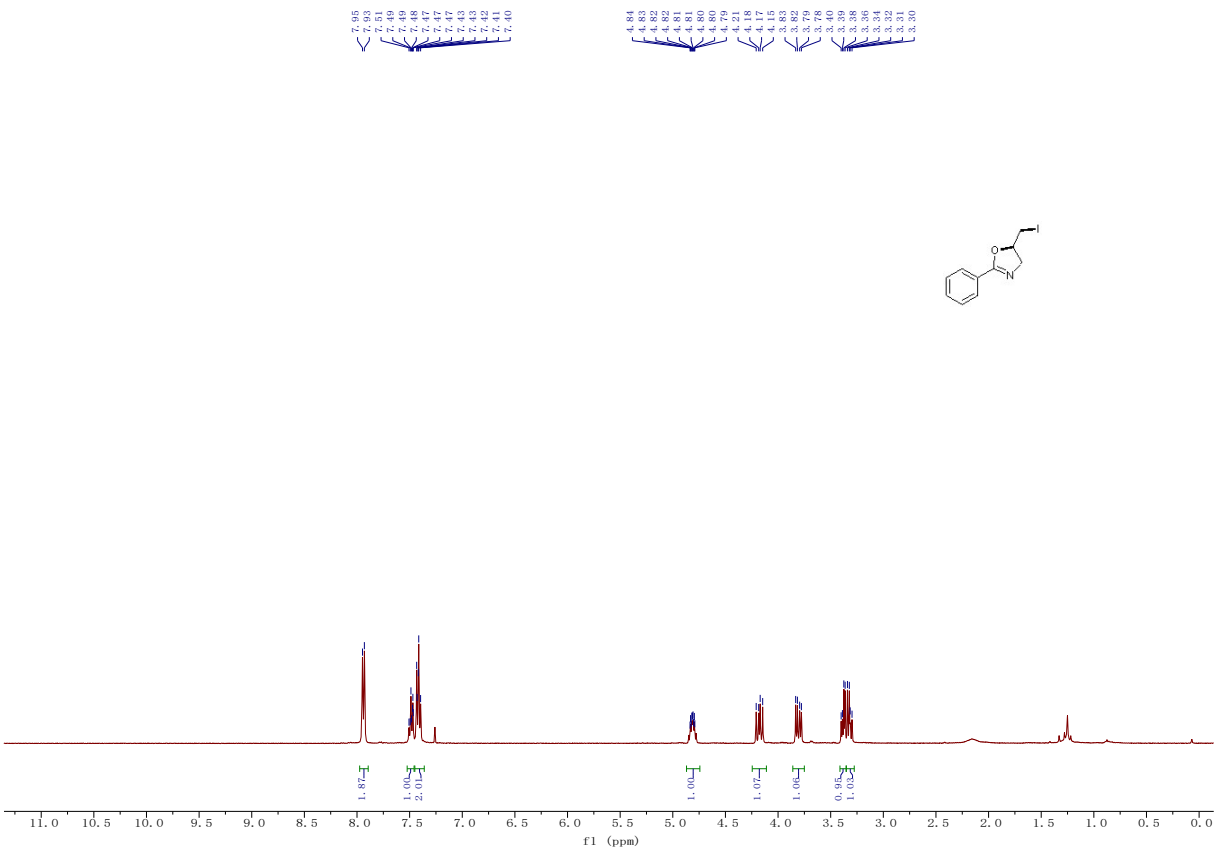


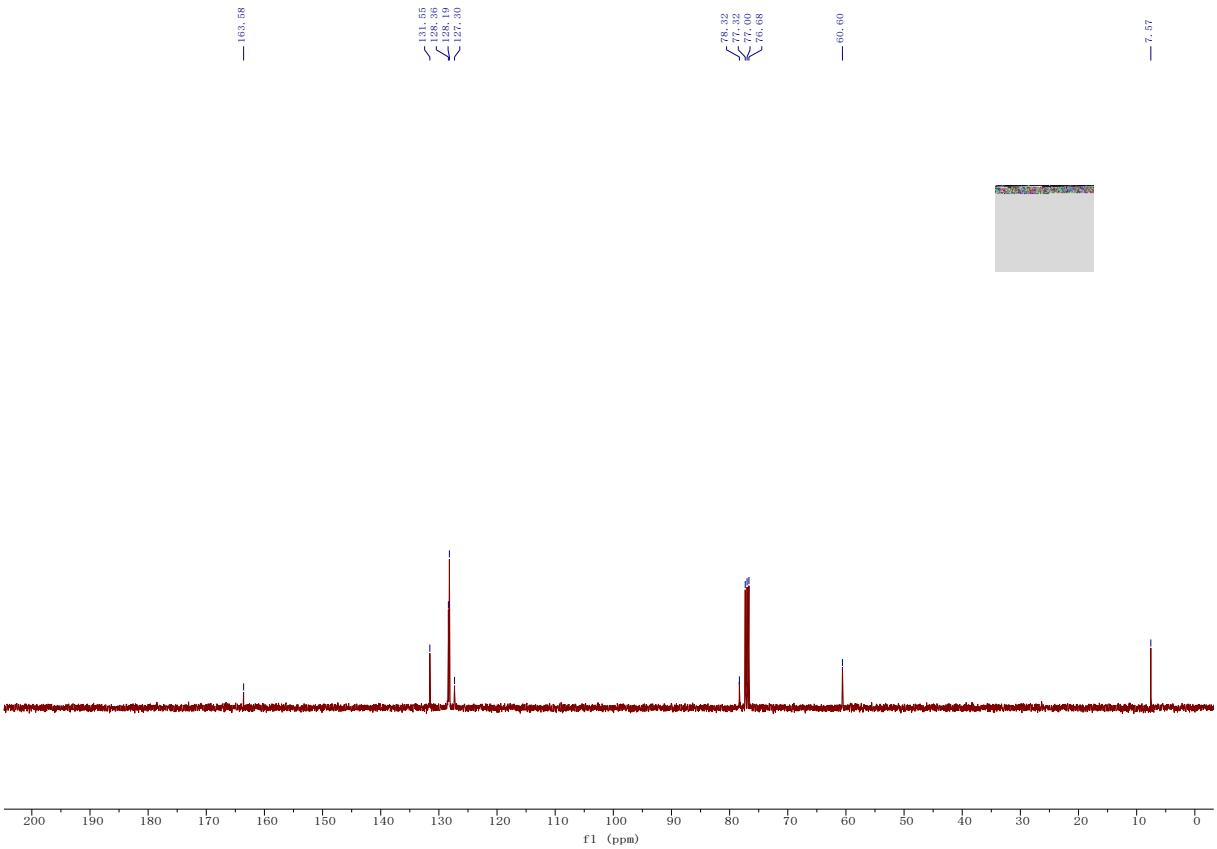
198.3 mg, yellow oil, yield: 73%
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 – 7.64 (m, 2H), 7.45 – 7.16 (m, 10H), 7.00 (d, *J* = 7.8 Hz, 2H), 5.37 (s, 2H), 4.40 – 4.27 (m, 1H), 3.82 (dd, *J* = 15.1, 9.6 Hz, 1H), 3.72 (s, 3H), 3.45 (dd, *J* = 15.1, 6.8 Hz, 1H), 2.90 – 2.81 (m, 3H), 2.78 (dd, *J* = 10.5, 4.9 Hz, 1H), 2.70 (s, 3H), 1.80 (m, 2H), 0.99 (t, *J* = 7.3 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 164.31, 156.40, 154.44, 143.01, 142.45, 141.02, 140.81, 136.43, 134.94, 134.70, 130.56, 130.22, 130.06, 129.33, 129.14, 127.25, 126.99, 125.75, 123.70, 123.56, 122.50, 122.32, 119.27, 109.49, 108.96, 78.33, 60.53, 46.96, 31.81, 29.72, 21.76, 16.84, 14.05, 7.10.
HRMS (ESI) Calc. for $C_{36}H_{34}IN_5O$ $[M+H]^+$: 680.1881; found: 680.1884.

[1] J. D. Haupt, M. Berger, S. R. Waldvogel, *Org. Lett.* **2019**, *21*, 242-245.
[2] M. Zhou, H.-Y. Zhao, S. Zhang, Y. Zhang, X. Zhang, *J. Am. Chem. Soc.* **2020**, *142*, 18191-18199.
[3] G.-Q. Liu, C.-H. Yang, Y.-M. Li, *J. Org. Chem.* **2015**, *80*, 11339-11350.

[4] C.-H. Yang, Z.-Q. Xu, L. Duan, Y.-M. Li, *Tetrahedron* **2017**, *73*, 6747-6753.
[5] K. Nagaraju, N. Rajesh, P. R. Krishna, *Synth. Commun.* **2018**, *48*, 1001-1007.
[6] O. Kentaro, Y. Takumi, S. Kosei, Y. Yoshinobu, *Bull. Chem. Soc. Jpn.* **2007**, *80*, 1824-1827.
[7] W. Yi, P.-F. Wang, M. Lu, Q.-Q. Liu, X. Bai, K.-D. Chen, J.-W. Zhang, G.-Q. Liu, *ACS Sustain. Chem. Eng.* **2019**, *7*, 16777-16785.

4. Copy of 1H and 13C NMR Spectra of Products

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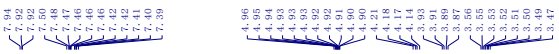
131.55
128.36
128.19
127.30
78.32
77.32
77.00
76.68
60.60
7.57
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.94
7.92
7.92
7.50
7.48
7.47
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7.46
7.46
7.42
7.42
7.41
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7.39
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4.93
4.93
4.92
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4.90
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4.14
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3.53
3.52
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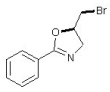
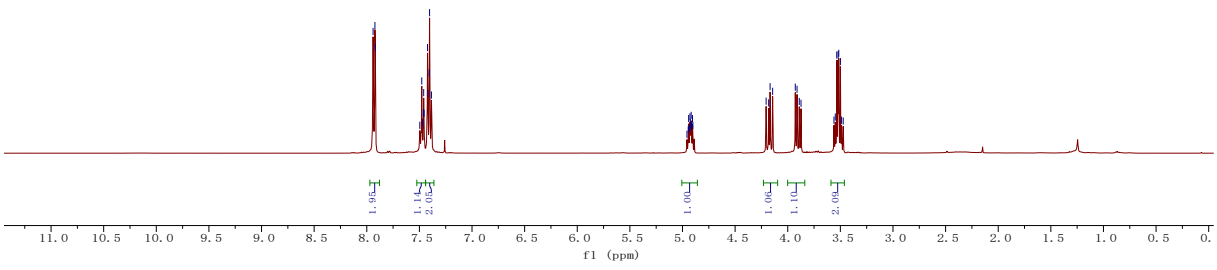
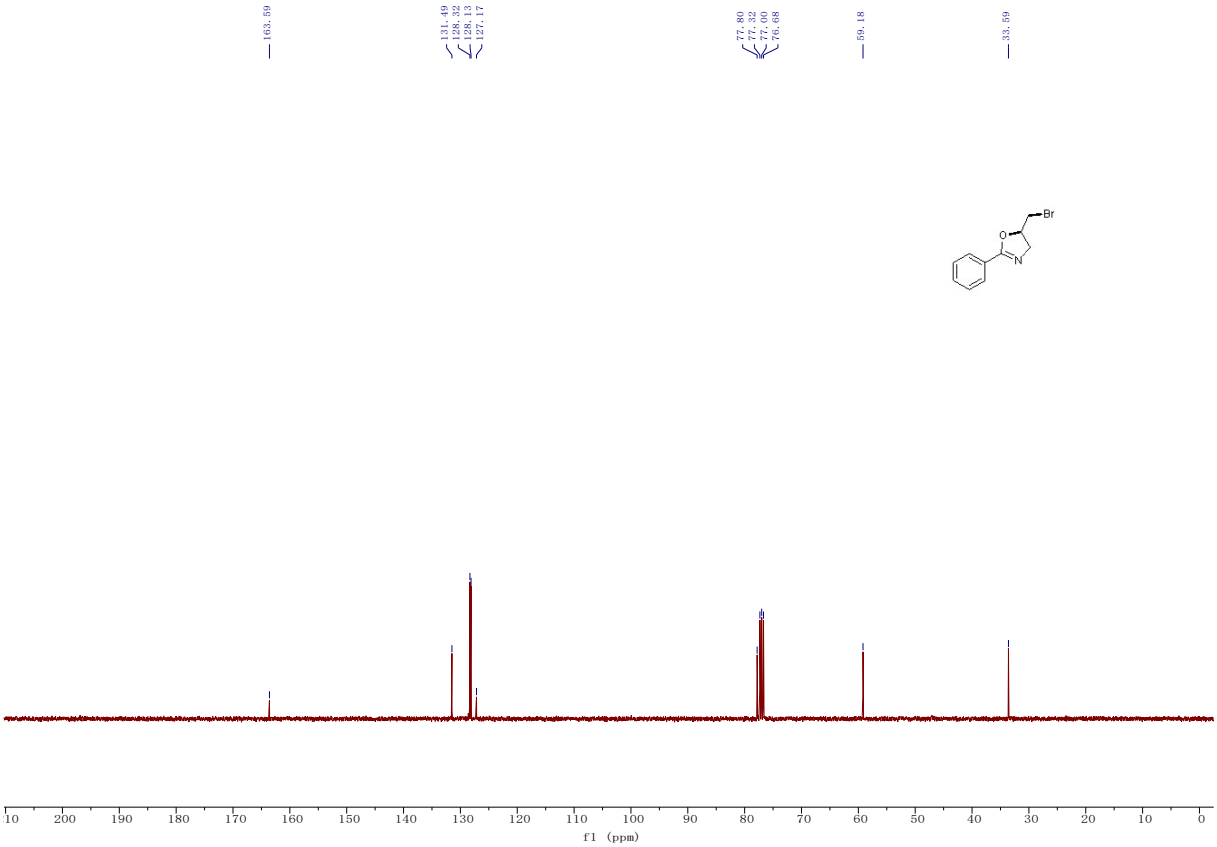
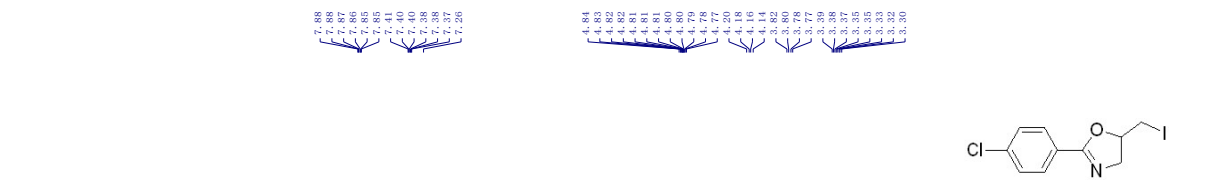
3.50
3.49
3.47


Br
O
N


1.95
1.14
2.05
1.00
1.06
1.10
2.09
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.
f1 (ppm)


163.59
131.49
128.32
128.13
127.17
77.80
77.22
77.00
76.68
59.18
33.59
Br
O
N
f1 (ppm)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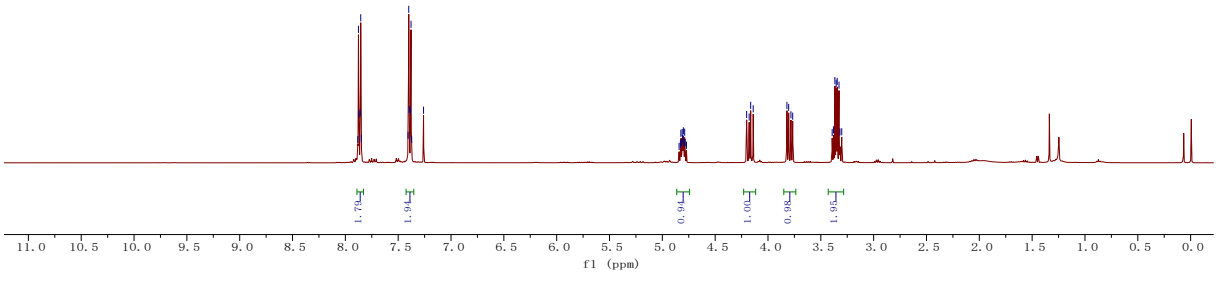
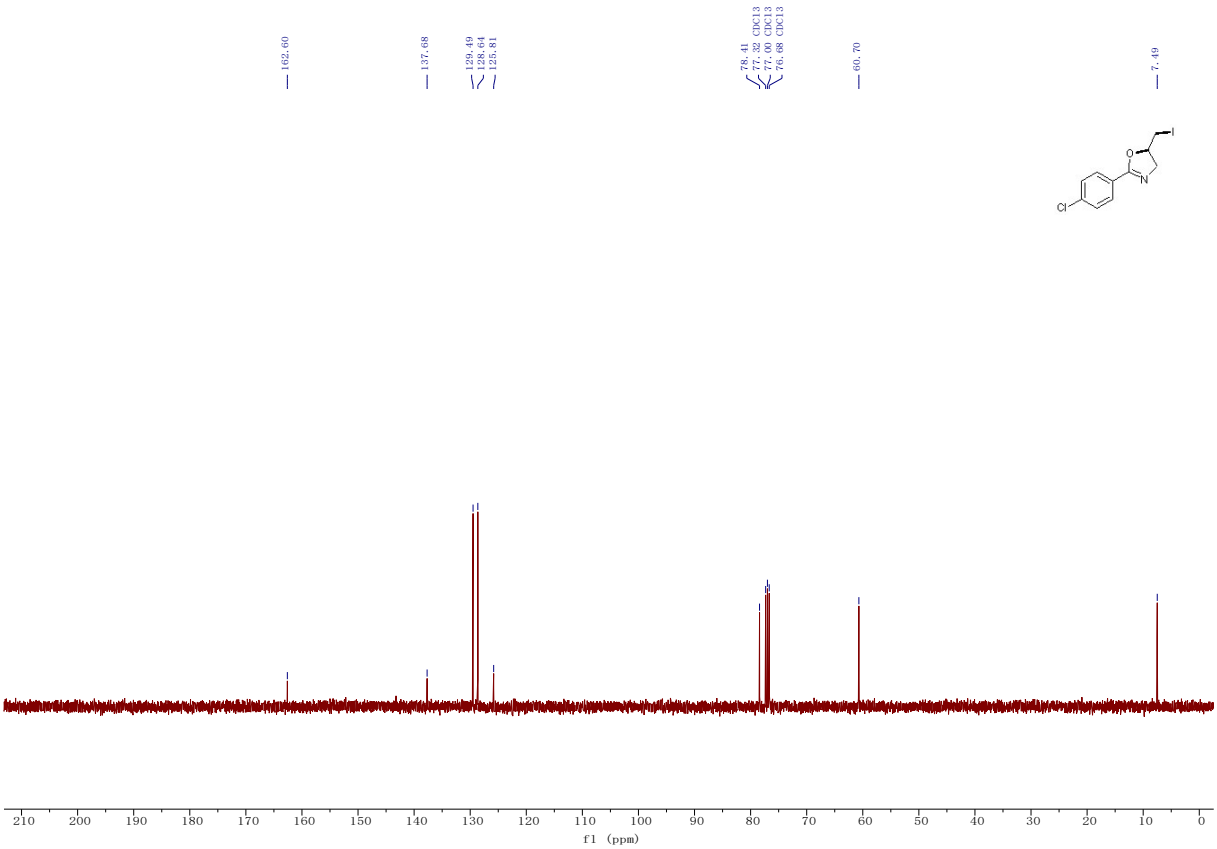
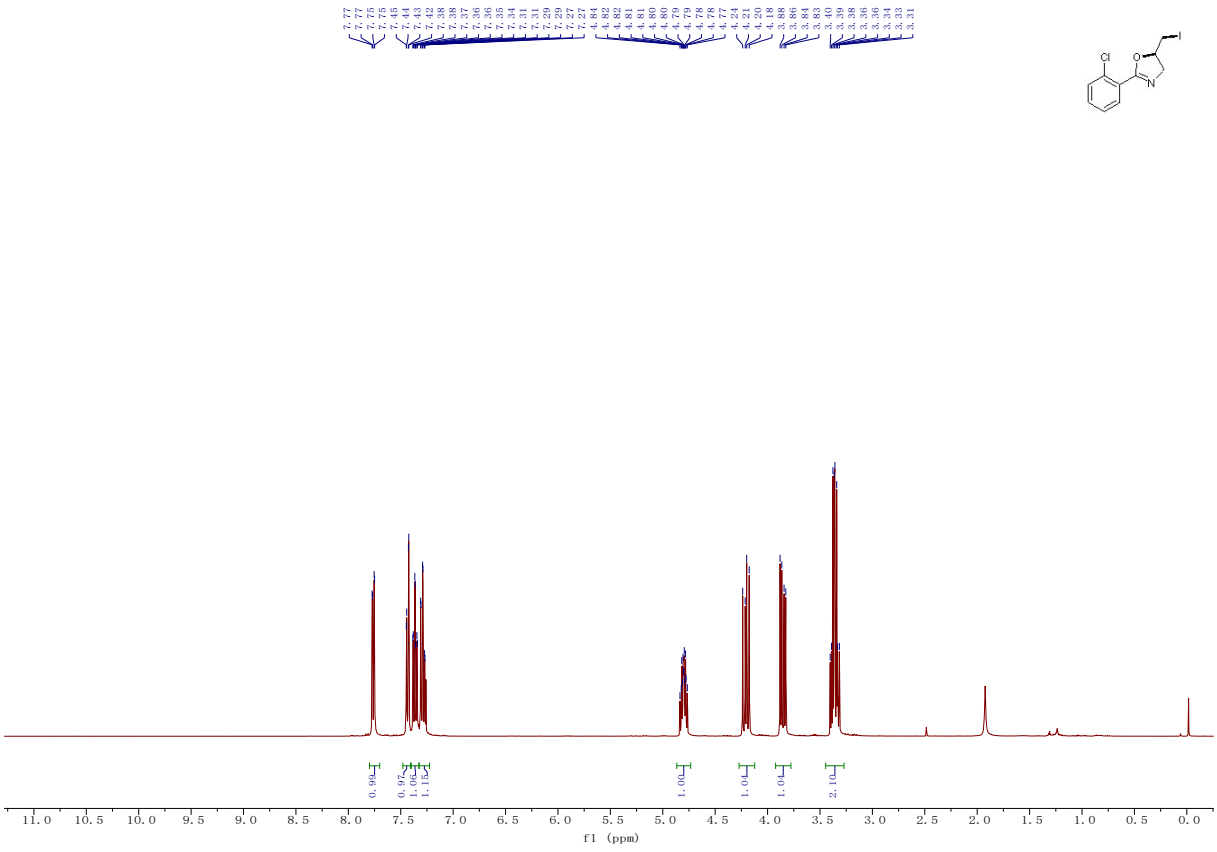
7.88
7.87
7.86
7.85
7.85
7.41
7.40
7.40
7.38
7.38
7.37
7.36
4.84
4.83
4.82
4.82
4.81
4.81
4.81
4.80
4.80
4.79
4.78
4.77
4.20
4.18
4.16
4.14
3.82
3.80
3.78
3.77
3.39
3.38
3.37
3.35
3.35
3.33
3.32
3.30

Cl
O
N
I


1.79
1.94
0.94
1.01
0.98
1.95
f1 (ppm)


162.60
137.68
129.49
128.64
125.81
78.41
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
60.70
7.49
O
N
Cl
I
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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7.77
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7.75
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7.43
7.42
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7.38
7.37
7.36
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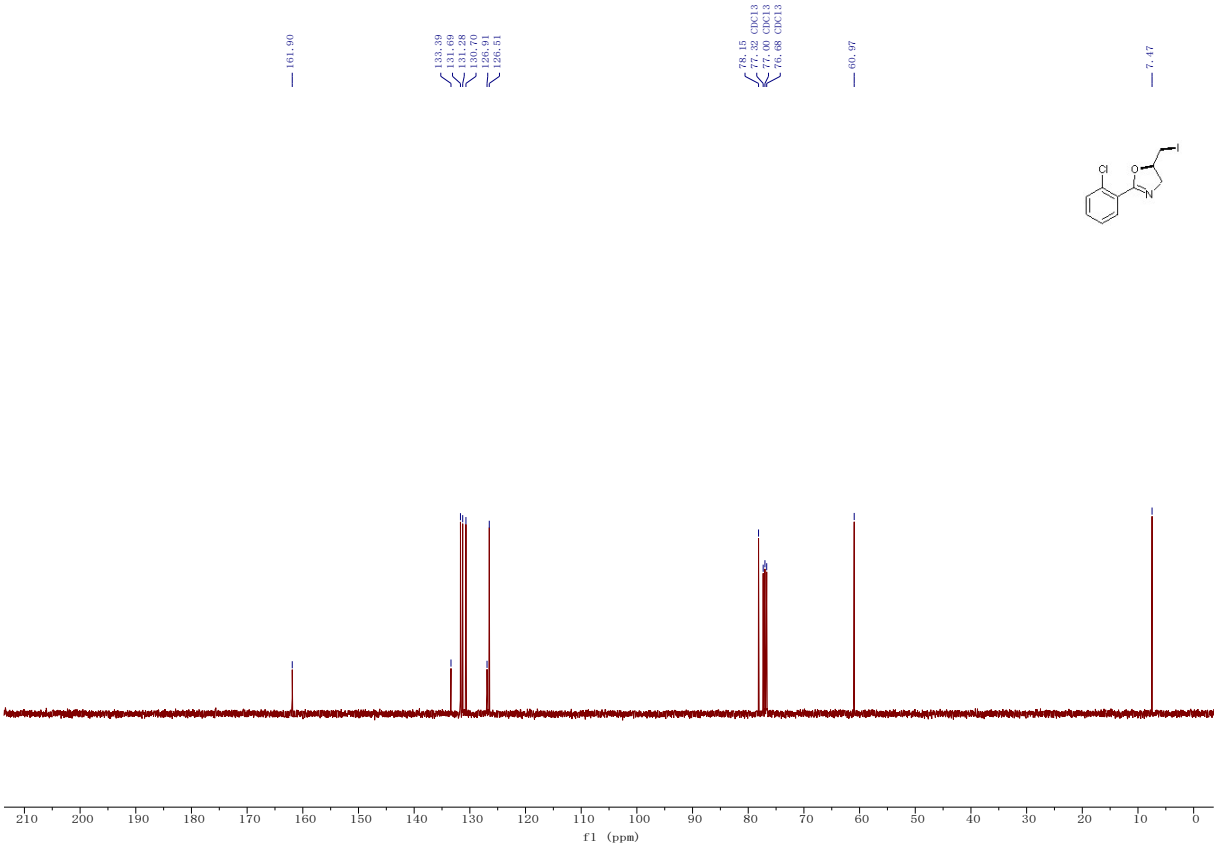
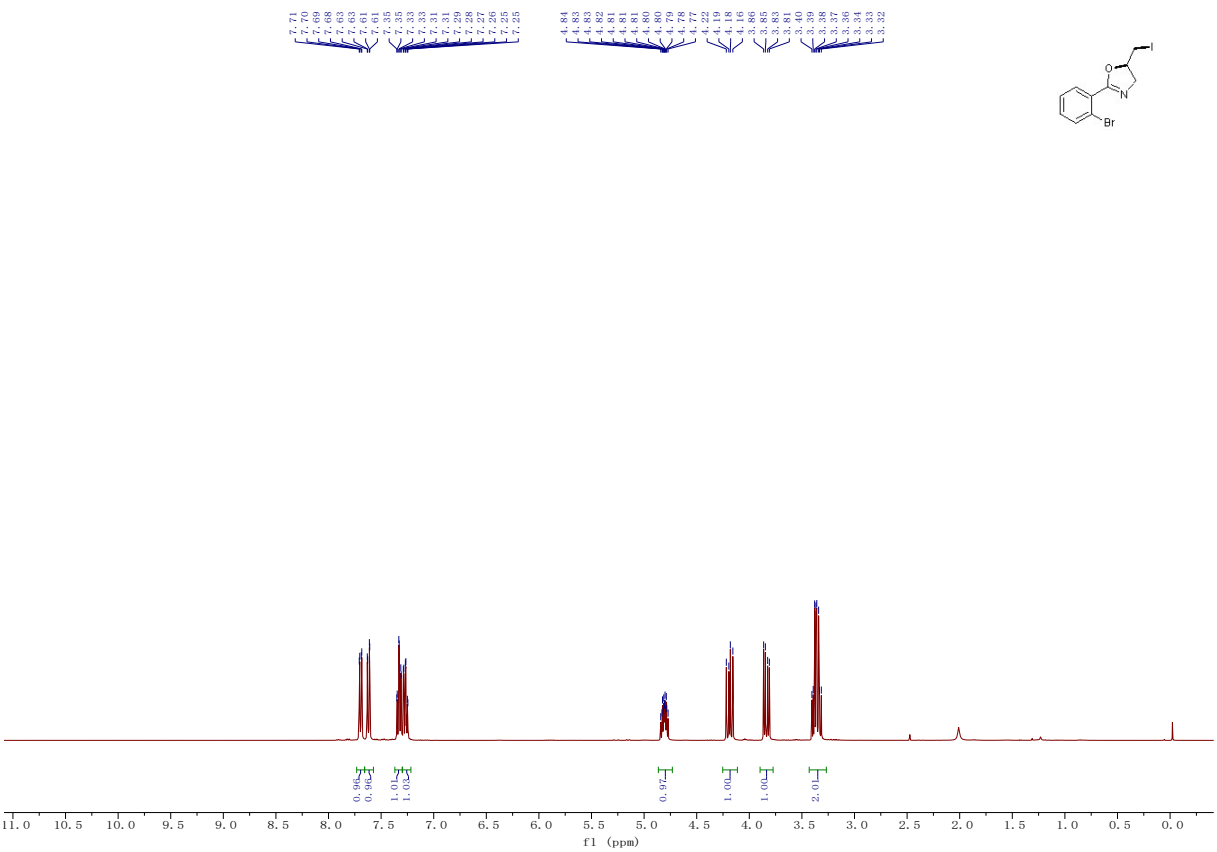
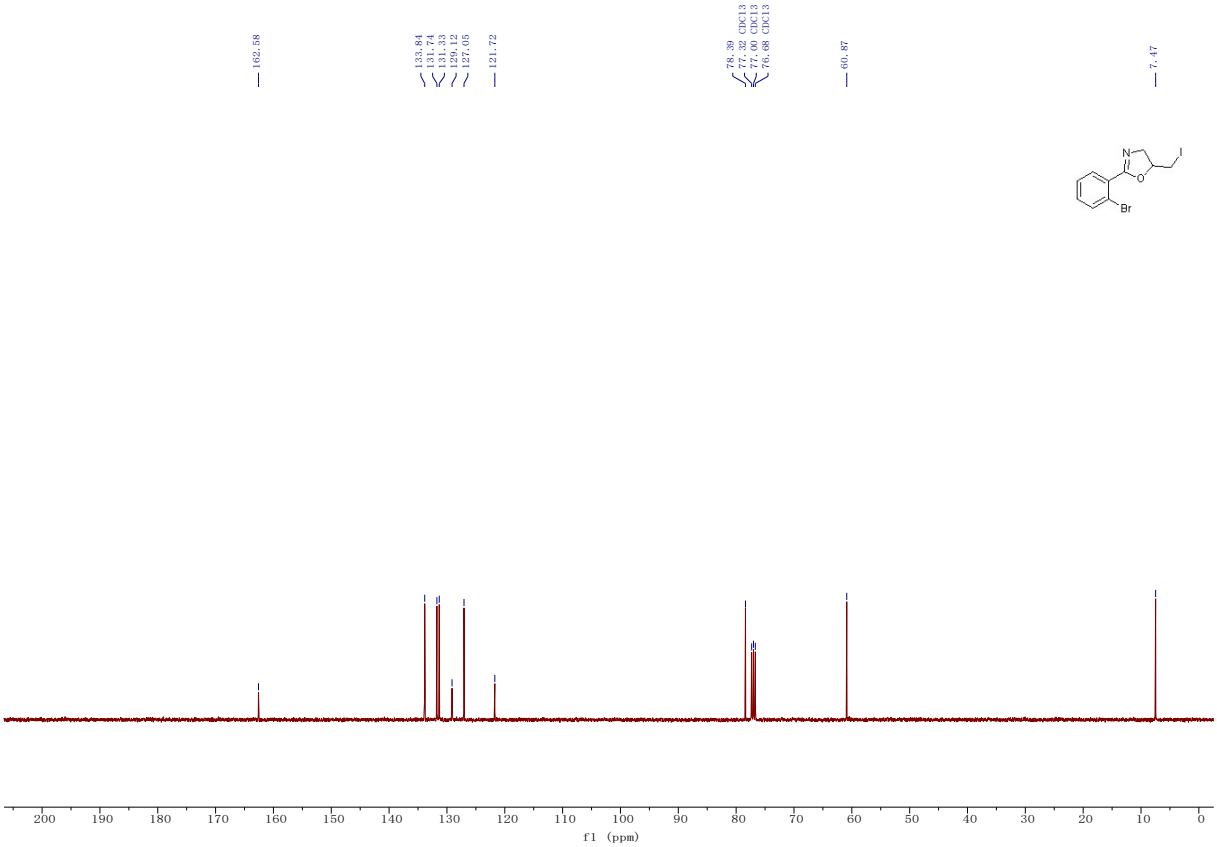
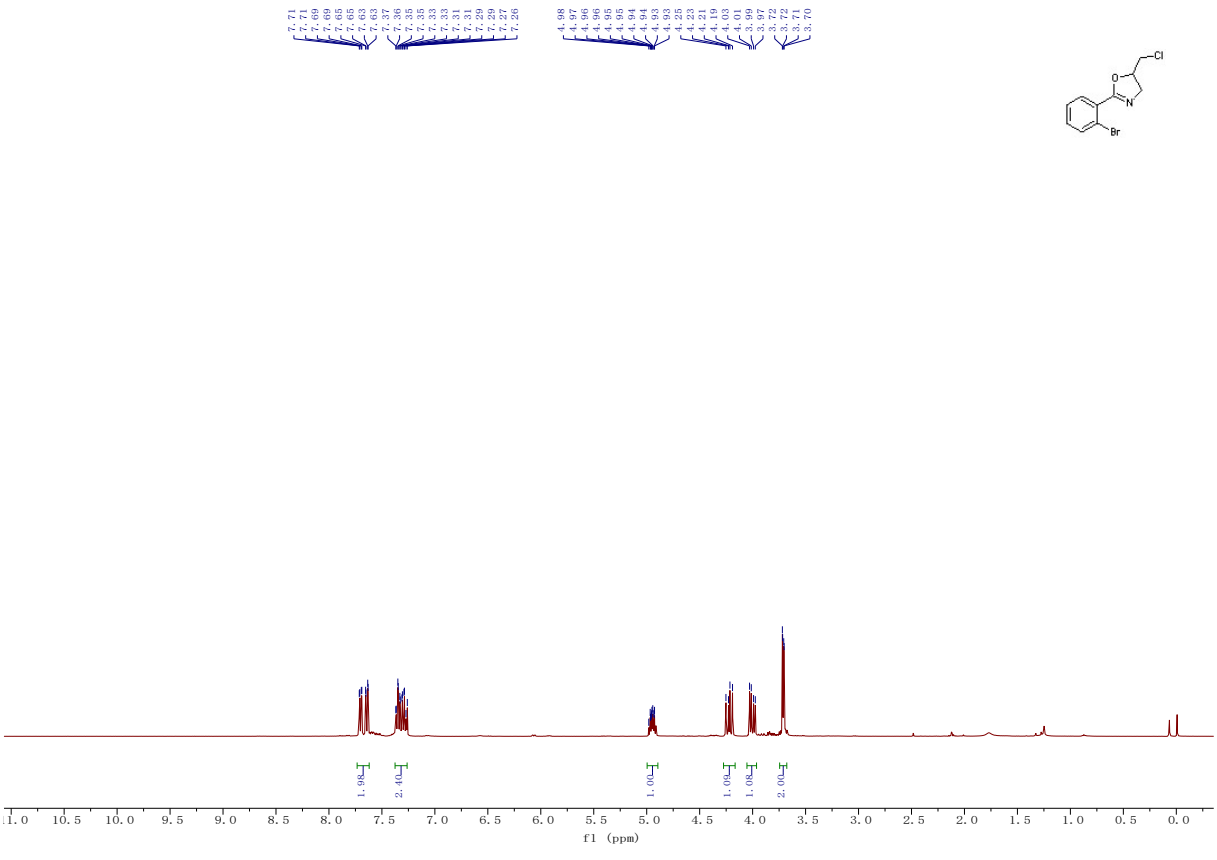
7.35
7.34
7.31
7.31
7.29
7.29
7.27
7.27
4.84
4.82
4.81
4.81
4.80
4.80
4.79
4.79
4.78
4.78
4.77
4.24
4.21
4.20
4.18
3.88
3.86
3.84
3.83
3.40
3.39
3.38
3.36
3.36
3.34
3.33
3.31
Cl
O
N
I
0.99
0.97
1.06
1.15
1.00
1.04
1.04
2.10
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


161.90
133.39
131.69
131.28
130.70
130.31
126.51
78.15
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
60.97
7.47
Cl
O
N
I
f1 (ppm)


7.71
7.70
7.69
7.68
7.63
7.63
7.61
7.61
7.35
7.35
7.33
7.33
7.31
7.31
7.29
7.28
7.27
7.26
7.25
7.25
4.84
4.83
4.83
4.82
4.81
4.81
4.81
4.80
4.80
4.79
4.79
4.78
4.77
4.22
4.19
4.18
4.16
3.86
3.85
3.83
3.81
3.40
3.39
3.38
3.37
3.36
3.34
3.33
3.32
O
N
Br
I
0.96
0.96
1.01
1.03
0.97
1.06
1.01
2.01
f1 (ppm)


162.58
133.84
131.74
131.33
129.12
127.05
121.72
78.39
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
60.87
7.47
N
O
I
Br
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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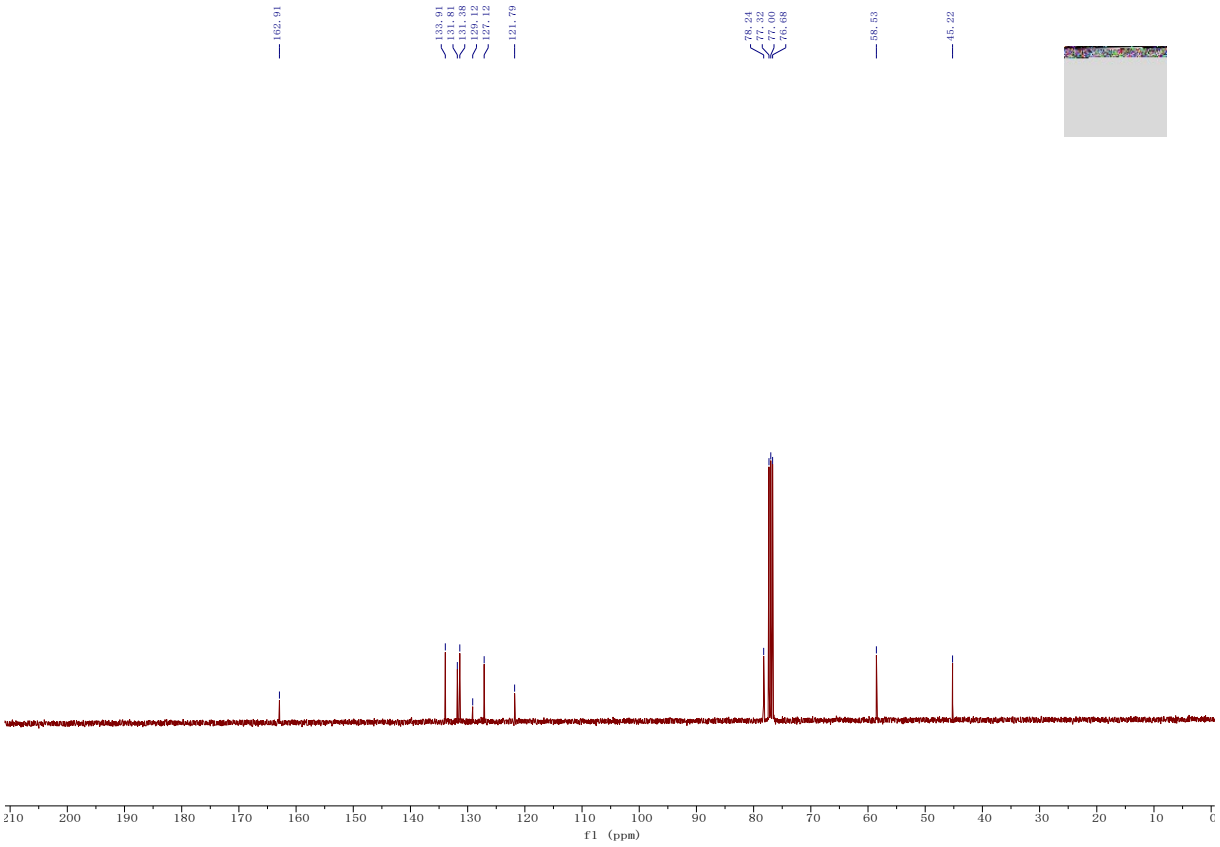
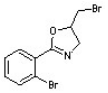
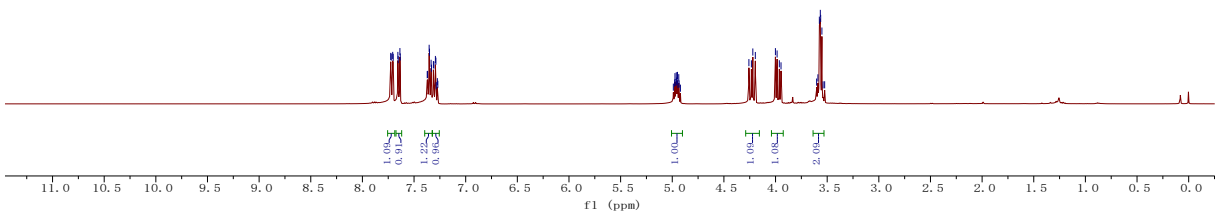
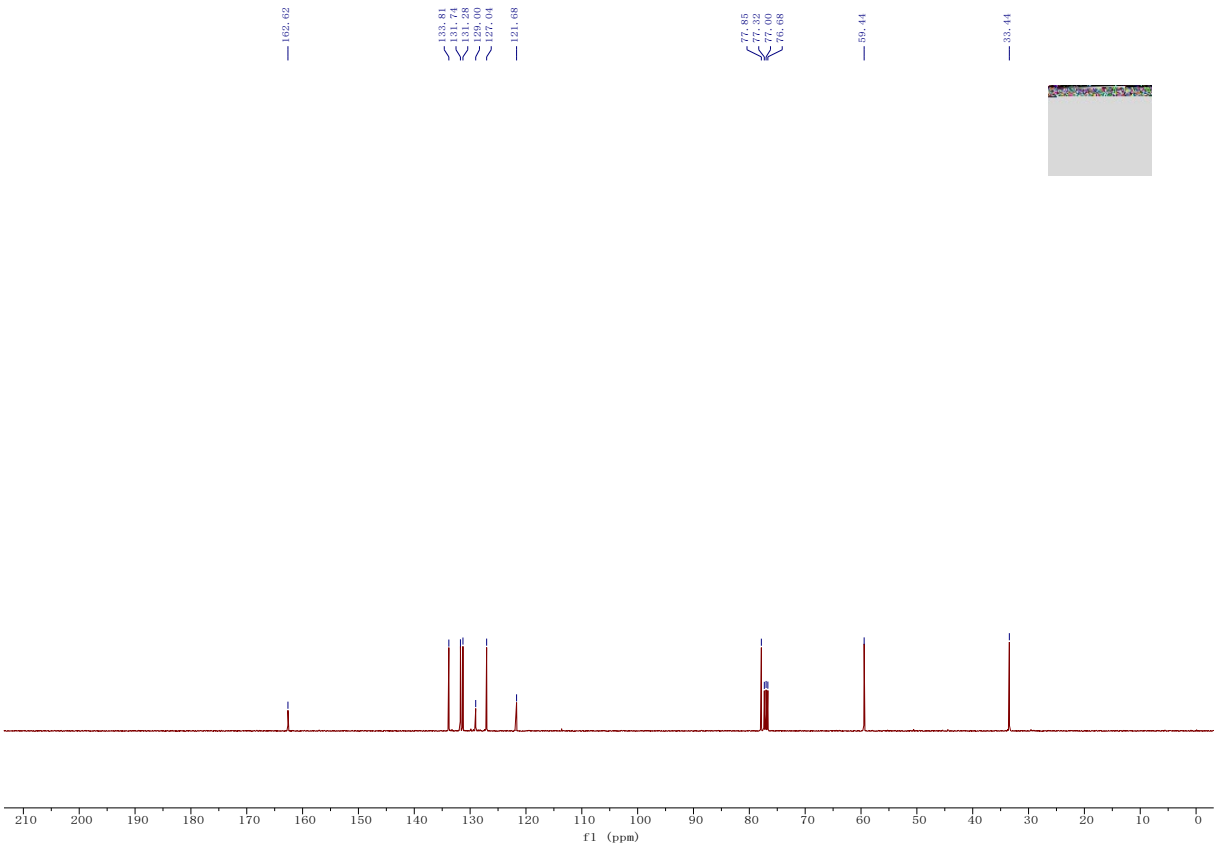
7.71
7.69
7.69
7.65
7.65
7.63
7.63
7.37
7.36
7.35
7.35
7.33
7.33
7.31
7.31
7.29
7.29
7.27
7.26
4.98
4.97
4.96
4.96
4.95
4.95
4.94
4.94
4.93
4.93
4.25
4.23
4.21
4.19
4.03
4.01
3.99
3.97
3.72
3.72
3.71
3.70
Cl
O
N
Br
1.98
2.40
1.00
1.09
1.08
2.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

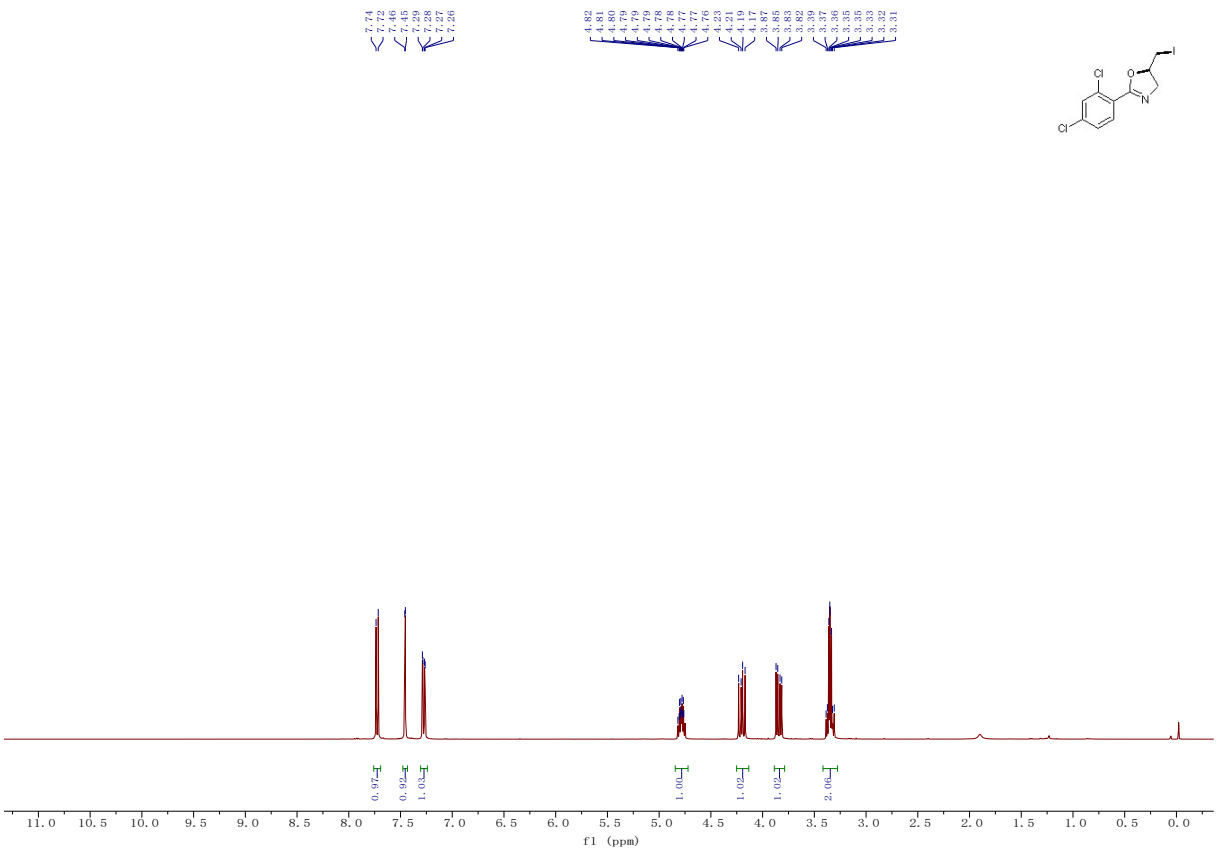

162.91
133.91
131.81
131.38
129.12
127.12
121.79
78.24
77.32
77.00
76.68
58.53
45.22
f1 (ppm)

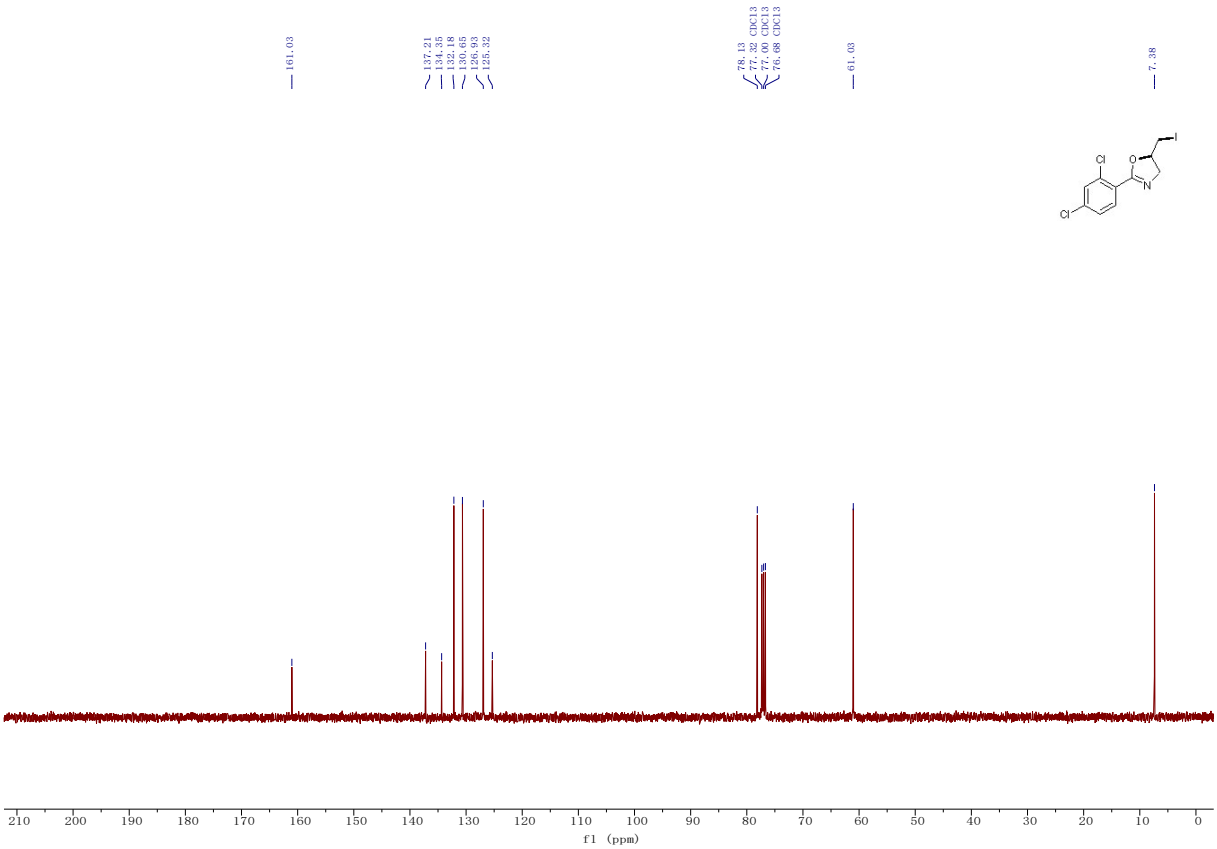

7.73
7.72
7.71
7.71
7.66
7.65
7.64
7.63
7.37
7.37
7.35
7.35
7.33
7.33
7.31
7.31
7.29
7.29
7.29
7.27
7.27
7.27
4.99
4.98
4.97
4.97
4.96
4.96
4.95
4.95
4.95
4.94
4.93
4.92
4.36
4.23
4.22
4.20
4.00
3.98
3.96
3.95
3.60
3.59
3.58
3.57
3.56
3.55
3.54
3.52

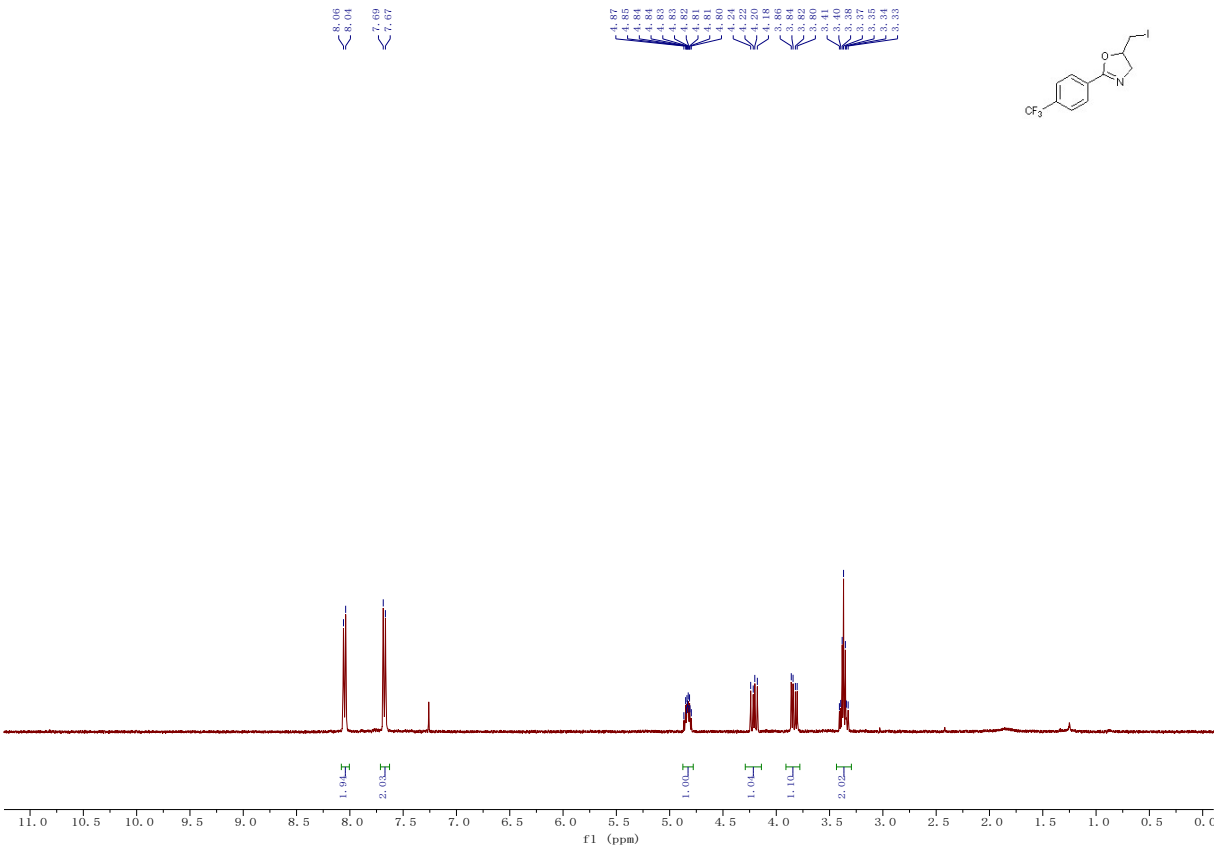

Br
O
N
Br

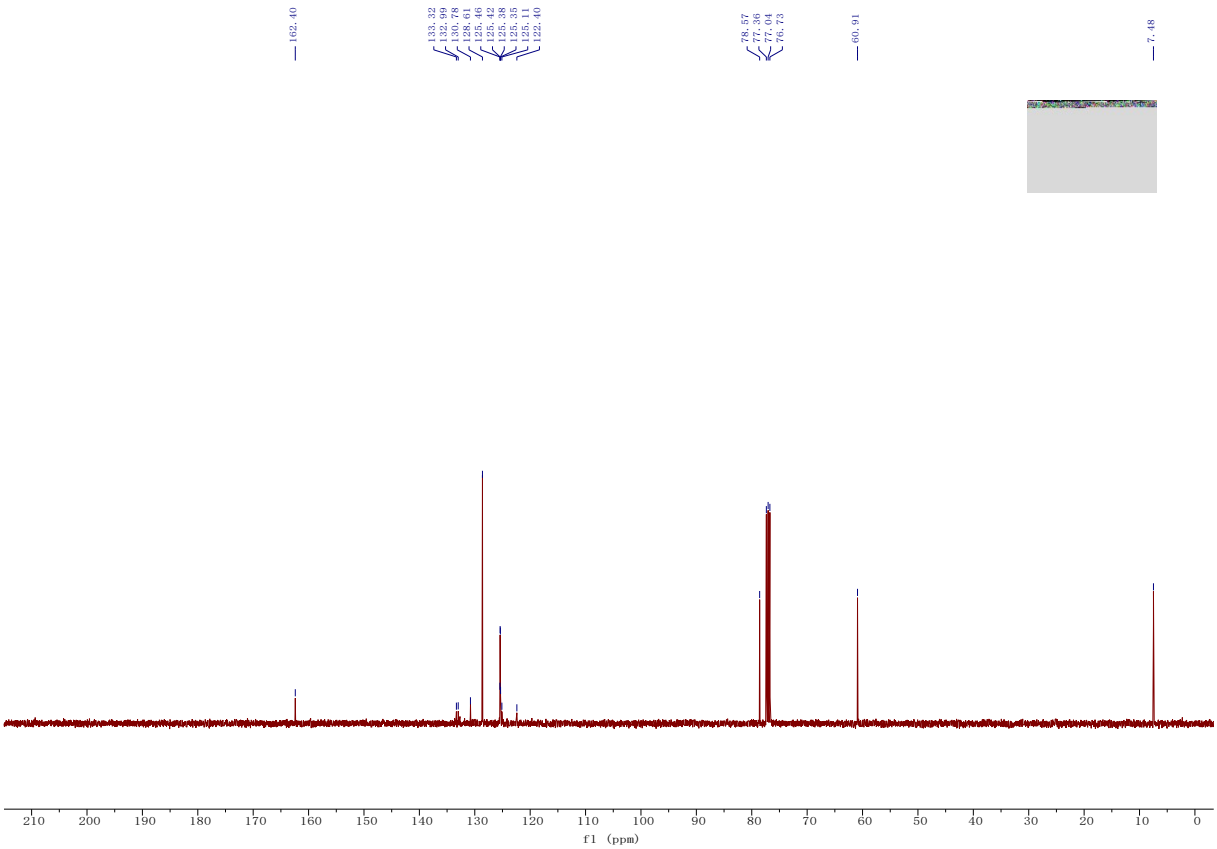

1.09
0.91
1.22
0.96
1.00
1.09
1.08
2.09
f1 (ppm)


162.62
133.81
131.74
131.28
129.00
127.04
121.68
77.85
77.32
77.00
76.68
59.44
33.44
f1 (ppm)


7.74
7.72
7.46
7.45
7.29
7.28
7.27
7.26
4.82
4.81
4.80
4.79
4.79
4.79
4.78
4.78
4.77
4.77
4.76
4.23
4.21
4.19
4.17
3.87
3.85
3.83
3.82
3.39
3.37
3.36
3.35
3.35
3.33
3.32
3.31
Cl
O
N
I
Cl
0.97
0.92
1.03
1.00
1.02
1.02
2.06
f1 (ppm)


161.03
137.21
134.35
132.18
130.65
126.93
125.32
78.13
77.32
77.00
76.68
CDCl3
61.03
7.38
Cl
Cl
O
N
I
f1 (ppm)


8.06
8.04
7.69
7.67
4.87
4.85
4.84
4.84
4.83
4.82
4.81
4.81
4.80
4.24
4.22
4.20
4.18
3.86
3.84
3.82
3.80
3.41
3.40
3.38
3.37
3.35
3.34
3.33
O
N
I
CF3
1.94
2.03
1.00
1.04
1.10
2.02
f1 (ppm)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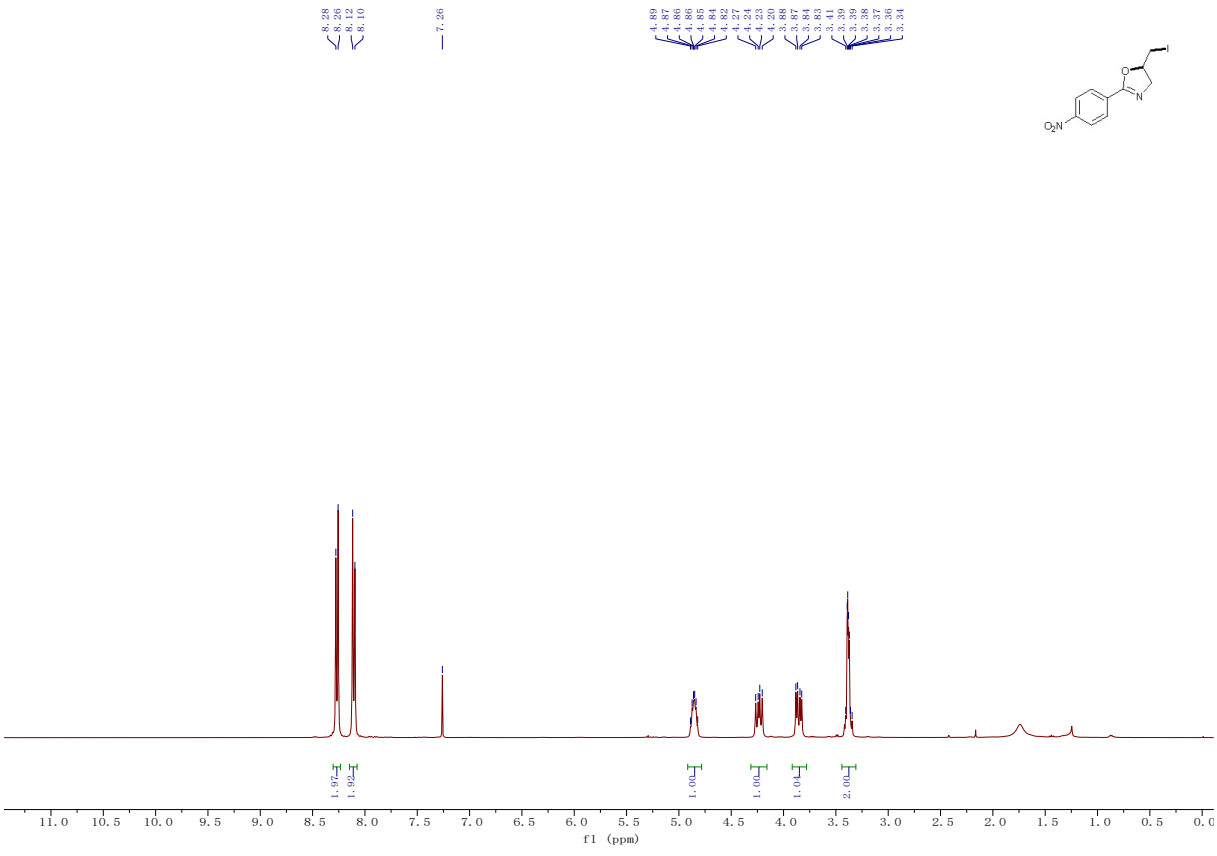
133.32
132.99
130.78
128.61
125.46
125.42
125.38
125.35
125.11
122.40
78.57
77.36
77.04
76.73
60.91
7.48
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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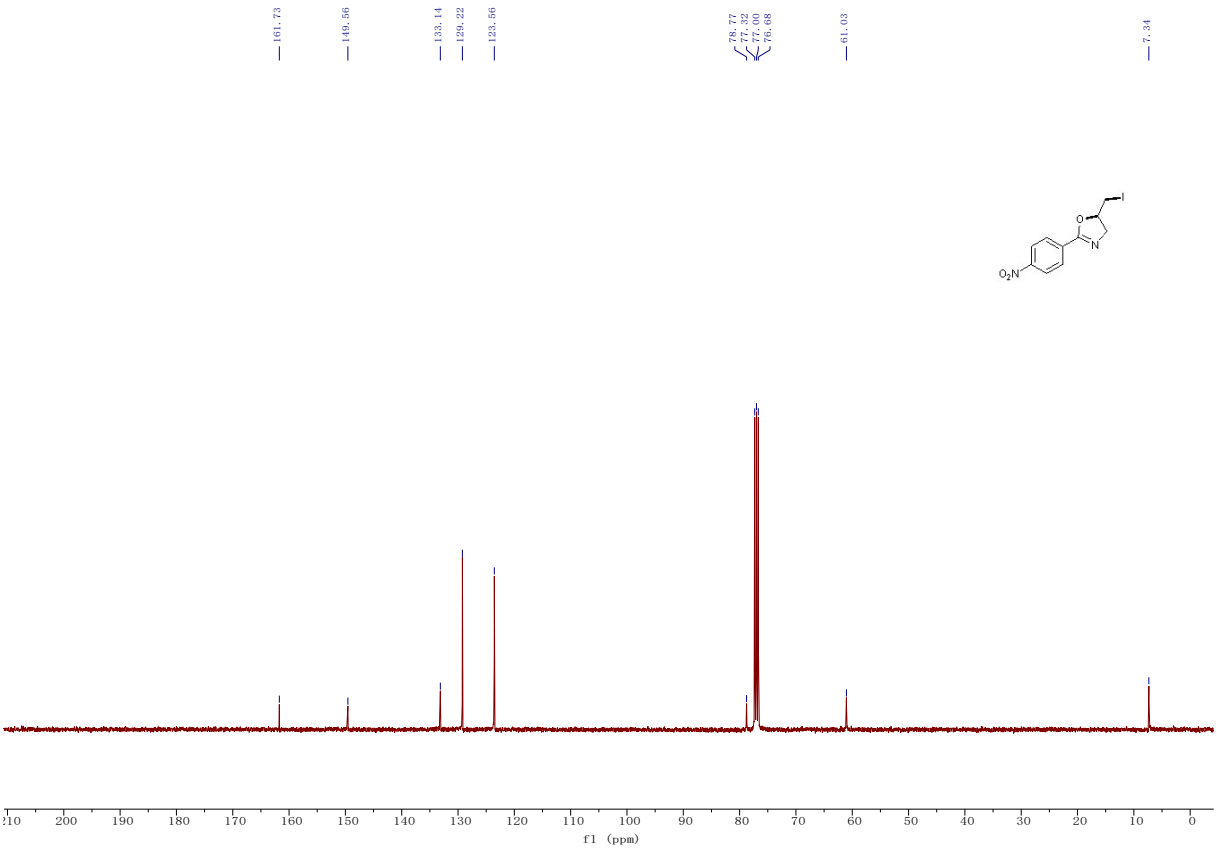
8.26
8.12
8.10
7.26
4.89
4.87
4.86
4.86
4.85
4.84
4.82
4.77
4.24
4.23
4.20
3.88
3.87
3.84
3.83
3.41
3.39
3.39
3.38
3.37
3.36
3.34
O
N
O2N
1.97
1.92
1.00
1.00
1.04
2.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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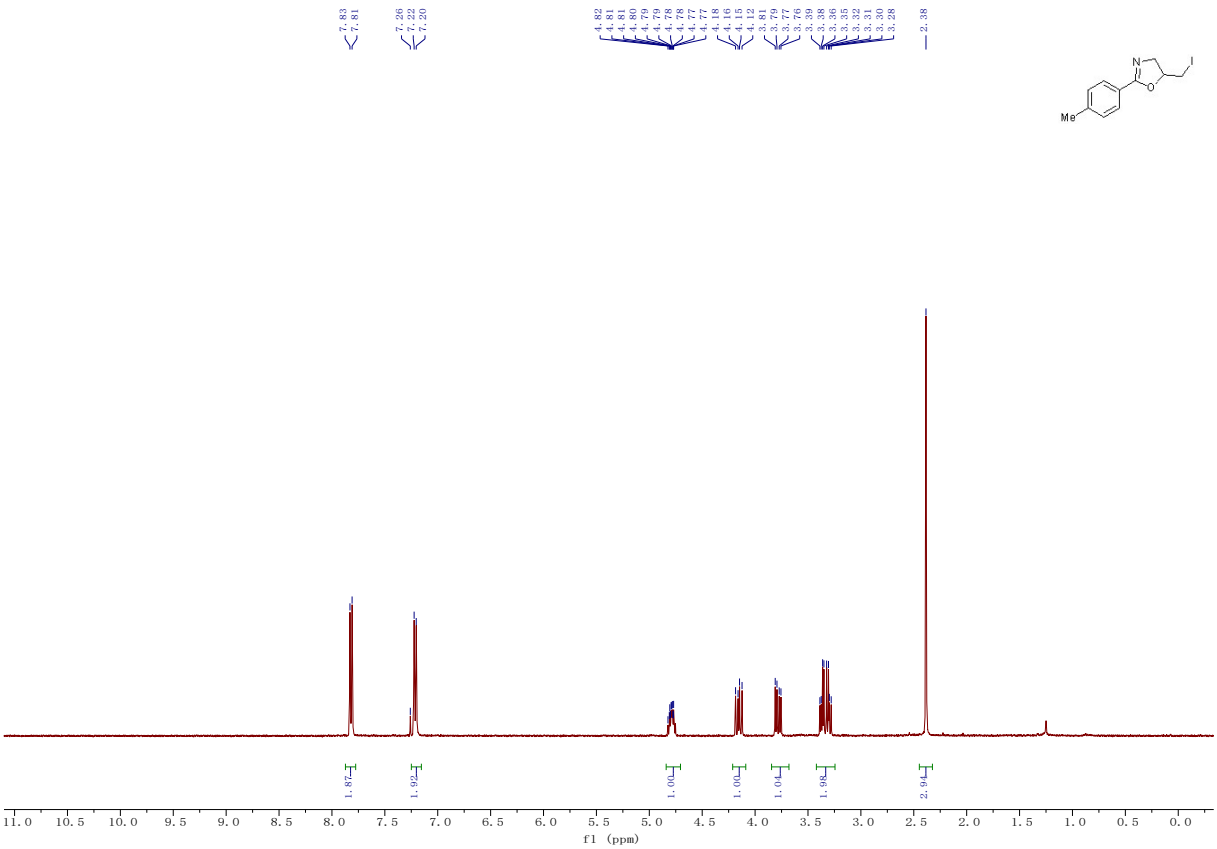
149.56
133.14
129.22
123.56
78.77
77.32
77.00
76.68
61.03
7.34
I
O
N
O2N
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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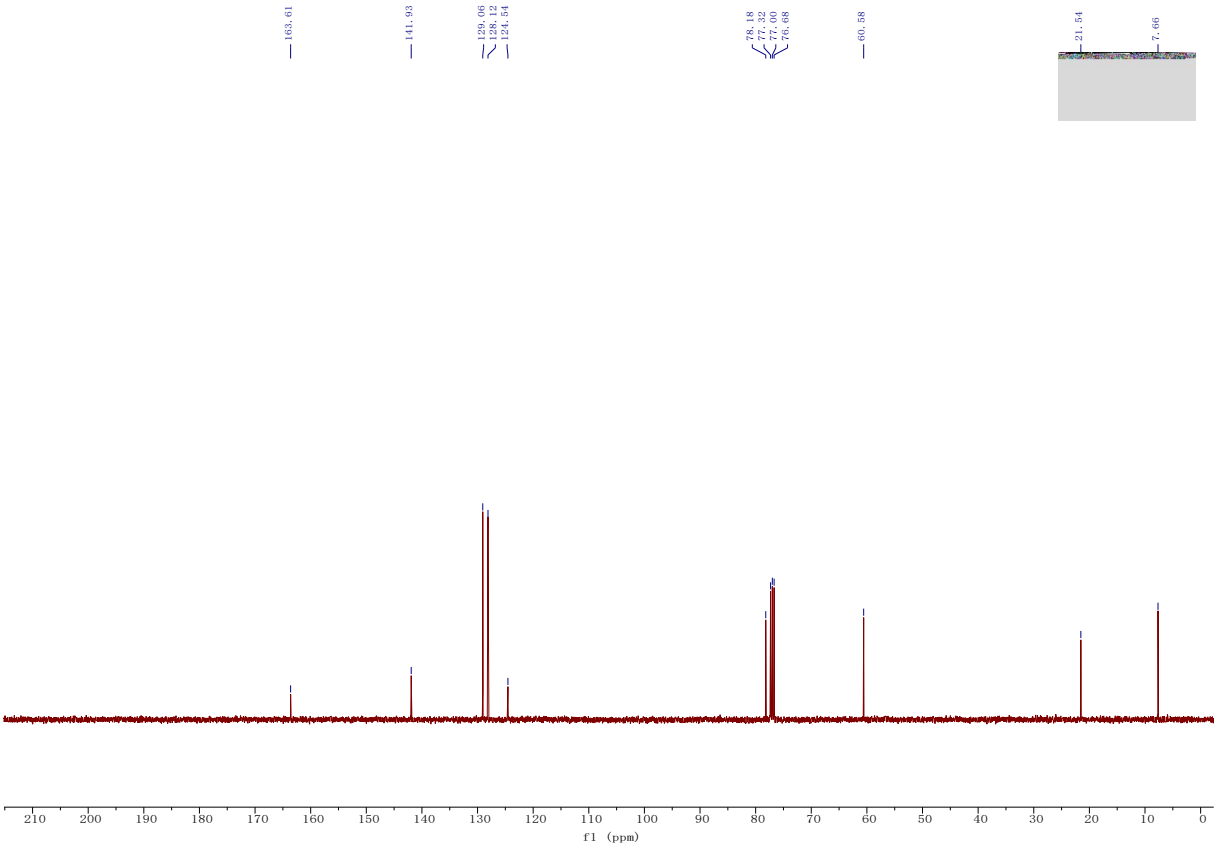
7.81
7.26
7.22
7.20
4.82
4.81
4.81
4.80
4.79
4.79
4.78
4.78
4.77
4.77
4.18
4.16
4.15
4.12
3.81
3.79
3.77
3.76
3.39
3.38
3.36
3.35
3.32
3.31
3.30
3.28
2.38
N
O
I
Me
1.87
1.92
1.00
1.00
1.04
1.98
2.94
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


163.61
141.93
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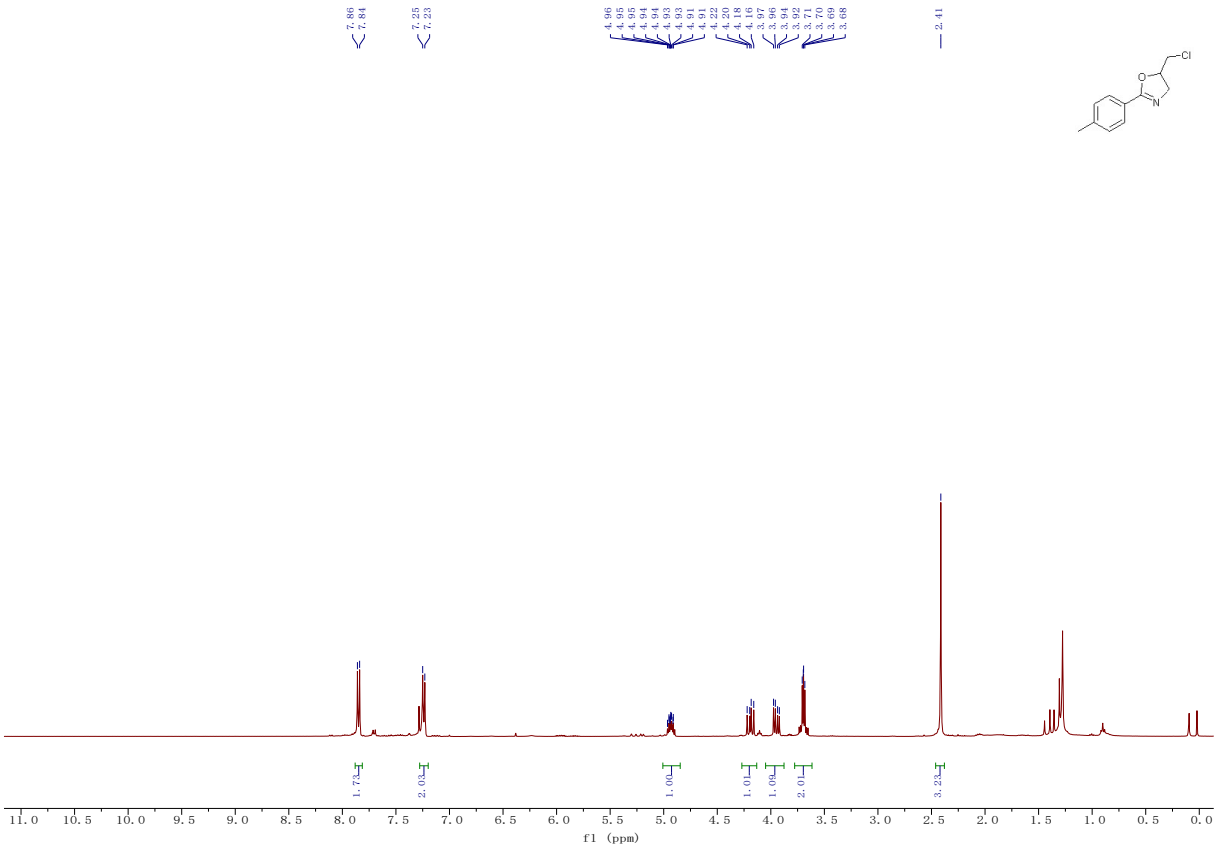
124.54
78.18
77.32
77.00
76.68
60.58
21.54
7.66
f1 (ppm)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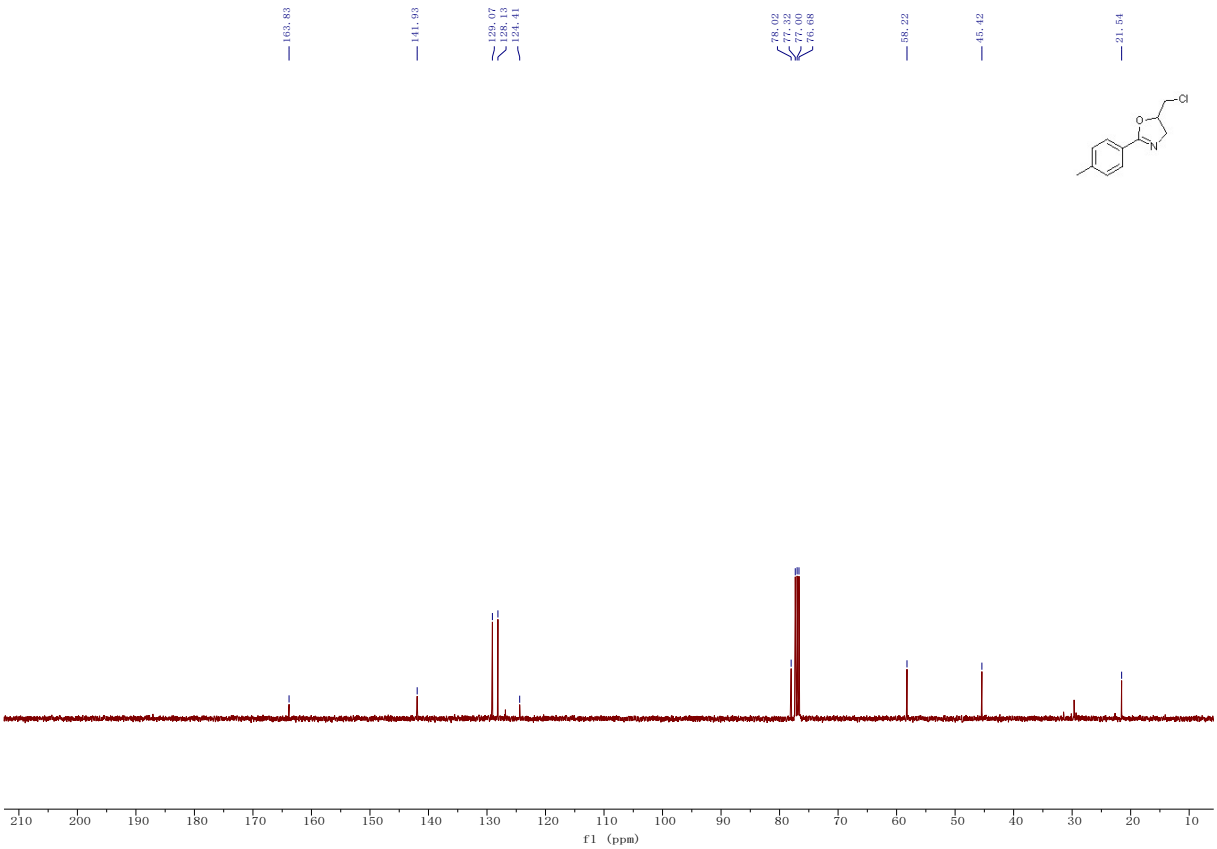
7.84
7.25
7.23
4.96
4.95
4.95
4.94
4.94
4.44
4.93
4.93
4.91
4.91
4.22
4.20
4.18
4.16
3.97
3.96
3.94
3.92
3.71
3.70
3.69
3.68
2.41
Cl
O
N
1.73
2.03
1.00
1.01
1.09
2.01
3.23
f1 (ppm)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141.93
129.07
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124.41
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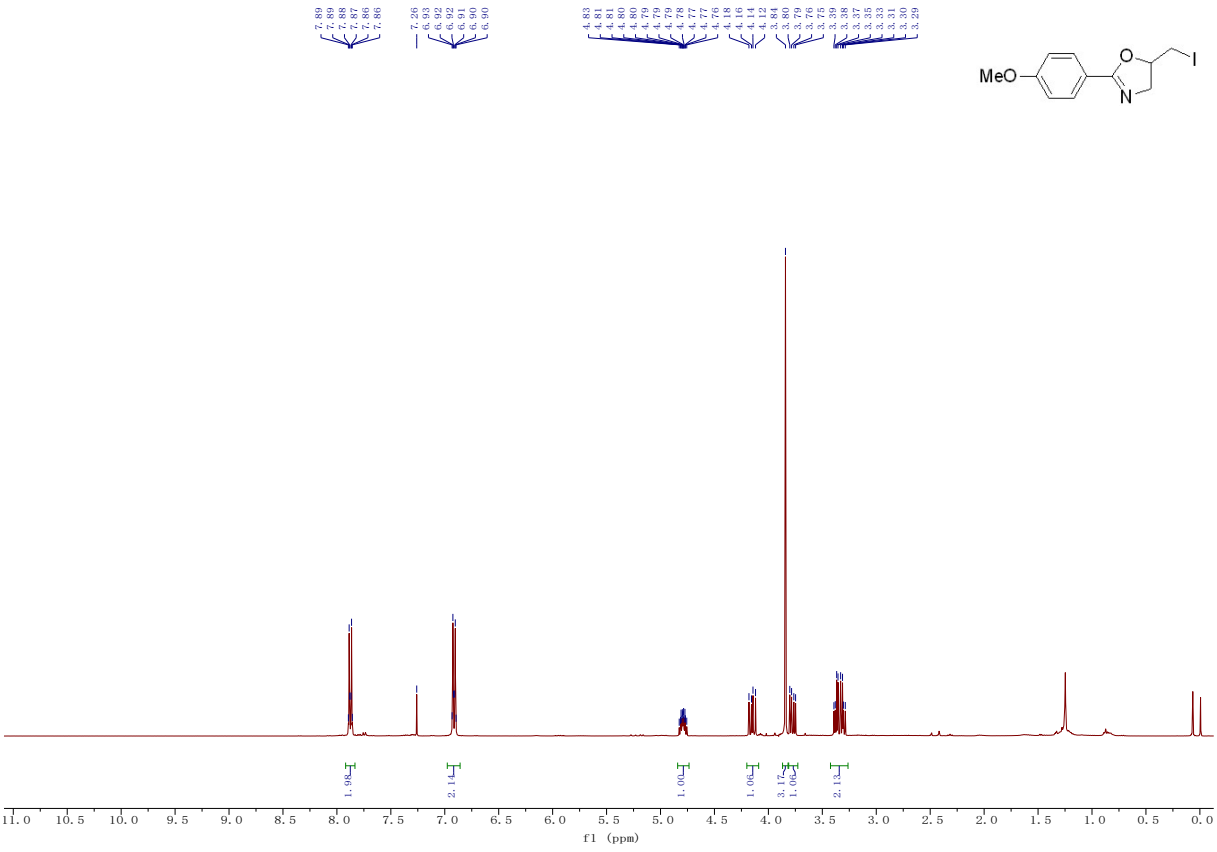
77.32
77.00
76.68
58.22
45.42
21.64
Cl
O
N
f1 (ppm)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7.89
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7.86
7.26
6.93
6.92
6.92
6.91
6.90
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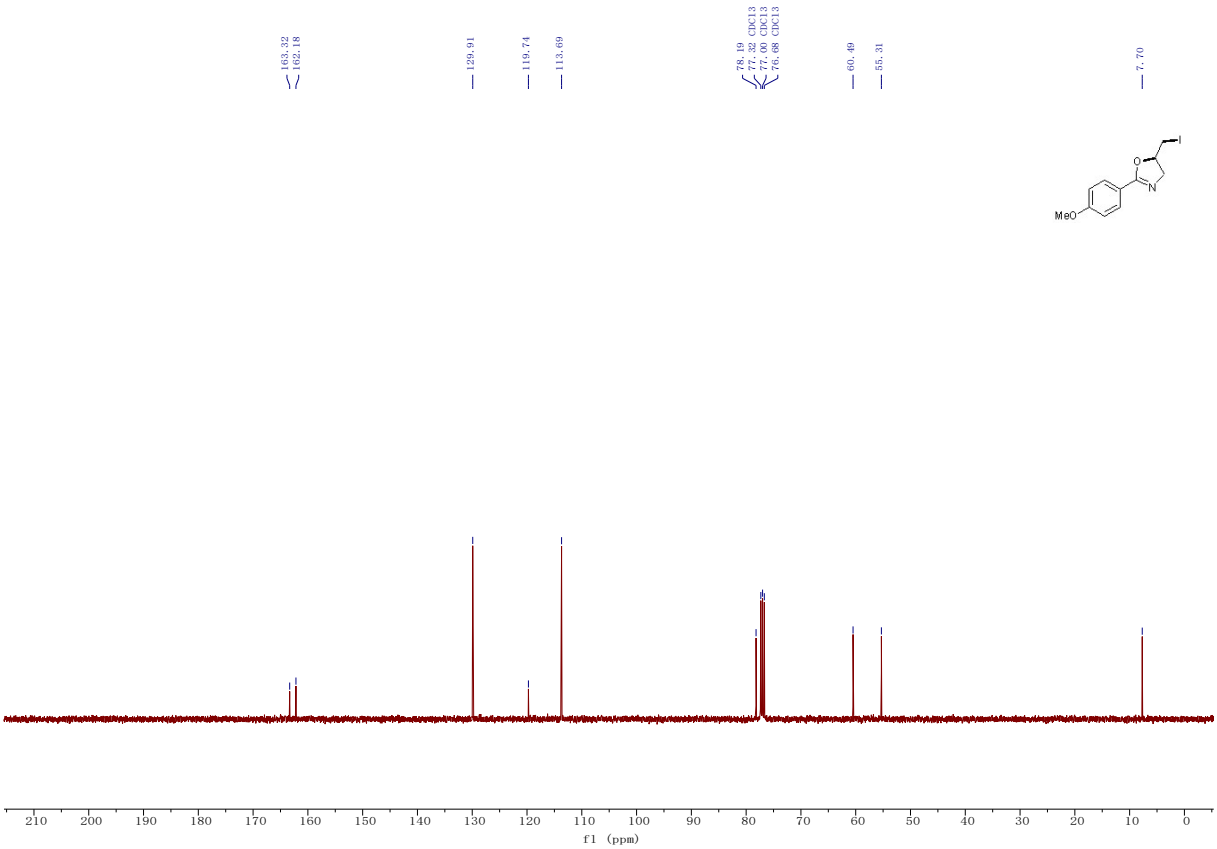
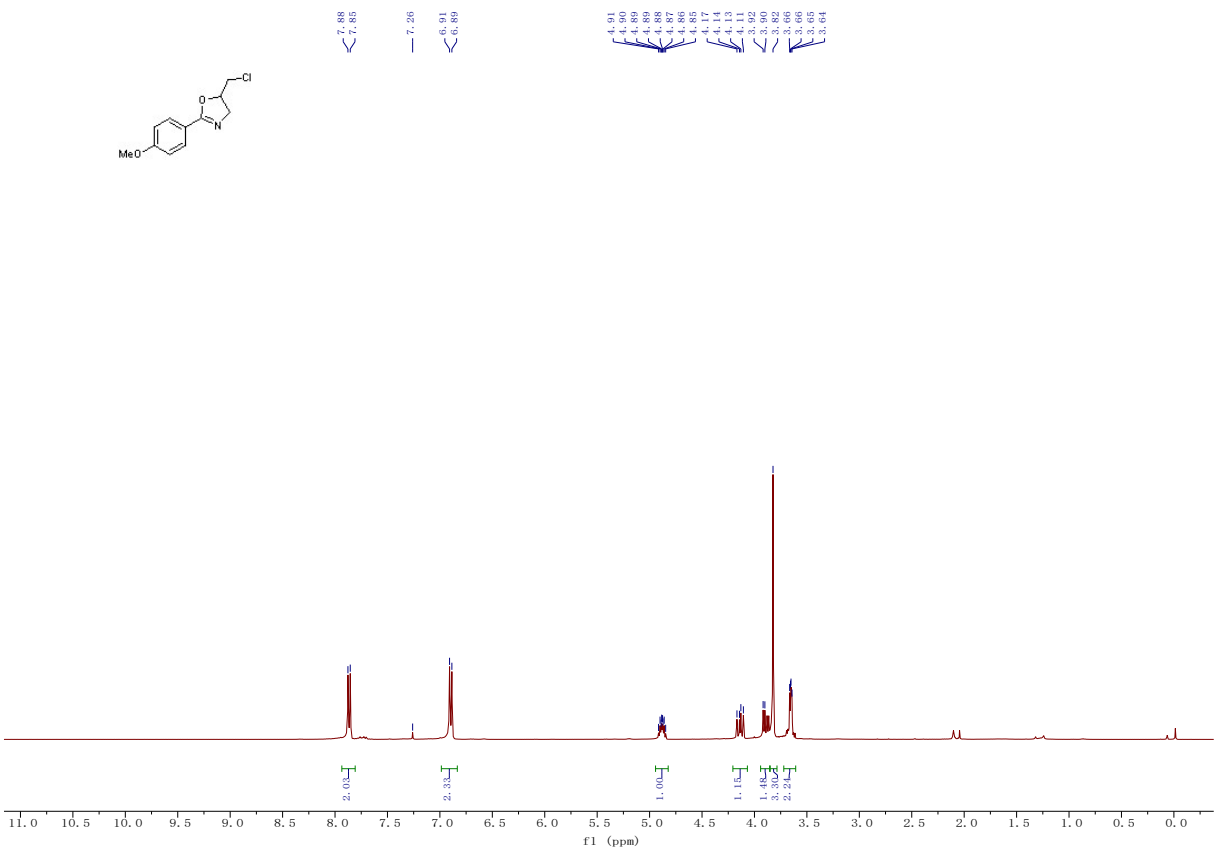
4.83
4.81
4.81
4.80
4.80
4.79
4.79
4.79
4.78
4.77
4.77
4.76
4.18
4.16
4.14
4.12
3.84
3.80
3.79
3.76
3.75
3.39
3.38
3.37
3.35
3.33
3.31
3.30
3.29
MeO
O
N
I
1.98
2.14
1.00
1.06
3.17
1.06
2.13
f1 (ppm)


163.32
162.18
129.91
119.74
113.69
78.19
77.32
77.00
76.68
CDCl3
CDCl3
CDCl3
60.49
55.31
7.70
MeO
f1 (ppm)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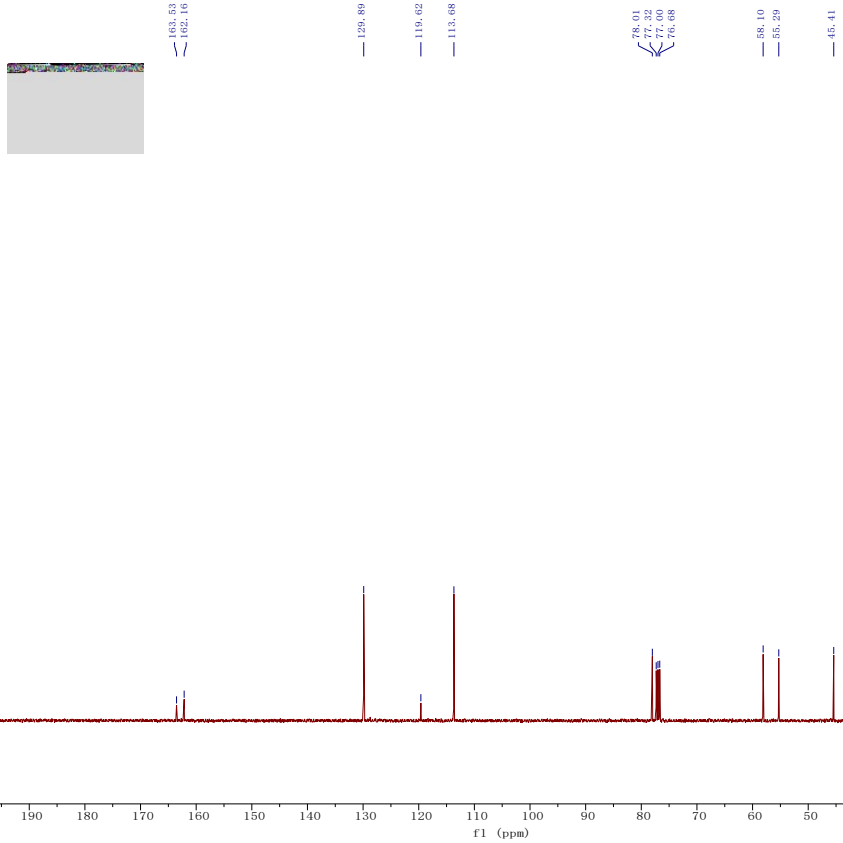
6.89
4.91
4.90
4.89
4.89
4.88
4.87
4.86
4.85
4.17
4.14
4.13
4.11
4.02
3.90
3.82
3.66
3.66
3.65
3.64
Cl
MeO
2.03
2.33
1.00
1.15
1.48
3.30
2.24
f1 (ppm)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162.16
129.89
119.62
113.68
78.01
77.32
77.00
76.68
58.10
55.29
45.41
f1 (ppm)


7.88
7.86
7.26
6.92
6.90
4.93
4.92
4.92
4.91
4.90
4.90
4.89
4.89
4.88
4.88
4.88
4.86
4.18
4.16
4.15
4.12
3.90
3.89
3.86
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3.55
3.53
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3.50
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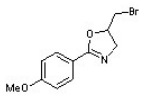
3.48
3.46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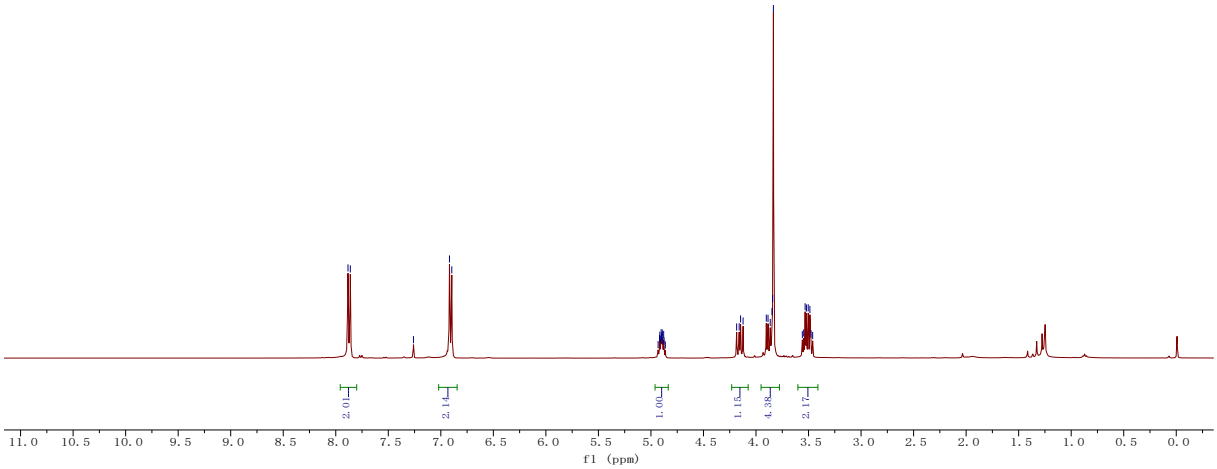
N
MeO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2.14
1.00
1.15
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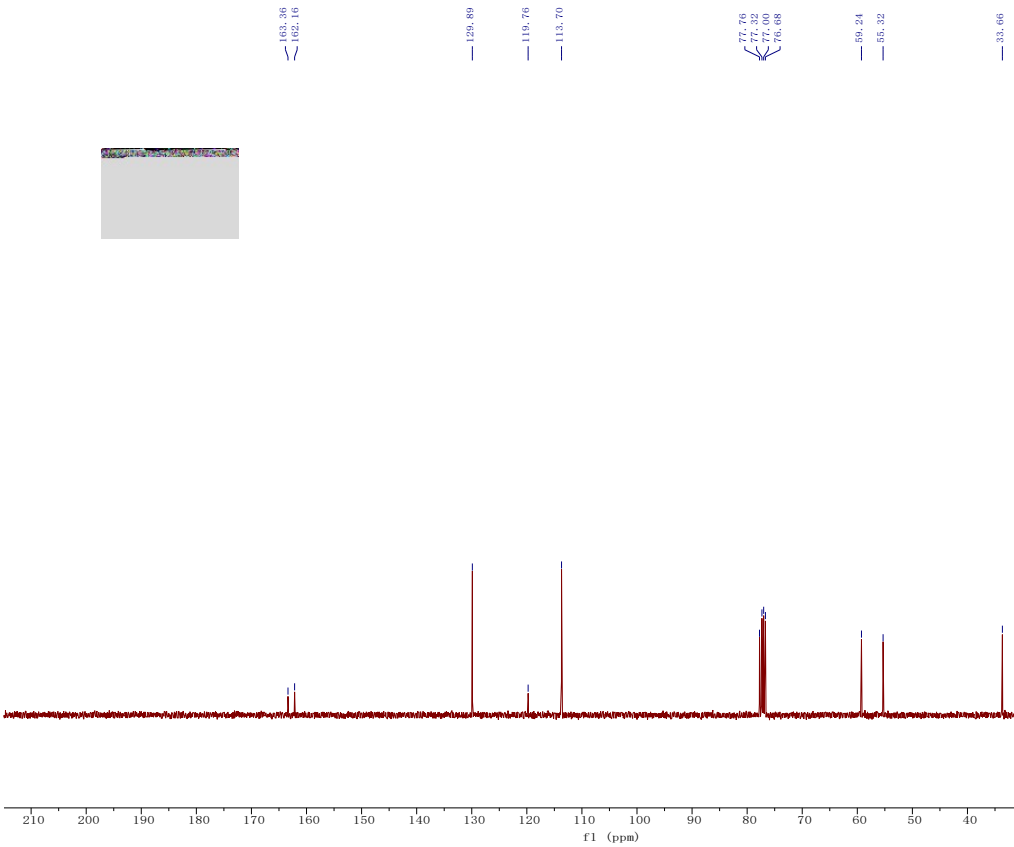
2.17
f1 (ppm)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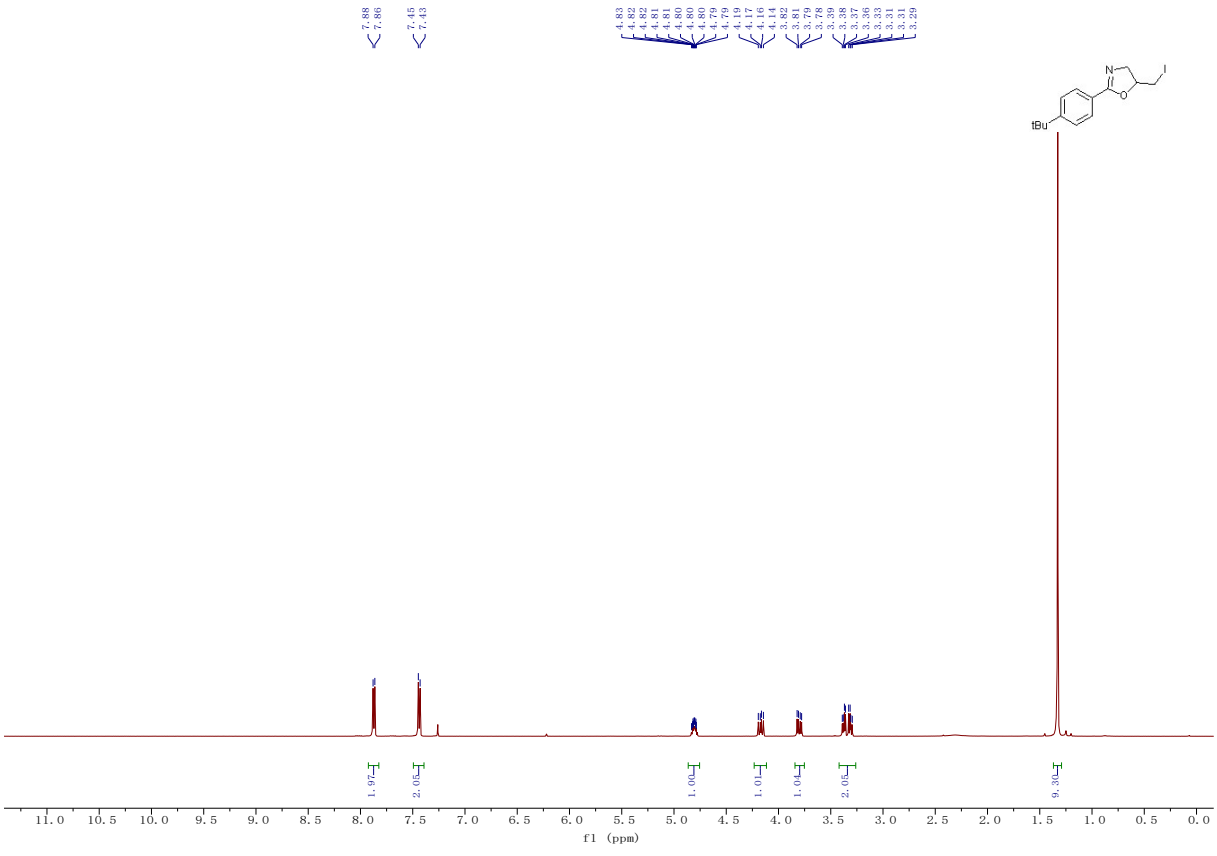
77.00
76.68
69.24
55.32
33.66
f1 (ppm)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7.86
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4.80
4.79
4.79
4.19
4.17
4.16
4.14
3.82
3.81
3.79
3.78
3.39
3.38
3.37
3.36
3.33
3.31
3.31
3.29
N
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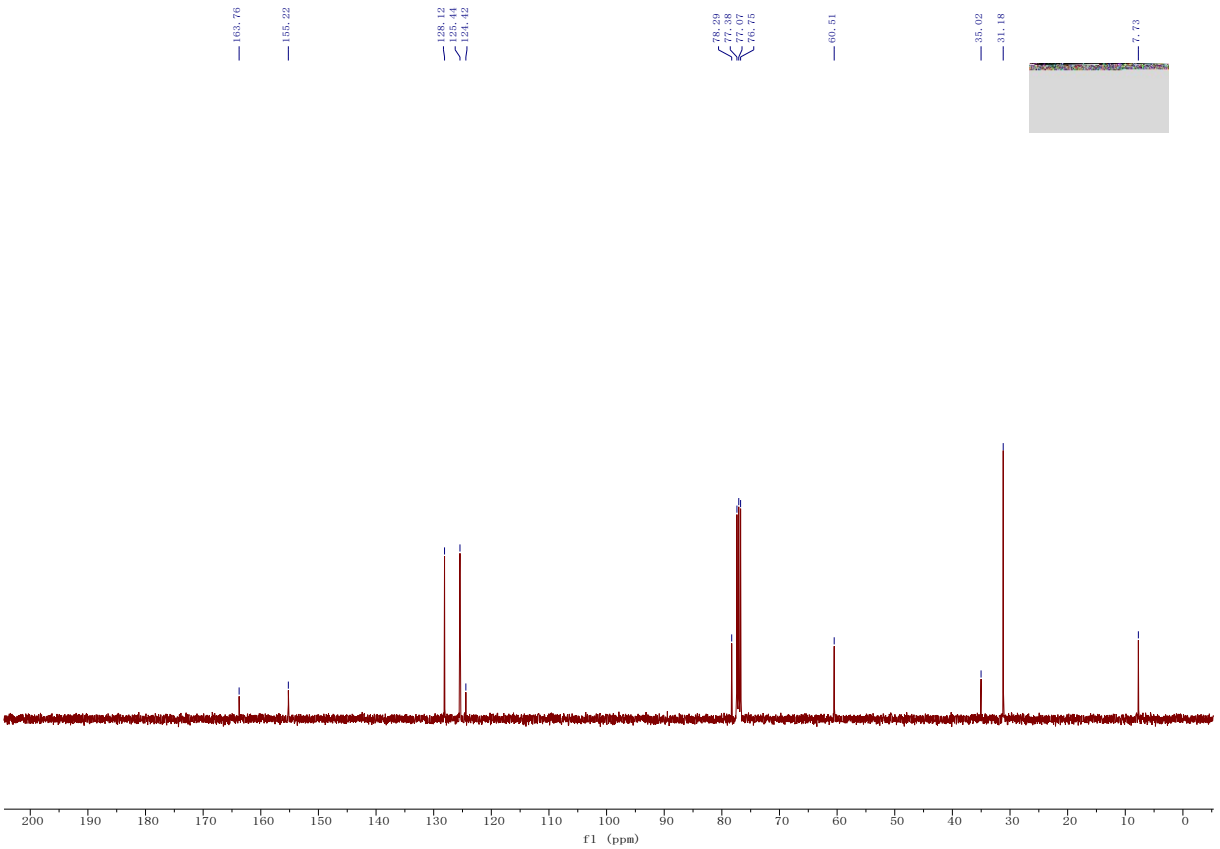
I
tBu
1.97
2.05
1.00
1.01
1.04
2.05
9.30
f1 (ppm)


163.76
155.22
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125.44
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77.07
76.75
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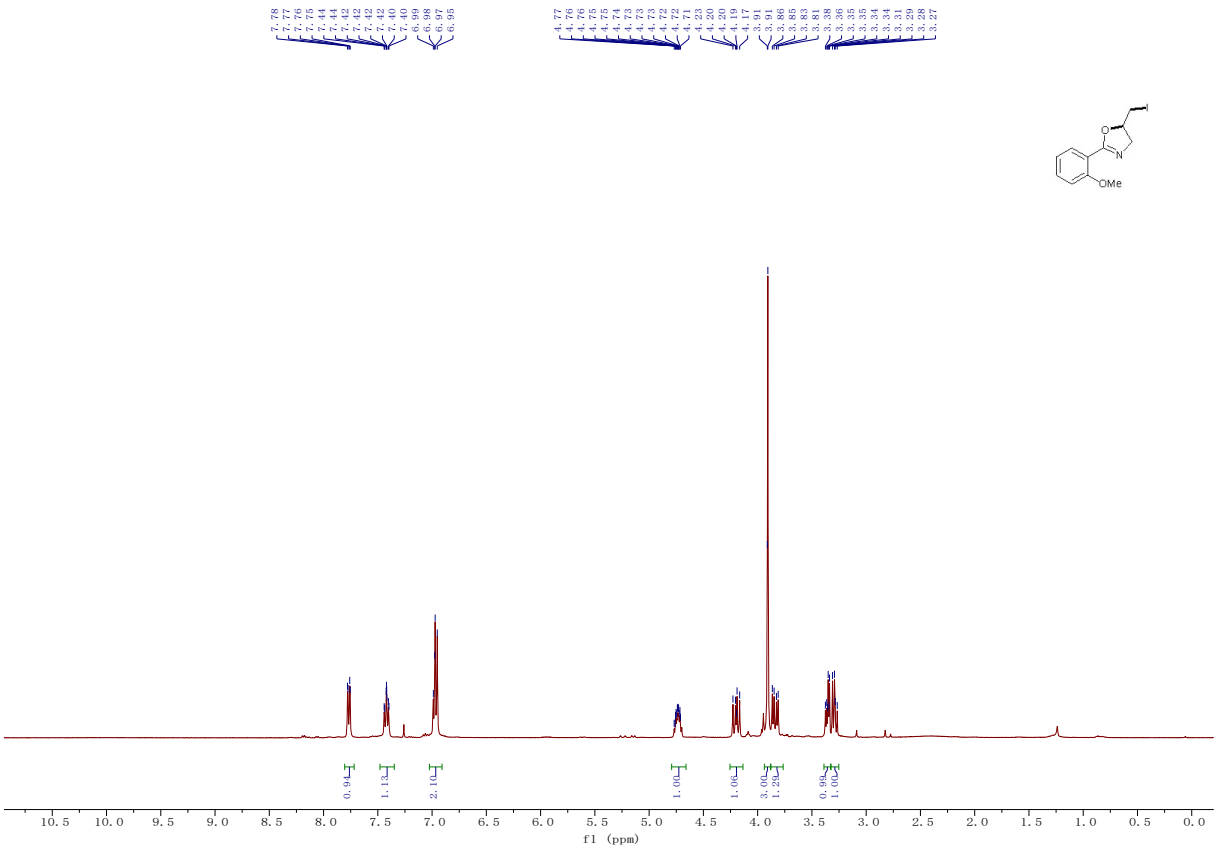
35.02
31.18
7.73
f1 (ppm)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7.42
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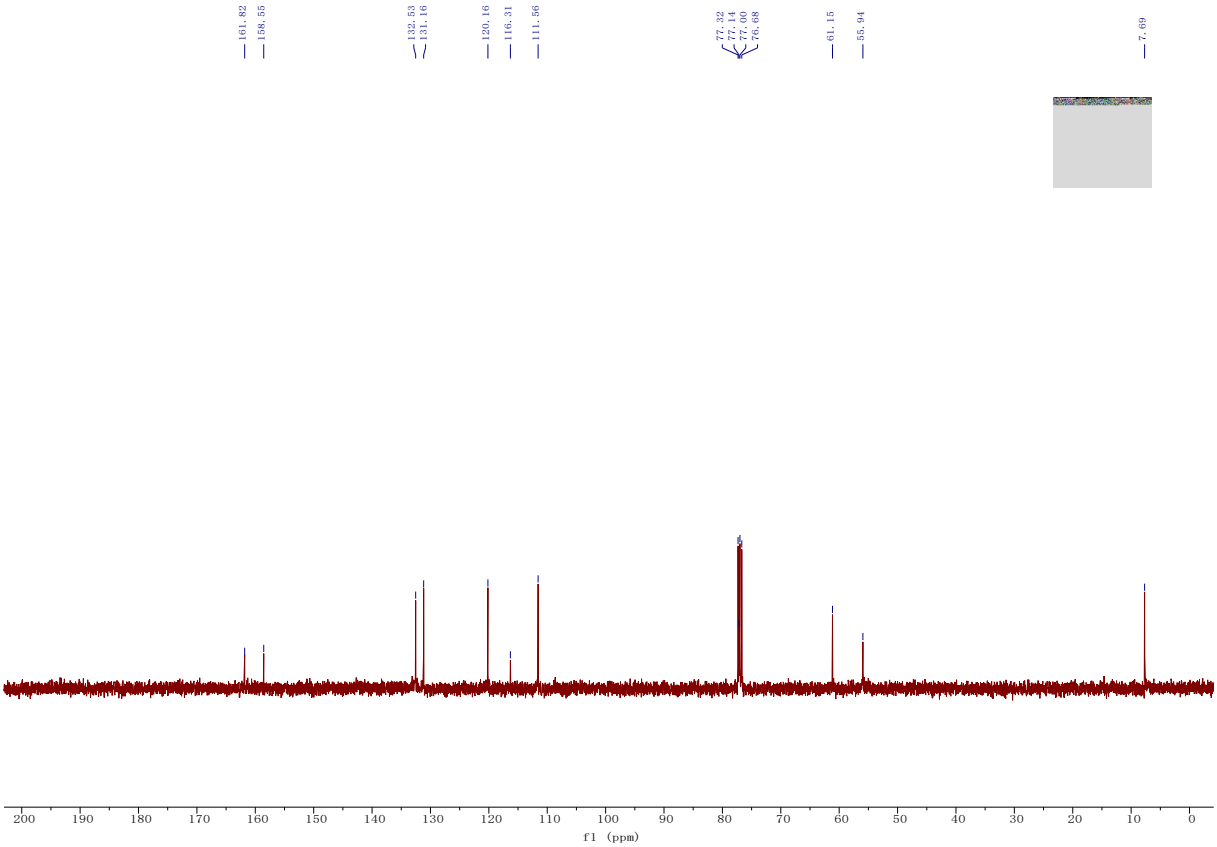
6.95
4.77
4.76
4.76
4.75
4.74
4.73
4.73
4.73
4.72
4.72
4.71
4.23
4.20
4.20
4.19
4.17
3.91
3.91
3.88
3.85
3.83
3.81
3.38
3.36
3.35
3.35
3.34
3.34
3.31
3.29
3.28
3.27
O
N
OMe
0.94
1.13
2.10
1.00
1.06
3.01
1.29
0.99
1.00
f1 (ppm)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158.55
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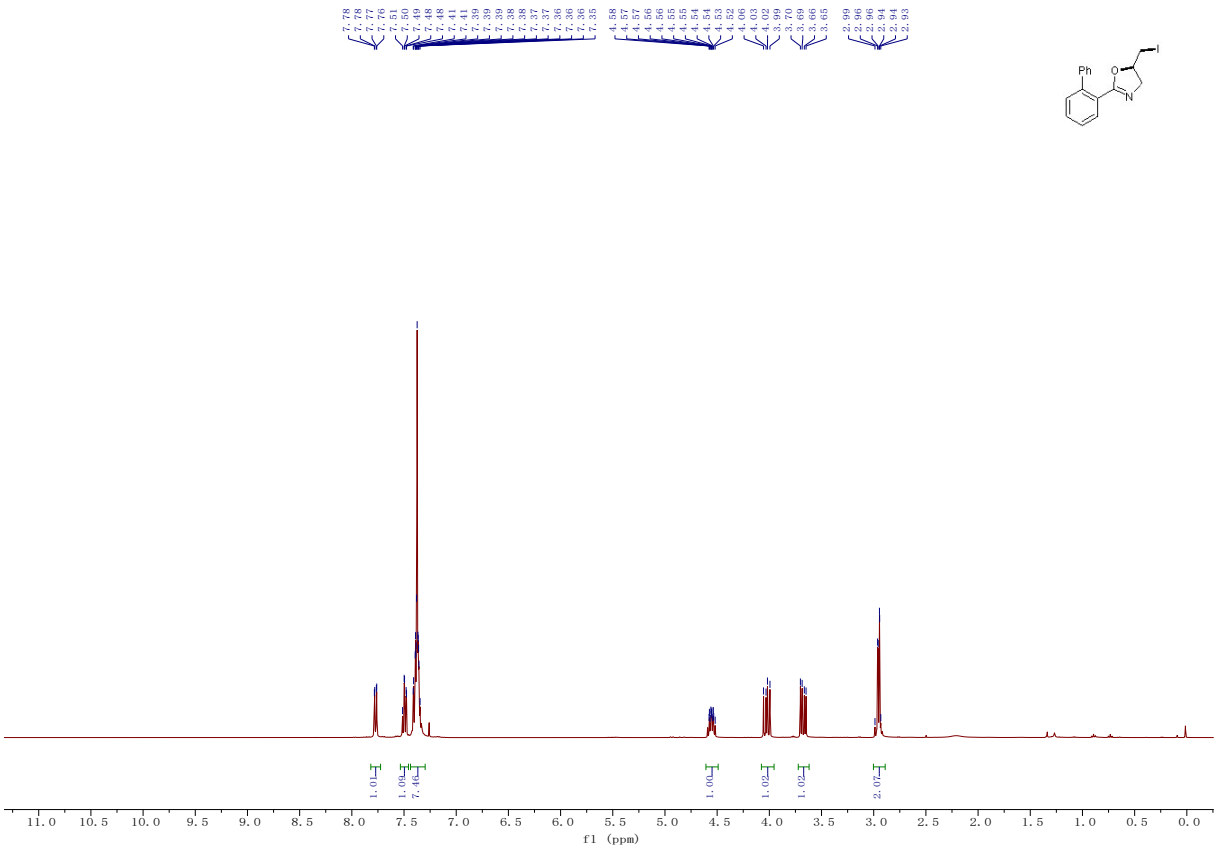
131.16
120.16
116.31
111.56
77.32
77.14
77.00
76.68
61.15
55.94
7.69
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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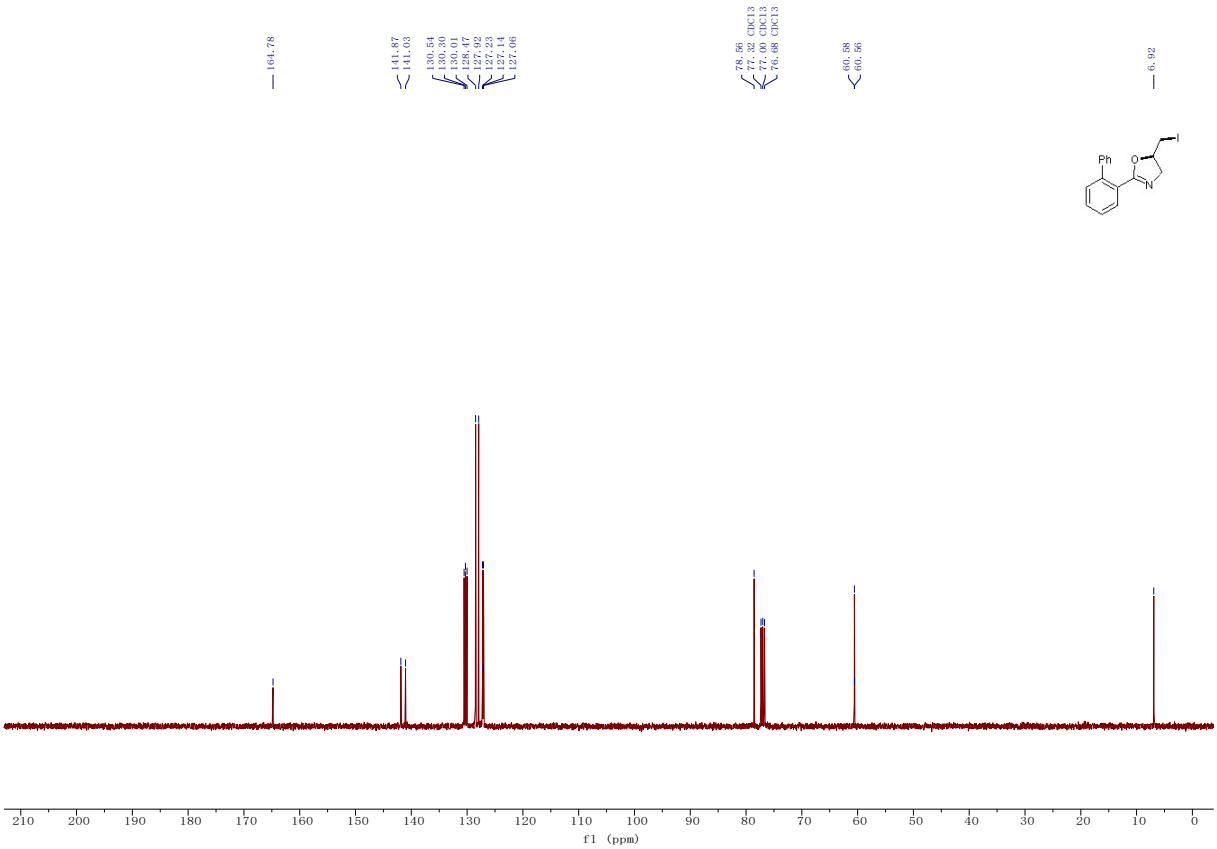
7.39
7.39
7.38
7.38
7.37
7.37
7.36
7.36
7.36
7.35
4.58
4.57
4.57
4.56
4.56
4.55
4.55
4.54
4.54
4.53
4.52
4.06
4.03
4.02
3.99
3.70
3.69
3.66
3.65
2.99
2.96
2.96
2.94
2.94
2.93
Ph
O
N
I
1.01
1.09
7.46
1.00
1.02
1.02
2.07
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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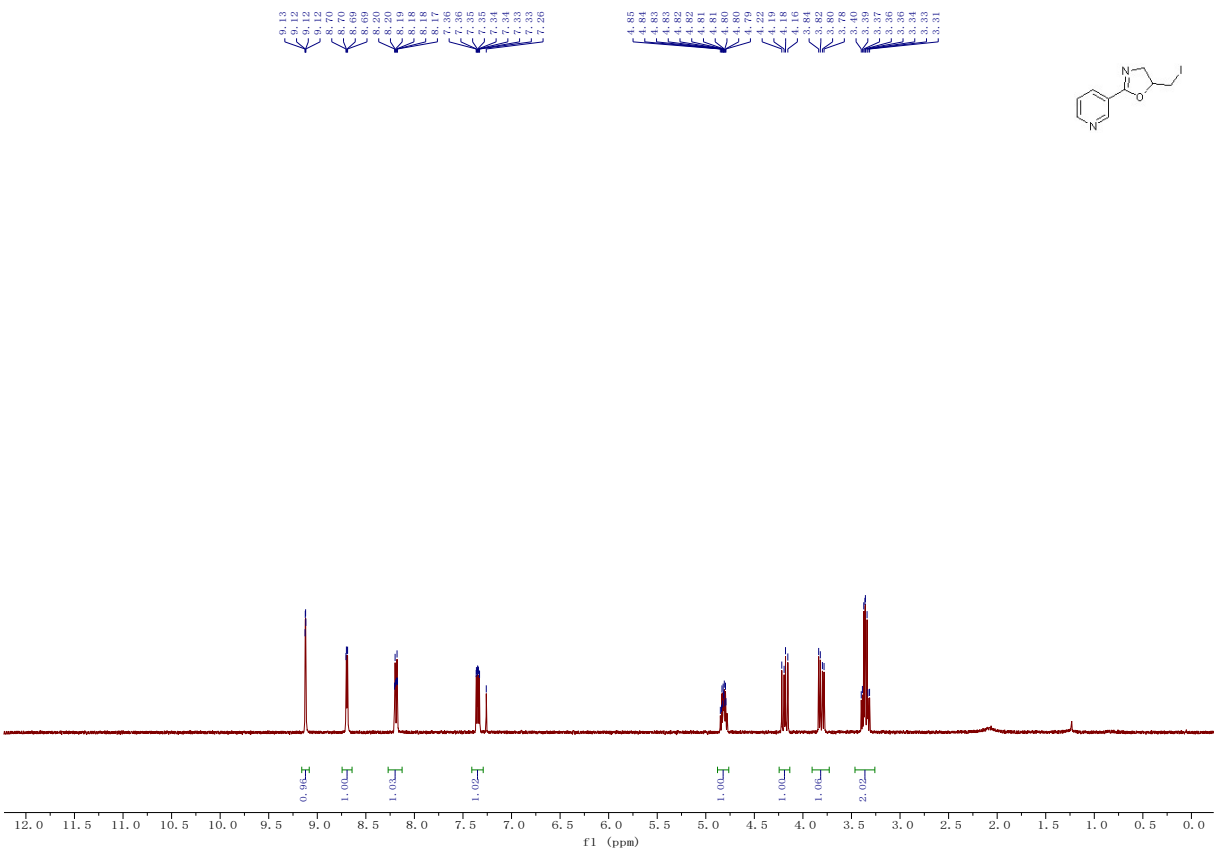
127.92
127.23
127.14
127.06
78.56
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
60.58
60.56
6.92
Ph
O
N
I
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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9.12
9.12
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8.69
8.69
8.20
8.20
8.19
8.18
8.18
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7.36
7.36
7.35
7.35
7.34
7.34
7.33
7.33
7.26
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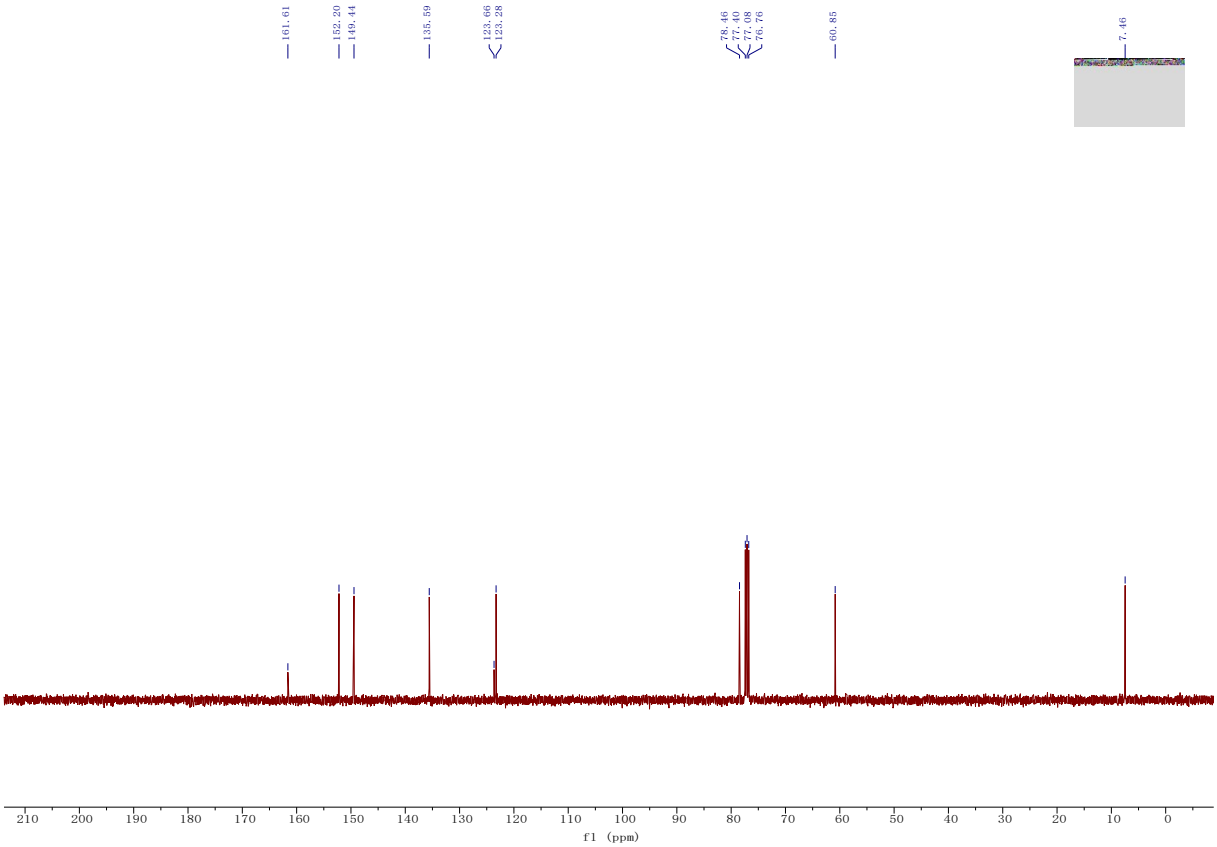
4.84
4.83
4.83
4.82
4.82
4.81
4.81
4.80
4.79
4.22
4.19
4.18
4.16
3.84
3.82
3.80
3.78
3.40
3.39
3.37
3.36
3.36
3.34
3.33
3.31
N
O
I
N
0.96
1.00
1.03
1.02
1.00
1.00
1.06
2.02
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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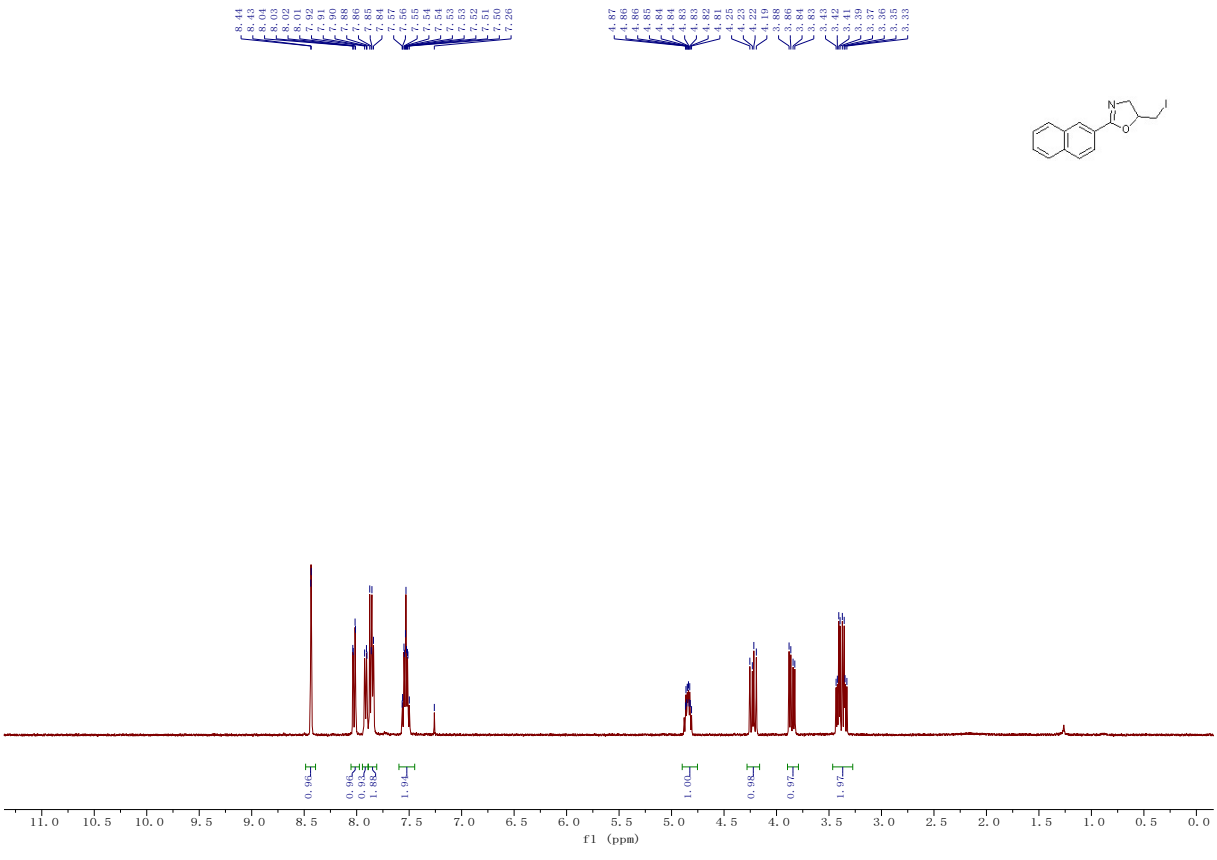
149.44
135.59
123.66
123.28
78.46
77.40
77.08
76.76
60.85
7.46
f1 (ppm)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8.02
8.01
7.92
7.91
7.90
7.88
7.86
7.85
7.84
7.57
7.56
7.55
7.54
7.54
7.53
7.53
7.52
7.51
7.50
7.26
4.87
4.86
4.86
4.85
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4.84
4.83
4.83
4.82
4.81
4.25
4.23
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4.19
3.88
3.86
3.84
3.83
3.43
3.42
3.41
3.39
3.37
3.36
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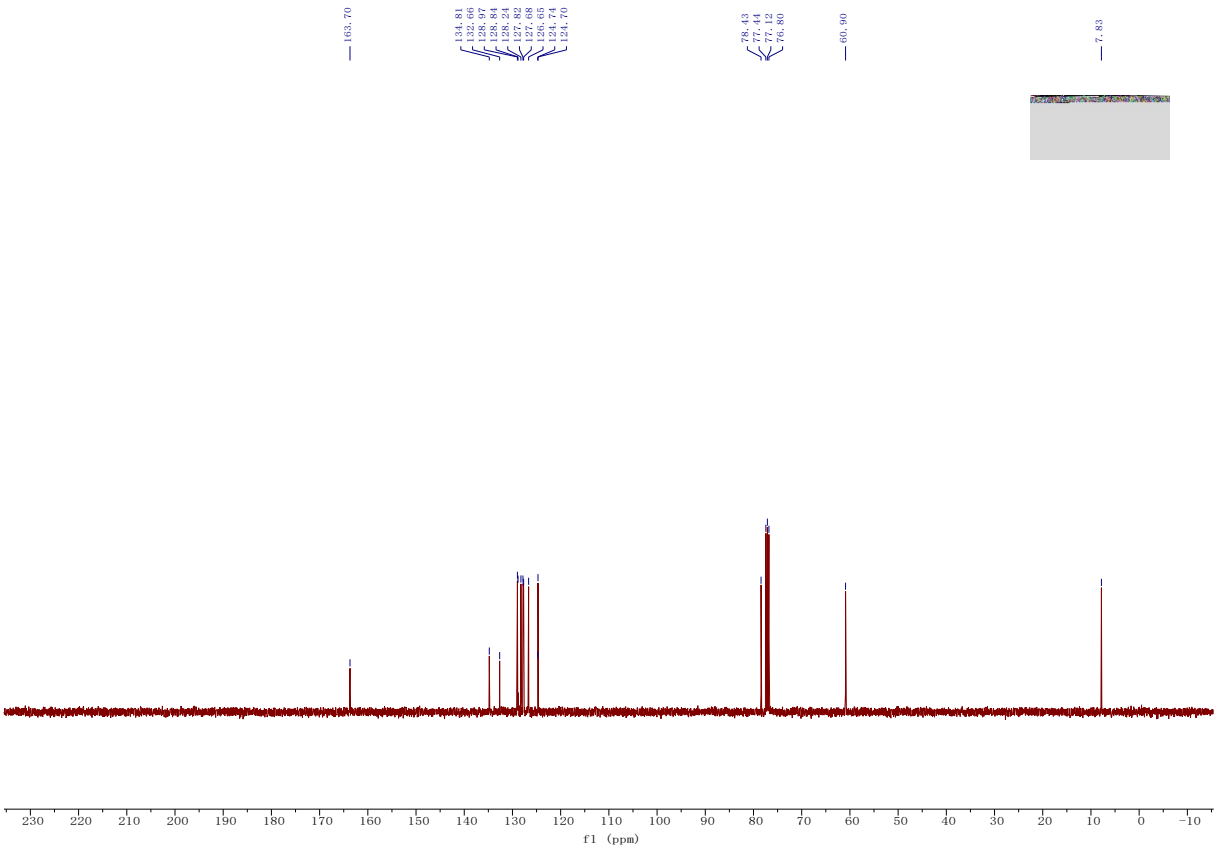
0.96
0.92
1.82
1.94
1.00
0.98
0.97
1.97
f1 (ppm)


163.70
134.81
132.66
128.97
128.84
128.24
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127.68
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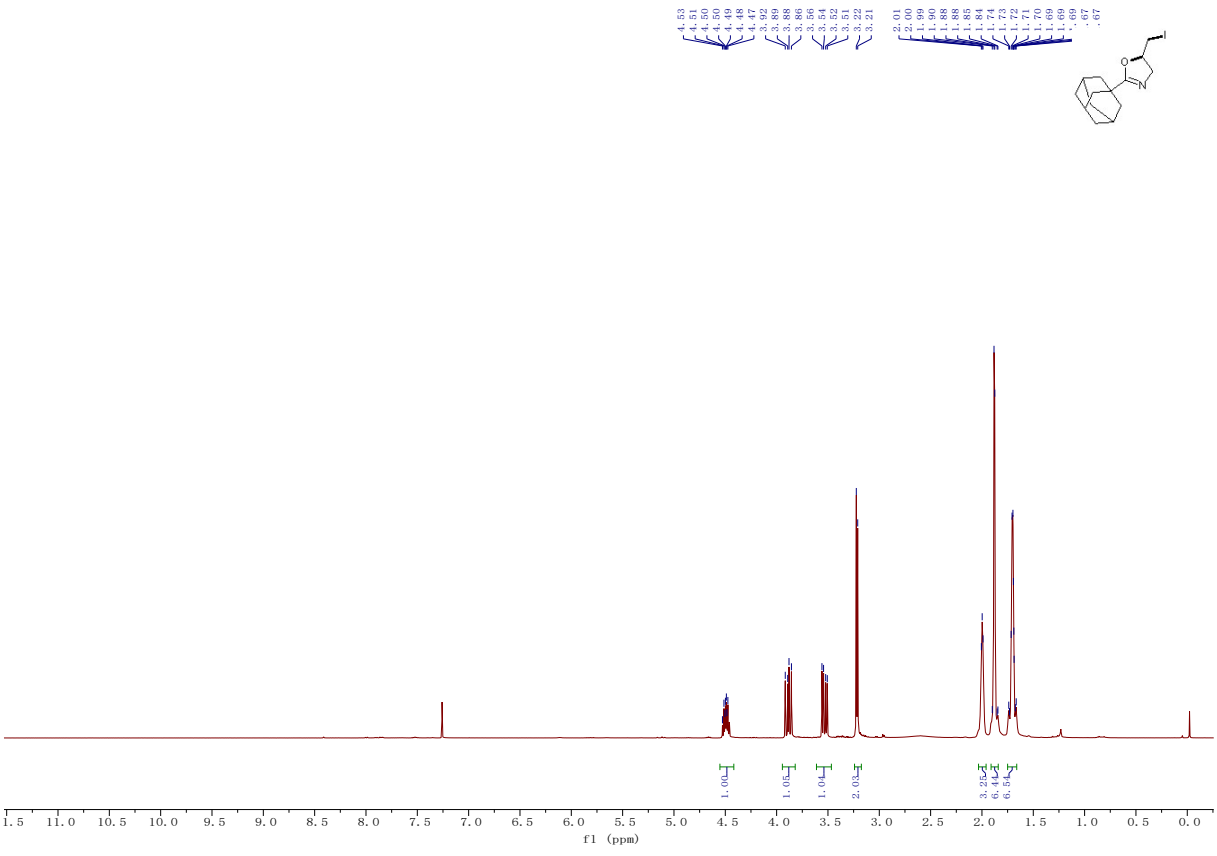
124.74
124.70
78.43
77.44
77.12
76.80
60.90
7.83
f1 (ppm)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3.56
3.54
3.52
3.51
3.22
3.21
2.01
2.00
1.99
1.90
1.88
1.88
1.85
1.84
1.74
1.73
1.72
1.71
1.70
1.69
1.69
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O
N
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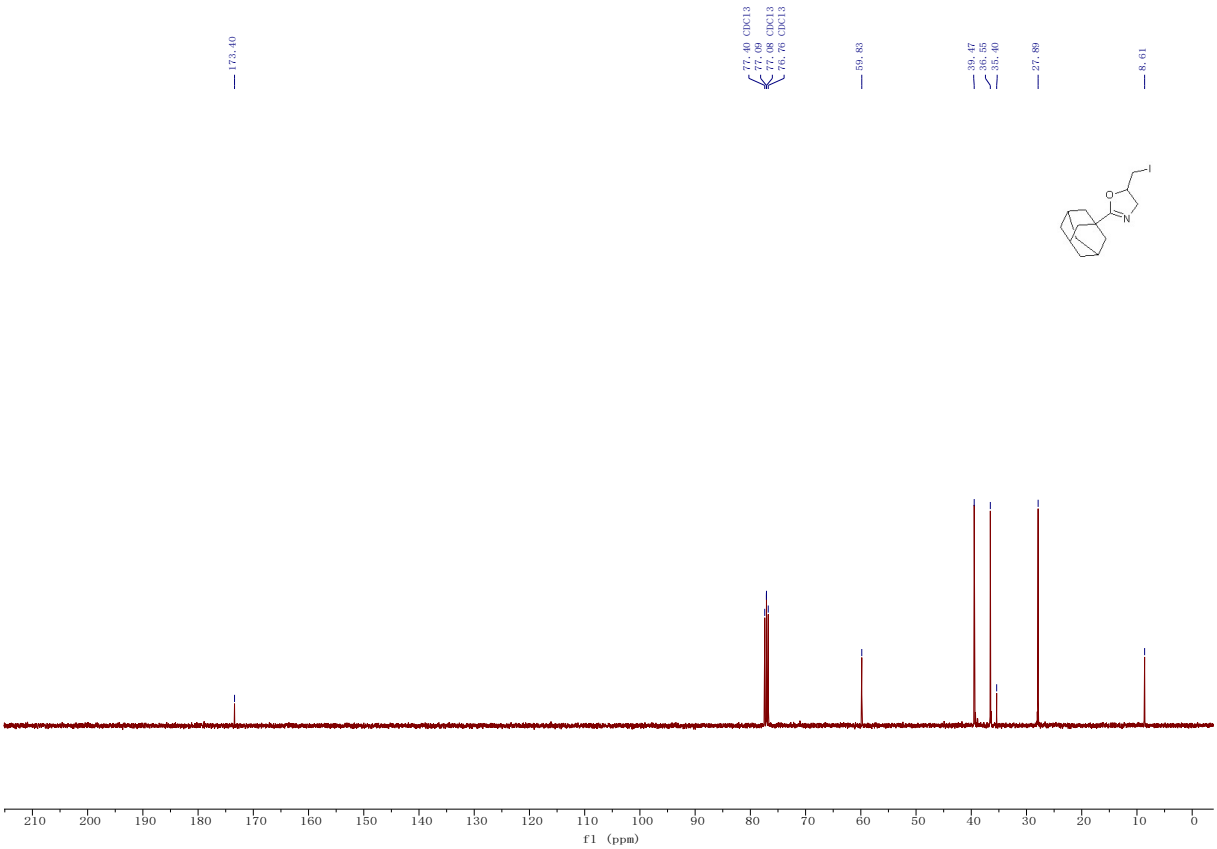
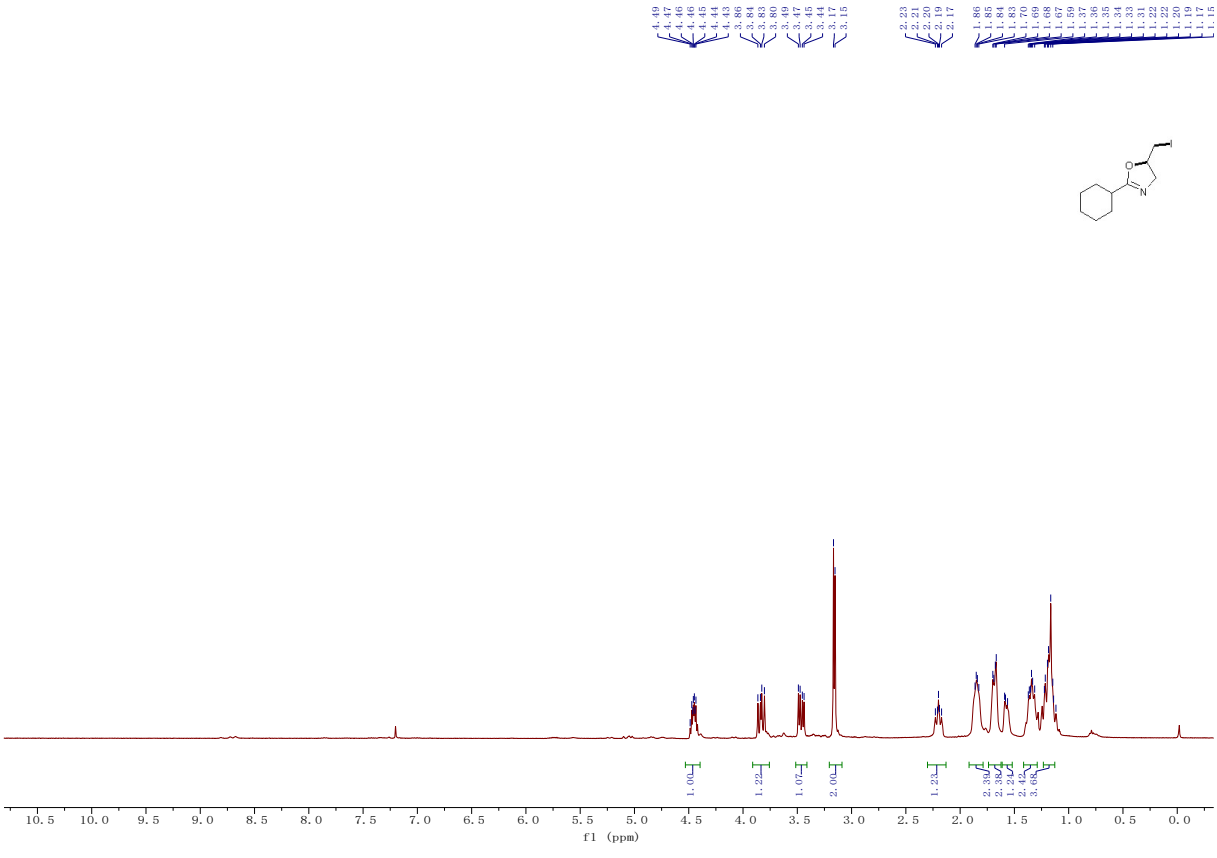
1.05
1.04
2.03
3.25
6.44
6.54
f1 (ppm)


173.40
77.40 CDCl3
77.09
77.08 CDCl3
76.76 CDCl3
59.83
39.47
36.55
35.40
27.89
8.61
O
N
I
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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3.80
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3.47
3.45
3.44
3.17
3.15
2.23
2.21
2.20
2.19
2.17
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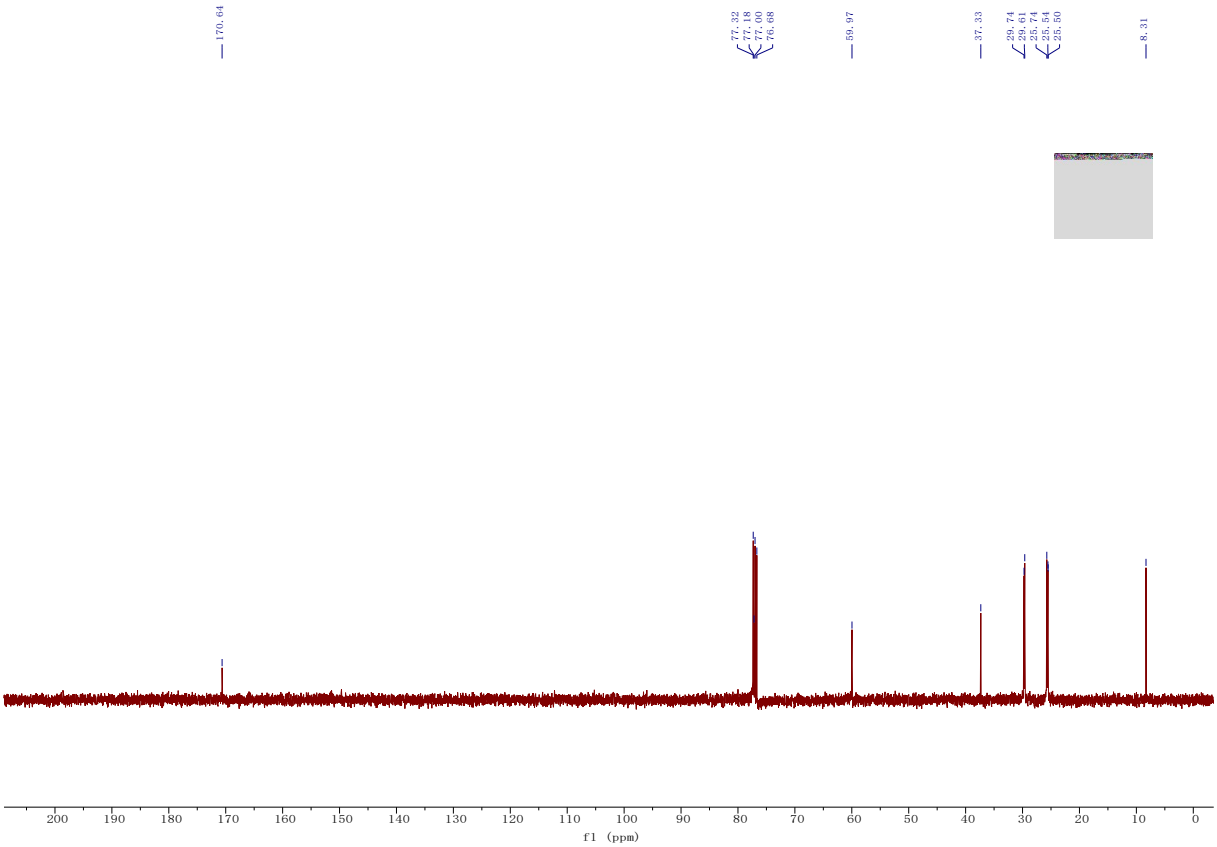
1.85
1.84
1.83
1.70
1.69
1.68
1.67
1.59
1.37
1.36
1.35
1.34
1.33
1.31
1.22
1.20
1.19
1.17
1.15
O
N
I
1.00
1.22
1.07
2.01
1.23
2.39
2.38
1.24
2.42
3.68
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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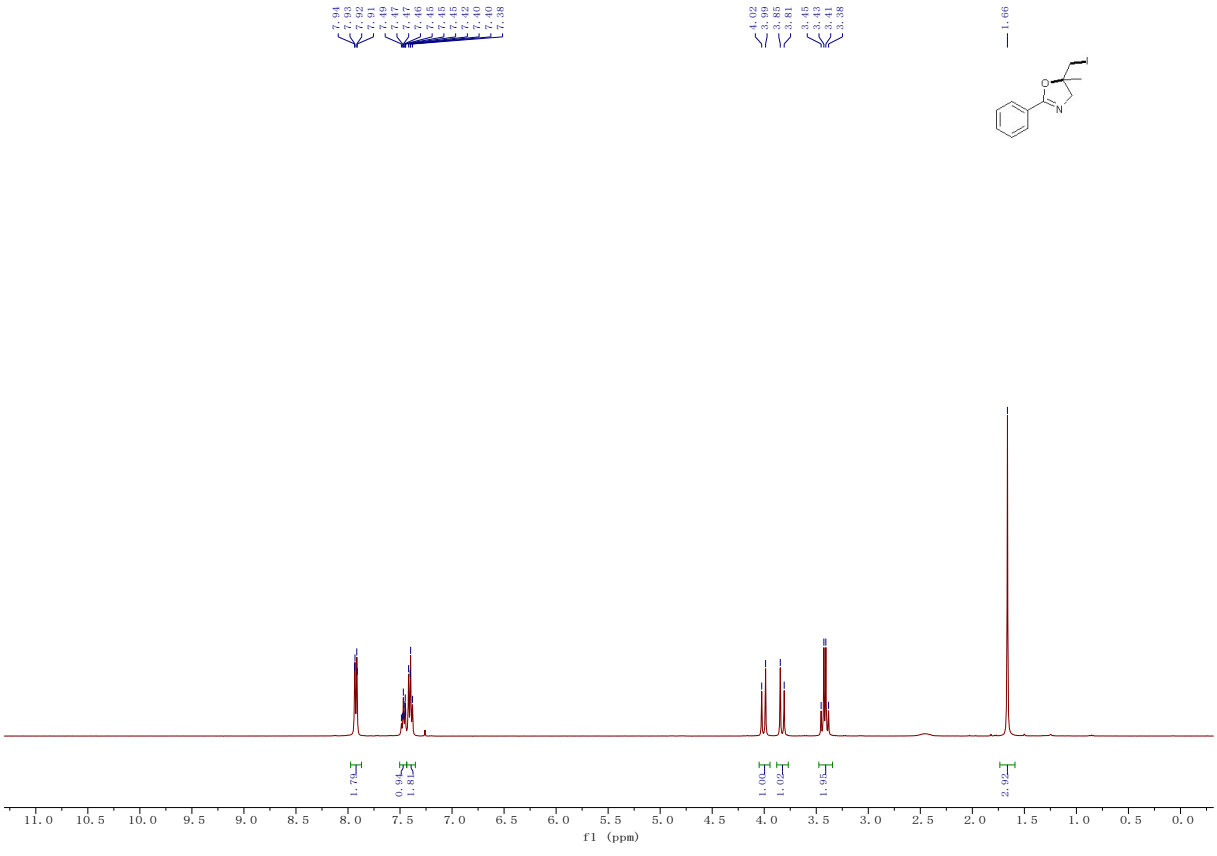
29.74
29.61
25.74
25.54
25.50
8.31
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.45
7.42
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7.40
7.38
4.02
3.99
3.85
3.81
3.45
3.43
3.41
3.38
1.66
1.79
0.94
1.81
1.00
1.02
1.95
2.92
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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3.5
3.0
2.5
2.0
1.5
1.0
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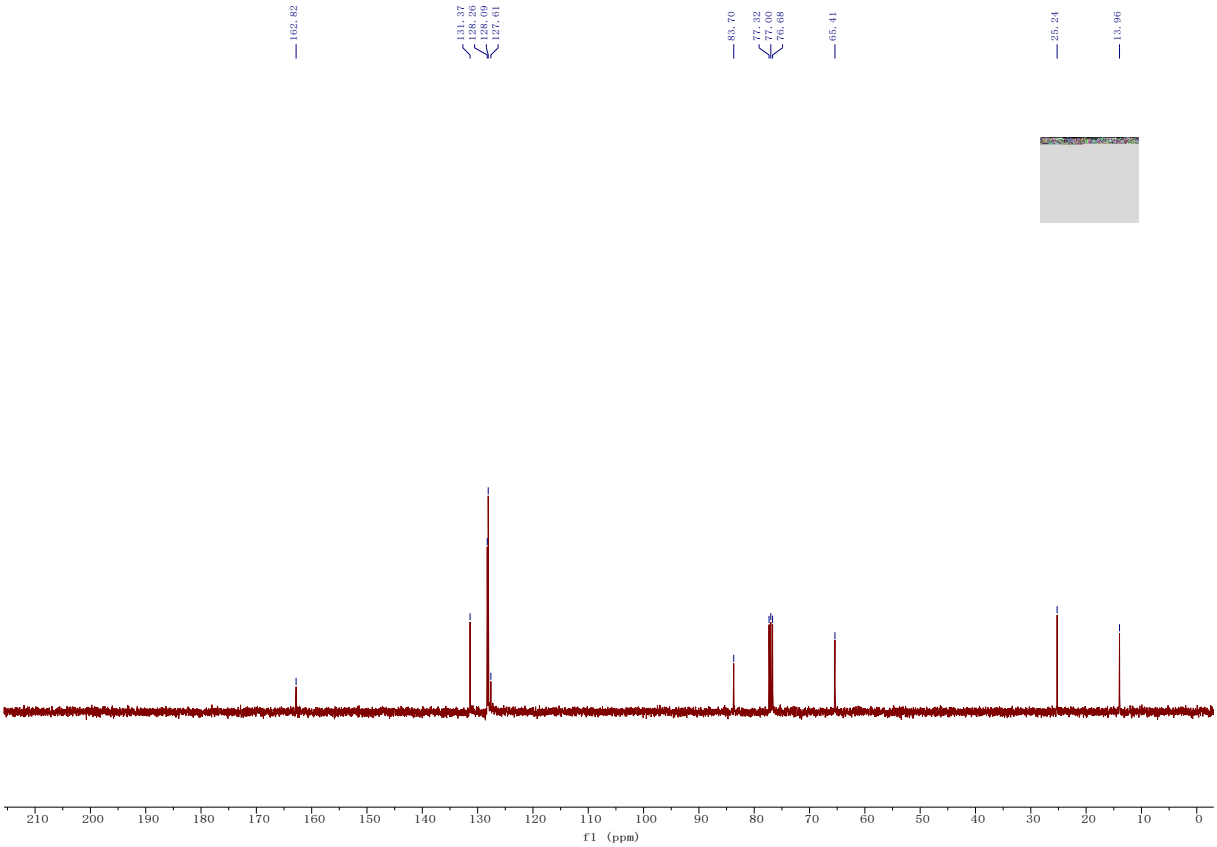
0.0
f1 (ppm)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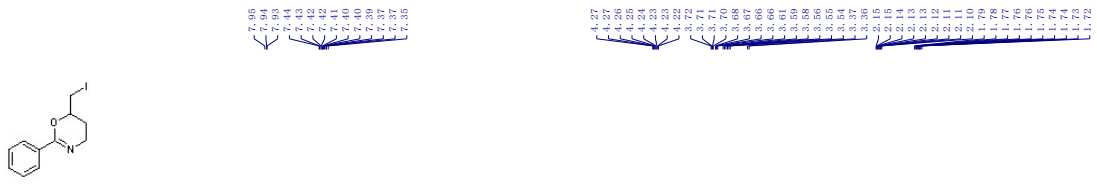
77.00
76.68
65.41
25.24
13.96
f1 (ppm)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4.26
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4.24
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4.23
4.22
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3.71
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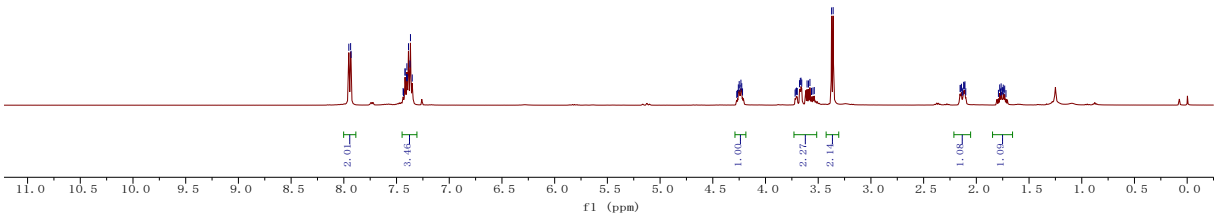
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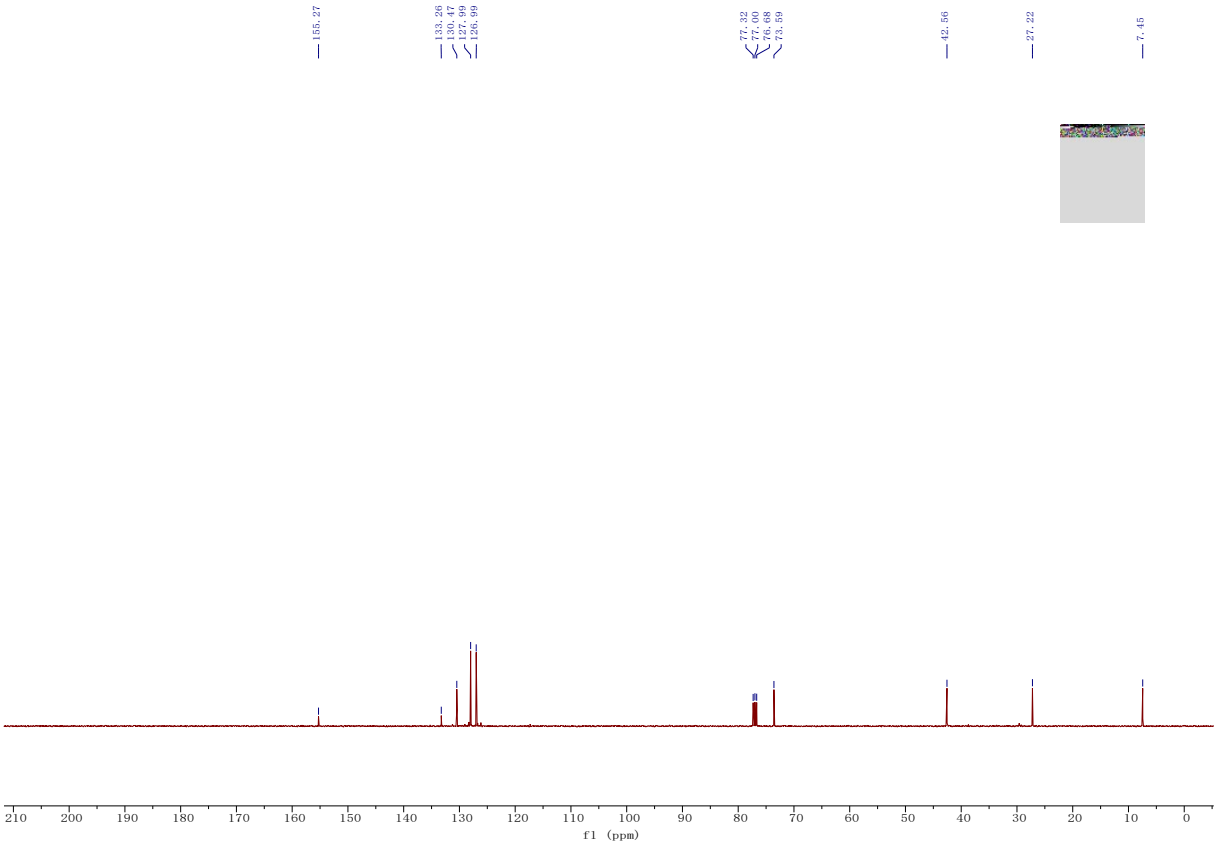
3.46
1.00
2.27
2.14
1.08
1.09
f1 (ppm)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133.26
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127.99
126.99
77.32
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76.68
73.59
42.56
27.22
7.45
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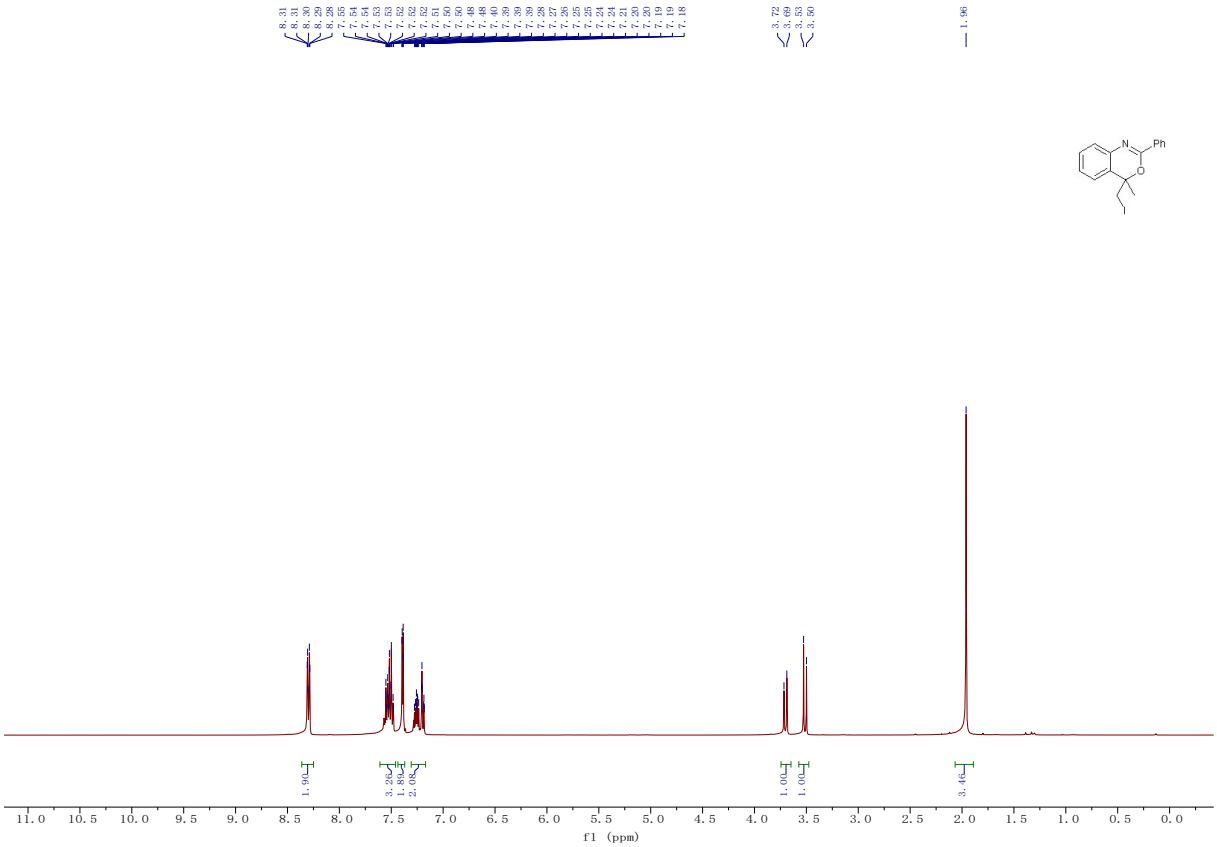
f1 (ppm)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7.18
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3.69
3.53
3.50
1.96
Ph
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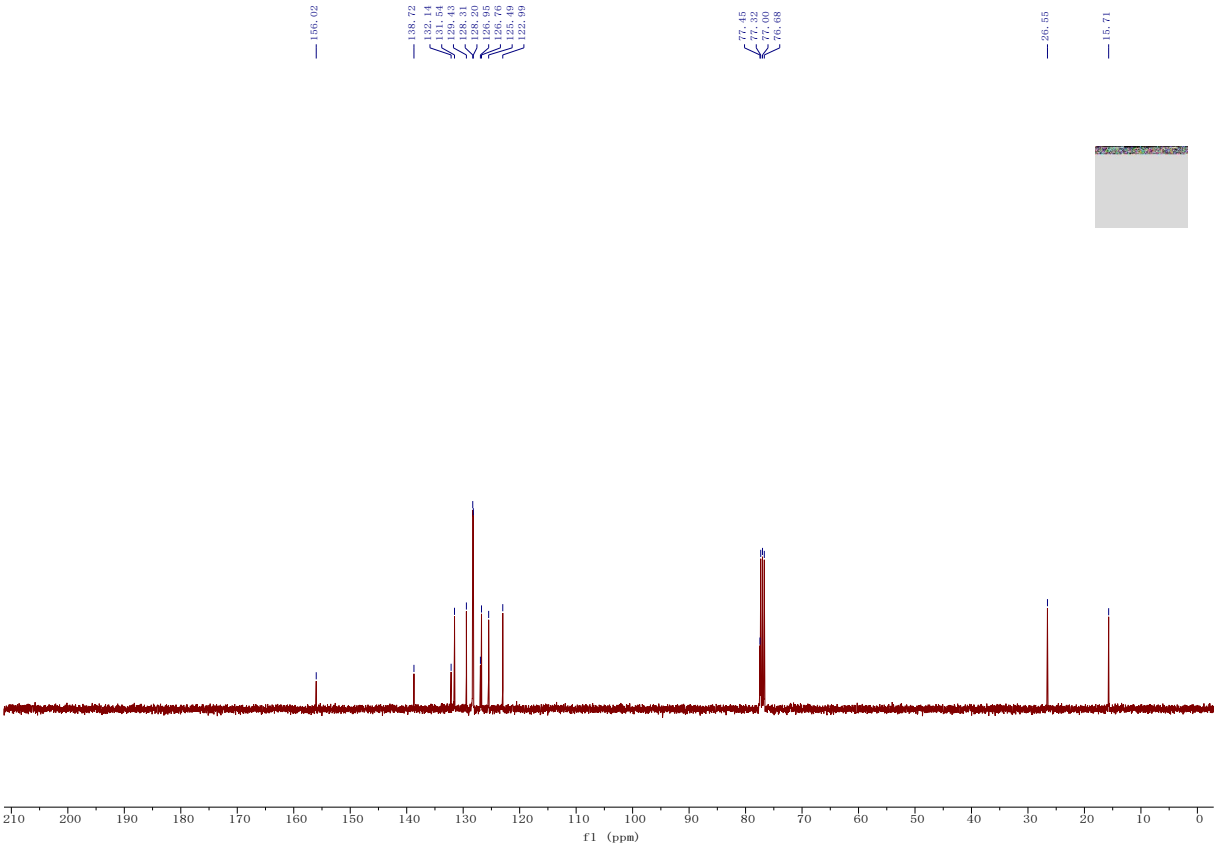
O
1.90
3.26
1.88
2.08
1.00
1.00
3.46
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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125.49
122.99
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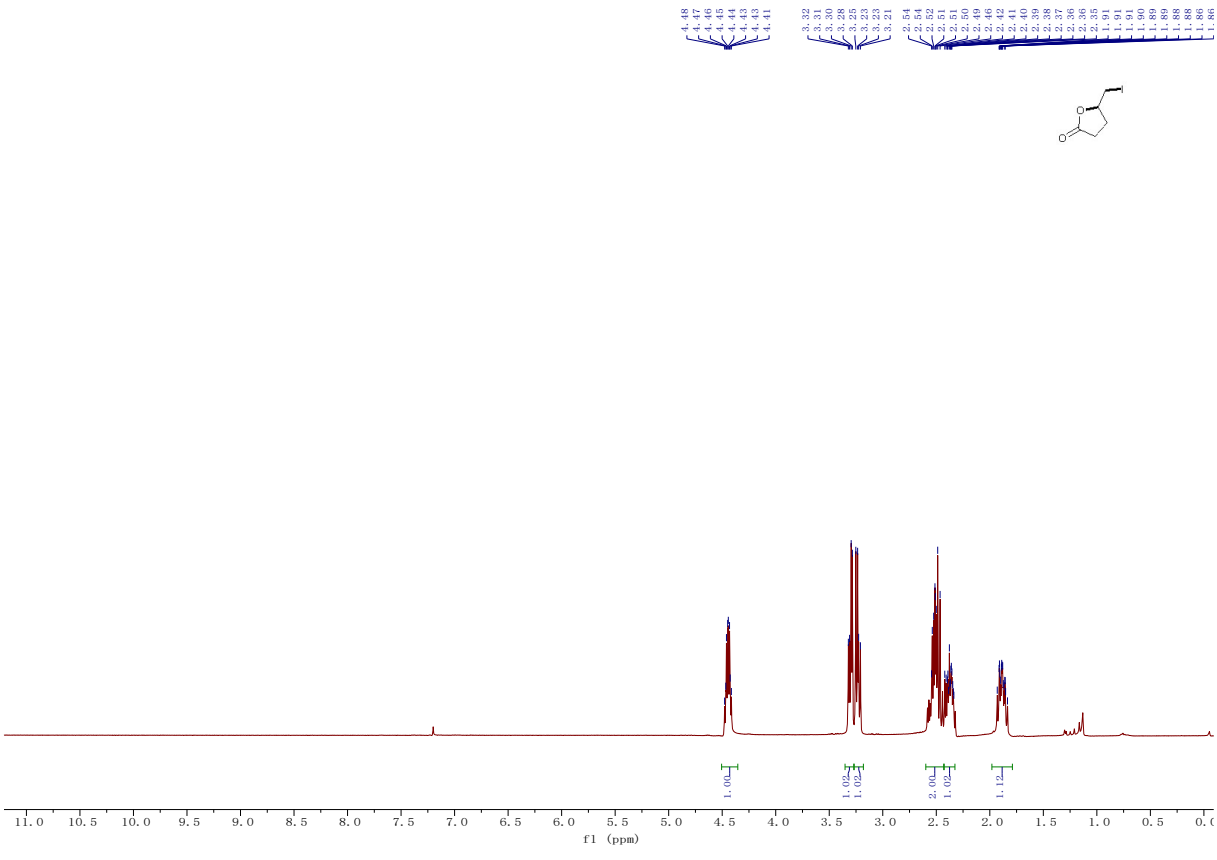
77.32
77.00
76.68
26.55
15.71
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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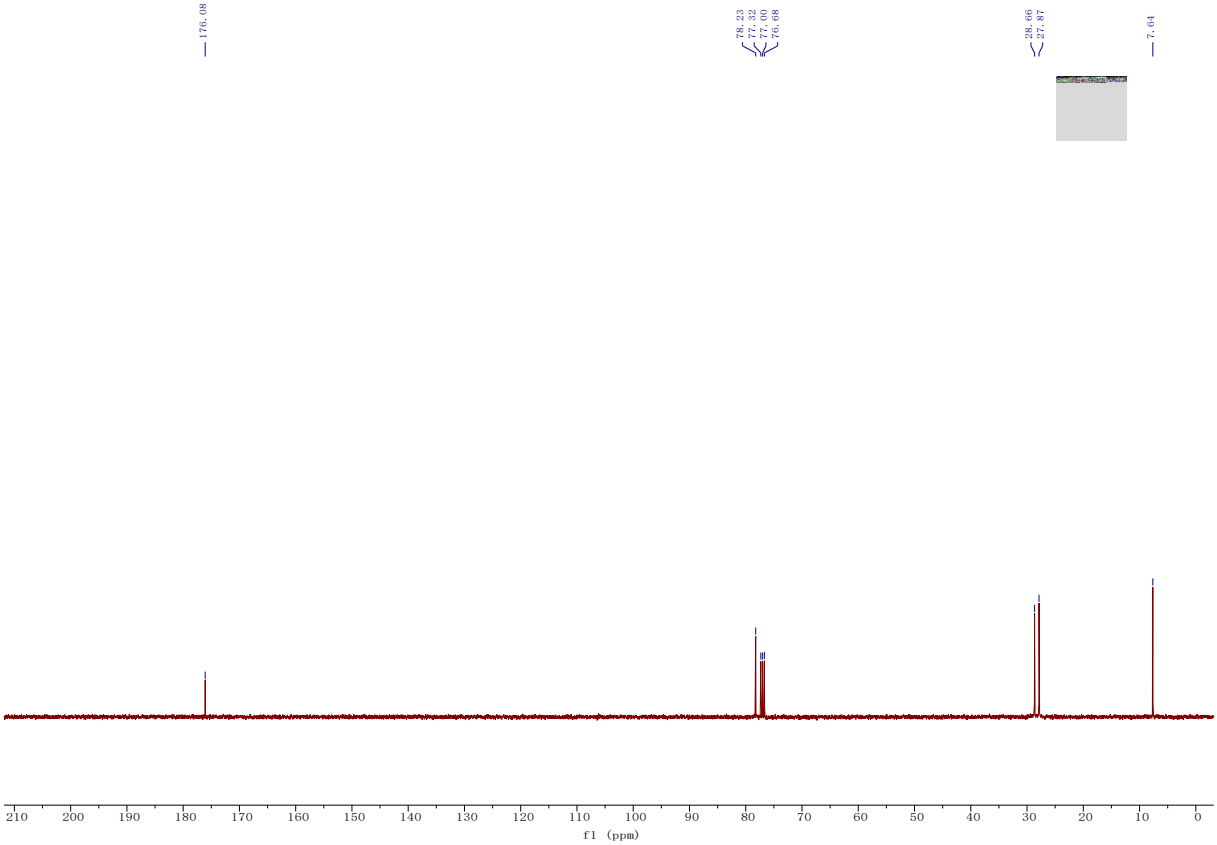
3.23
3.23
3.21
2.54
2.54
2.52
2.51
2.51
2.50
2.49
2.46
2.42
2.41
2.40
2.39
2.38
2.37
2.36
2.36
2.35
1.91
1.91
1.91
1.91
1.90
1.89
1.89
1.88
1.88
1.88
1.86
1.86
1.00
1.02
1.02
2.01
1.02
1.12
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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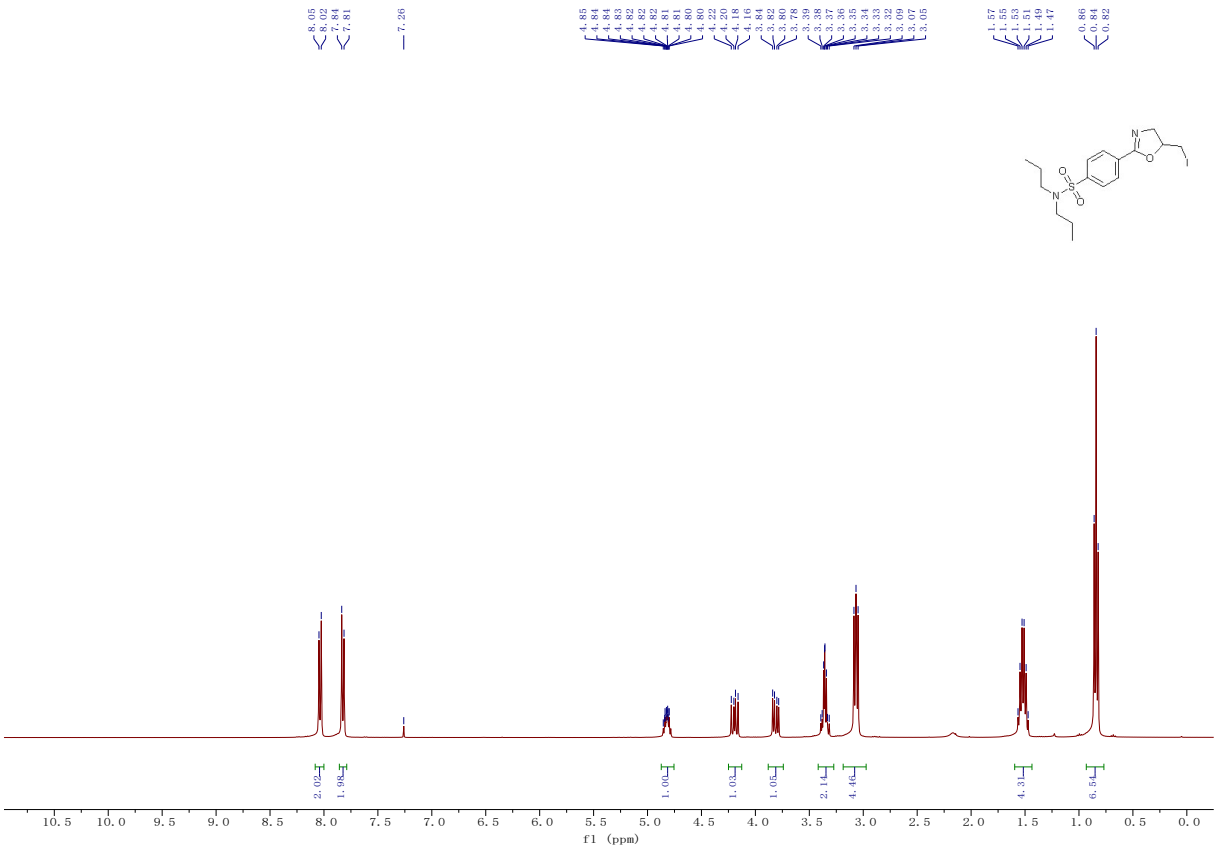
78.23
77.32
77.00
76.68
28.66
27.87
7.64
f1 (ppm)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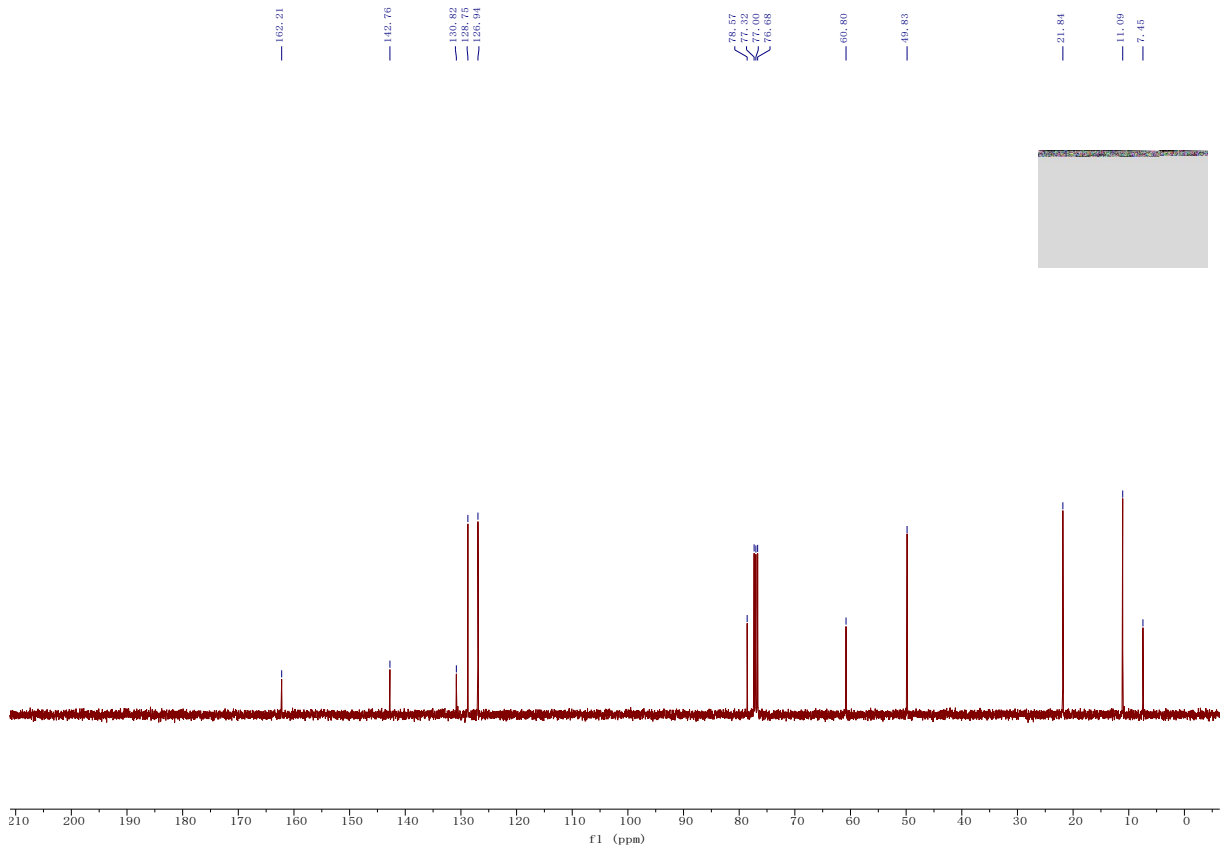
8.02
7.84
7.81
7.26
4.85
4.84
4.84
4.83
4.82
4.82
4.82
4.82
4.81
4.81
4.80
4.80
4.22
4.20
4.18
4.16
3.84
3.82
3.80
3.78
3.39
3.38
3.37
3.36
3.35
3.34
3.33
3.32
3.09
3.07
3.05
1.57
1.55
1.53
1.51
1.49
1.47
0.86
0.84
0.82
2.02
1.98
1.00
1.03
1.05
2.14
4.46
4.31
6.54
f1 (ppm)


162.21
142.76
130.82
128.75
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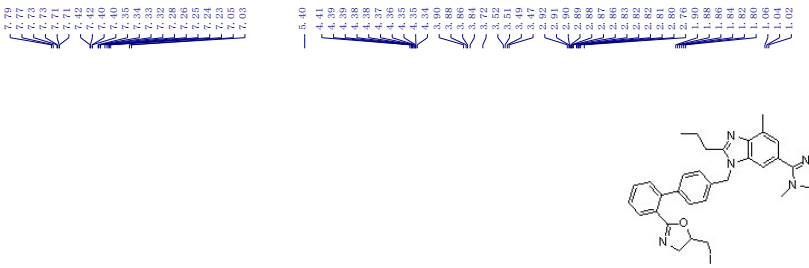
78.57
77.32
77.00
76.68
60.80
49.83
21.84
11.09
7.45
f1 (ppm)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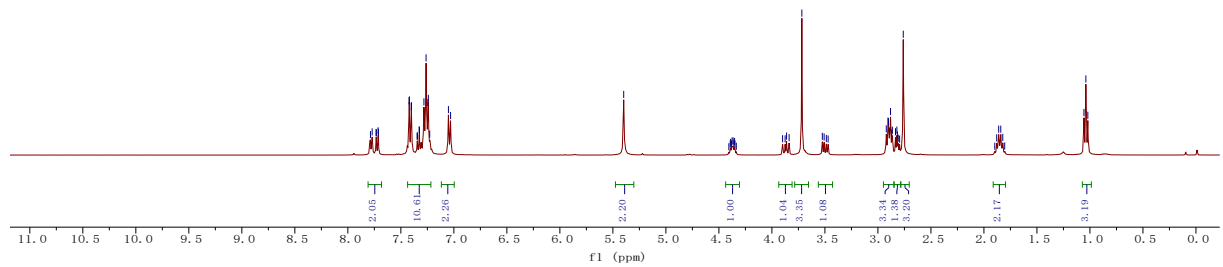
7.77
7.73
7.71
7.42
7.40
7.35
7.34
7.33
7.32
7.26
7.25
7.24
7.23
7.05
7.03
5.40
4.41
4.39
4.38
4.37
4.36
4.35
4.34
3.90
3.88
3.86
3.84
3.72
3.52
3.51
3.49
3.47
2.92
2.91
2.90
2.89
2.88
2.87
2.86
2.83
2.82
2.82
2.81
2.80
2.76
1.90
1.88
1.86
1.84
1.82
1.80
1.06
1.04
1.02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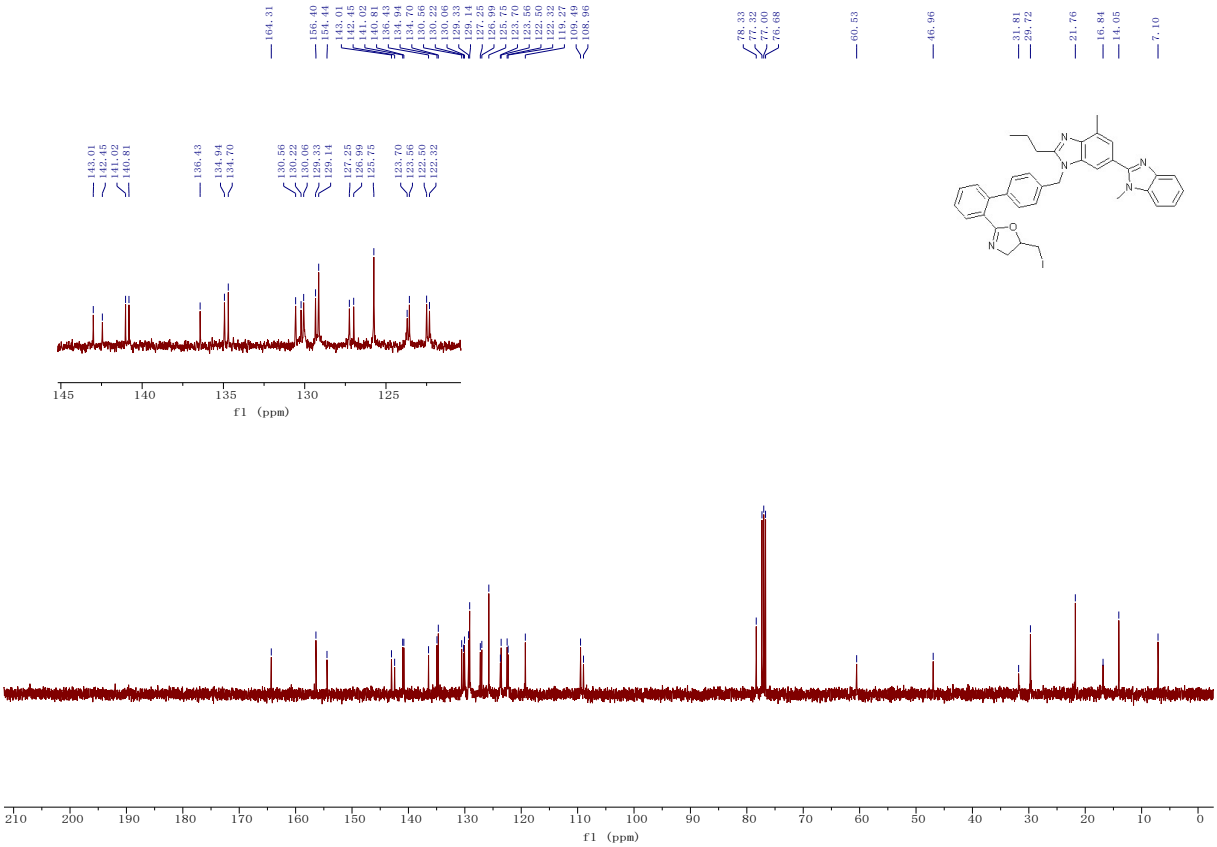
10.61
2.26
2.20
1.00
1.04
3.35
1.08
3.34
1.38
3.20
2.17
3.19
f1 (ppm)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156.40
154.44
143.01
142.45
141.02
140.81
136.43
134.94
134.70
130.56
130.22
130.06
129.33
129.14
127.25
126.99
125.75
123.70
123.56
122.50
122.32
119.27
109.49
108.96
78.33
77.32
77.00
76.68
60.53
46.96
31.81
29.72
21.76
16.84
14.05
7.10
f1 (ppm)