

## Supporting Information

### **A transition-metal-free & diazo-free styrene cyclopropanation**

Ana G. Herraiz<sup>a,b</sup>, Marcos G. Suero<sup>a\*</sup>

<sup>a</sup>*Institute of Chemical Research of Catalonia (ICIQ), The Barcelona Institute of Science and Technology, Av. Països Catalans 16 – 43007 Tarragona (Spain)*

<sup>b</sup>*Departament de Química Analítica i Química Orgànica, Universitat Rovira i Virgili, calle Marcel·lí Domingo, 1, 43007 Tarragona (Spain).*

[mgsuero@iciq.es](mailto:mgsuero@iciq.es)

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## 1. General Information.

All reagents were used as purchased and used without further purification (*E*)- anethole was purchased from Aldrich and used without further purification. Unless otherwise stated, reactions were carried out under argon atmosphere. Anhydrous solvents (DMF, DMSO, THF) were dried by passing through an activated alumina column on a PureSolv<sup>TM</sup> solvent purification system (Innovative Technologies, Inc., MA). CH<sub>3</sub>CN (Panreac, HPLC grade) and EtOH (Scharlau, analytical solvent) were used without any further purification. Blue LED strips (Leroy Merlin Ref. 15379721), white LED strips (Leroy Merlin Ref. 16640246) and compact fluorescent lamp (CFL) (Leroy Merlin Ref. 14640402) were used as visible light sources. Analytical thin layer chromatography was carried out using TLC-aluminum sheets with 0.2 mm of silica gel (Merck GF234). Visualization was achieved using ultraviolet light (254 nm) and chemical staining with vanillin or basic potassium permanganate solutions as appropriate. Flash column chromatography was performed on silica gel (Aldrich, 230-400 mesh). Organic solutions were concentrated under reduced pressure on a Büchi rotatory evaporator. NMR spectra were recorded at 298 K (unless otherwise stated) on a Bruker Avance 300, Bruker Avance 400 Ultrashield and Bruker Avance 500 Ultrashield apparatuses. Chemical shifts ( $\delta$ ) are quoted in ppm relative to residual solvent and coupling constants (*J*) are quoted in hertz (Hz). Multiplicity is reported with the following abbreviations: s = singlet, d = doublet, t = triplet, q = quartet, dt = doublet of triplets, td = triplet of doublets, ddd = doublet of doublet of doublets. Melting points were measured using open glass capillaries in a Büchi B540 apparatus. Infrared spectra were recorded on a Bruker Tensor 27. Mass spectra were recorded on a Waters LCT Premier spectrometer. Ad = 1-adamantanyl

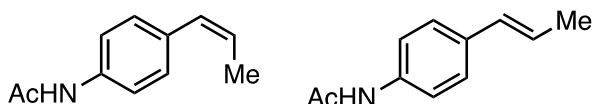
## 2. Synthesis of styrenes 1.

**General Procedure A:** Potassium *tert*-butoxide (1.0 M solution in THF, 16.1 mL, 16.1 mmol) was added to a suspension of the corresponding phosphonium salt (6.46 mmol) in THF (6.5 mL) at 0 °C. After this, the resulting reaction mixture was stirred for 30 min at room temperature. Then, the corresponding aldehyde or ketone (6.46 mmol) was added and stirred until complete conversion (the reaction was monitored by TLC). The mixture was quenched with saturated NH<sub>4</sub>Cl aqueous solution (10 mL) and extracted with AcOEt (3 x 10 mL). The combined organic extracts were washed with brine and dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated *in vacuo*. The crude residues were purified by flash column chromatography on silica gel (AcOEt/hexane mixtures) affording the corresponding alkenes **1**.

**General Procedure B:** To a solution of the corresponding triphenylphosphonium bromide salt (1.2 eq, 7.7 mmol) in dry THF (0.3 M) at -78 °C, *n*-Butyllithium (1.6 M in hexanes, 1.2 eq, 5.25 ml) was added dropwise. The temperature of the reaction mixture was allowed to rise to 0 °C for 30 min and then the corresponding aldehyde or ketone (1.0 eq, 7.0 mmol) dissolved in THF (7 ml) was added dropwise over 20 min. The reaction mixture was stirred for 20 min at 0 °C and then allowed to warm up to room temperature and stirred until complete conversion (the reaction was monitored by TLC). The mixture was quenched with saturated aqueous NH<sub>4</sub>Cl (10 mL) and extracted with AcOEt (3 x 10 mL). The combined organic extracts were washed with brine and dried over anhydrous

$\text{Na}_2\text{SO}_4$  and concentrated *in vacuo*. The crude residues were purified by flash column chromatography on silica gel (AcOEt/hexane mixtures) affording the corresponding alkenes **1**.

### ***N*-(4-(prop-1-en-1-yl)phenyl)acetamide **1c****



Prepared according to **General Procedure A** using *N*-(4-formylphenyl)acetamide (1.05 g, 6.46 mmol), ethyltriphenylphosphonium bromide (3.0 g, 8.08 mmol) and THF (7.0 mL). Purification by flash chromatography on silica gel (hexane/AcOEt, 2:1) provided a mixture of alkenes (560 mg, 49 % yield, 1:1.5 *E/Z*) as a white solid;

**m.p.** 115-118 °C.

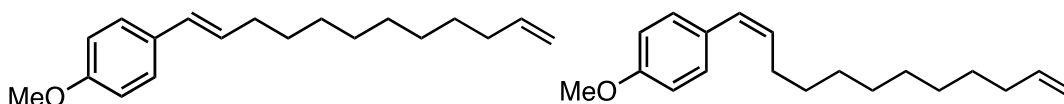
**IR** 3286, 3011, 2406, 1642, 1512, 1413, 841.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.36 (d, *J* = 7.5 Hz, 2H, *Z*), 7.32 (d, *J* = 8.5 Hz, 2H, *E*), 7.20 – 7.12 (m, 4H), 6.31 – 6.20 (m, 2H), 6.06 (dq, *J* = 15.7, 6.6 Hz, 1H, *E*), 5.65 (dq, *J* = 11.6, 7.2 Hz, 1H, *E*), 2.07 (s, 3H, *Z*), 2.06 (s, 3H, *E*), 1.79 (dd, *J* = 7.2, 1.9 Hz, 3H, *Z*), 1.78 – 1.75 (m, 3H, *E*).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 168.38, 168.28, 136.27, 133.95, 130.43, 129.57, 129.30, 126.52, 126.47, 125.18, 120.03, 119.92, 119.64, 119.53, 24.75, 24.71, 18.59, 14.77.

**HRMS** (ESI) calculated for C<sub>11</sub>H<sub>14</sub>NO [M+H]<sup>+</sup> *m/z*: 176.1070, found: 176.1074.

### **1-(deca-1,9-dien-1-yl)-4-methoxybenzene **1d****



Prepared according to **General Procedure A** using undec-10-enal (1 ml, 5.00 mmol), (4-methoxybenzyl)triphenylphosphonium bromide (2.32 g, 5.00 mmol) and THF (6.5 mL). Purification by flash chromatography on silica gel (hexane/EtOAc 50:1) provided a mixture of alkenes (750 mg, 62 % yield, 3:2 *E/Z*) as a colorless oil;

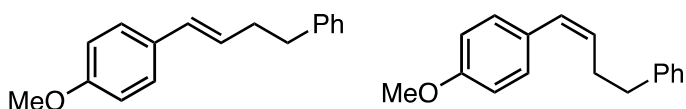
**IR** 2921, 2850, 1606, 1508, 1243, 1173, 1036.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.28 (d, *J* = 8.7 Hz, 2H, *Z*), 7.23 (d, *J* = 8.7 Hz, 2H, *E*), 6.88 (d, *J* = 8.8 Hz, 2H, *Z*), 6.84 (d, *J* = 8.8 Hz, 2H, *E*), 6.34 (td, *J* = 6.5, 6.1, 1.8 Hz, 1H, *E*), 6.30 (d, *J* = 1.5 Hz, 1H, *Z*), 6.09 (dt, *J* = 15.8, 6.9 Hz, 1H, *E*), 5.82 (m, 2H), 5.58 (dt, *J* = 11.7, 7.2 Hz, 1H, *Z*), 5.05 – 4.95 (m, 2H, *E*), 4.94 (ddt, *J* = 10.2, 2.4, 1.2 Hz, 2H, *Z*), 3.82 (s, 3H, *Z*), 3.80 (s, 3H, *E*), 2.32 (qd, *J* = 7.3, 1.9 Hz, 2H, *Z*), 2.18 (qd, *J* = 7.0, 1.5 Hz, 2H, *E*), 2.05 (m, 4H), 1.51 – 1.24 (m, 24H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 158.75, 158.28, 139.38, 131.83, 130.97, 130.68, 130.06, 129.22, 129.16, 128.22, 127.10, 114.24, 114.04, 113.67, 55.42, 55.37, 33.96, 33.16, 30.18, 29.67, 29.62, 29.60, 29.59, 29.50, 29.36, 29.29, 29.26, 29.09, 29.09, 28.79.

**HRMS** (ESI) calculated for C<sub>19</sub>H<sub>29</sub>O [M+H]<sup>+</sup> *m/z*: 273.2213, found: 273.2215.

### 1-methoxy-4-(4-phenylbut-1-en-1-yl)benzene 1h



Prepared according to **General Procedure A** using 4-methoxybenzaldehyde (1.0 ml, 8.80 mmol), triphenyl(3-phenylpropyl)phosphonium bromide (4.84 g, 10.5 mmol) and THF (8.0 mL). Purification by flash chromatography on silica gel (hexane) provided a mixture of alkenes (1.30 g, 62 % yield, 4:1 *E/Z*) as a colorless oil;

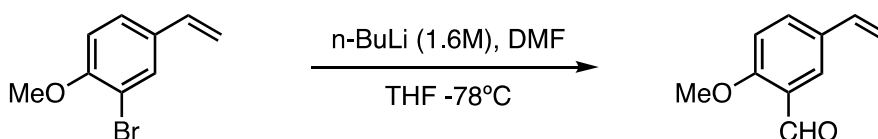
**IR** 1506, 1243, 1173, 962.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.35 – 7.10 (m, 14H), 6.88 – 6.78 (m, 4H), 6.36 (m, 2H), 6.12 (dt, *J* = 15.7, 6.8 Hz, 1H, *E*), 5.61 (dt, *J* = 11.4, 6.9 Hz, 1H, *Z*), 3.81 (s, 3H, *Z*), 3.80 (s, 3H, *E*), 2.78 (m, 4H, *Z,E*), 2.70 – 2.61 (m, 2H, *Z*), 2.56 – 2.47 (m, 2H, *E*).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 158.90, 158.89, 142.03, 141.93, 130.73, 130.41, 130.06, 129.85, 128.96, 128.62 (2C), 128.48 (2C), 127.97, 127.21 (2C), 126.04, 125.98, 114.07, 113.73, 55.44, 55.40, 36.29, 36.19, 35.02, 30.60.

**HRMS** (ESI) calculated for C<sub>17</sub>H<sub>19</sub>O [M+H]<sup>+</sup> *m/z*: 293.1430, found: 293.1427.

### 2-Methoxy-5-vinylbenzaldehyde 1ai



To a solution of 2-bromo-1-methoxy-4-vinylbenzene<sup>1</sup> (470 mg, 2.20 mmol) in THF (8.1 mL) at -78 °C, n-butyllithium (1.6 M in hexanes, 1.6 mL, 2.60 mmol) was added dropwise for 10 min and the resulting reaction mixture was stirred for 15 min. Then, *N,N*-dimethylformamide (1.7 mL, 22.0 mmol) was added at the same temperature, and the mixture was stirred for 2 h. The reaction mixture was quenched with saturated aqueous NH<sub>4</sub>Cl and extracted with AcOEt (3 x 10 mL). The organic layers were combined, dried (Na<sub>2</sub>SO<sub>4</sub>) and concentrated in vacuo. Purification by flash chromatography on silica gel (hexane/AcOEt, 20:1) provided the corresponding aldehyde (230 mg, 65% yield) as a colourless oil;

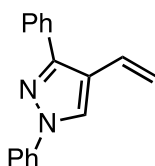
**IR** 1677, 1603, 1495, 1387, 1253, 1020, 905.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 10.46 (s, 1H), 7.87 (d, *J* = 2.4 Hz, 1H), 7.60 (ddd, *J* = 8.6, 2.4, 0.5 Hz, 1H), 6.96 (d, *J* = 8.6 Hz, 1H), 6.66 (dd, *J* = 17.6, 10.9 Hz, 1H), 5.69 (dd, *J* = 17.6, 0.7 Hz, 1H), 5.22 (dd, *J* = 10.9, 0.8 Hz, 1H), 3.94 (s, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 189.87, 161.55, 135.31, 133.60, 130.65, 126.24, 124.81, 113.54, 111.95, 55.96.

**HRMS** (ESI) calculated for C<sub>10</sub>H<sub>11</sub>O<sub>2</sub> [M+H]<sup>+</sup> *m/z*: 163.0754, found: 163.0759.

### 1,3-diphenyl-4-vinyl-1H-pyrazole 1a



Prepared according to **General Procedure B** using 1,3-diphenyl-1H-pyrazole-4-carbaldehyde (650 mg, 2.65 mmol), methyltriphenylphosphonium bromide (1.14 g, 3.18 mmol) and THF (6.5 mL). Purification by flash chromatography on silica gel (hexane/AcOEt, 20:1) provided the corresponding alkene (250 mg, 35 % yield) as a colorless oil;

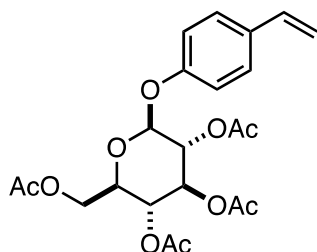
**IR** 1597, 1541, 1502, 1447, 1354, 956.

**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>) δ 8.10 (s, 1H), 7.80 – 7.68 (m, 4H), 7.51 – 7.26 (m, 6H), 6.72 (ddd, *J* = 17.6, 11.0, 0.7 Hz, 1H), 5.58 (dd, *J* = 17.6, 1.5 Hz, 1H), 5.21 (dd, *J* = 11.0, 1.5 Hz, 1H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 151.51, 140.06, 133.31, 129.56, 128.64, 128.59, 128.18, 127.34, 126.64, 124.82, 120.73, 119.21, 114.24.

**HRMS** (ESI) calculated for C<sub>17</sub>H<sub>15</sub>N<sub>2</sub> [M+H]<sup>+</sup> *m/z*: 247.1230, found: 247.1230.

### (2*R*,3*R*,4*S*,5*R*,6*S*)-2-(acetoxymethyl)-6-(4-vinylphenoxy)tetrahydro-2*H*-pyran-3,4,5-triyl triacetate



Prepared according to **General Procedure B** using (2*R*,3*R*,4*S*,5*R*,6*S*)-2-(acetoxymethyl)-6-(4-formylphenoxy)tetrahydro-2*H*-pyran-3,4,5-triyl triacetate<sup>2</sup> (1.78 g, 3.96 mmol), methyltriphenylphosphonium bromide (2.7 g, 5.9 mmol) and THF (8.0 mL). Purification by flash chromatography on silica gel (gradient: hexane/EtOAc 10:1 to 1:1) provided the desired alkene (670 mg, 38 % yield) as a white solid;

**m.p.** 109-112 °C.

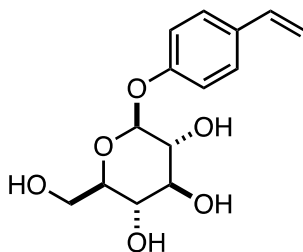
**IR** 1741, 1507, 1364, 1208, 1031, 905.

**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>) δ 7.34 (d, *J* = 8.7 Hz, 2H), 6.95 (d, *J* = 8.7 Hz, 2H), 6.66 (dd, *J* = 17.6, 10.9 Hz, 1H), 5.64 (dd, *J* = 17.6, 0.9 Hz, 1H), 5.35 – 5.24 (m, 2H), 5.23 – 5.12 (m, 2H), 5.11 – 5.02 (m, 1H), 4.29 (dd, *J* = 12.3, 5.3 Hz, 1H), 4.17 (dd, *J* = 12.3, 2.5 Hz, 1H), 3.86 (ddd, *J* = 9.9, 5.3, 2.5 Hz, 1H), 2.08 (s, 3H), 2.06 (s, 3H), 2.05 (s, 3H), 2.04 (s, 3H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 170.73, 170.40, 169.55, 169.45, 156.64, 136.00, 133.23, 127.54, 117.18, 113.17, 99.27, 72.87, 72.22, 71.33, 68.45, 62.10, 20.84, 20.78, 20.77, 20.74.

**HRMS** (ESI) calculated for  $C_{22}H_{26}NaO_{10}$   $[M+Na]^+$   $m/z$ : 473.1418, found: 473.1410.  
 $[\alpha]^{25}_D = -16.6$  ( $c = 0.12$ ,  $CH_2Cl_2$ ).

**(2R,3S,4S,5R,6S)-2-(hydroxymethyl)-6-(4-vinylphenoxy)tetrahydro-2H-pyran-3,4,5-triol**



To a round bottom flask was added (2R,3R,4S,5R,6S)-2-(acetoxymethyl)-6-(4-vinylphenoxy)tetrahydro-2H-pyran-3,4,5-triyl triacetate **4c** (650 mg, 1.44 mmol, 1 equiv.) and  $K_2CO_3$  (2.40 g, 17.0 mmol, 12 equiv.). MeOH (15 ml) was added and the reaction mixture was stirred for 1h at room temperature. Then, a NaOH solution (0.1M, 30ml) was added and the aqueous phase was extracted with AcOEt (3 x 50 ml). The combined organic layers were washed with brine and dried over  $Na_2SO_4$ . After concentration under vacuo, the corresponding white solid (370 mg, 91% yield) was used without further purification.

**m.p.** 168-172 °C.

**IR** 2916, 2849, 2359, 1106, 1245, 1074, 1058, 840.

**$^1H$  NMR** (400 MHz, MeOD)  $\delta$  7.44 (d,  $J = 8.7$  Hz, 2H), 7.13 (d,  $J = 8.7$  Hz, 2H), 6.75 (dd,  $J = 17.6, 10.9$  Hz, 1H), 5.72 (dd,  $J = 17.6, 1.1$  Hz, 1H), 5.20 (dd,  $J = 10.9, 1.0$  Hz, 1H), 5.01 – 4.97 (m, 1H), 3.97 (dd,  $J = 12.0, 2.1$  Hz, 1H), 3.78 (dd,  $J = 12.1, 5.3$  Hz, 1H), 3.60 – 3.43 (m, 4H).

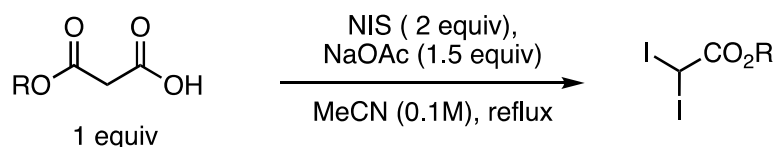
**$^{13}C$  NMR** (101 MHz, MeOD)  $\delta$  157.51, 136.15, 132.11, 126.92, 116.39, 111.03, 100.90, 76.83, 76.66, 73.57, 70.04, 61.17.

**HRMS** (ESI) calculated for  $C_{14}H_{18}NaO_6$   $[M+Na]^+$   $m/z$ : 305.0996, found: 305.0997

$[\alpha]^{25}_D = -38.5$  ( $c = 0.11$ ,  $CH_2Cl_2$ ).

### 3. Synthesis of *gem*-diiodomethyl carbonyl reagents

#### General procedure C:



To a round bottom flask with a stir bar was added NIS (20 mmol, 2 equiv), NaOAc (15 mmol, 1.5 equiv) and the corresponding malonate monoester (10 mmol, 1 equiv). The reaction was degassed and filled with argon (x 3) and MeCN (80 ml, 0.12M) was added. A condenser was placed and the reaction mixture was stirred under reflux. The reaction was monitored by TLC and it was quenched upon complete consumption of the malonate monoester (2-4 h). A  $Na_2S_2O_3$  saturated aqueous solution (50 ml) was added and

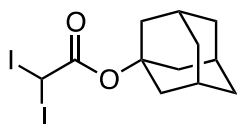
extracted with Et<sub>2</sub>O (3 x 50 ml). Combined organic layers were dried over MgSO<sub>4</sub> and concentrated. The residue was purified by flash chromatography on silica gel (hexane 100% to hexane/EtOAc 50:1). Once the product was eluted from the column, the corresponding fractions were concentrated. If the test tubes were left in the bench the *gem*-diiodo reagent will slowly decompose and a pink color will appear (I<sub>2</sub> formation). In this case, a spatula of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> could be added to the collected tubes and stirred until the pink color disappeared, the solid was then removed by filtration and the desired product was obtained after concentration.

Note:

If the *gem*-diiodo reagent was a liquid, it was kept in solution in MeCN (1.0–0.5M) at -30°C under argon atmosphere. 1,2-Dichloroethane was used as external standard to calculate the concentration.

If the *gem*-diiodo reagent was a solid, it was kept as a solid at -30°C.

### Adamantan-1-yl 2,2-diiodoacetate **2a**



Prepared according to **General Procedure C** using 3-((adamantan-1-yl)oxy)-3-oxopropanoic acid<sup>3</sup> (10.3 g, 43 mmol, 1 equiv). Purification by flash chromatography on silica gel (gradient: hexane/EtOAc 100:0 to 100:1) provided the corresponding product (12 g, 65%) as a white pale solid.

**m.p.** 88–91 °C.

**IR** 2907, 2850, 1713, 1246, 1120, 1048.

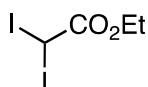
**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>) δ 5.27 (s, 1H), 2.20 (m, 3H), 2.13 (m, 6H), 1.68 (m, 6H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 164.91, 84.27, 40.71, 36.16, 31.03, -40.50.

**HRMS** (ESI) calculated for C<sub>12</sub>H<sub>16</sub>I<sub>2</sub>NaO<sub>2</sub> [M+Na]<sup>+</sup> m/z: 468.9132, found: 468.9120.

The crystal structure of **2a** has been deposited at the Cambridge Crystallographic Data Centre, CCDC 1915890. The crystal of **2a** used for the single-crystal X-ray diffraction experiment was grown from a solution of **2a** in CH<sub>3</sub>CN.

### Ethyl 2,2-diiodoacetate **2b**



Prepared according to **General Procedure C** using 3-ethoxy-3-oxopropanoic acid (1.2 ml, 0.2 mmol, 1 equiv). Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided the corresponding product (40–65% yield) that was kept in a MeCN solution (1.0–0.5M).

**IR** 2921, 2850, 1718, 1247, 1118, 1022, 858.

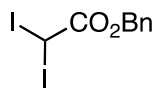
**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>) δ 5.34 (s, 1H), 4.28 (q, *J* = 7.1 Hz, 2H), 1.31 (t, *J* = 7.1 Hz, 3H).



<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 166.41, 63.71, 13.76, -45.25.

HRMS (APCI) calculated for C<sub>4</sub>H<sub>7</sub>I<sub>2</sub>O<sub>2</sub> [M+H]<sup>+</sup> m/z: 340.8530, found: 340.8527.

### Benzyl 2,2-diiodoacetate 2b



Prepared according to **General Procedure C** using 3-(benzyloxy)-3-oxopropanoic acid (1.2 ml, 0.2 mmol, 1 equiv). Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided the corresponding product (45% yield) that was kept in a MeCN solution (0.6M).

IR 2917, 2848, 1512, 1251, 1118, 1022, 837.

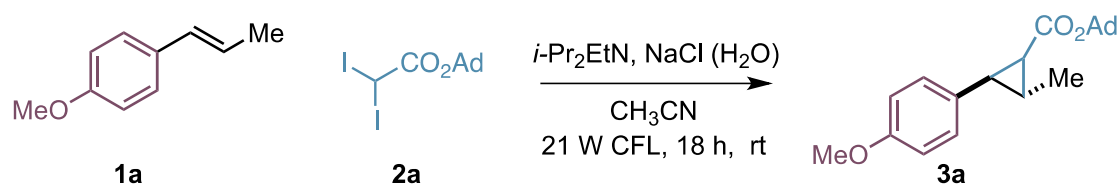
<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 7.46 – 7.32 (m, 6H), 5.37 (s, 1H), 5.24 (s, 2H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 166.07, 134.66 (2C), 128.76, 128.38, 69.10, -45.75.

MS calculated for C<sub>4</sub>H<sub>7</sub>I<sub>2</sub>O<sub>2</sub> [M] m/z: 401.86, found (and fragmentations): 401.7; 275.0; 148; 107.0; 91.1; 77.0.

## 4. Visible-light-driven cyclopropanation of styrenes: evaluation of reaction parameters.

### General procedure D:

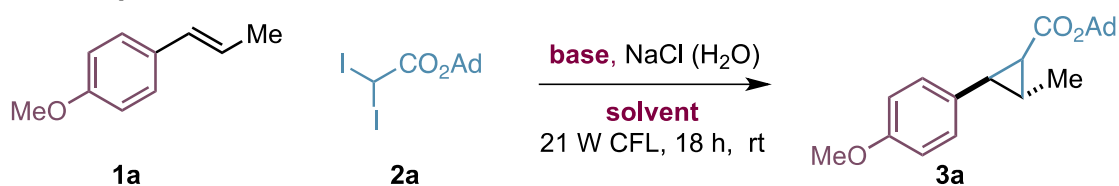


To a 10 mL reaction tube equipped with a stirring bar was added adamantan-1-yl 2,2-diiodoacetate (45 mg, 0.10 mmol, 1 equiv) and the tube was sealed with a septum, degassed and filled with argon. MeCN (1ml), *E*-anethole **1a** (15  $\mu$ L, 0.10 mmol, 1 equiv), *i*-Pr<sub>2</sub>EtN (0.20 mmol, 2 equiv) and aqueous NaCl (1.25 M, 0.5 mL) were added. The reaction mixture was degassed by two freeze-pump-thaw cycles under argon. After that, the reaction tube was irradiated under the light source (21W CFL) at a distance of 7 cm and a mini-fan was kept on top to maintain room temperature. After 18 h, the reaction mixture was passed through a short pad of silica gel and eluted with dichloromethane. The solvent was removed under *vacuum* and 1,2-dimethoxyethane (10  $\mu$ L, 0.10 mmol, 1 equiv) was added as internal standard. The mixture was then analysed by <sup>1</sup>H-NMR.

**Table 1.** Control experiments

Entry	Modification of the conditions	Yield <b>3a</b> [%]
1	with Ru(bpy) <sub>3</sub> (PF <sub>6</sub> ) <sub>2</sub> 1 mol %	63
2	none	60
3	without <i>i</i> -Pr <sub>2</sub> EtN	0
4	under dark	0
5	no NaCl in H <sub>2</sub> O	17
6	under air	10
7	2 equivalents of <b>1a</b>	91 <sup>a</sup>
8	white LEDs as light source	42
9	blue LEDs as light source	44

<sup>a</sup> 1 equiv of **2a** and 2 equiv of *i*-Pr<sub>2</sub>EtN were added after 4h of reaction time, isolated yield.

**General procedure E:**

To a 10 mL reaction tube equipped with a stirring bar was added adamantan-1-yl 2,2-diodoacetate (45 mg, 0.10 mmol, 1 equiv) and the tube was sealed with a septum, degassed and filled with argon. The corresponding solvent (1 mL), *E*-anethole **1a** (15  $\mu$ L, 0.10 mmol, 1 equiv), the corresponding amine (0.20 mmol, 2 equiv) and aqueous NaCl (1.25 M, 0.5 mL) were added. The reaction mixture was degassed by two freeze-pump-thaw cycles under argon. After that, the reaction tube was irradiated under the light source (21W CFL) at a distance of 7 cm and a mini-fan was kept on top to maintain room temperature. After 18 h, the reaction mixture was passed through a short pad of silica gel and eluted with dichloromethane. The solvent was removed under *vacuum* and 1,2-dimethoxyethane (10  $\mu$ L, 0.10 mmol, 1 equiv) was added as internal standard. The mixture was then analysed by <sup>1</sup>H-NMR.

**Table 2.** Evaluation of solvents and amines.

Entry	Solvent	Base	Yield <b>3a</b> [%]
1	MeCN	Et <sub>3</sub> N	41
2	MeCN	<i>i</i> -Pr <sub>2</sub> NH	17
3	MeCN	DABCO	11
-----			
4	Acetone	<i>i</i> -Pr <sub>2</sub> EtN	n.d.
5	DCM	<i>i</i> -Pr <sub>2</sub> EtN	16
6	THF	<i>i</i> -Pr <sub>2</sub> EtN	35

## 5. Visible-light-driven cyclopropanation of styrenes: reaction scope.

### **General procedure F (ratio 1:2 styrene:*gem*-diiodo)**

To a 10 mL reaction tube equipped with a stirring bar was added the *gem*-diiodo reagent **2** (0.20 mmol, 1 equiv) and the tube was sealed with a septum, degassed and filled with argon. MeCN (2ml), the corresponding styrene (0.2 mmol, 1 equiv), *i*-Pr<sub>2</sub>EtN (0.40 mmol, 2 equiv) and aqueous NaCl (1.25 M, 1 mL) were added. The reaction mixture was degassed by two freeze-pump-thaw cycles under argon. After that, the reaction tube was irradiated under with visible light (21W CFL at a distance of 7 cm) and a mini-fan was kept on top to maintain room temperature. After 4 h *gem*-diiodo reagent (0.20 mmol, 1 equiv) and *i*-Pr<sub>2</sub>EtN (0.40 mmol, 2 equiv) were added and the reaction mixture was stirred for 14 more hours. Then, the reaction mixture was passed through a short pad of silica gel and eluted with dichloromethane. The solvent was removed under *vacuum* and the residue was purified by column chromatography on neutral silica gel to give the corresponding cyclopropanes. Ratio of isomers was determined from the crude reaction mixture using <sup>1</sup>H NMR spectroscopy.

### **General procedure G (ratio 2:1 styrene:*gem*-diiodo)**

To a 10 mL reaction tube equipped with a stirring bar was added the *gem*-diiodo reagent **2** (0.20 mmol, 1 equiv) and the tube was sealed with a septum, degassed and filled with argon. MeCN (2ml), the corresponding styrene (0.4 mmol, 2 equiv), *i*-Pr<sub>2</sub>EtN (0.40 mmol, 2 equiv) and aqueous NaCl (1.25 M, 1 mL) were added. The reaction mixture was degassed by two freeze-pump-thaw cycles under argon. After that, the reaction tube was irradiated under with visible light (21W CFL at a distance of 7 cm) and a mini-fan was kept on top to maintain room temperature. After 18 h, the reaction mixture was passed through a short pad of silica gel and eluted with dichloromethane. The solvent was removed under *vacuum* and the residue was purified by column chromatography on neutral silica gel to give the corresponding cyclopropanes. Ratio of isomers was determined from the crude reaction mixture using <sup>1</sup>H NMR spectroscopy.

#### *Note:*

The *cis/trans* configuration of cyclopropanes was determined based on the value of the *J* coupling constants for the benzylic and  $\alpha$ -protons to the carbonyl group. The <sup>3</sup>*J*<sub>*cis*</sub> (7-13 Hz) is always larger than <sup>3</sup>*J*<sub>*trans*</sub> (2-7 Hz), and this can be reliably used for the

assignment of relative configuration assignment. In some cases, 2D-NMR (HMBC, HMQC and NOESY) were measured.

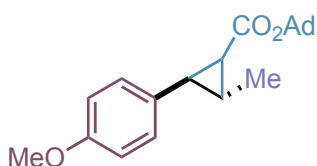
**Procedure for a 1g-scale reaction with *trans*-anethole:**

To a 250 ml round-bottom-flask was added adamantan-1-yl 2,2-diiodoacetate **2a** (3.0 g, 6.76 mmol, 1 equiv) and the flask was degassed and filled with argon (x 3). Then, CH<sub>3</sub>CN (80 mL, previously degassed by bubbling argon for 15 min), *E*-anethole **1a** (1.0 g, 6.75 mmol, 1 equiv), aqueous NaCl (1.25 M, 30 mL, previously degassed by bubbling argon for 15 min) and *i*-Pr<sub>2</sub>EtN (1.2 mL, 2 equiv) were added. A balloon filled with argon was placed and the reaction mixture was irradiated with 2 x 21 W CFL as visible-light source at a distance of 5 cm and stirred at room temperature. After 4 h *i*-Pr<sub>2</sub>EtN (0.6 mL, 1 equiv) was added and the reaction mixture was stirred for 13 h. The reaction mixture was quenched with water (150 ml) and extracted with AcOEt (3 x 100 ml). Combined organic layers were washed with brine, dried over anhydrous MgSO<sub>4</sub> and concentrated *in vacuo*. Purification by flash chromatography (gradient: hexane/EtOAc 100:0 to 50:1) provided the corresponding cyclopropanes **3a** (1.75 g, 75 %) as colorless oils and the remaining *E*-anethole (0.14 g, 15%).



**Figure 1.** Set-up for a 1g-scale reaction.

**Adamantan-1-yl(2*R*\*,3*R*\*)-2-(4-methoxyphenyl)-3-methylcyclopropane-1-carboxylate **3a****



Prepared according to **General procedure F** using (*E*)-anethole **1a** (30 mg, 0.2 mmol, 1 equiv) and adamantan-1-yl 2,2-diiodoacetate **2a**. Purification by flash chromatography on silica gel (gradient: hexane/EtOAc 100:0 to 100:1) provided the corresponding cyclopropanes (62 mg, 91%). Ratio of isomers was determined to be 1:1.1 (*trans*:*cis*).

*trans*-isomer:

IR 2906, 2850, 1712, 1514, 1245, 1171, 1054.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.00 (d, *J* = 8.5 Hz, 2H), 6.81 (d, *J* = 8.6 Hz, 2H), 3.78 (s, 3H), 2.27 (app. t, *J* = 5.7 Hz, 1H), 2.16 (s, 3H), 2.14 (s, 6H), 1.87 (dd, *J* = 9.3, 5.0 Hz,

1H), 1.68 (d,  $J = 14.9$  Hz, 6H), 1.54 (ddd,  $J = 15.4, 13.1, 6.8$  Hz, 1H), 1.32 (d,  $J = 6.2$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  170.82, 158.20, 133.14, 127.23, 113.99, 80.73, 55.47, 41.70, 36.39, 31.27, 31.00, 30.46, 25.17, 12.08.

HRMS (ESI) calculated for  $\text{C}_{22}\text{H}_{28}\text{NaO}_3$   $[\text{M}+\text{Na}]^+$   $m/z$ : 363.1931, found: 363.1923.

*cis-isomer*:

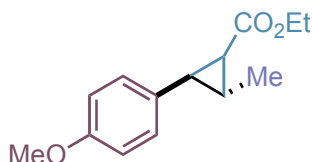
IR 2907, 2850, 1716, 1514, 1246, 1165, 1059.

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.15 (d,  $J = 8.3$  Hz, 2H), 6.82 – 6.78 (m, 2H), 3.78 (s, 3H), 2.23 (dd,  $J = 9.2, 6.8$  Hz, 1H), 2.03 (s, 3H), 1.95 – 1.88 (m, 1H), 1.87 – 1.76 (m, 6H), 1.67 (dd,  $J = 9.3, 5.0$  Hz, 1H), 1.55 (s, 6H), 1.23 (d,  $J = 6.1$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  170.13, 158.31, 130.41, 129.37, 113.42, 55.42, 41.25, 36.28, 33.37, 31.11, 30.86, 19.35, 17.84.

HRMS (ESI) calculated for  $\text{C}_{22}\text{H}_{28}\text{NaO}_3$   $[\text{M}+\text{Na}]^+$   $m/z$ : 363.1931, found: 363.1929.

#### Ethyl (2*R*\*,3*R*\*)-2-(4-methoxyphenyl)-3-methylcyclopropane-1-carboxylate **3b**<sup>4</sup>



Prepared according to **General procedure F** using (*E*)-anethole **1a** (30 mg, 0.2 mmol, 1 equiv) and ethyl 2,2-diiodoacetate **2b**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided the corresponding cyclopropanes (41 mg, 88%). Ratio of isomers was determined to be 1:1. Data are consistent with those reported in the literature.

*trans-isomer*:

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.01 (d,  $J = 8.5$  Hz, 2H), 6.81 (d,  $J = 8.7$  Hz, 2H), 4.17 (qd,  $J = 7.1, 1.7$  Hz, 2H), 3.77 (s, 3H), 2.37 (dd,  $J = 6.5, 5.1$  Hz, 1H), 1.93 (dd,  $J = 9.2, 5.0$  Hz, 1H), 1.62 (m, 1H), 1.34 (d,  $J = 6.2$  Hz, 3H), 1.28 (t,  $J = 7.2$  Hz, 3H).

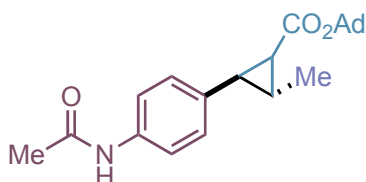
$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  171.94, 158.33, 132.77, 127.33, 114.05, 60.54, 55.48, 31.92, 29.24, 25.30, 14.54, 12.16.

*cis-isomer*:

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.15 (d,  $J = 8.6$  Hz, 2H), 6.79 (d,  $J = 8.7$  Hz, 2H), 3.89 (qd,  $J = 7.1, 1.7$  Hz, 2H), 3.77 (s, 3H), 2.29 (dd,  $J = 8.9, 7.1$  Hz, 1H), 2.02 (app. hept,  $J = 6.1$  Hz, 1H), 1.77 (dd,  $J = 9.2, 5.0$  Hz, 1H), 1.26 (d,  $J = 6.1$  Hz, 3H), 1.02 (t,  $J = 7.1$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  171.16, 158.38, 130.23, 128.90, 113.45, 60.21, 55.34, 33.77, 30.12, 19.98, 17.89, 14.27.

#### Adamantan-1-yl(2*R*\*,3*R*\*)-2-(4-acetamidophenyl)-3-methylcyclopropane-1-carboxylate **3c**



Prepared according to **General procedure F** using *N*-(4-(prop-1-en-1-yl)phenyl)acetamide **1c** (35 mg, 0.2 mmol, 1 equiv) and adamantan-1-yl 2,2-diiodoacetate **2a**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 50:0 to 10:1) provided a mixture of the corresponding cyclopropanes (35 mg, 48%). Ratio of isomers was determined to be 1:1.

*trans* and *cis*-isomer:

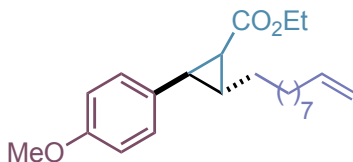
**IR** 2908, 2851, 1663, 1517, 1171, 1054.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.38 (d, *J* = 7.8 Hz, 4H), 7.17 (d, *J* = 8.3 Hz, 2H), 7.00 (d, *J* = 8.4 Hz, 2H), 2.32 – 2.20 (m, 2H), 2.14 (d, *J* = 4.3 Hz, 15H), 2.03 (s, 3H), 1.96 – 1.89 (m, 2H), 1.83 (q, *J* = 11.8, 11.4 Hz, 6H), 1.74 – 1.69 (m, 1H), 1.66 (s, 6H), 1.54 (s, 7H), 1.31 (d, *J* = 6.2 Hz, 3H), 1.23 (d, *J* = 6.0 Hz, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 170.50, 169.91, 168.15, 168.06, 136.99, 136.25, 135.99, 133.08, 129.82, 126.57, 119.99, 119.20, 80.74, 80.20, 41.55, 41.15, 36.24, 36.12, 33.38, 31.25, 31.05, 30.86, 30.72, 30.50, 25.22, 24.63, 24.55, 19.27, 17.65, 11.93.

**HRMS** (ESI) calculated for C<sub>23</sub>H<sub>29</sub>NNaO<sub>3</sub> [M+Na]<sup>+</sup> *m/z*: 390.2040, found: 390.2031.

**Ethyl (2*R*\*,3*R*\*)-2-(dec-9-en-1-yl)-3-(4-methoxyphenyl)cyclopropane-1-carboxylate **3d****



Prepared according to **General procedure F** using 1-(dodeca-1,11-dien-1-yl)-4-methoxybenzene **1d** (55 mg, 0.2 mmol, 1 equiv) and ethyl 2,2-diiodoacetate **2b**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided the corresponding cyclopropanes (46 mg, 64%). Ratio of isomers was determined to be 1:1.

*trans*-isomer:

**IR** 2924, 2852, 1123, 1516, 1247, 1175.

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.05 – 6.99 (m, 2H), 6.84 – 6.78 (m, 2H), 5.81 (ddt, *J* = 16.9, 10.2, 6.7 Hz, 1H), 4.99 (dq, *J* = 17.1, 1.7 Hz, 1H), 4.93 (ddt, *J* = 10.2, 2.3, 1.2 Hz, 1H), 4.16 (q, *J* = 7.2 Hz, 2H), 3.78 (s, 3H), 2.41 (dd, *J* = 6.7, 5.0 Hz, 1H), 2.07 – 2.00 (m, 2H), 1.94 (dd, *J* = 9.1, 5.0 Hz, 1H), 1.77 – 1.68 (m, 1H), 1.67 – 1.59 (m, 1H), 1.54 (dq, *J* = 9.0, 7.0 Hz, 1H), 1.42 – 1.24 (m, 15H).

**<sup>13</sup>C NMR** (75 MHz, CDCl<sub>3</sub>) δ 172.09, 158.28, 139.38, 132.82, 127.43, 114.24, 114.01, 60.54, 55.45, 33.95, 31.39, 31.07, 29.67, 29.65, 29.57, 29.42, 29.25, 29.08, 28.62, 26.93, 14.51.

**HRMS** (ESI) calculated for C<sub>23</sub>H<sub>34</sub>NaO<sub>3</sub> [M+Na]<sup>+</sup> *m/z*: 381.2400, found: 381.2392.

*cis-isomer:*

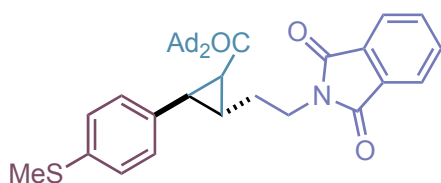
**IR** 2923, 2852, 1727, 1514, 1246, 1174, 1037;

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.15 (d, *J* = 8.5 Hz, 2H), 6.79 (d, *J* = 8.7 Hz, 2H), 5.81 (ddt, *J* = 17.0, 10.2, 6.7 Hz, 1H), 5.02 – 4.96 (m, 1H), 4.95 – 4.90 (m, 1H), 3.90 (qd, *J* = 7.1, 1.3 Hz, 2H), 3.77 (s, 3H), 2.30 (dd, *J* = 9.0, 7.1 Hz, 1H), 2.07 – 2.01 (m, 2H), 1.98 (qd, *J* = 6.9, 5.2 Hz, 1H), 1.79 (dd, *J* = 9.2, 5.0 Hz, 1H), 1.54 – 1.44 (m, 3H), 1.36 (m, 11H), 1.02 (t, *J* = 7.1 Hz, 3H).

**<sup>13</sup>C NMR** (75 MHz, CDCl<sub>3</sub>) δ 171.25, 158.36, 139.38, 130.30, 129.06, 114.27, 113.45, 60.20, 55.33, 33.95, 33.05, 32.74, 29.66, 29.55, 29.46, 29.25, 29.17, 29.06, 28.90, 25.83, 14.27.

**HRMS** (ESI) calculated for C<sub>23</sub>H<sub>34</sub>NaO<sub>3</sub> [M+Na]<sup>+</sup> *m/z*: 381.2400, found: 381.2394.

**(Adamantan-1-yl(2*R*\*,3*R*\*)-2-(2-(1,3-dioxoisindolin-2-yl)ethyl)-3-(4-(methylthio)phenyl)cyclopropane-1-carboxylate 3e**



Prepared according to **General procedure G** using 2-(4-(4-(methylthio)phenyl)but-3-en-1-yl)isindoline-1,3-dione<sup>5</sup> **1e** (65 mg, 0.2 mmol, 2 equiv) and adamantan-1-yl 2,2-diiodoacetate **2a** and 4 ml of MeCN. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 10:1) provided the corresponding cyclopropanes (33 mg, 61%). Ratio of isomers was determined to be 1:0.7 (*trans*:*cis*).

*trans-isomer:*

**IR**: 2909, 2851, 1711, 1394, 1178.

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.77 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.68 (dd, *J* = 5.4, 3.0 Hz, 2H), 7.09 (d, *J* = 8.4 Hz, 2H), 6.88 (d, *J* = 8.3 Hz, 2H), 3.88 – 3.72 (m, 2H), 2.44 (s, 3H), 2.28 (dd, *J* = 6.5, 5.1 Hz, 1H), 2.23 – 2.00 (m, 11H), 1.92 (dd, *J* = 9.2, 5.0 Hz, 1H), 1.65 (m, 6H), 1.57 – 1.49 (m, 1H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 170.35, 168.41, 137.42, 136.02, 133.92, 132.30, 127.21, 126.67, 123.30, 81.16, 41.52, 37.73, 36.33, 30.99, 30.74, 29.65, 28.35, 26.06, 16.47.

**HRMS** (ESI) calculated for C<sub>31</sub>H<sub>33</sub>NNaO<sub>4</sub>S [M+Na]<sup>+</sup> *m/z*: 538.2023, found: 538.2033.

**<sup>1</sup>H-<sup>13</sup>C HSQC, HMBC** and **<sup>1</sup>H-<sup>1</sup>H NOESY** spectra were measured.

*cis-isomer:*

**IR** 2908, 2820, 1708, 1394, 1362, 1055.

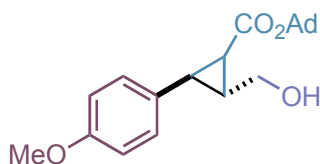
**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>) δ 7.82 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.70 (dd, *J* = 5.5, 3.0 Hz, 2H), 7.17 – 7.06 (m, 4H), 3.89 (t, *J* = 6.9 Hz, 2H), 2.45 (s, 3H), 2.29 (dd, *J* = 9.5, 6.2 Hz, 1H), 2.04 (d, *J* = 8.6 Hz, 3H), 1.99 – 1.93 (m, 1H), 1.88 (m, 2H), 1.82 – 1.75 (m, 6H), 1.72 (m, 1H), 1.55 (m, 6H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 169.19, 168.46, 136.30, 134.03, 133.65, 132.21, 129.92, 126.70, 123.36, 80.52, 41.13, 36.25, 31.97, 31.53, 31.03, 30.84, 29.59, 22.17, 16.48.

**HRMS** (ESI) calculated for C<sub>31</sub>H<sub>33</sub>NNaO<sub>4</sub>S [M+Na]<sup>+</sup> *m/z*: 538.2023, found: 538.2023.

**<sup>1</sup>H-<sup>13</sup>C HSQC, HMBC** and **<sup>1</sup>H-<sup>1</sup>H NOESY** spectra were measured

**Adamantan-1-yl (2*R*\*,3*R*\*)-2-(hydroxymethyl)-3-(4-methoxyphenyl)cyclopropane-1-carboxylate **3f****



Prepared according to **General procedure F** using (*E*)-3-(4-methoxyphenyl)prop-2-en-1-ol **1f** (32 mg, 0.2 mmol, 1 equiv) and adamantan-1-yl 2,2-diiodoacetate **2a**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 20:0 to 4:1) provided the corresponding cyclopropanes (66 mg, 93%). Ratio of isomers was determined to be 1:1.

*trans*-isomer:

IR 2908, 2851, 1718, 1513, 830.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.03 (d, *J* = 8.6 Hz, 2H), 6.82 (d, *J* = 8.7 Hz, 2H), 4.07 (dd, *J* = 11.9, 4.8 Hz, 1H), 3.91 (dd, *J* = 11.9, 7.7 Hz, 1H), 3.78 (s, 3H), 2.60 – 2.55 (m, 1H), 2.17 (s, 3H), 2.13 (d, *J* = 3.3 Hz, 6H), 1.95 (dd, *J* = 8.9, 5.2 Hz, 1H), 1.87 (dddd, *J* = 9.0, 7.8, 6.5, 4.8 Hz, 1H), 1.66 (d, *J* = 3.0 Hz, 6H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 171.86, 158.52, 131.88, 127.53, 114.10, 81.58, 60.21, 55.49, 41.54, 36.32, 32.24, 31.00, 29.35, 28.96.

HRMS (ESI) calculated for C<sub>22</sub>H<sub>28</sub>NaO<sub>4</sub> [M+Na]<sup>+</sup> *m/z*: 379.1880, found: 379.1880.

*cis*-isomer:

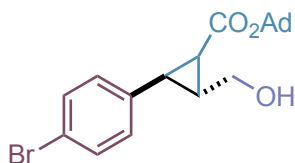
IR 2908, 2851, 1715, 1454, 1244, 835.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.17 (d, *J* = 8.3 Hz, 2H), 6.81 (d, *J* = 8.7 Hz, 2H), 3.77 (s, 3H), 3.70 (dd, *J* = 6.4, 2.1 Hz, 2H), 2.43 (dd, *J* = 9.6, 6.6 Hz, 1H), 2.25 (qd, *J* = 6.4, 5.0 Hz, 1H), 2.03 (m, 3H), 1.90 (dd, *J* = 9.5, 5.1 Hz, 1H), 1.87 – 1.76 (m, 6H), 1.54 (m, 6H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 169.35, 158.54, 130.48, 128.29, 113.54, 80.62, 64.54, 55.44, 41.20, 36.23, 30.85, 29.53, 27.74, 26.81.

HRMS (ESI) calculated for C<sub>22</sub>H<sub>28</sub>NaO<sub>4</sub> [M+Na]<sup>+</sup> *m/z*: 379.1880, found: 379.1865.

**Adamantan-1-yl (2*R*\*,3*R*\*)-2-(4-bromophenyl)-3-(hydroxymethyl)cyclopropane-1-carboxylate **3g****



Prepared according to **General procedure G** using (*E*)-3-(4-bromophenyl)prop-2-en-1-ol **1g** (85 mg, 0.4 mmol, 2 equiv) and adamantan-1-yl 2,2-diiodoacetate **2a**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 20:0 to 4:1) provided the corresponding cyclopropanes (34 mg, 42%). Ratio of isomers was determined to be 1:1.

*trans*-isomer:

IR 2911, 2851, 1714, 1352, 1180, 1055, 805.



**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.42 – 7.38 (m, 2H), 7.00 – 6.93 (m, 2H), 4.06 (dt, *J* = 11.7, 5.1 Hz, 1H), 3.90 (dt, *J* = 11.9, 7.4 Hz, 1H), 2.61 – 2.55 (m, 1H), 2.20 (dd, *J* = 7.0, 5.7 Hz, 1H), 2.18 (s, 3H), 2.13 (d, *J* = 3.4 Hz, 6H), 1.99 (dd, *J* = 9.1, 5.2 Hz, 1H), 1.71 – 1.62 (m, 6H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 171.38, 138.97, 131.68, 128.19, 120.35, 81.91, 59.98, 41.52, 36.29, 32.38, 31.00, 29.56, 28.81.

**HRMS** (ESI) calculated for C<sub>21</sub>H<sub>25</sub>BrNaO<sub>3</sub> [M+Na]<sup>+</sup> *m/z*: 427.0879, found: 427.0880.

*cis-isomer*:

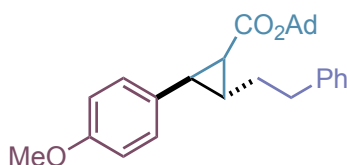
**IR** 2908, 2815, 1715, 1192, 1054.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.43 – 7.35 (m, 2H), 7.18 – 7.10 (m, 2H), 3.73 (dd, *J* = 6.2, 1.4 Hz, 2H), 2.43 (dd, *J* = 9.6, 6.7 Hz, 1H), 2.26 (qd, *J* = 6.3, 5.1 Hz, 1H), 2.05 (t, *J* = 3.2 Hz, 3H), 1.96 (dd, *J* = 9.6, 5.1 Hz, 1H), 1.89 – 1.72 (m, 6H), 1.69 – 1.51 (m, 6H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 168.98, 135.43, 131.26, 131.17, 120.70, 80.99, 64.16, 41.27, 36.22, 30.87, 29.41, 27.64, 26.78.

**HRMS** (ESI) calculated for C<sub>21</sub>H<sub>25</sub>BrNaO<sub>3</sub> [M+Na]<sup>+</sup> *m/z*: 427.0879, found: 427.0869.

**Adamantan-1-yl (2*R*\*,3*R*\*)-2-(4-methoxyphenyl)-3-phenethylcyclopropane-1-carboxylate 3h**



Prepared according to **General procedure G** using 1-methoxy-4-(4-phenylbut-1-en-1-yl)benzene **1h** (95 mg, 0.4 mmol, 2 equiv) and adamantan-1-yl 2,2-diiodoacetate **2a**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided the corresponding cyclopropanes (63 mg, 73%). Ratio of isomers was determined to be 1:1.

*trans-isomer*:

**IR** 2906, 2850, 1711, 1453, 1056, 1163.

**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>) δ 7.37 – 7.29 (m, 2H), 7.25 (d, *J* = 6.9 Hz, 3H), 7.06 – 6.98 (m, 2H), 6.90 – 6.81 (m, 2H), 3.84 (s, 3H), 2.84 – 2.66 (m, 2H), 2.41 (dd, *J* = 6.6, 5.0 Hz, 1H), 2.22 (m, 9H), 2.19 – 2.01 (m, 2H), 1.97 (dd, *J* = 9.2, 5.0 Hz, 1H), 1.73 (d, *J* = 2.8 Hz, 6H), 1.58 (dq, *J* = 9.1, 7.3 Hz, 1H).

**<sup>13</sup>C NMR** (75 MHz, CDCl<sub>3</sub>) δ 170.92, 158.22, 142.04, 132.87, 128.67, 128.46, 127.35, 125.91, 113.94, 80.79, 55.46, 41.66, 36.36, 35.91, 30.98, 30.71, 30.66, 29.70, 28.97.

**HRMS** (ESI) calculated for C<sub>29</sub>H<sub>34</sub>NaO<sub>3</sub> [M+Na]<sup>+</sup> *m/z*: 453.2400, found: 453.2397.

*cis-isomer*:

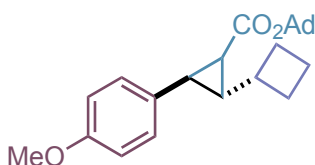
**IR** 2908, 2851, 1718, 1514, 1246, 1173, 1058, 1037.

**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>) δ 7.37 – 7.29 (m, 2H), 7.28 – 7.22 (m, 3H), 7.20 – 7.11 (m, 2H), 6.88 – 6.79 (m, 2H), 3.82 (s, 3H), 2.85 (t, *J* = 7.6 Hz, 2H), 2.32 (dd, *J* = 9.3, 6.6 Hz,

1H), 2.12 – 2.04 (m, 3H), 2.03 – 1.95 (m, 1H), 1.92 (m, 1H), 1.91 – 1.82 (m, 6H), 1.80 – 1.69 (m, 2H), 1.60 (m, 6H).

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 169.91, 158.35, 141.89, 130.46, 129.21, 128.63, 128.52, 126.03, 113.41, 80.19, 55.43, 41.23, 36.28, 35.57, 34.95, 32.27, 30.85, 30.06, 24.79.  
HRMS (ESI) calculated for C<sub>29</sub>H<sub>34</sub>NaO<sub>3</sub> [M+Na]<sup>+</sup> m/z: 453.2400, found: 453.2379.

**Adamantan-1-yl (2R\*,3R\*)-2-cyclobutyl-3-(4-methoxyphenyl)cyclopropane-1-carboxylate 3i**



Prepared according to **General procedure F** using 1-(2-cyclobutylvinyl)-4-methoxybenzene **1i** (38 mg, 0.2 mmol, 1 equiv) and adamantan-1-yl 2,2-diiodoacetate **2a**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided the corresponding cyclopropanes (42 mg, 55%). Ratio of isomers was determined to be 1:1.

*trans-isomer:*

IR 2910, 2851, 1715, 1515, 1454, 1247, 1058, 825.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.12 – 6.90 (m, 2H), 6.92 – 6.71 (m, 2H), 3.78 (s, 3H), 2.59 – 2.47 (m, 1H), 2.34 (dd, *J* = 6.5, 5.0 Hz, 1H), 2.18 – 2.13 (m, 4H), 2.12 (d, *J* = 3.0 Hz, 6H), 2.05 – 1.97 (m, 1H), 1.90 – 1.74 (m, 5H), 1.66 (d, *J* = 3.2 Hz, 6H), 1.62 – 1.53 (m, 1H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 170.81, 158.21, 133.07, 127.45, 113.97, 80.53, 55.48, 41.57, 36.91, 36.39, 34.29, 31.00, 30.46, 29.94, 28.76, 28.59, 18.63.

HRMS (ESI) calculated for C<sub>25</sub>H<sub>32</sub>NaO<sub>3</sub> [M+Na]<sup>+</sup> m/z: 403.2244, found: 403.2247.

<sup>1</sup>H-<sup>13</sup>C HSQC and HMBC spectra were measured.

*cis-isomer:*

IR 2909, 2851, 1722, 1514, 1246, 1160, 1056.

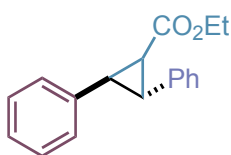
<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 7.21 – 7.08 (m, 2H), 6.93 – 6.68 (m, 2H), 3.78 (s, 3H), 2.39 – 2.25 (m, 2H), 2.02 (m, 6H), 1.92 – 1.75 (m, 10H), 1.73 (dd, *J* = 9.4, 5.2 Hz, 1H), 1.55 (m, 6H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 170.13, 158.35, 130.56, 129.41, 113.46, 80.15, 55.45, 41.29, 37.30, 36.30, 30.88, 30.01, 29.65, 27.95, 27.01, 26.66, 18.32.

HRMS (ESI) calculated for C<sub>25</sub>H<sub>32</sub>NaO<sub>3</sub> [M+Na]<sup>+</sup> m/z: 403.2244, found: 403.2247.

<sup>1</sup>H-<sup>13</sup>C HSQC and HMBC spectra were measured.

**Ethyl (2R\*,3R\*)-2,3-diphenylcyclopropane-1-carboxylate 3j<sup>4</sup>**

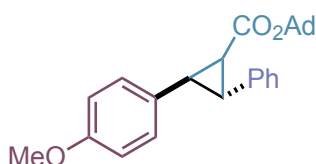


Prepared according to **General procedure G** using *trans*-stilbene **1j** (72 mg, 0.4 mmol, 2 equiv) and ethyl 2,2-diiodoacetate **2b**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided the corresponding cyclopropane (30 mg, 56%). Data are consistent with those reported on the literature. Note: reaction performed with *cis*-stilbene gave the same stereoisomer with lower yield (15%)

**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>) δ 7.40 – 7.14 (m, 10H), 3.97 (q, *J* = 7.1 Hz, 2H), 3.24 (dd, *J* = 7.0, 5.3 Hz, 1H), 2.95 (dd, *J* = 9.6, 7.1 Hz, 1H), 2.44 (dd, *J* = 9.6, 5.2 Hz, 1H), 1.06 (t, *J* = 7.1 Hz, 3H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 173.56, 137.68, 133.51, 132.43, 128.32, 127.77, 127.55, 126.41, 125.64, 124.93, 124.72, 60.91, 26.56, 24.29, 17.18, 14.43.

### Adamantan-1-yl(2*R*\*,3*R*\*)-2-(4-methoxyphenyl)-3-phenylcyclopropane-1-carboxylate **3k**



Prepared according to **General procedure F** using (*E*)-1-methoxy-4-styrylbenzene **1k** (42 mg, 0.2 mmol, 1 equiv) and adamantan-1-yl 2,2-diiodoacetate **2a**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided a mixture the corresponding cyclopropanes (37 mg, 46%). Ratio of isomers was determined to be 1:1 (*trans*:*cis*).

*trans* and *cis*-isomer:

**IR**: 2907, 2850, 1716, 1514, 1246, 1172, 1055, 907.

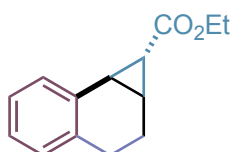
**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.40 – 7.32 (m, 5H), 7.32 – 7.22 (m, 7H), 7.22 – 7.15 (m, 2H), 6.92 – 6.83 (m, 4H), 3.83 (s, 3H), 3.82 (s, 3H), 3.09 (ddd, *J* = 9.8, 6.9, 5.2 Hz, 2H), 2.84 (ddd, *J* = 9.6, 6.8, 2.4 Hz, 2H), 2.30 (ddd, *J* = 14.8, 9.6, 5.2 Hz, 2H), 2.08 (m, 6H), 1.98 – 1.81 (m, 12H), 1.59 (m, 12H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 169.10, 169.03, 158.59, 158.51, 140.26, 136.75, 132.11, 130.44, 129.51, 128.66 (2C), 128.13, 127.93, 126.83, 126.72, 126.60, 114.13, 113.63, 80.74, 80.71, 55.49, 55.44, 41.31, 41.23, 36.28, 36.25, 34.22, 33.93, 32.39, 32.22, 30.90, 30.88, 29.19, 28.29.

**HRMS** (ESI) calculated for C<sub>27</sub>H<sub>30</sub>NaO<sub>3</sub> [M+Na]<sup>+</sup> *m/z*: 425.2087, found: 425.2084.

**<sup>1</sup>H-<sup>13</sup>C HSQC, HMBC** and **<sup>1</sup>H-<sup>1</sup>H NOESY** spectra were measured.

### Ethyl 1*a*,2,3,7*b*-tetrahydro-1*H*-cyclopropa[*a*]naphthalene-1-carboxylate **3l**<sup>6</sup>



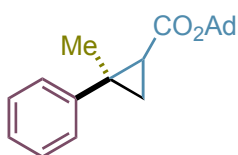
Prepared according to **General procedure G** using 1,2-dihydronaphthalene **1l** (50 mg, 0.4 mmol, 2 equiv) and ethyl 2,2-diiodoacetate **2b**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided the corresponding *trans*-cyclopropanes (21 mg, 48%). Data are consistent with those reported on literature.

**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>) δ 7.29 (dd, *J* = 7.0, 2.0 Hz, 1H), 7.19 – 7.07 (m, 2H), 7.05 – 6.99 (m, 1H), 4.16 (q, *J* = 7.1 Hz, 2H), 2.66 (dd, *J* = 16.3, 5.8 Hz, 1H), 2.55 (dd, *J* = 9.0, 3.5 Hz, 1H), 2.52 – 2.38 (m, 1H), 2.19 (m, 2H), 2.07 (dd, *J* = 4.5, 3.5 Hz, 1H), 1.80 (tdd, *J* = 14.2, 5.8, 3.4 Hz, 1H), 1.27 (t, *J* = 7.2 Hz, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 173.37, 135.21, 133.91, 128.98, 128.75, 126.39, 126.12, 60.72, 26.53, 25.50, 24.41, 23.27, 18.71, 14.42.

**<sup>1</sup>H-<sup>13</sup>C HSQC** and **HMBC** spectra were measured.

### Adamantan-1-yl 2-methyl-2-phenylcyclopropane-1-carboxylate **3m**



Prepared according to **General procedure G** using prop-1-en-2-ylbenzene **1m** (47 mg, 0.4 mmol, 2 equiv) and adamantan-1-yl 2,2-diiodoacetate **2a**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided a mixture the corresponding cyclopropanes (56 mg, 90%). Ratio of isomers was determined to be 1:1.

*trans* and *cis*-isomer:

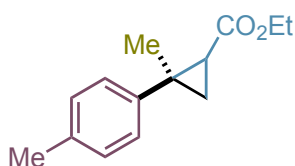
**IR** 2928, 1697, 1375, 1514, 1301, 710.

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.35 – 7.26 (m, 8H), 7.23 (m, 2H), 2.22 – 2.14 (m, 12H), 2.04 (m, 3H), 1.92 (dd, *J* = 8.3, 6.1 Hz, 1H), 1.88 – 1.64 (m, 11H), 1.61 – 1.55 (m, 6H), 1.54 (s, 3H), 1.47 (s, 3H), 1.41 – 1.33 (m, 2H), 1.09 (dd, *J* = 7.7, 4.5 Hz, 1H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 171.23, 170.33, 146.40, 142.31, 129.17, 128.53, 128.23, 127.29, 126.61, 126.41, 80.68, 80.03, 41.70, 41.14, 36.41, 36.27, 31.88, 31.01, 30.84, 29.91, 29.79, 29.46, 28.85, 20.54, 19.74, 19.38.

**HRMS** (ESI) calculated for C<sub>21</sub>H<sub>26</sub>NaO<sub>2</sub> [M+Na]<sup>+</sup> *m/z*: 333.1825, found: 333.1825.

### Ethyl (2*R*)-2-methyl-2-(*p*-tolyl)cyclopropane-1-carboxylate **3n**



Prepared according to **General procedure F** using 1-methyl-4-(prop-1-en-2-yl)benzene **1n** (26 mg, 0.2 mmol, 1 equiv) and ethyl 2,2-diiodoacetate **2b**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided the corresponding cyclopropanes (40 mg, 91%). Ratio of isomers was determined to be 1:1.

*trans-isomer:*

**IR** 2925, 1724, 1381, 1175, 817.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.23 – 7.17 (m, 2H), 7.14 – 7.08 (m, 2H), 4.19 (qd, *J* = 7.1, 3.1 Hz, 2H), 2.32 (s, 3H), 1.94 (dd, *J* = 8.3, 6.0 Hz, 1H), 1.51 (s, 3H), 1.44 – 1.37 (m, 2H), 1.30 (t, *J* = 7.1 Hz, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 172.44, 143.18, 136.20, 129.25, 127.33, 60.61, 30.46, 28.01, 21.11, 20.98, 20.11, 14.57.

**HRMS** (ESI) calculated for C<sub>14</sub>H<sub>18</sub>NaO<sub>2</sub> [M+Na]<sup>+</sup> *m/z*: 241.1195, found: 241.1199.

**<sup>1</sup>H-<sup>13</sup>C HSQC** and **HMBC** spectra were measured.

*cis-isomer:*

**IR** 2957, 2923, 1730, 1516, 1162.

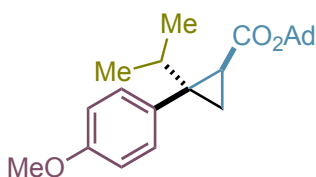
**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.19 – 7.12 (m, 2H), 7.10 – 7.04 (m, 2H), 3.86 (qd, *J* = 7.1, 4.7 Hz, 2H), 2.30 (d, *J* = 0.8 Hz, 3H), 1.88 (dd, *J* = 7.8, 5.4 Hz, 1H), 1.76 – 1.72 (m, 1H), 1.44 (s, 3H), 1.12 (dd, *J* = 7.8, 4.6 Hz, 1H), 0.99 (t, *J* = 7.1 Hz, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 171.51, 138.98, 136.25, 129.03, 128.75, 60.23, 31.91, 28.79, 28.62, 21.24, 19.68, 14.19.

**HRMS** (ESI) calculated for C<sub>14</sub>H<sub>18</sub>NaO<sub>2</sub> [M+Na]<sup>+</sup> *m/z*: 241.1195, found: 241.1191.

**<sup>1</sup>H-<sup>13</sup>C HSQC** and **HMBC** spectra were measured.

### Adamantan-1-yl-2-isopropyl-2-(4-methoxyphenyl)cyclopropane-1-carboxylate **3o**



Prepared according to **General procedure G** using 1-methoxy-4-(3-methylbut-1-en-2-yl)benzene **1o** (69 mg, 0.4 mmol, 2 equiv) and adamantan-1-yl 2,2-diiodoacetate **2a**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 50:1) provided the corresponding cyclopropanes (55 mg, 75%). Ratio of isomers was determined to be 1:25 (*trans:cis*).

*trans-isomer:*

**IR** 2911, 2852, 1719, 1513, 1172, 1061.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.24 – 7.15 (m, 2H), 6.85 – 6.77 (m, 2H), 3.79 (s, 3H), 2.18 (s, 6H), 1.96 – 1.89 (m, 1H), 1.87 (dd, *J* = 8.0, 5.8 Hz, 1H), 1.69 (m, 3H), 1.55 (s, 6H), 1.28 (dd, *J* = 5.8, 4.3 Hz, 1H), 1.16 (dd, *J* = 8.0, 4.3 Hz, 1H), 0.86 (d, *J* = 6.9 Hz, 3H), 0.80 (d, *J* = 6.8 Hz, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 171.78, 158.42, 133.78, 132.42, 113.01, 80.43, 55.37, 41.62, 40.92, 36.45, 31.03, 30.18, 29.85, 28.78, 20.39, 20.26.

**HRMS** (ESI) calculated for C<sub>24</sub>H<sub>32</sub>NaO<sub>3</sub> [M+Na]<sup>+</sup> *m/z*: 391.2244, found: 391.2241.

**<sup>1</sup>H-<sup>13</sup>C HSQC**, **HMBC** and **<sup>1</sup>H-<sup>1</sup>H NOESY** spectra were measured.

*cis-isomer:*

**IR** 2907, 1717, 1512, 1244, 1168, 1056.

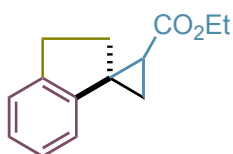
**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>) δ 7.09 (d, *J* = 8.8 Hz, 2H), 6.79 (d, *J* = 8.7 Hz, 2H), 3.78 (s, 3H), 2.06 (s, 3H), 1.89 (s, 6H), 1.75 (dd, *J* = 7.8, 5.4 Hz, 1H), 1.58 (d, *J* = 3.4 Hz, 6H), 1.50 – 1.44 (m, 1H), 1.24 – 1.10 (m, 1H), 1.03 (dd, *J* = 7.8, 4.2 Hz, 1H), 0.95 (d, *J* = 6.8 Hz, 3H), 0.80 (d, *J* = 6.7 Hz, 3H).

**<sup>13</sup>C NMR** (75 MHz, CDCl<sub>3</sub>) δ 170.64, 158.37, 132.30, 129.99, 112.95, 79.92, 55.31, 41.33, 40.88, 38.89, 36.33, 30.88, 28.56, 20.17, 19.59, 19.54.

**HRMS** (ESI) calculated for C<sub>24</sub>H<sub>32</sub>NaO<sub>3</sub> [M+Na]<sup>+</sup> *m/z*: 391.2244, found: 391.2247.

**<sup>1</sup>H-<sup>13</sup>C HSQC, HMBC and <sup>1</sup>H-<sup>1</sup>H NOESY** spectra were measured.

### Ethyl 2',3'-dihydrospiro[cyclopropane-1,1'-indene]-2-carboxylate **3p**



Prepared according to **General procedure G** using 1-methylene-2,3-dihydro-1*H*-indene **1p** (52 mg, 0.4 mmol, 2 equiv) and ethyl 2,2-diiodoacetate **2b**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided a mixture of the corresponding cyclopropanes (17 mg, 39%). Ratio of isomers was determined to be 1:1. Data are consistent with those reported in the literature.

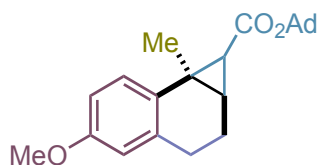
*trans* and *cis*-isomer:

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.25 – 7.06 (m, 7H), 6.73 – 6.69 (m, 1H), 4.17 (q, *J* = 7.2 Hz, 2H, *trans*), 4.07 – 3.88 (m, 2H, *cis*), 3.16 – 2.96 (m, 3H), 2.90 (dd, *J* = 14.9, 8.3 Hz, 1H, *cis*), 2.44 – 2.34 (m, 1H, *cis*), 2.33 – 2.21 (m, 2H, *trans*), 2.12 – 2.07 (m, 1H, *cis*), 2.03 (dd, *J* = 8.4, 6.1 Hz, 1H, *trans*), 1.92 (dd, *J* = 12.7, 8.1 Hz, 1H, *cis*), 1.90 – 1.85 (m, 1H, *cis*), 1.70 – 1.63 (m, 1H, *trans*), 1.42 (m, 2H), 1.26 (t, *J* = 7.2 Hz, 3H, *trans*), 1.12 (t, *J* = 7.2 Hz, 3H, *cis*).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 172.07, 170.69, 145.81, 145.72, 144.35, 141.60, 126.88, 126.80, 126.78, 125.83, 124.66, 124.15, 123.31, 118.79, 60.58, 60.47, 38.09, 37.98, 37.02, 31.44, 30.65, 30.62, 29.82, 29.61, 22.47, 18.39, 14.53, 14.33.

**<sup>1</sup>H-<sup>13</sup>C HSQC, HMBC and <sup>1</sup>H-<sup>1</sup>H NOESY** spectra were measured.

### Adamantan-1-yl 5-methoxy-7b-methyl-1a,2,3,7b-tetrahydro-1*H*-cyclopropa[*a*]naphthalene-1-carboxylate **3q**



Prepared according to **General procedure G** using 7-methoxy-4-methyl-1,2-dihydronaphthalene **1q** (70 mg, 0.4 mmol, 2 equiv) and adamantan-1-yl 2,2-diiodoacetate **2a**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 50:1) provided the corresponding cyclopropanes (32 mg, 60%). Ratio of isomers was determined to be 1:1.

*trans-isomer:*

**IR** 2907, 2820, 1710, 1498, 1244, 1169.

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.37 (d, *J* = 8.5 Hz, 1H), 6.76 (dd, *J* = 8.5, 2.7 Hz, 1H), 6.60 (d, *J* = 2.6 Hz, 1H), 3.78 (s, 3H), 2.72 – 2.59 (m, 1H), 2.57 – 2.47 (m, 1H), 2.15 (s, 3H), 2.11 (d, *J* = 2.5 Hz, 6H), 2.11 – 2.04 (m, 1H), 1.99 – 1.93 (m, 1H), 1.89 (d, *J* = 9.9 Hz, 1H), 1.84 (tdd, *J* = 12.8, 5.6, 3.2 Hz, 1H), 1.67 (m, 6H), 1.60 (s, 3H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 171.08, 157.56, 135.88, 132.48, 127.31, 114.40, 111.71, 80.55, 55.41, 41.66, 36.39, 30.98, 30.19, 29.61, 26.79, 26.59, 18.75, 17.17.

**HRMS** (ESI) calculated for C<sub>24</sub>H<sub>30</sub>NaO<sub>3</sub> [M+Na]<sup>+</sup> *m/z*: 389.2087, found: 389.2089.

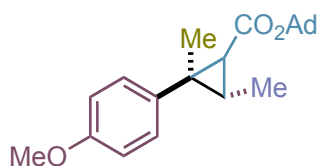
*cis-isomer:*

**IR** 2906, 2849, 1721, 1503, 1158, 1038, 814.

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.19 (d, *J* = 8.4 Hz, 1H), 6.72 (dd, *J* = 8.4, 2.2 Hz, 1H), 6.64 (d, *J* = 2.6 Hz, 1H), 3.77 (s, 3H), 2.81 – 2.72 (m, 1H), 2.46 (ddd, *J* = 14.9, 4.7, 2.9 Hz, 1H), 2.26 – 2.17 (m, 1H), 2.15 – 2.07 (m, 1H), 2.03 (s, 3H), 1.84 (d, *J* = 2.9 Hz, 6H), 1.72 (d, *J* = 8.5 Hz, 1H), 1.57 (d, *J* = 3.5 Hz, 1H), 1.55 (t, *J* = 2.8 Hz, 6H), 1.46 (s, 3H). **<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 169.89, 157.68, 140.29, 129.56, 112.92, 112.11, 79.97, 56.38, 55.29, 41.34, 36.31, 34.40, 30.86, 29.53, 27.52, 27.40, 25.01, 19.23.

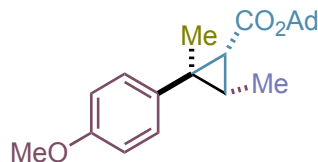
**HRMS** (ESI) calculated for C<sub>24</sub>H<sub>30</sub>NaO<sub>3</sub> [M+Na]<sup>+</sup> *m/z*: 389.2087, found: 389.2088.

### Adamantan-1-yl (2*R*\*,3*S*\*)-2-(4-methoxyphenyl)-2,3-dimethylcyclopropane-1-carboxylate **3r**



Prepared according to **General procedure F** using 1-(but-2-en-2-yl)-4-methoxybenzene **1r** (26 mg, 0.2 mmol, 1 equiv) and adamantan-1-yl 2,2-diiodoacetate **2a**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided the corresponding cyclopropanes (67 mg, 94%). Ratio of isomers was determined to be 1:1.

*trans-isomer:*



**IR** 2907, 2851, 1715, 1514, 1243, 1169, 1056.

**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>) δ 7.19 (d, *J* = 8.8 Hz, 2H), 6.82 (d, *J* = 8.8 Hz, 2H), 3.78 (s, 3H), 2.16 (m, 9H), 1.79 (d, *J* = 9.1 Hz, 1H), 1.67 (s, 6H), 1.58 (m, 1H), 1.46 (s, 3H), 1.35 (d, *J* = 6.5 Hz, 3H).

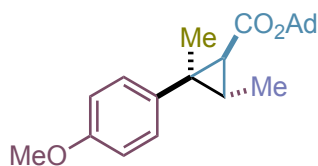
**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 170.98, 158.03, 140.84, 128.56, 113.85, 80.36, 55.44, 41.81, 36.45, 32.23, 31.01, 30.20, 25.91, 15.60, 8.46.

**HRMS** (ESI) calculated for C<sub>23</sub>H<sub>34</sub>NaO<sub>3</sub> [M+Na]<sup>+</sup> *m/z*: 377.2087, found: 377.208.

**<sup>1</sup>H-<sup>1</sup>H NOESY** spectrum was measured.



*cis-isomer:*



**IR** 2907, 2850, 1719, 1513, 1246, 1058, 830.

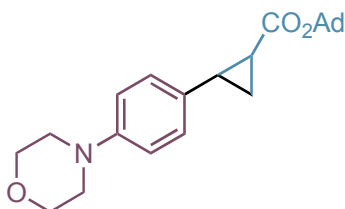
**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>) δ 7.15 (d, *J* = 8.8 Hz, 2H), 6.80 (d, *J* = 8.8 Hz, 2H), 3.77 (s, 3H), 2.10 (m, 4H), 1.99 – 1.77 (m, 7H), 1.56 (s, 6H), 1.38 (s, 3H), 1.26 (d, *J* = 6.3 Hz, 3H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 170.79, 158.14, 136.49, 130.01, 113.66, 79.89, 55.37, 41.30, 36.53, 36.31, 35.70, 30.87, 24.27, 23.15, 12.77.

**HRMS** (ESI) calculated for C<sub>23</sub>H<sub>34</sub>NaO<sub>3</sub> [M+Na]<sup>+</sup> *m/z*: 377.2087, found: 377.2077.

**<sup>1</sup>H-<sup>1</sup>H NOESY** spectrum was measured.

### Adamantan-1-yl-2-(4-morpholinophenyl)cyclopropane-1-carboxylate **3u**



Prepared according to **General procedure F** using 4-(4-vinylphenyl)morpholine **1u** (76 mg, 0.2 mmol, 1 equiv) adamantan-1-yl-2,2-diiodoacetate **2a**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided the corresponding cyclopropanes (68 mg, 88%). Ratio of isomers was determined to be 1:1 (*trans:cis*).

*trans-isomer:*

**IR** 2907, 2850, 1712, 1177, 927.

**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>) δ 7.01 (d, *J* = 8.6 Hz, 2H), 6.83 (d, *J* = 8.8 Hz, 2H), 3.89 – 3.81 (m, 4H), 3.15 – 3.07 (m, 4H), 2.37 (ddd, *J* = 9.2, 6.5, 4.1 Hz, 1H), 2.17 (m, 3H), 2.11 (m, 6H), 1.74 (ddd, *J* = 8.4, 5.2, 4.1 Hz, 1H), 1.65 (m, 6H), 1.47 (ddd, *J* = 9.4, 5.3, 4.4 Hz, 1H), 1.17 (ddd, *J* = 8.4, 6.4, 4.4 Hz, 1H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 172.64, 150.08, 132.12, 127.10, 116.02, 80.65, 67.04, 49.75, 41.57, 36.35, 30.98, 25.41, 25.19, 16.85.

**HRMS** (ESI) calculated for C<sub>24</sub>H<sub>32</sub>NO<sub>3</sub> [M+H]<sup>+</sup> *m/z*: 382.2377, found: 382.2370.

*trans-isomer:*

**IR** 2908, 2850, 1716, 1518, 1177, 1057.

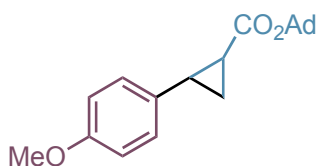
**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.17 (d, *J* = 8.5 Hz, 2H), 6.82 (d, *J* = 8.7 Hz, 2H), 3.87 – 3.82 (m, 4H), 3.13 – 3.08 (m, 4H), 2.45 (app. q, *J* = 8.5 Hz, 1H), 2.02 (m, 3H), 1.92 (ddd, *J* = 9.3, 7.7, 5.6 Hz, 1H), 1.86 – 1.73 (m, 6H), 1.57 (dd, *J* = 7.5, 5.3 Hz, 1H), 1.54 (d, *J* = 3.1 Hz, 6H), 1.19 (ddd, *J* = 8.6, 7.7, 4.9 Hz, 1H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 170.18, 150.19, 130.37, 128.73, 115.59, 80.14, 67.09, 49.93, 41.16, 36.29, 30.87, 24.66, 22.93, 10.88.

**HRMS** (ESI) calculated for C<sub>24</sub>H<sub>32</sub>NO<sub>3</sub> [M+H]<sup>+</sup> *m/z*: 382.2377, found: 382.2370.



### Adamantan-1-yl-2-(4-methoxyphenyl)cyclopropane-1-carboxylate **3v**



Prepared according to **General procedure F** using 1-methoxy-4-vinylbenzene **1v** (27 mg, 0.2 mmol, 1 equiv) and adamantan-1-yl 2,2-diiodoacetate **2a**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided the corresponding cyclopropanes (59 mg, 91%). Ratio of isomers was determined to be 1:1.

*trans-isomer:*

**IR** 2909, 2851, 1715, 1515, 1176, 1057.

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.02 (d, *J* = 8.3 Hz, 2H), 6.82 (d, *J* = 8.3 Hz, 2H), 3.78 (s, 3H), 2.40 (d, *J* = 9.8 Hz, 1H), 2.16 (s, 3H), 2.12 (s, 6H), 1.74 (dq, *J* = 8.6, 4.5 Hz, 1H), 1.66 (s, 6H), 1.47 (dt, *J* = 9.0, 4.5 Hz, 1H) 1.16 (m, 1H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 172.64, 141.37, 132.68, 127.39, 114.02, 80.72, 55.48, 41.58, 36.37, 31.00, 25.38, 25.20, 16.92.

**HRMS** (ESI) calculated for C<sub>21</sub>H<sub>26</sub>NaO<sub>3</sub> [M+Na]<sup>+</sup> *m/z*: 349.1774, found: 349.1783.

*cis-isomer:*

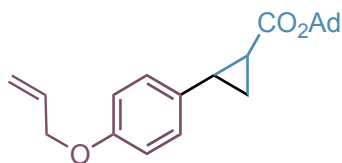
**IR** 2906, 2850, 1716, 1514, 1246, 1172, 1056.

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.22 – 7.15 (m, 2H), 6.85 – 6.78 (m, 2H), 3.78 (s, 3H), 2.46 (q, *J* = 8.6 Hz, 1H), 2.07 – 2.00 (m, 3H), 1.93 (ddd, *J* = 9.3, 7.7, 5.6 Hz, 1H), 1.88 – 1.76 (m, 6H), 1.60 – 1.50 (m, 7H), 1.21 (ddd, *J* = 8.6, 7.8, 4.9 Hz, 1H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 170.16, 158.42, 130.62, 129.19, 113.46, 80.23, 55.45, 41.22, 36.29, 30.87, 24.66, 22.81, 11.02.

**HRMS** (ESI) calculated for C<sub>21</sub>H<sub>26</sub>NaO<sub>3</sub> [M+Na]<sup>+</sup> *m/z*: 349.1774, found: 349.1771.

### Adamantan-1-yl 2-(4-(allyloxy)phenyl)cyclopropane-1-carboxylate **3w**



Prepared according to **General procedure F** using 1-(allyloxy)-4-vinylbenzene **1w** (32 mg, 0.2 mmol, 1 equiv) and adamantan-1-yl 2,2-diiodoacetate **1a**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided the corresponding cyclopropanes (51 mg, 72%). Ratio of isomers was determined to be 1:1.

*trans-isomer:*

**IR** 2909, 1715, 1513, 1244, 1177, 822.

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.04 – 6.97 (m, 2H), 6.89 – 6.79 (m, 2H), 6.04 (ddt, *J* = 17.2, 10.5, 5.3 Hz, 1H), 5.40 (dq, *J* = 17.3, 1.6 Hz, 1H), 5.28 (dq, *J* = 10.5, 1.4 Hz, 1H), 4.51 (dt, *J* = 5.3, 1.6 Hz, 2H), 2.38 (ddd, *J* = 9.2, 6.4, 4.1 Hz, 1H), 2.16 (m, 3H), 2.12 (m,

6H), 1.75 (ddd,  $J = 8.3, 5.3, 4.2$  Hz, 1H), 1.66 (m, 6H), 1.47 (ddd,  $J = 9.4, 5.3, 4.4$  Hz, 1H), 1.16 (ddd,  $J = 8.4, 6.5, 4.4$  Hz, 1H).

$^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  172.62, 157.37, 133.48, 132.87, 127.36, 117.76, 114.91, 80.71, 69.06, 41.58, 36.37, 31.00, 25.39, 25.20, 16.93.

**HRMS** (ESI) calculated for  $\text{C}_{23}\text{H}_{28}\text{NaO}_3$   $[\text{M}+\text{Na}]^+$   $m/z$ : 375,1931, found: 375,1929.

*cis-isomer:*

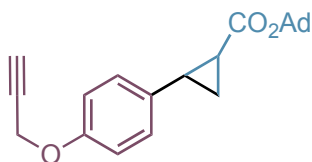
**IR:** 1909, 2851, 1721, 1512, 1178, 1057, 832.

$^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.23 – 7.14 (m, 2H), 6.88 – 6.79 (m, 2H), 6.04 (ddt,  $J = 17.3, 10.5, 5.3$  Hz, 1H), 5.39 (dq,  $J = 17.2, 1.7$  Hz, 1H), 5.26 (dq,  $J = 10.5, 1.5$  Hz, 1H), 4.51 (dt,  $J = 5.3, 1.6$  Hz, 2H), 2.45 (q,  $J = 8.5$  Hz, 1H), 2.02 (m, 3H), 1.93 (ddd,  $J = 9.3, 7.7, 5.6$  Hz, 1H), 1.87 – 1.74 (m, 6H), 1.55 (m, 7H), 1.20 (ddd,  $J = 8.6, 7.8, 4.9$  Hz, 1H).

$^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  170.13, 157.43, 133.60, 130.60, 129.38, 117.62, 114.34, 80.24, 69.00, 41.22, 36.29, 30.87, 24.64, 22.88, 10.96.

**HRMS** (ESI) calculated for  $\text{C}_{23}\text{H}_{28}\text{NaO}_3$   $[\text{M}+\text{Na}]^+$   $m/z$ : 375,1931, found: 375,1942.

### Adamantan-1-yl 2-(4-(prop-2-yn-1-yloxy)phenyl)cyclopropane-1-carboxylate 3x



Prepared according to **General procedure F** using 1-(prop-2-yn-1-yloxy)-4-vinylbenzene<sup>7</sup> **1x** (63 mg, 0.2 mmol, 1 equiv) and adamantan-1-yl-2,2-diiodoacetate **2a**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided a mixture of the corresponding cyclopropanes (47 mg, 67%). Ratio of isomers was determined to be 1:1.

*trans-isomer:*

**IR** 3289, 2907, 2850, 1710, 1512, 1774, 821.

$^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.03 (d,  $J = 8.7$  Hz, 2H), 6.89 (d,  $J = 8.7$  Hz, 2H), 4.66 (d,  $J = 2.4$  Hz, 2H), 2.50 (t,  $J = 2.4$  Hz, 1H), 2.39 (ddd,  $J = 9.2, 6.4, 4.1$  Hz, 1H), 2.19 – 2.14 (m, 3H), 2.12 (m, 6H), 1.75 (ddd,  $J = 8.4, 5.3, 4.2$  Hz, 1H), 1.66 (m, 6H), 1.48 (ddd,  $J = 9.5, 5.3, 4.4$  Hz, 1H), 1.17 (ddd,  $J = 8.4, 6.4, 4.4$  Hz, 1H).

$^{13}\text{C NMR}$  (126 MHz,  $\text{CDCl}_3$ )  $\delta$  172.55, 156.27, 133.71, 127.38, 115.09, 80.76, 78.73, 75.61, 56.04, 41.57, 36.35, 30.98, 25.35, 25.24, 16.95.

**HRMS** (ESI) calculated for  $\text{C}_{23}\text{H}_{26}\text{NaO}_3$   $[\text{M}+\text{Na}]^+$   $m/z$ : 373.1774, found: 373.1778.

*cis-isomer*

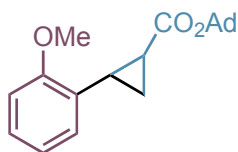
**IR** 3303, 2909, 2851, 1715, 1512, 1184, 907.

$^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.20 (d,  $J = 8.8$  Hz, 2H), 6.88 (d,  $J = 8.8$  Hz, 2H), 4.66 (d,  $J = 2.4$  Hz, 2H), 2.49 – 2.43 (m, 2H), 2.02 (m, 3H), 1.93 (ddd,  $J = 9.3, 7.7, 5.6$  Hz, 1H), 1.86 – 1.73 (m, 6H), 1.58 – 1.55 (m, 1H), 1.55 (m, 6H), 1.21 (ddd,  $J = 8.7, 7.7, 4.9$  Hz, 1H).

$^{13}\text{C NMR}$  (126 MHz,  $\text{CDCl}_3$ )  $\delta$  170.10, 156.38, 130.65, 130.23, 114.49, 80.29, 78.84, 75.41, 56.01, 41.20, 36.26, 30.86, 24.59, 22.88, 10.98.

**HRMS** (ESI) calculated for  $C_{23}H_{26}NaO_3$   $[M+Na]^+$   $m/z$ : 373.1774, found: 373.1763.

### Adamantan-1-yl 2-(2-methoxyphenyl)cyclopropane-1-carboxylate **3y**



Prepared according to **General procedure F** using 1-methoxy-2-vinylbenzene **1y** (54 mg, 0.2 mmol, 1 equiv) and adamantan-1-yl 2,2-diiodoacetate **2a**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided the corresponding cyclopropanes (27 mg, 41%). Ratio of isomers was determined to be 1:1.

*trans-isomer:*

**IR** 2907, 1716, 1456, 1246, 1179, 865.

**$^1H$  NMR** (400 MHz,  $CDCl_3$ )  $\delta$  7.21 – 7.12 (m, 1H), 6.90 – 6.81 (m, 3H), 3.84 (s, 3H), 2.66 (ddd,  $J = 9.2, 6.7, 4.4$  Hz, 1H), 2.17 (m, 3H), 2.14 (m, 6H), 1.77 (ddd,  $J = 8.3, 5.2, 4.4$  Hz, 1H), 1.67 (m, 6H), 1.47 (ddd,  $J = 9.3, 5.2, 4.2$  Hz, 1H), 1.21 (ddd,  $J = 8.3, 6.7, 4.2$  Hz, 1H).

**$^{13}C$  NMR** (101 MHz,  $CDCl_3$ )  $\delta$  173.01, 158.46, 128.96, 127.41, 125.89, 120.53, 110.56, 80.47, 55.66, 41.60, 36.40, 31.00, 24.00, 20.93, 15.80.

**HRMS** (ESI) calculated for  $C_{21}H_{26}NaO_3$   $[M+Na]^+$   $m/z$ : 349,1774, found: 349,1783.

*cis-isomer:*

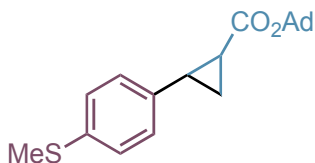
**IR** 2908, 1718, 1456, 1246, 1179, 1058.

**$^1H$  NMR** (400 MHz,  $CDCl_3$ )  $\delta$  7.23 – 7.16 (m, 2H), 6.89 (td,  $J = 7.5, 1.1$  Hz, 1H), 6.81 (dd,  $J = 8.1, 1.1$  Hz, 1H), 3.83 (s, 3H), 2.42 (q,  $J = 8.4$  Hz, 1H), 2.06 – 1.95 (m, 4H), 1.83 – 1.70 (m, 6H), 1.55 – 1.46 (m, 7H), 1.24 (ddd,  $J = 8.4, 7.9, 4.8$  Hz, 1H).

**$^{13}C$  NMR** (101 MHz,  $CDCl_3$ )  $\delta$  170.59, 159.15, 130.53, 127.80, 125.88, 120.08, 109.79, 55.49, 41.13, 36.29, 30.83, 22.21, 21.25, 11.02.

**HRMS** (ESI) calculated for  $C_{21}H_{26}NaO_3$   $[M+Na]^+$   $m/z$ : 349,1774, found: 349, 349,1770.

### Adamantan-1-yl 2-(4-(methylthio)phenyl)cyclopropane-1-carboxylate **3z**



Prepared according to **General procedure F** using methyl(4-vinylphenyl)sulfane **1z** (30 mg, 0.2 mmol, 1 equiv) adamantan-1-yl-2,2-diiodoacetate **2a**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided the corresponding cyclopropanes (50 mg, 73%). Ratio of isomers was determined to be 1:1 (*trans:cis*).

*trans-isomer:*

**IR** 2908, 2850, 1715, 1181, 1056, 814.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.18 (d, *J* = 8.4 Hz, 2H), 7.01 (d, *J* = 8.3 Hz, 2H), 2.46 (s, 3H), 2.39 (ddd, *J* = 9.2, 6.4, 4.1 Hz, 1H), 2.20 – 2.14 (m, 3H), 2.12 (m, 6H), 1.78 (ddd, *J* = 8.4, 5.3, 4.1 Hz, 1H), 1.72 – 1.60 (m, 6H), 1.50 (ddd, *J* = 9.1, 5.3, 4.4 Hz, 1H), 1.19 (ddd, *J* = 8.4, 6.4, 4.5 Hz, 1H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 172.38, 137.84, 136.21, 127.30, 126.81, 80.87, 41.56, 36.34, 30.99, 25.54, 25.46, 17.12, 16.48.

**HRMS** (ESI) calculated for C<sub>21</sub>H<sub>26</sub>NaO<sub>2</sub>S [M+Na]<sup>+</sup> *m/z*: 365.1546, found: 365.1542.

*cis-isomer:*

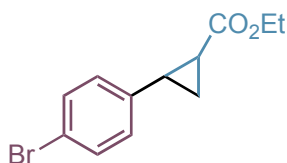
**IR** 2909, 2850, 1715, 1181, 1057, 813.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.19 (s, 4H), 2.45 (m, 4H), 2.02 (s, 3H), 1.96 (ddd, *J* = 9.3, 7.8, 5.6 Hz, 1H), 1.88 – 1.71 (m, 6H), 1.61 – 1.56 (m, 1H), 1.55 (s, 6H), 1.23 (ddd, *J* = 8.6, 7.7, 5.0 Hz, 1H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 169.98, 136.31, 134.30, 130.15, 126.82, 80.41, 41.20, 36.27, 30.86, 24.86, 22.96, 16.55, 10.96.

**HRMS** (ESI) calculated for C<sub>21</sub>H<sub>26</sub>NaO<sub>2</sub>S [M+Na]<sup>+</sup> *m/z*: 365.1546, found: 365.1528.

### Ethyl-2-(4-bromophenyl)cyclopropane-1-carboxylate **2aa**<sup>8</sup>



Prepared according to **General procedure G** using 4-bromostyrene **1aa** (73 mg, 0.4 mmol, 2 equiv) and ethyl 2,2-diiodoacetate **2b**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided the corresponding cyclopropanes (33 mg, 61%). Ratio of isomers was determined to be 1:0.7 (trans:cis). Data are consistent with those reported in the literature.

*trans-isomer:*

**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>) δ 7.41 (d, *J* = 8.4 Hz, 2H), 6.99 (d, *J* = 8.3 Hz, 2H), 4.19 (q, *J* = 7.1 Hz, 2H), 2.49 (ddd, *J* = 9.1, 6.5, 4.2 Hz, 1H), 1.88 (ddd, *J* = 8.4, 5.3, 4.2 Hz, 1H), 1.61 (ddd, *J* = 9.3, 5.3, 4.6 Hz, 1H), 1.30 (t, *J* = 7.1 Hz, 3H), 1.27 (m, 1H).

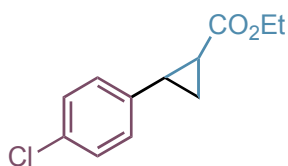
**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 173.26, 139.34, 131.65, 128.10, 120.30, 60.98, 25.70, 24.31, 17.14, 14.40.

*cis-isomer:*

**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>) δ 7.38 (d, *J* = 8.5 Hz, 2H), 7.14 (d, *J* = 8.2 Hz, 2H), 3.90 (q, *J* = 7.1 Hz, 2H), 2.50 (appar q, *J* = 8.6 Hz, 1H), 2.08 (ddd, *J* = 9.2, 7.9, 5.7 Hz, 1H), 1.67 (appar dt, *J* = 7.5, 5.4 Hz, 1H), 1.34 (ddd, *J* = 8.6, 7.8, 5.1 Hz, 1H), 1.02 (t, *J* = 7.1 Hz, 3H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 170.91, 135.79, 131.16, 131.12, 120.68, 60.50, 25.04, 21.96, 14.24, 11.45.

### Ethyl-2-(4-chlorophenyl)cyclopropane-1-carboxylate **3ab**<sup>8</sup>



Prepared according to **General procedure G** using 4-chlorostyrene **1ab** (55 mg, 0.4 mmol, 2 equiv) and ethyl 2,2-diiodoacetate **2b**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided the corresponding cyclopropanes (18 mg, 40%). Ratio of isomers was determined to be 1:0.8 (*trans*:*cis*). Data are consistent with those reported in the literature.

*trans*-isomer:

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.24 (d, *J* = 8.5 Hz, 2H), 7.03 (d, *J* = 8.4 Hz, 2H), 4.17 (qd, *J* = 7.1, 0.8 Hz, 2H), 2.49 (ddd, *J* = 10.5, 6.5, 4.2 Hz, 1H), 1.86 (ddd, *J* = 8.4, 5.3, 4.2 Hz, 1H), 1.60 (ddd, *J* = 9.2, 5.4, 4.6 Hz, 1H), 1.31 – 1.23 (m, 4H).

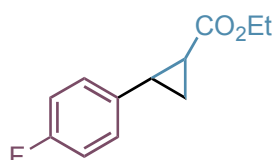
**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 173.30, 138.79, 132.34, 128.72, 127.73, 60.97, 25.64, 24.32, 17.16, 14.40.

*cis*-isomer:

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.25 – 7.16 (m, 4H), 3.90 (q, *J* = 7.1 Hz, 2H), 2.52 (q, *J* = 8.5 Hz, 1H), 2.08 (ddd, *J* = 9.2, 7.9, 5.6 Hz, 1H), 1.67 (dt, *J* = 7.5, 5.4 Hz, 1H), 1.34 (ddd, *J* = 8.7, 7.8, 5.1 Hz, 1H), 1.02 (t, *J* = 7.1 Hz, 3H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 170.93, 135.25, 132.56, 130.78, 128.18, 60.49, 24.97, 21.98, 14.24, 11.48.

### Ethyl-2-(4-fluorophenyl)cyclopropane-1-carboxylate **3ac**<sup>9</sup>



Prepared according to **General procedure G** using 4-fluorostyrene **1ac** (49 mg, 0.4 mmol, 2 equiv) and ethyl 2,2-diiodoacetate **2b**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided the corresponding cyclopropanes (15 mg, 36%). Ratio of isomers was determined to be 1:0.6 (*trans*:*cis*). Data are consistent with those reported in the literature.

*trans*-isomer:

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.00 – 6.93 (m, 2H), 6.90 – 6.83 (m, 2H), 4.07 (q, *J* = 7.1 Hz, 2H), 2.40 (ddd, *J* = 9.2, 6.5, 4.2 Hz, 1H), 1.74 (ddd, *J* = 8.4, 5.3, 4.2 Hz, 1H), 1.48 (ddd, *J* = 9.2, 5.3, 4.6 Hz, 1H), 1.18 (t, *J* = 7.1 Hz, 3H), 1.16 – 1.12 (m, 1H).

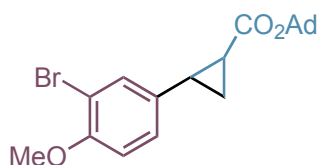
**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 173.42, 161.76 (d, *J* = 244.5 Hz), 135.88 (d, *J* = 3.2 Hz), 127.94 (d, *J* = 7.9 Hz), 115.43 (d, *J* = 21.6 Hz), 60.91, 25.57, 24.15, 17.03, 14.41.

*cis-isomer:*

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.15 – 7.08 (m, 2H), 6.88 – 6.81 (m, 2H), 3.79 (q, *J* = 7.1 Hz, 2H), 2.43 (q, *J* = 8.4 Hz, 1H), 1.96 (ddd, *J* = 9.2, 7.9, 5.6 Hz, 1H), 1.56 (dt, *J* = 7.4, 5.3 Hz, 1H), 1.23 (ddd, *J* = 8.7, 7.8, 5.1 Hz, 1H), 0.91 (t, *J* = 7.1 Hz, 3H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 171.01, 161.85 (d, *J* = 244.8 Hz), 132.40 (d, *J* = 3.2 Hz), 130.91 (d, *J* = 7.9 Hz), 114.88 (d, *J* = 21.4 Hz), 60.41, 24.84, 21.87, 14.23, 11.52.

### Adamantan-1-yl 2-(3-bromo-4-methoxyphenyl)cyclopropane-1-carboxylate **3ae**



Prepared according to **General procedure F** using 2-bromo-1-methoxy-4-vinylbenzene **1ad** (43 mg, 0.2 mmol, 1 equiv) and adamantan-1-yl-2,2-diiodoacetate **2a**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided the corresponding cyclopropanes (58 mg, 71%). Ratio of isomers was determined to be 1:1 (*trans:cis*).

*trans-isomer:*

**IR** 2909, 2851, 1714, 1501, 1190, 1056.

**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>) δ 7.26 (d, *J* = 1.9 Hz, 1H), 7.02 (dd, *J* = 8.5, 2.3 Hz, 1H), 6.80 (d, *J* = 8.5 Hz, 1H), 3.86 (s, 3H), 2.36 (ddd, *J* = 9.2, 6.4, 4.1 Hz, 1H), 2.16 (s, 3H), 2.12 (d, *J* = 2.9 Hz, 6H), 1.74 (ddd, *J* = 8.5, 5.3, 4.1 Hz, 1H), 1.66 (d, *J* = 3.0 Hz, 6H), 1.48 (ddd, *J* = 9.4, 5.3, 4.5 Hz, 1H), 1.15 (ddd, *J* = 8.5, 6.4, 4.5 Hz, 1H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 172.26, 154.65, 134.33, 131.23, 126.64, 112.02, 117.90, 80.94, 56.49, 41.57, 36.35, 31.00, 25.16, 24.79, 16.84.

**HRMS** (ESI) calculated for C<sub>21</sub>H<sub>25</sub>BrNaO<sub>3</sub> [M+Na]<sup>+</sup> *m/z*: 427.0879, found: 427.0879.

*cis-isomer:*

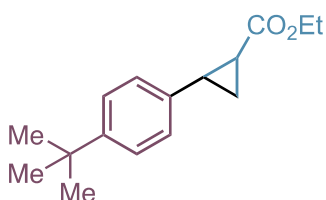
**IR** 2908, 2850, 1718, 1500, 1180, 1054.

**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>) δ 7.45 (dd, *J* = 2.2, 0.7 Hz, 1H), 7.17 (ddd, *J* = 8.4, 2.2, 0.8 Hz, 1H), 6.81 (d, *J* = 8.5 Hz, 1H), 3.87 (s, 3H), 2.43 (q, *J* = 8.4 Hz, 1H), 2.13 – 2.00 (m, 3H), 1.99 – 1.92 (m, 1H), 1.85 (m, 6H), 1.56 (t, *J* = 3.4 Hz, 6H), 1.55 – 1.48 (m, 1H), 1.22 (ddd, *J* = 8.7, 7.8, 5.0 Hz, 1H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 169.89, 154.73, 134.51, 130.99, 129.65, 111.53, 111.09, 80.62, 56.46, 41.31, 36.28, 30.91, 24.08, 22.82, 11.11.

**HRMS** (ESI) calculated for C<sub>21</sub>H<sub>25</sub>BrNaO<sub>3</sub> [M+Na]<sup>+</sup> *m/z*: 427.0879, found: 427.0879.

### Ethyl-2-(4-(*tert*-butyl)phenyl)cyclopropane-1-carboxylate **3af**<sup>10</sup>



Prepared according to **General procedure F** using 1 1-(*tert*-butyl)-4-vinylbenzene **1ae** (32 mg, 0.2 mmol, 1 equiv) and ethyl 2,2-diiodoacetate **2b**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided the corresponding cyclopropanes (37 mg, 75%). Ratio of isomers was determined to be 1:1. Data are consistent with those reported on the literature.

*trans*-isomer:

**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>) δ 7.31 (d, *J* = 8.4 Hz, 2H), 7.04 (d, *J* = 8.3 Hz, 2H), 4.16 (q, *J* = 7.0 Hz, 2H), 2.49 (ddd, *J* = 9.3, 6.5, 4.2 Hz, 1H), 1.88 (ddd, *J* = 8.4, 5.3, 4.2 Hz, 1H), 1.62 – 1.54 (m, 1H), 1.33 – 1.30 (m, 1H), 1.30 (s, 9H), 1.29 – 1.23 (t, *J* = 7.2, 3H).

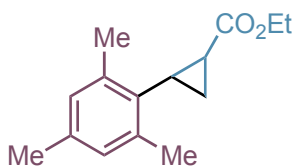
**<sup>13</sup>C NMR** (75 MHz, CDCl<sub>3</sub>) δ 173.68, 149.62, 137.26, 125.99, 125.53, 60.78, 34.56, 31.47, 26.00, 24.28, 17.10, 14.41.

*cis*-isomer:

**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>) δ 7.29 (d, *J* = 9.3 Hz, 2H), 7.21 (d, *J* = 8.5 Hz, 2H), 3.88 (m, 2H), 2.56 (appar. q, *J* = 8.6 Hz, 1H), 2.06 (ddd, *J* = 9.2, 7.8, 5.7 Hz, 1H), 1.71 (appar. dt, *J* = 7.5, 5.3 Hz, 1H), 1.33 (m, 1H), 1.30 (s, 9H), 1.00 – 0.88 (t, *J* = 7.2, Hz, 3H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 171.26, 149.54, 133.65, 129.06, 124.92, 60.25, 34.54, 31.49, 25.21, 21.99, 14.05, 11.28.

### Ethyl-2-mesitylcyclopropane-1-carboxylate **3ag**



Prepared according to **General procedure G** using 1,3,5-trimethyl-2-vinylbenzene **1af** (58 mg, 0.4 mmol, 2 equiv) ethyl 2,2-diiodoacetate **2b**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided the corresponding cyclopropanes (19 mg, 40%). Ratio of isomers was determined to be 1:1.1 (*trans*:*cis*).

*trans*-isomer:

**IR** 2969, 2923, 1724, 1179, 908.

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 6.83 (s, 2H), 4.29 – 4.16 (m, 2H), 2.34 (s, 6H), 2.30 (m, 1H), 2.25 (s, 3H), 1.71 (dt, *J* = 8.3, 4.9 Hz, 1H), 1.66 (ddd, *J* = 9.2, 5.1, 4.1 Hz, 1H), 1.31 (t, *J* = 7.1 Hz, 3H), 1.13 (ddd, *J* = 8.3, 7.1, 4.1 Hz, 1H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 174.49, 138.52, 136.51, 133.28, 129.04, 60.74, 23.33, 23.29, 20.94, 20.58, 17.73, 14.55.

**HRMS** (ESI) calculated for C<sub>15</sub>H<sub>20</sub>NaO<sub>2</sub> [M+Na]<sup>+</sup> *m/z*: 255.1356, found: 255.1345.

*cis*-isomer:

**IR** 2973, 2919, 1740, 1210, 715.

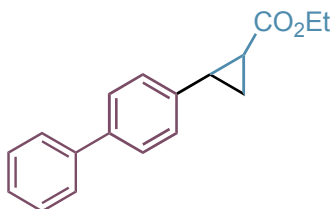
**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 6.78 (s, 2H), 3.92 (qd, *J* = 7.1, 1.6 Hz, 2H), 2.30 (s, 1H), 2.29 – 2.24 (m, 6H), 2.22 (d, *J* = 0.8 Hz, 3H), 2.14 (td, *J* = 8.2, 5.3 Hz, 1H), 1.62 (dt, *J* = 8.2, 5.0 Hz, 1H), 1.57 – 1.51 (m, 1H), 1.02 (t, *J* = 7.1 Hz, 3H).



$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  172.33, 138.30, 135.89, 130.67, 128.99, 60.24, 23.02, 21.21, 21.00, 20.63, 16.15, 14.09.

HRMS (ESI) calculated for  $\text{C}_{15}\text{H}_{20}\text{NaO}_2$   $[\text{M}+\text{Na}]^+$   $m/z$ : 255.1356, found: 255.255.1351.

### Ethyl-2-([1,1'-biphenyl]-4-yl)cyclopropane-1-carboxylate **3ag**<sup>11</sup>



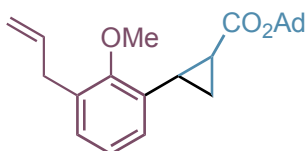
Prepared according to **General procedure F** using 1,3,5-trimethyl-2-vinylbenzene **1ah** (36 mg, 0.2 mmol, 1 equiv) ethyl 2,2-diiodoacetate **2b**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided a mixture the corresponding cyclopropanes (37 mg, 69%). Ratio of isomers was determined to be 3:2 (*trans*:*cis*). Data are consistent with those reported on the literature.

*trans* and *cis*-isomer:

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.57 (m, 3H), 7.51 (m, 4H), 7.48 – 7.38 (m, 4H), 7.38 – 7.31 (m, 4H), 7.17 (d,  $J$  = 8.2 Hz, 2H), 4.18 (q,  $J$  = 7.1 Hz, 2H), 3.96 – 3.85 (q,  $J$  = 7.1 Hz, 2H), 2.61 (m, 2H), 2.11 (ddd,  $J$  = 9.3, 7.8, 5.7 Hz, 1H), 1.94 (ddd,  $J$  = 8.4, 5.3, 4.2 Hz, 1H), 1.75 (dt,  $J$  = 7.5, 5.3 Hz, 1H), 1.63 (dt,  $J$  = 9.4, 4.9 Hz, 1H), 1.42 – 1.32 (m, 2H), 1.28 (t,  $J$  = 7.1 Hz, 3H), 0.99 (t,  $J$  = 7.1 Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  171.14, 141.12, 139.60, 135.87, 129.85, 128.84, 127.24, 127.15, 126.74, 60.40, 25.36, 22.13, 14.20, 11.44.

### Adamantan-1-yl 2-(3-allyl-2-methoxyphenyl)cyclopropane-1-carboxylate **3ai**



Prepared according to **General procedure G** using 1-allyl-2-methoxy-3-vinylbenzene **1ah** (80 mg, 0.4 mmol, 2 equiv) and adamantan-1-yl 2,2-diiodoacetate **2a**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided the corresponding cyclopropanes (45 mg, 62%). Ratio of isomers was determined to be 1:1.

*trans*-isomer:

IR 2909, 1717, 1179, 1056, 1009, 765.

$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.05 (dd,  $J$  = 7.6, 1.9 Hz, 1H), 6.98 (t,  $J$  = 7.5 Hz, 1H), 6.71 (dd,  $J$  = 7.5, 1.9 Hz, 1H), 6.06 – 5.90 (m, 1H), 5.15 – 5.01 (m, 2H), 3.79 (s, 3H), 3.44 (dt,  $J$  = 6.6, 1.6 Hz, 2H), 2.70 (ddd,  $J$  = 9.2, 6.7, 4.4 Hz, 1H), 2.16 (s, 3H), 2.12 (d,  $J$  = 2.9 Hz, 6H), 1.75 (ddd,  $J$  = 8.3, 5.3, 4.3 Hz, 1H), 1.66 (d,  $J$  = 3.1 Hz, 6H), 1.54 (ddd,  $J$  = 9.3, 5.2, 4.4 Hz, 1H), 1.25 (ddd,  $J$  = 8.3, 6.6, 4.4 Hz, 1H).



**<sup>13</sup>C NMR** (75 MHz, CDCl<sub>3</sub>) δ 172.49, 157.43, 137.40, 133.52, 133.25, 128.61, 124.27, 123.45, 115.95, 80.72, 61.28, 41.58, 36.35, 34.09, 30.98, 25.29, 20.80, 15.83.

**HRMS** (ESI) calculated for C<sub>24</sub>H<sub>30</sub>NaO<sub>3</sub> [M+Na]<sup>+</sup> m/z: 389.2087, found: 389.2086.

*cis-isomer:*

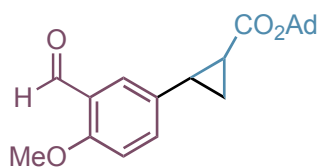
**IR** 2908, 1720, 1463, 1181, 1057, 1009.

**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>) δ 7.11 – 6.85 (m, 3H), 5.98 (ddt, *J* = 17.9, 9.2, 6.4 Hz, 1H), 5.10 – 4.96 (m, 2H), 3.81 (s, 3H), 3.42 (dt, *J* = 6.4, 1.6 Hz, 2H), 2.61 (q, *J* = 8.6 Hz, 1H), 2.11 – 1.99 (m, 1H), 2.00 (m, 3H), 1.85 – 1.69 (m, 6H), 1.65 (dt, *J* = 7.7, 5.3 Hz, 1H), 1.53 (m, 6H), 1.27 (ddd, *J* = 8.7, 7.8, 5.2 Hz, 1H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 170.12, 158.42, 137.70, 132.46, 129.77, 129.08, 128.47, 123.40, 115.62, 80.26, 61.06, 41.15, 36.27, 34.05, 30.85, 23.76, 21.09, 10.72.

**HRMS** (ESI) calculated for C<sub>24</sub>H<sub>30</sub>NaO<sub>3</sub> [M+Na]<sup>+</sup> m/z: 389.2087, found: 389.2088.

### Adamantan-1-yl 2-(3-formyl-4-methoxyphenyl)cyclopropane-1-carboxylate **3aj**



Prepared according to **General procedure G** using 2-methoxy-5-vinylbenzaldehyde **1ai** (65 mg, 0.4 mmol, 2 equiv) and adamantan-1-yl 2,2-diiodoacetate **2a**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 50:1) provided the corresponding cyclopropanes (40 mg, 56%). Ratio of isomers was determined to be 0.7:1 (*trans*:*cis*).

*trans-isomer:*

**IR**: 2909, 2851, 1715, 1682, 1500, 1253, 1178, 1056.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 10.43 (s, 1H), 7.52 (d, *J* = 2.5 Hz, 1H), 7.34 (dd, *J* = 8.6, 2.5 Hz, 1H), 6.91 (d, *J* = 8.6 Hz, 1H), 3.91 (s, 3H), 2.41 (ddd, *J* = 9.2, 6.4, 4.2 Hz, 1H), 2.17 (d, *J* = 4.5 Hz, 3H), 2.12 (d, *J* = 3.0 Hz, 6H), 1.78 (ddd, *J* = 8.5, 5.3, 4.2 Hz, 1H), 1.67 (q, *J* = 2.2 Hz, 6H), 1.50 (ddd, *J* = 9.2, 5.3, 4.5 Hz, 1H), 1.20 (ddd, *J* = 8.5, 6.4, 4.5 Hz, 1H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 189.82, 172.26, 160.72, 134.52, 133.06, 125.65, 124.77, 111.94, 80.96, 55.96, 41.57, 36.36, 31.01, 25.10, 24.90, 16.78.

**HRMS** (ESI) calculated for C<sub>22</sub>H<sub>26</sub>NaO<sub>4</sub> [M+Na]<sup>+</sup> m/z: 377.1723, found: 377.1725.

*cis-isomer:*

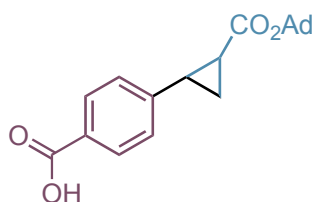
**IR**: 2908, 2850, 1716, 1682, 1498, 1253, 1178, 1057, 1025.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 10.43 (s, 1H), 7.74 (dd, *J* = 2.4, 0.8 Hz, 1H), 7.45 (ddd, *J* = 8.5, 2.4, 0.8 Hz, 1H), 6.91 (d, *J* = 8.6 Hz, 1H), 3.91 (s, 3H), 2.45 (td, *J* = 8.7, 7.3 Hz, 1H), 2.03 (t, *J* = 3.2 Hz, 3H), 1.97 (ddd, *J* = 9.2, 7.8, 5.6 Hz, 1H), 1.89 – 1.76 (m, 6H), 1.58 (m, 1H), 1.56 – 1.52 (m, 6H), 1.30 – 1.22 (m, 1H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 189.86, 170.01, 160.77, 137.04, 134.52, 129.85, 129.65, 111.37, 80.55, 55.90, 41.25, 36.25, 30.87, 24.28, 22.62, 11.27.

**HRMS** (ESI) calculated for C<sub>22</sub>H<sub>26</sub>NaO<sub>4</sub> [M+Na]<sup>+</sup> m/z: 377.1723, found: 377.1728.

#### 4-(2-(((adamantan-1-yl)oxy)carbonyl)cyclopropyl)benzoic acid **3ak**



Prepared according to **General procedure F** using 4-vinylbenzoic acid **1aj** (30 mg, 0.2 mmol, 1 equiv) and adamantan-1-yl-2,2-diiodoacetate **2a**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 10:1 to 4:1) provided a mixture of the corresponding cyclopropanes (26 mg, 38%). Ratio of isomers was determined to be 1:1.

*trans* and *cis*-isomer:

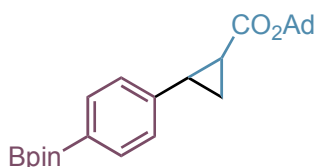
**IR** 2909, 2850, 1690, 1688, 1609, 1284, 1180, 1054.

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 8.02 (d, *J* = 4.9 Hz, 2H), 8.00 (d, *J* = 5.0 Hz, 2H), 7.38 (d, *J* = 8.1 Hz, 2H), 7.17 (d, *J* = 8.3 Hz, 2H), 2.55 (q, *J* = 8.5 Hz, 1H), 2.48 (ddd, *J* = 9.1, 6.3, 4.1 Hz, 1H), 2.18 – 2.15 (m, 6H), 2.13 (m, 6H), 2.09 – 2.04 (m, 1H), 1.91 (ddd, *J* = 8.5, 5.5, 4.1 Hz, 1H), 1.80 (dt, *J* = 14.5, 11.4 Hz, 6H), 1.72 – 1.64 (m, 7H), 1.60 (ddd, *J* = 9.1, 5.5, 4.6 Hz, 1H), 1.54 (d, *J* = 3.0 Hz, 6H), 1.34 – 1.30 (m, 1H), 1.30 – 1.27 (m, 1H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 171.91, 171.46, 171.27, 169.64, 147.36, 143.72, 130.54, 129.94, 129.81, 127.52, 127.39, 126.21, 81.24, 80.81, 41.56, 41.25, 36.33, 36.22, 31.01, 30.84, 26.17, 25.87, 25.30, 23.37, 17.86, 11.24.

**HRMS** (ESI) calculated for C<sub>21</sub>H<sub>23</sub>O<sub>4</sub> [M-H]<sup>-</sup> m/z: 339.1602. found: 339.1610.

#### Adamantan-1-yl-2-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)cyclopropane-1-carboxylate **3al**



Prepared according to **General procedure G** using 4,4,5,5-tetramethyl-2-(4-vinylphenyl)-1,3,2-dioxaborolane<sup>12</sup> **1ak** (92 mg, 0.4 mmol, 2 equiv) and adamantan-1-yl 2,2-diiodoacetate **2a**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 50:1 to 10:1) provided a mixture of the corresponding cyclopropanes (30 mg, 35%). Ratio of isomers was determined to be 1:1.

*trans* and *cis*-isomer:

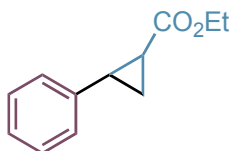
**IR** 2911, 2851, 1720, 1359, 1181, 1144, 1056.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.71 (d, *J* = 7.7 Hz, 4H), 7.28 (m, 2H), 7.08 (d, *J* = 8.1 Hz, 2H), 2.56 – 2.47 (m, 1H), 2.45 – 2.39 (m, 1H), 2.16 (m, 6H), 2.06 (m, *J* = 44.7 Hz, 12H), 1.98 – 1.93 (m, 1H), 1.83 (m, 1H), 1.77 (m, 6H), 1.66 (m, 6H), 1.62 (m, 1H), 1.48 (m, 1H), 1.33 (s, 24H), 1.19 – 1.09 (m, 2H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 172.34, 170.02, 144.13, 140.54, 135.07, 134.55, 129.13, 125.86, 125.58, 125.55, 83.88, 83.78, 80.88, 80.43, 41.56, 41.13, 36.35, 36.23, 30.99, 30.85, 29.85, 26.11, 25.78, 25.52, 25.06, 24.98, 24.88, 17.49.

**HRMS** (ESI) calculated for C<sub>26</sub>H<sub>35</sub>NaO<sub>4</sub><sup>11</sup>B [M+Na]<sup>+</sup> m/z: 445.2521, found: 445.2535.

### Ethyl 2-phenylcyclopropane-1-carboxylate **3am**<sup>13</sup>



Prepared according to **General procedure G** using styrene **1aI** (42 mg, 0.4 mmol, 2 equiv) and ethyl 2,2-diiodoacetate **2b**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided the corresponding cyclopropanes (26 mg, 67%). Ratio of isomers was determined to be 1:1. Data are consistent with those reported in the literature.

*trans*-isomer:

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.32 – 7.26 (m, 2H), 7.23 – 7.16 (m, 1H), 7.12 – 7.07 (m, 2H), 4.17 (q, *J* = 7.1 Hz, 2H), 2.52 (ddd, *J* = 9.2, 6.5, 4.1 Hz, 1H), 1.90 (ddd, *J* = 8.4, 5.3, 4.2 Hz, 1H), 1.60 (ddd, *J* = 9.2, 5.3, 4.5 Hz, 1H), 1.34 – 1.30 (m, 1H), 1.30 (t, *J* = 7.2, 3H).

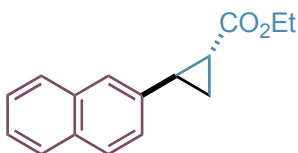
**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 173.58, 140.29, 128.62, 126.62, 126.33, 60.86, 26.32, 24.34, 17.22, 14.43.

*cis*-isomer:

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.25 (m, 5H), 3.86 (q, *J* = 7.1 Hz, 2H), 2.61 – 2.52 (m, 1H), 2.07 (ddd, *J* = 9.3, 7.8, 5.6 Hz, 1H), 1.71 (dt, *J* = 7.5, 5.4 Hz, 1H), 1.35 – 1.30 (m, 1H), 0.96 (t, *J* = 7.1 Hz, 3H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 171.14, 136.72, 129.45, 128.03, 126.78, 60.32, 25.61, 21.96, 14.16, 11.26.

### Ethyl-2-(naphthalen-2-yl)cyclopropane-1-carboxylate **3an**



Prepared according to **General procedure G** using 4-bromostyrene **1am** (62 mg, 0.4 mmol, 2 equiv) and ethyl 2,2-diiodoacetate **2b**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided the corresponding cyclopropanes (28 mg, 57%). Ratio of isomers was determined to be 3:1 (*trans*:*cis*).

*trans*-isomer:

**IR** 2977, 1722, 1368, 1181, 814.

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.78 (m, 3H), 7.57 (dd, *J* = 1.7, 0.9 Hz, 1H), 7.44 (dddd, *J* = 17.4, 8.2, 6.8, 1.4 Hz, 2H), 7.21 (dd, *J* = 8.5, 1.8 Hz, 1H), 4.20 (qd, *J* = 7.2, 0.9 Hz, 2H), 2.69 (ddd, *J* = 9.2, 6.5, 4.1 Hz, 1H), 2.01 (ddd, *J* = 8.4, 5.3, 4.1 Hz, 1H), 1.67 (ddd, *J* = 9.2, 5.3, 4.6 Hz, 1H), 1.43 (ddd, *J* = 8.4, 6.5, 4.6 Hz, 1H), 1.30 (t, *J* = 7.2 Hz, 3H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 173.56, 137.68, 133.51, 132.43, 128.32, 127.77, 127.55, 126.41, 125.64, 124.93, 124.72, 60.91, 26.56, 24.29, 17.18, 14.43.

**HRMS** (ESI) calculated for C<sub>16</sub>H<sub>16</sub>NaO<sub>2</sub> [M+Na]<sup>+</sup> *m/z*: 263.1043, found: 263.1045.

*cis-isomer*:

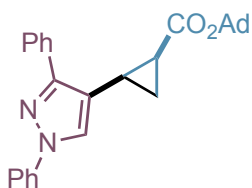
**IR** 2978, 1738, 1365, 1228.

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.81 – 7.76 (m, 3H), 7.75 – 7.71 (m, 2H), 7.47 – 7.37 (m, 2H), 3.82 (q, *J* = 7.1 Hz, 2H), 2.77 – 2.69 (m, 1H), 2.16 (ddd, *J* = 9.3, 7.8, 5.6 Hz, 1H), 1.85 (ddd, *J* = 7.5, 5.6, 5.1 Hz, 1H), 1.41 (ddd, *J* = 8.7, 7.8, 5.1 Hz, 1H), 0.91 (t, *J* = 7.1 Hz, 3H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 171.13, 134.26, 133.36, 132.57, 128.07, 127.80, 127.80, 127.73, 127.53, 126.01, 125.59, 60.35, 25.84, 22.15, 14.16, 11.50.

**HRMS** (ESI) calculated for C<sub>16</sub>H<sub>16</sub>NaO<sub>2</sub> [M+Na]<sup>+</sup> *m/z*: 263.1043, found: 263.1042.

### Adamantan-1-yl 2-(1,3-diphenyl-1*H*-pyrazol-4-yl)cyclopropane-1-carboxylate **3ao**



Prepared according to **General procedure G** using 1,3-diphenyl-4-vinyl-1*H*-pyrazole **1an** (99 mg, 0.4 mmol, 2 equiv) and adamantan-1-yl 2,2-diiodoacetate **2a**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided the corresponding cyclopropanes (64 mg, 73%). Ratio of isomers was determined to be 1:2 (trans:cis).

*trans-isomer*:

**IR**: 1909, 2851, 1714, 1503, 1191, 1156, 907.

**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>) δ 7.96 – 7.88 (m, 2H), 7.74 – 7.69 (m, 2H), 7.67 (d, *J* = 0.9 Hz, 1H), 7.49 – 7.41 (m, 4H), 7.41 – 7.33 (m, 1H), 7.31 – 7.23 (m, 1H), 2.43 (dddd, *J* = 8.9, 6.5, 4.3, 0.8 Hz, 1H), 2.19 (s, 3H), 2.15 (d, *J* = 2.8 Hz, 6H), 1.76 (ddd, *J* = 8.3, 5.1, 4.3 Hz, 1H), 1.69 (s, 6H), 1.59 – 1.54 (m, 1H), 1.19 (ddd, *J* = 8.3, 6.5, 4.2 Hz, 1H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 172.37, 152.00, 140.12, 133.34, 129.53, 128.64, 128.00, 127.69, 126.39, 125.81, 121.24, 118.95, 80.91, 41.60, 36.37, 31.01, 24.87, 17.46, 16.45.

**HRMS** (ESI) calculated for C<sub>29</sub>H<sub>31</sub>N<sub>2</sub>O<sub>2</sub> [M+Na]<sup>+</sup> *m/z*: 439.2380, found: 439.2378.

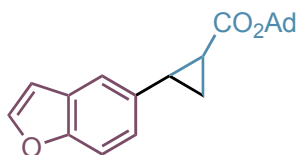
*cis-isomer*:

**IR** 2908, 2850, 1716, 1598, 1184, 1056.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.99 – 7.93 (m, 2H), 7.79 – 7.70 (m, 3H), 7.50 – 7.40 (m, 4H), 7.40 – 7.34 (m, 1H), 7.31 – 7.23 (m, 1H), 2.36 (q, *J* = 8.5 Hz, 1H), 2.12 (ddd, *J* = 9.0, 7.8, 5.7 Hz, 1H), 2.01 – 1.92 (m, 3H), 1.79 (m, 6H), 1.53 (dt, *J* = 7.2, 5.3 Hz, 1H), 1.48 (m, 6H), 1.35 (td, *J* = 8.2, 5.0 Hz, 1H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  169.45, 152.45, 140.13, 133.46, 129.32, 128.43, 127.80, 127.78, 127.72, 126.07, 118.84, 117.10, 80.50, 41.05, 36.04, 30.66, 23.78, 16.20, 12.18.  
HRMS (ESI) calculated for  $\text{C}_{29}\text{H}_{31}\text{N}_2\text{O}_2$   $[\text{M}+\text{Na}]^+$   $m/z$ : 439.2380, found: 439.2374.

### Adamantan-1-yl 2-(benzofuran-5-yl)cyclopropane-1-carboxylate 3ap



Prepared according to **General procedure G** using 5-vinylbenzofuran **1ao** (58 mg, 0.4 mmol, 2 equiv) and adamantan-1-yl 2,2-diiodoacetate **2a**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 50:1) provided the corresponding cyclopropanes (61 mg, 90%). Ratio of isomers was determined to be 1:1 (*trans*:*cis*).

*trans*-isomer:

IR 2908, 1720, 1385, 1172, 1057, 772.

$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.60 (d,  $J$  = 2.2 Hz, 1H), 7.40 (d,  $J$  = 8.5 Hz, 1H), 7.33 (d,  $J$  = 1.8 Hz, 1H), 7.04 (dd,  $J$  = 8.5, 1.9 Hz, 1H), 6.70 (dd,  $J$  = 2.2, 1.0 Hz, 1H), 2.54 (ddd,  $J$  = 9.2, 6.4, 4.2 Hz, 1H), 2.22 – 2.09 (m, 9H), 1.83 (ddd,  $J$  = 8.4, 5.3, 4.2 Hz, 1H), 1.72 – 1.61 (m, 6H), 1.56 – 1.51 (m, 1H), 1.26 (ddd,  $J$  = 8.4, 6.4, 4.4 Hz, 1H).

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  172.61, 153.99, 145.58, 135.12, 127.71, 123.04, 118.68, 111.29, 106.48, 80.78, 41.59, 36.37, 31.00, 26.04, 25.51, 17.25.

HRMS (ESI) calculated for  $\text{C}_{22}\text{H}_{24}\text{NaO}_3$   $[\text{M}+\text{Na}]^+$   $m/z$ : 359.1618, found: 359.1623.

*cis*-isomer:

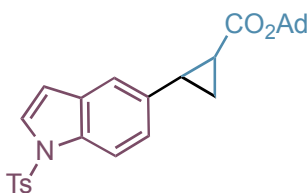
IR 2908, 2850, 1714, 1177, 1057, 874.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.58 (d,  $J$  = 2.2 Hz, 1H), 7.50 – 7.47 (m, 1H), 7.39 (d,  $J$  = 8.5 Hz, 1H), 7.22 (dd,  $J$  = 8.5, 1.8 Hz, 1H), 6.71 (dd,  $J$  = 2.3, 0.9 Hz, 1H), 2.62 (q,  $J$  = 8.5 Hz, 1H), 2.03 – 1.93 (m, 4H), 1.82 – 1.68 (m, 6H), 1.65 (dt,  $J$  = 7.4, 5.3 Hz, 1H), 1.54 – 1.44 (m, 6H), 1.27 (ddd,  $J$  = 8.7, 7.7, 4.9 Hz, 1H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  170.16, 154.10, 145.21, 131.64, 127.25, 126.29, 121.99, 110.72, 106.63, 80.25, 41.19, 36.24, 30.82, 25.31, 22.90, 11.30.

HRMS (ESI) calculated for  $\text{C}_{22}\text{H}_{24}\text{NaO}_3$   $[\text{M}+\text{Na}]^+$   $m/z$ : 359.1618, found: 359.162.

### Adamantan-1-yl 2-(1-tosyl-1H-indol-5-yl)cyclopropane-1-carboxylate 3aq



Prepared according to **General procedure G** using 1-tosyl-5-vinyl-1H-indole **1ap** (119 mg, 0.4 mmol, 2 equiv) and adamantan-1-yl 2,2-diiodoacetate **2a**. Purification by flash

chromatography on silica gel (gradient: hexane:EtOAc 50:1 to 10:1) provided the corresponding cyclopropanes ( 57 mg, 58%). Ratio of isomers was determined to be 1:1.

*trans-isomer:*

**IR** 2908, 2850, 1712, 1449, 1170, 1127, 1055, 558.

**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>) δ 7.87 (d, *J* = 8.6 Hz, 1H), 7.79 – 7.67 (m, 2H), 7.53 (d, *J* = 3.6 Hz, 1H), 7.24 – 7.15 (m, 3H), 7.03 (dd, *J* = 8.6, 1.8 Hz, 1H), 6.57 (dd, *J* = 3.7, 0.8 Hz, 1H), 2.48 (ddd, *J* = 9.2, 6.4, 4.2 Hz, 1H), 2.33 (s, 3H), 2.16 (s, 3H), 2.11 (m, 6H), 1.79 (ddd, *J* = 8.4, 5.3, 4.1 Hz, 1H), 1.65 (m, 6H), 1.51 (ddd, *J* = 9.5, 5.3, 4.5 Hz, 1H), 1.22 (ddd, *J* = 8.4, 6.5, 4.5 Hz, 1H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 172.44, 145.05, 135.81, 135.41, 133.74, 131.13, 130.01, 126.99, 126.91, 123.28, 118.80, 113.59, 108.98, 80.84, 41.57, 36.35, 30.99, 25.93, 25.57, 21.70, 17.23.

**HRMS** (ESI) calculated for C<sub>29</sub>H<sub>31</sub>NNaO<sub>4</sub>S [M+Na]<sup>+</sup> *m/z*: 512.1866, found: 512.1874.

*cis-isomer:*

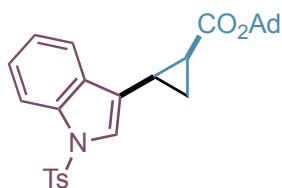
**IR** 2908, 2850, 1715, 1370, 1170, 1128, 1056.

**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>) δ 7.87 (d, *J* = 8.5 Hz, 1H), 7.78 – 7.69 (m, 2H), 7.51 (d, *J* = 3.7 Hz, 1H), 7.42 – 7.37 (m, 1H), 7.26 – 7.21 (m, 1H), 7.21 – 7.16 (m, 2H), 6.58 (dd, *J* = 3.7, 0.8 Hz, 1H), 2.56 (q, *J* = 8.5 Hz, 1H), 2.32 (s, 3H), 1.97 (ddd, *J* = 9.4, 7.7, 5.6 Hz, 1H), 1.89 (s, 3H), 1.74 – 1.59 (m, 7H), 1.43 (q, *J* = 12.6 Hz, 6H), 1.24 (ddd, *J* = 8.7, 7.6, 4.9 Hz, 1H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 170.03, 144.91, 135.61, 133.83, 132.28, 130.67, 129.91, 126.90, 126.72, 126.54, 122.12, 112.98, 109.19, 80.20, 41.08, 36.18, 30.76, 25.20, 22.98, 21.65, 11.09.

**HRMS** (ESI) calculated for C<sub>29</sub>H<sub>31</sub>NNaO<sub>4</sub>S [M+Na]<sup>+</sup> *m/z*: 512.1866, found: 512.1857.

### Adamantan-1-yl 2-(1-tosyl-1*H*-indol-3-yl)cyclopropane-1-carboxylate **3ar**



Prepared according to **General procedure F** using 1-tosyl-3-vinyl-1*H*-indole **1aq** (60 mg, 0.2 mmol, 1 equiv) and adamantan-1-yl 2,2-diiodoacetate **2a**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 50:1 to 10:1) provided the corresponding cyclopropanes ( 34 mg, 35%). Ratio of isomers was determined to be 1:3.

*trans-isomer:*

**IR** 2912, 2851, 1715, 1174, 1126.

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.95 (dt, *J* = 8.2, 0.9 Hz, 1H), 7.76 – 7.71 (d, *J* = 8.0 Hz, 2H), 7.56 (dt, *J* = 7.9, 1.0 Hz, 1H), 7.32 (ddd, *J* = 8.4, 7.2, 1.3 Hz, 1H), 7.26 – 7.23 (m, 2H), 7.22 (d, *J* = 8.0 Hz, 2H), 2.40 (dddd, *J* = 9.0, 6.4, 4.2, 1.2 Hz, 1H), 2.34 (s, 3H), 2.22 – 2.17 (m, 3H), 2.14 (m, 6H), 1.78 (ddd, *J* = 8.3, 5.1, 4.2 Hz, 1H), 1.72 – 1.62 (m, 6H), 1.50 (ddd, *J* = 9.2, 5.2, 4.2 Hz, 1H), 1.23 – 1.18 (m, 1H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 172.46, 145.06, 135.40, 135.36, 131.03, 130.03, 126.93, 125.11, 123.35, 122.82, 122.26, 119.70, 113.87, 81.04, 41.58, 36.35, 31.01, 23.17, 21.72, 16.76, 15.26.

**HRMS** (ESI) calculated for C<sub>29</sub>H<sub>31</sub>NNaO<sub>4</sub>S [M+Na]<sup>+</sup> m/z: 512.1866, found: 512.1864.

*cis-isomer:*

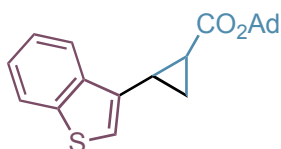
**IR** 2909, 2850, 1718, 1447, 1368, 1171, 1128.

**<sup>1</sup>H NMR** (300 MHz, CDCl<sub>3</sub>) δ 7.91 – 7.85 (m, 1H), 7.78 – 7.71 (m, 2H), 7.63 – 7.57 (m, 1H), 7.38 (d, *J* = 1.4 Hz, 1H), 7.30 – 7.16 (m, 3H), 2.39 – 2.32 (m, 1H), 2.31 (s, 3H), 2.07 (ddd, *J* = 9.1, 7.7, 5.7 Hz, 1H), 1.85 (s, 2H), 1.61 – 1.56 (m, 3H), 1.56 – 1.52 (m, 1H), 1.51 – 1.34 (m, 12H), 1.34 Z– 1.26 (m, 1H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 169.44, 144.62, 135.60, 135.11, 131.86, 129.84, 127.08, 125.19, 124.70, 123.06, 120.06, 119.17, 113.58, 80.20, 40.78, 36.13, 30.73, 21.80, 21.68, 15.21, 10.46.

**HRMS** (ESI) calculated for C<sub>29</sub>H<sub>31</sub>NNaO<sub>4</sub>S [M+Na]<sup>+</sup> m/z: 512.1866, found: 512.1868.

### Adamantan-1-yl 2-(benzo[*b*]thiophen-3-yl)cyclopropane-1-carboxylate **3as**



Prepared according to **General procedure G** using 3-vinylbenzo[*b*]thiophene **1ar** (64 mg, 0.4 mmol, 2 equiv) and adamantan-1-yl 2,2-diiodoacetate **2a**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided the corresponding cyclopropanes (27 mg, 38%). Ratio of isomers was determined to be 1:1.

*trans-isomer:*

**IR** 2908, 2850, 1715, 1350, 1178, 1056.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.90 – 7.81 (m, 2H), 7.43 (ddd, *J* = 8.0, 7.1, 1.3 Hz, 1H), 7.37 (td, *J* = 7.5, 7.1, 1.4 Hz, 1H), 7.01 (d, *J* = 1.1 Hz, 1H), 2.60 (dddd, *J* = 9.0, 6.4, 4.3, 1.2 Hz, 1H), 2.24 – 2.14 (m, 9H), 1.82 (ddd, *J* = 8.3, 5.1, 4.2 Hz, 1H), 1.71 – 1.61 (m, 6H), 1.59 – 1.53 (m, 1H), 1.28 (ddd, *J* = 7.5, 6.0, 3.7 Hz, 1H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 172.59, 140.54, 139.46, 135.78, 124.73, 124.29, 122.99, 122.03, 120.97, 80.94, 41.60, 36.37, 31.02, 23.20, 19.84, 15.20.

**HRMS** (ESI) calculated for C<sub>22</sub>H<sub>24</sub>NaO<sub>2</sub>S [M+Na]<sup>+</sup> m/z: 375.1389, found: 375.1389.

*cis-isomer:*

**IR** 2907, 2849, 1718, 1270, 1182, 1056.

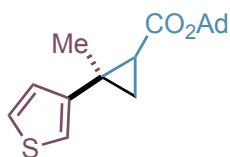
**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.91 (ddd, *J* = 7.9, 1.4, 0.7 Hz, 1H), 7.82 (ddd, *J* = 7.8, 1.3, 0.7 Hz, 1H), 7.40 – 7.35 (m, 1H), 7.32 (td, *J* = 7.6, 7.2, 1.1 Hz, 1H), 7.16 (d, *J* = 1.4 Hz, 1H), 2.51 (dddd, *J* = 9.2, 8.5, 7.2, 1.4 Hz, 1H), 2.11 (ddd, *J* = 9.1, 7.8, 5.6 Hz, 1H), 1.93 – 1.83 (m, 3H), 1.66 (ddd, *J* = 7.2, 5.6, 4.8 Hz, 1H), 1.54 – 1.50 (m, 6H), 1.50 – 1.39 (m, 6H), 1.36 (ddd, *J* = 8.4, 7.9, 4.8 Hz, 1H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 169.82, 140.24, 139.96, 132.77, 124.39, 124.09, 124.02, 122.77, 122.37, 80.23, 40.79, 36.17, 30.75, 21.87, 18.72, 10.87.

**HRMS** (ESI) calculated for C<sub>22</sub>H<sub>24</sub>NaO<sub>2</sub>S [M+Na]<sup>+</sup> m/z: 375.1389, found: 375.1400.



### Adamantan-1-yl-2-methyl-2-(thiophen-3-yl)cyclopropane-1-carboxylate **3at**



Prepared according to **General procedure G** using 3-(prop-1-en-2-yl)thiophene **1as** (50 mg, 0.4 mmol, 2 equiv) and adamantan-1-yl 2,2-diiodoacetate **2a**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 50:1) provided the corresponding cyclopropanes (45 mg, 71%). Ratio of isomers was determined to be 1:1.

*trans-isomer:*

**IR** 2906, 2850, 1714, 1259, 1174, 1050, 775.

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.24 (dd, *J* = 5.0, 3.0 Hz, 1H), 6.99 (dd, *J* = 3.0, 1.4 Hz, 1H), 6.84 (dd, *J* = 5.1, 1.4 Hz, 1H), 2.17 (s, 3H), 2.16 – 2.10 (m, 6H), 1.90 (dd, *J* = 8.3, 6.2 Hz, 1H), 1.71 – 1.61 (m, 6H), 1.55 (s, 3H), 1.40 (dd, *J* = 6.2, 4.8 Hz, 1H), 1.32 (dd, *J* = 8.3, 4.8 Hz, 1H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 170.71, 147.68, 125.86, 125.58, 119.34, 80.79, 40.69, 36.38, 30.99, 30.74, 25.75, 22.08, 17.86.

**HRMS** (ESI) calculated for C<sub>19</sub>H<sub>24</sub>NaO<sub>2</sub>S [M+Na]<sup>+</sup> *m/z*: 339.1389, found: 339.1385.

**<sup>1</sup>H-<sup>13</sup>C HSQC** and **HMBC** spectra were measured.

*cis-isomer:*

**IR** 2908, 2851, 2359, 2340, 1722, 1266, 1168, 1062, 787.

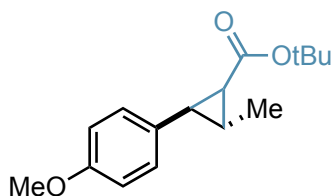
**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.20 (dd, *J* = 5.0, 3.0 Hz, 1H), 7.04 (dd, *J* = 3.0, 1.3 Hz, 1H), 7.02 (dd, *J* = 5.0, 1.3 Hz, 1H), 2.04 (m, 3H), 1.88 – 1.77 (m, 6H), 1.75 (dd, *J* = 7.7, 5.7 Hz, 1H), 1.71 – 1.68 (m, 1H), 1.56 (q, *J* = 3.1, 2.7 Hz, 6H), 1.45 (s, 3H), 1.05 (dd, *J* = 7.7, 4.6 Hz, 1H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 169.94, 142.88, 128.48, 124.82, 122.16, 80.08, 41.10, 36.29, 30.86, 30.37, 27.84, 26.73, 19.74.

**HRMS** (ESI) calculated for C<sub>19</sub>H<sub>24</sub>NaO<sub>2</sub>S [M+Na]<sup>+</sup> *m/z*: 339.1389, found: 339.1385.

**<sup>1</sup>H-<sup>13</sup>C HSQC** and **HMBC** spectra were measured.

### *Tert*-butyl(2*R*\*,3*R*\*)-2-(4-methoxyphenyl)-3-methylcyclopropane-1-carboxylate **3au**



Prepared according to **General procedure F** using (*E*)-anethole **1a** (30 mg, 0.2 mmol, 1 equiv) and *tert*-butyl 2,2-diiodoacetate. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided the corresponding cyclopropanes (33 mg, 63%). Ratio of isomers was determined to be 1:1.



cis-isomer:

**IR** 2973, 2930, 1716, 1515, 1245, 1147, 1036;

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.15 (d, *J* = 8.5 Hz, 2H), 6.79 (d, *J* = 8.7 Hz, 2H), 3.77 (s, 3H), 2.23 (dd, *J* = 9.1, 6.9 Hz, 1H), 1.93 (dt, *J* = 12.3, 6.1 Hz, 1H), 1.68 (dd, *J* = 9.3, 5.1 Hz, 1H), 1.23 (d, *J* = 6.1 Hz, 3H), 1.18 (s, 9H);

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 170.41, 158.32, 130.41, 129.32, 113.39, 80.04, 55.40, 33.29, 31.13, 28.05, 19.18, 17.83.

**HRMS** (ESI) calculated for C<sub>16</sub>H<sub>23</sub>O<sub>2</sub> [M+H]<sup>+</sup> *m/z*: 247.1693, found: 247.1699.

*trans*-isomer:

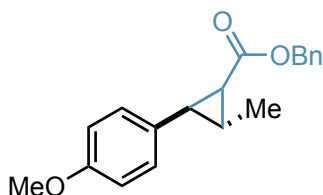
**IR** 2976, 1716, 1516, 1247, 1153;

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.00 (d, *J* = 8.6 Hz, 2H), 6.81 (d, *J* = 8.7 Hz, 2H), 3.78 (s, 3H), 2.29 (dd, *J* = 6.5, 5.0 Hz, 1H), 1.87 (dd, *J* = 9.3, 5.0 Hz, 1H), 1.54 – 1.50 (m, 1H), 1.47 (s, 9H), 1.31 (d, *J* = 6.2 Hz, 3H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 171.08, 158.24, 133.13, 127.25, 114.02, 80.59, 55.49, 31.22, 30.38, 28.45, 25.15, 12.07.

**HRMS** (ESI) calculated for C<sub>16</sub>H<sub>22</sub>NaO<sub>2</sub> [M+Na]<sup>+</sup> *m/z*: 269.1512, found: 269.1511

### Benzyl (2*R*\*,3*R*\*)-2-(4-methoxyphenyl)-3-methylcyclopropane-1-carboxylate **3av**



Prepared according to **General procedure F** using (*E*)-anethole **1a** (30 mg, 0.2 mmol, 1 equiv) and benzyl 2,2-diiodoacetate. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided the corresponding cyclopropanes (42 mg, 71%). Ratio of isomers was determined to be 1:1.

cis-isomer:

**IR** 2951, 2942, 1697, 1515, 1245, 1036;

**<sup>1</sup>H NMR** <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.39 – 7.31 (m, 5H), 7.01 (d, *J* = 8.6 Hz, 2H), 6.81 (d, *J* = 8.7 Hz, 2H), 5.16 (s, 2H), 3.78 (s, 3H), 2.41 (dd, *J* = 6.7, 4.9 Hz, 1H), 2.00 (dd, *J* = 9.2, 4.9 Hz, 1H), 1.65 (dt, *J* = 9.2, 6.4 Hz, 1H), 1.35 (d, *J* = 6.2 Hz, 3H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 170.41, 158.32, 130.41, 129.32, 113.39, 80.04, 55.40, 33.29, 31.13, 28.05, 19.18, 17.83. **<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 171.81, 158.36, 136.31, 132.58, 128.70, 128.35, 128.31, 127.33, 114.06, 66.47, 55.47, 32.21, 29.17, 25.55, 12.21.

**HRMS** (ESI) calculated for C<sub>19</sub>H<sub>20</sub>NaO<sub>3</sub> [M+Na]<sup>+</sup> *m/z*: 319.1305, found: 319.1300.

*trans*-isomer:

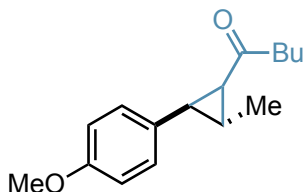
**IR** 2948, 1540, 1258, 782

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.29 – 7.25 (m, 4H), 7.14 – 7.09 (m, 3H), 6.76 (d, *J* = 8.7 Hz, 2H), 4.97 – 4.76 (m, 2H), 3.78 (s, 3H), 2.32 (dd, *J* = 9.2, 6.9 Hz, 1H), 2.05 (ddd, *J* = 6.8, 6.0, 5.0 Hz, 1H), 1.84 (dd, *J* = 9.2, 5.1 Hz, 1H), 1.26 (d, *J* = 6.1 Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  171.03, 158.42, 136.24, 130.24, 128.70, 128.49, 128.30, 128.08, 113.53, 66.16, 55.31, 34.02, 30.07, 20.22, 17.87.

HRMS (ESI) calculated for  $\text{C}_{19}\text{H}_{20}\text{NaO}_3$   $[\text{M}+\text{Na}]^+$   $m/z$ : 319.1305, found: 319.1298.

#### 1-((2*R*\*,3*R*\*)-2-(4-methoxyphenyl)-3-methylcyclopropyl)pentan-1-one 4a



Prepared according to **General procedure F** using (*E*)-anethole **1a** (30 mg, 0.2 mmol, 1 equiv) and 1,1-diiodohexan-2-one<sup>14</sup> **2c**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided the corresponding cyclopropanes (16 mg, 33%). Ratio of isomers was determined to be 3:2 (*trans*:*cis*).

*trans*-isomer:

IR 2915, 2358, 1697, 1516, 1259, 714.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.03 (d,  $J$  = 8.6 Hz, 2H), 6.84 (d,  $J$  = 8.7 Hz, 2H), 3.81 (s, 3H), 2.60 – 2.56 (m, 2H), 2.54 – 2.51 (m, 1H), 2.26 (dd,  $J$  = 9.1, 4.9 Hz, 1H), 1.79 (m, 1H), 1.63 (m, 2H), 1.36 (q,  $J$  = 7.4 Hz, 2H), 1.25 (d,  $J$  = 6.1 Hz, 3H), 0.94 (t,  $J$  = 7.3 Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  208.38, 158.36, 133.29, 127.38, 114.14, 55.59, 45.06, 37.30, 32.92, 28.32, 26.43, 22.67, 14.13, 11.67.

HRMS (ESI) calculated for  $\text{C}_{16}\text{H}_{22}\text{NaO}_2$   $[\text{M}+\text{Na}]^+$   $m/z$ : 269.1512, found: 269.1500.

*cis*-isomer:

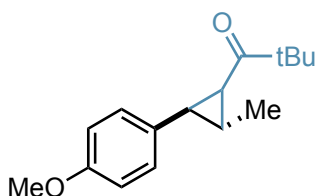
IR 2924, 1697, 1514, 1258, 720.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.09 (d,  $J$  = 8.6 Hz, 2H), 6.77 (d,  $J$  = 8.7 Hz, 2H), 3.76 (s, 3H), 2.43 – 2.36 (m, 1H), 2.37 – 2.23 (m, 2H), 2.19 – 2.08 (m, 2H), 1.39 (m, 2H), 1.24 (d,  $J$  = 5.8 Hz, 3H), 1.14 (tt,  $J$  = 15.1, 7.3 Hz, 2H), 0.80 (t,  $J$  = 7.3 Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  206.69, 158.32, 130.09, 128.45, 113.45, 55.32, 44.21, 38.47, 37.13, 25.97, 22.40, 20.40, 18.05, 13.95.

HRMS (ESI) calculated for  $\text{C}_{16}\text{H}_{23}\text{O}_2$   $[\text{M}+\text{H}]^+$   $m/z$ : 247.1693, found: 247.1699.

#### 1-((2*R*\*,3*R*\*)-2-(4-methoxyphenyl)-3-methylcyclopropyl)-2,2-dimethylpropan-1-one 4b



Prepared according to **General procedure F** using (*E*)-anethole **1a** (30 mg, 0.2 mmol, 1 equiv) and 1,1-diiodo-3,3-dimethylbutan-2-one<sup>15</sup> **2d**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 100:1) provided the

corresponding cyclopropanes (24 mg, 49%). Ratio of isomers was determined to be 3:2 (*trans*:*cis*).

*trans*-isomer:

**IR** 2917, 2358, 1680, 1516, 721.

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.02 (d, *J* = 8.6 Hz, 2H), 6.82 (d, *J* = 8.7 Hz, 2H), 3.78 (s, 3H), 2.50 (dd, *J* = 6.6, 5.0 Hz, 1H), 2.43 (dd, *J* = 9.2, 5.0 Hz, 1H), 1.75 (dt, *J* = 9.2, 6.3 Hz, 1H), 1.19 (s, 9H), 1.18 (d, *J* = 6.1 Hz, 3H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 212.24, 158.21, 133.34, 127.50, 114.02, 55.47, 44.37, 32.92, 32.19, 28.00, 26.46, 11.57.

**HRMS** (ESI) calculated for C<sub>16</sub>H<sub>22</sub>NaO<sub>2</sub> [M+Na]<sup>+</sup> *m/z*: 269.1512, found: 269.1522

*cis*-isomer:

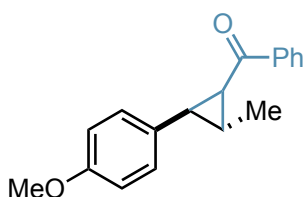
**IR** 2915, 1697, 1521, 1259, 715.

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.08 (d, *J* = 8.5 Hz, 2H), 6.77 (d, *J* = 8.7 Hz, 2H), 3.76 (s, 3H), 2.39 (dd, *J* = 9.2, 6.8 Hz, 1H), 2.33 (dd, *J* = 9.2, 5.3 Hz, 1H), 2.19 – 2.11 (m, 1H), 1.25 (d, *J* = 6.0 Hz, 3H), 1.02 (s, 9H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 210.25, 158.31, 130.04, 128.42, 113.30, 55.29, 43.82, 37.64, 34.61, 26.31, 20.31, 18.09.

**HRMS** (ESI) calculated for C<sub>16</sub>H<sub>22</sub>NaO<sub>2</sub> [M+Na]<sup>+</sup> *m/z*: 269.1512, found: 269.1511.

#### **((2*R*\*,3*R*\*)-2-(4-methoxyphenyl)-3-methylcyclopropyl)(phenyl) methanone 4c**



Prepared according to **General procedure G** using using (*E*)-anethole **1a** (60 mg, 0.4 mmol, 2 equiv) and 2,2-diiodo-1-phenylethan-1-one<sup>16</sup> **2e**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 50:1) provided the corresponding cyclopropanes (25 mg, 47%). Ratio of isomers was determined to be 1:0.8 (*trans*:*cis*). Data are consistent with those reported in the literature.

*trans*-isomer:

**IR** 2955, 2928, 1724, 1666, 1515, 1246, 1219.

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 8.04 – 7.98 (m, 2H), 7.58 – 7.53 (m, 1H), 7.47 (dd, *J* = 8.2, 6.9 Hz, 2H), 7.10 (d, *J* = 8.6 Hz, 2H), 6.84 (d, *J* = 8.7 Hz, 2H), 3.79 (s, 3H), 2.92 (dd, *J* = 9.2, 5.0 Hz, 1H), 2.78 (dd, *J* = 6.7, 5.0 Hz, 1H), 2.00 (m, 1H), 1.28 (d, *J* = 6.2 Hz, 3H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 197.71, 158.34, 138.98, 133.11, 132.78, 128.65, 128.18, 127.56, 114.07, 55.49, 34.65, 32.61, 28.75, 11.80.

**HRMS** (ESI) calculated for C<sub>18</sub>H<sub>19</sub>O<sub>2</sub> [M+H]<sup>+</sup> *m/z*: 267.1380, found: 267.1371.

*cis*-isomer:

**IR** 2923, 2851, 1668, 1515, 1247, 1216.

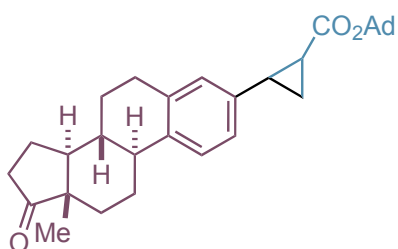
**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.92 – 7.87 (m, 2H), 7.54 – 7.46 (m, 1H), 7.41 (ddd, *J* = 8.2, 6.7, 1.2 Hz, 2H), 7.16 – 7.09 (m, 2H), 6.76 – 6.70 (m, 2H), 3.72 (s, 3H), 2.79 (dd, *J*

= 9.2, 5.2 Hz, 1H), 2.64 (dd,  $J = 9.2, 6.9$  Hz, 1H), 2.45 – 2.38 (m, 1H), 1.34 (d,  $J = 6.0$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  196.49, 158.33, 138.98, 132.51, 130.11, 128.53, 128.32, 128.09, 113.52, 55.25, 38.35, 35.87, 20.74, 18.16.

HRMS (ESI) calculated for  $\text{C}_{18}\text{H}_{18}\text{NaO}_2$   $[\text{M}+\text{Na}]^+$   $m/z$ : 289.1199, found: 289.1195.

**Adamantan-1-yl 2-((8R,9S,13S,14S)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthren-3-yl)cyclopropane-1-carboxylate 6a**



Prepared according to **General procedure F** using (8R,9S,13S,14S)-13-methyl-3-vinyl-6,7,8,9,11,12,13,14,15,16-decahydro-17H-cyclopenta[a]phenanthren-17-one<sup>17</sup> (56 mg, 0.2 mmol, 1 equiv) and adamantan-1-yl 2,2-diiodoacetate **2a**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 20:1) provided a mixture of the corresponding cyclopropanes (47 mg, 50%). Ratio of isomers was determined to be 1:1.

*trans-isomer:*

IR 2907, 2851, 1714, 1455, 1261, 1176, 1055.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.20 (d,  $J = 8.1$  Hz, 1H), 6.90 – 6.85 (m, 1H), 6.84 (d,  $J = 8.3$  Hz, 1H), 2.91 – 2.84 (m, 2H), 2.50 (dd,  $J = 18.9, 8.6$  Hz, 1H), 2.40 (d,  $J = 10.3$  Hz, 1H), 2.36 (ddd,  $J = 9.4, 6.6, 4.3$  Hz, 1H), 2.27 (td,  $J = 10.7, 4.3$  Hz, 1H), 2.15 (m, 4H), 2.11 (m, 6H), 2.08 – 1.99 (m, 2H), 1.96 (d,  $J = 9.4$  Hz, 1H), 1.79 (dt,  $J = 8.7, 4.7$  Hz, 1H), 1.66 (m, 6H), 1.62 (m, 1H), 1.57 – 1.38 (m, 6H), 1.20 (ddd,  $J = 8.4, 6.4, 4.5$  Hz, 1H), 0.90 (s, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  221.02, 172.53, 138.23, 138.04, 136.71, 136.71, \* 127.01, 126.81, \* 125.63, 125.62, \* 123.63, 123.45, \* 80.73, 50.61, 48.13, 44.41, 41.58, 38.35, 36.35, 36.00, 31.72, 30.98, 29.51, 26.63, 25.90, 25.59, 25.49, 21.72, 17.07, 17.04, \* 13.97. \* These  $^{13}\text{C}$  signals may belong to a rotamer.

HRMS (ESI) calculated for  $\text{C}_{32}\text{H}_{40}\text{NaO}_3$   $[\text{M}+\text{Na}]^+$   $m/z$ : 495.2870, found: 495.2865

$^1\text{H}$ - $^{13}\text{C}$  HSQCed spectrum was measured.

*cis-isomer:*

IR 2908, 2851, 1737, 1260, 1176, 1057.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.18 (d,  $J = 8.2$  Hz, 1H), 7.05 (t,  $J = 7.4$  Hz, 1H), 6.99 (d,  $J = 7.1$  Hz, 1H), 2.93 – 2.81 (m, 2H), 2.54 – 2.38 (m, 3H), 2.28 (td,  $J = 10.0, 3.9$  Hz, 1H), 2.11 (d,  $J = 3.3$  Hz, 1H), 2.09 – 1.95 (m, 6H), 1.92 (ddd,  $J = 9.4, 7.7, 5.6$  Hz, 1H), 1.84 – 1.69 (m, 6H), 1.65 – 1.59 (m, 1H), 1.55 (d,  $J = 5.2$  Hz, 8H), 1.50 – 1.26 (m, 4H), 1.23 – 1.17 (m, 1H), 0.89 (s, 3H).

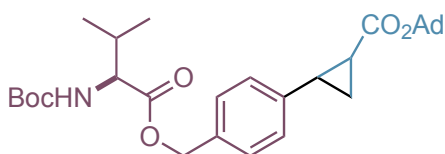
$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  221.09, 221.08, \* 170.35, 170.32, \* 138.08, 138.07, \* 135.91, 135.85, \* 134.73, 130.41, 130.33, \* 127.27, 127.20, \* 125.10, 124.99, \* 80.14,

80.11, \* 50.67, 50.64, \* 48.15, 48.13, \* 44.54, 44.46, \* 41.15, 41.14, \* 38.53, 38.49, \* 36.31, 36.01, 31.75, 31.74, \* 30.88, 29.58, 29.53, \* 26.71, 26.04, 25.95, \* 24.86, 24.83, \* 22.64, 22.53, \* 21.75, 13.93, 10.99, 10.98. \* These  $^{13}\text{C}$  signals may belong to a rotamer.

**HRMS** (ESI) calculated for  $\text{C}_{32}\text{H}_{40}\text{NaO}_3$   $[\text{M}+\text{Na}]^+$   $m/z$ : 495.2870, found: 495.2872.

$^1\text{H}$ - $^{13}\text{C}$  HSQCed spectrum was measured.

**Adamantan-1-yl 2-(4-(((tert-butoxycarbonyl)-L-valyl)oxy)phenyl)cyclopropane-1-carboxylate 6b**



Prepared according to **General procedure F** using 4-vinylphenyl (*tert*-butoxycarbonyl)-*L*-valinat<sup>18</sup> (66 mg, 0.2 mmol, 1 equiv) and adamantan-1-yl 2,2-diiodoacetate **2a**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 100:0 to 20:1) provided a mixture of the corresponding cyclopropanes (63 mg, 61%). Ratio of isomers was determined to be 1:1.

*trans* and *cis*-isomer:

**IR** 2909, 2851, 1715, 1365, 1176, 1056.

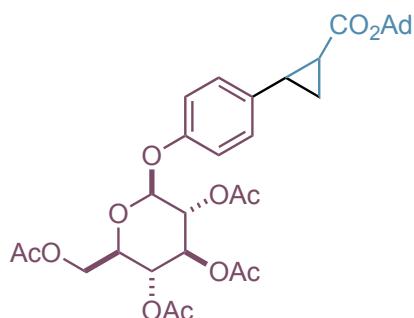
**$^1\text{H}$  NMR** (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.29 – 7.23 (m, 6H), 7.11 – 7.05 (m, 2H), 5.20 – 5.06 (m, 4H), 5.02 (d,  $J$  = 8.1 Hz, 2H), 4.31 – 4.21 (m, 2H), 2.50 (q,  $J$  = 8.8 Hz, 1H), 2.45 – 2.40 (m, 1H), 2.17 (m, 5H), 2.12 (d,  $J$  = 3.0 Hz, 6H), 2.02 (m, 3H), 2.00 – 1.95 (m, 1H), 1.85 – 1.72 (m, 7H), 1.66 (m, 6H), 1.60 (dtd,  $J$  = 7.2, 5.4, 1.5 Hz, 1H), 1.57 – 1.50 (m, 7H), 1.44 (d,  $J$  = 3.1 Hz, 18H), 1.27 – 1.20 (m, 2H), 0.93 (dd,  $J$  = 6.9, 1.8 Hz, 6H), 0.84 (d,  $J$  = 7.0 Hz, 6H).

**$^{13}\text{C}$  NMR** (126 MHz,  $\text{CDCl}_3$ )  $\delta$  172.45, 172.43, 172.29, 169.93, 155.81 (2C), 141.11, 137.50, 133.69, 133.62, 129.89, 128.73, 128.07, 126.43, 80.92, 80.41, 79.89, 79.89, 66.94, 66.78, 58.68 (2C), 41.55, 41.18, 36.33, 36.22, 31.50, 31.45, 30.98, 30.83, 28.45 (2C), 25.65, 25.61, 25.02, 22.90, 19.14, 17.62, 17.59, 17.57, 17.28, 11.01.

**HRMS** (ESI) calculated for  $\text{C}_{31}\text{H}_{43}\text{NNaO}_6$   $[\text{M}+\text{Na}]^+$   $m/z$ : 548.2983, found: 548.2974.

$^1\text{H}$ - $^{13}\text{C}$  HSQC spectrum was measured.

**(2*R*,3*R*,4*S*,5*R*,6*S*)-2-(acetoxymethyl)-6-(4-(2-((adamantan-1-yl)oxy)carbonyl)cyclopropyl) phenoxy)tetrahydro-2*H*-pyran-3,4,5-triyl triacetate 6c**



Prepared according to **General procedure F** using (2*R*,3*R*,4*S*,5*R*,6*S*)-2-(acetoxymethyl)-6-(4-vinylphenoxy)tetrahydro-2*H*-pyran-3,4,5-triyl triacetate (90 mg, 0.2 mmol, 1 equiv) and adamantan-1-yl-2,2-diiodoacetate **2a**. Purification by flash chromatography on silica gel (gradient: hexane:EtOAc 50:1 to 10:1) provided a mixture of the corresponding cyclopropanes (79 mg, 62%). Ratio of isomers was determined to be 1:1.

*trans*-isomer:

**IR** 2910, 2852, 1752, 1715, 1217, 1047.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.02 (d, *J* = 8.7 Hz, 2H), 6.90 (d, *J* = 8.7 Hz, 2H), 5.32 – 5.21 (m, 2H), 5.16 (t, *J* = 9.5 Hz, 1H), 5.05 – 4.99 (m, 1H), 4.28 (dd, *J* = 12.3, 5.2 Hz, 1H), 4.16 (dd, *J* = 12.3, 2.5 Hz, 1H), 3.83 (ddd, *J* = 10.0, 5.2, 2.5 Hz, 1H), 2.39 (ddd, *J* = 9.2, 6.4, 4.2 Hz, 1H), 2.16 (s, 3H), 2.11 (m, 6H), 2.08 (s, 3H), 2.05 (s, 3H), 2.04 (s, 3H), 2.03 (s, 3H), 1.75 (ddd, *J* = 8.4, 5.3, 4.1 Hz, 1H), 1.66 (m, 6H), 1.49 (ddd, *J* = 9.4, 5.3, 4.5 Hz, 1H), 1.16 (td, *J* = 4.3, 2.0 Hz, 1H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 172.39, 170.73, 170.40, 169.53, 169.44, 155.61, 135.75, 127.45, 117.30, 99.56, 80.89, 72.87, 72.17, 71.32, 68.43, 62.08, 41.57, 36.34, 30.98, 25.37, 25.27, 20.86, 20.78, 20.77, 20.74, 17.05.

**HRMS** (ESI) calculated for C<sub>34</sub>H<sub>42</sub>NaO<sub>12</sub> [M+Na]<sup>+</sup> *m/z*: 665.2559, found: 665.2568.

*cis*-isomer:

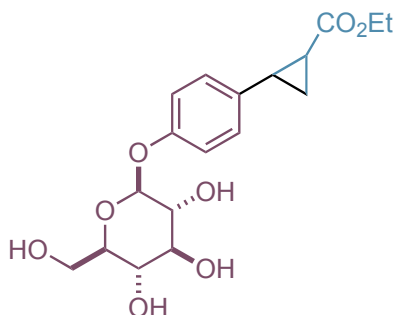
**IR** 2909, 2852, 1752, 1221, 1047, 908.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.18 (d, *J* = 8.5 Hz, 2H), 6.89 (d, *J* = 8.7 Hz, 2H), 5.32 – 5.21 (m, 2H), 5.19 – 5.11 (m, 1H), 5.05 – 5.00 (m, 1H), 4.28 (ddt, *J* = 12.3, 5.3, 1.5 Hz, 1H), 4.20 – 4.10 (m, 1H), 3.88 – 3.78 (m, 1H), 2.45 (q, *J* = 8.5 Hz, 1H), 2.09 – 2.00 (m, 12H), 1.99 – 1.91 (m, 1H), 1.88 – 1.74 (m, 6H), 1.70 – 1.63 (m, 3H), 1.54 (dd, *J* = 4.8, 2.4 Hz, 7H), 1.28 – 1.19 (m, 1H).

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 170.83, 170.50, 170.15, 169.65, 169.53, 155.88, 132.34, 130.85, 116.70, 99.65, 80.47, 73.01, 72.25, 71.45, 68.54, 62.19, 41.34, 36.34, 30.94, 24.72, 22.88, 20.94, 20.94, 20.87, 20.84, 11.27.

**HRMS** (ESI) calculated for C<sub>34</sub>H<sub>42</sub>NaO<sub>12</sub> [M+Na]<sup>+</sup> *m/z*: 665.2559, found: 665.2552.

**Ethyl 2-(4-(((2*S*,3*R*,4*S*,5*S*,6*R*)-3,4,5-trihydroxy-6-(hydroxymethyl)tetrahydro-2*H*-pyran-2-yl)oxy)phenyl)cyclopropane-1-carboxylate **6d****



To a 10 mL reaction tube equipped with a stirring bar was added (2*R*,3*S*,4*S*,5*R*,6*S*)-2-(hydroxymethyl)-6-(4-vinylphenoxy)tetrahydro-2*H*-pyran-3,4,5-triol (56 mg, 0.2 mmol, 1 equiv) and the tube was sealed with a septum, degassed and filled with argon. MeCN

(4ml), *i*-Pr<sub>2</sub>EtN (0.40 mmol, 2 equiv), ethyl 2,2-diiodoacetate **2b** (0.2 mmol, 400 ml, 0.5 M) and H<sub>2</sub>O (1 mL) were added. The reaction mixture was degassed by two freeze-pump-thaw cycles under argon. After that, the reaction tube was irradiated under visible light (21W CFL at a distance of 7 cm) and a mini-fan was kept on top to maintain room temperature. After 3 h, ethyl 2,2-diiodoacetate (0.2 mmol, 400 ml, 0.5 M) and *i*-Pr<sub>2</sub>EtN (0.40 mmol, 2 equiv) were added and the reaction mixture was stirred for 14h more hours. Then, H<sub>2</sub>O (2 ml) was added and the aqueous layer was extracted with EtOAc (3x5 ml). Combined organic layers were dried over NaSO<sub>4</sub> and concentrated under vacuum. The H-NMR yield was calculated after a flash chromatography on neutral silica (gradient dichloromethane:MeOH 50:1 to 15:1). 1,3,5-trimethoxybenzene (0.02 mmol) was added as internal standard and the H-NMR yield was determined to be 30%. Ratio of isomers was determined to be 1:1. The two diastereoisomers could be separated from the starting material by HPLC (PFP C18, 100 x 4.6mm, H<sub>2</sub>O / MeOH 75:25, 1mL/min).

**IR** 2912, 2860, 1240, 1047, 840.

**<sup>1</sup>H NMR** (500 MHz, CD<sub>3</sub>OD) δ 7.20 – 7.15 (m, 2H), 7.06 (d, *J* = 8.8 Hz, 2H), 7.02 (d, *J* = 8.9 Hz, 2H), 6.99 (dd, *J* = 8.7, 1.8 Hz, 2H), 4.15 (q, *J* = 7.1 Hz, 2H), 3.92 – 3.85 (m, 2H), 3.85 (m, 2H), 3.69 (m, 2H), 3.49 – 3.31 (m, 10H), 2.57 (q, *J* = 8.5 Hz, 1H), 2.42 (ddd, *J* = 9.3, 6.6, 4.2 Hz, 1H), 2.11 – 2.02 (m, 1H), 1.82 (dddd, *J* = 8.4, 5.0, 4.1, 0.7 Hz, 1H), 1.65 – 1.57 (m, 1H), 1.49 (ddd, *J* = 9.5, 5.3, 4.5 Hz, 1H), 1.38 – 1.29 (m, 2H), 1.27 (t, *J* = 7.1 Hz, 3H), 1.00 (t, *J* = 7.1 Hz, 3H).

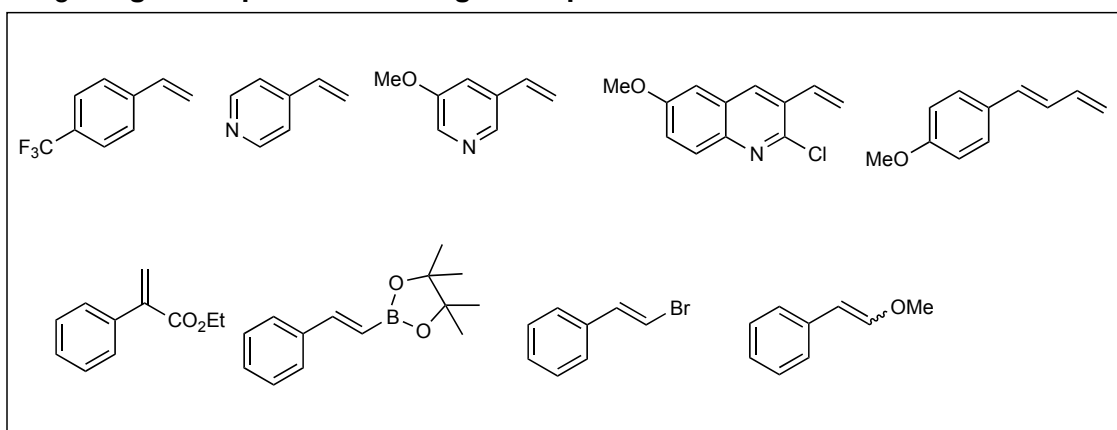
**<sup>13</sup>C NMR** (126 MHz, CD<sub>3</sub>OD) δ 173.76, 171.60, 156.52, 156.50, 133.72, 129.92, 126.79, 116.50, 115.85, 115.81, 101.04, 100.99, 76.73, 76.69, 76.58 (2C), 73.51 (2C), 69.97 (2C), 61.10 (2C), 60.47, 59.92, 25.18, 24.43, 23.52, 21.26, 15.71, 13.13, 13.03, 10.21.

**HRMS** (ESI) calculated for C<sub>18</sub>H<sub>24</sub>NaO<sub>8</sub> [M+Na]<sup>+</sup> *m/z*: 391.1363, found: 391.1364.

**<sup>1</sup>H-<sup>13</sup>C HSQC, HMBC and <sup>1</sup>H-<sup>1</sup>H NOESY** spectra were measured.

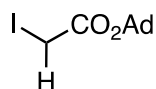
### Limitations of the photocyclopropanation reaction of styrenes.

Figure 2 shows nine styrenes that did not conduct to the corresponding cyclopropane by using our **general procedure F** or **general procedure G**.



**Figure 2.** Unsuccessful styrenes.

## 6. Synthesis of adamantan-1-yl 2-iodoacetate **7**



To a 10 mL reaction tube equipped with a stirring bar was added adamantan-1-yl 2,2-diiodoacetate **2a** (45 mg, 0.10 mmol, 1 equiv) and the tube was sealed with a septum, degassed and filled with argon. MeCN (1ml), *i*-Pr<sub>2</sub>EtN (0.20 mmol, 2 equiv) and aqueous NaCl solution (1.25 M, 0.5 mL) were added. The reaction mixture was degassed by two freeze-pump-thaw cycles under argon. After that, the reaction tube was irradiated under the light source (21W CFL) at a distance of 7 cm and a mini-fan was kept on top to maintain room temperature. After 18 h, the reaction mixture was passed through a short pad of silica gel and eluted with dichloromethane. The solvent was removed under *vacuum* and the residue was purified by column chromatography on neutral silica gel (hexane) to give the desired product in 68% isolated yield.

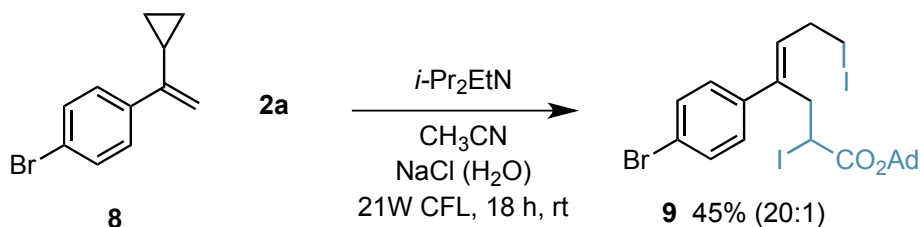
IR 2906, 1438, 1719, 1454, 1249, 1050, 966.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 3.61 (s, 2H), 2.20 (s, 3H), 2.13 (m, 6H), 1.67 (m, 6H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 167.71, 82.51, 40.98, 36.19, 30.93, -2.17.

HRMS (ESI) calculated for C<sub>12</sub>H<sub>17</sub>INaO<sub>2</sub> [M+Na]<sup>+</sup> m/z: 343.0165, found: 343.0154.

## 7. Synthesis of adamantan-1-yl (*E*)-4-(4-bromophenyl)-2,7-diiodohept-4-enoate **9**



To a 10 mL reaction tube equipped with a stirring bar was added the adamantan-1-yl 2,2-diiodoacetate **2a** (90 mg, 0.20 mmol, 1 equiv) and 1-bromo-4-(1-cyclopropylvinyl)benzene<sup>5</sup> (29 mg, 0.2 mmol, 1 equiv). The tube was sealed with a septum, degassed and filled with argon. MeCN (2ml), *i*-Pr<sub>2</sub>EtN (0.40 mmol, 2 equiv) and aqueous NaCl solution (1.25 M, 1 mL) were added. The reaction mixture was degassed by two freeze-pump-thaw cycles under argon. After that, the reaction tube was irradiated under visible light (21W CFL at a distance of 7 cm) and a mini-fan was kept on top to maintain room temperature. The reaction mixture was stirred for 18 h. Then, the reaction mixture was passed through a short pad of silica gel and washed with dichloromethane. The solvent was removed under *vacuum* and the residue was purified by column chromatography on neutral silica gel (gradient: hexane:EtOAc 200:0 to 50:1) to give product **9** (56 mg, 45% yield). Ratio of isomers was determined to be 20:1.

IR 2908, 2820, 1723, 1259, 1051, 716.



**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.46 (d, *J* = 8.5 Hz, 2H), 7.16 (d, *J* = 8.5 Hz, 2H), 5.68 (t, *J* = 7.2 Hz, 1H), 4.03 (t, *J* = 7.7 Hz, 1H), 3.25 (t, *J* = 7.0 Hz, 2H), 3.22 – 3.08 (m, 2H), 2.92 – 2.76 (m, 2H), 2.12 – 2.09 (m, 3H), 2.04 (m, 6H), 1.66 – 1.62 (m, 6H).

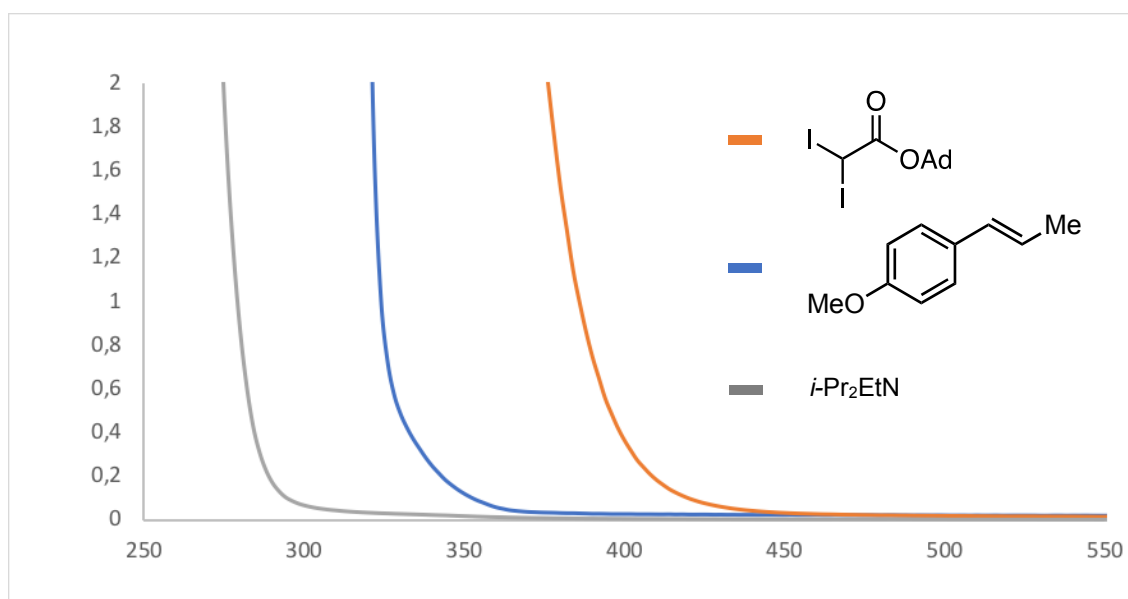
**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 169.62, 139.88, 138.40, 131.74, 131.44, 128.47, 121.56, 82.34, 41.31, 40.75, 36.06, 32.39, 30.79, 21.20, 4.74.

**HRMS** (ESI) calculated for C<sub>23</sub>H<sub>27</sub>BrI<sub>2</sub>NaO<sub>2</sub> [M+Na]<sup>+</sup> *m/z*: 690.9176, found: 690.9166.

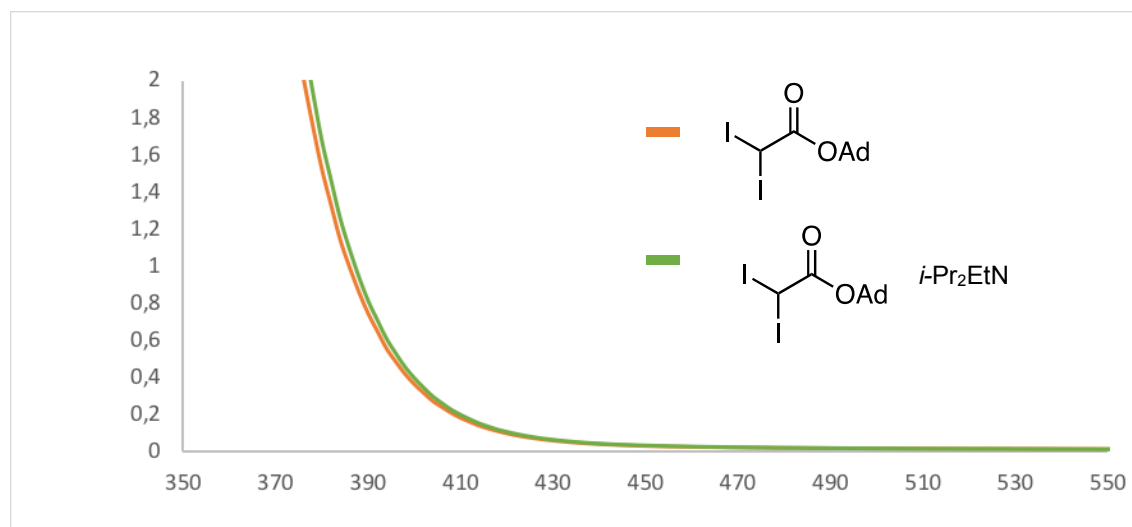
**<sup>1</sup>H-<sup>13</sup>C HSQC, HMBC and <sup>1</sup>H-<sup>1</sup>H NOESY** spectra were measured.

## 8. Optical Absorption Spectra

All the spectra were recorded in acetonitrile, using 0.1 M concentration solutions, in a 1 cm Hellma Quartz cuvette with a cap provided with a Teflon septum. Solutions were degassed with nitrogen just before the optical absorption spectrum was recorded.



**Figure 3.** Absorption spectra of *E*-anethole (1a), 2a, *i*-Pr<sub>2</sub>EtN.



**Figure 4.** Absorption spectra of 2a and an equimolar mixture of 1a and *i*-Pr<sub>2</sub>EtN.

## 9. Differential scanning calorimetry (DSC) analysis of *gem*-diiodomethyl carbonyl reagent 2a.

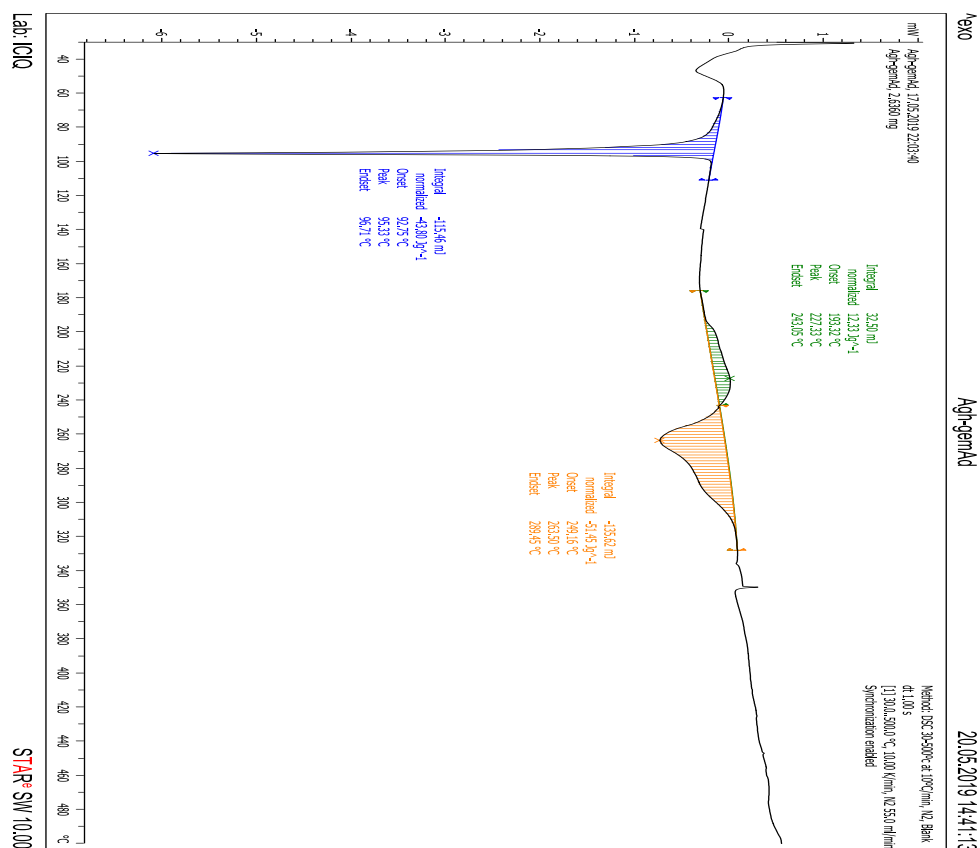


Figure 5. DSC measurement curve for 2a.

## 10. Quantum yield determination

The quantum yield was determined following a reported procedure in Cismesia, M. A.; Yoon, T. P. *Chem. Sci.* **2015**, *6*, 5426.<sup>19</sup> Reactions were conducted in a 1 cm square quartz cuvette and capped with a rubber septum in a fluorometer. Fluorolog Horiba Jobin Yvon spectrofluorimeter equipped with photomultiplier [or *InGaAs* if using the nitrogen cooled detector] detector, double monochromator and Xenon light source was used as the light source. All solutions were carefully prepared in a dark room under red light, and all solutions were wrapped in aluminum foil and stored in the dark.

### Determination of Photon Flux using Ferrioxalate Actinometry

The photon flux of the spectrophotometer was determined by standard ferrioxalate actinometry.<sup>19</sup>

Two solutions were prepared:

**Solution A:** a 0.15M solution of potassium ferrioxalate trihydrate was prepared in 0.05M H<sub>2</sub>SO<sub>4</sub> solution.

**Solution B:** A buffered solution of phenanthroline was prepared by dissolving phenanthroline (0.025 g, 0.14 mmol) and NaOAc (5.63 g, 68.6 mmol) in 25 mL of 0.5 M aq H<sub>2</sub>SO<sub>4</sub>.

To determine the photon flux of the spectrophotometer, 2.0 mL of the ferrioxalate solution A was placed in a cuvette and irradiated for 30 seconds at  $\lambda = 410$  nm with an emission slit width at 5.0 nm. After irradiation, 0.35 mL of the phenanthroline solution was added to the cuvette. The solution was then allowed to rest for 1 h to allow the ferrous ions to completely coordinate to the phenanthroline. The absorbance of the solution was measured at 510 nm. The same procedure was repeated for 20 seconds and 10 seconds.

A non-irradiated sample was also prepared and the absorbance at 510 nm measured. For the calculation of photon flux the following equations were used:

$$\text{mol Fe}^{2+} = (V \cdot \Delta A) / (l \cdot \epsilon) \quad \text{equation 1}$$

$$\text{Photon flux} = (\text{mol Fe}^{2+}) / (\phi \cdot t \cdot f) \quad \text{equation 2}$$

Where V is the total volume of the solution,  $\Delta A$  is the difference in absorption between the irradiated and non-irradiated solutions, l is the path length, and  $\epsilon$  is the molar absorptivity at 510 nm (11,110 L mol<sup>-1</sup>cm<sup>-1</sup>),  $\phi$  is the quantum yield for the ferrioxalate actinometer, t is the time (s), and  $f > 0.999$ .

**Table 3.** photon flux determination

experiment	irradiation time (s)	Abs (510)	mol (Fe <sup>2+</sup> )	Photon flux
blanc	0 seg	0,097	-	
1	10	0,732	1,35919E-07	1,34573E-08
2	20	1,347	2,6464E-07	1,3101E-08
3	30	1,967	3,95901E-07	1,3066E-08

The average of photon flux was calculated to be: 1,308E-8 einstein/s

#### Determination of the Quantum Yield of the photocyclopropanation

Under argon atmosphere, a cuvette was charged with anethole (0.1mmol, 1 equiv), DIPEA (0.2mmol, 2 equiv), reagent **2a** (0.1mmol, 1 equiv) and 2ml of MeCN (previously degassed) and 0,5ml of water (previously degassed). The sample was stirred and irradiated ( $\lambda = 410$  nm, slit width= 5.0 nm) for 5400 s (90 min). After irradiation, the solution was passed through a silica plug. The yield of product formed was determined by <sup>1</sup>H NMR. The quantum yield was determined using eq 3.

$$\phi = (\text{mol product}) / (\text{photon flux} \cdot t \cdot f) \quad \text{equation 3}$$

Where f is the fraction of light absorbed at 410nm of the reaction mixture (equation 4).

$$f = 1 - 10^{-A} \quad \text{equation 4}$$

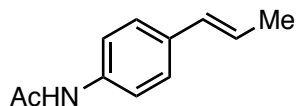
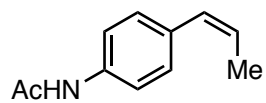
**Table 4.** Quantum yield determination

Abs at 410nm	f	experiment	irradiation time	Yield	Quantum Yield
0,154	0,29	1	90min, 5400s	31%	1,47
		2	93min, 5580s	33%	1,51

The average of quantum yield was calculated to be: 1,49

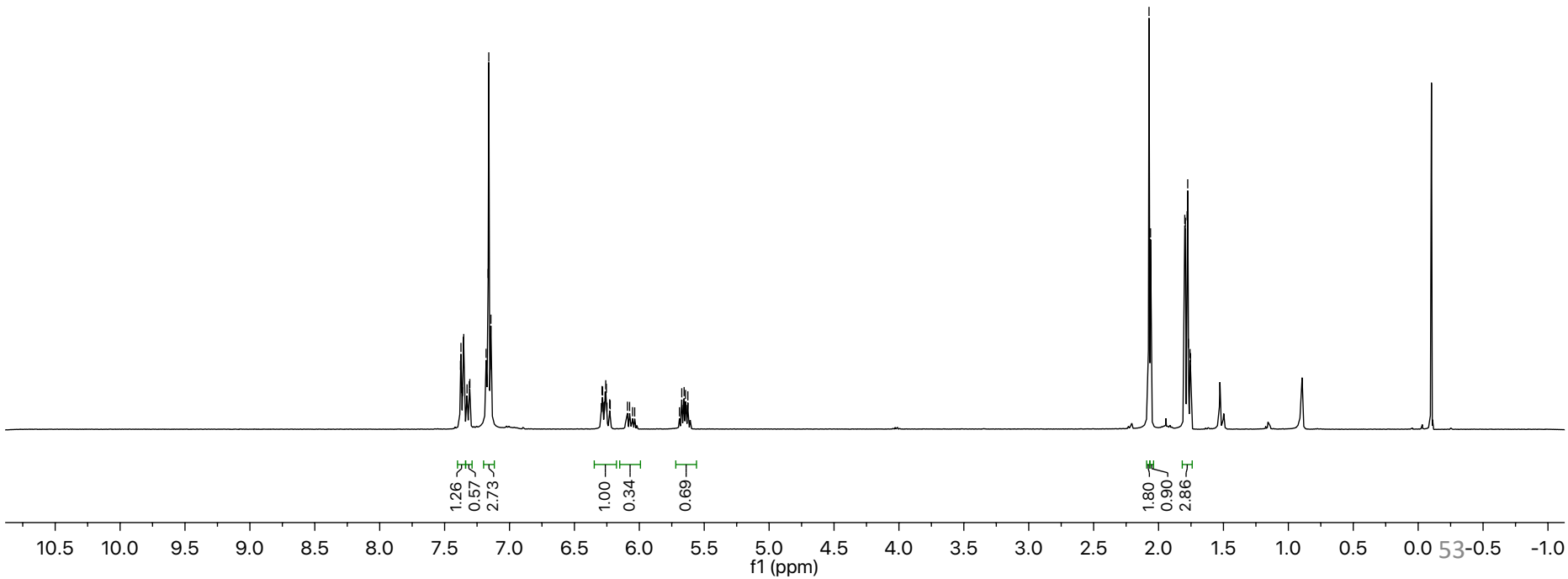
## 11. References

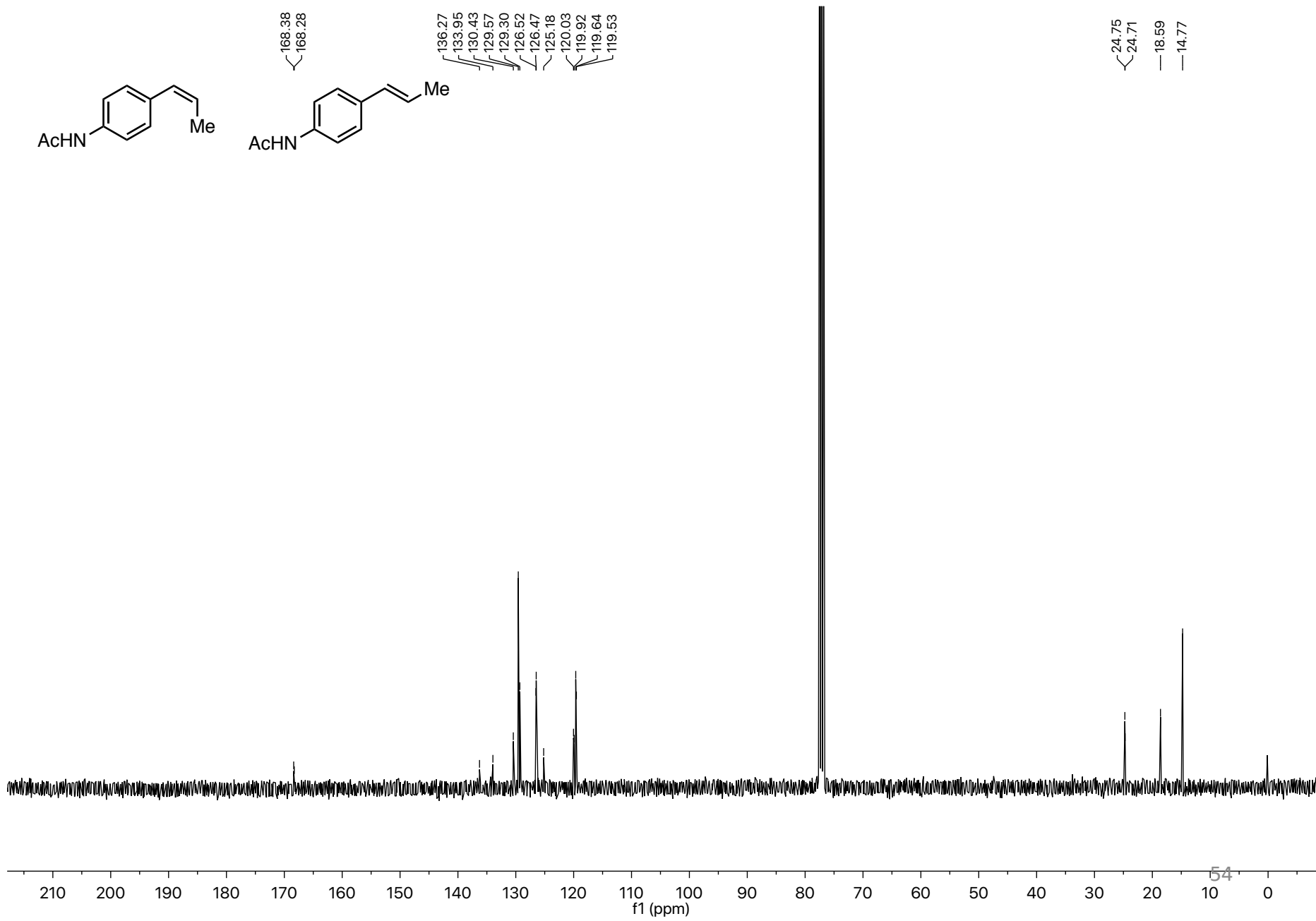
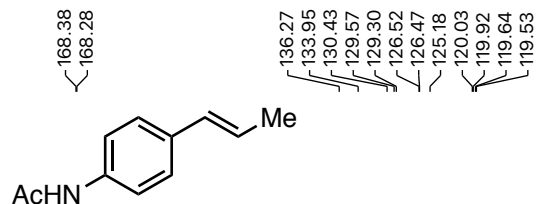
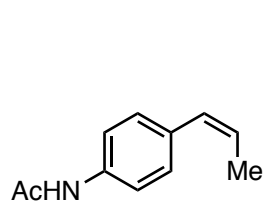
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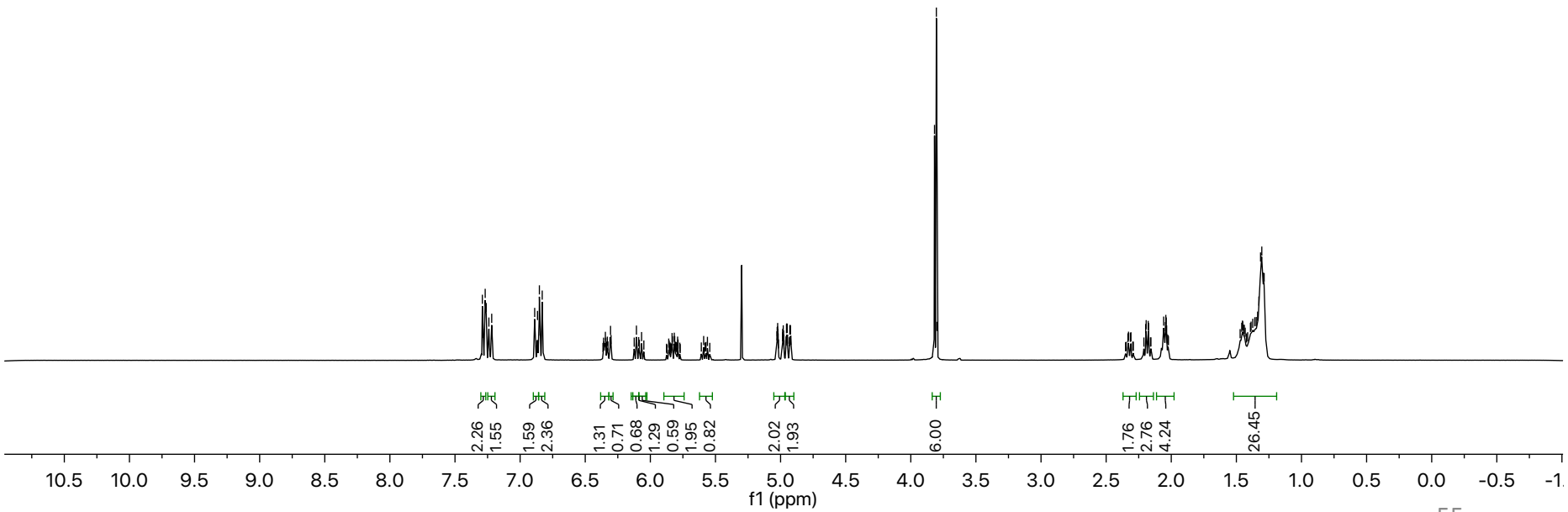
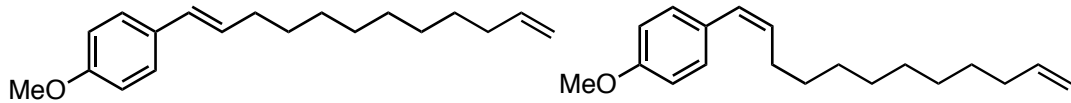
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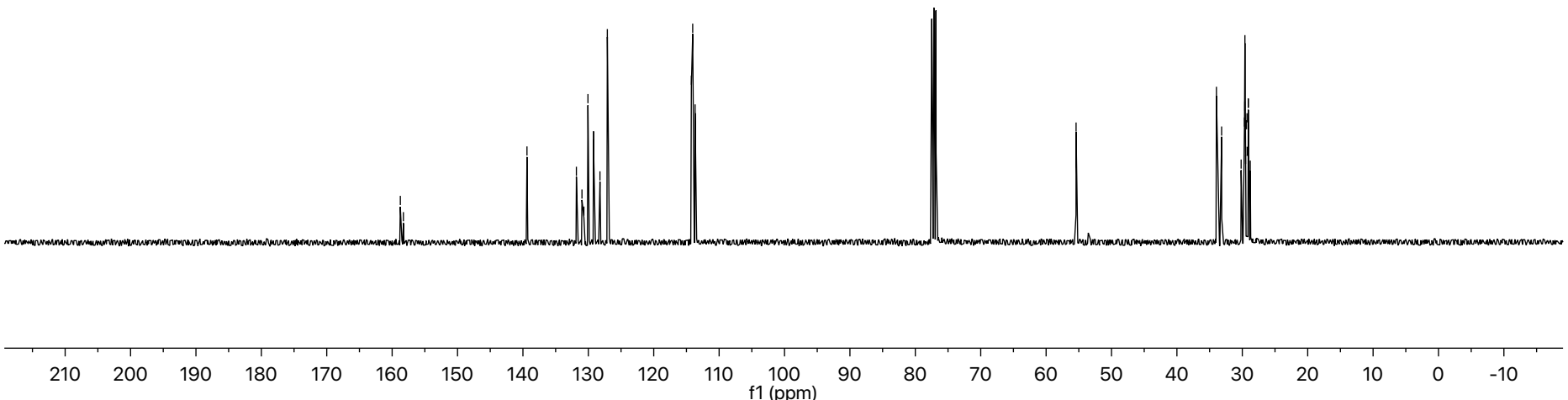
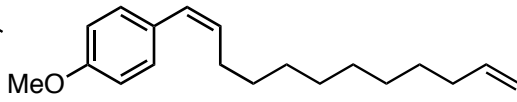
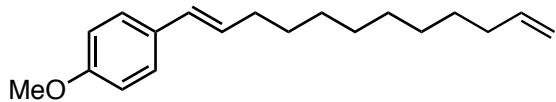
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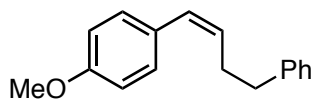
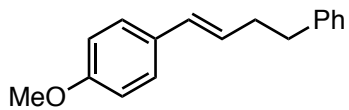
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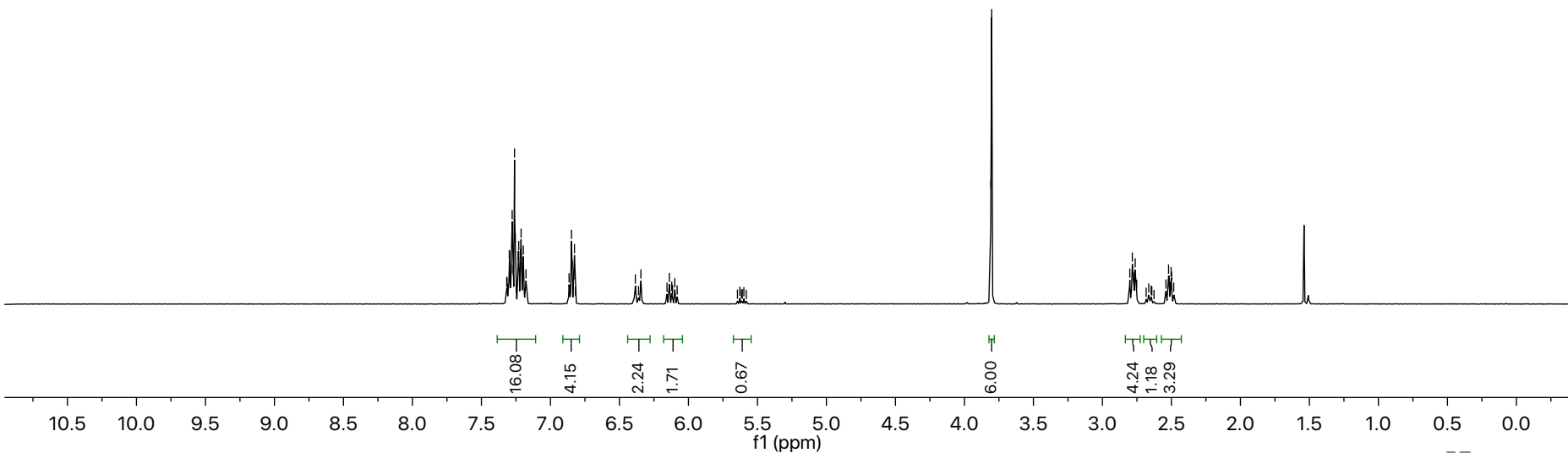


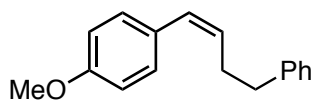
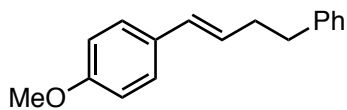




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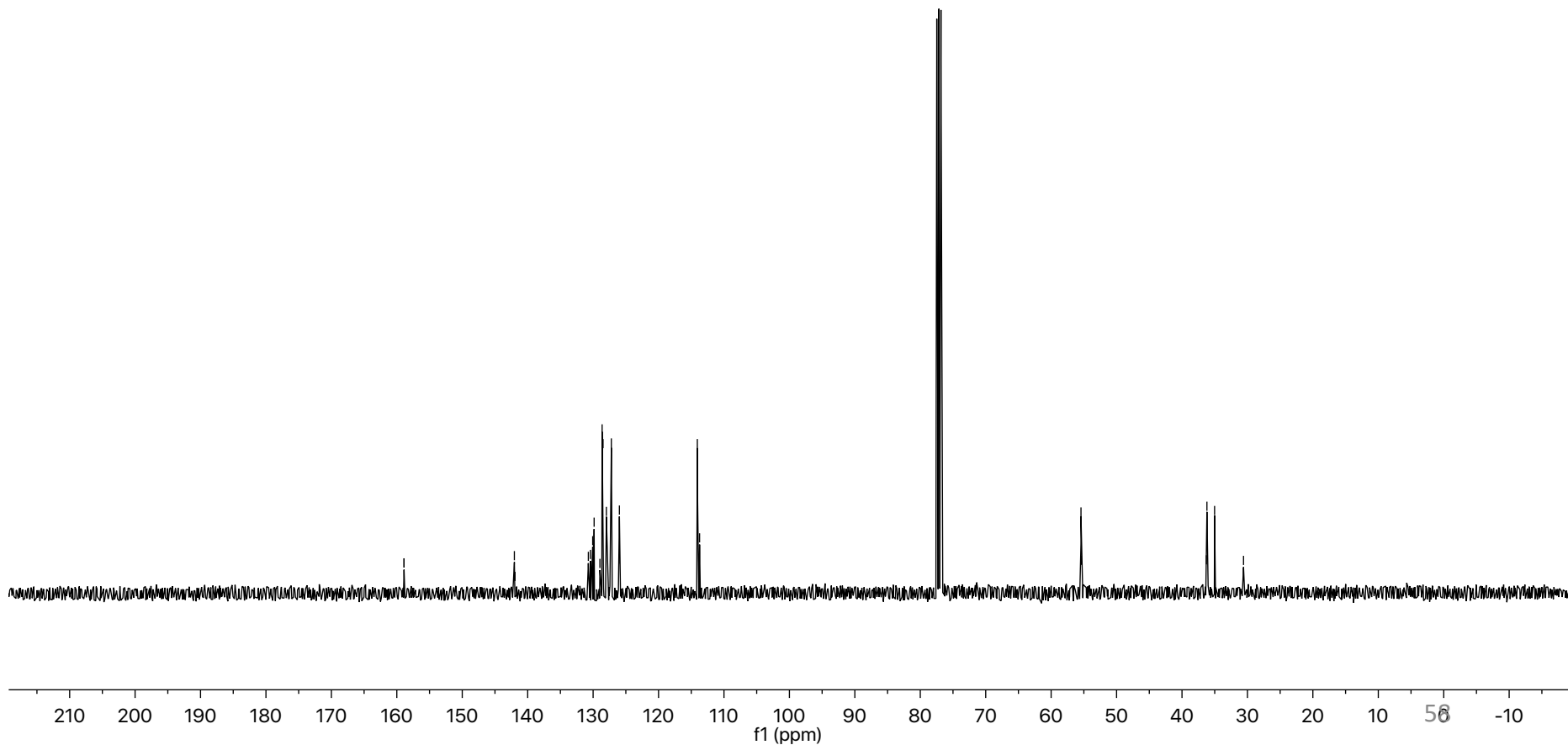


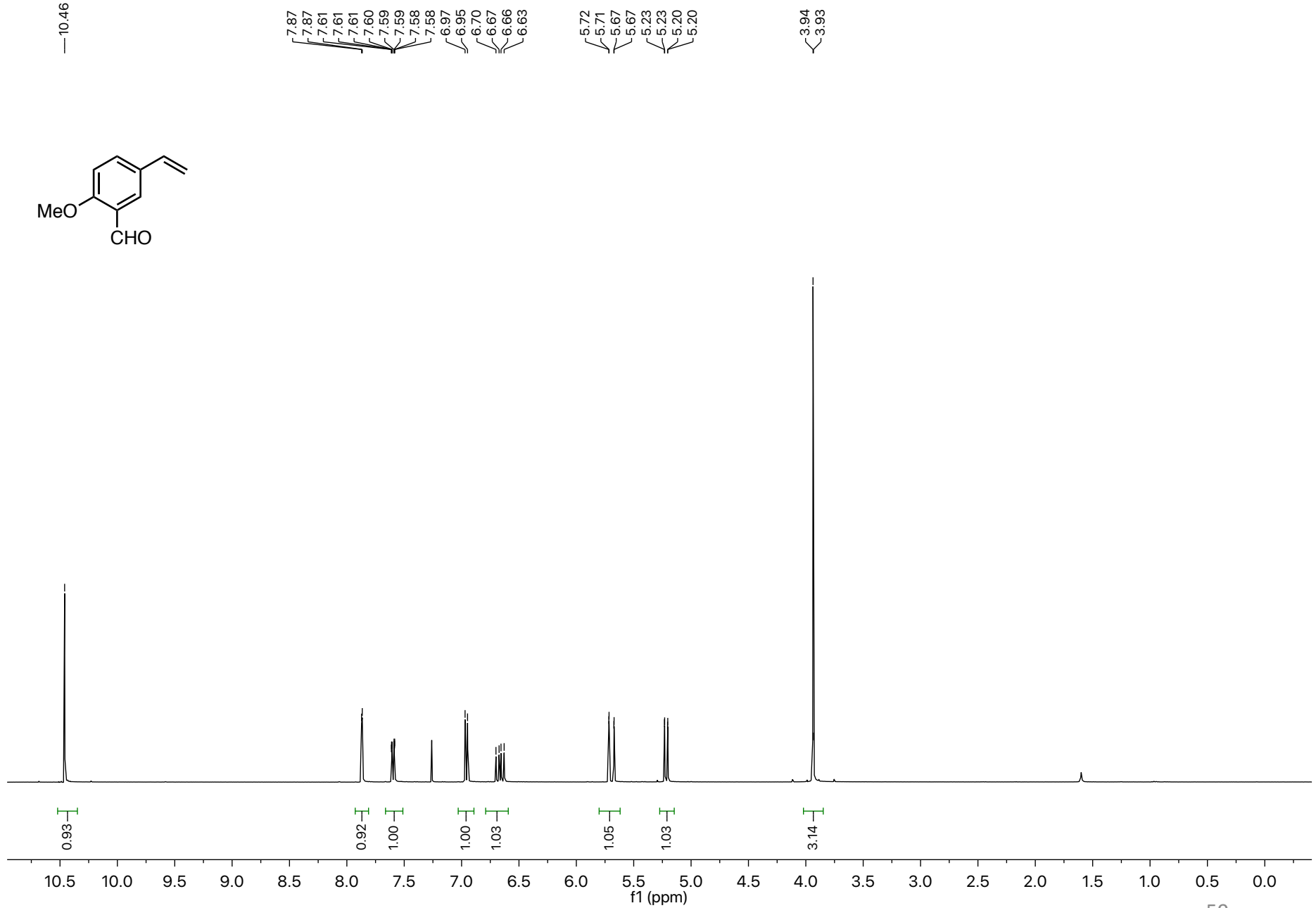
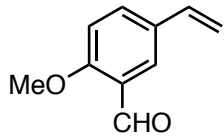


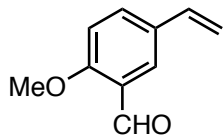
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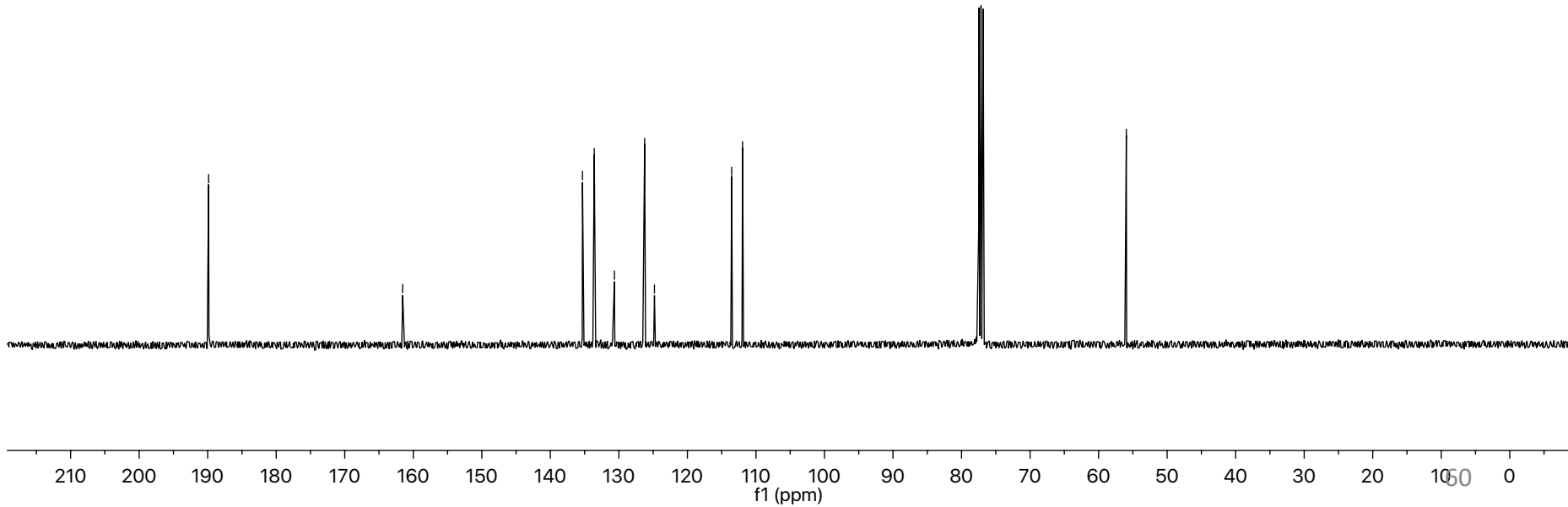
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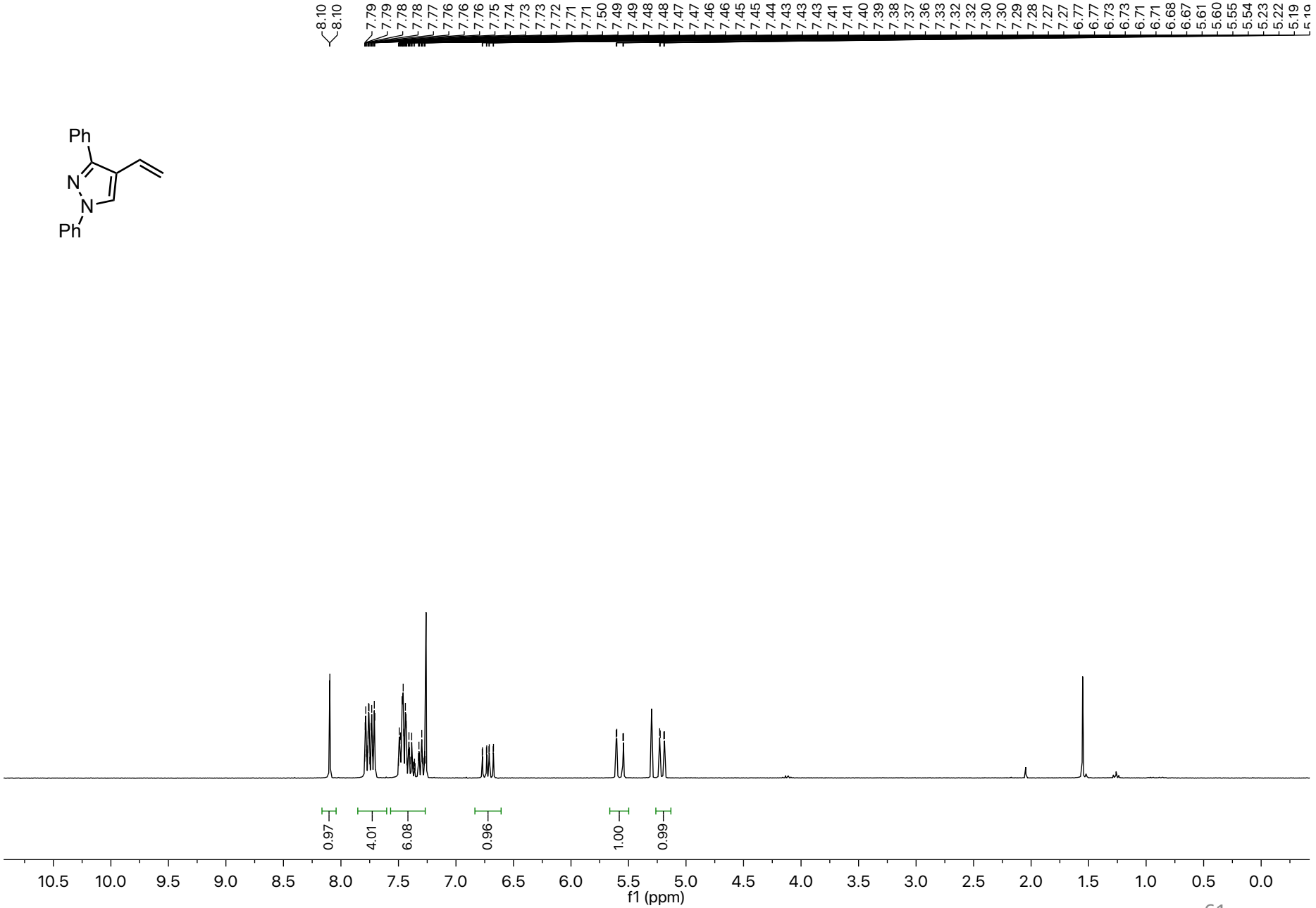
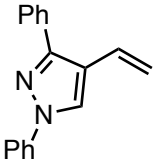
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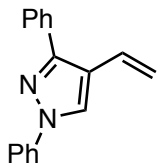
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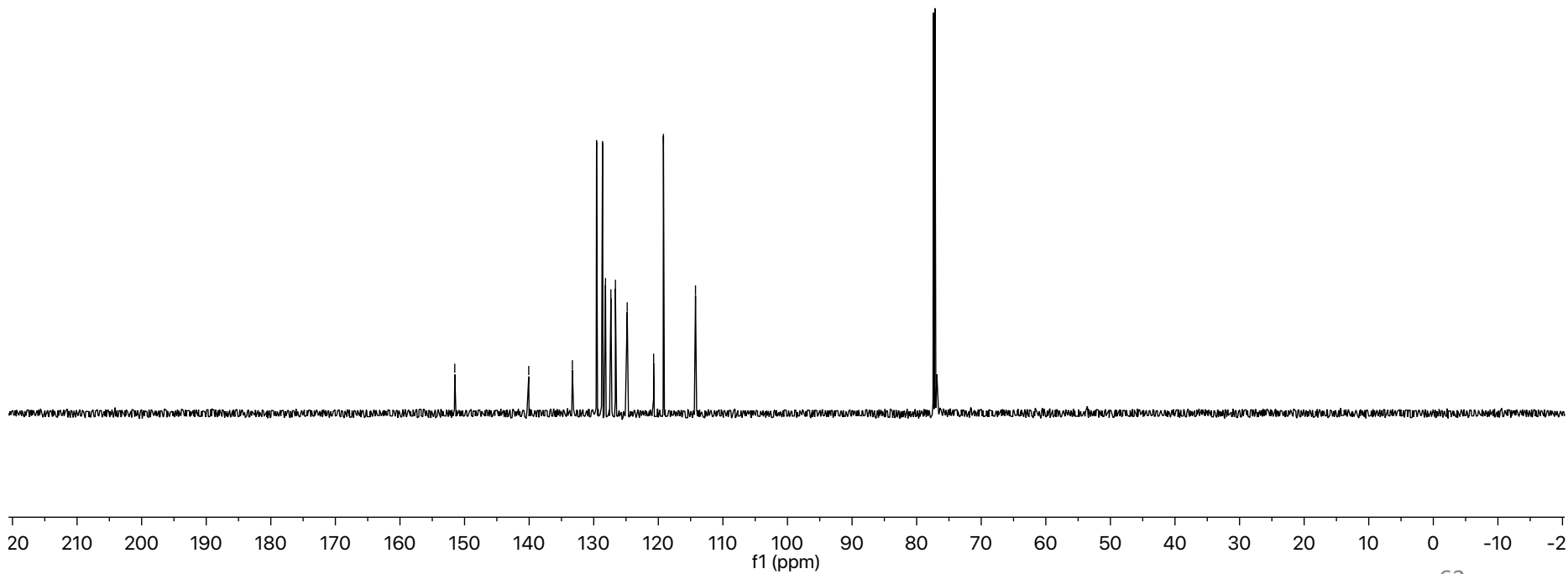
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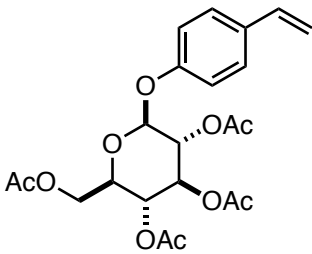






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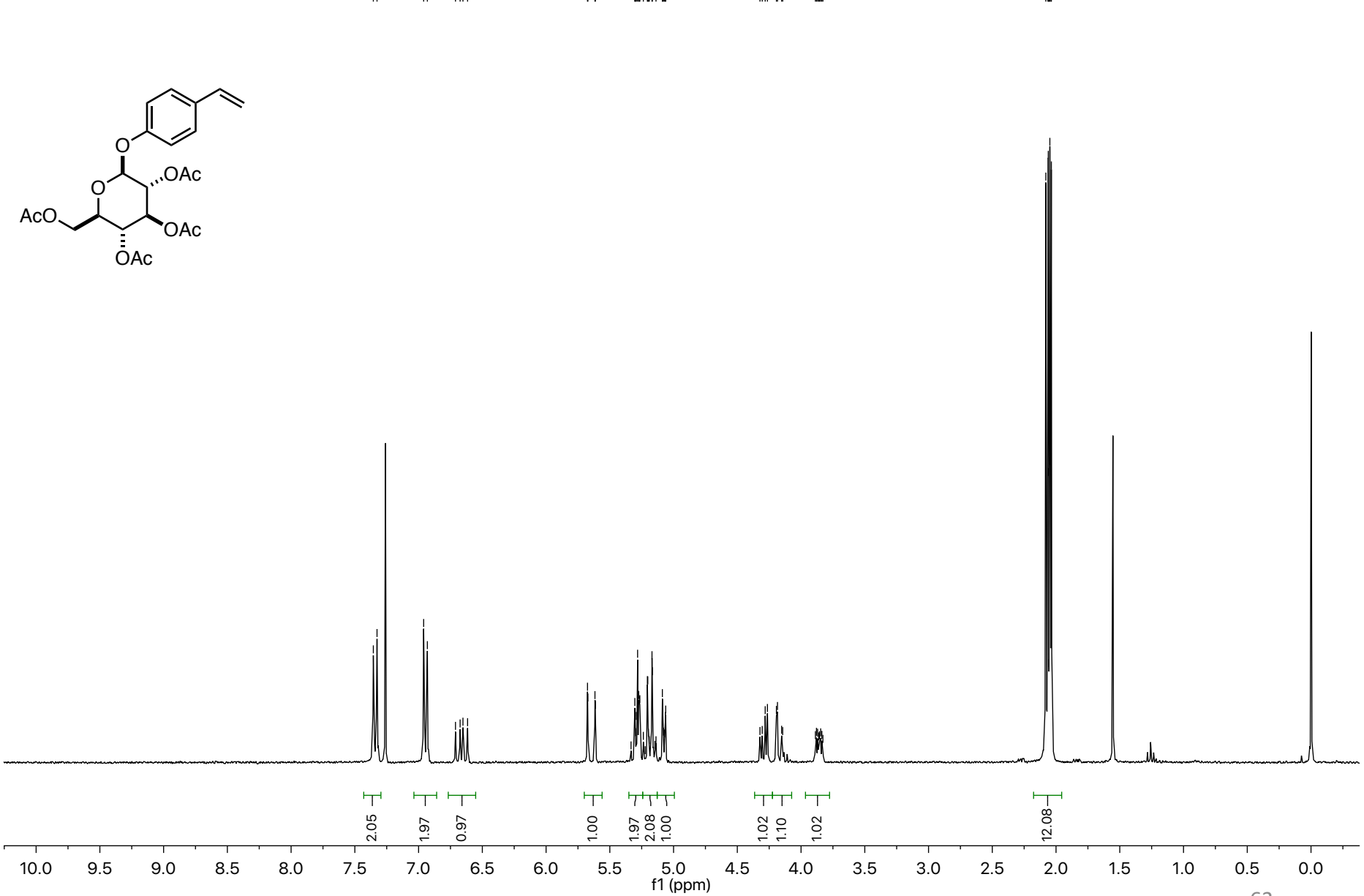


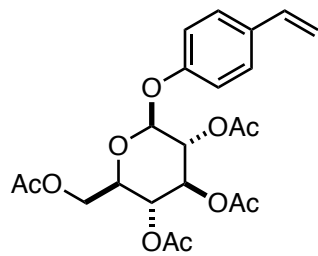


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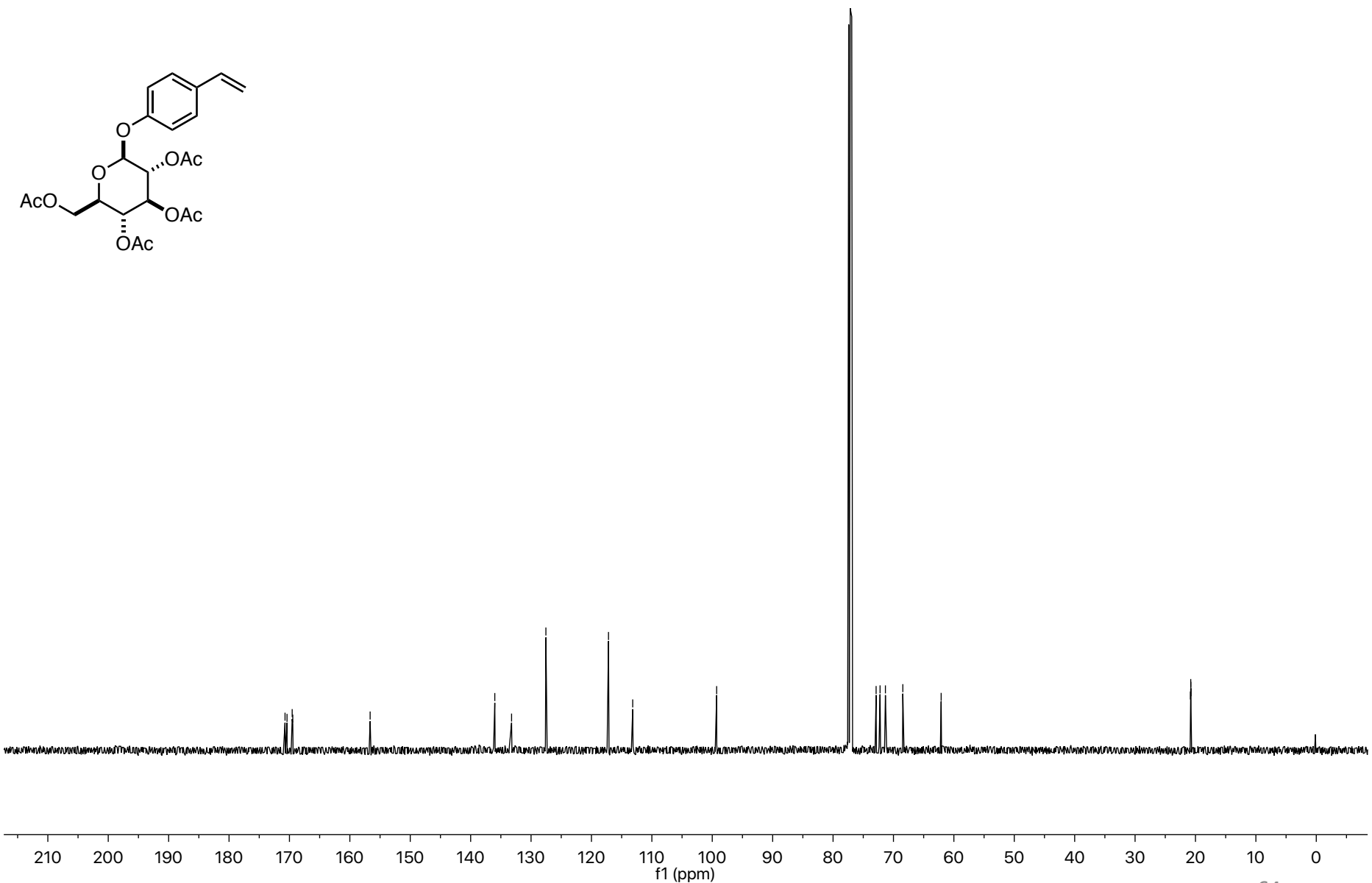
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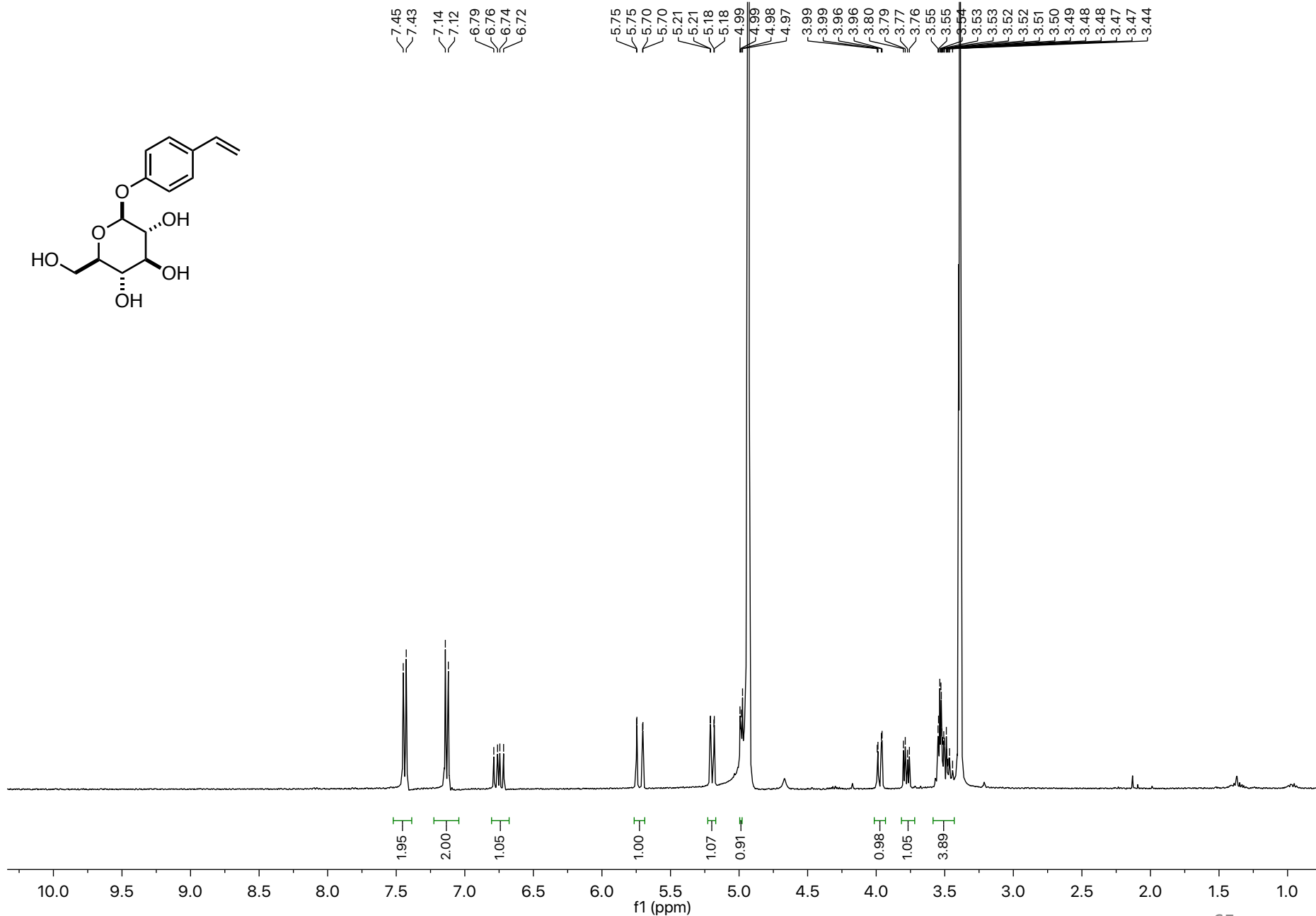
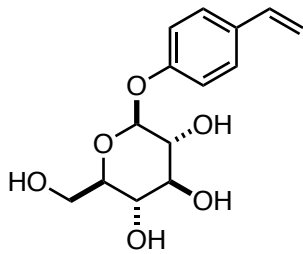
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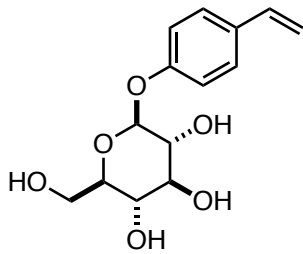
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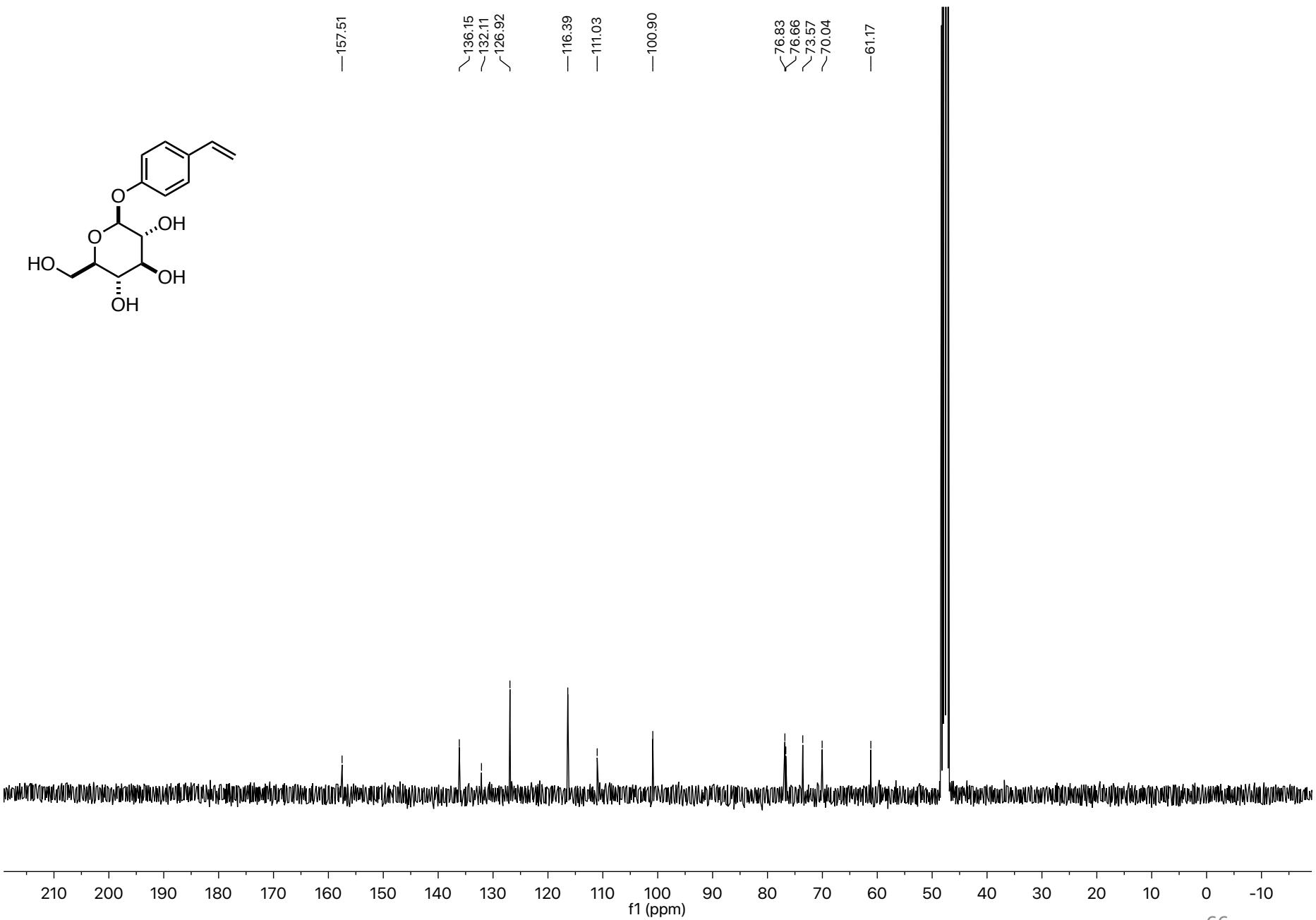
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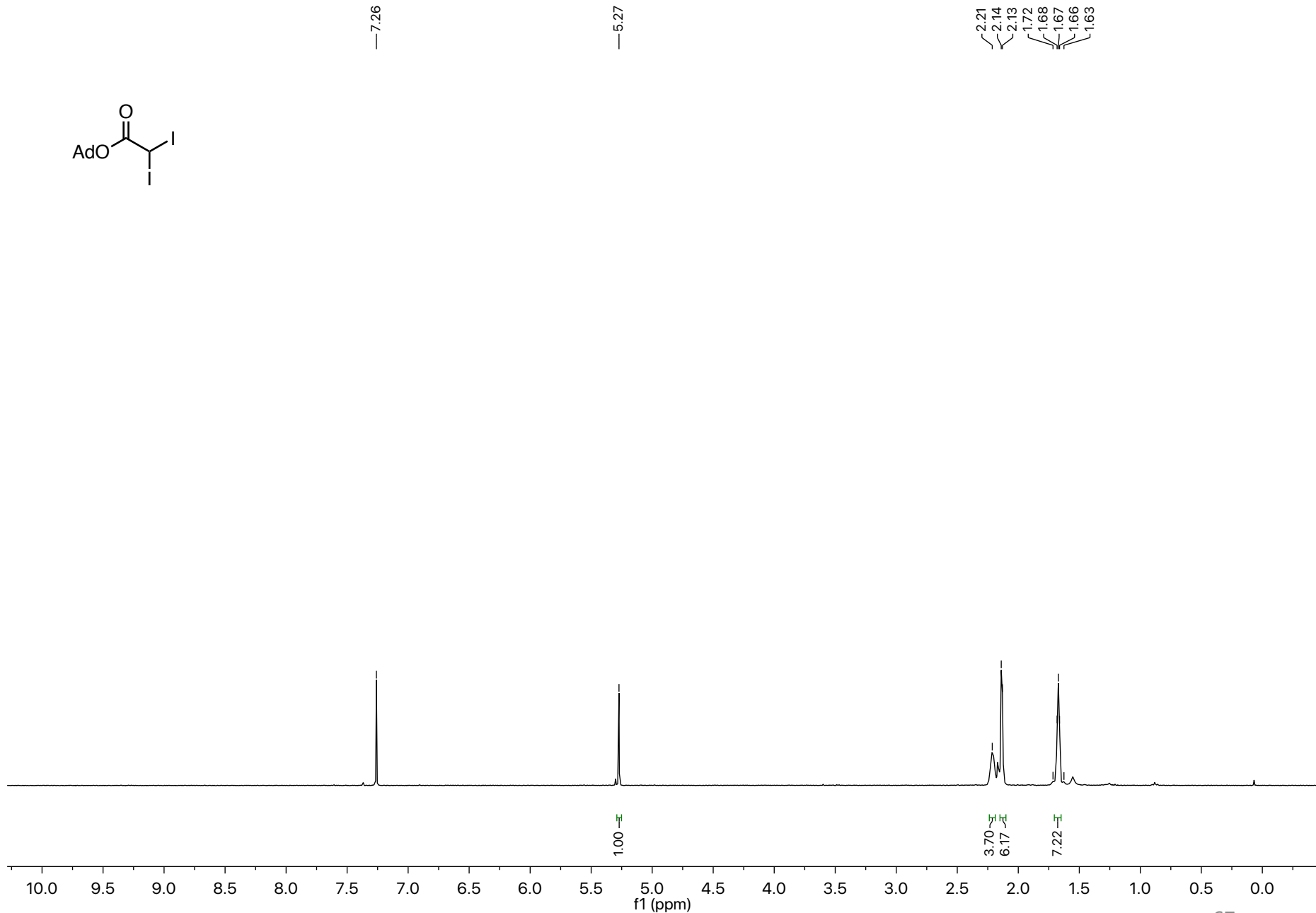
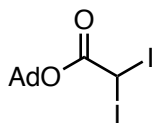
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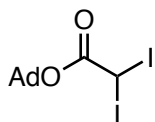
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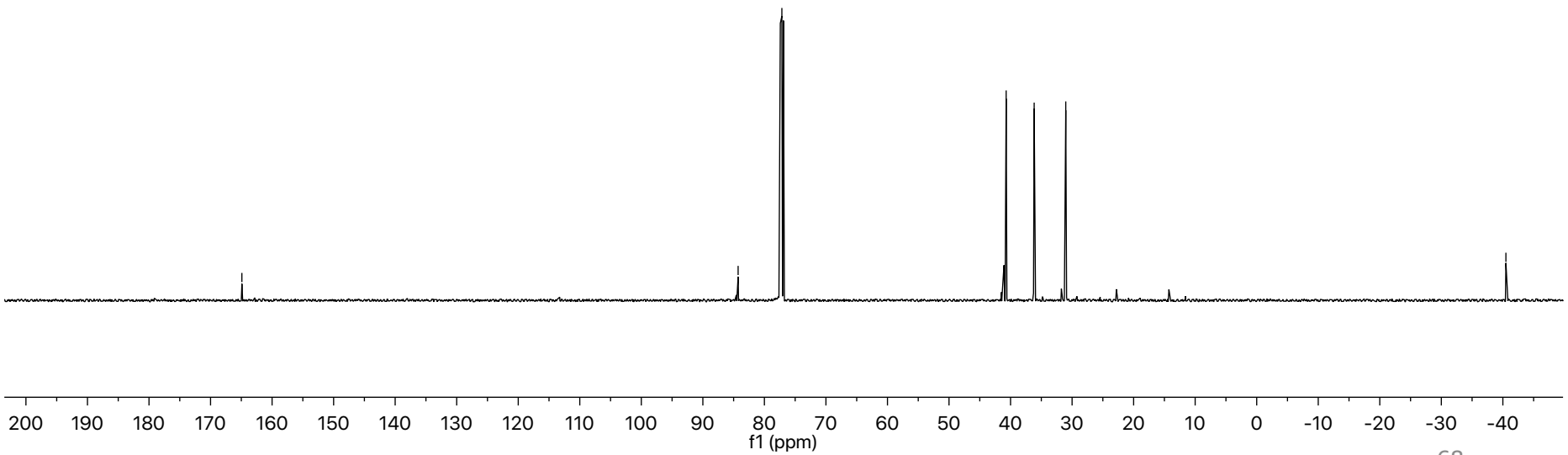
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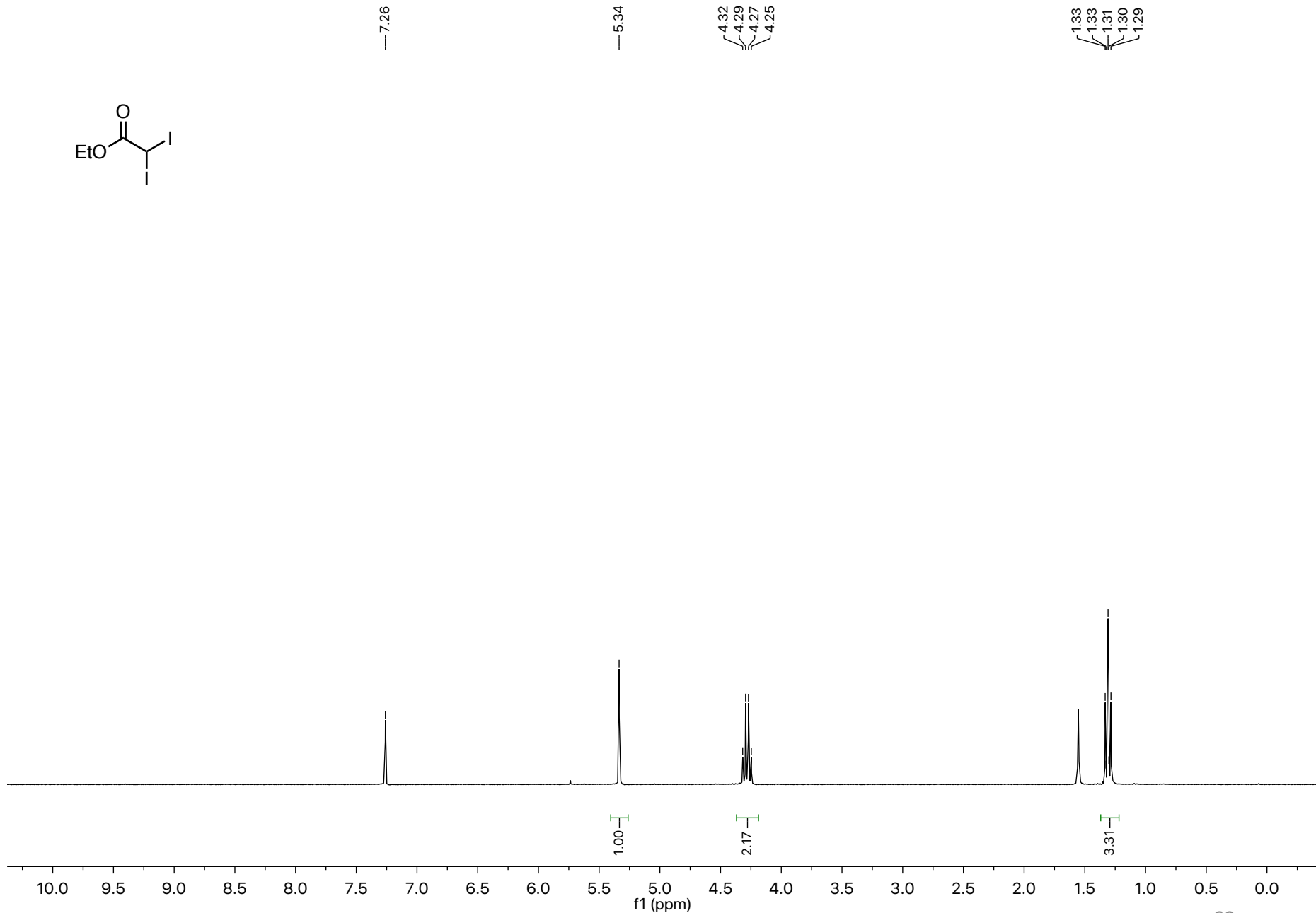
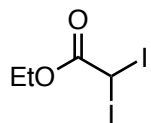
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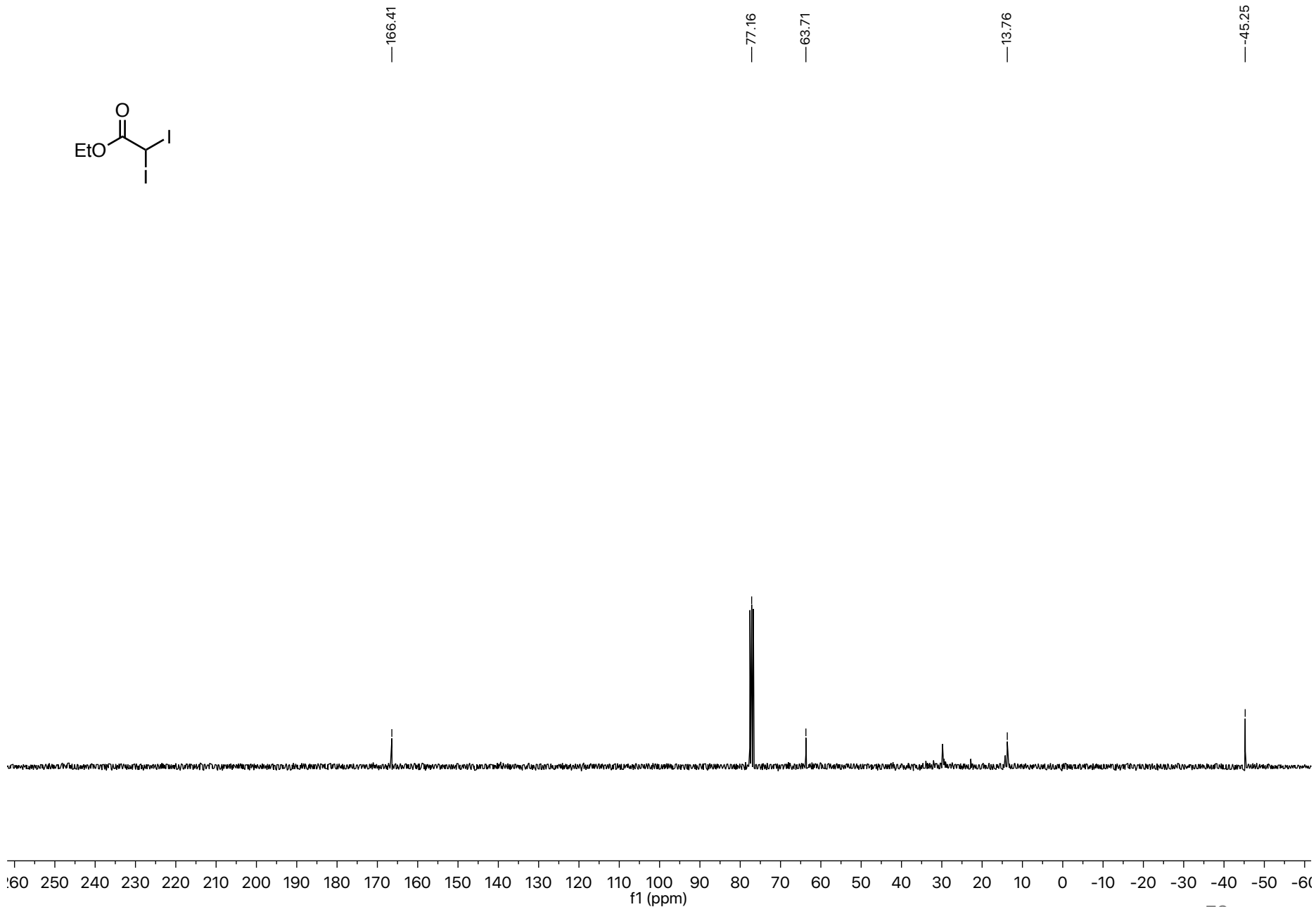
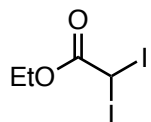
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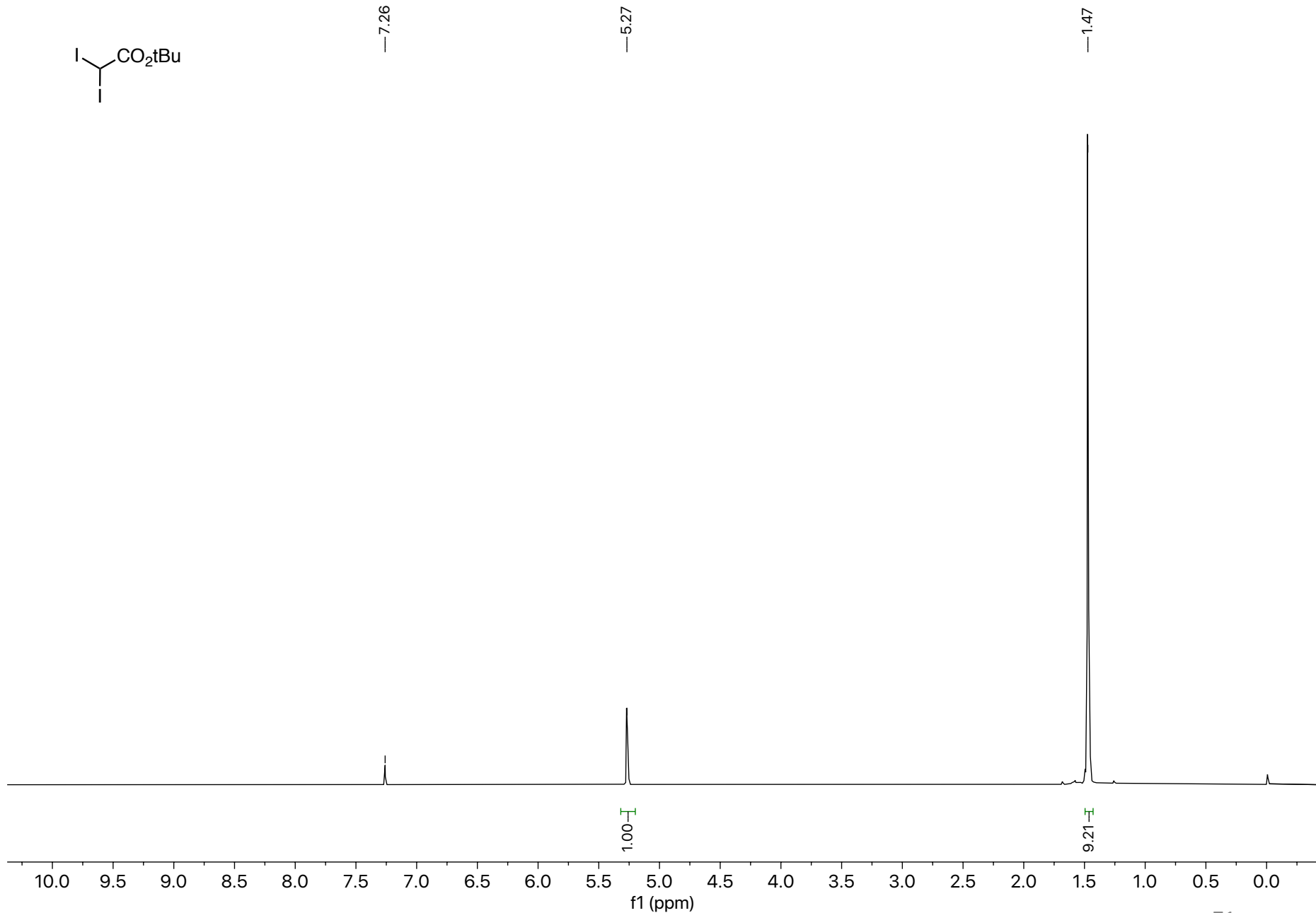
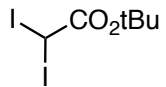
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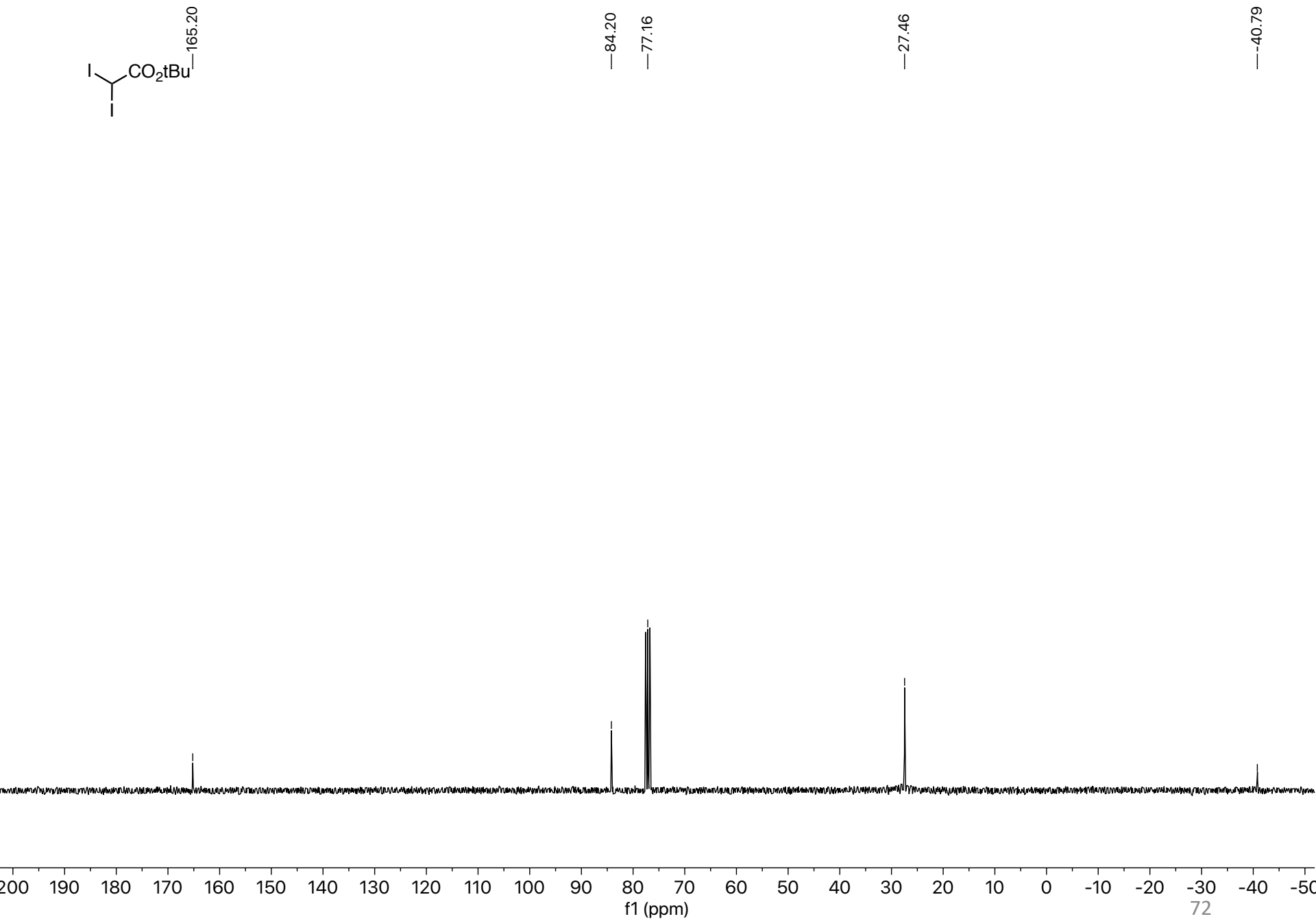
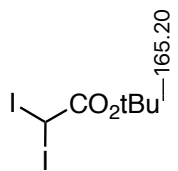
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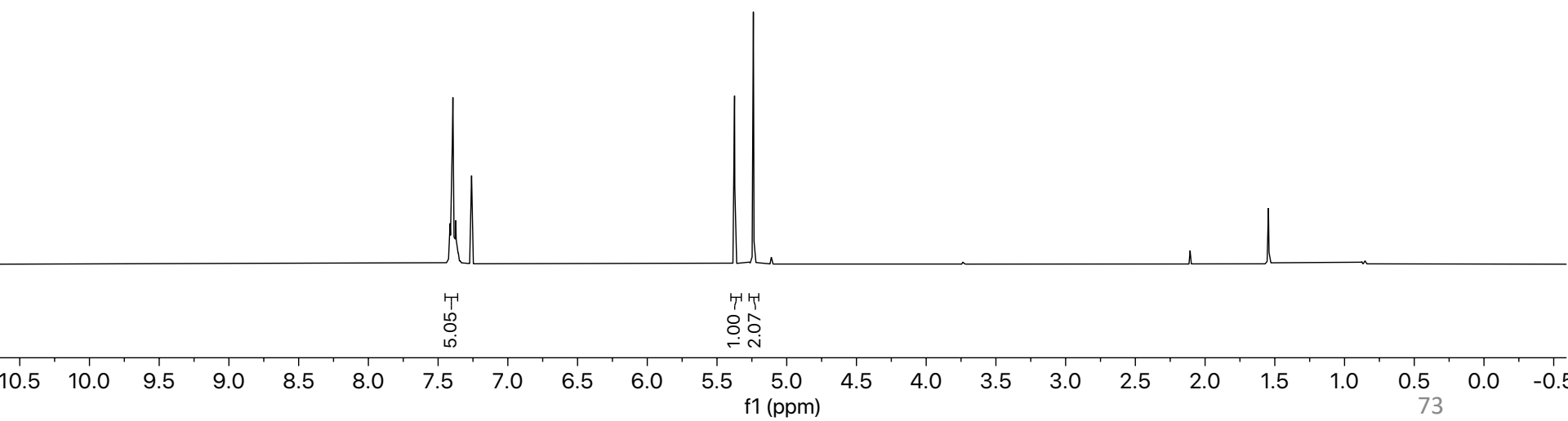
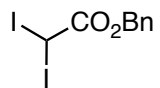


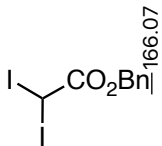












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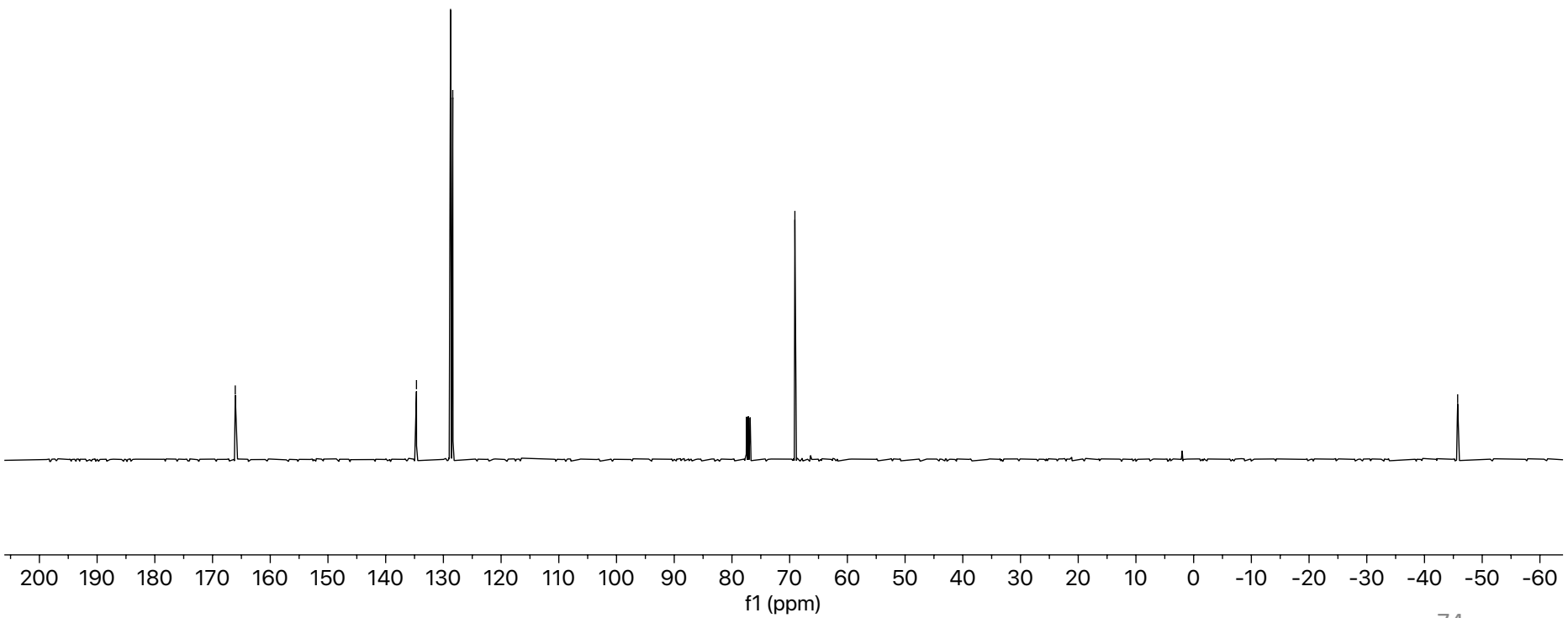
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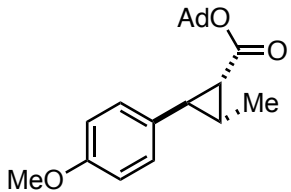
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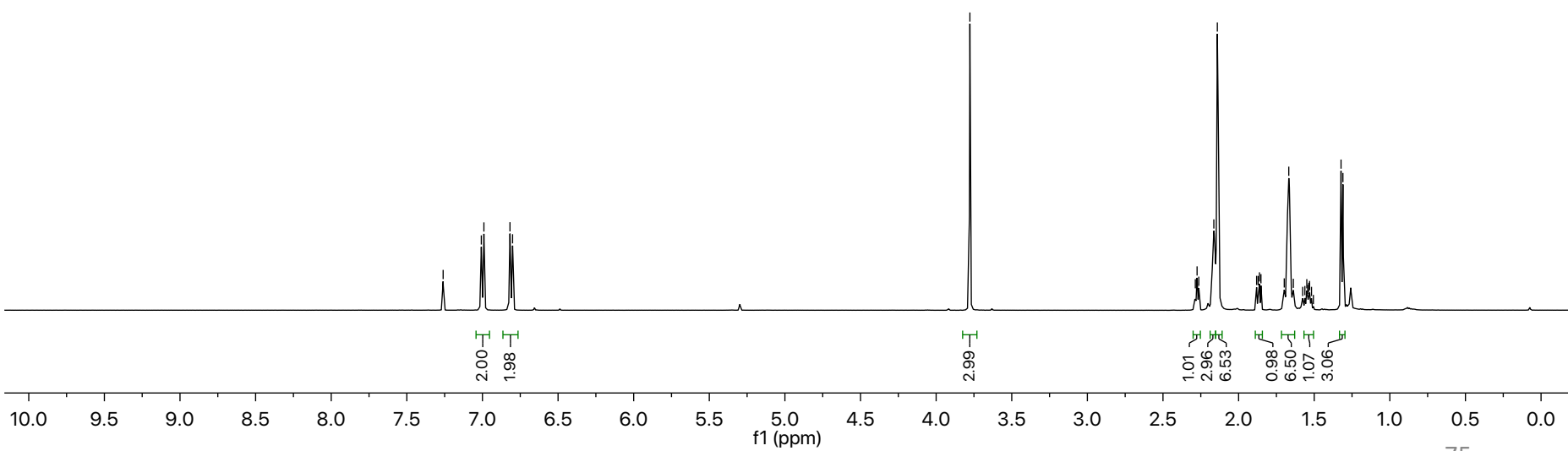


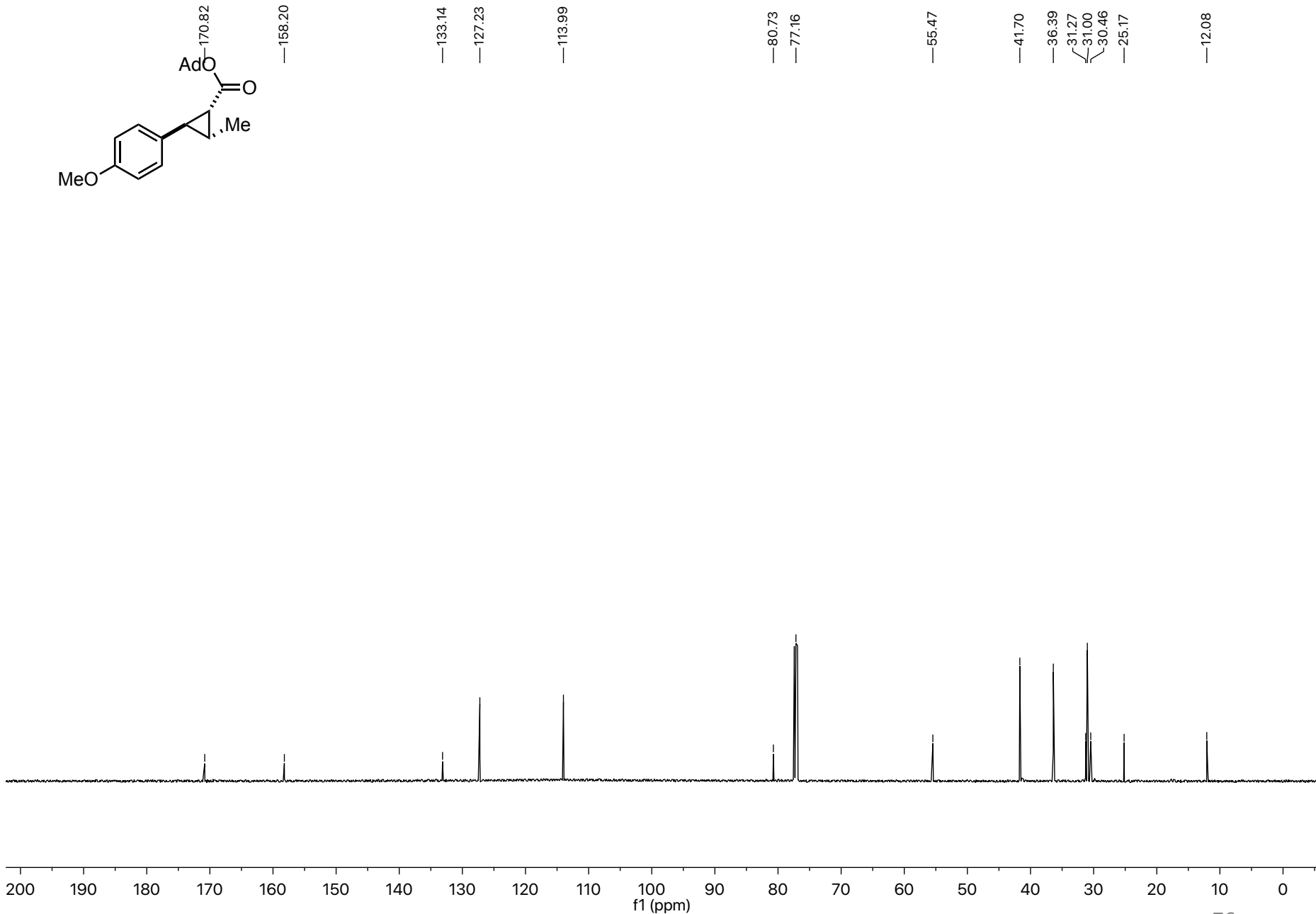
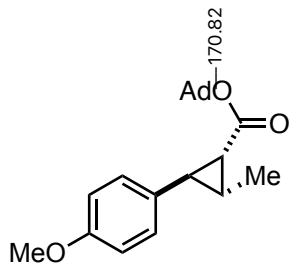


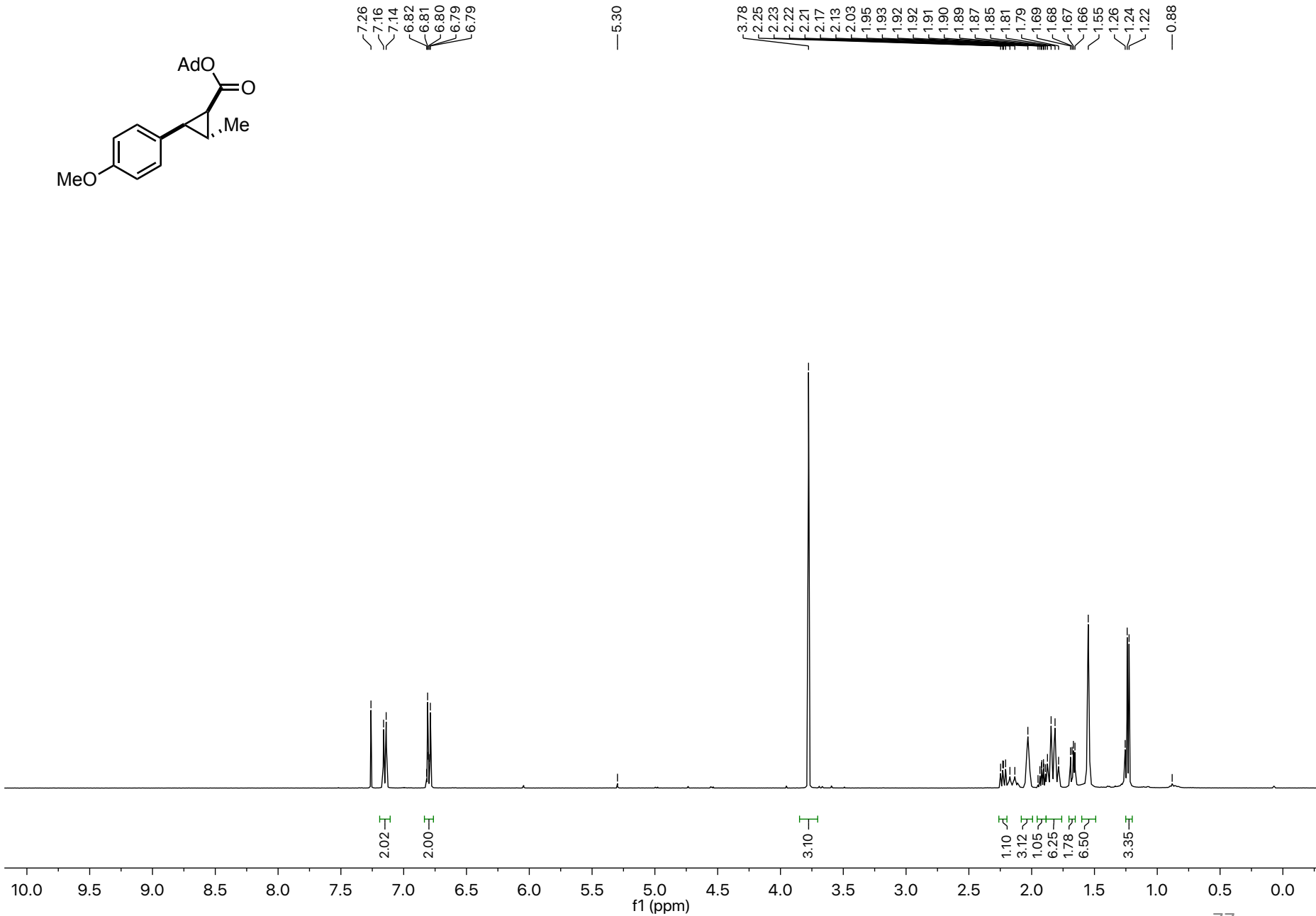
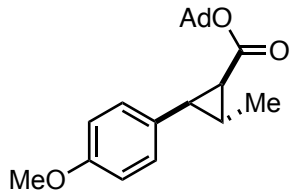
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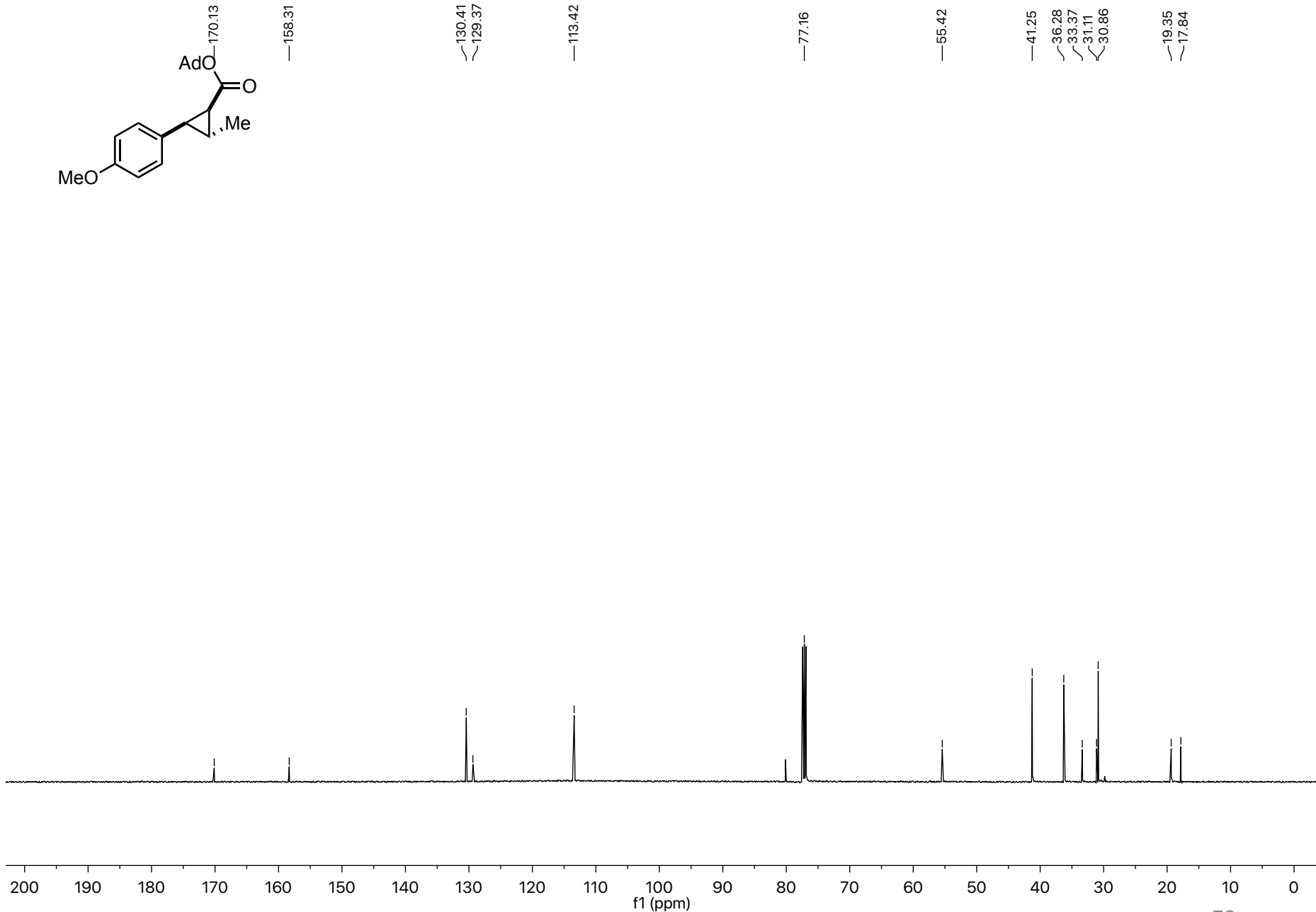
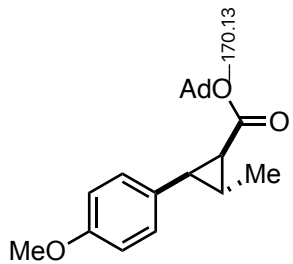
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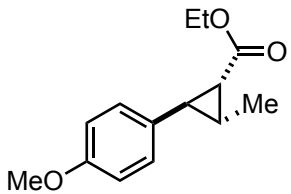
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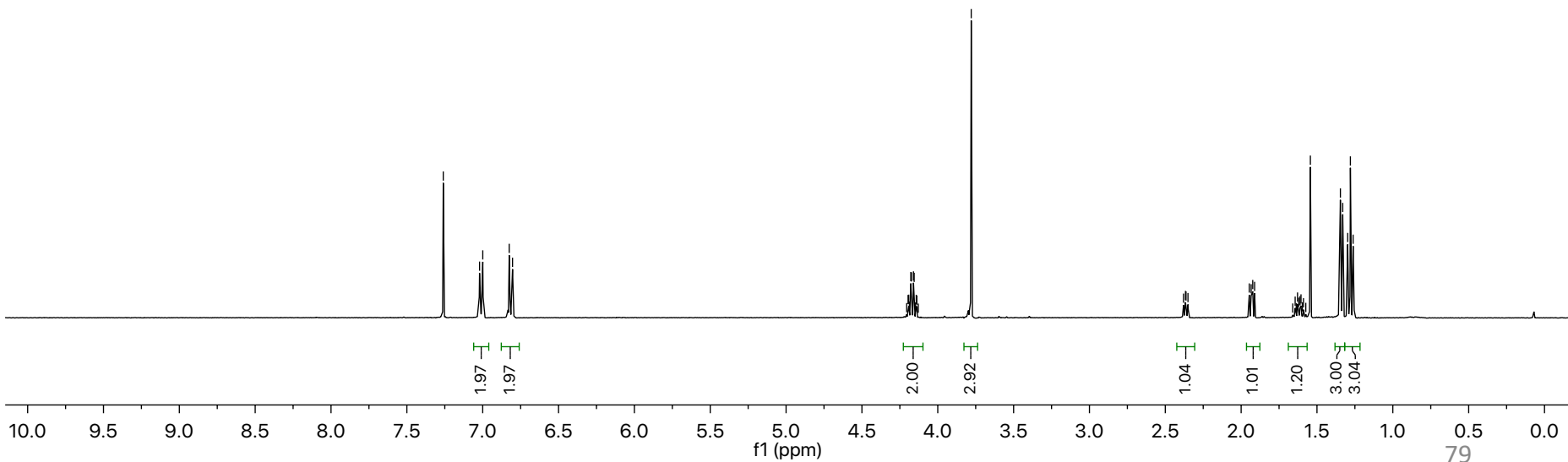


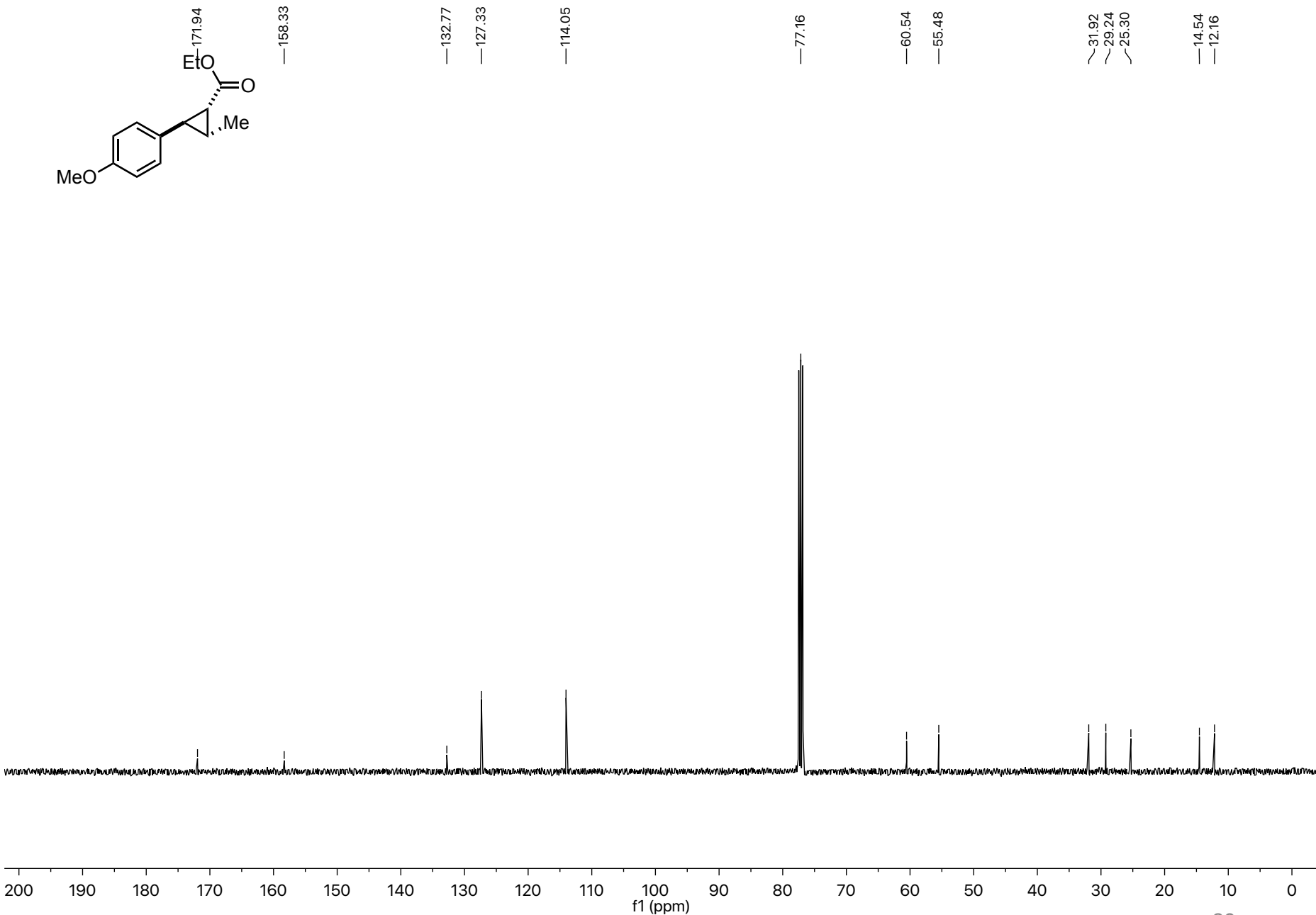
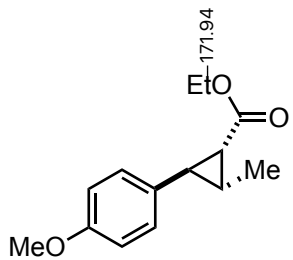


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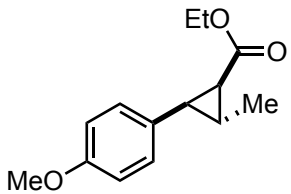
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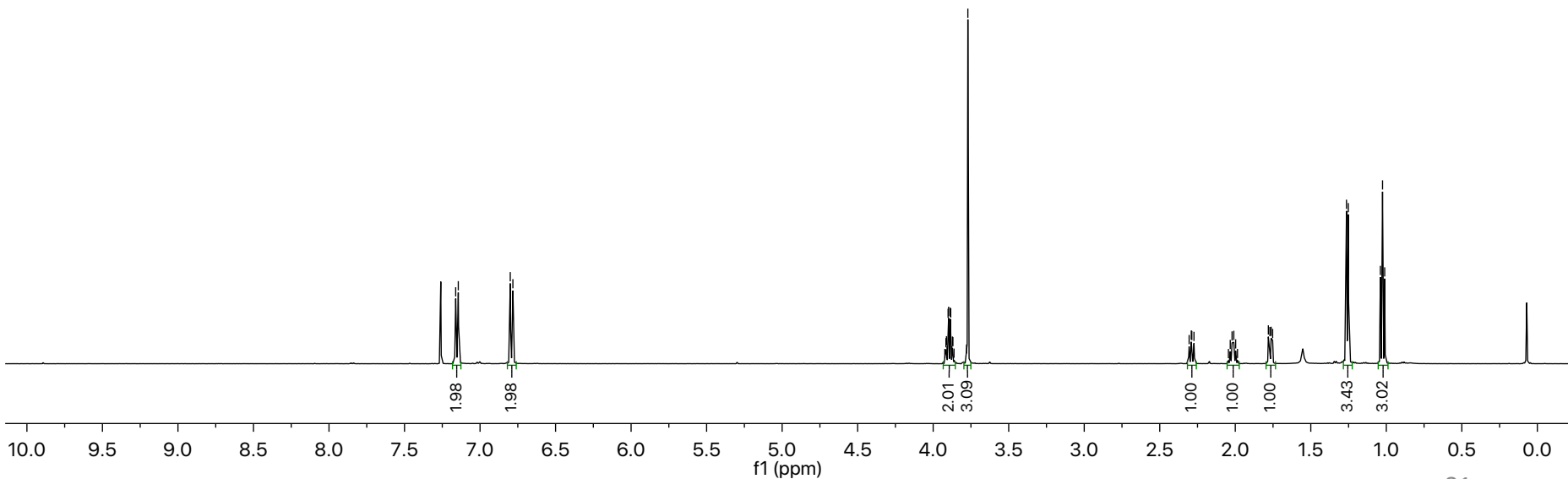


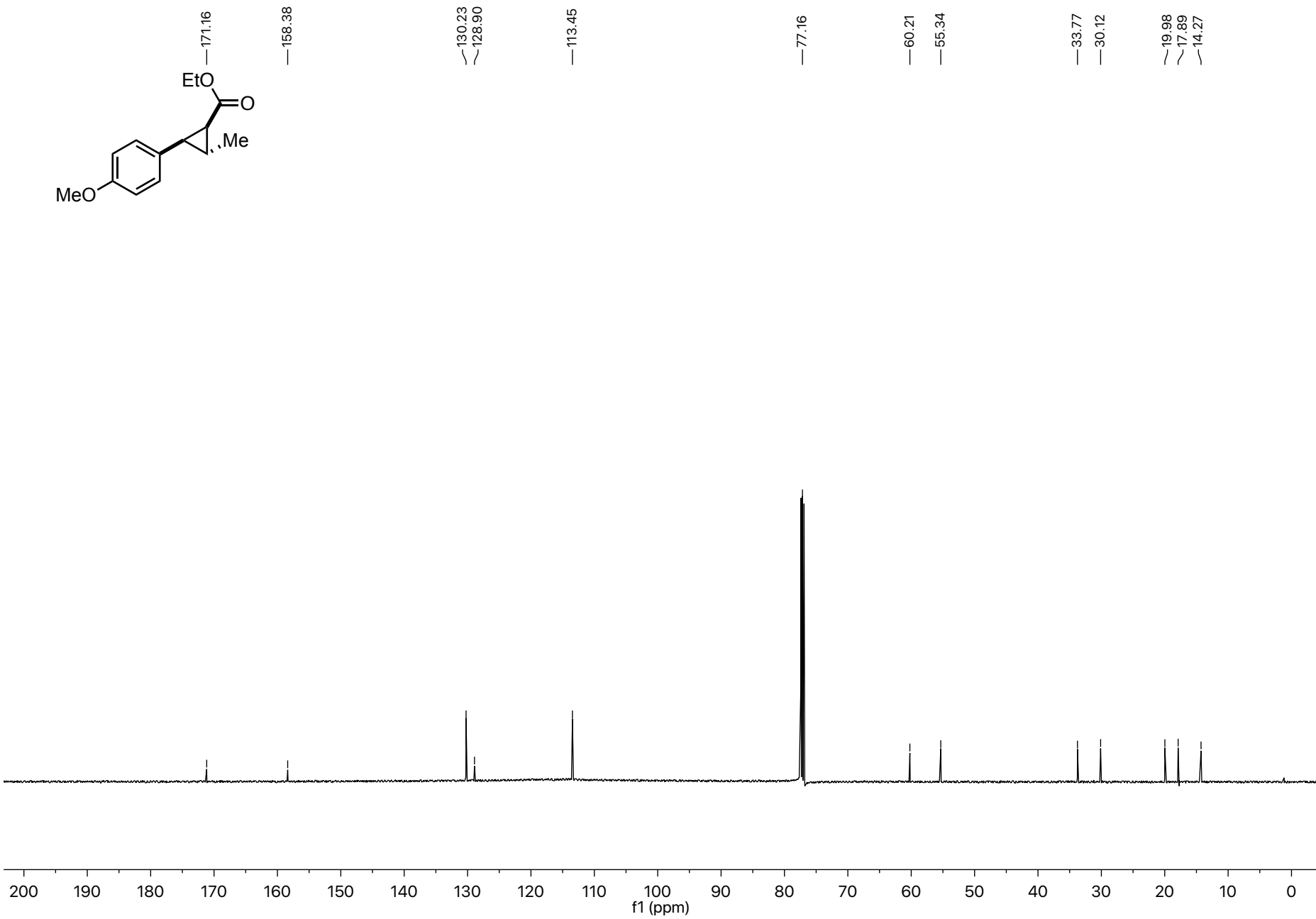
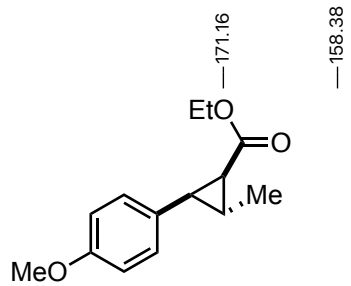


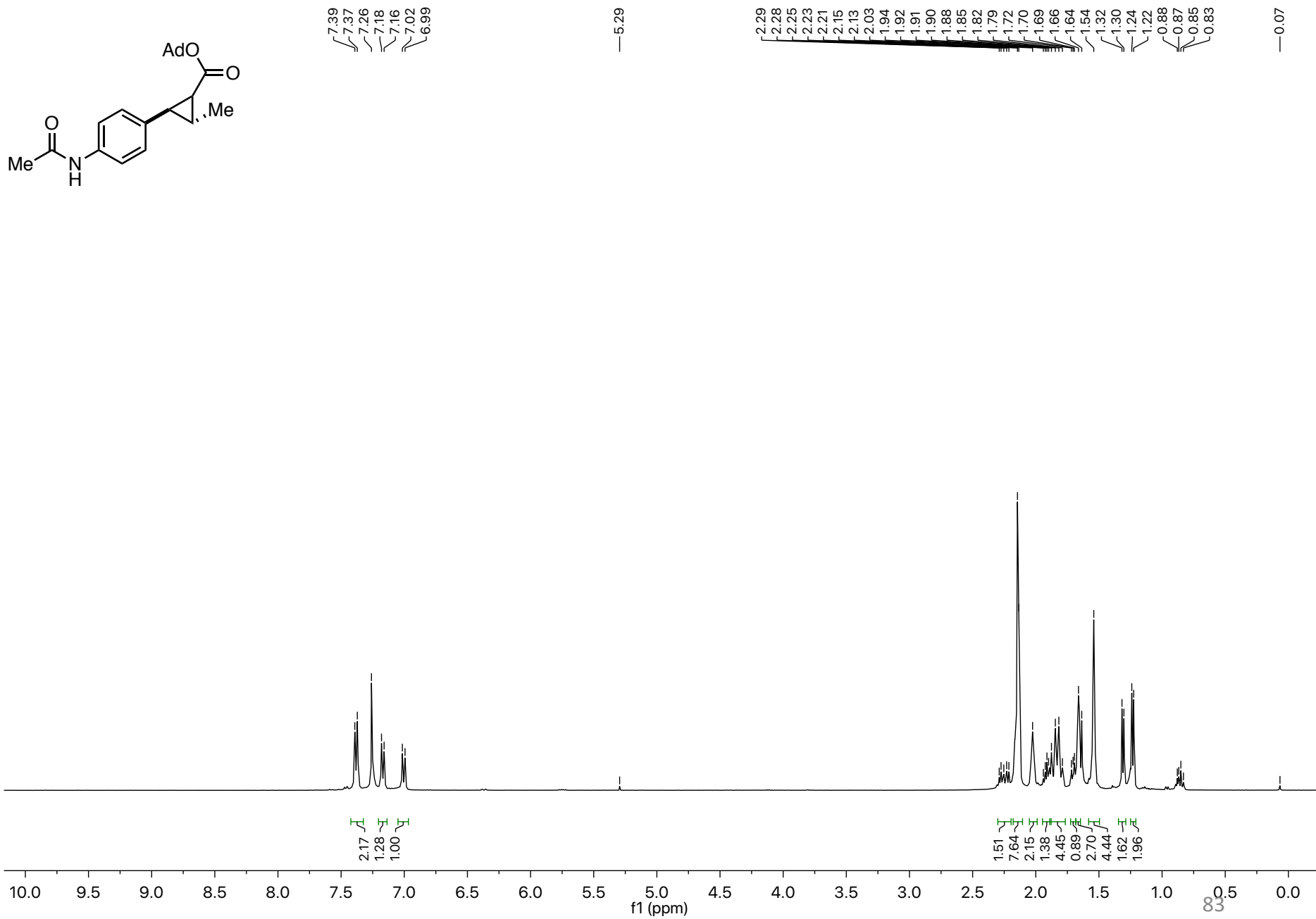
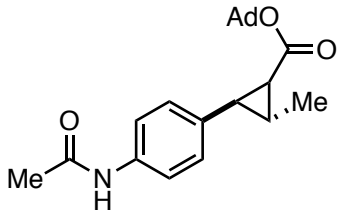


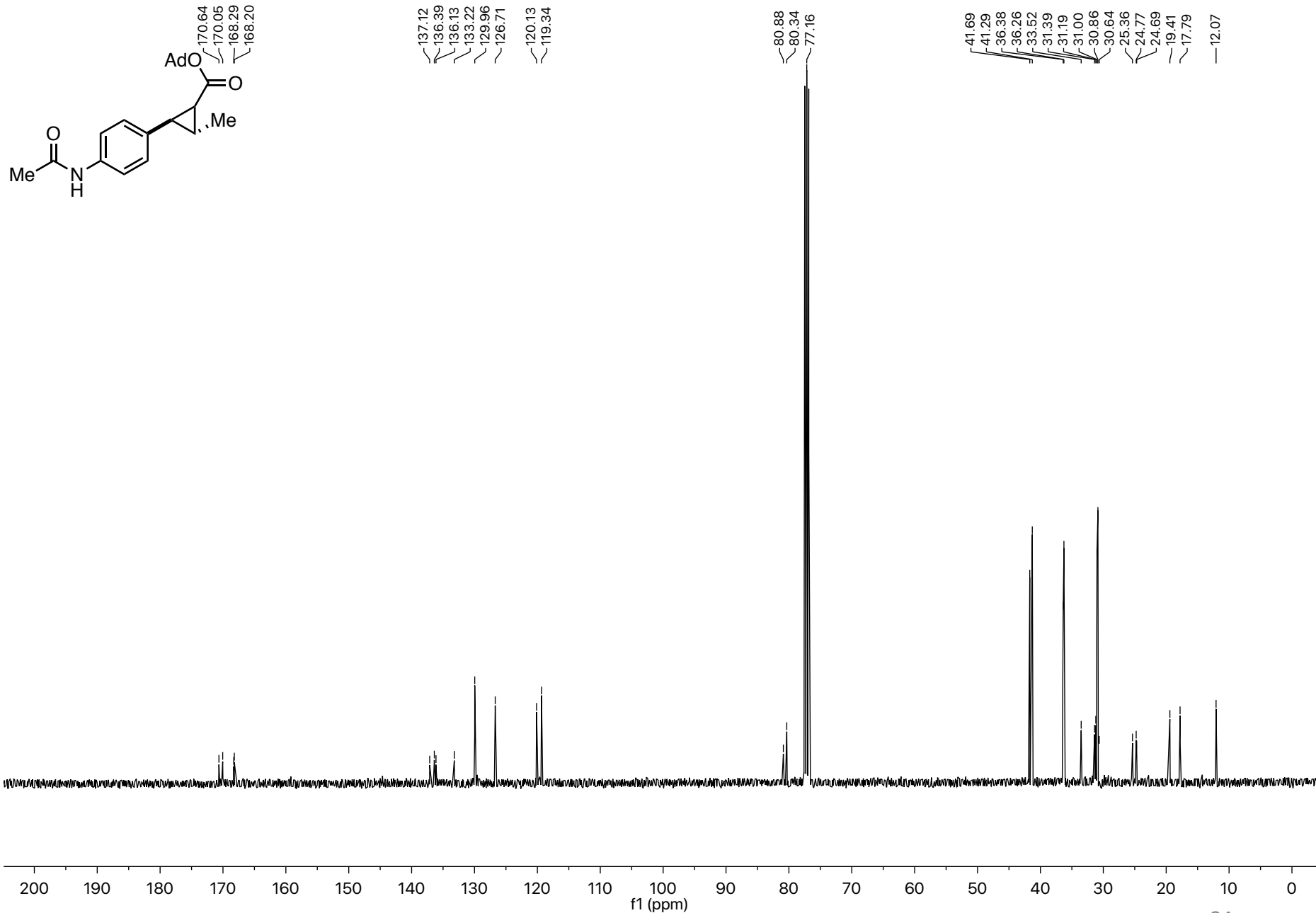
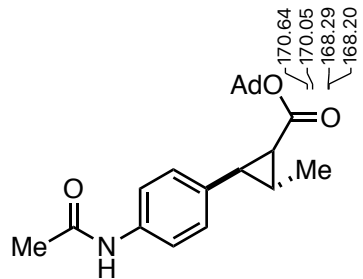
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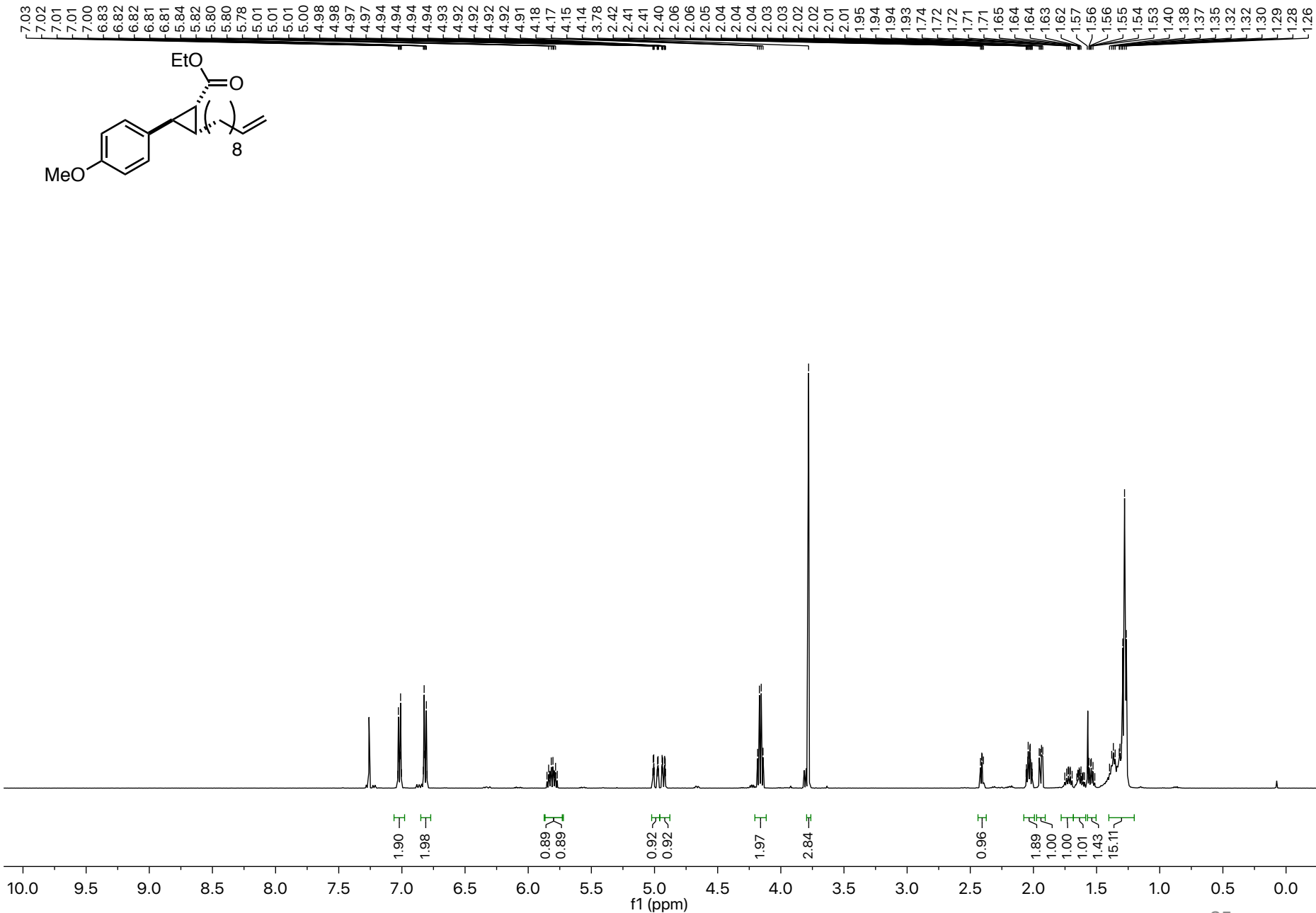
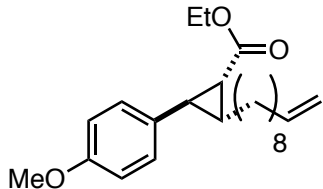
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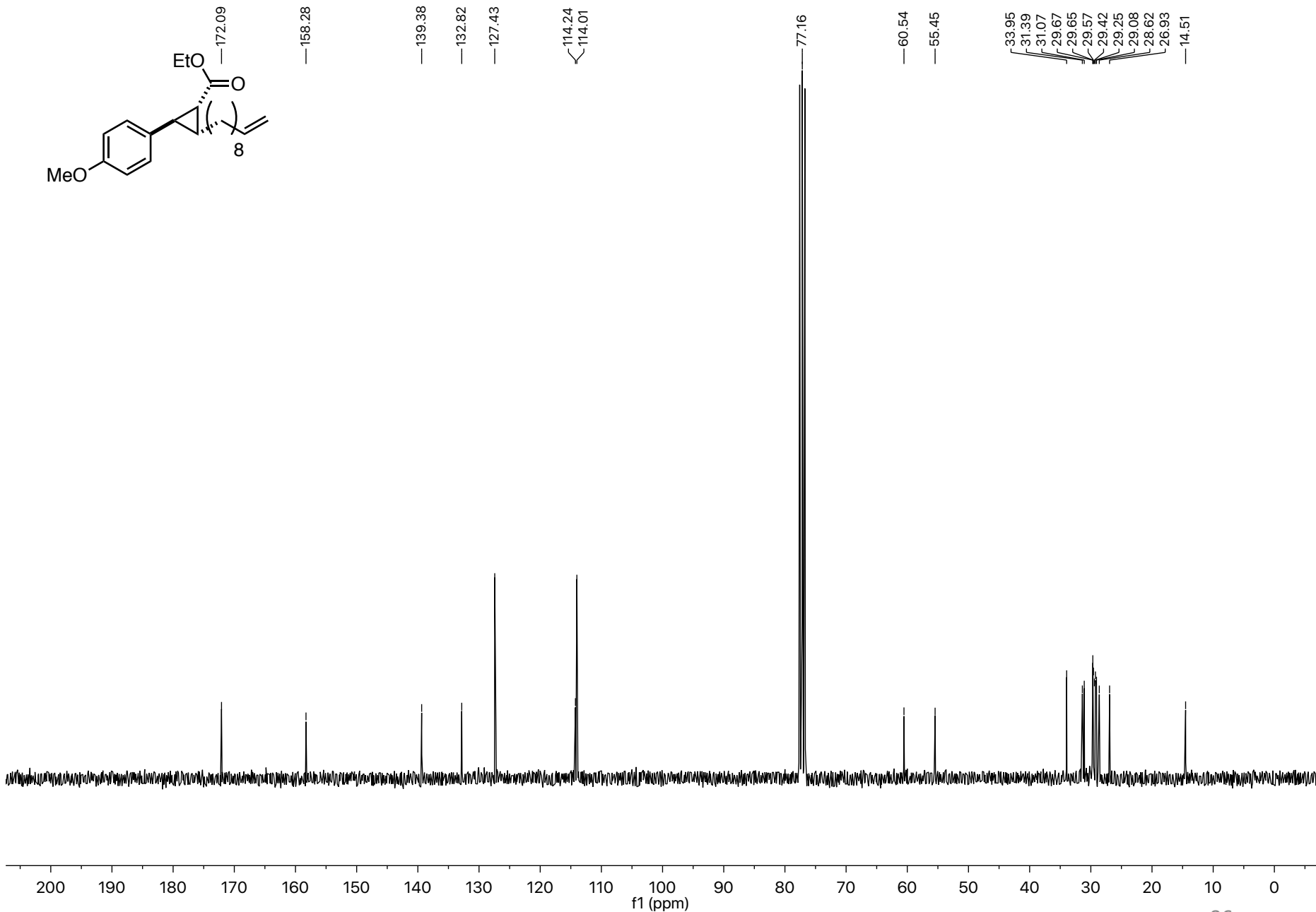
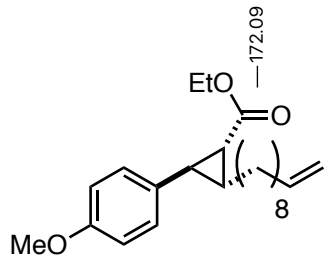




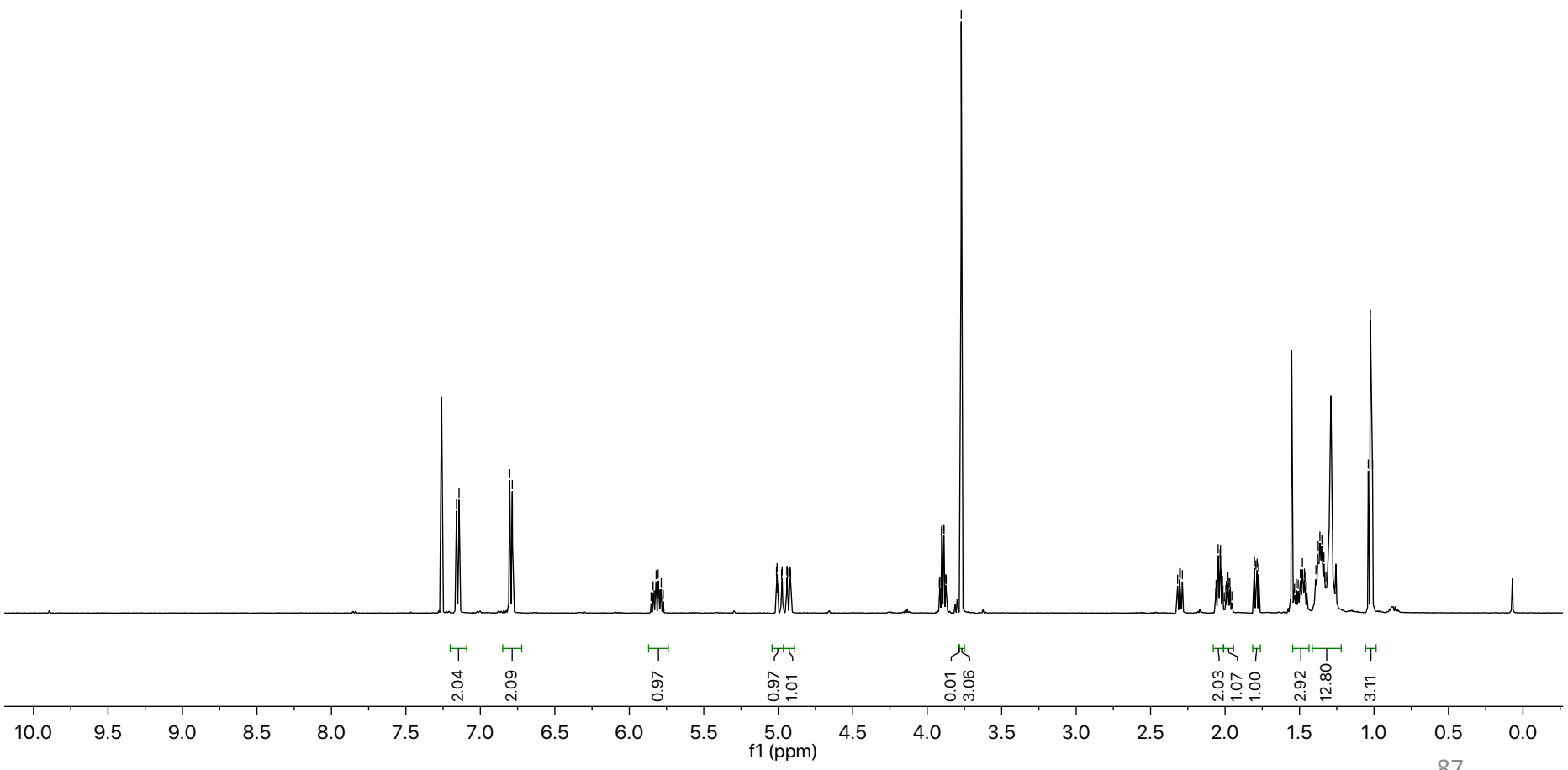
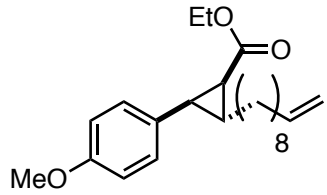


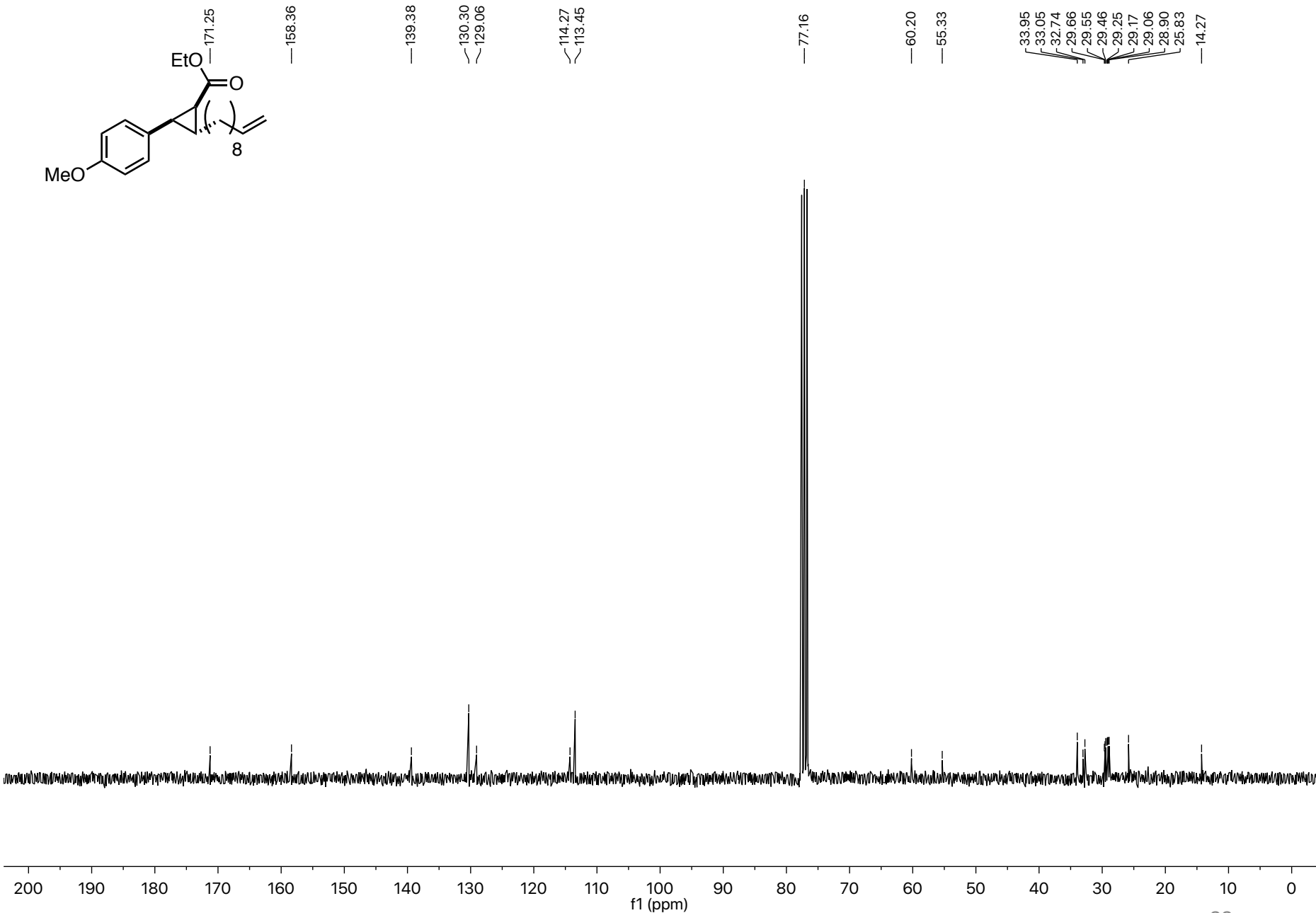
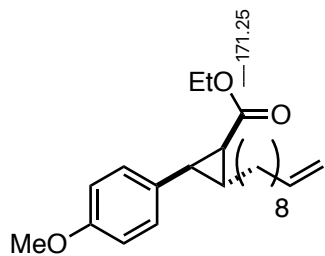




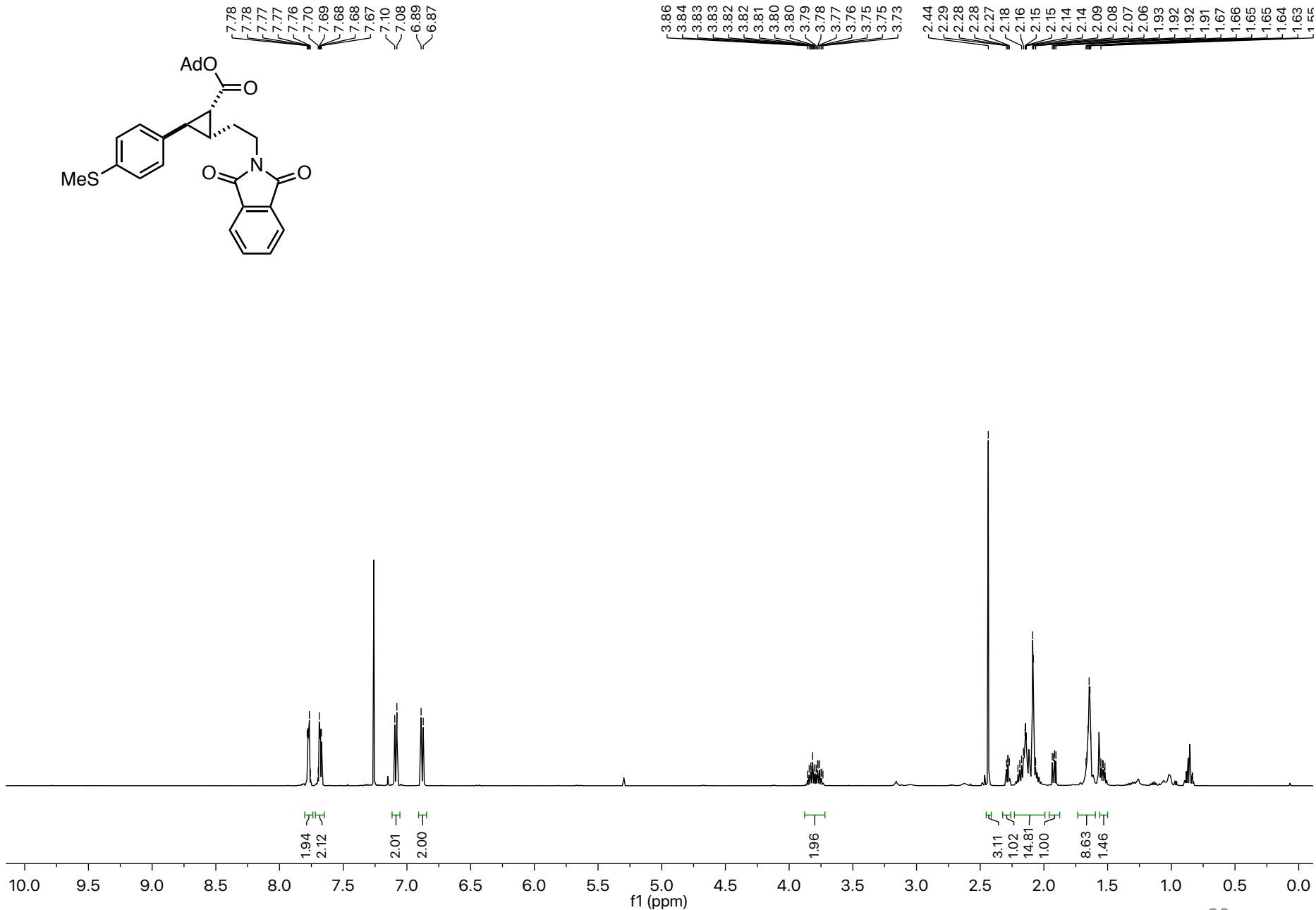
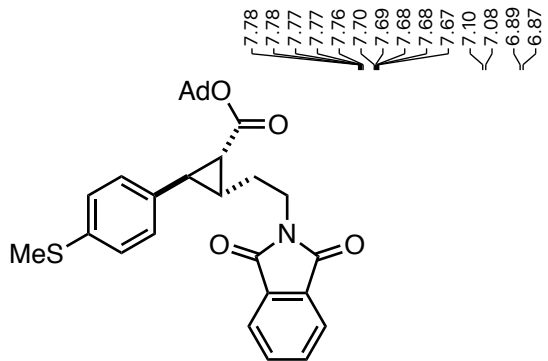


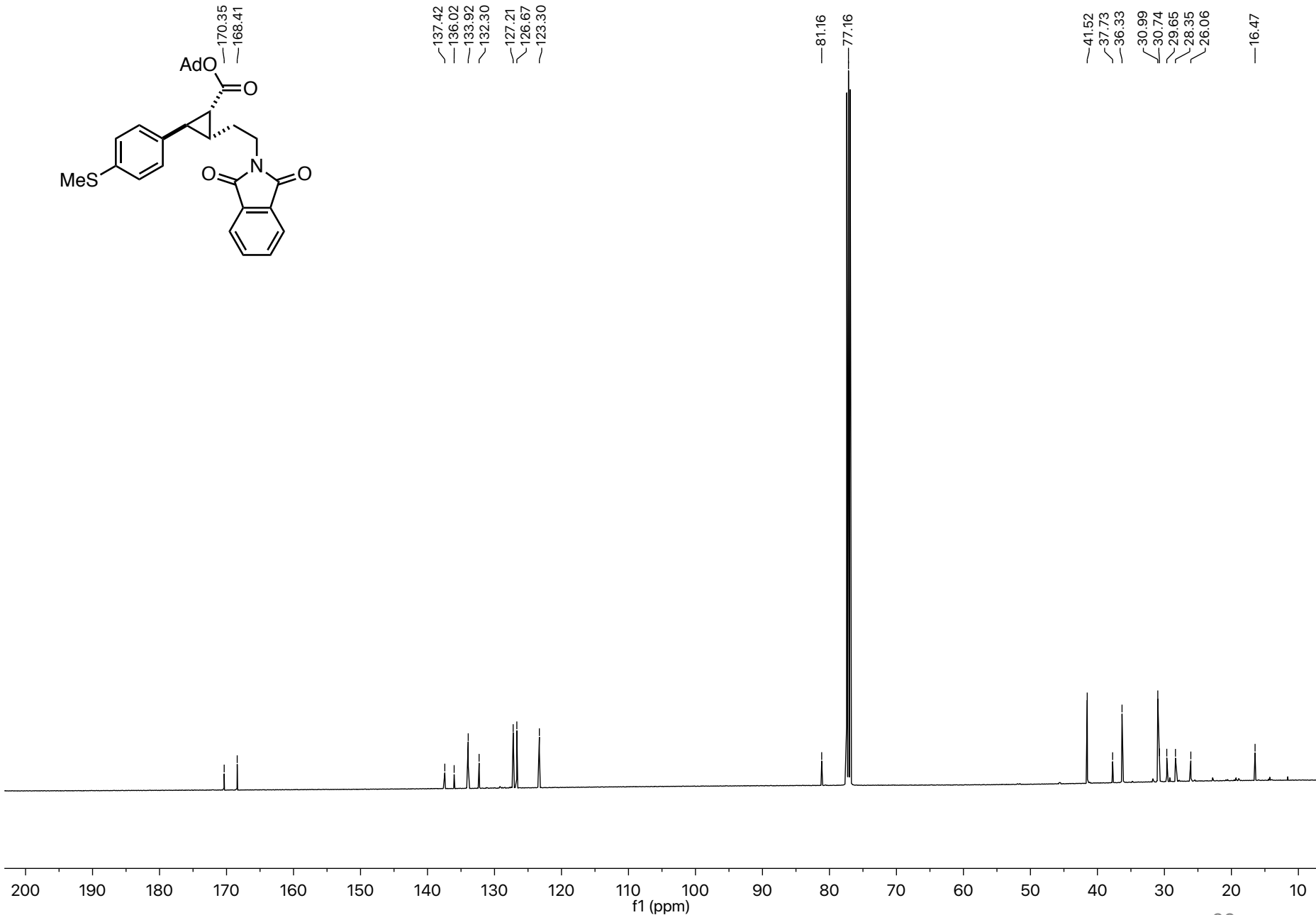
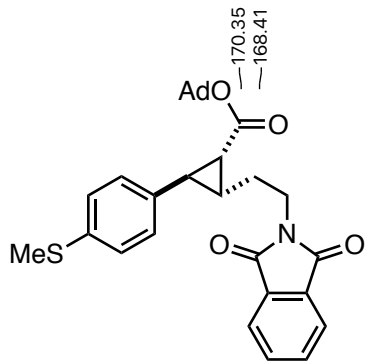
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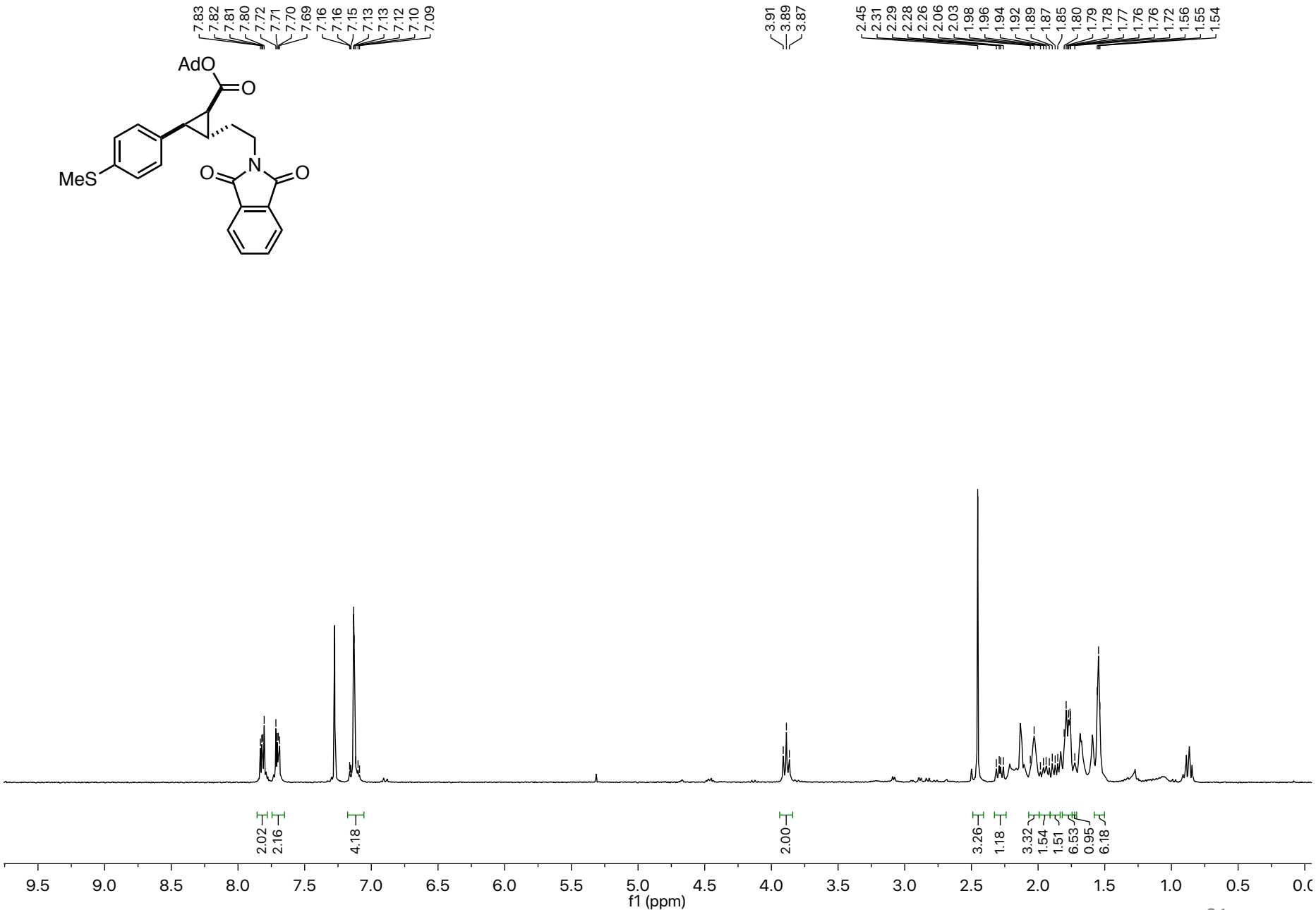
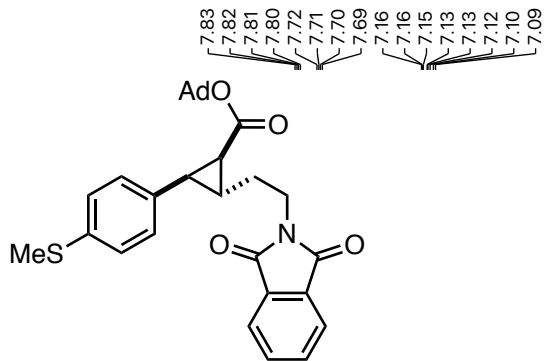


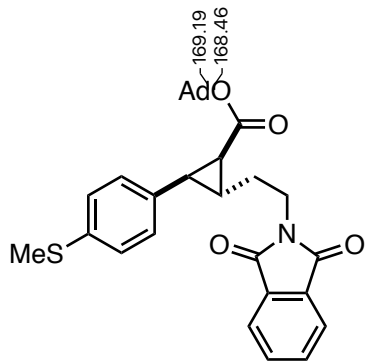








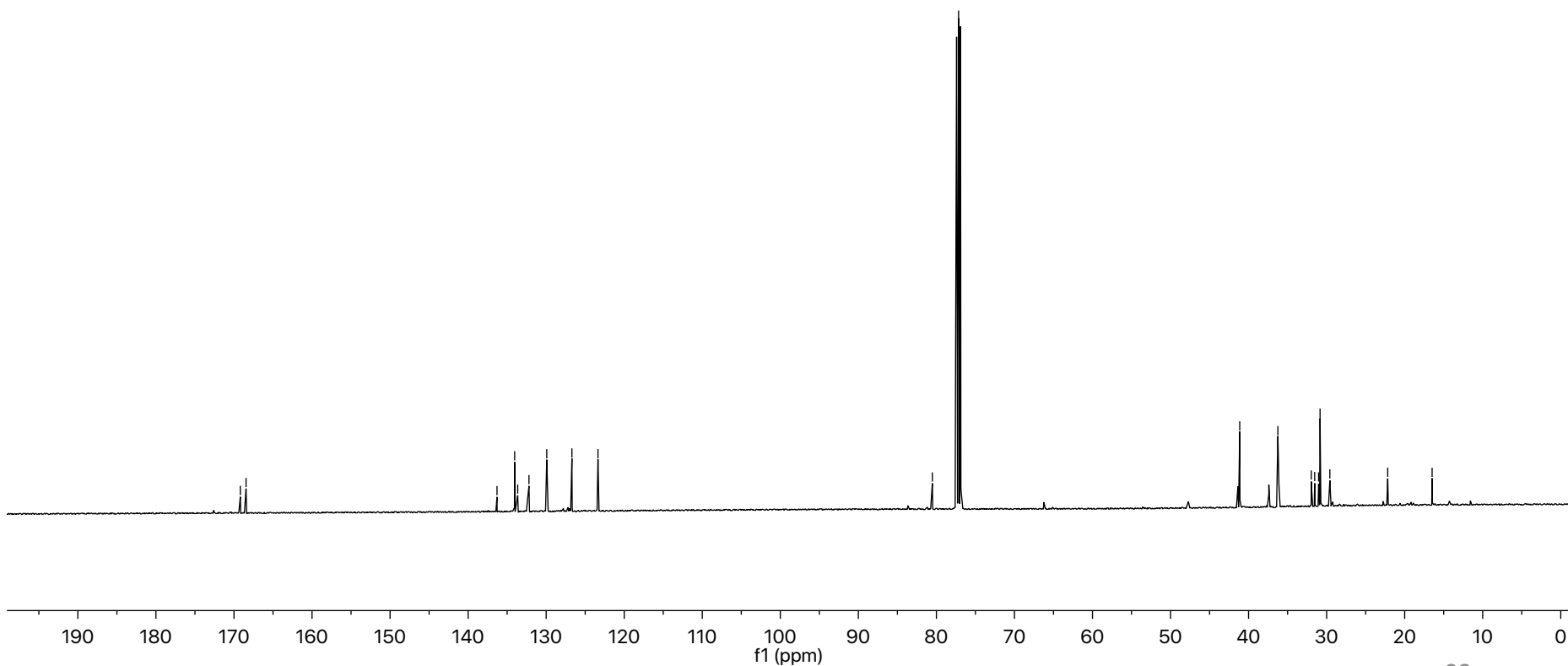


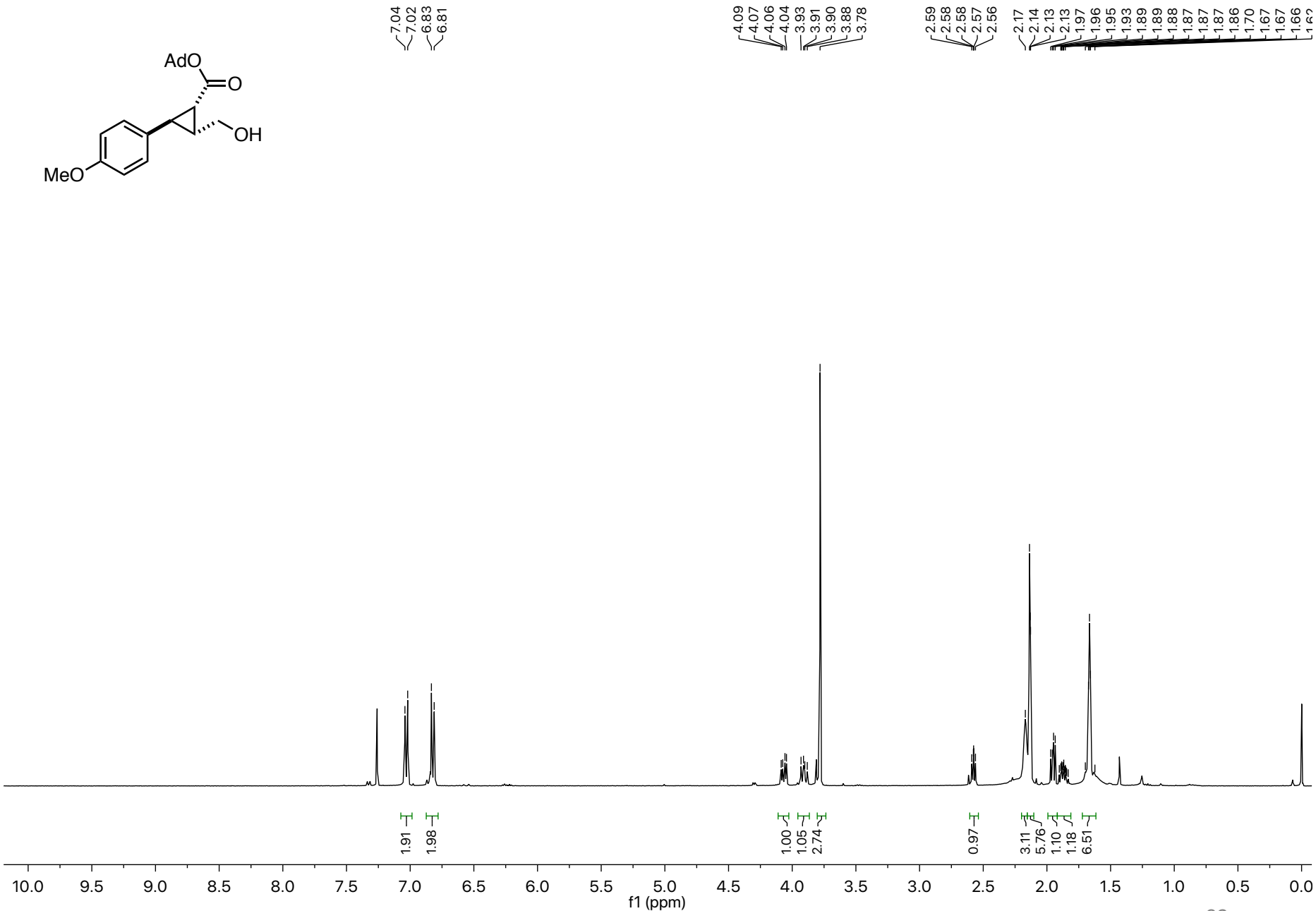
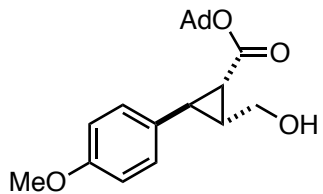


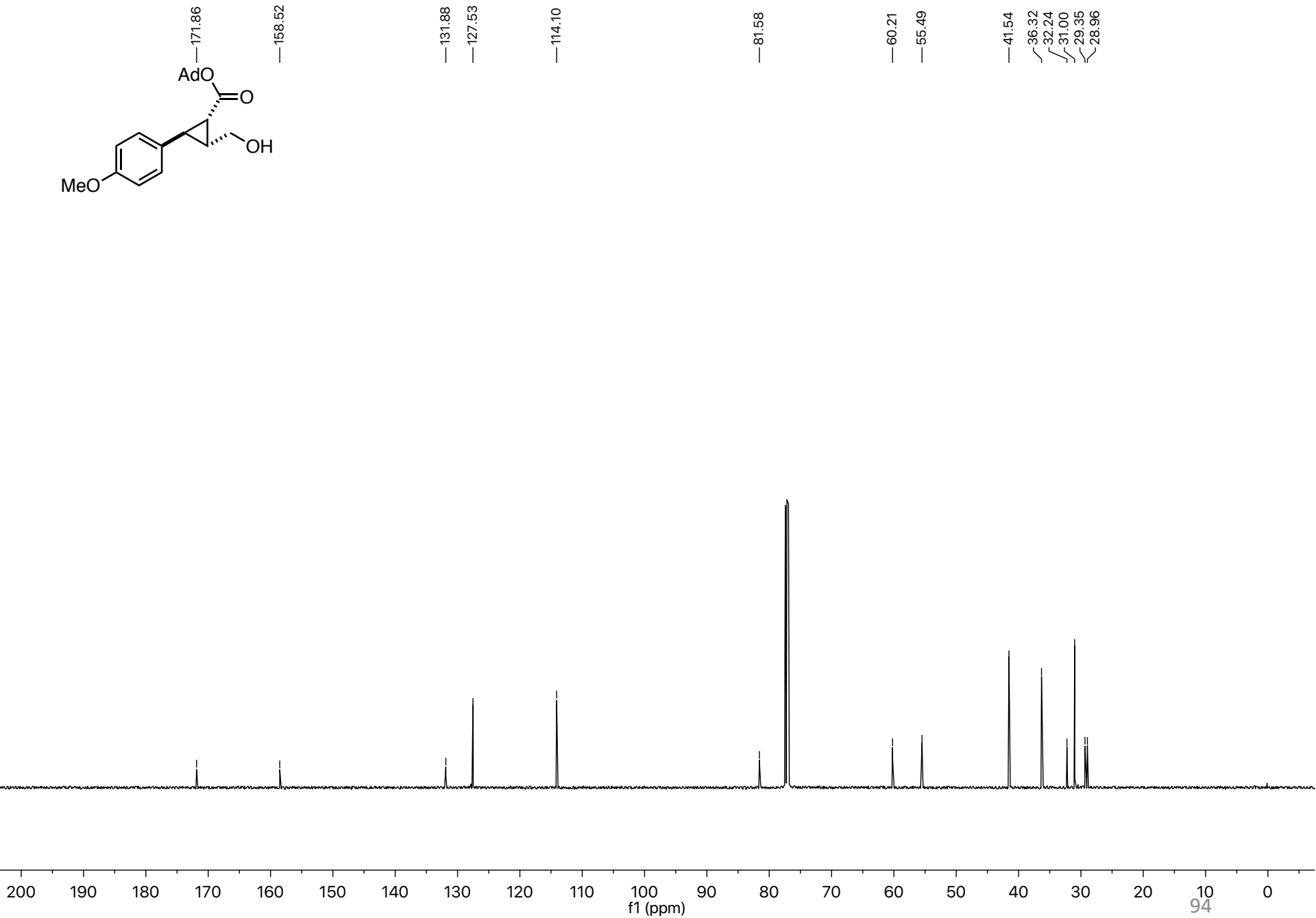
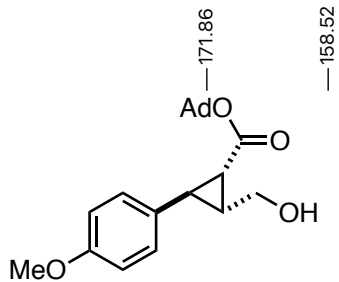
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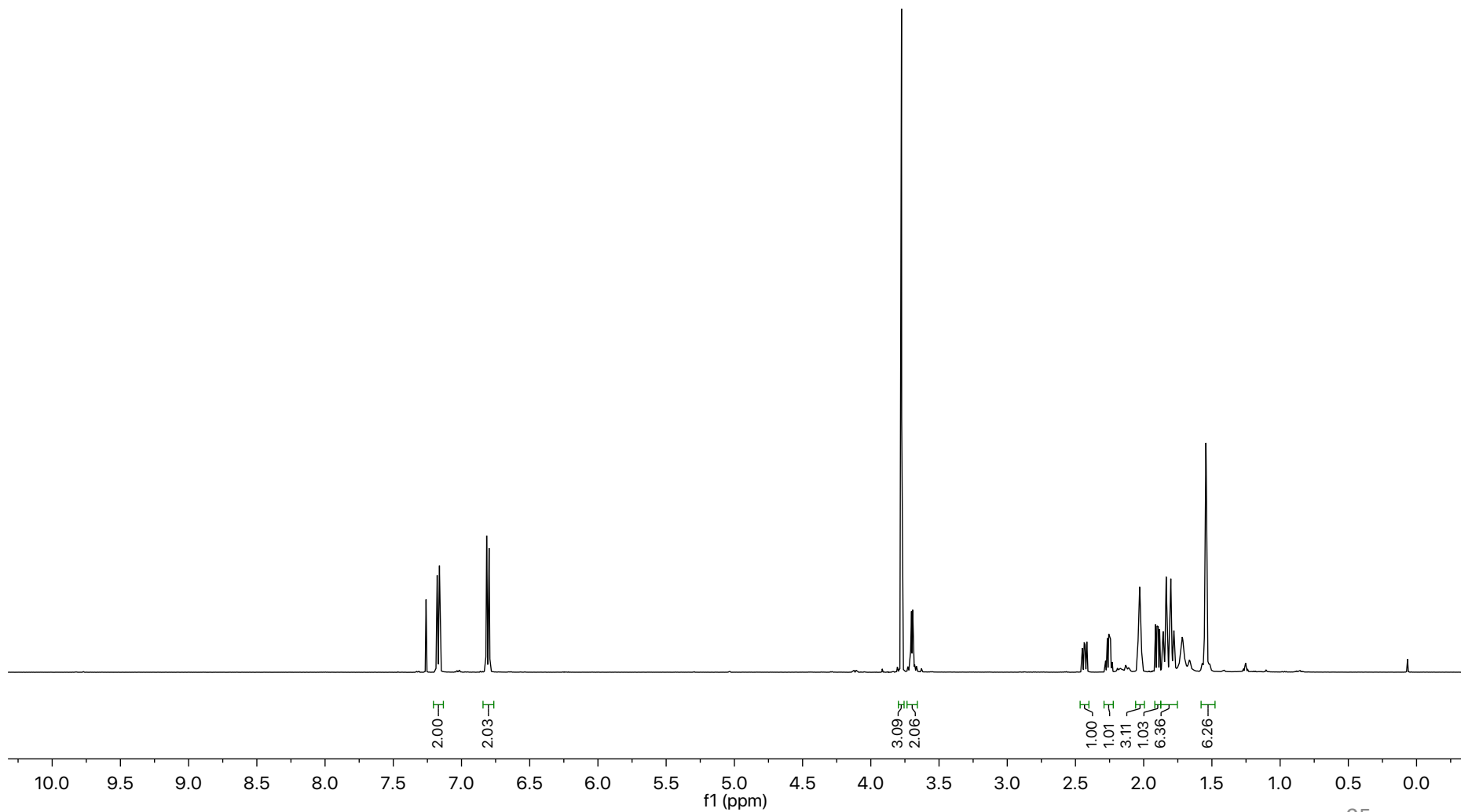
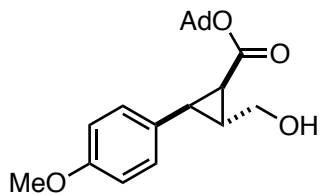
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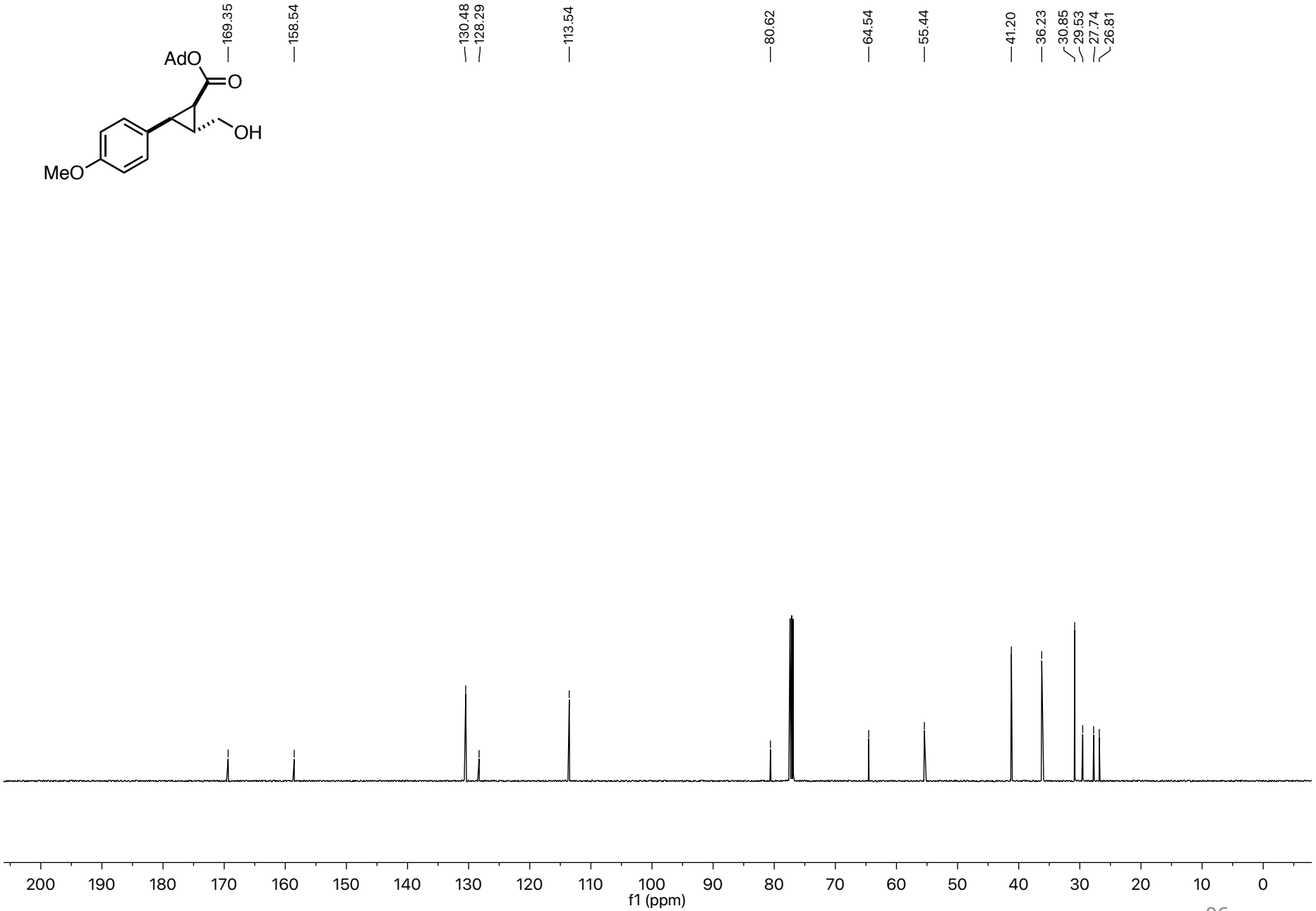
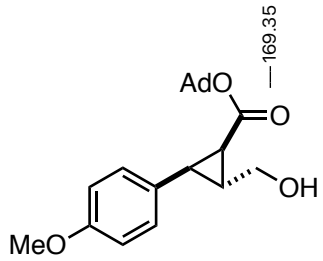
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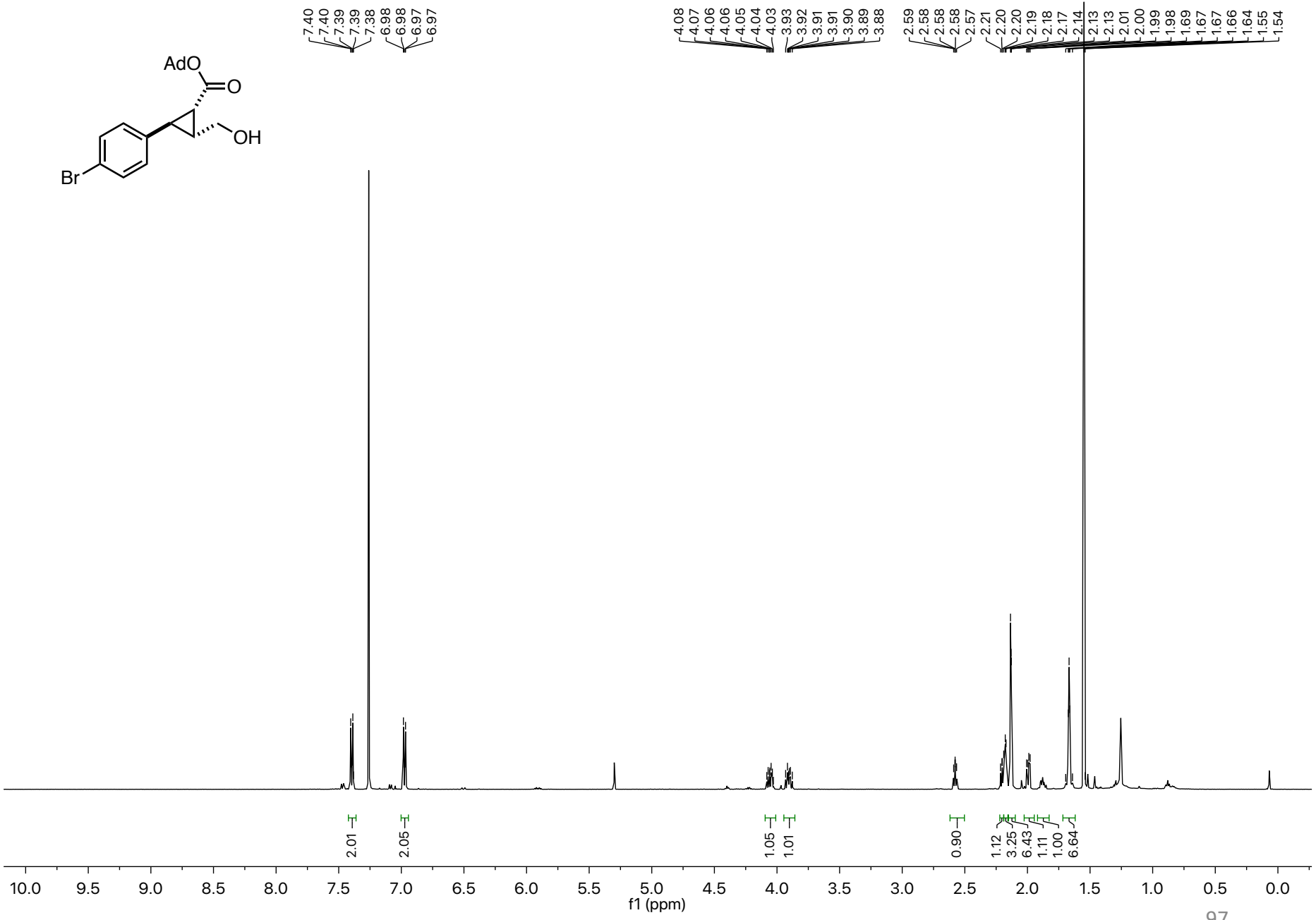
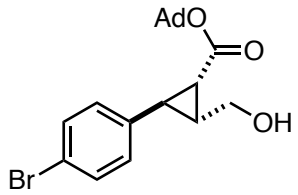


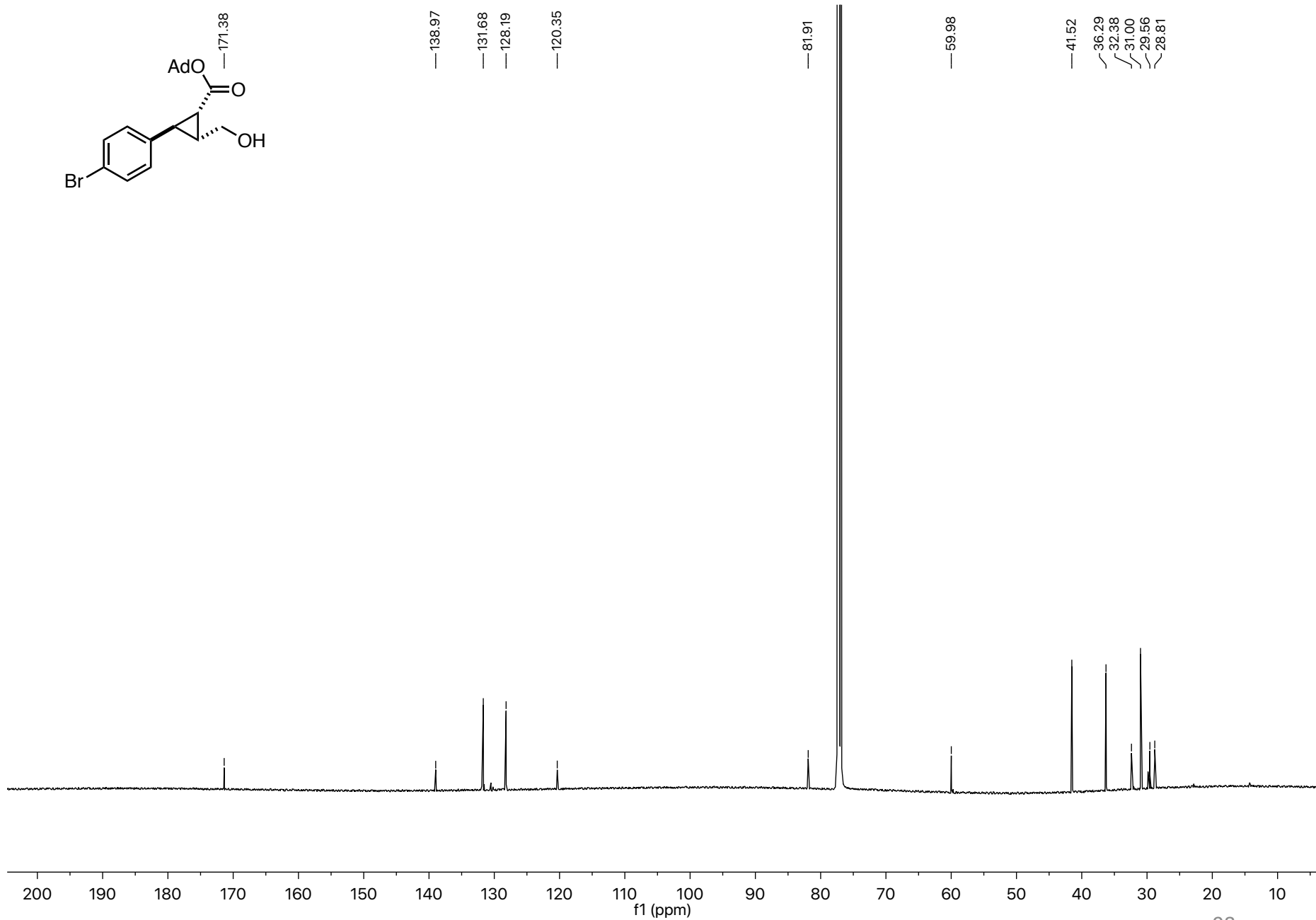
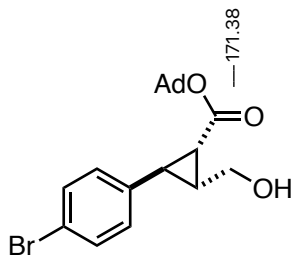


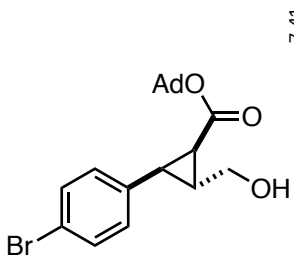






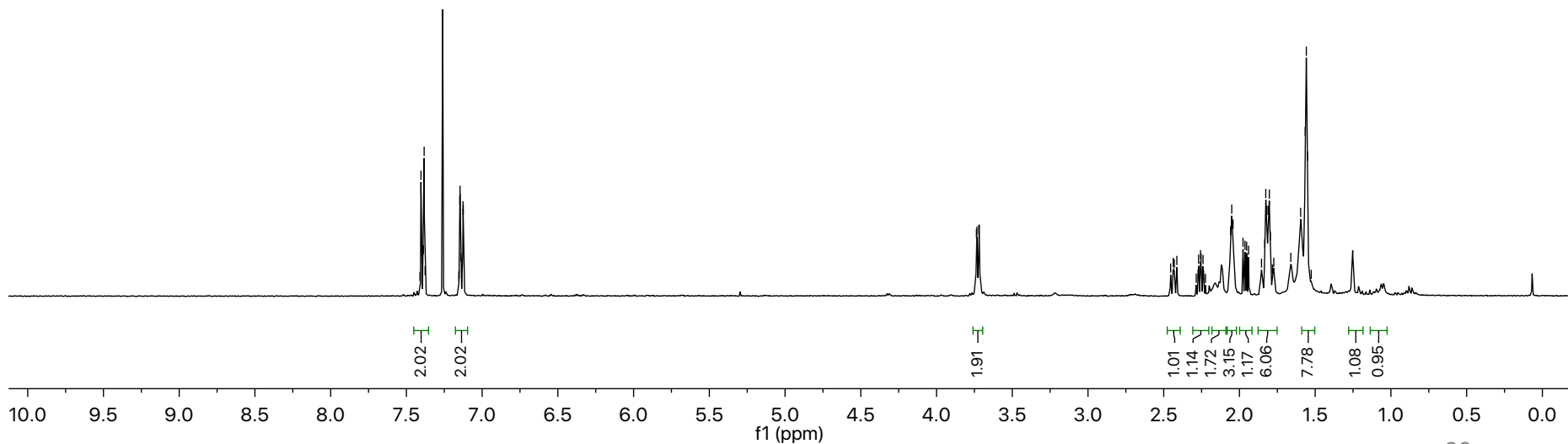


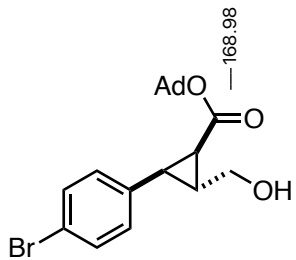




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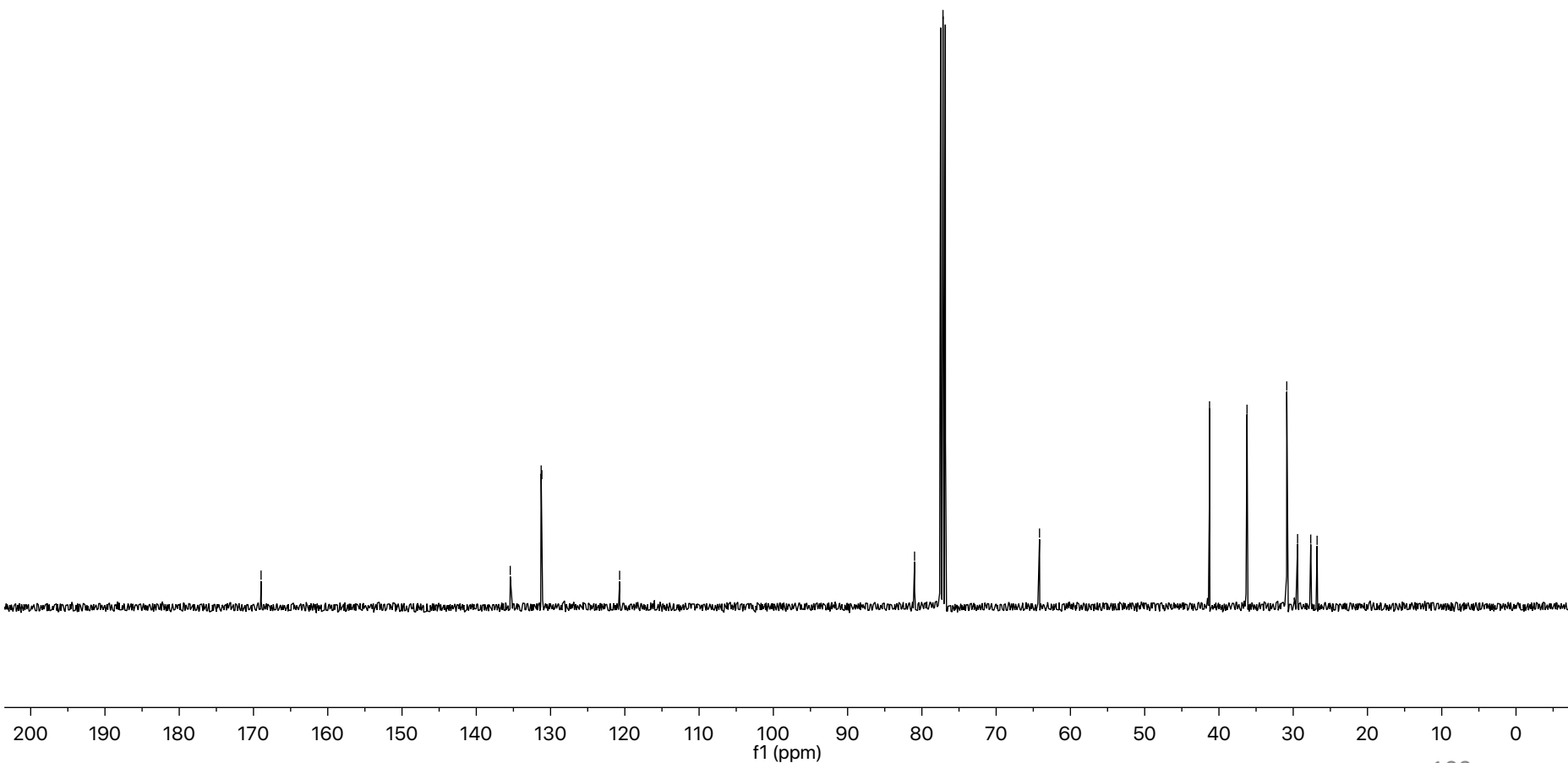
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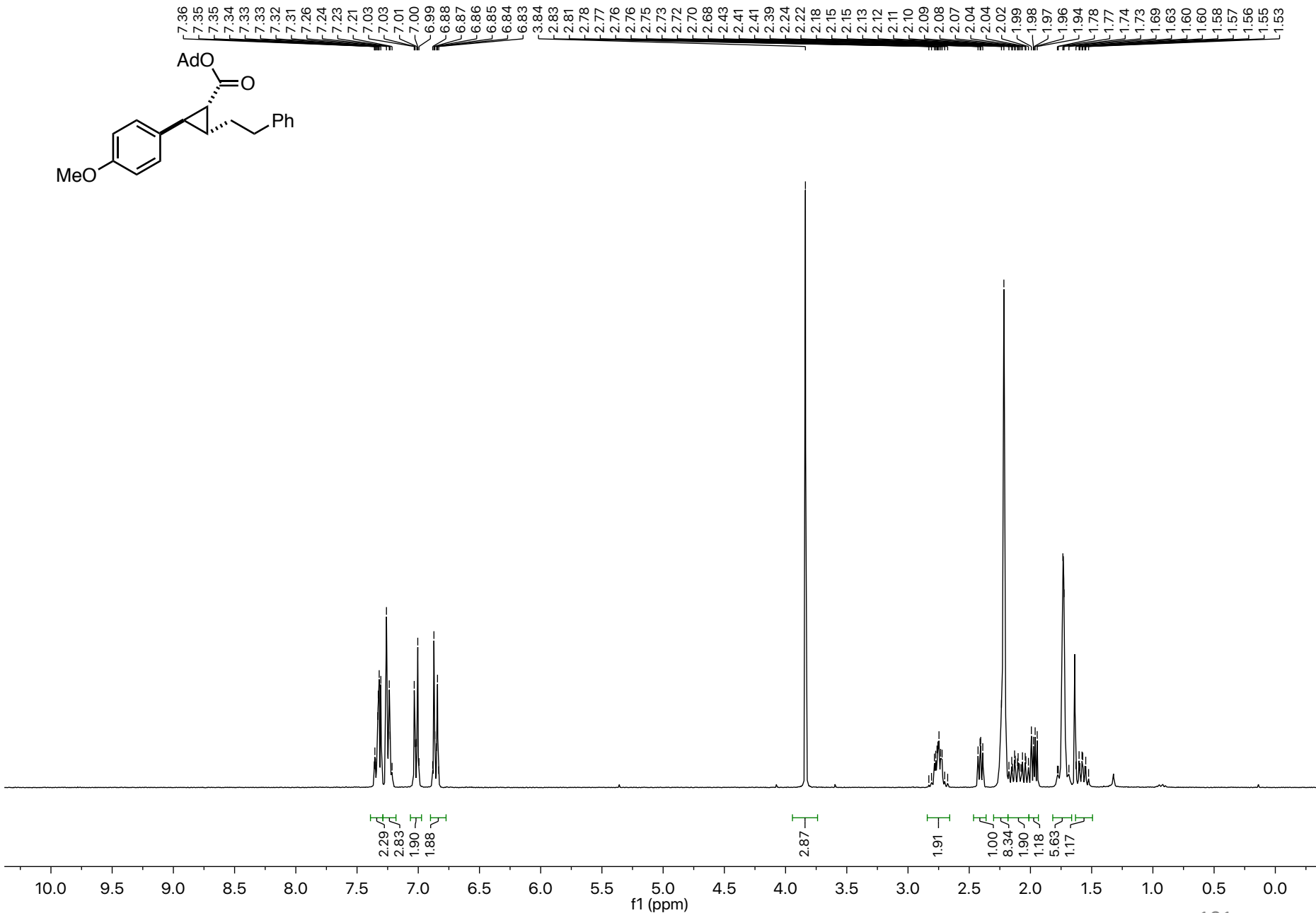
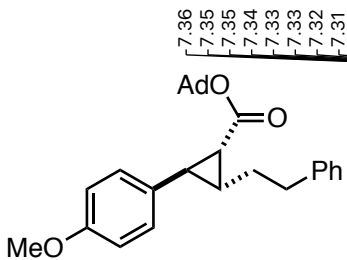
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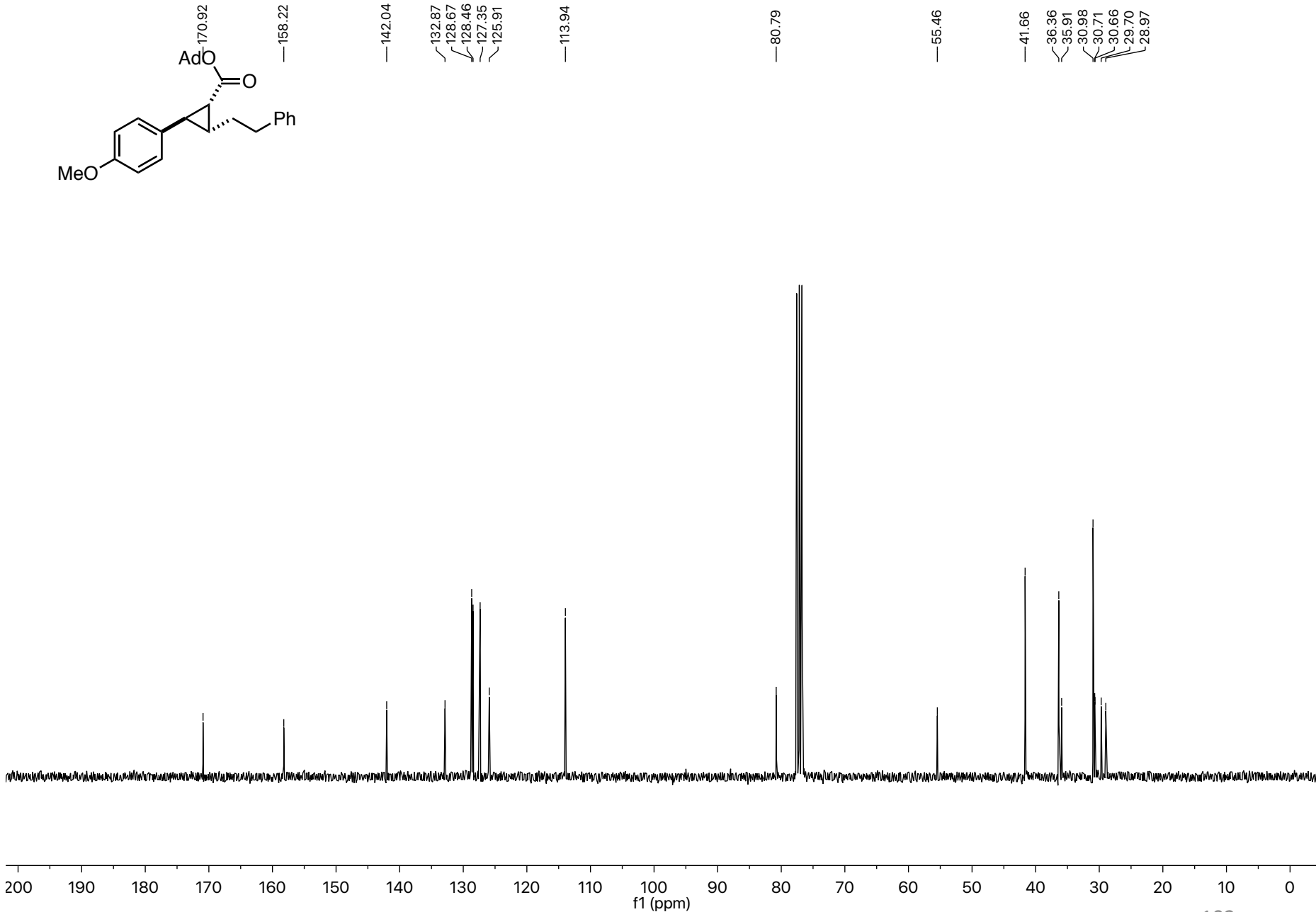
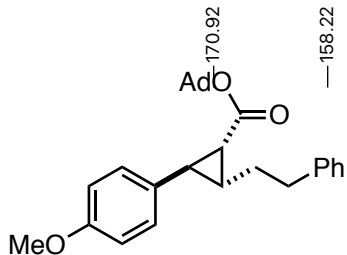
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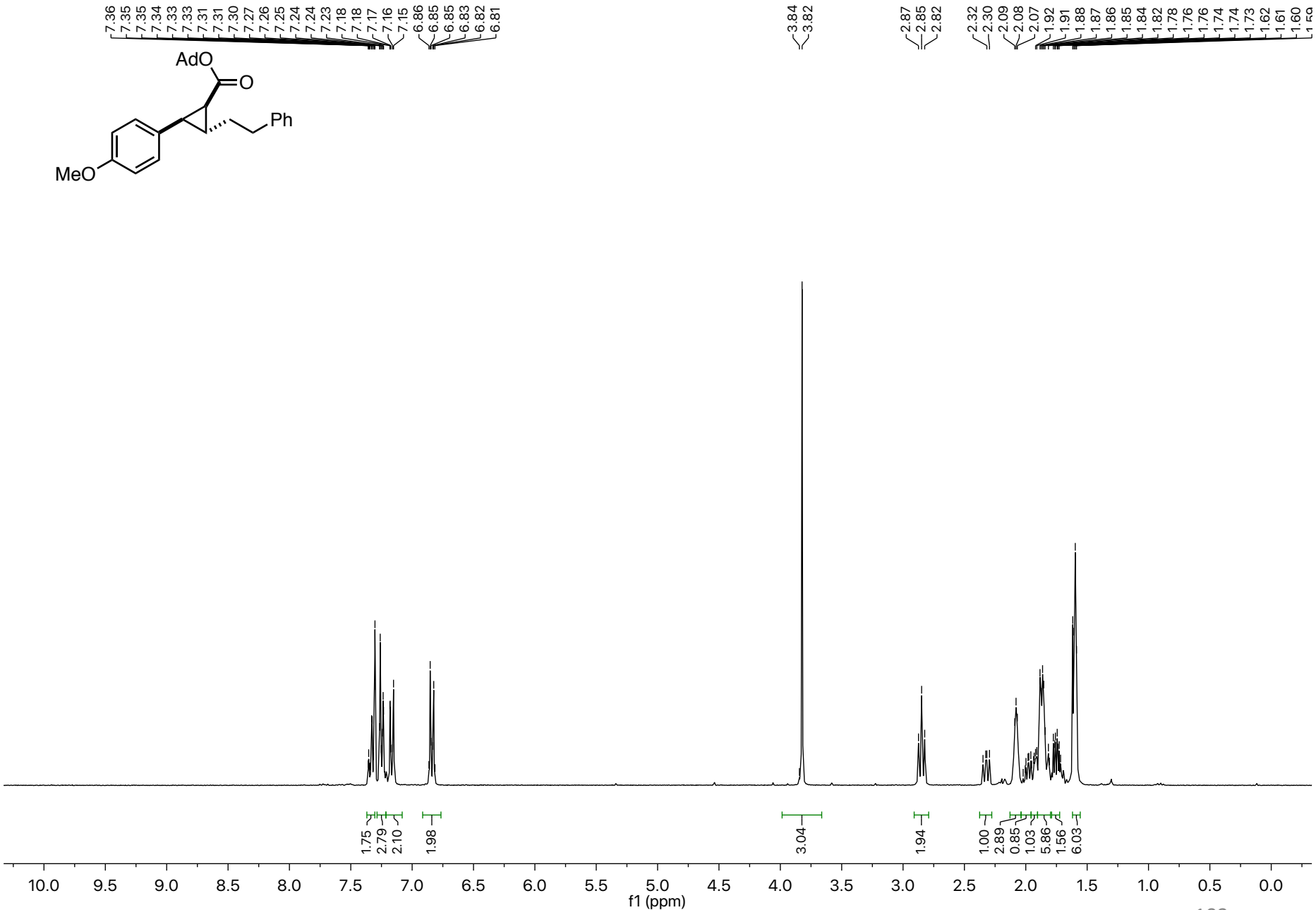
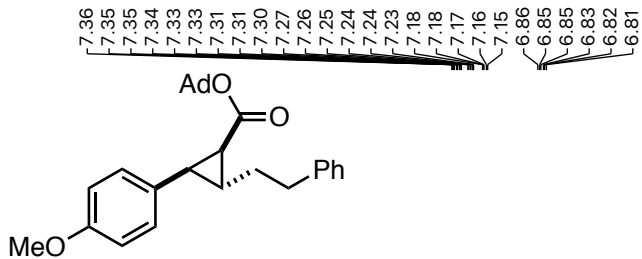
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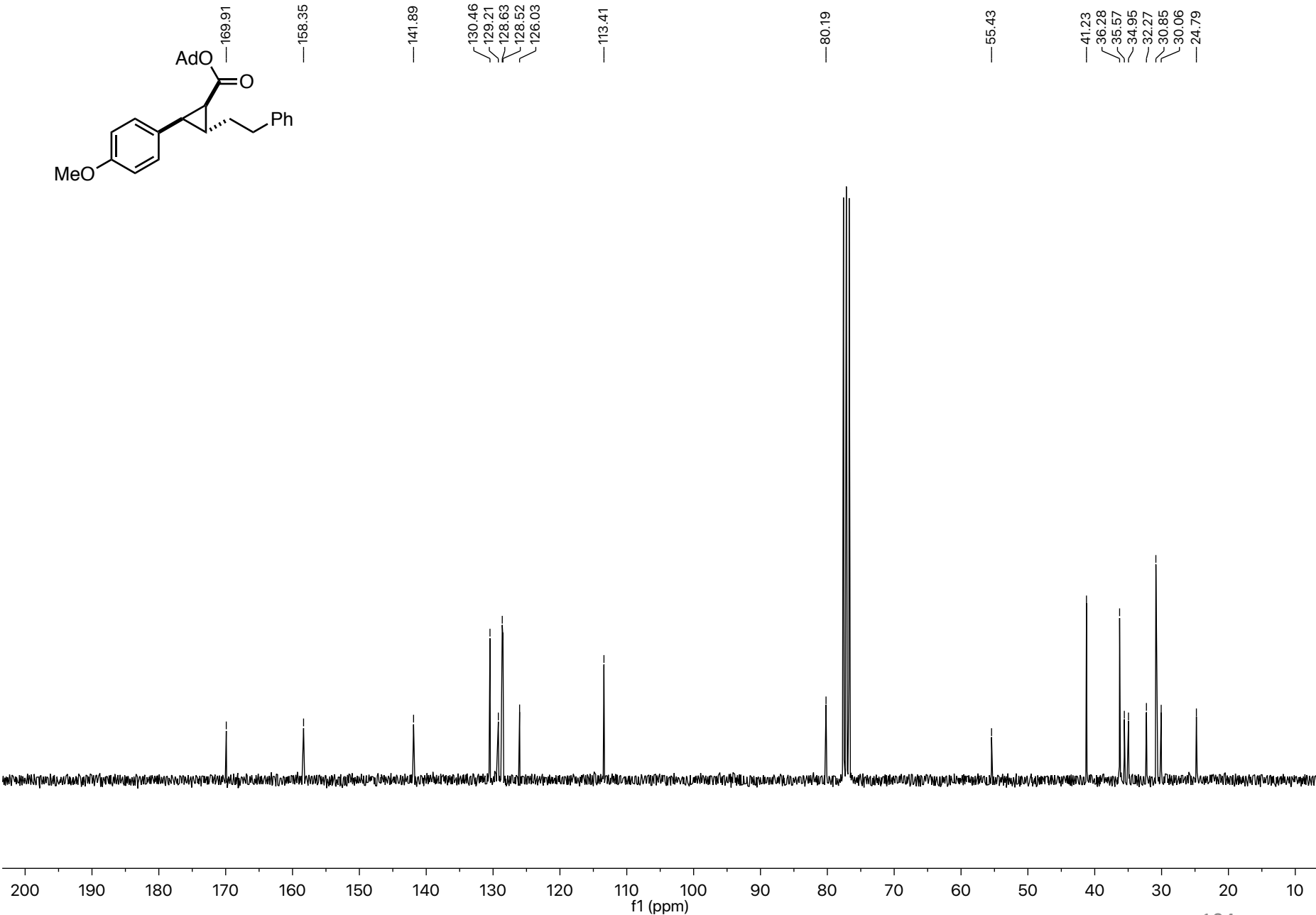
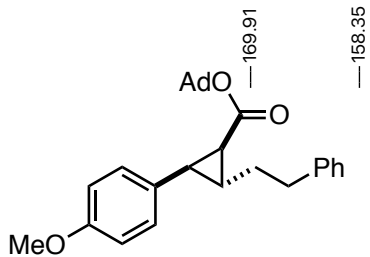
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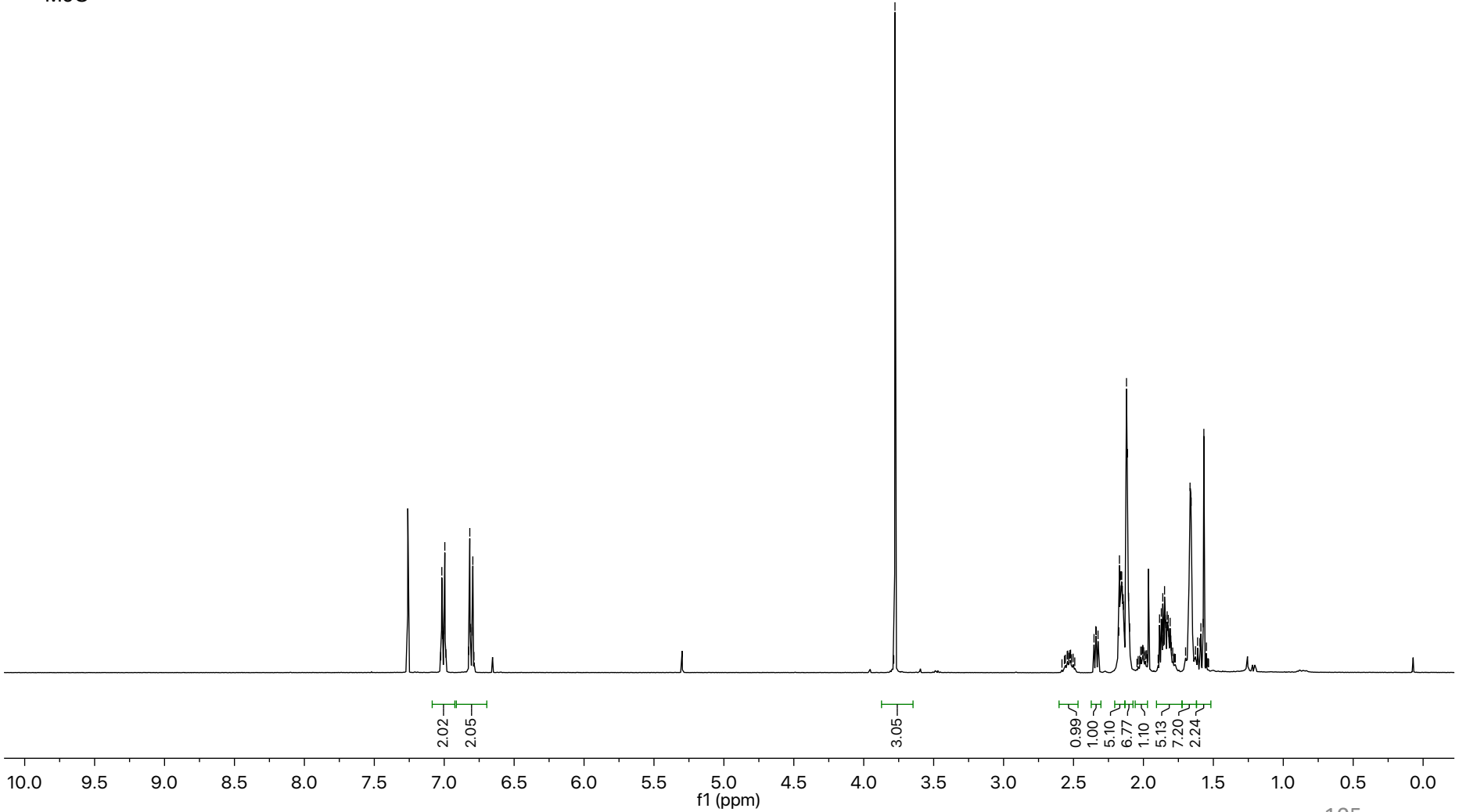
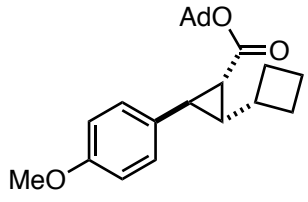


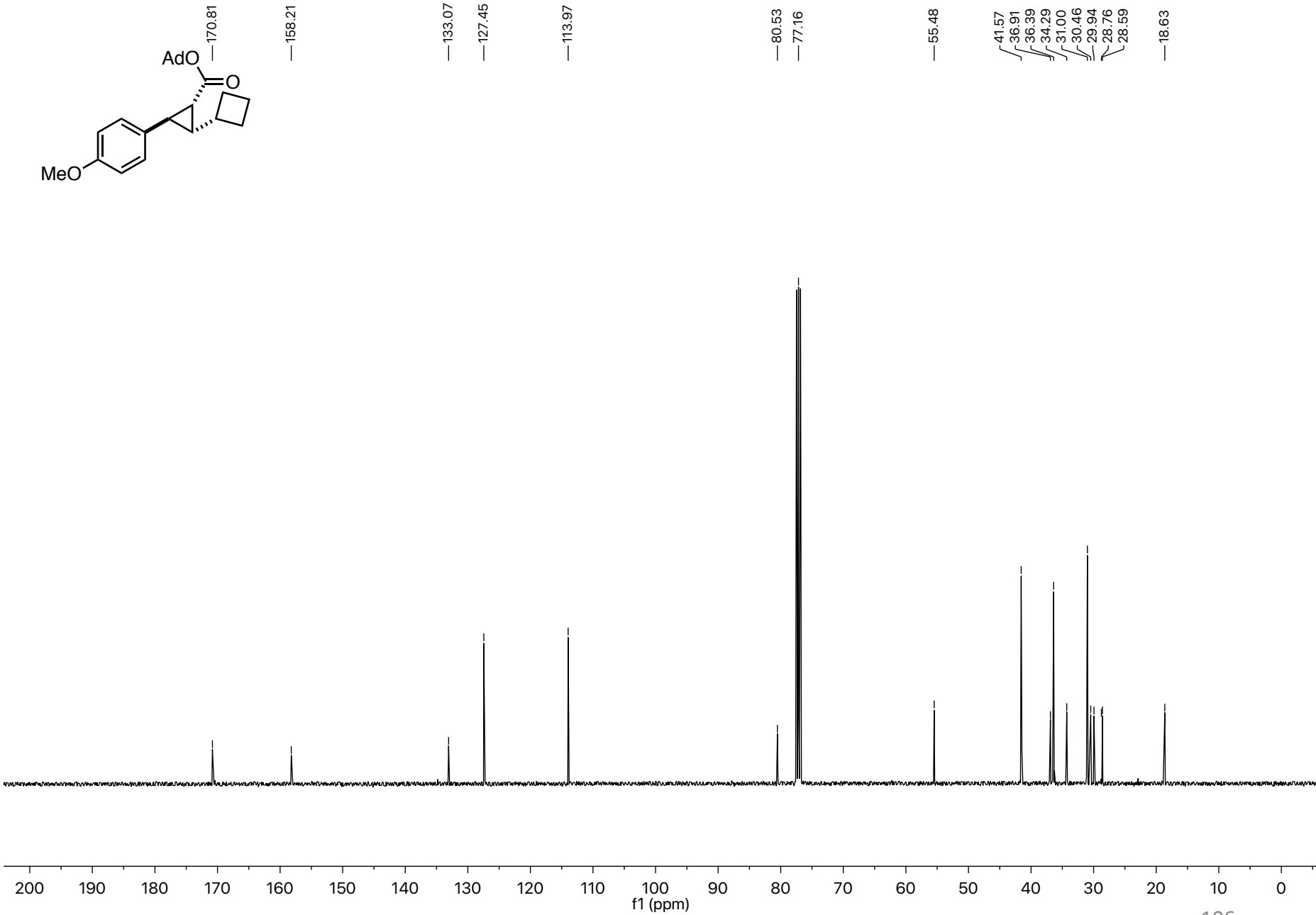
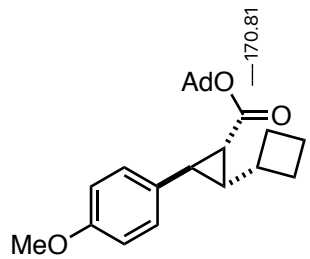


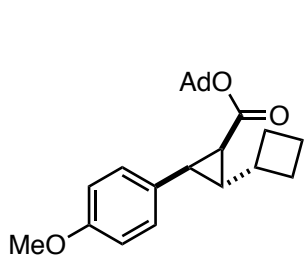




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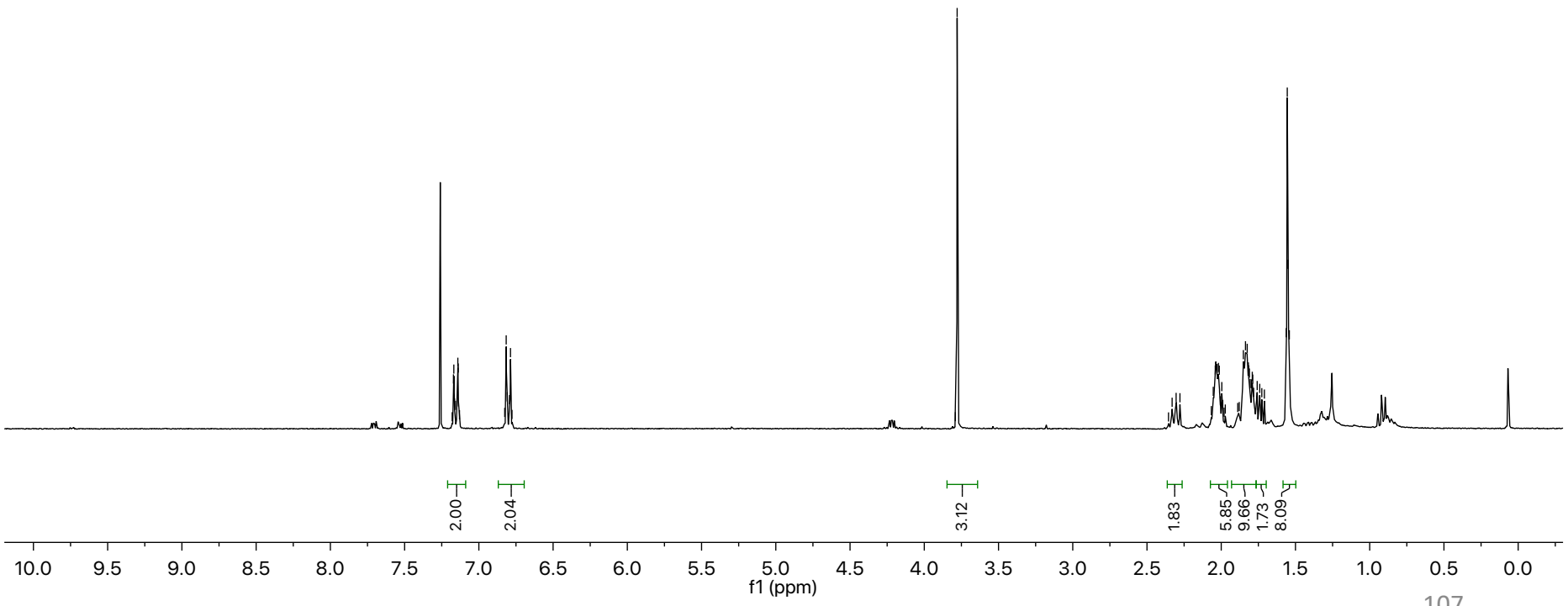


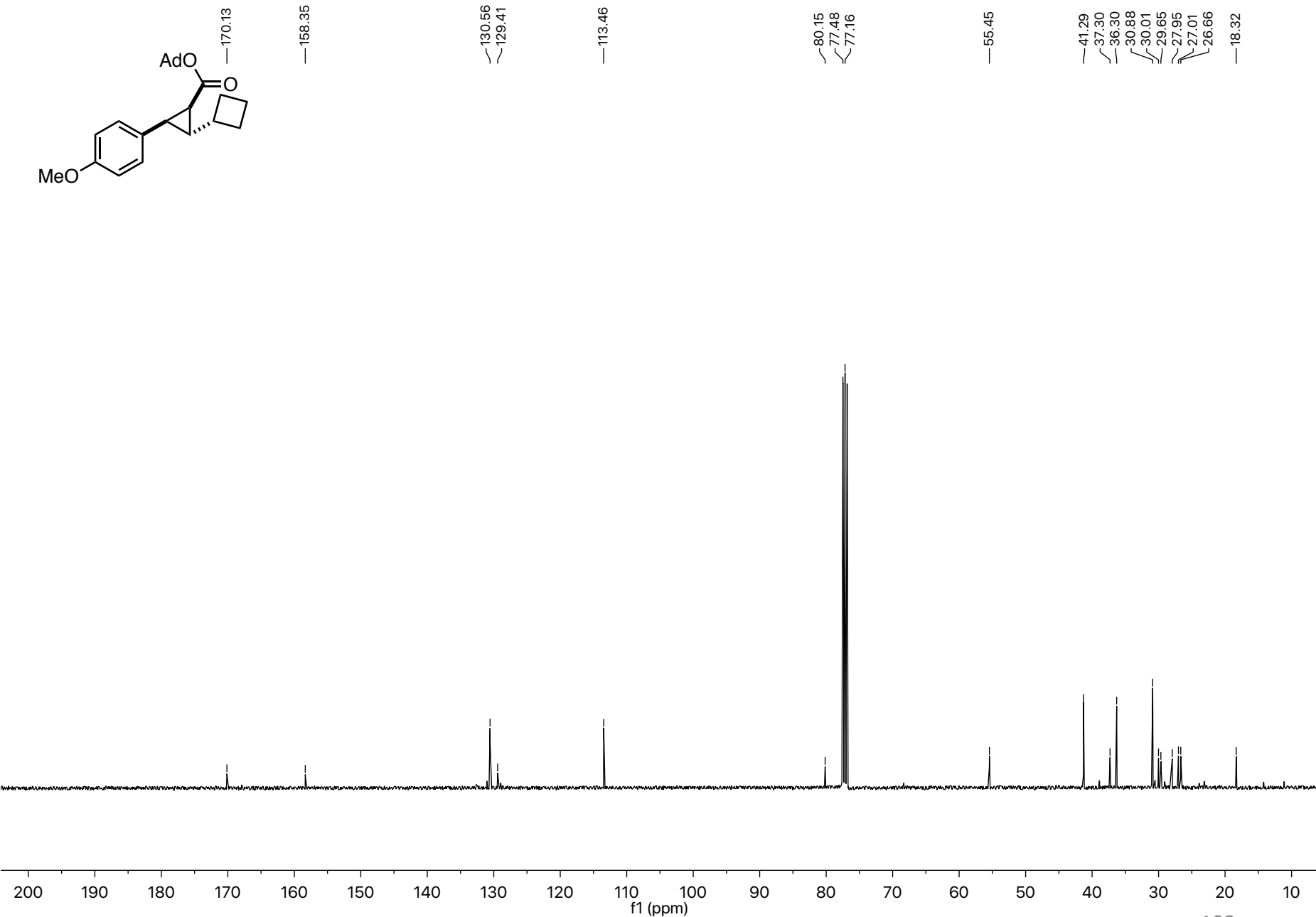
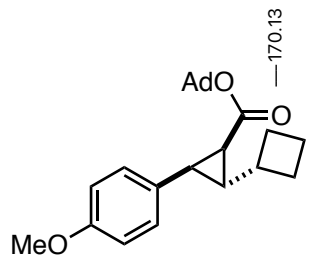


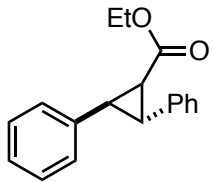


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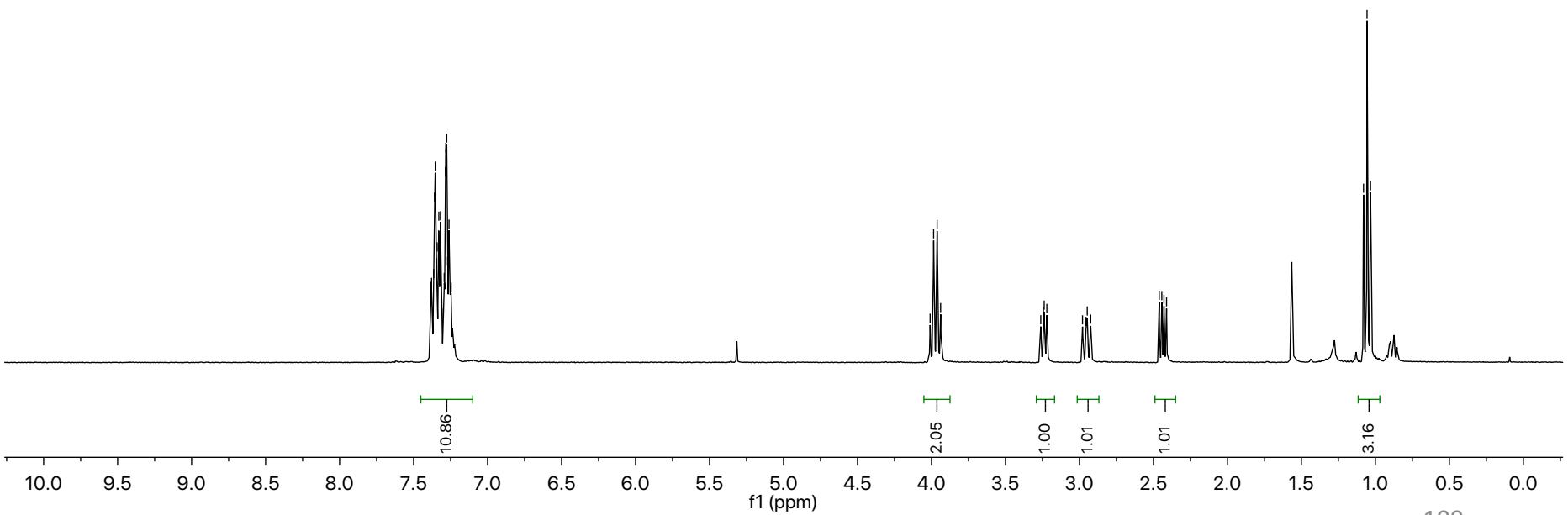


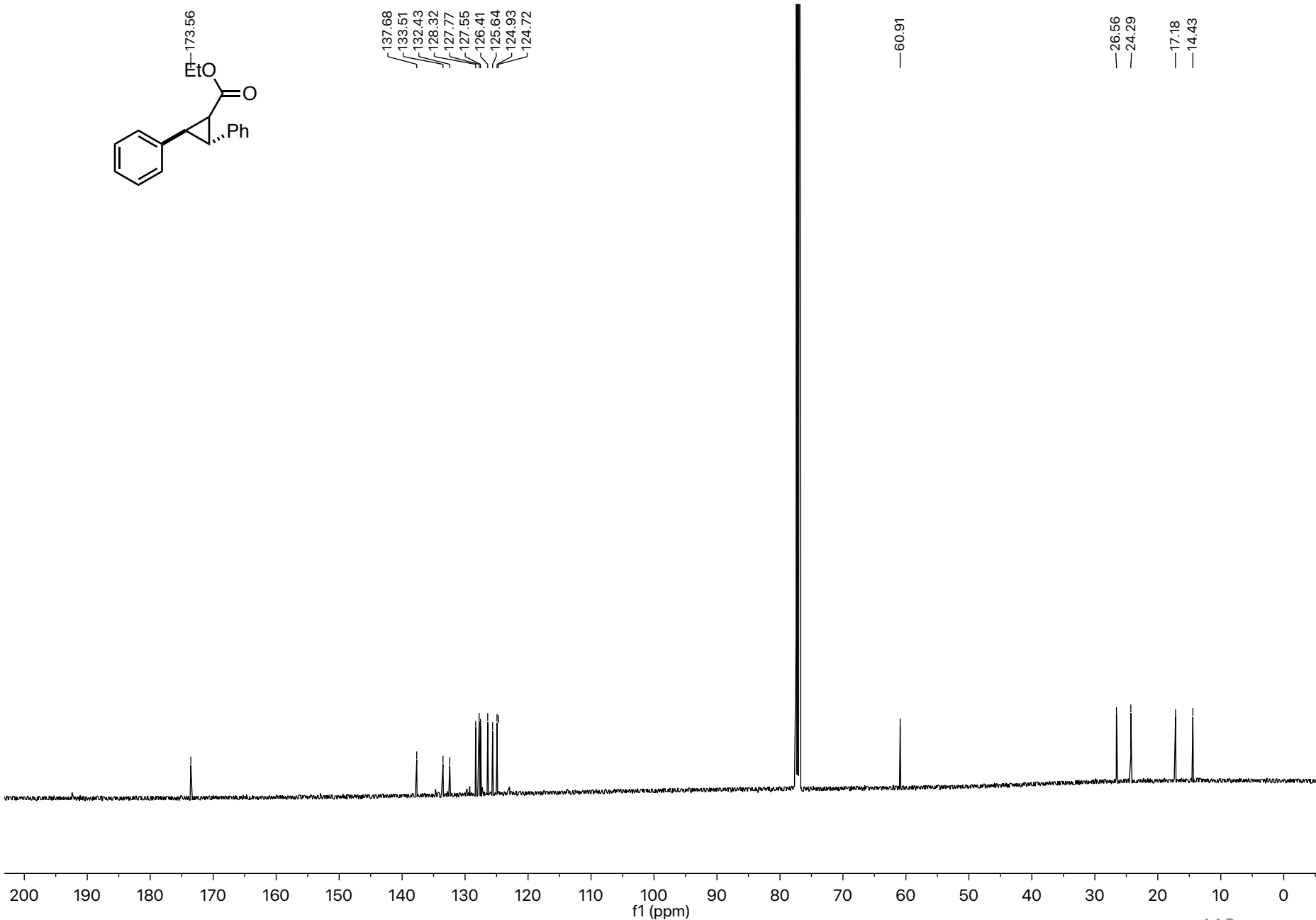
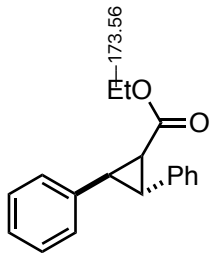


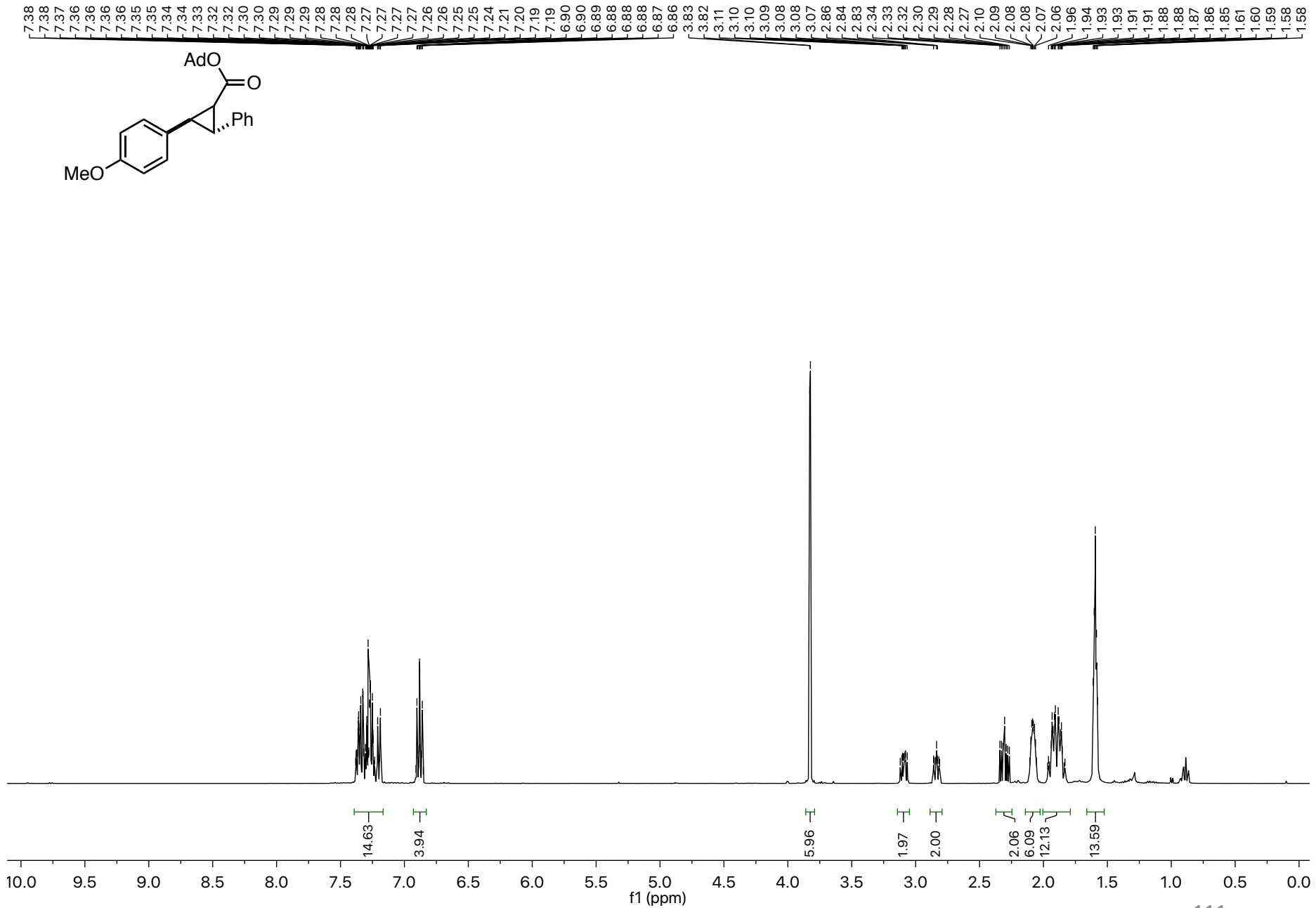
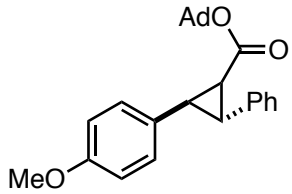
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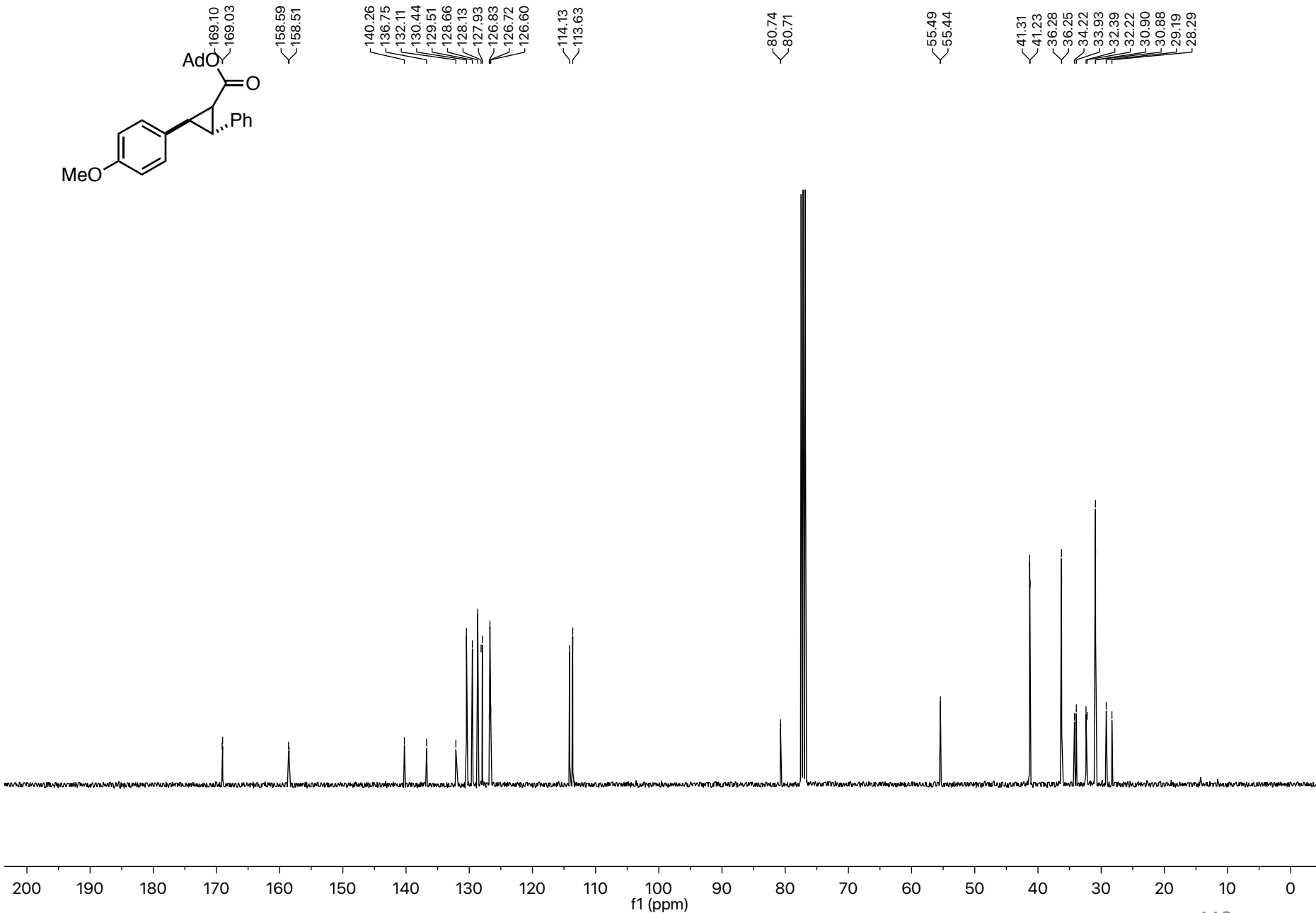
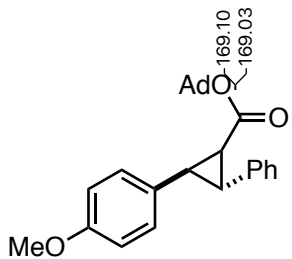
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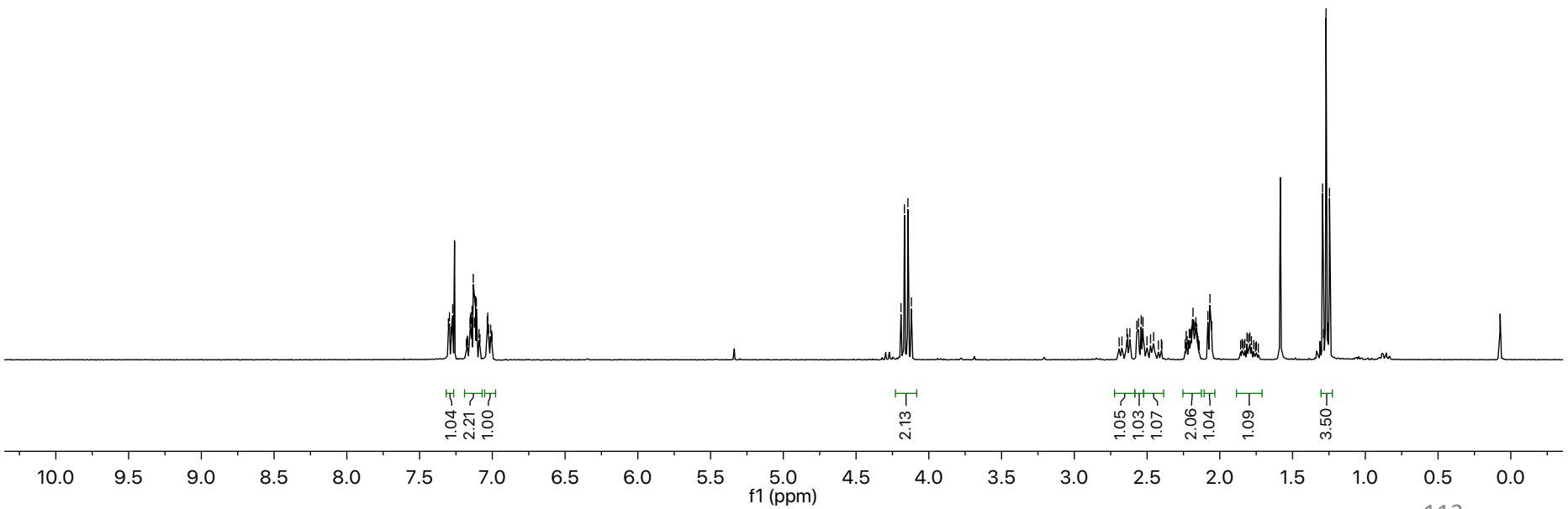
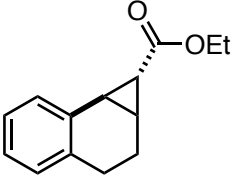


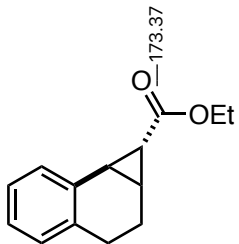






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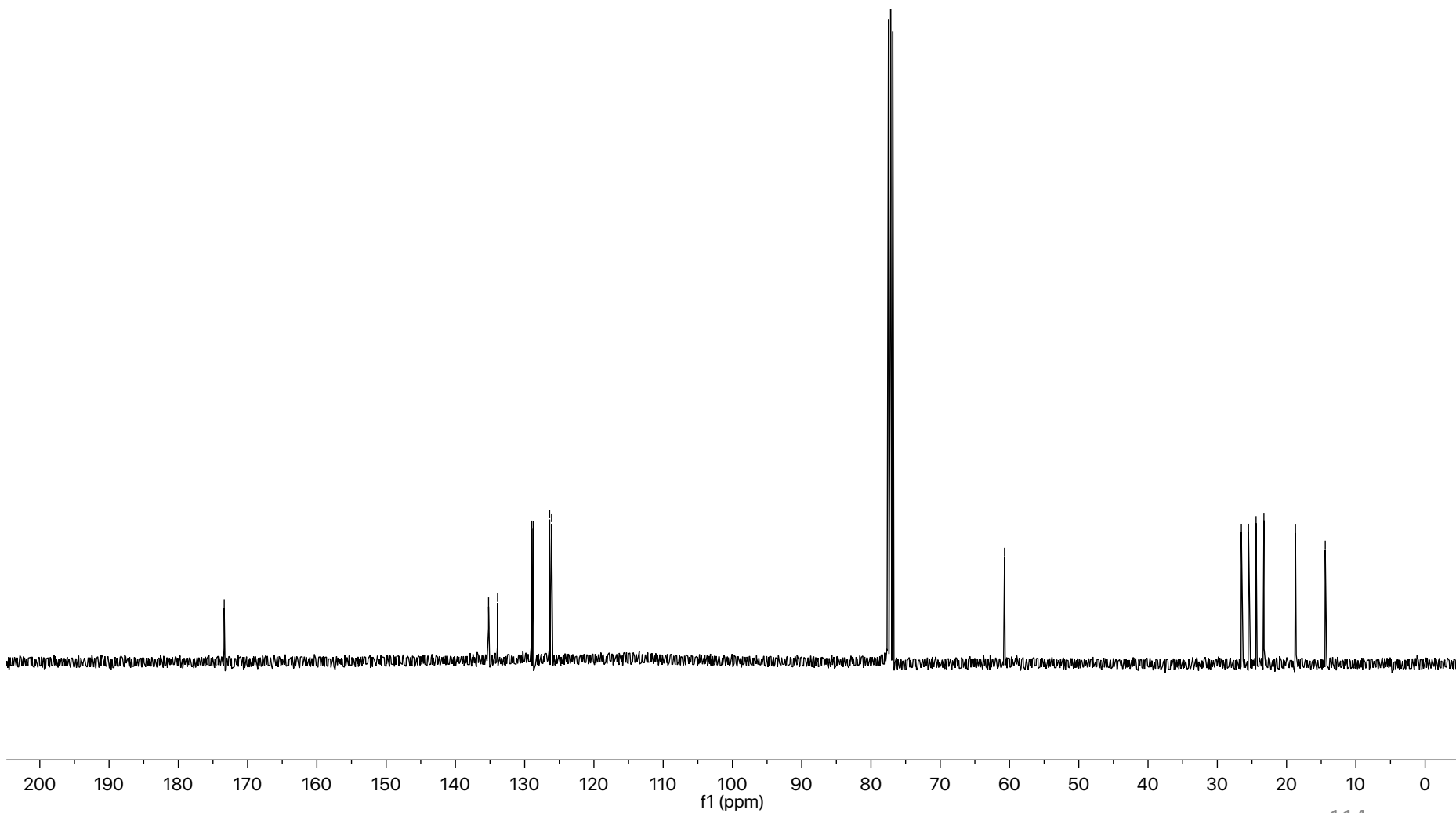


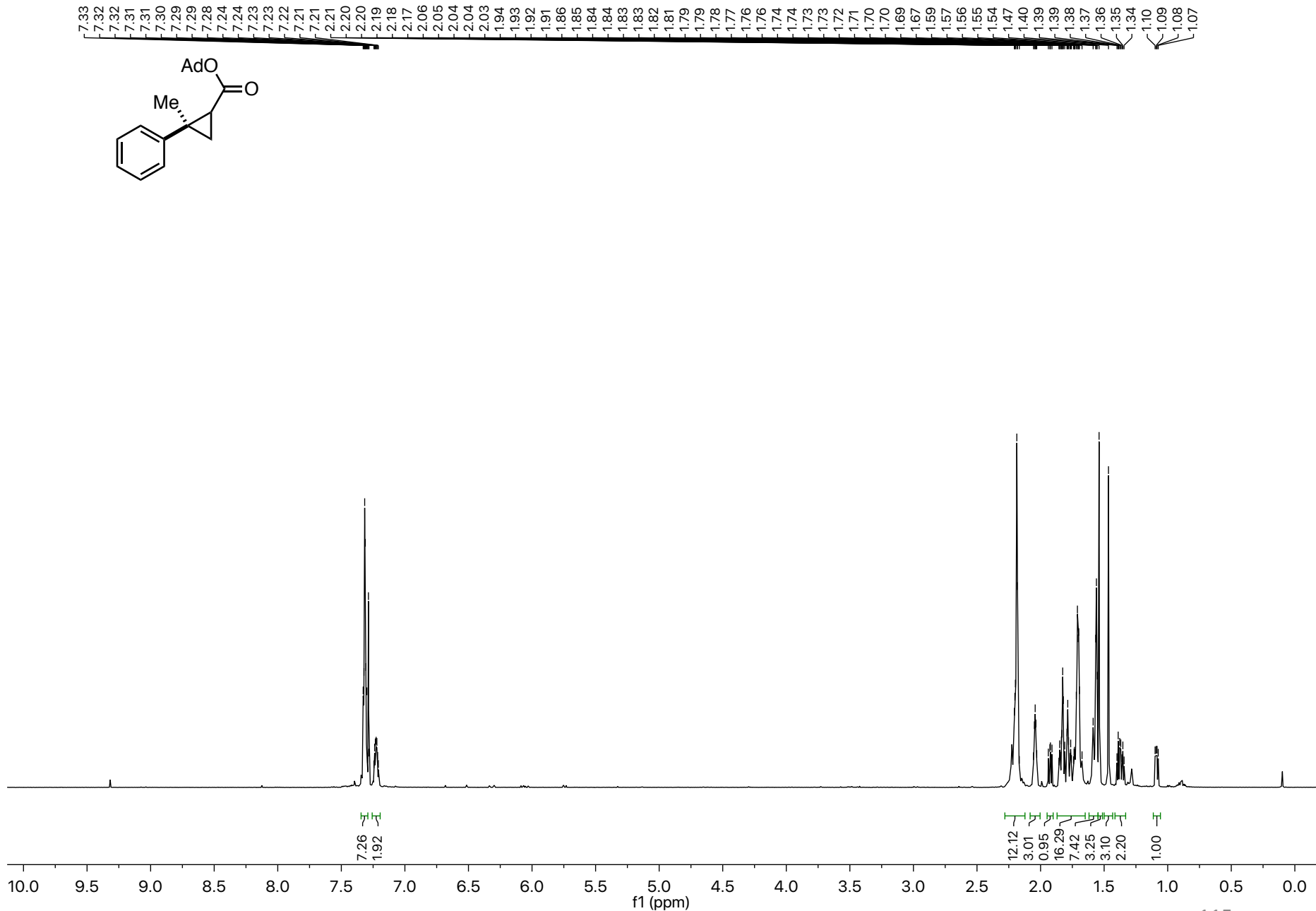
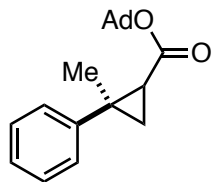


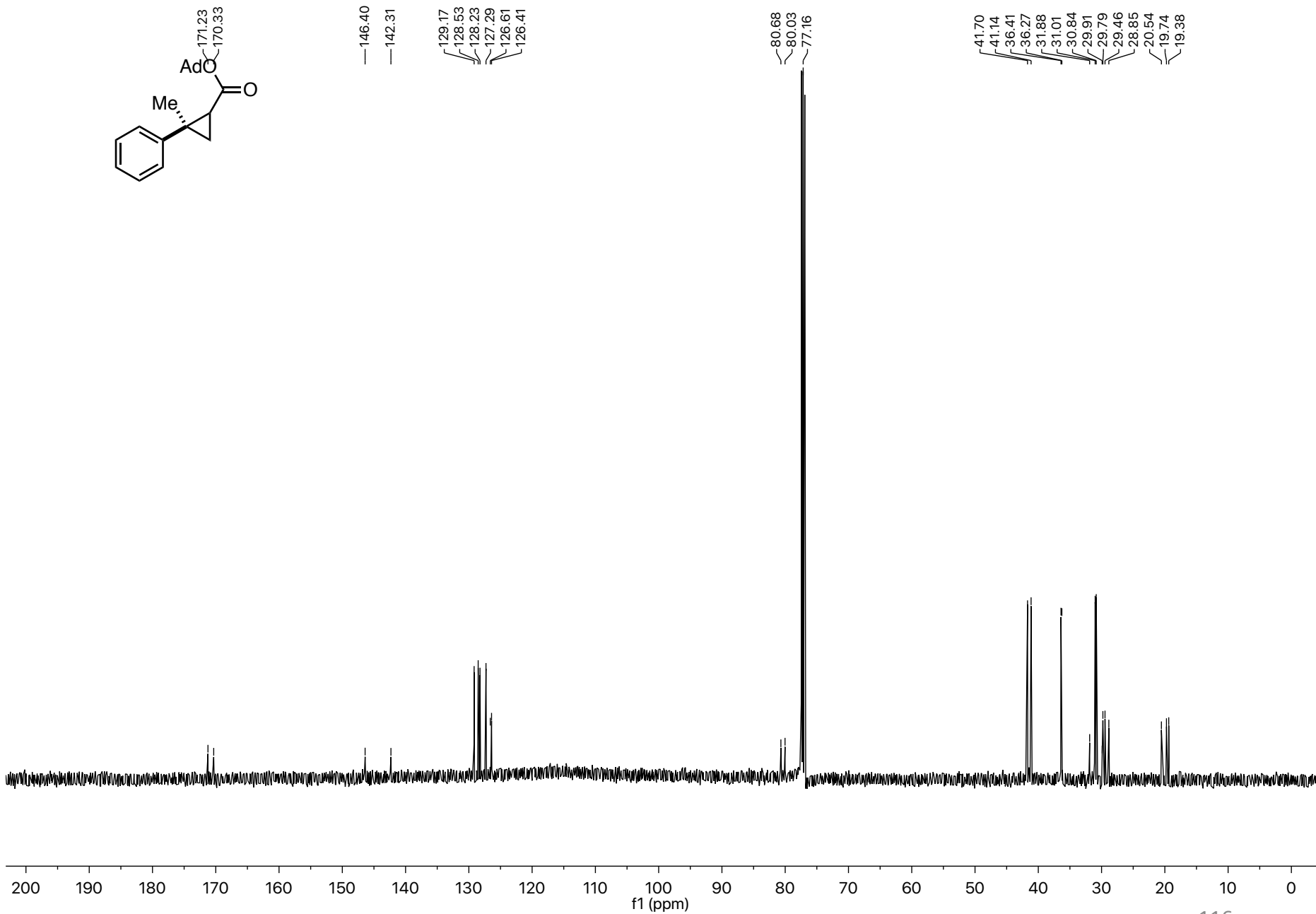
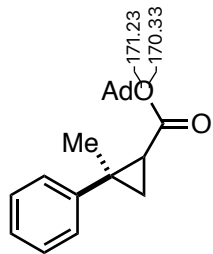
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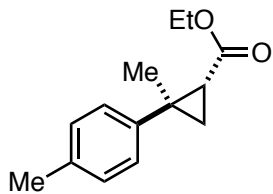
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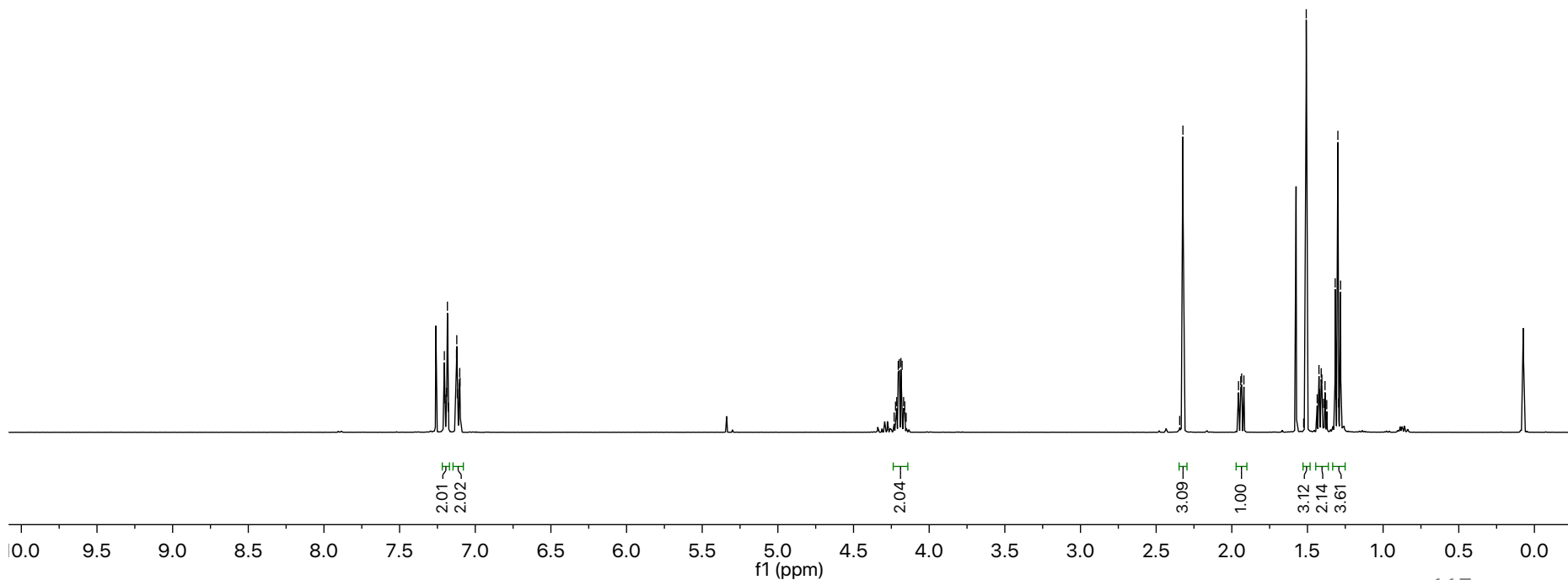


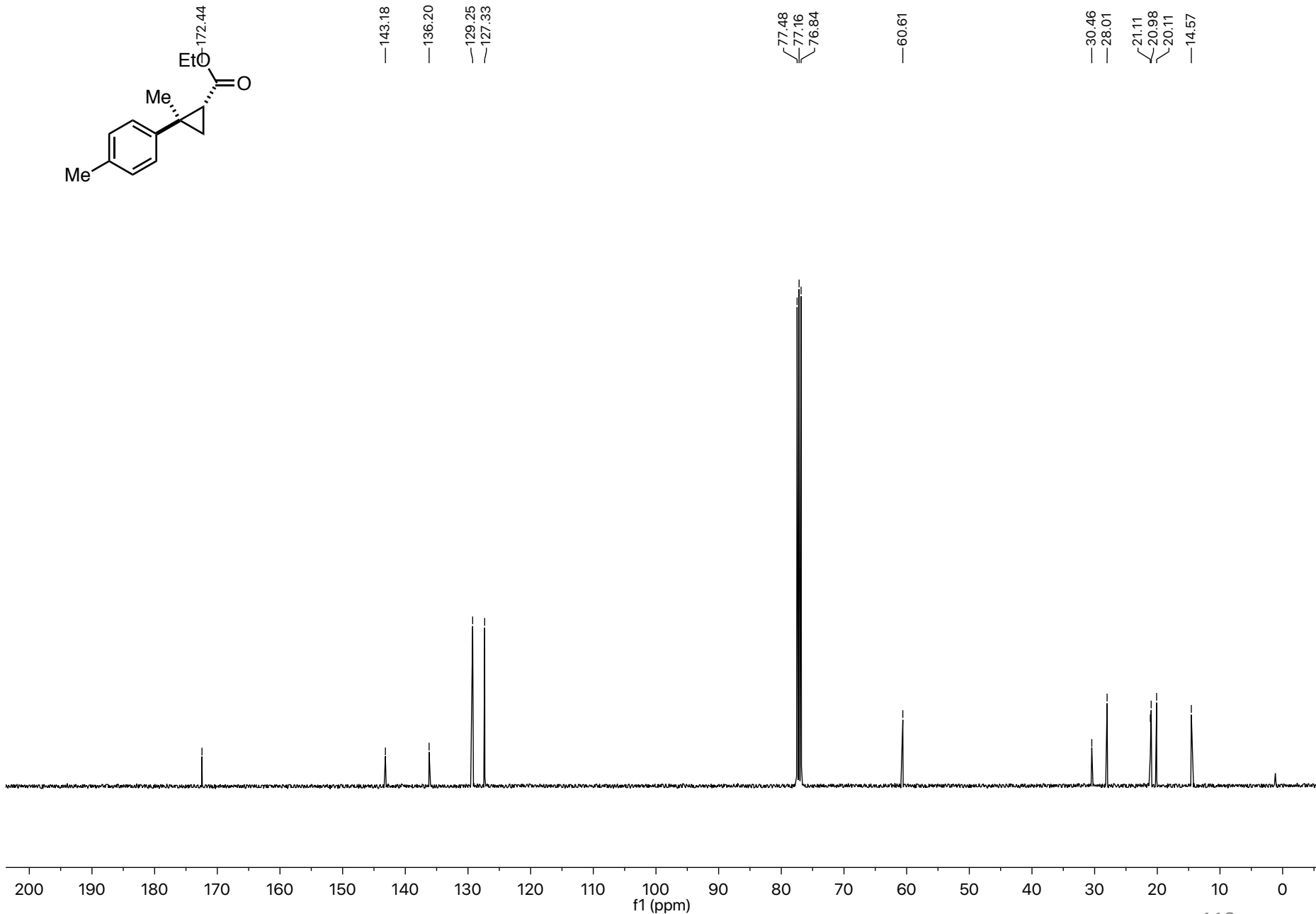
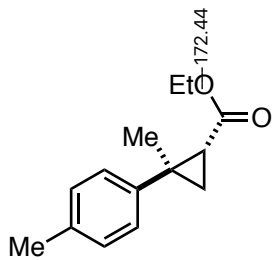


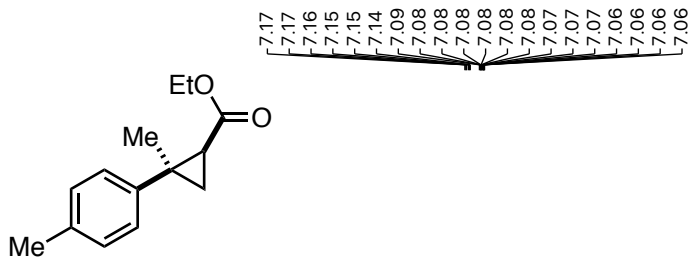
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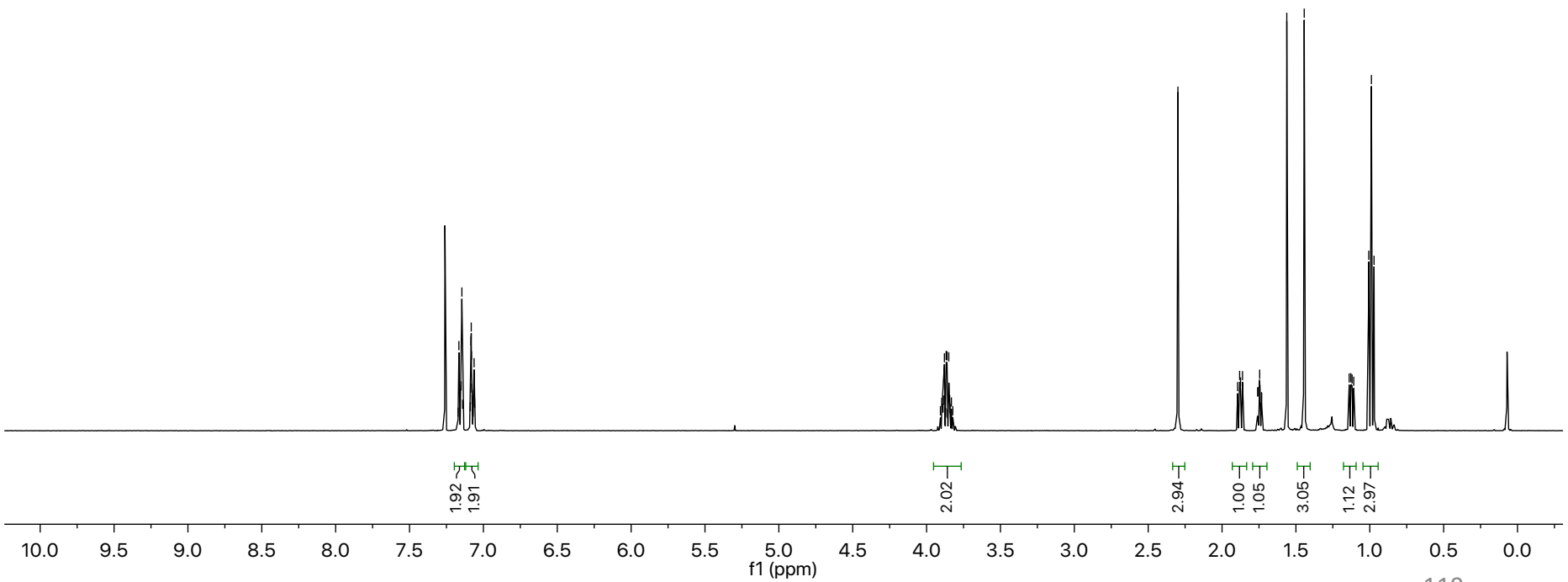


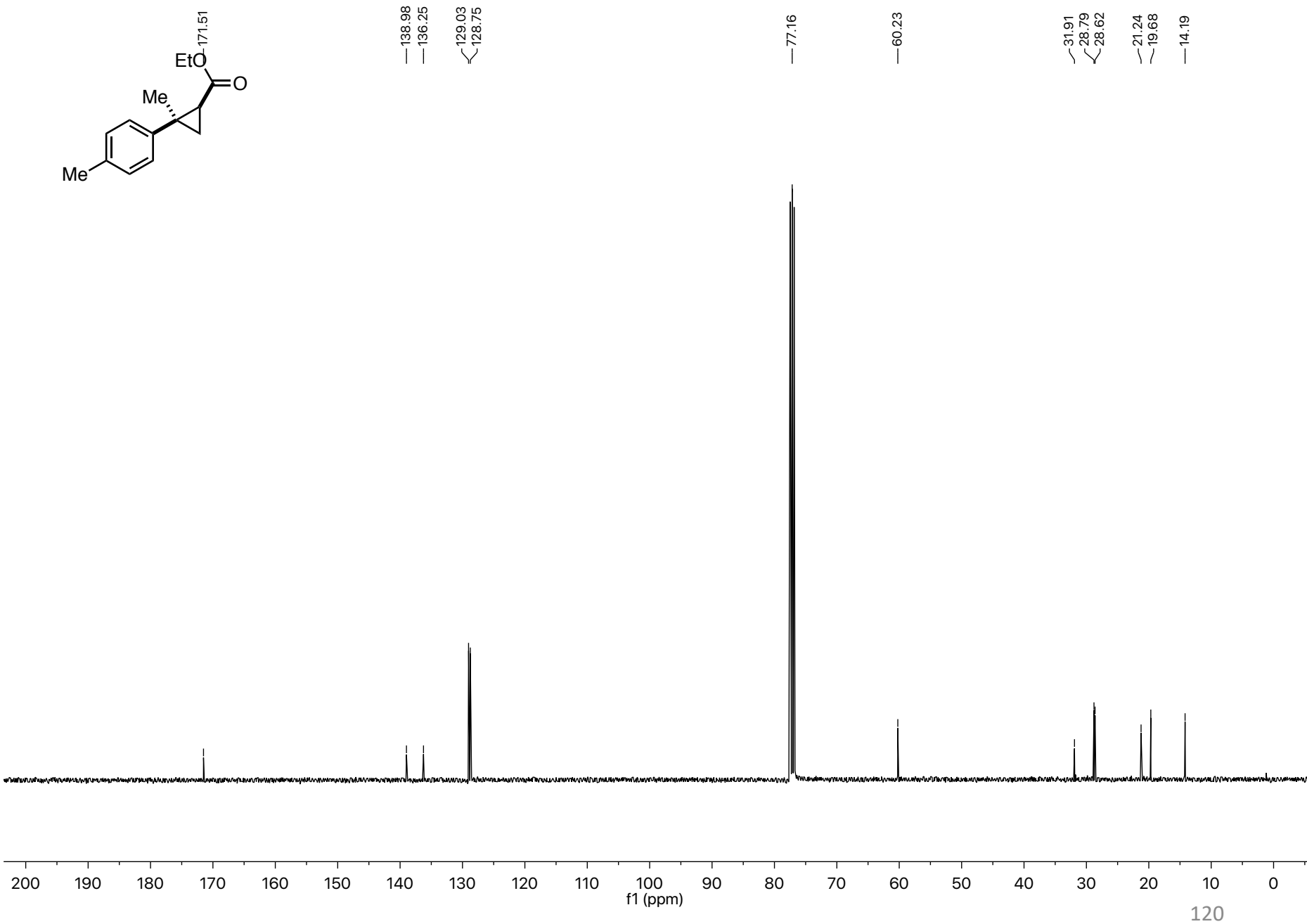
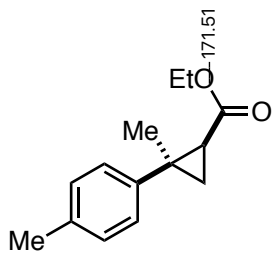


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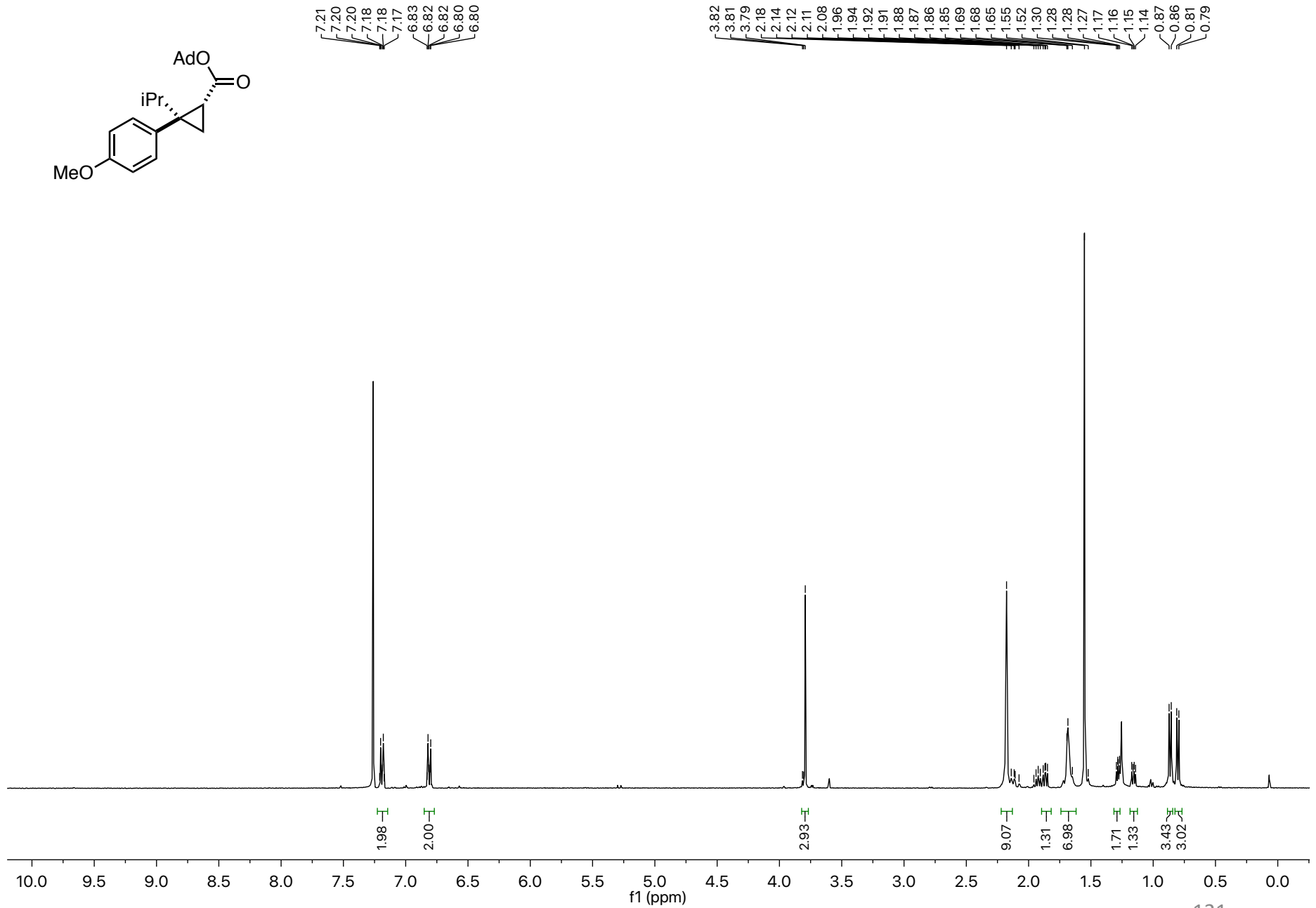
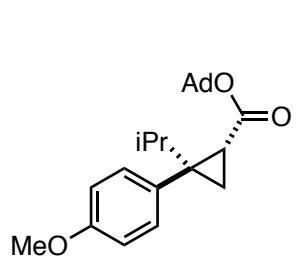
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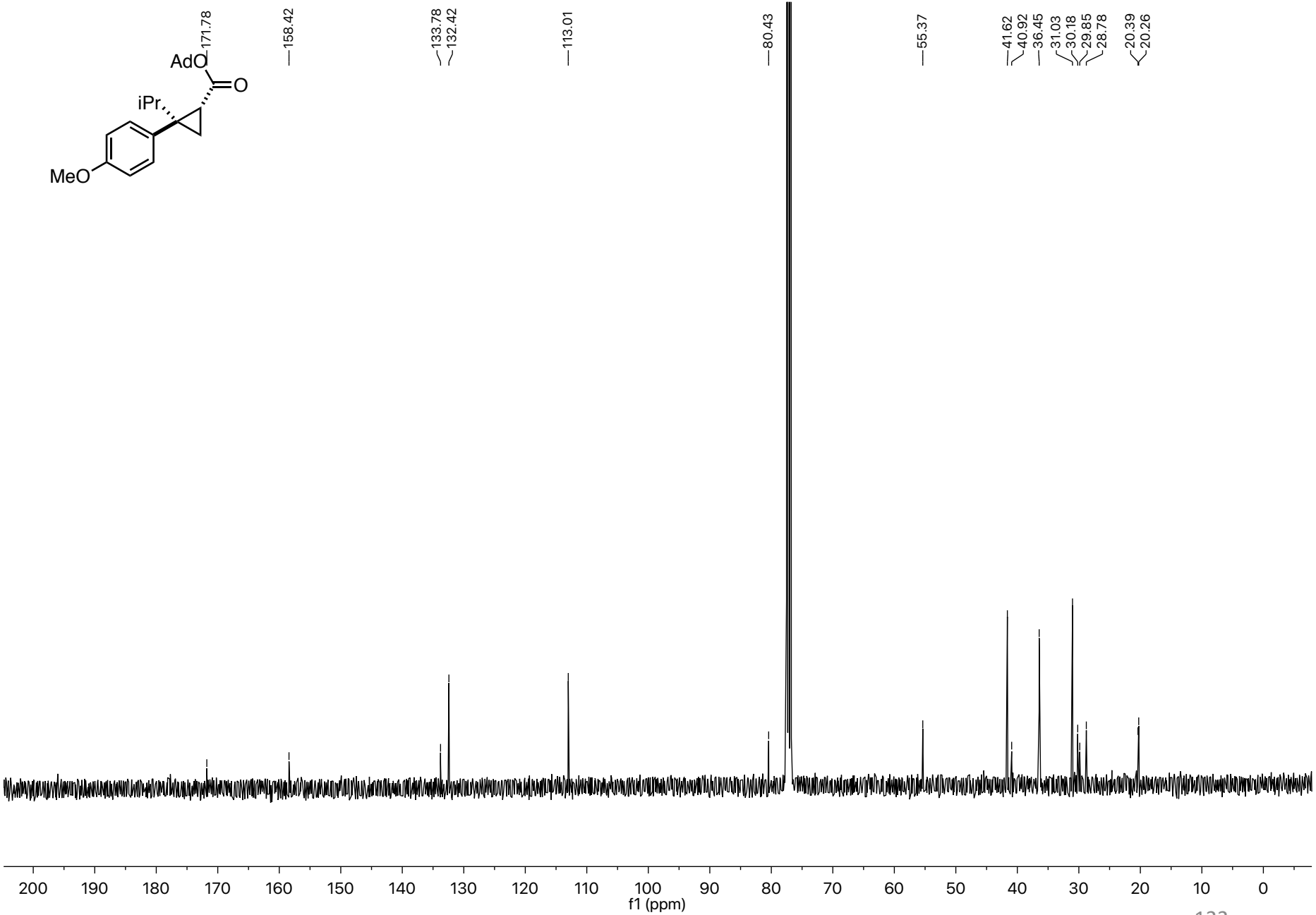
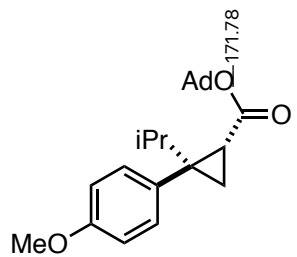
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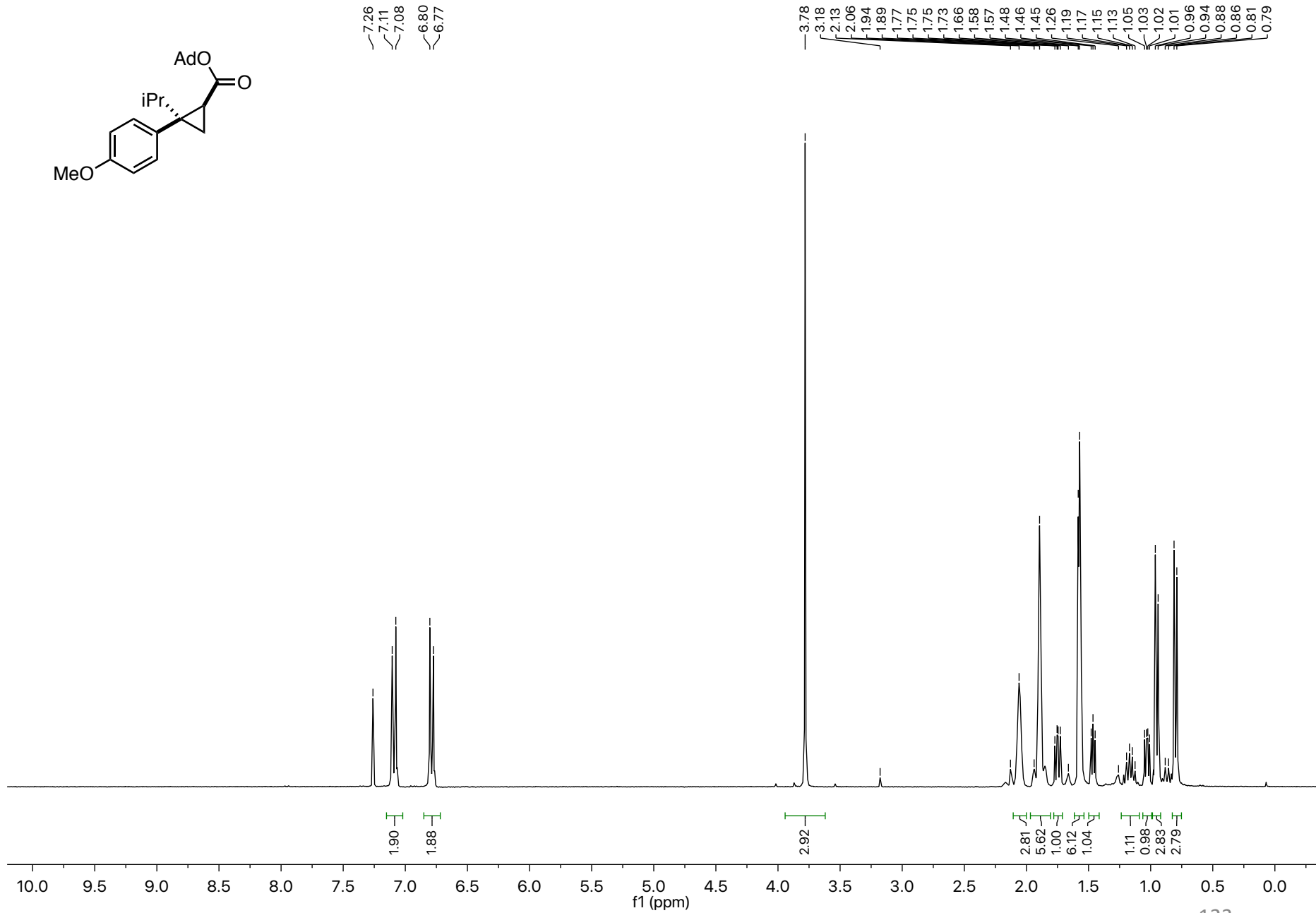
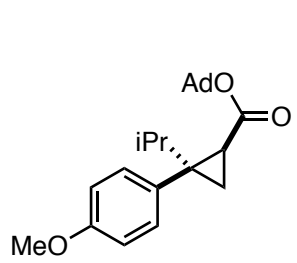


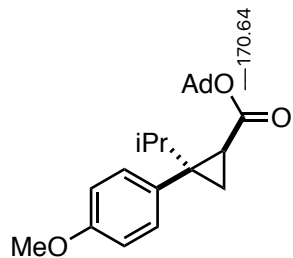












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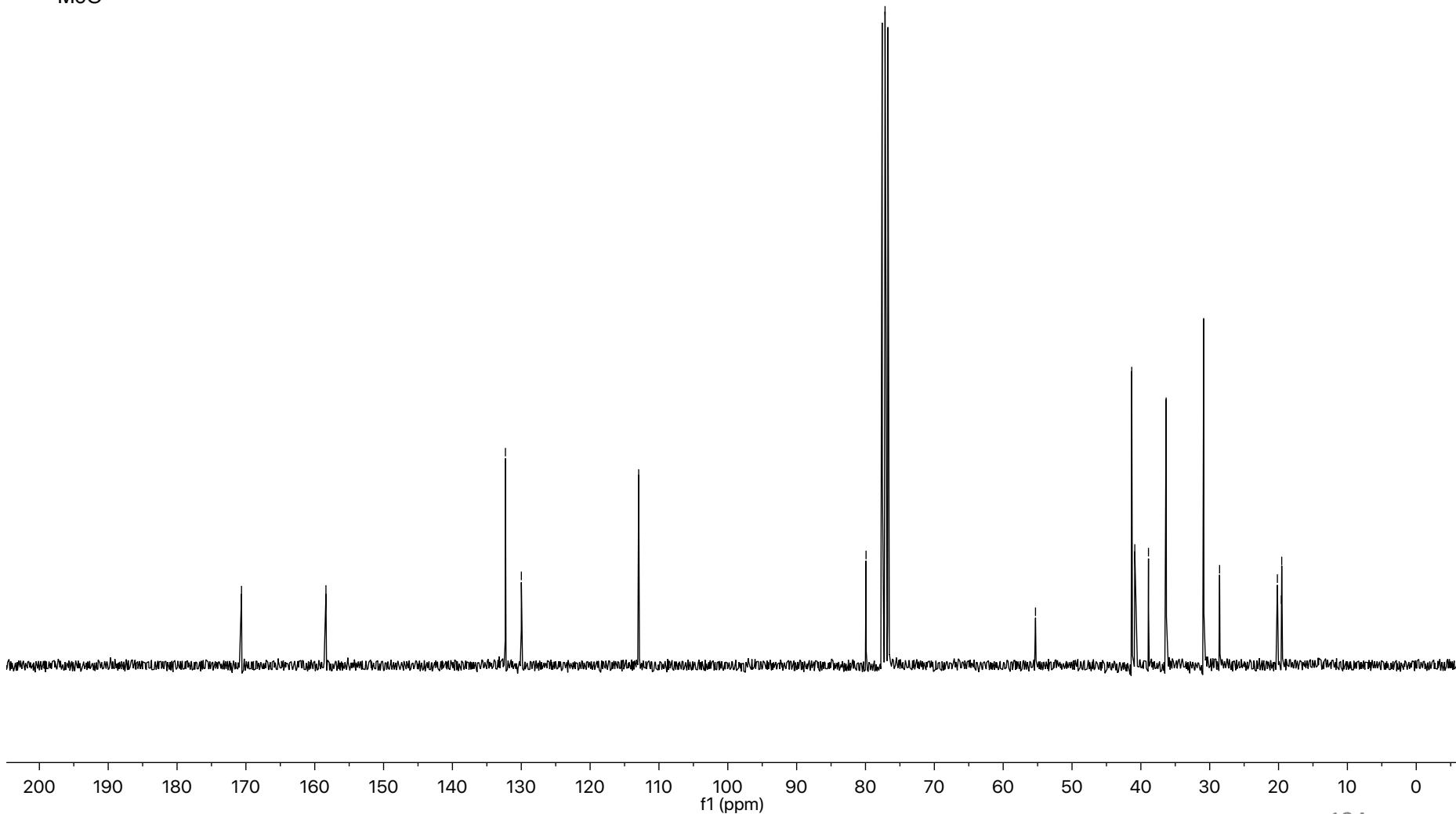
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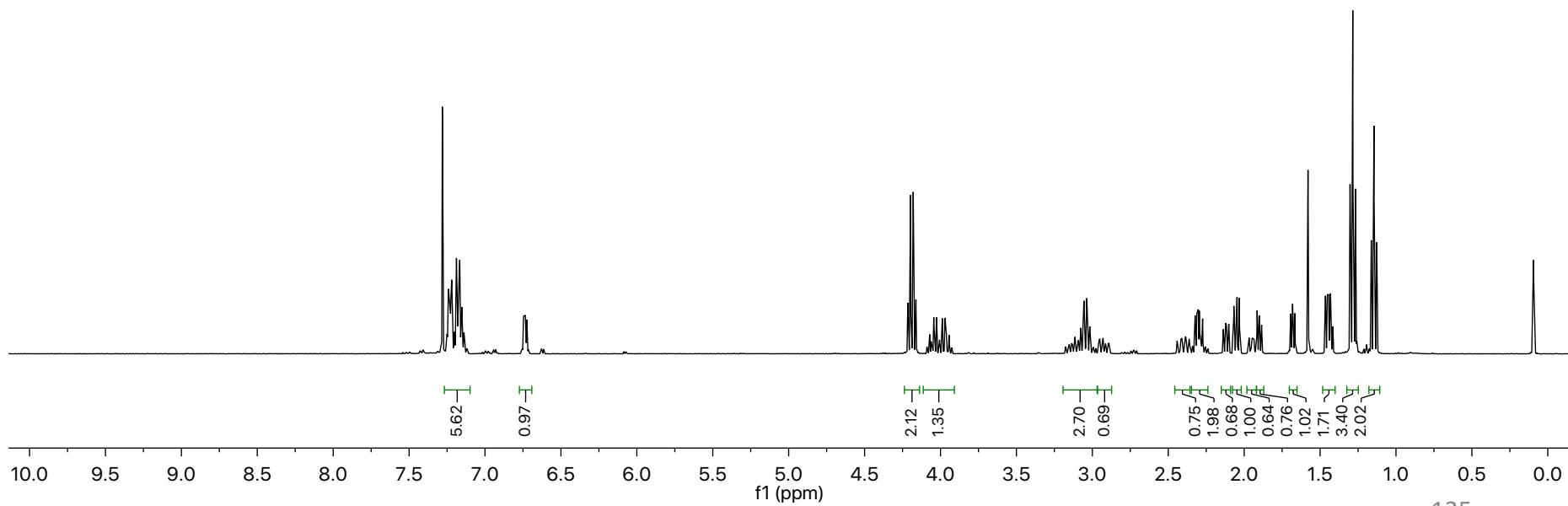
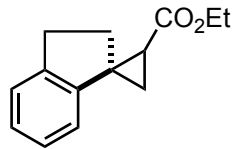
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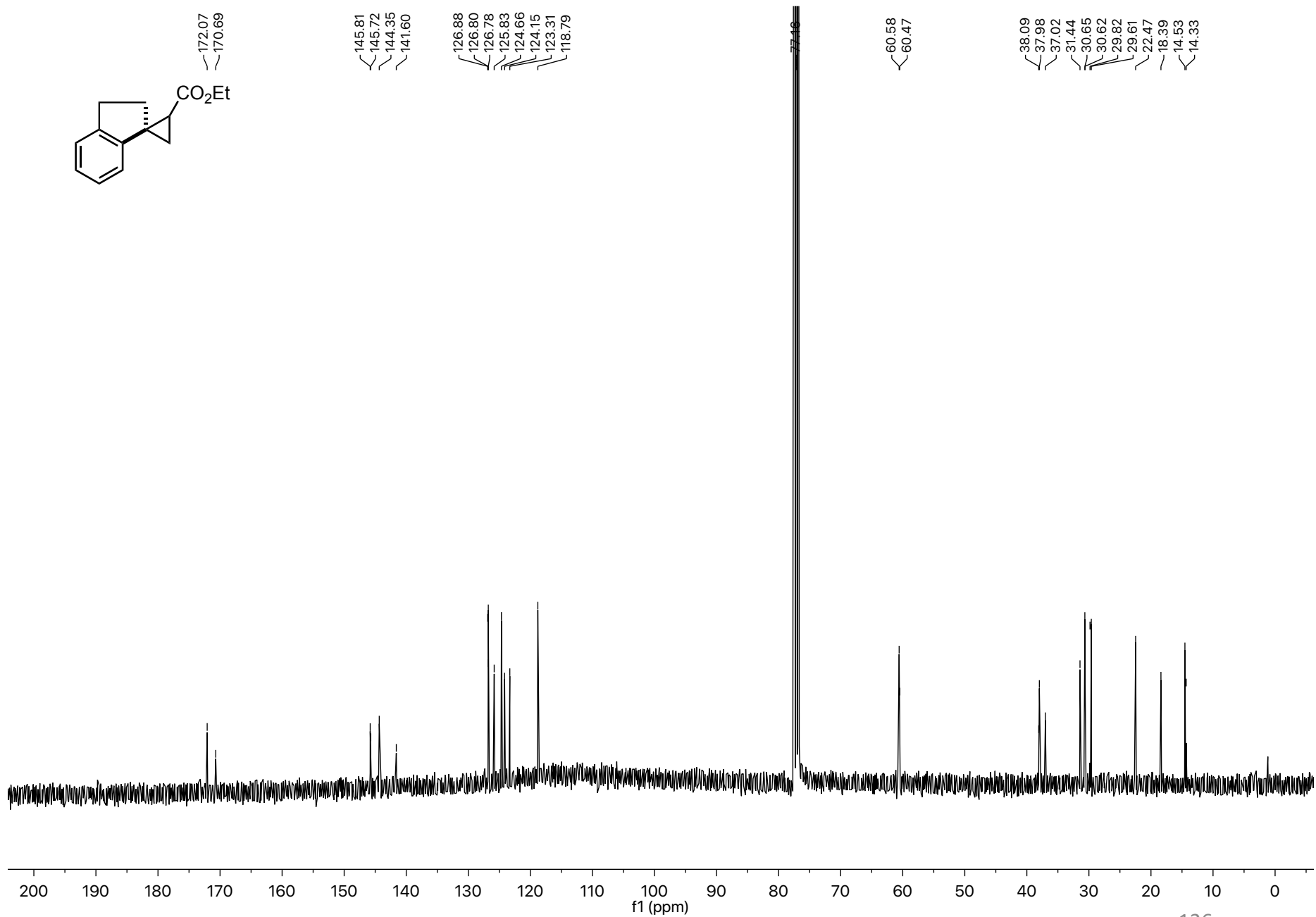
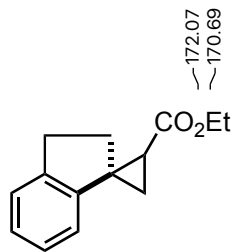
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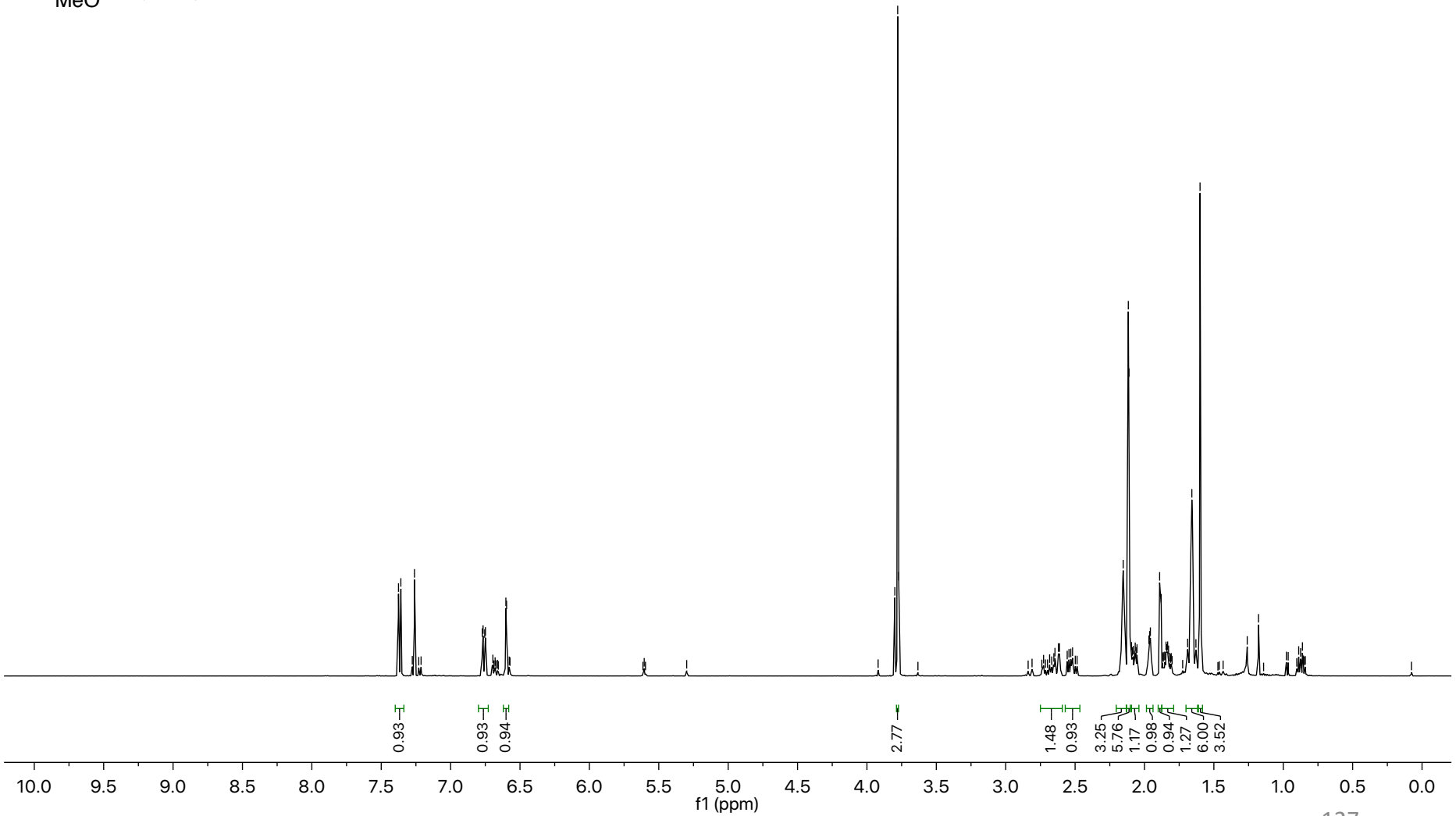
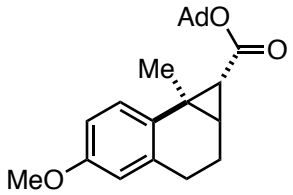
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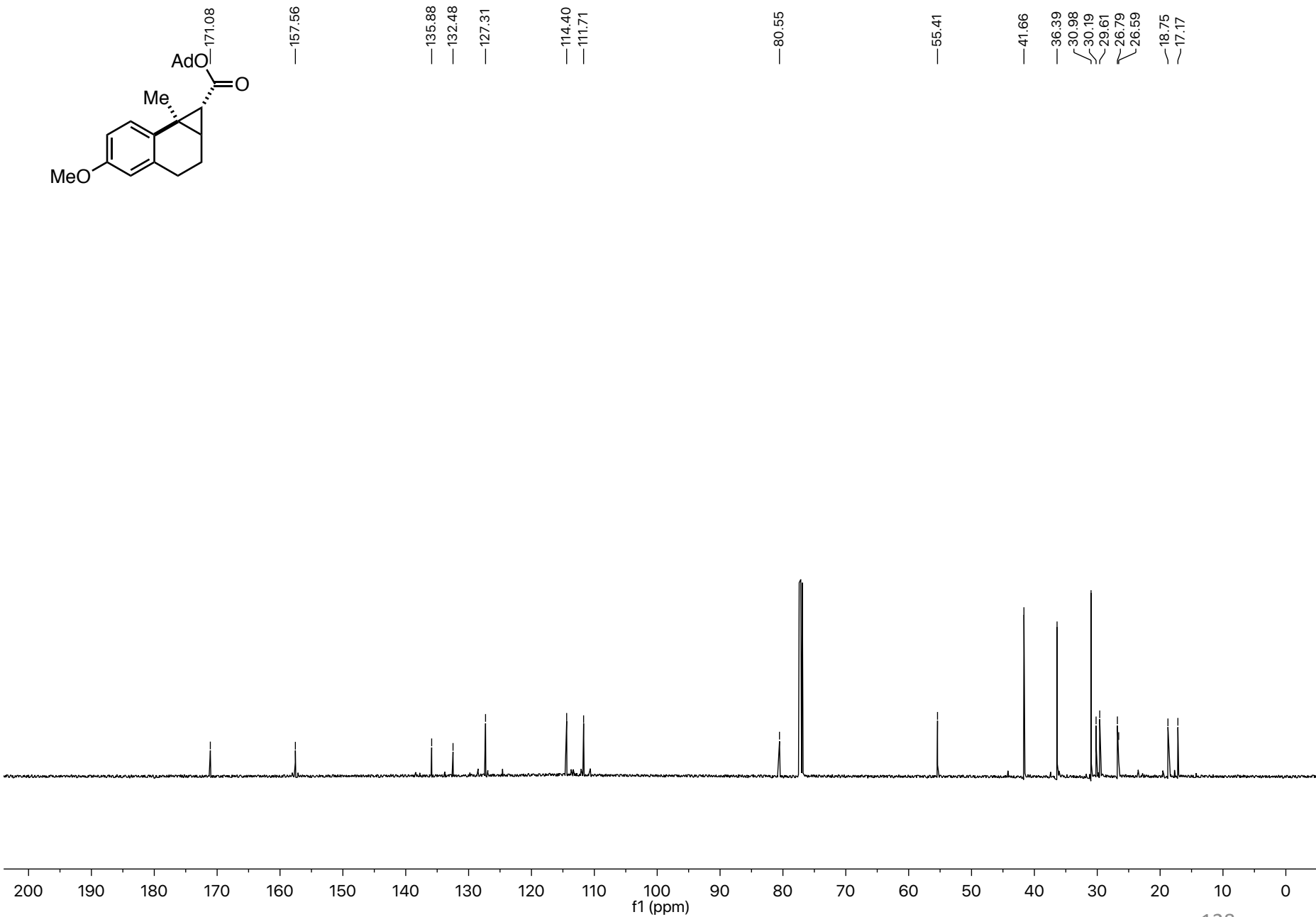
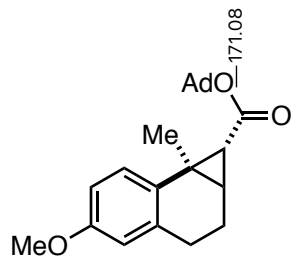




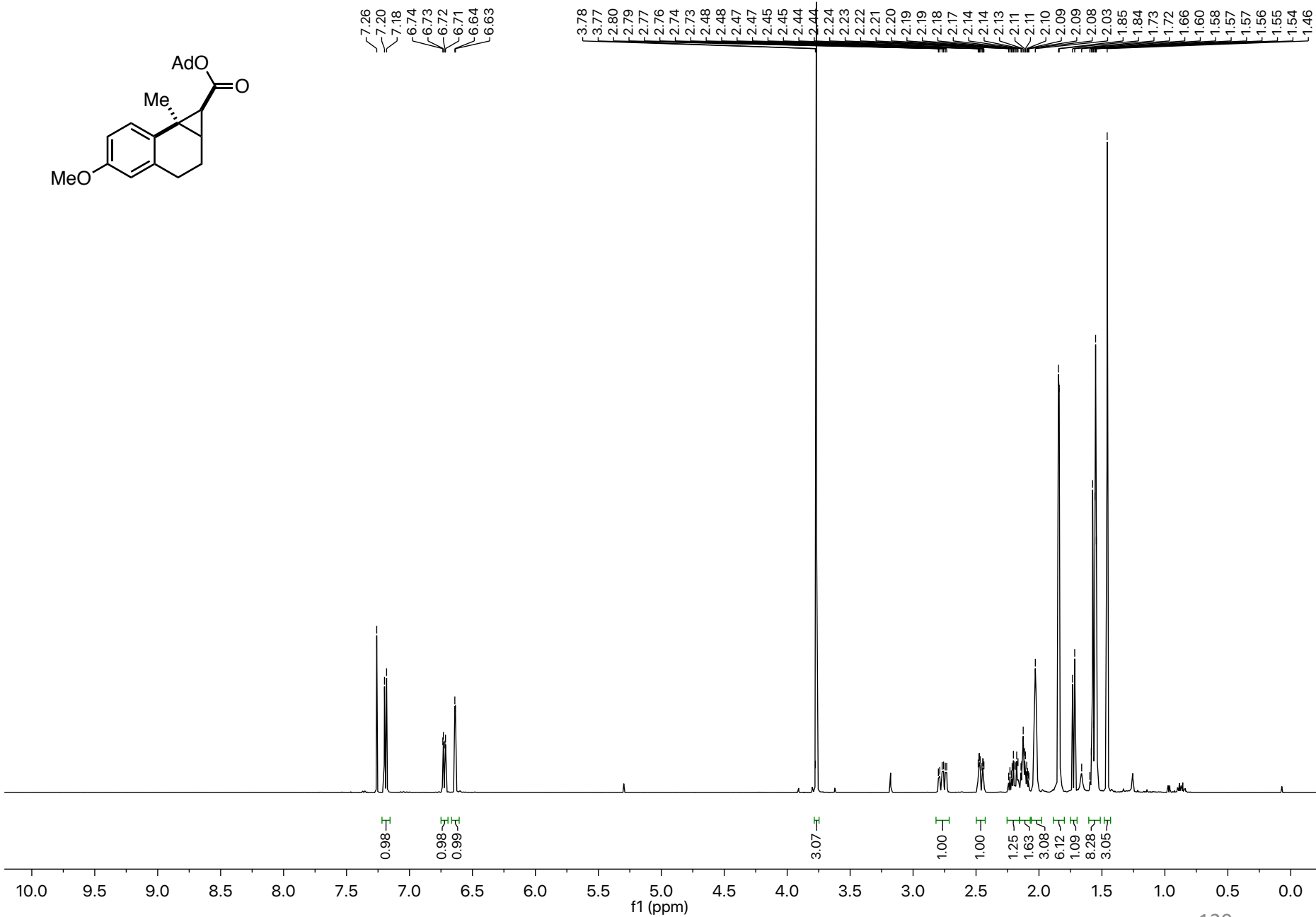
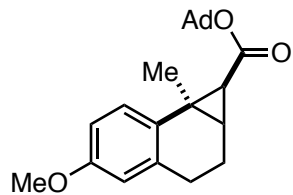


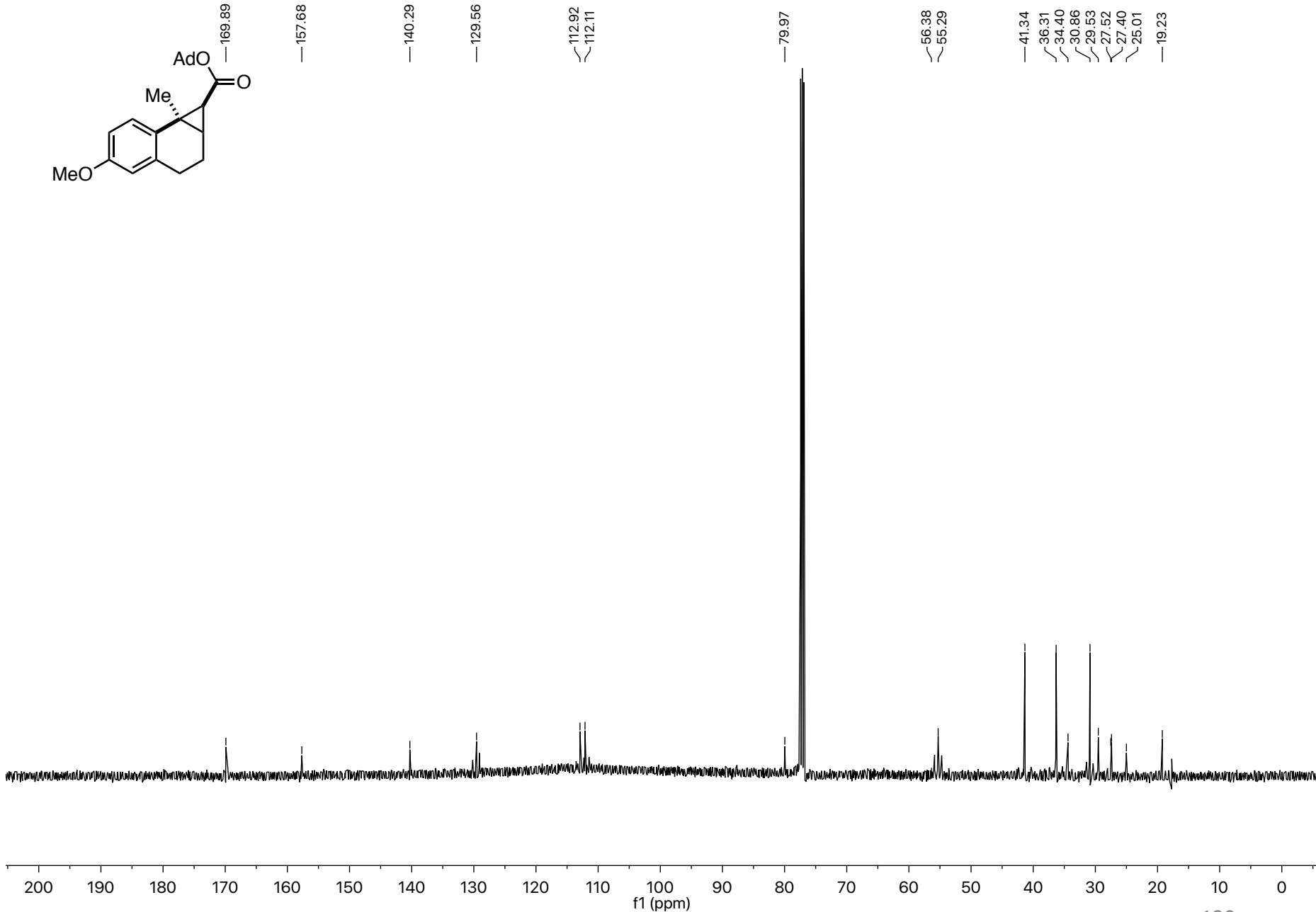
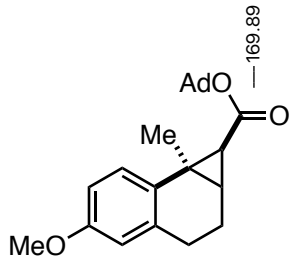
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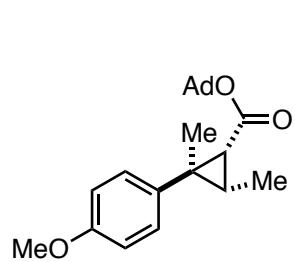








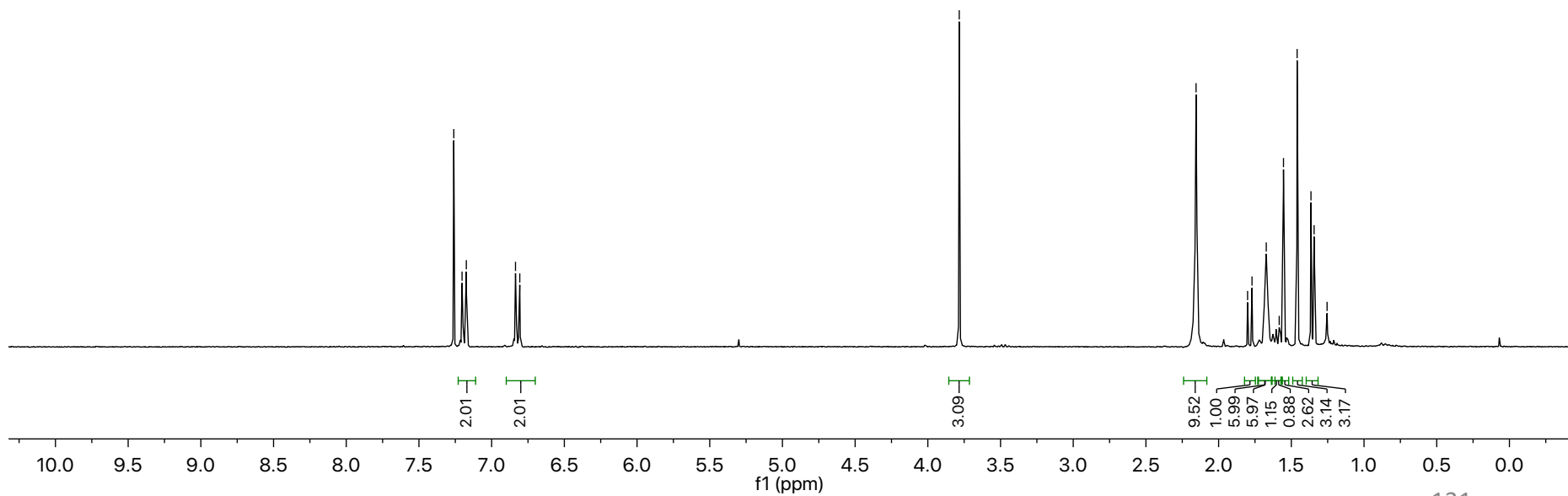


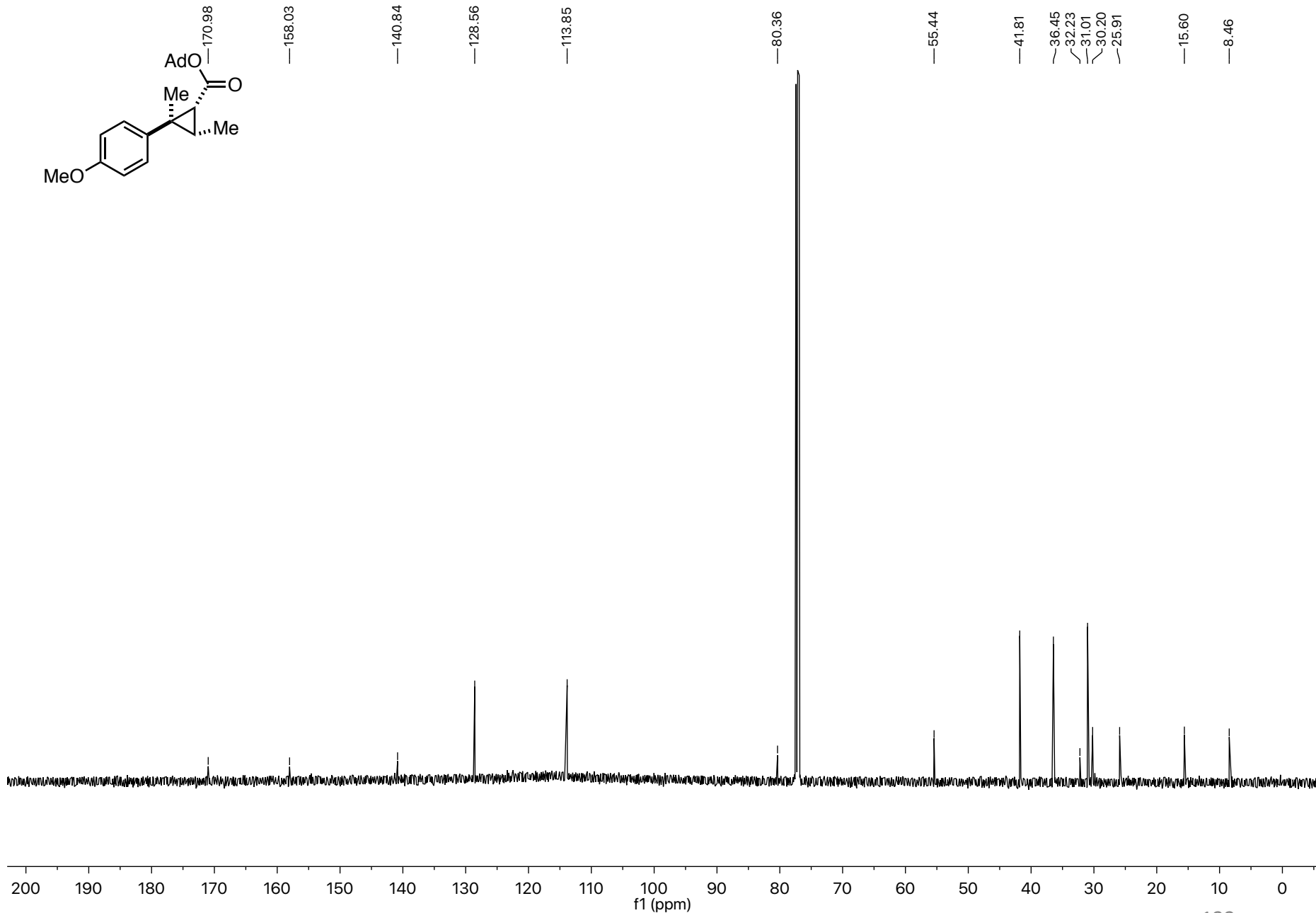
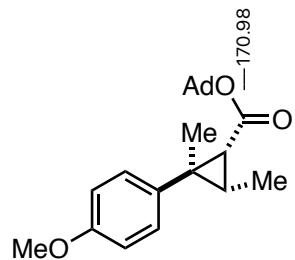


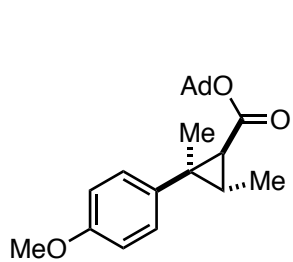
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6.81

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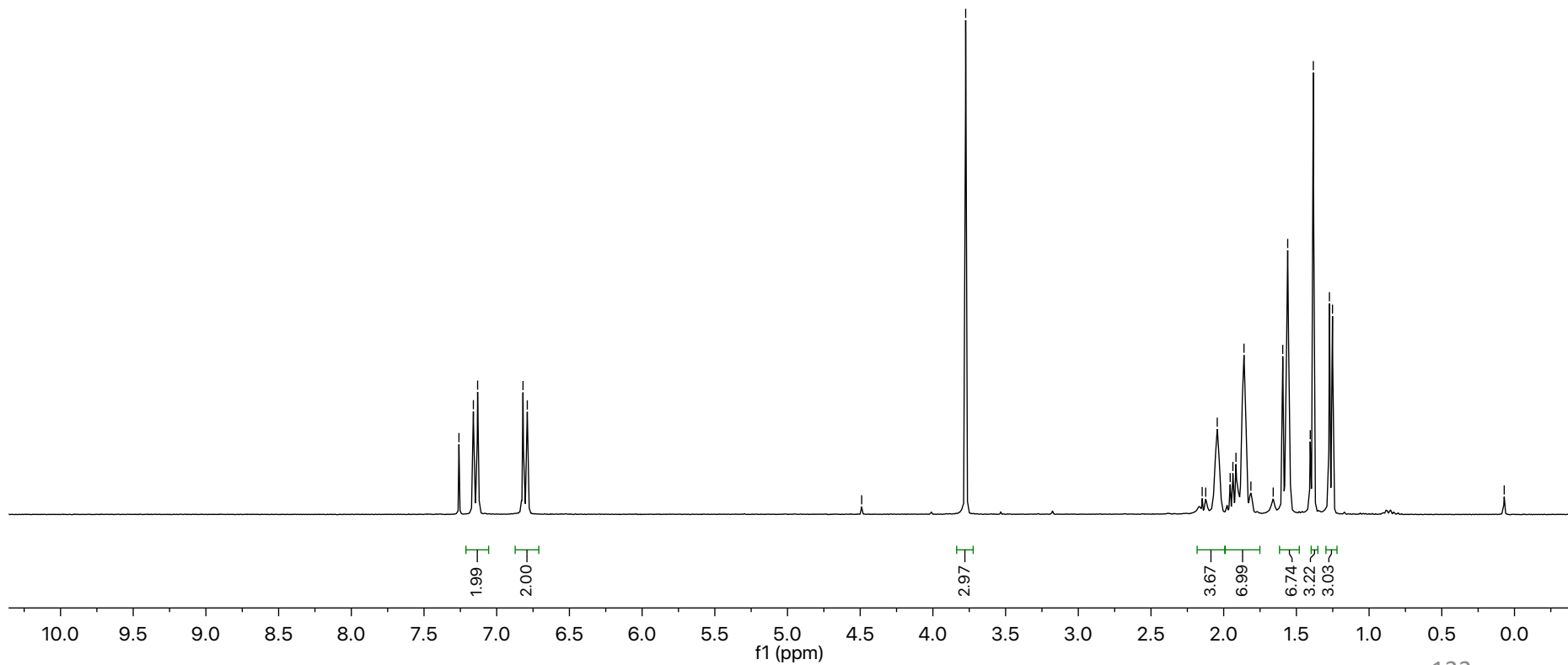
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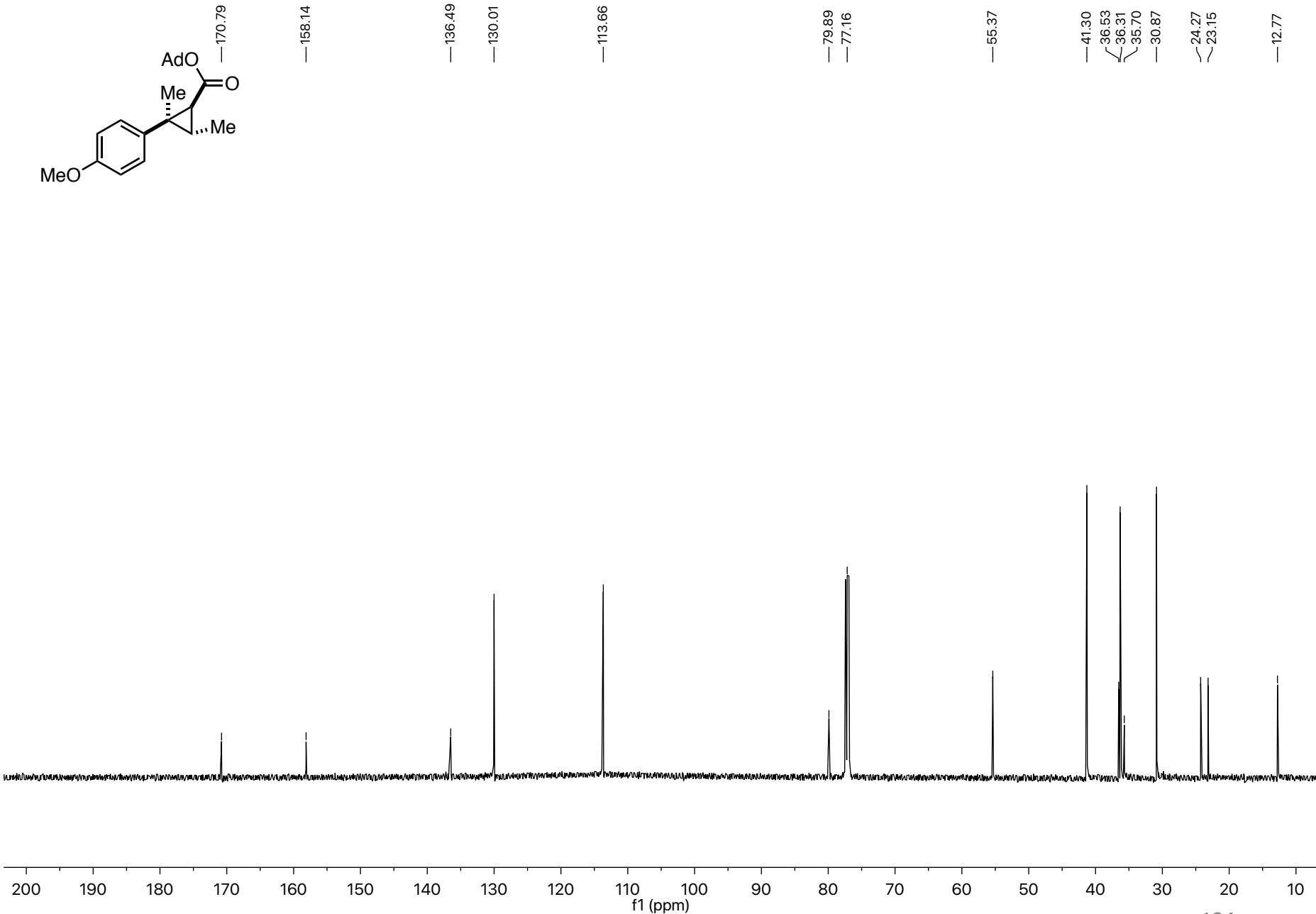
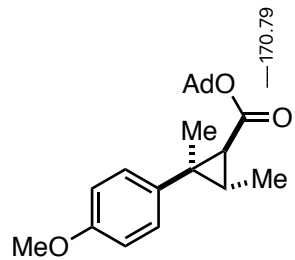
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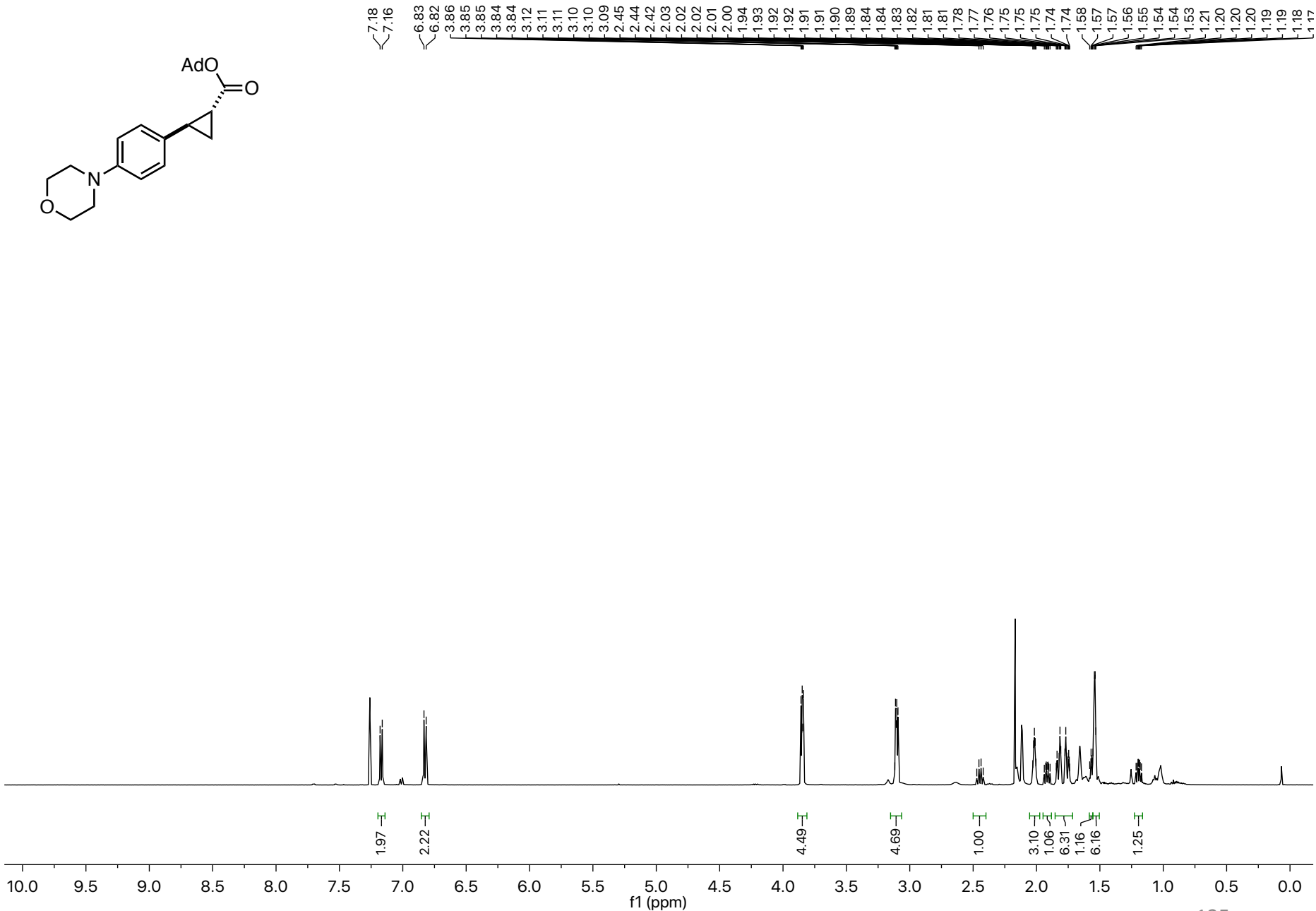
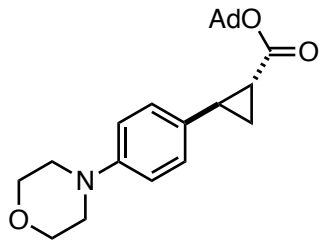
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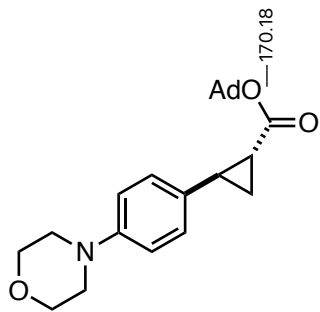
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—150.19

~130.37  
~128.73

—115.59

—80.14  
—77.16

—67.09

—49.93

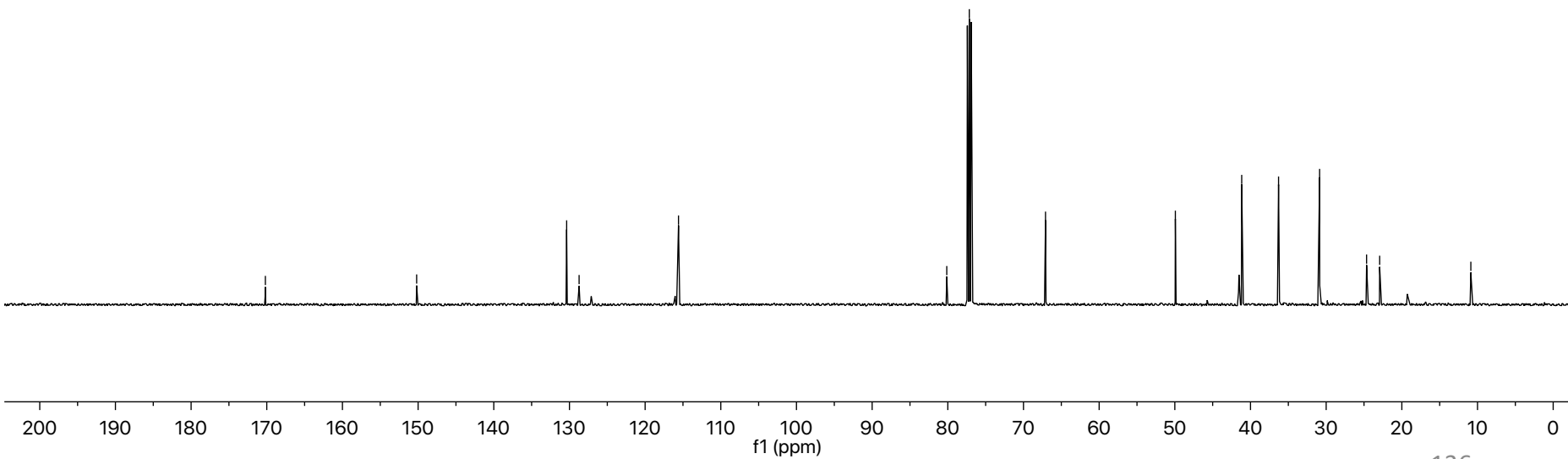
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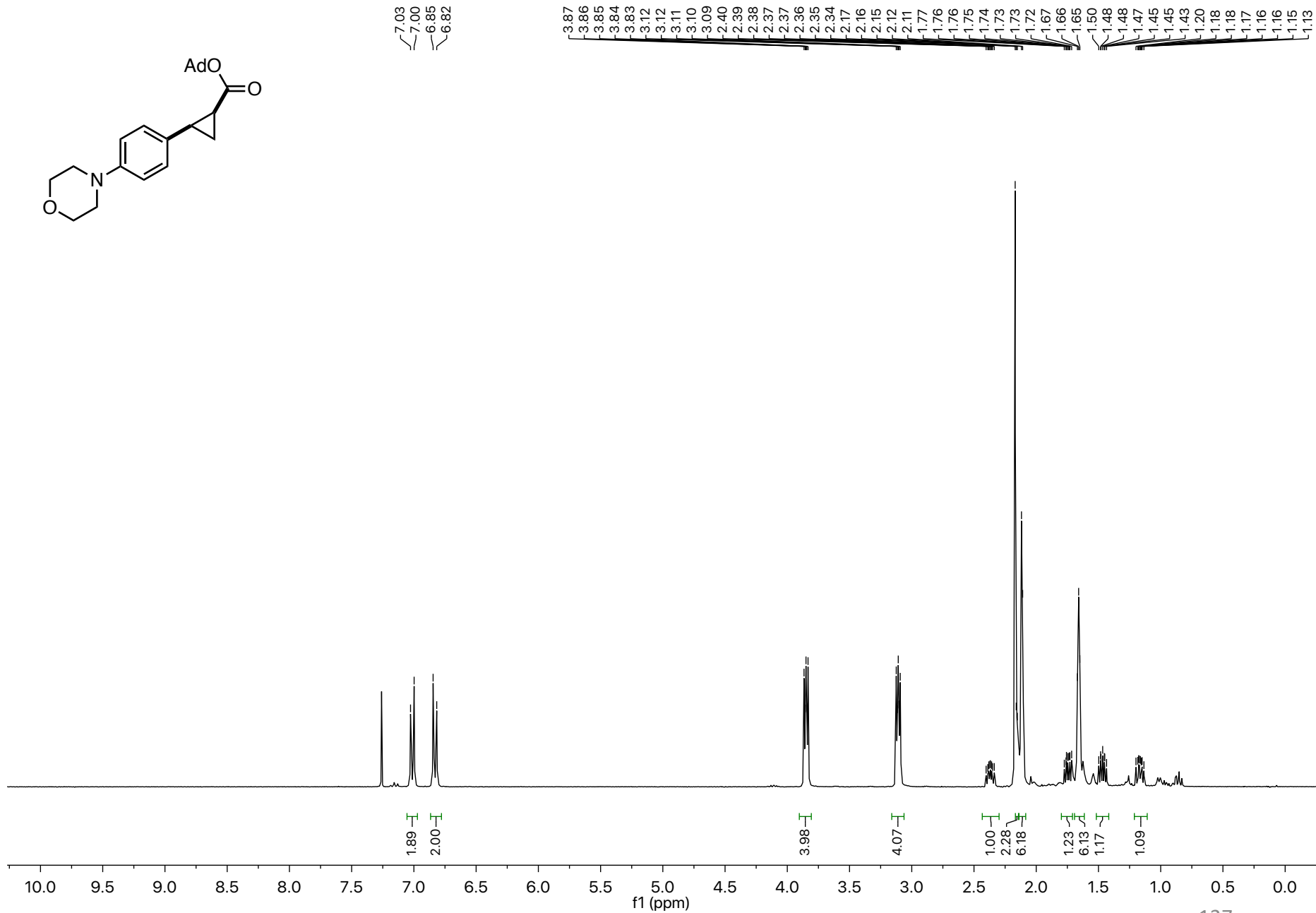
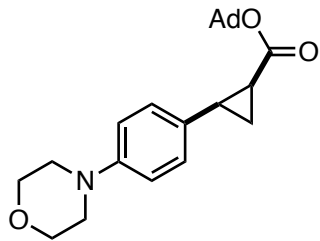
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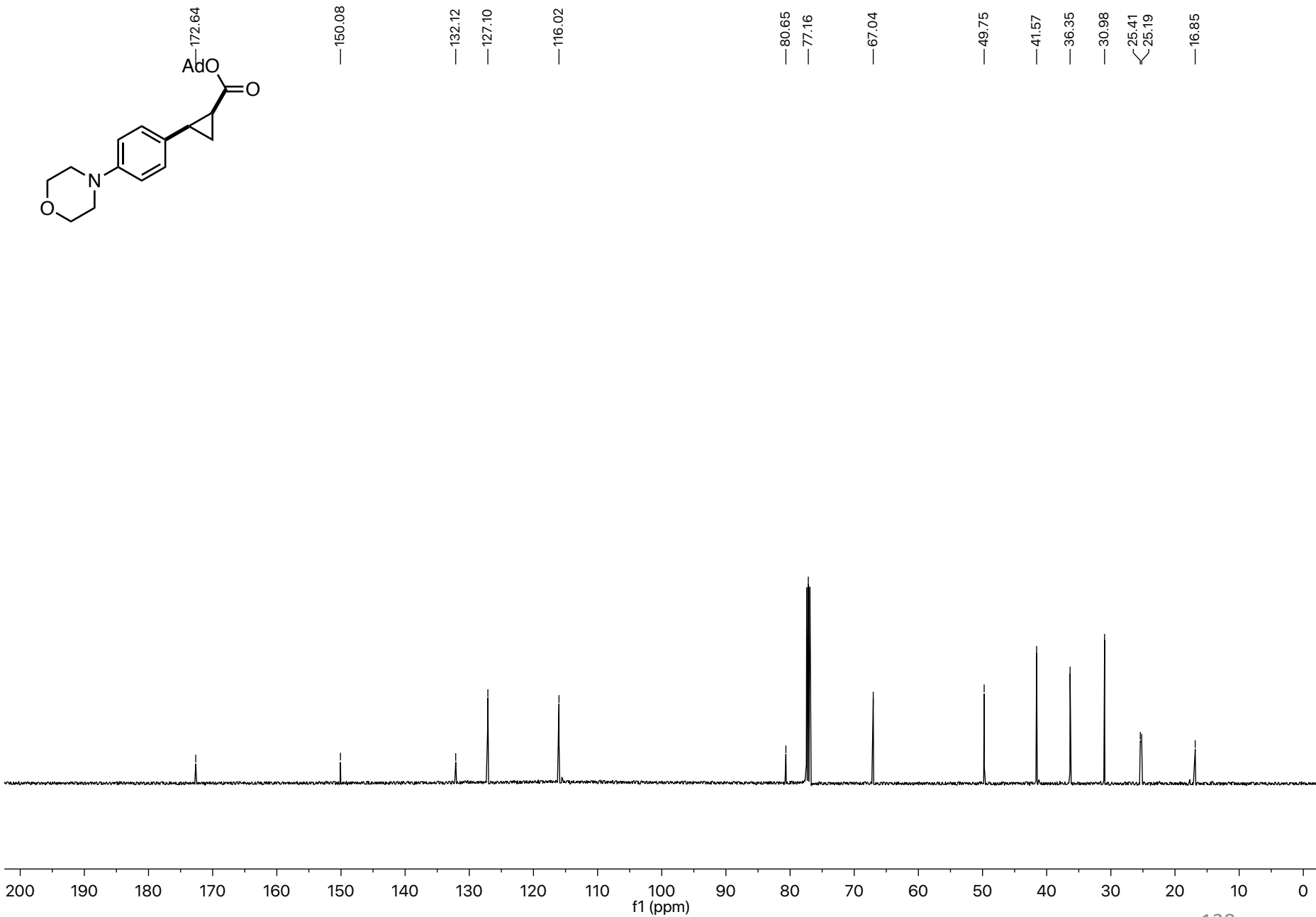
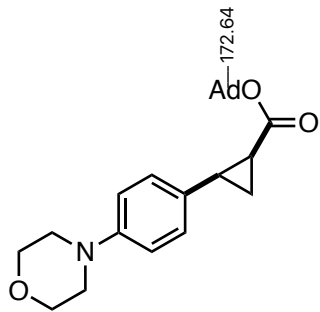
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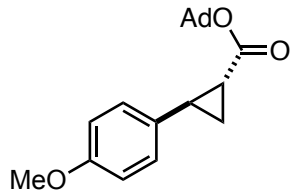
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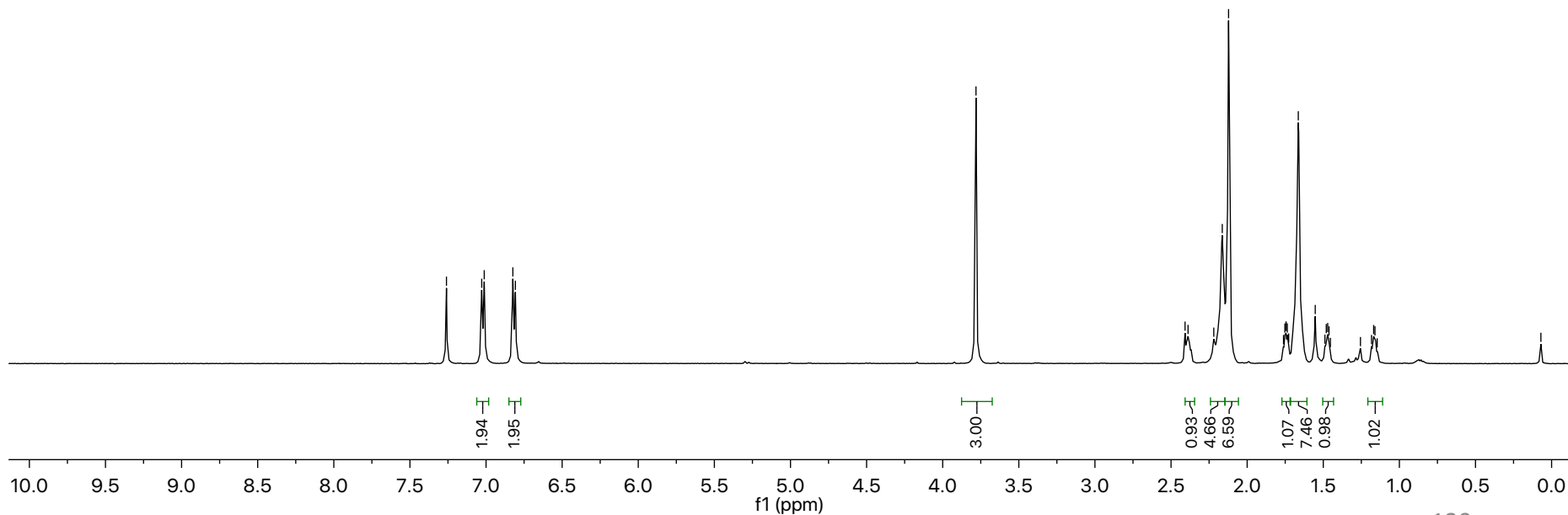


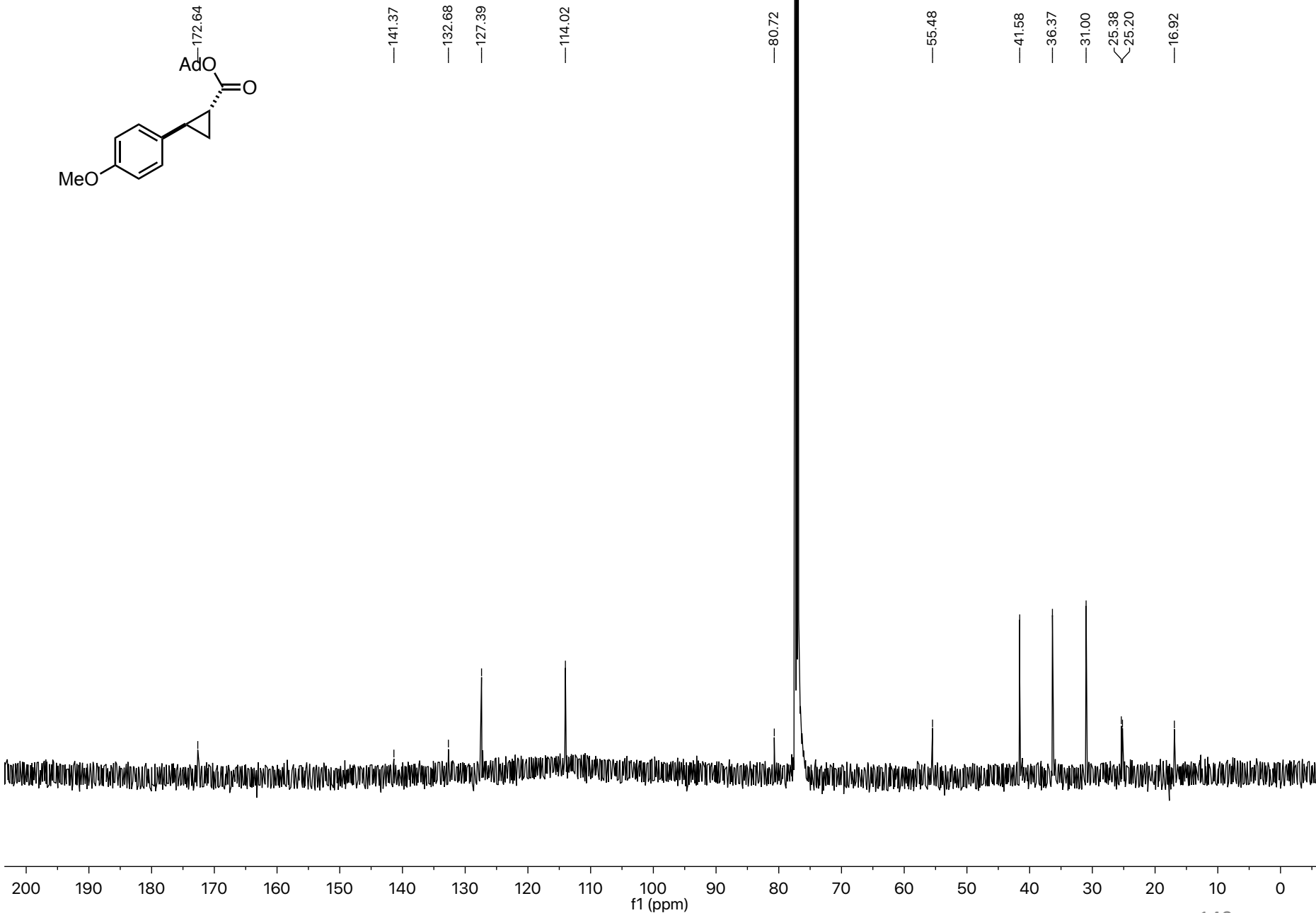
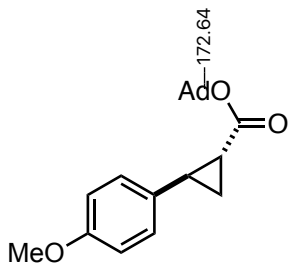


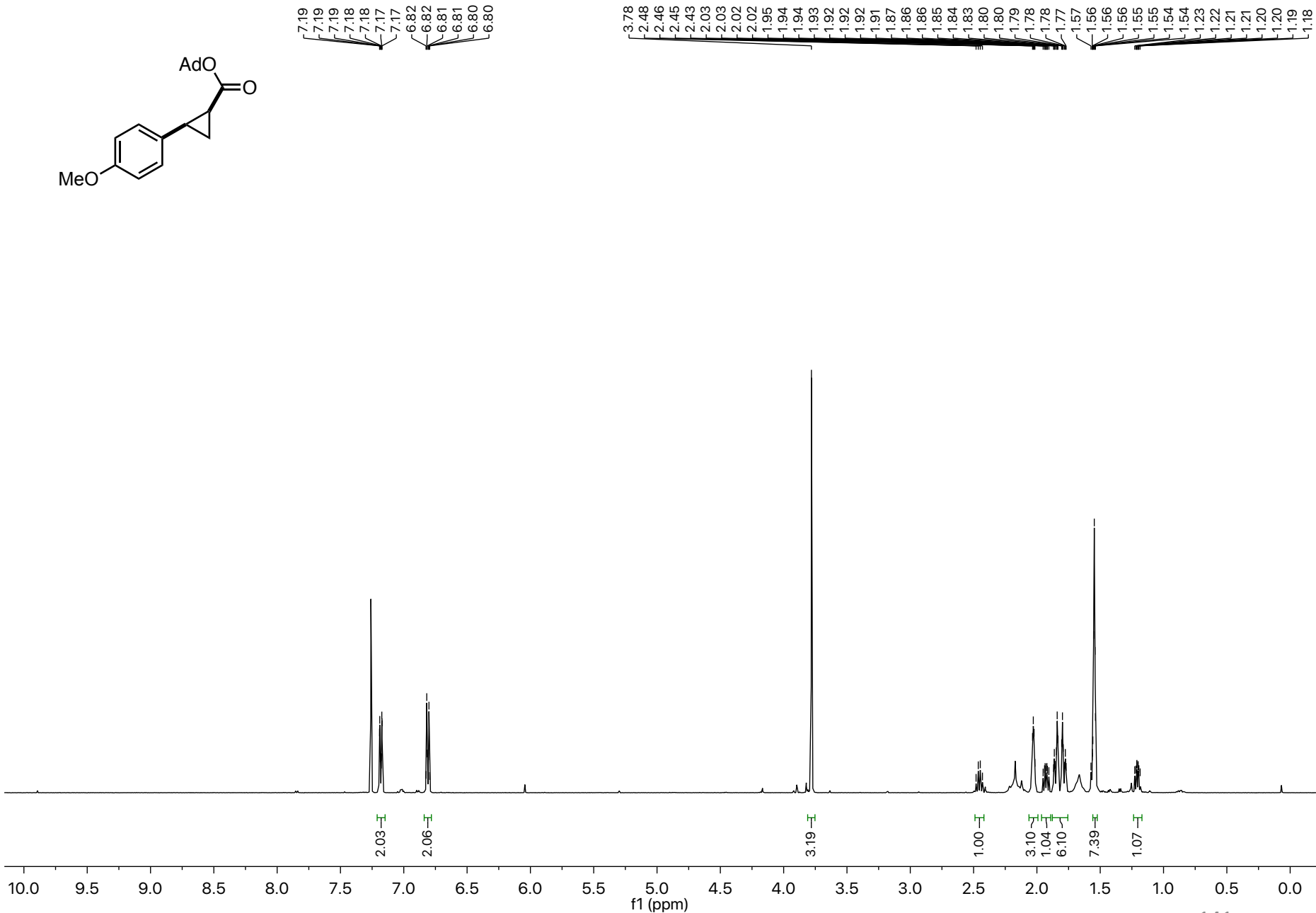
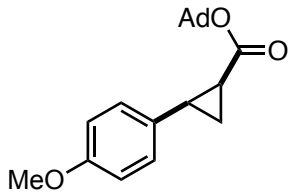
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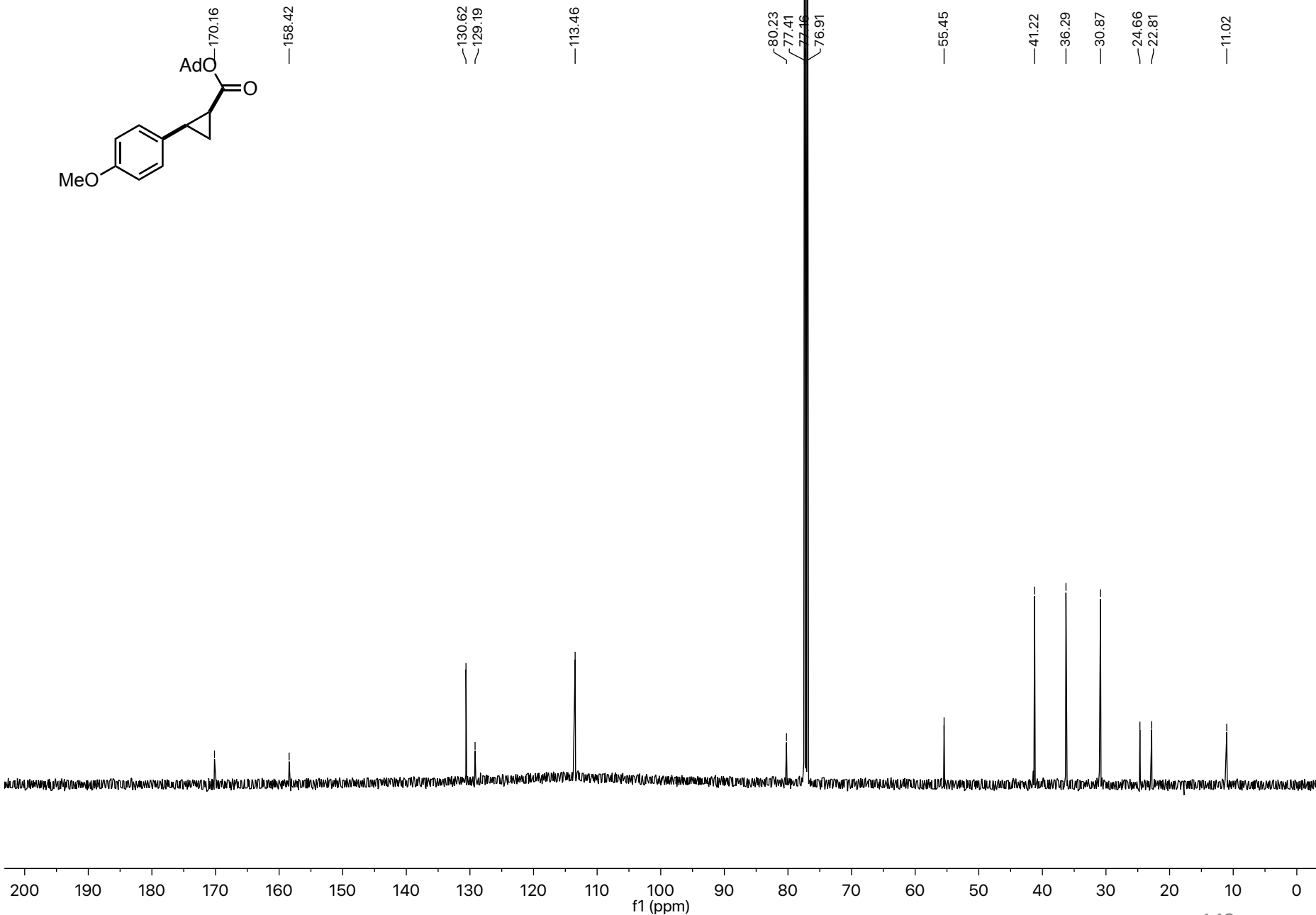
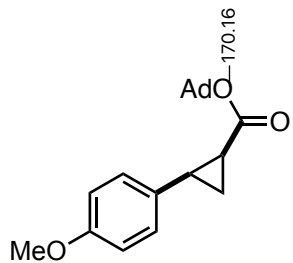
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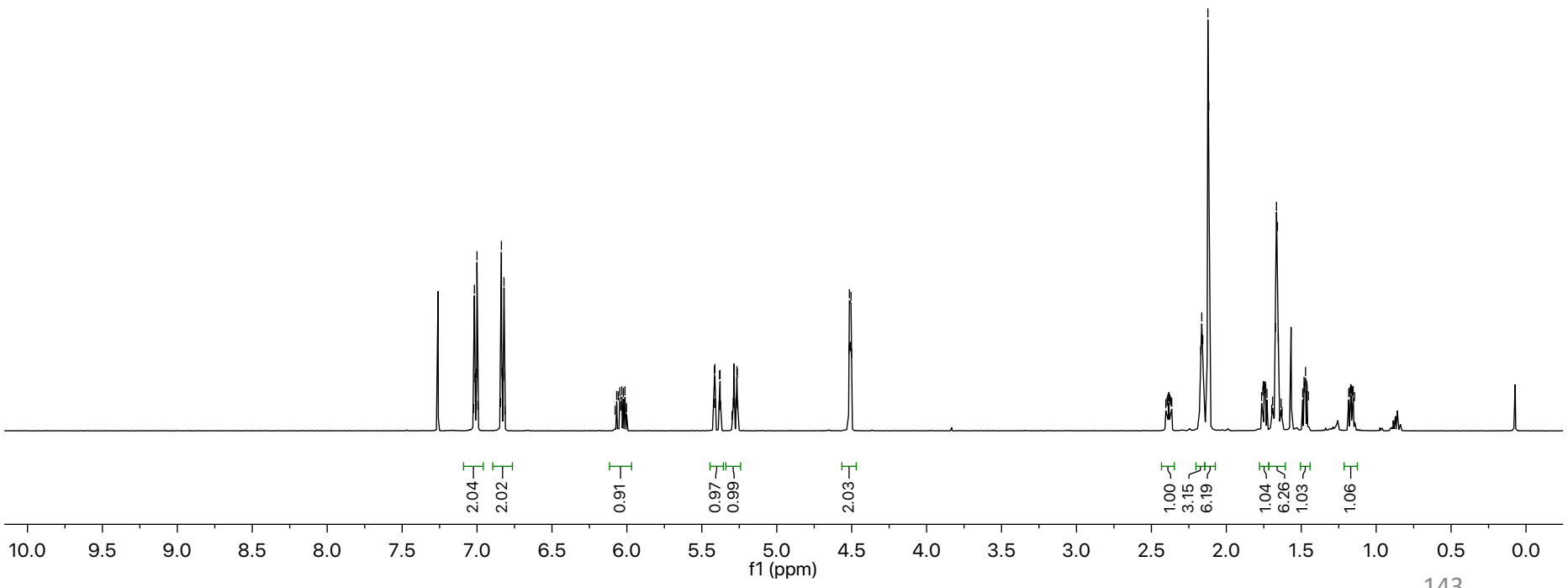
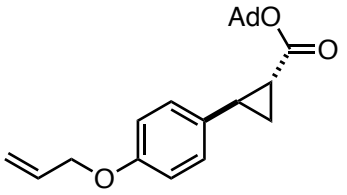


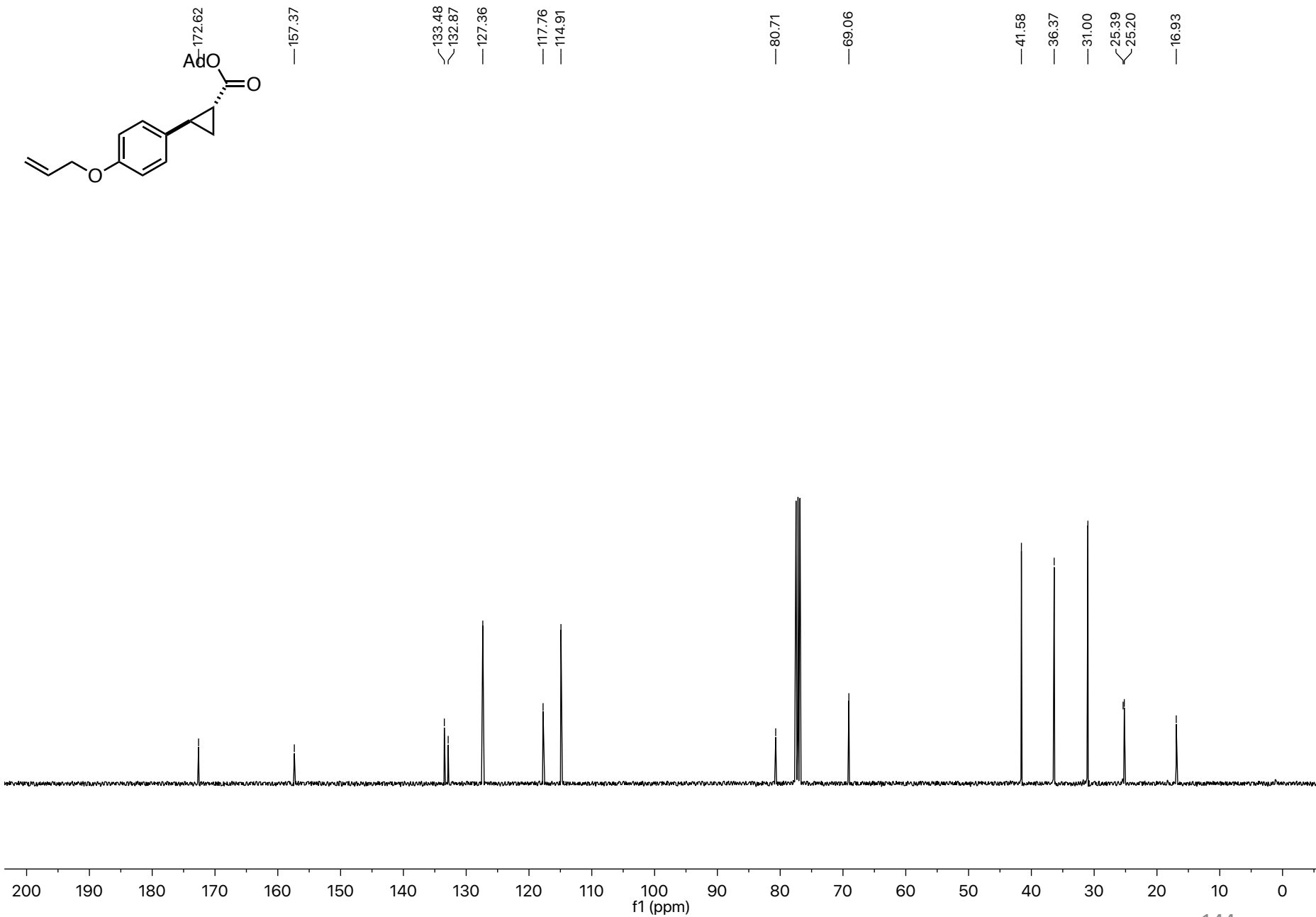
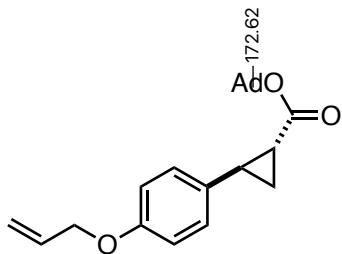






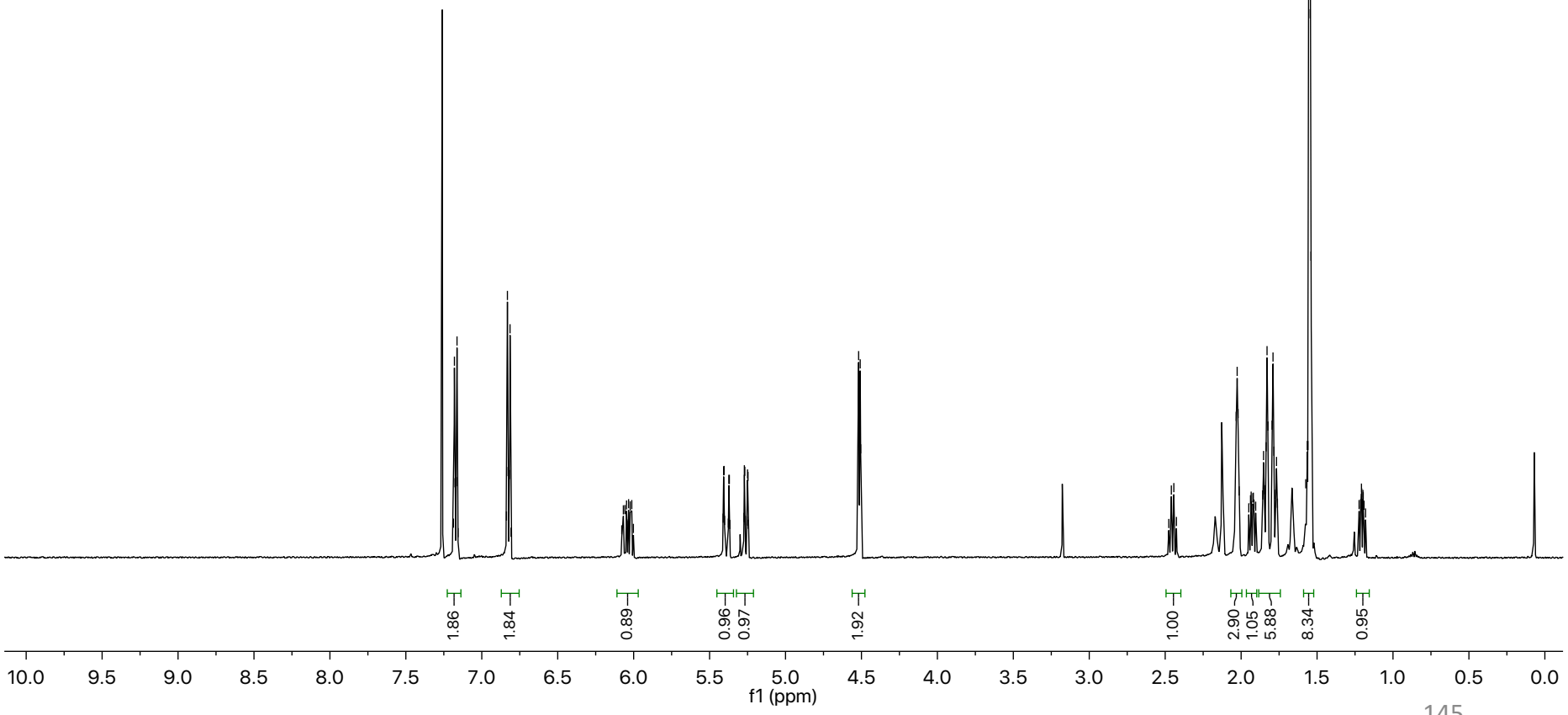
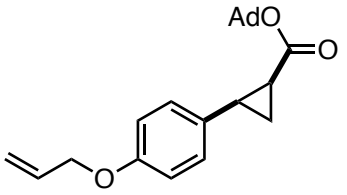
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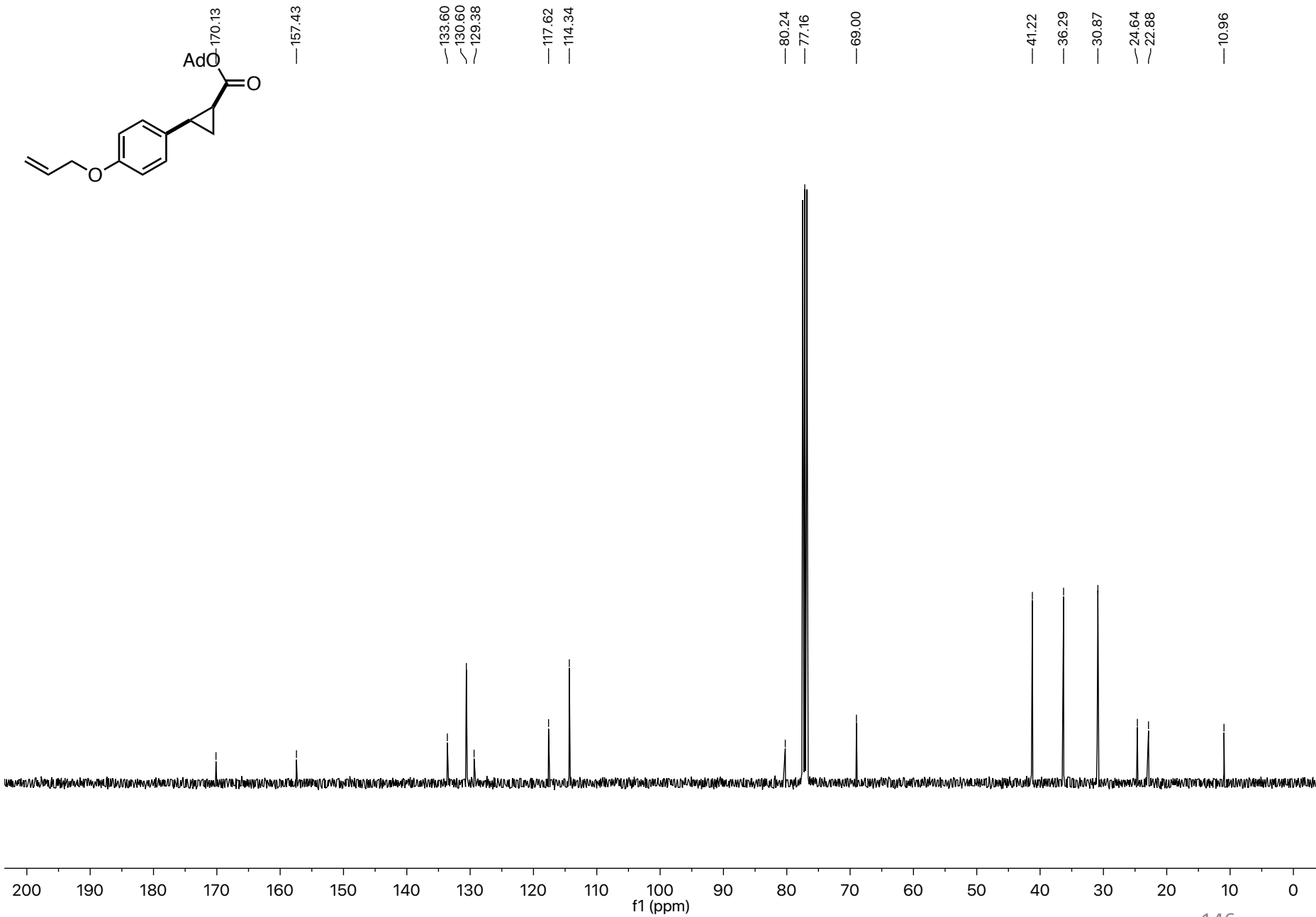
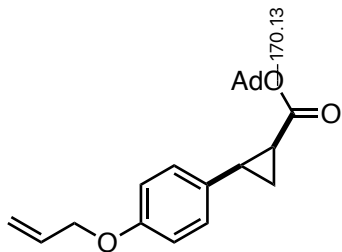


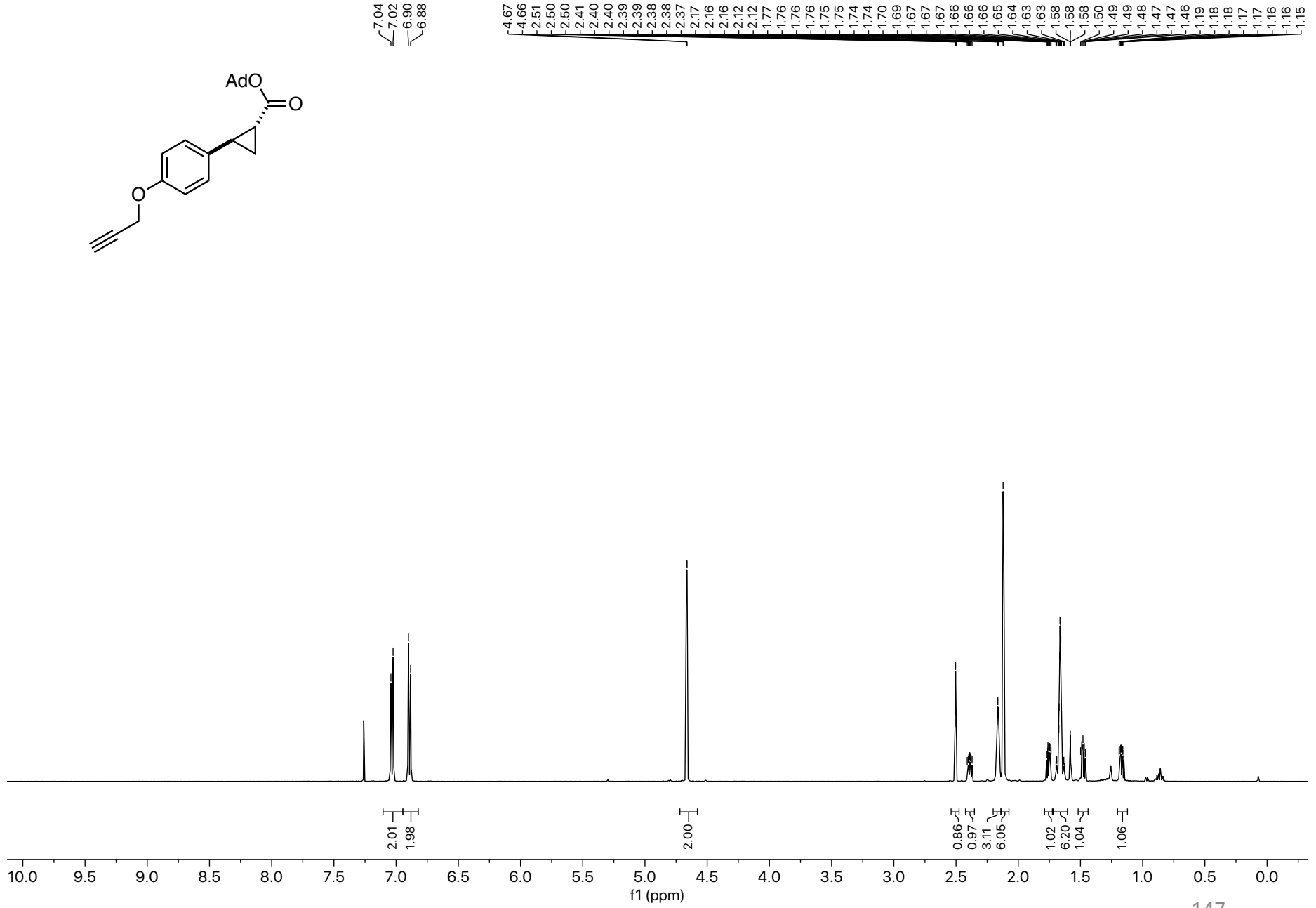
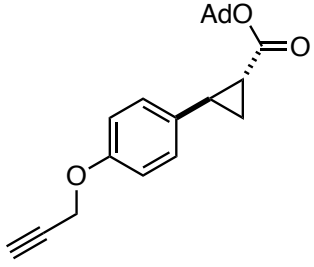


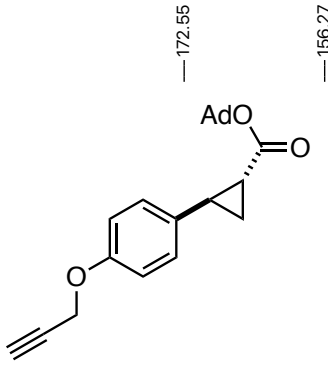


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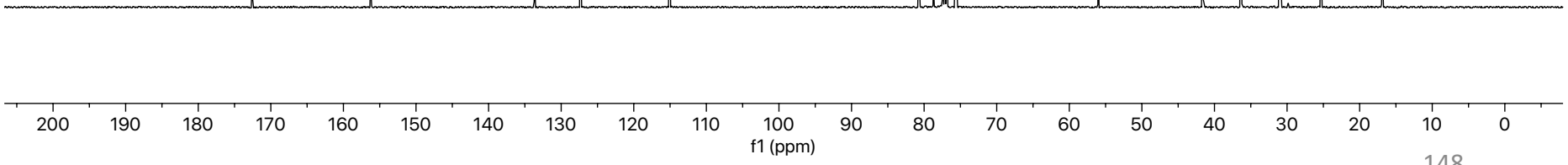
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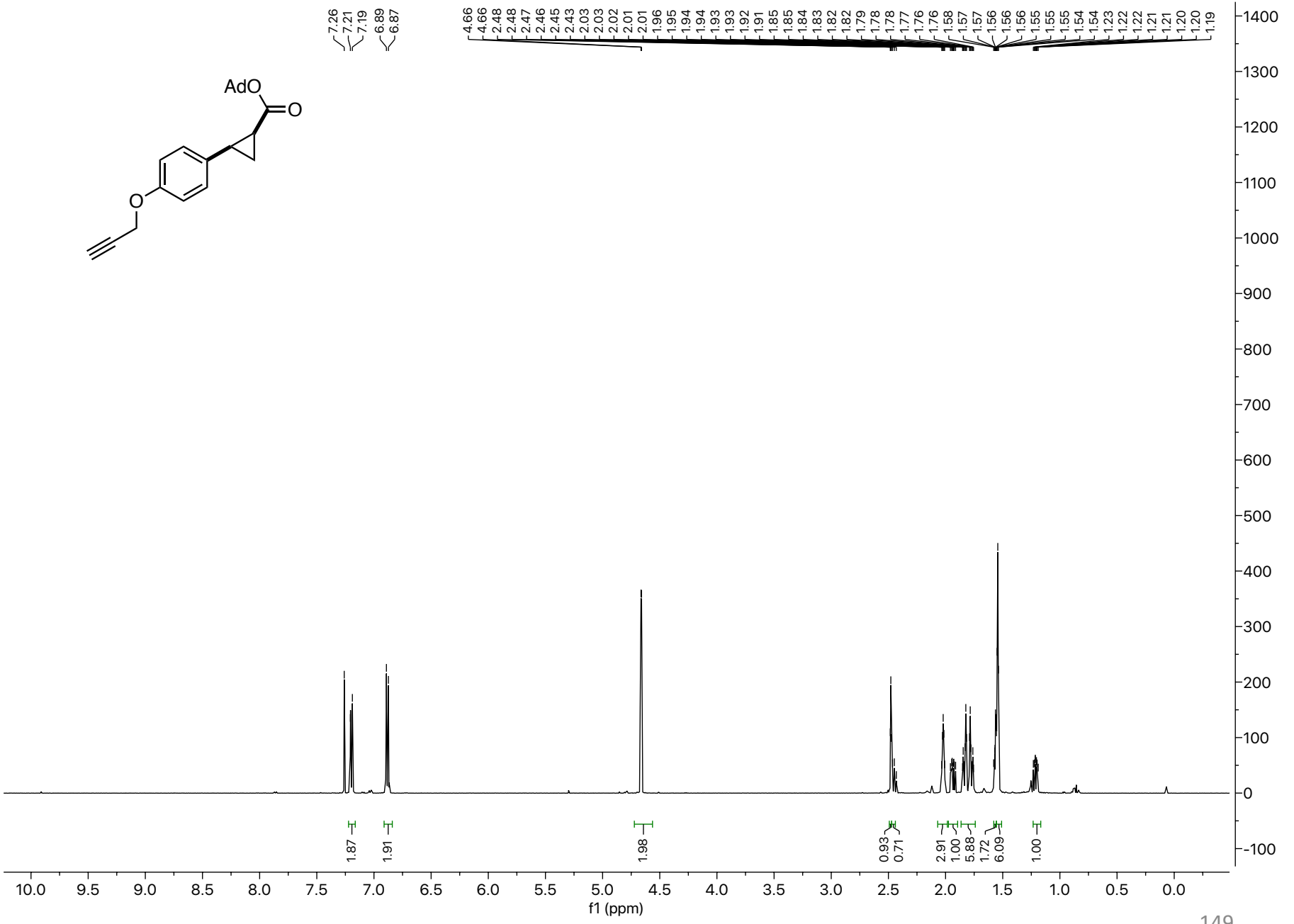
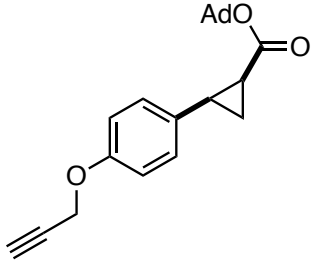
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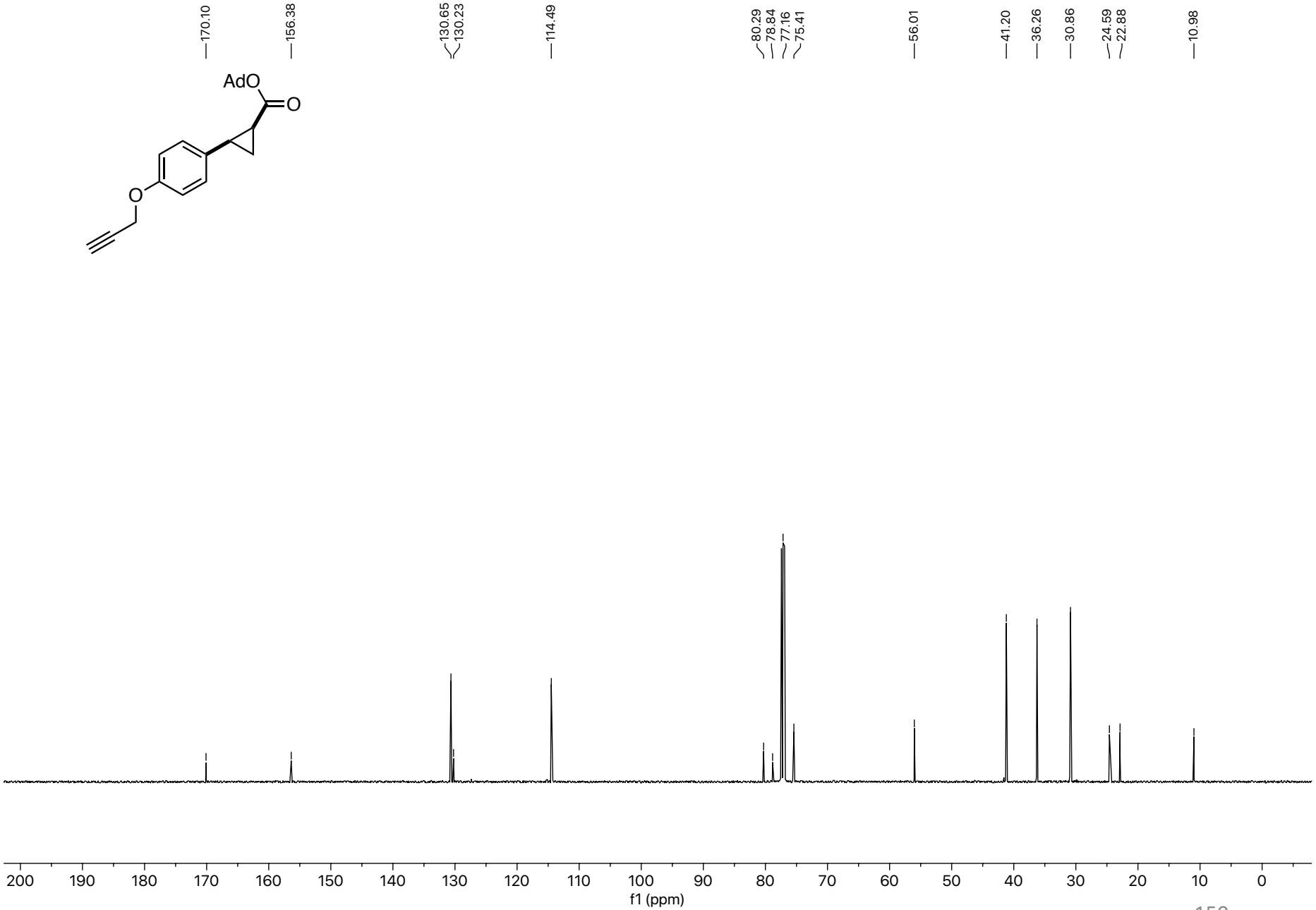
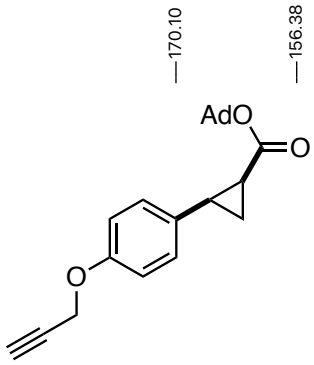
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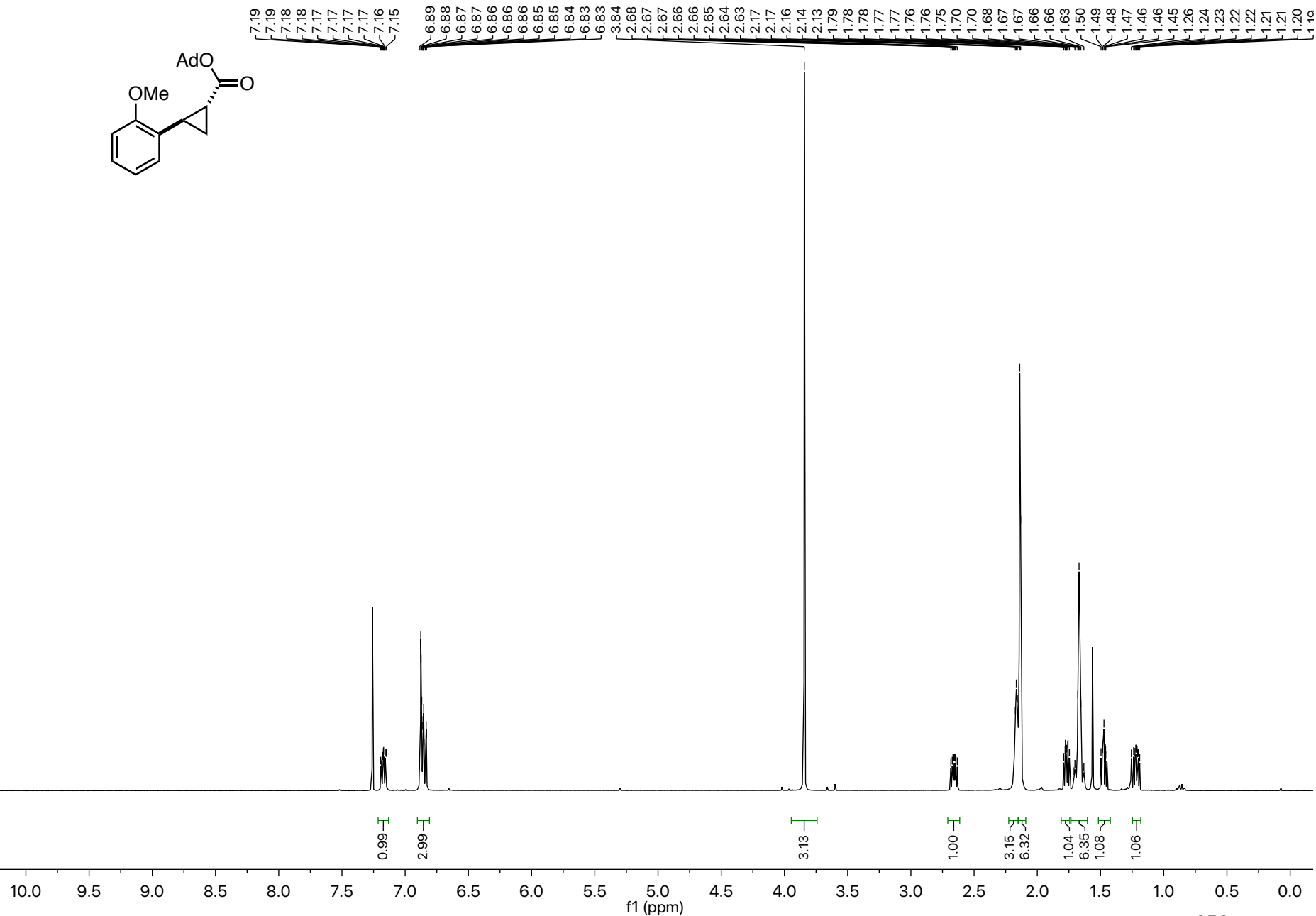
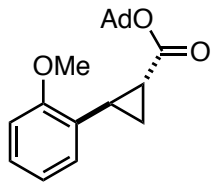
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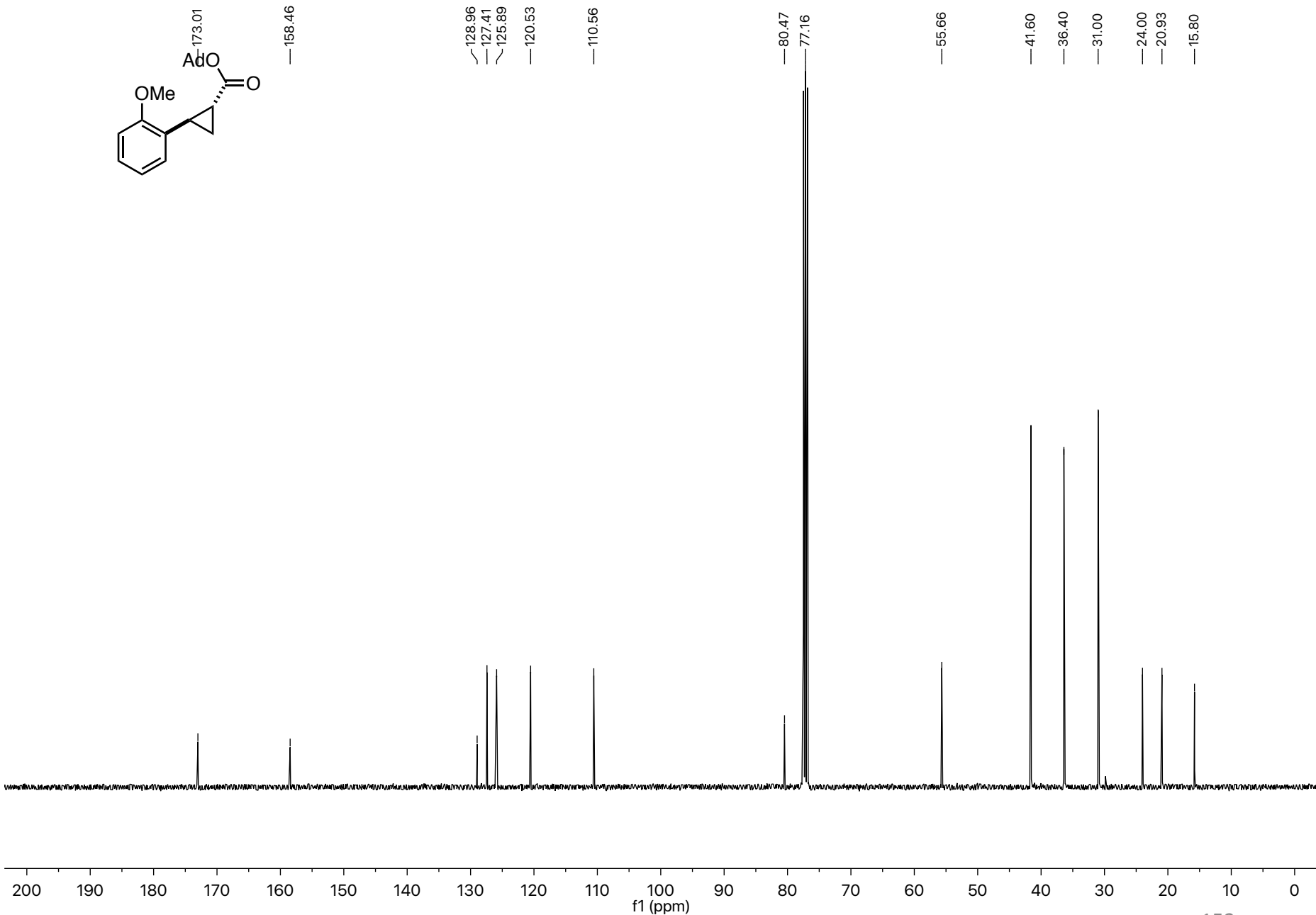
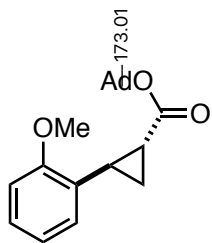
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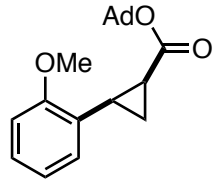




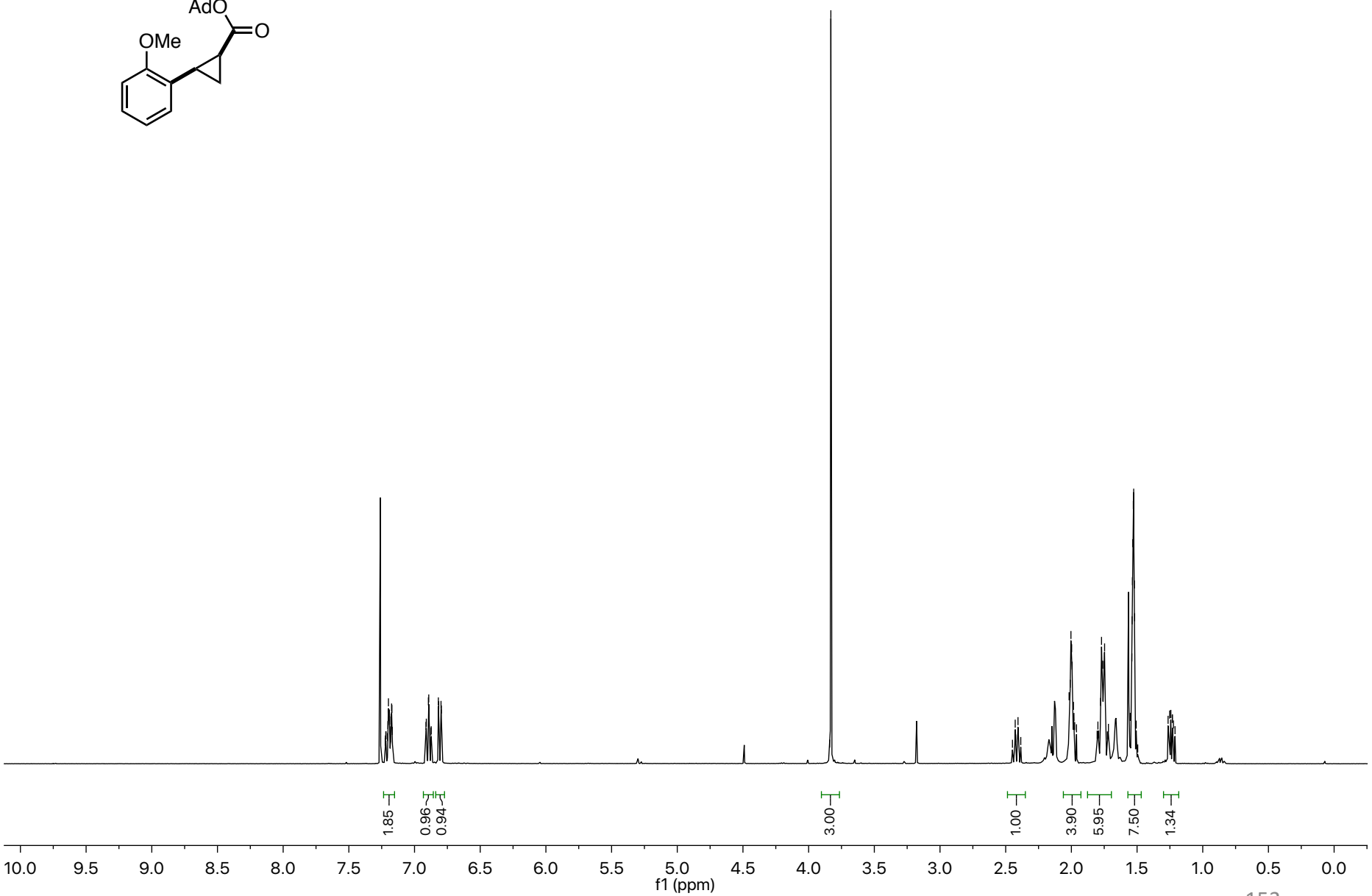


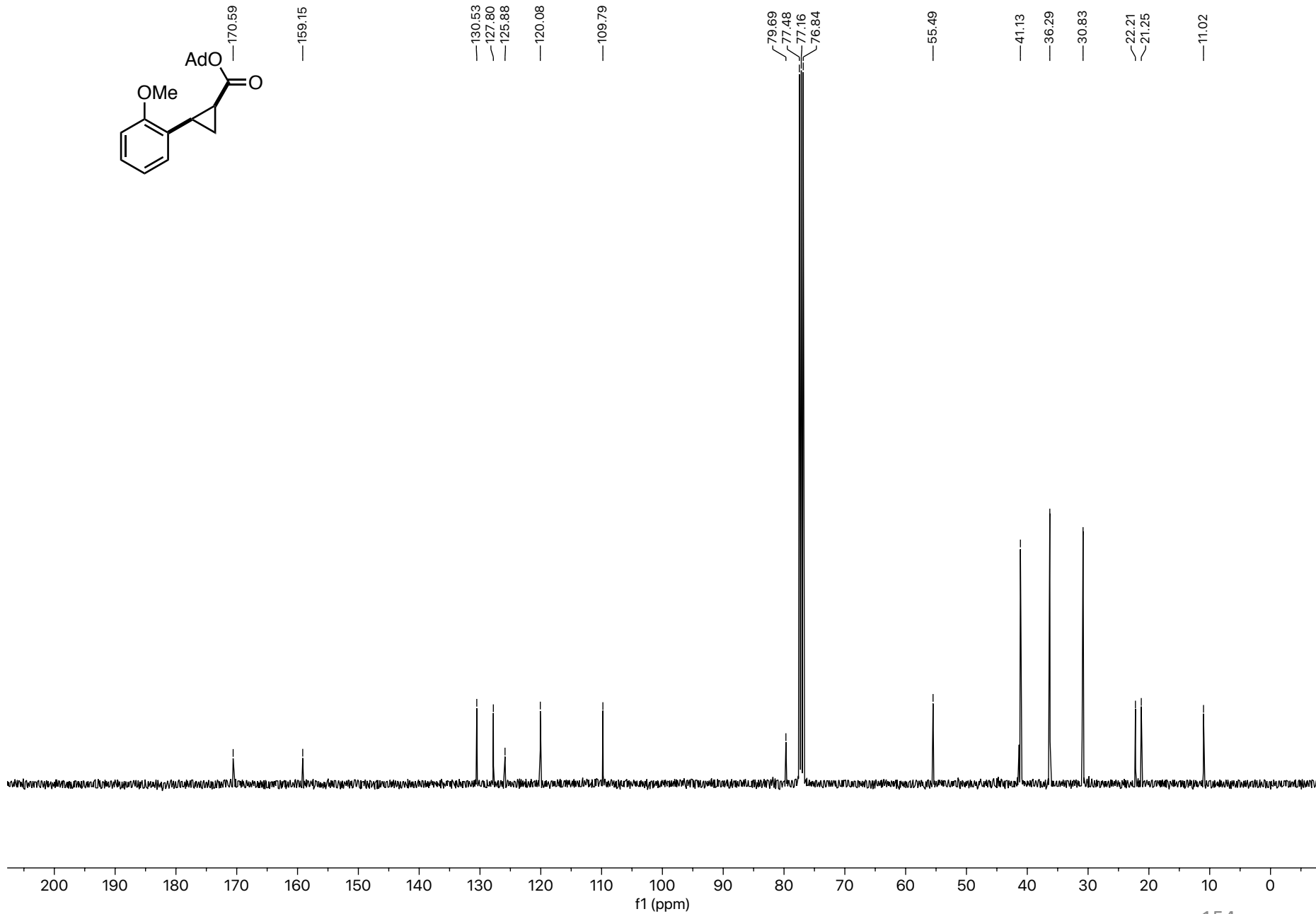
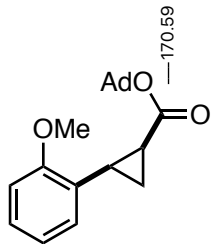


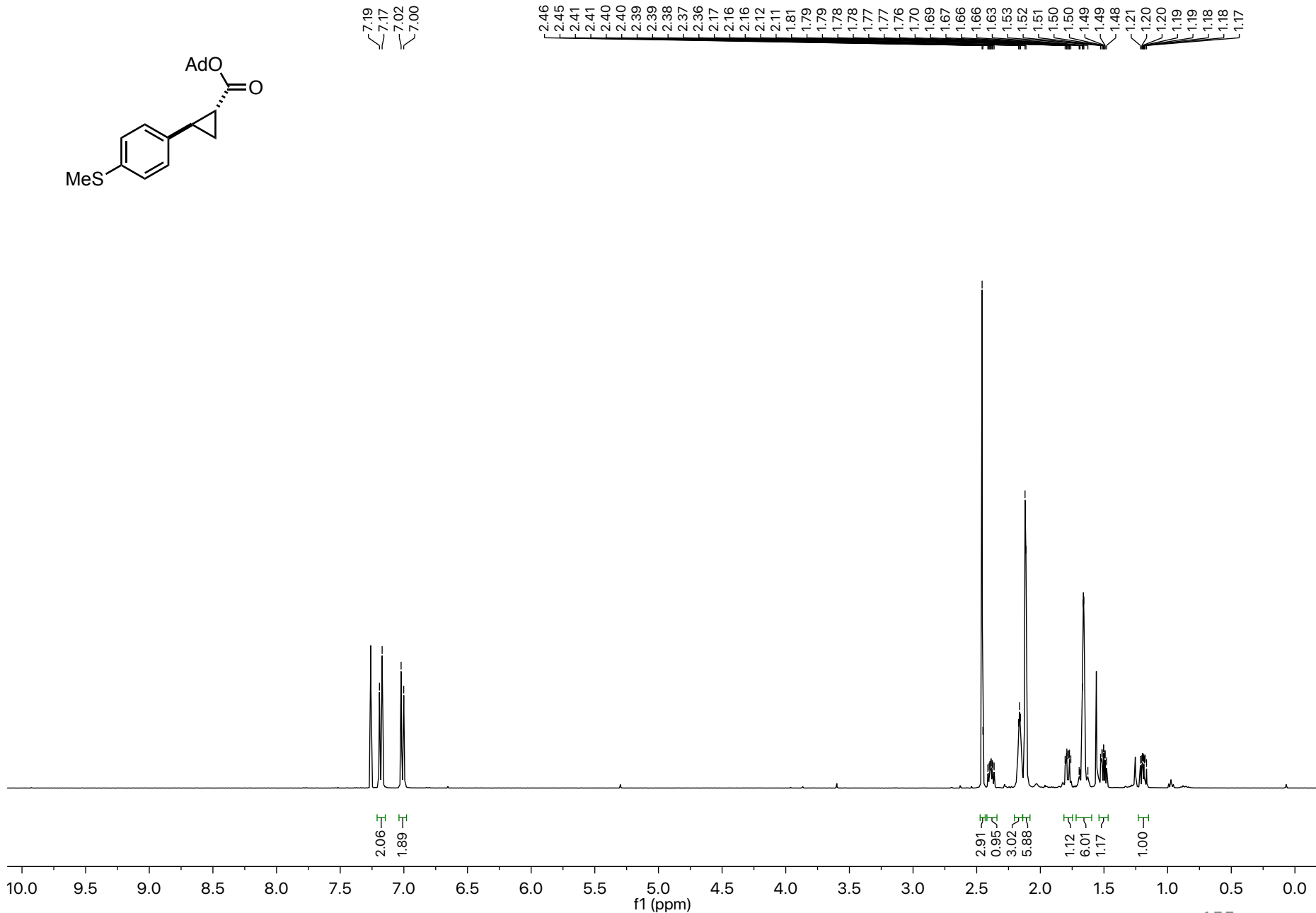
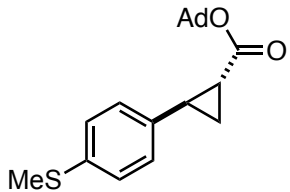
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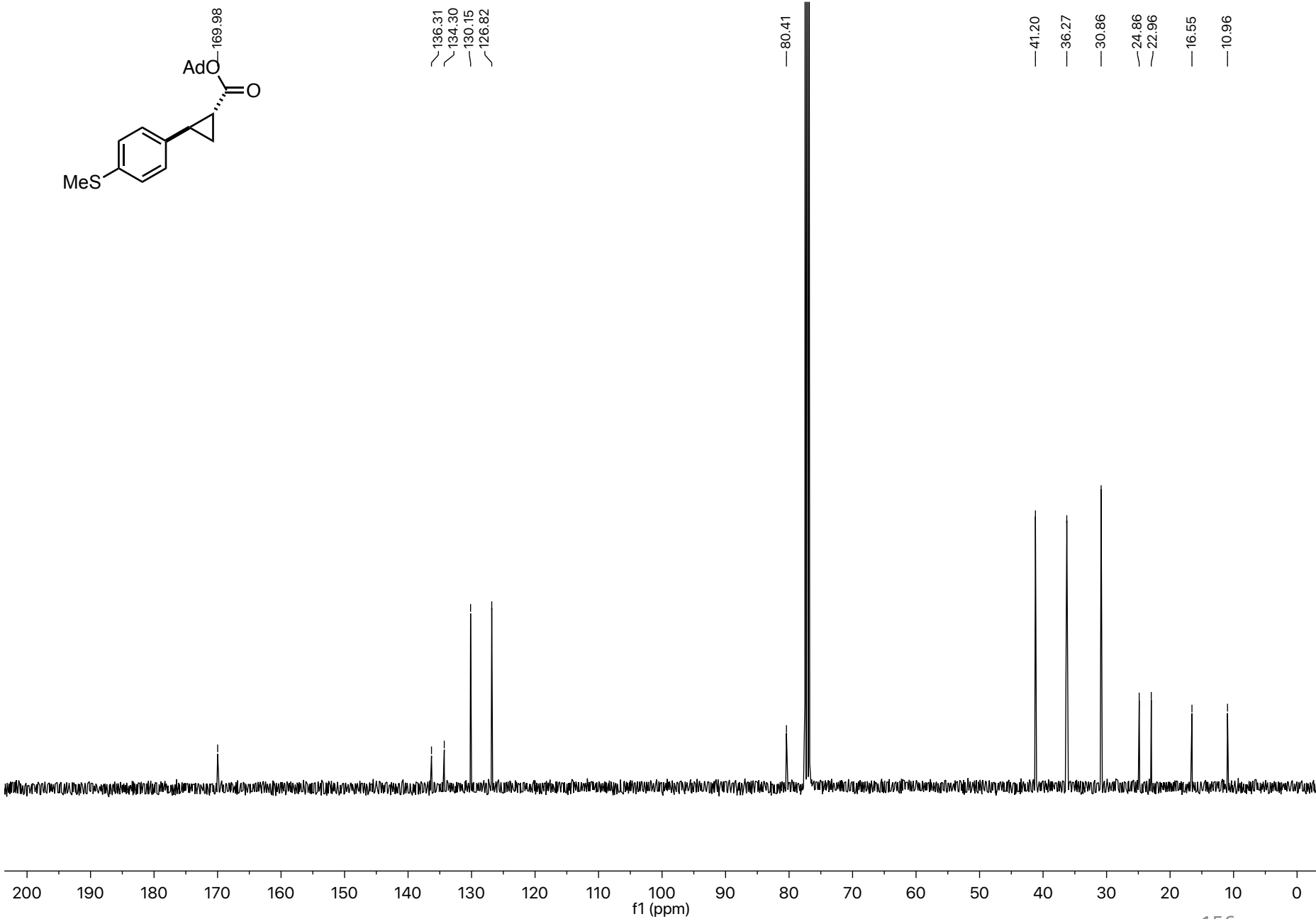
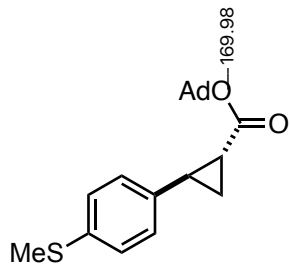


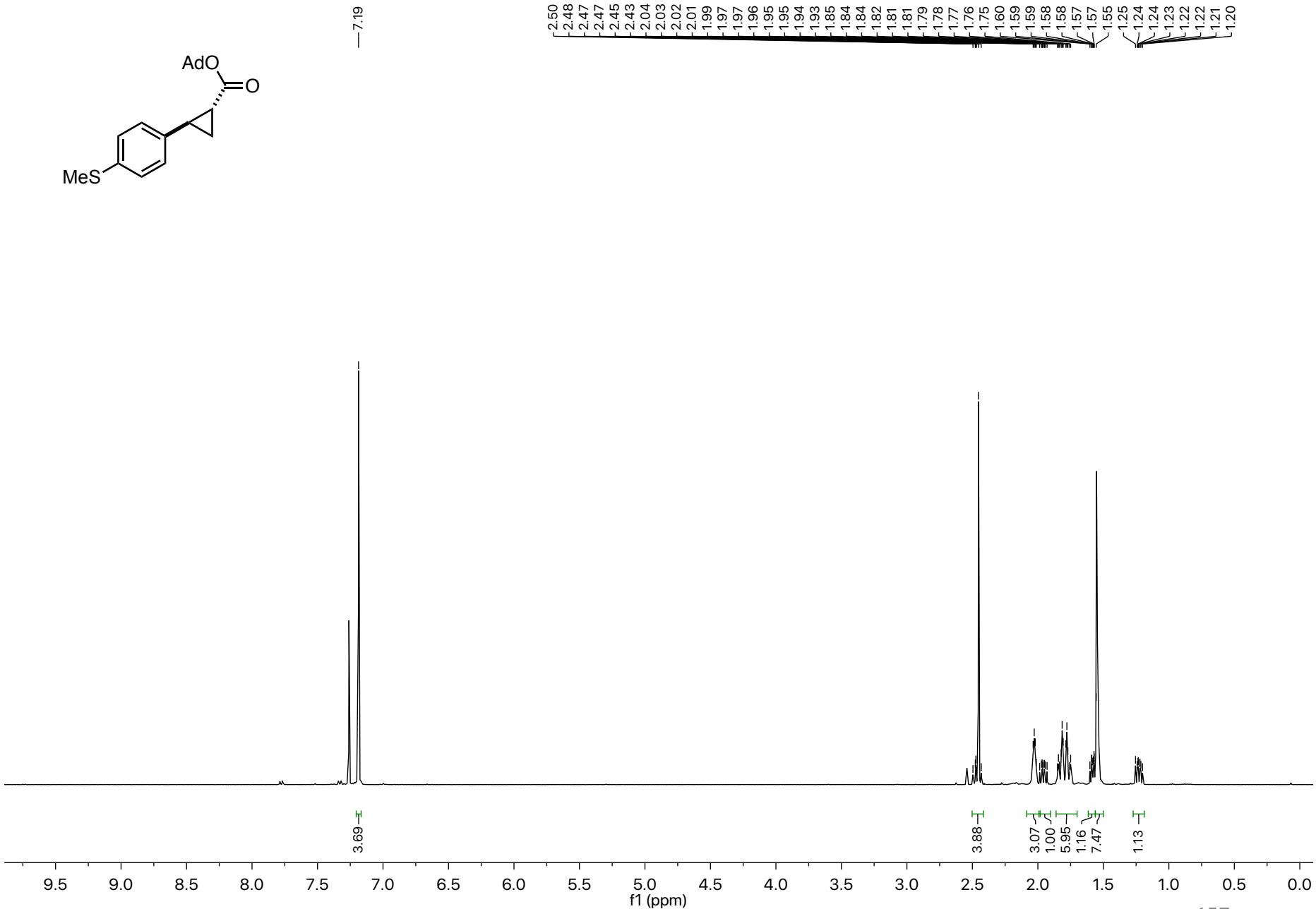
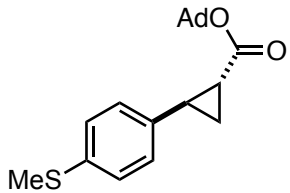
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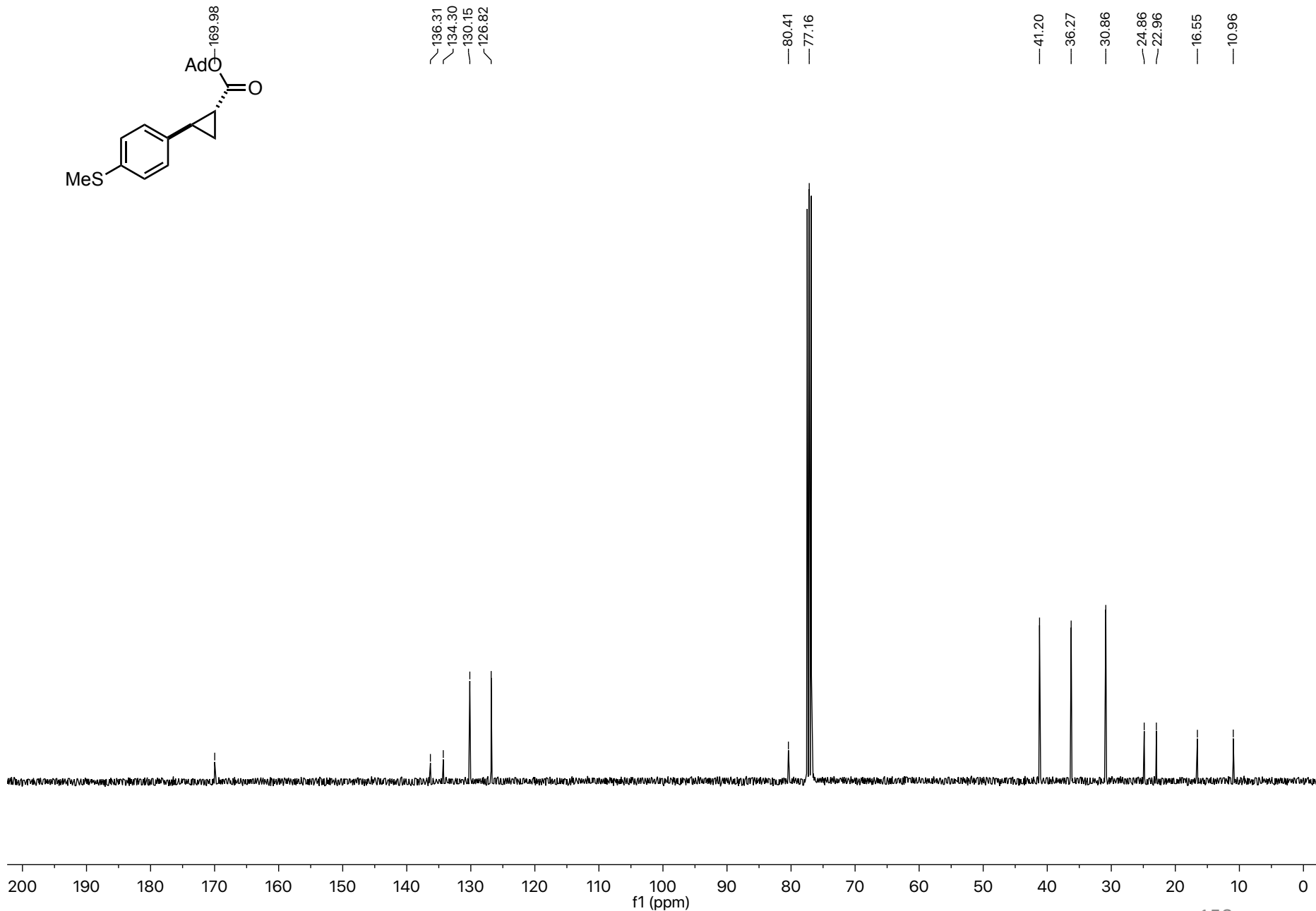
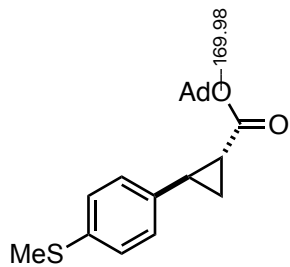


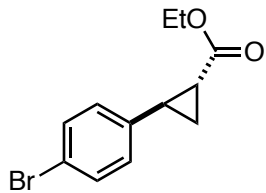












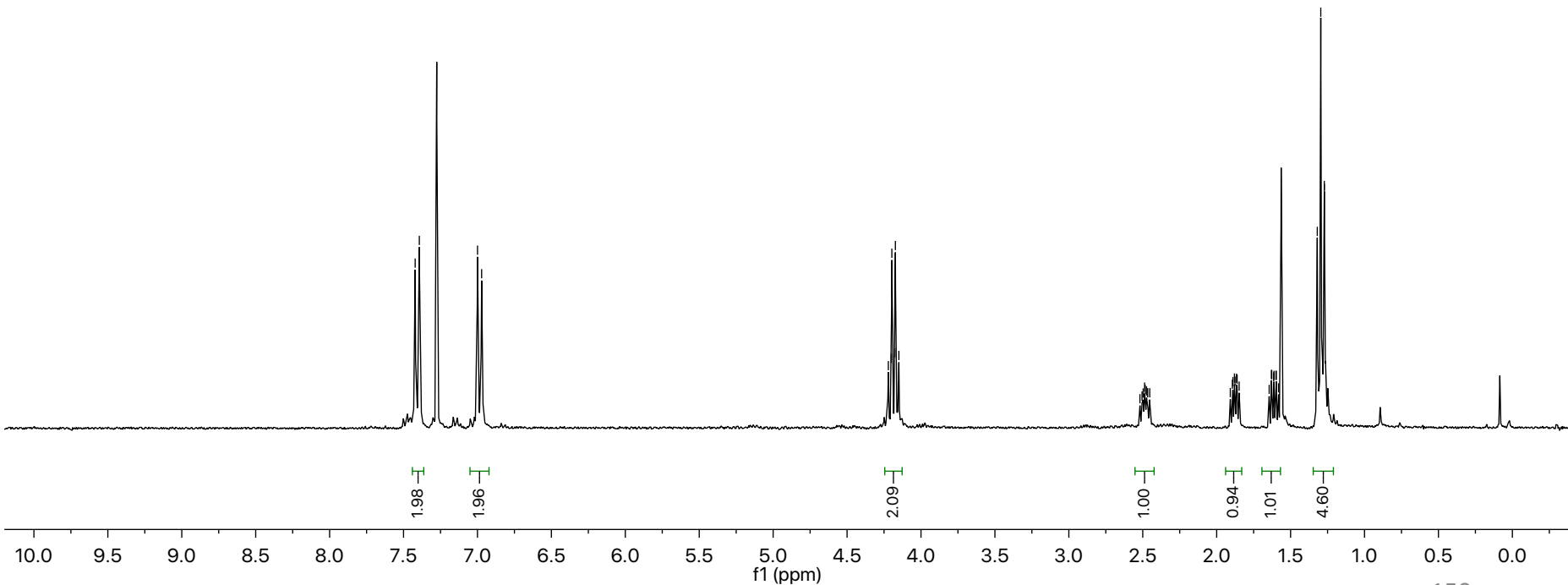
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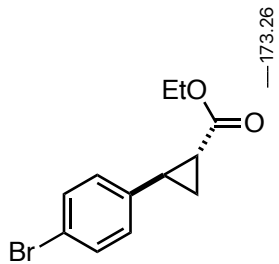
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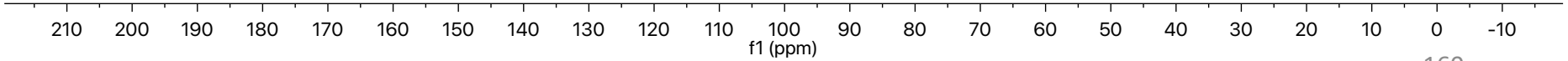
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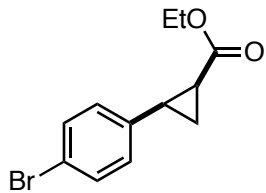
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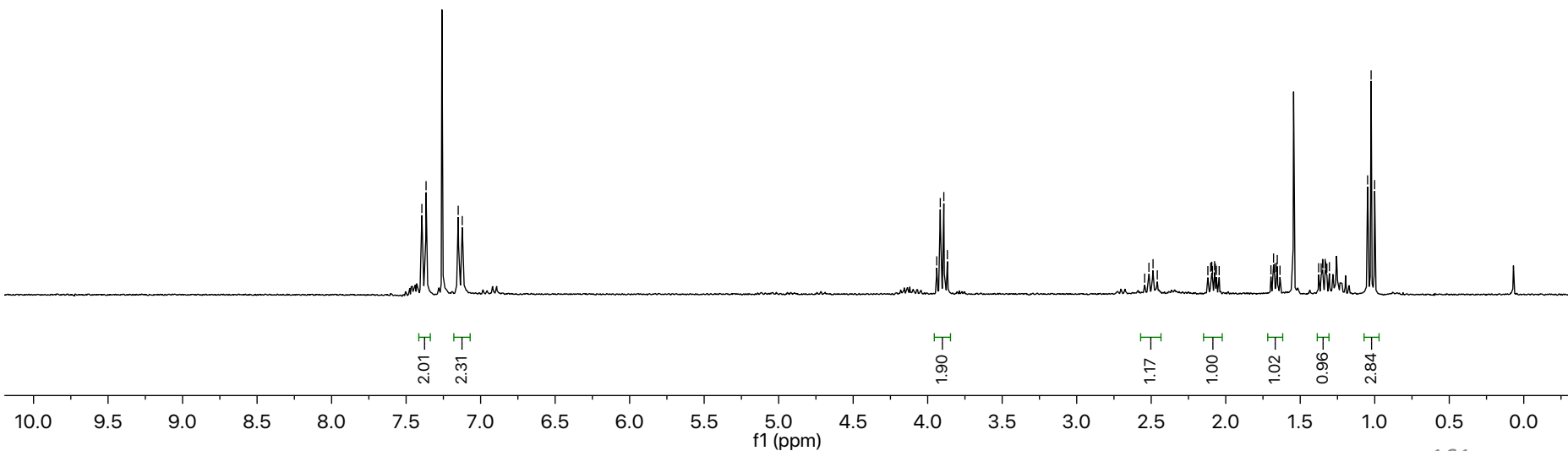


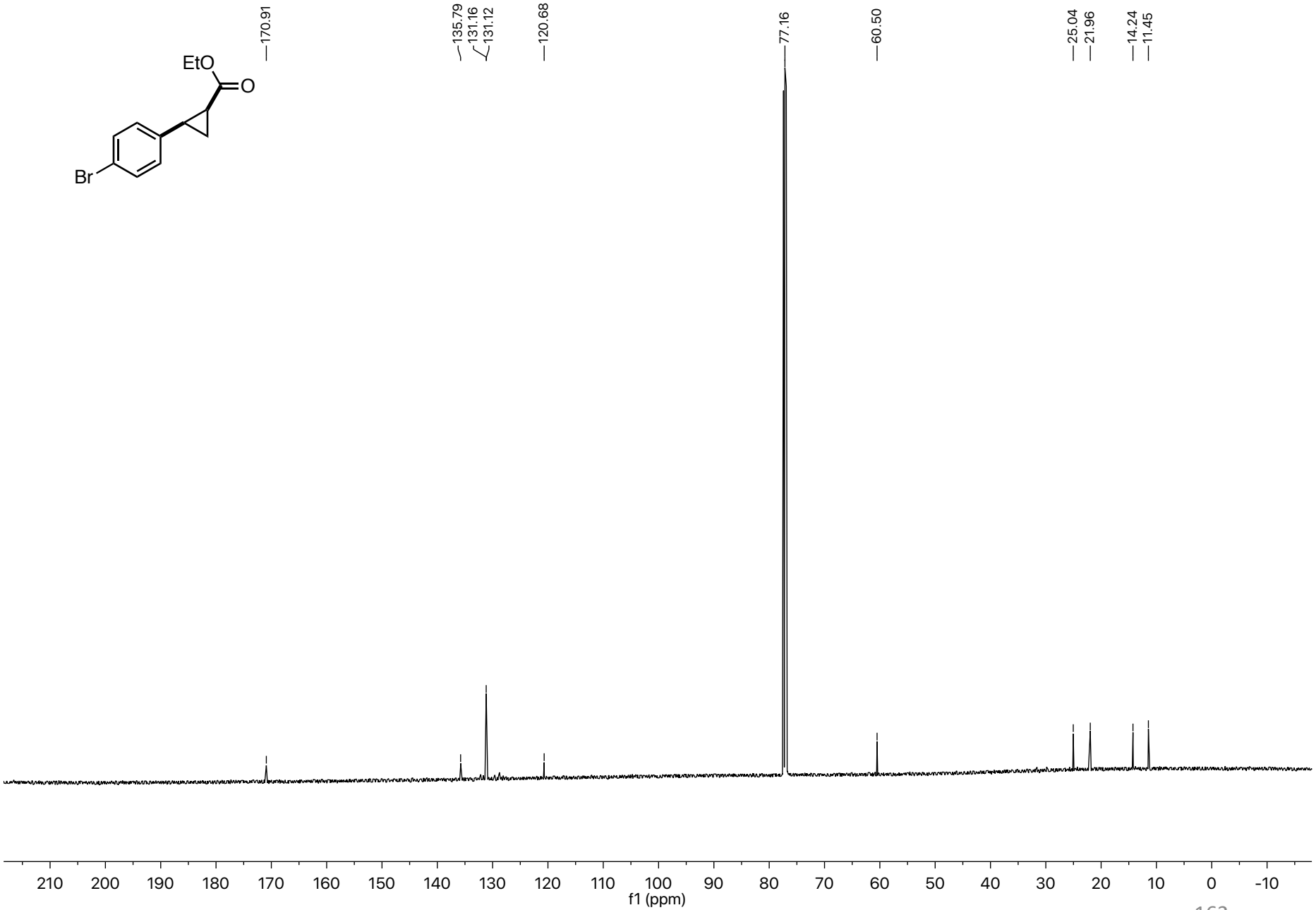
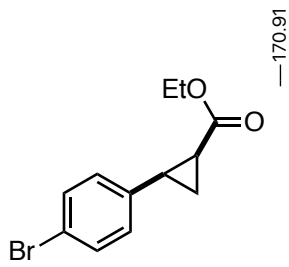


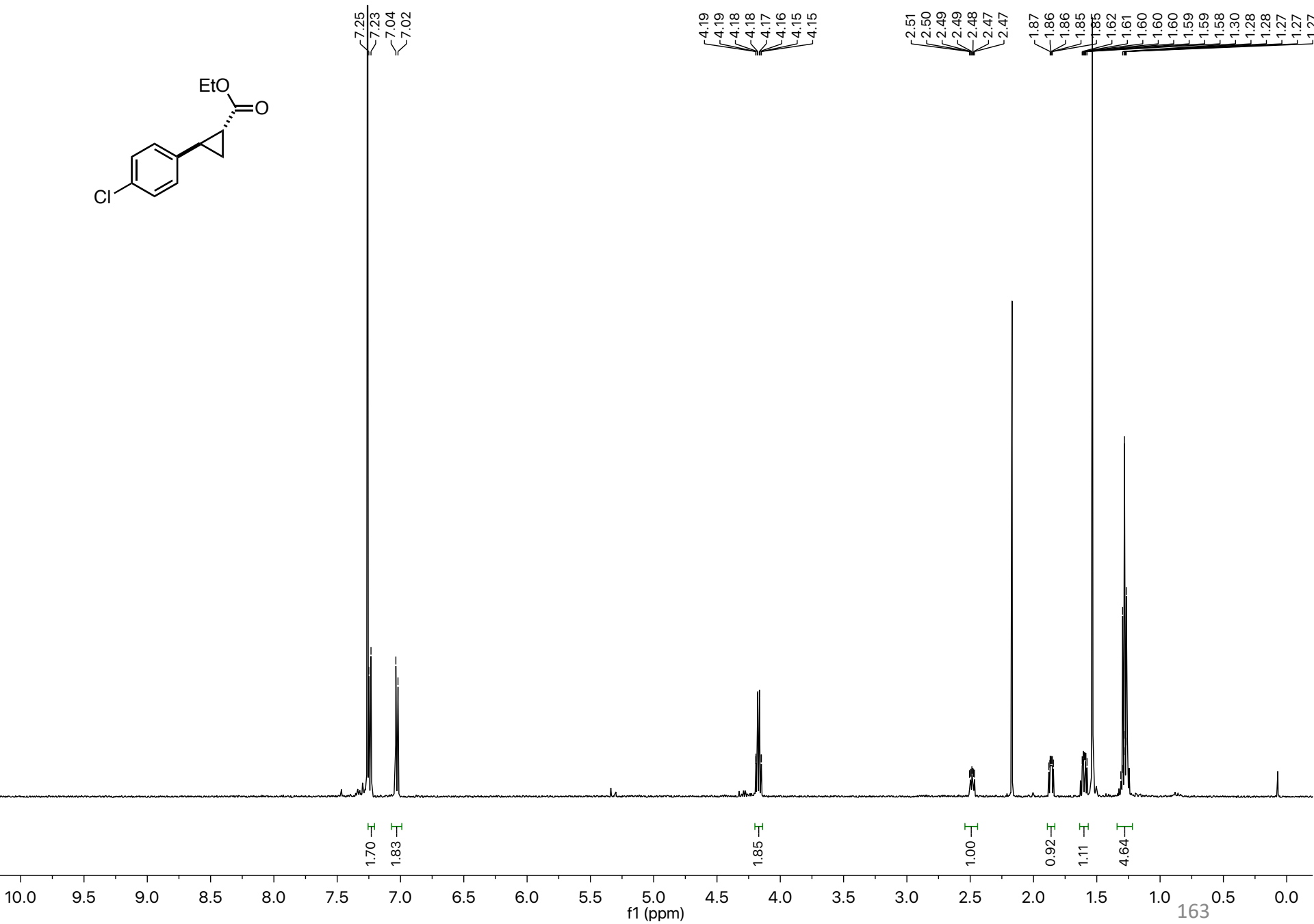
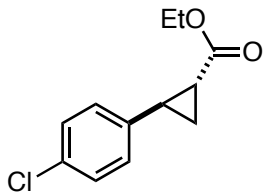
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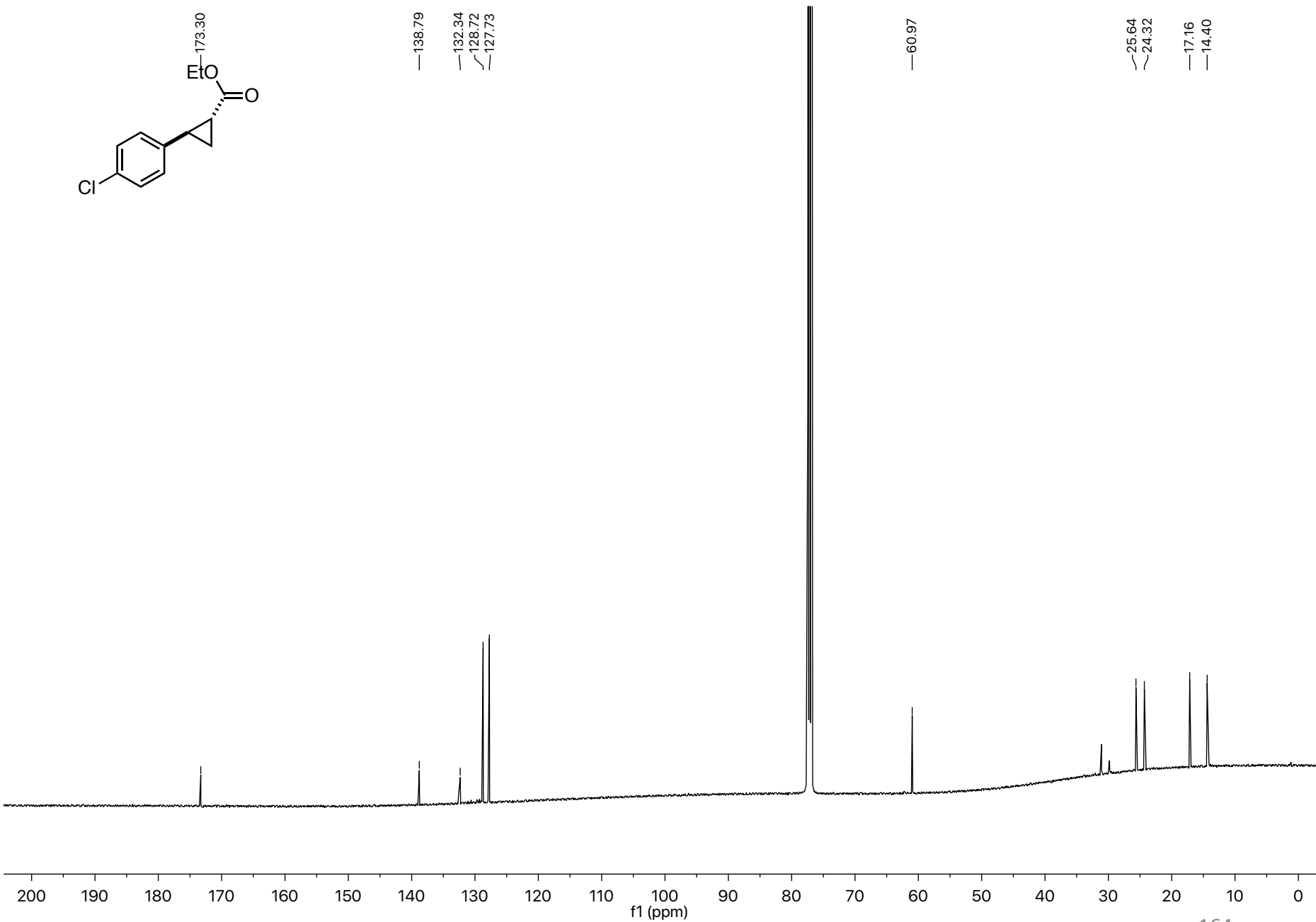
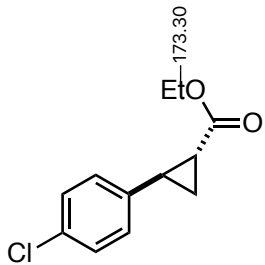
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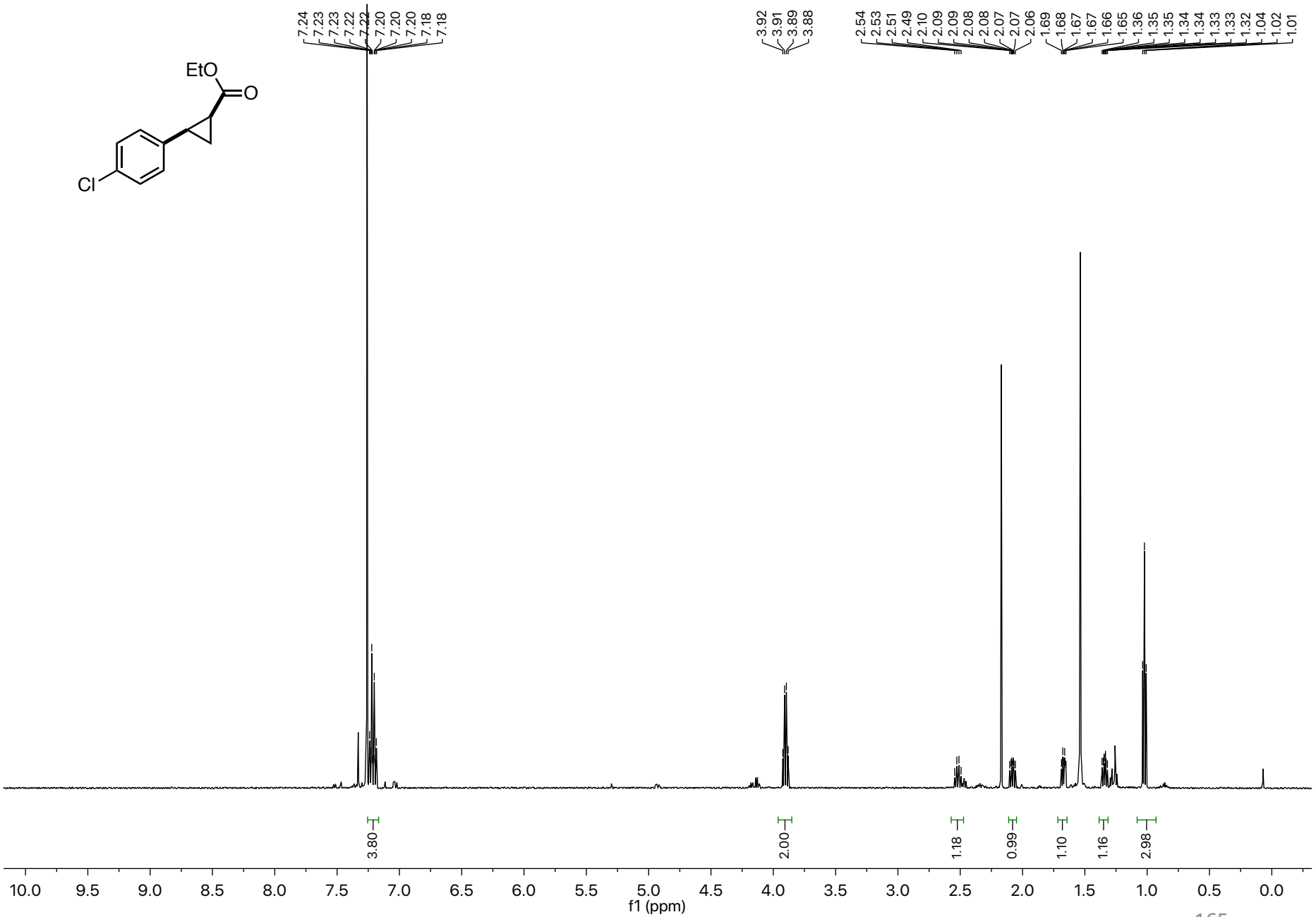
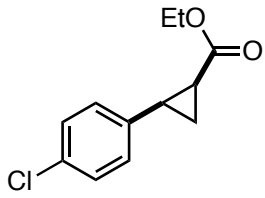
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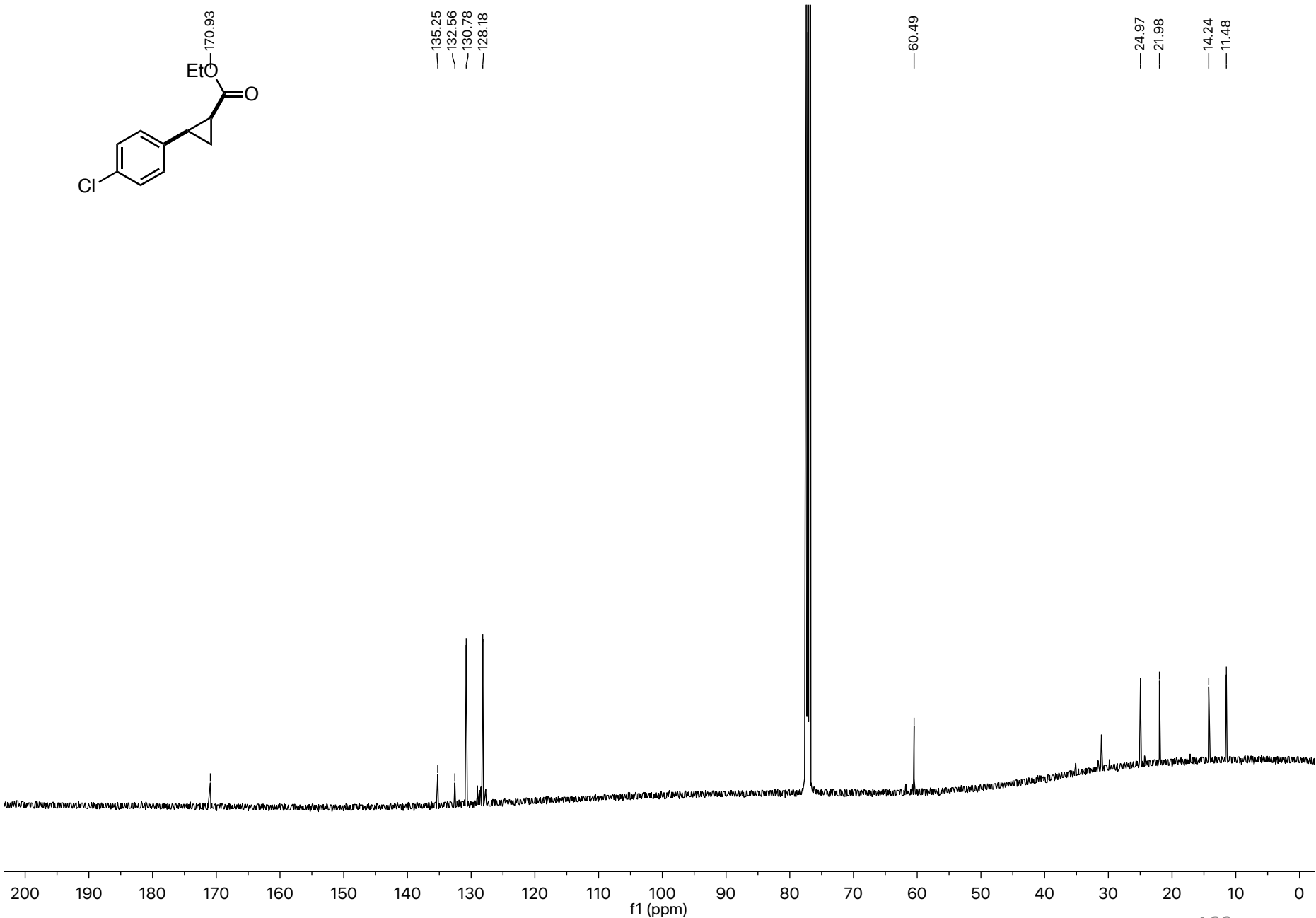
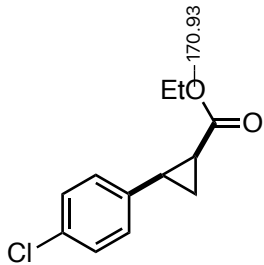


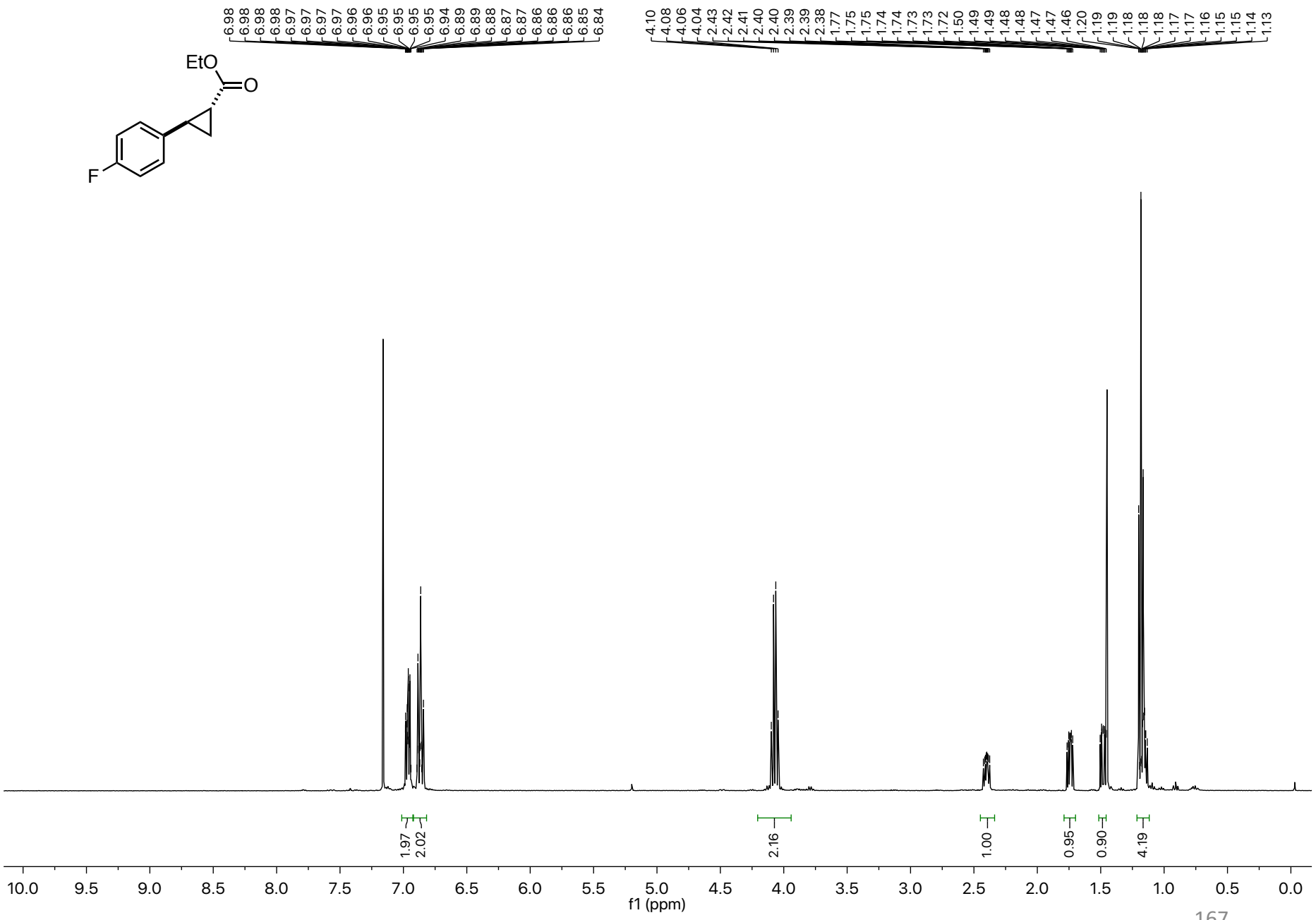
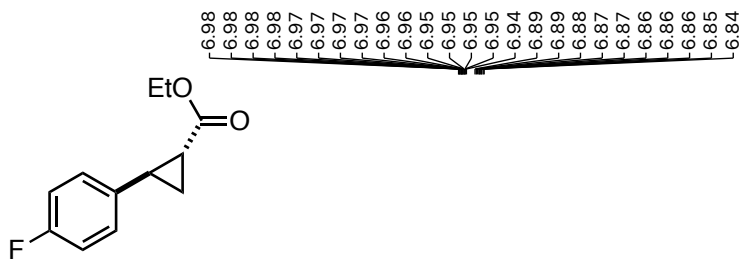


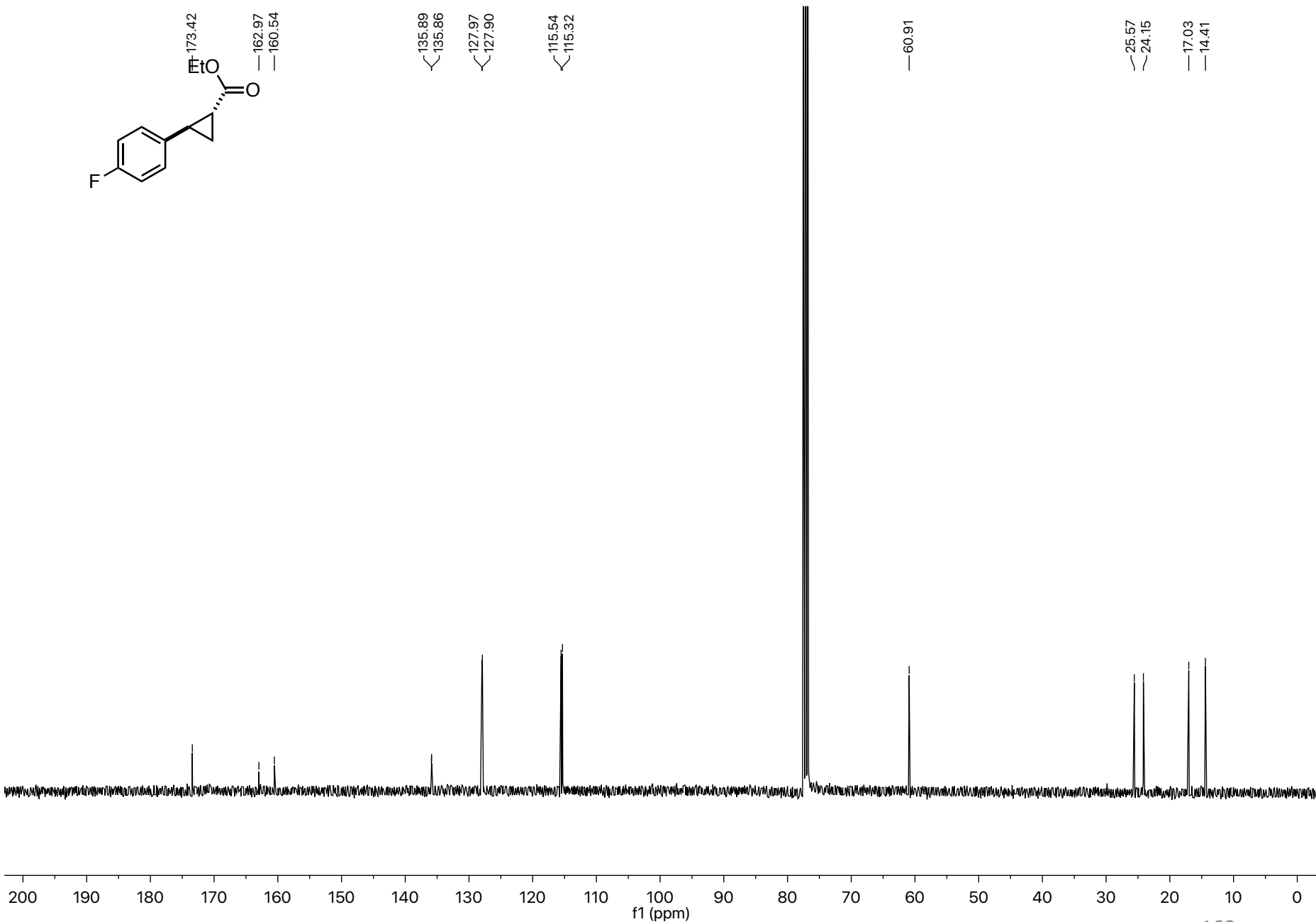
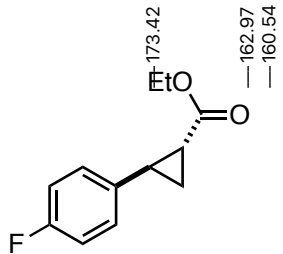




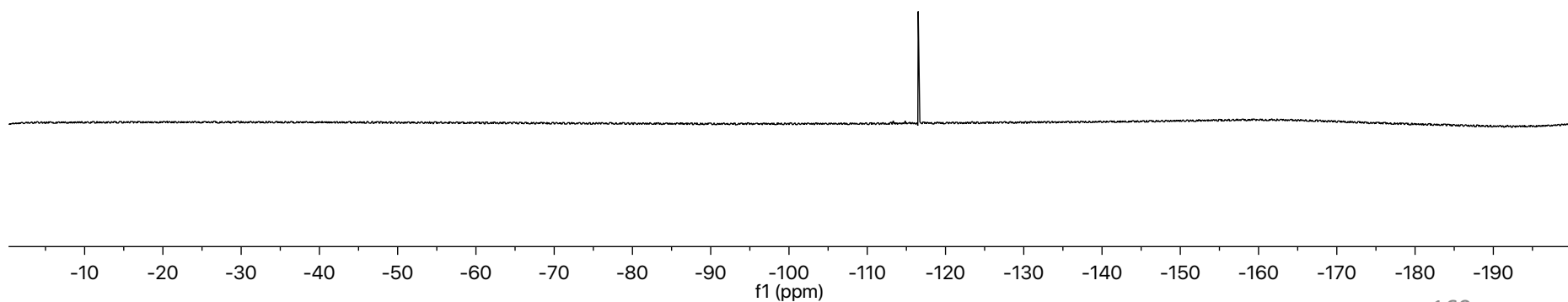
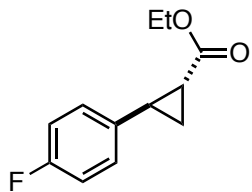


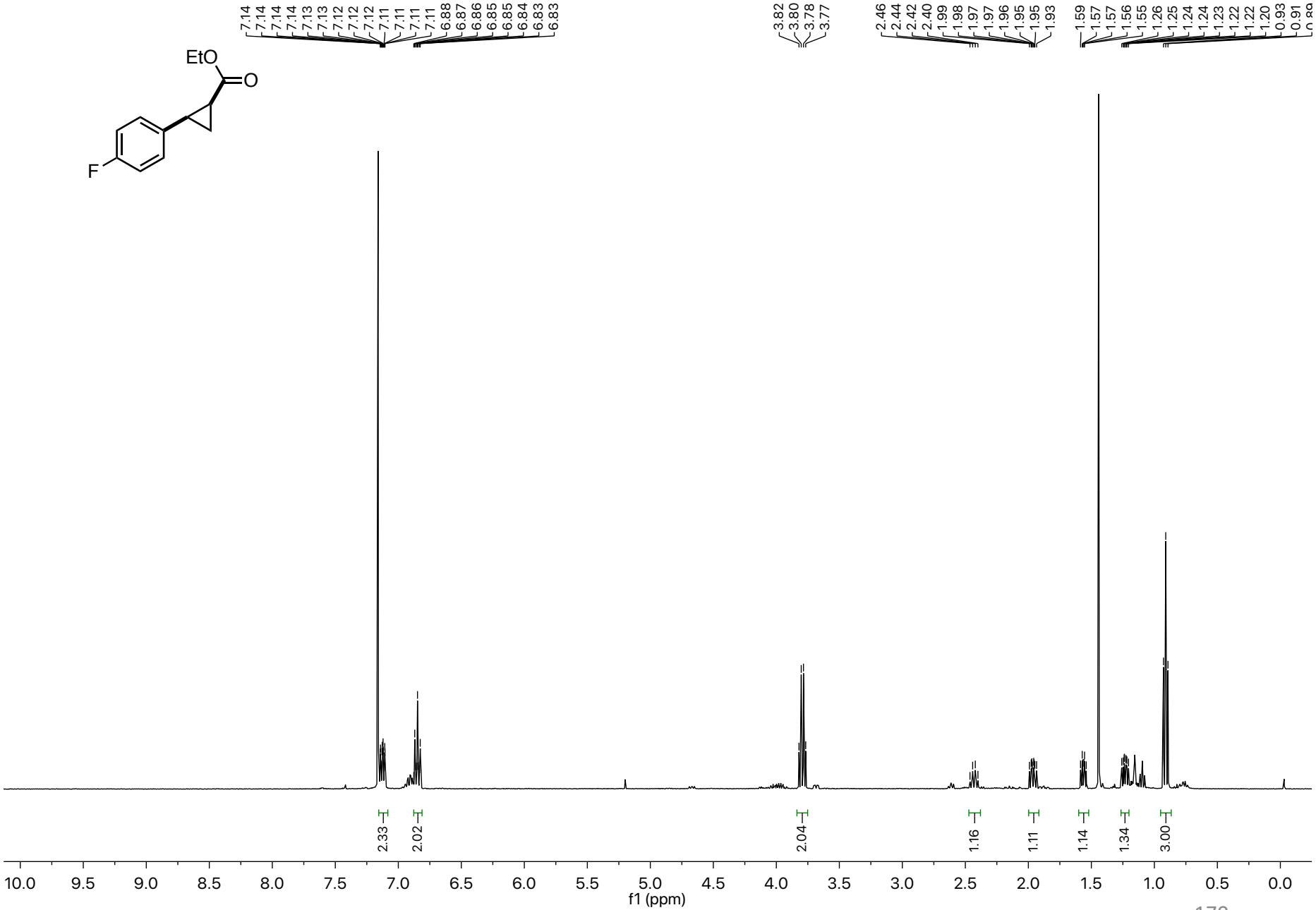
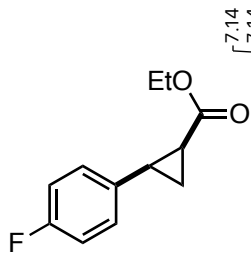


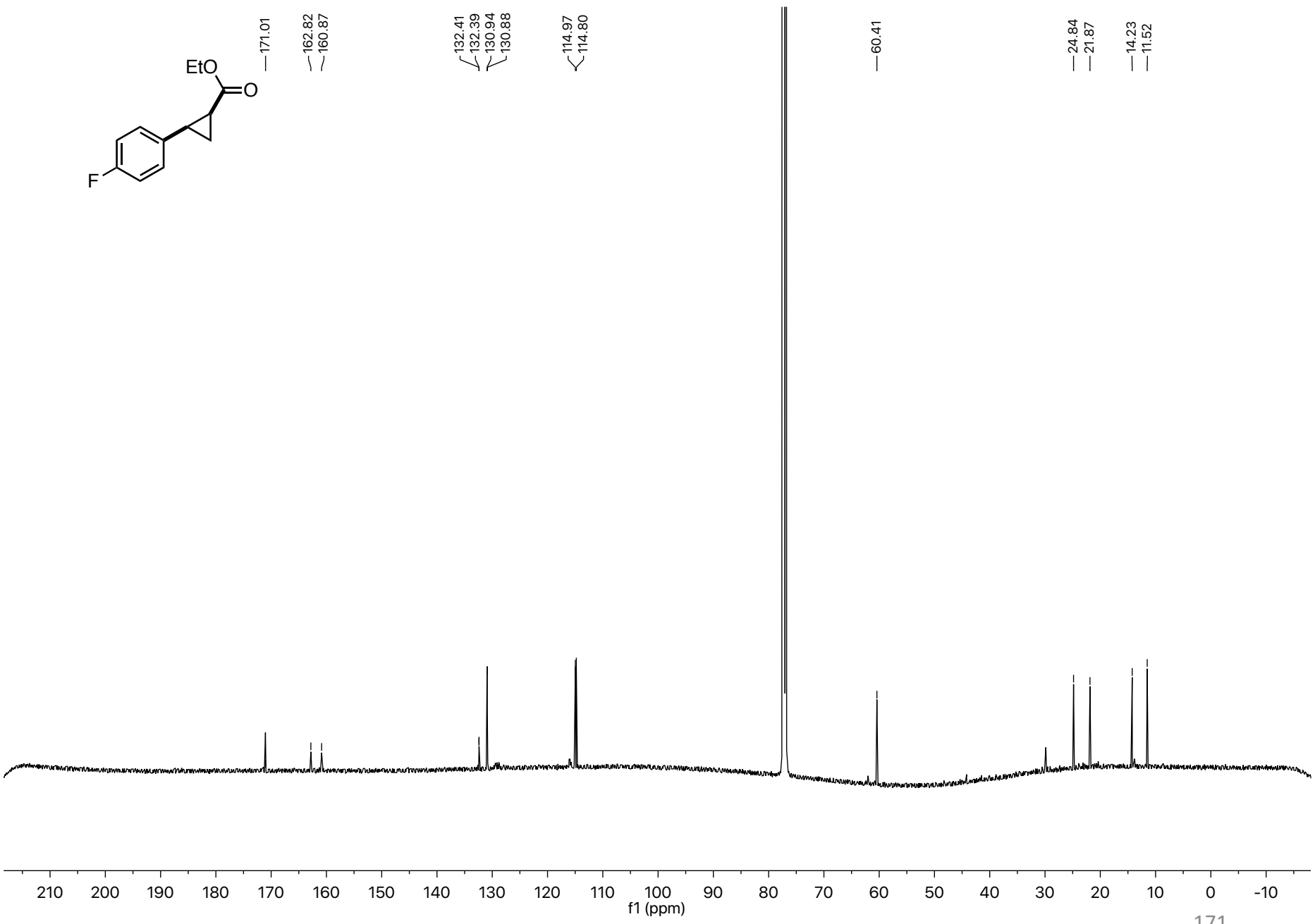
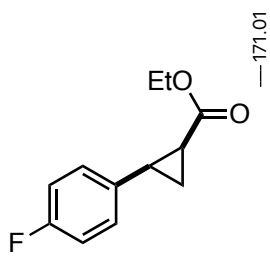


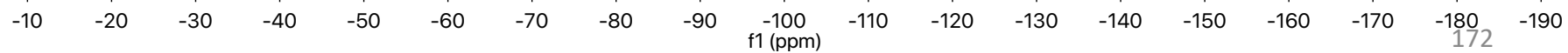
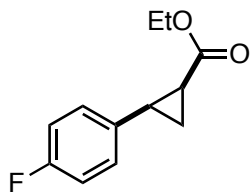




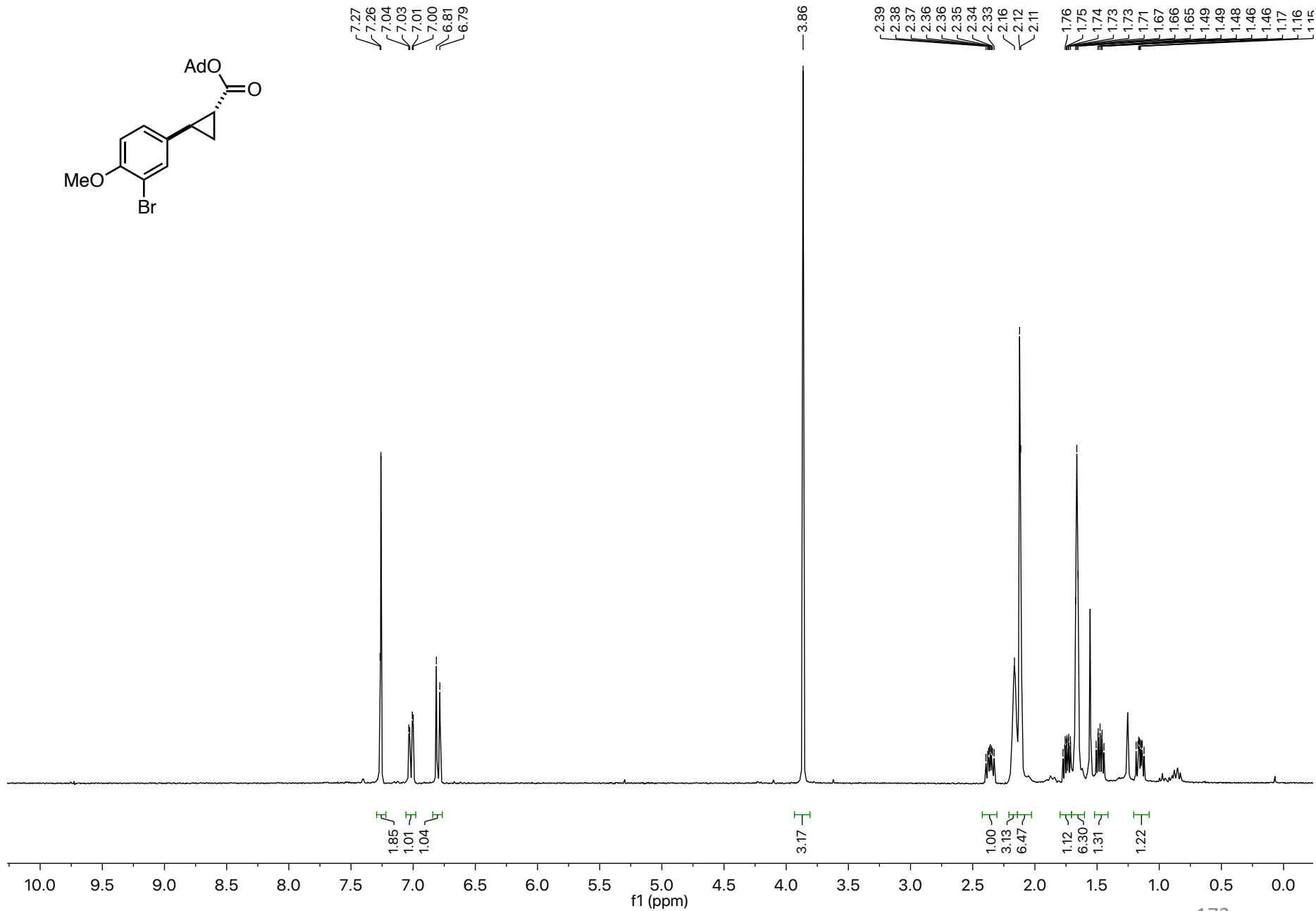
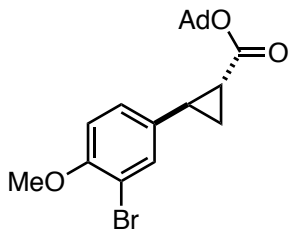


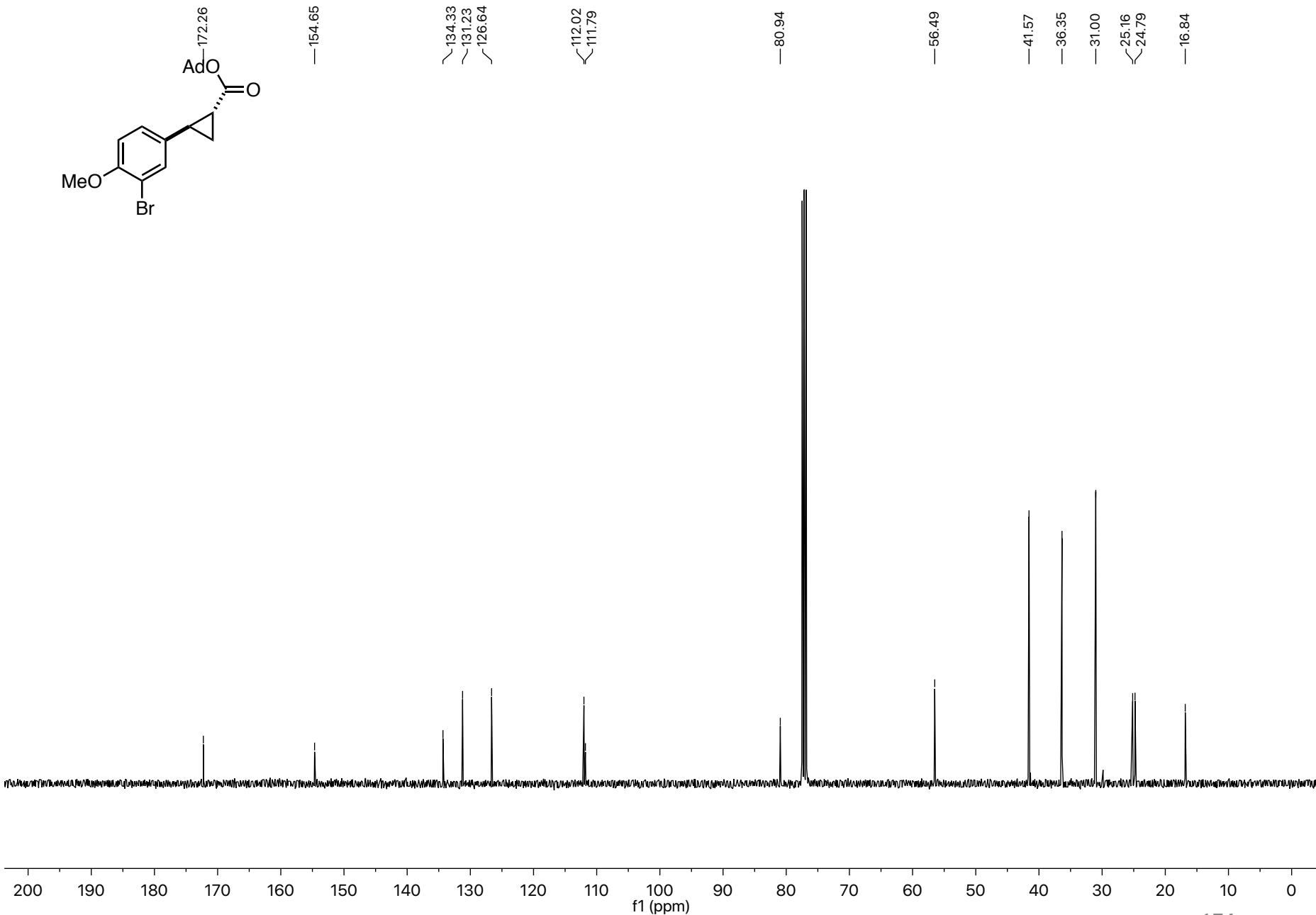
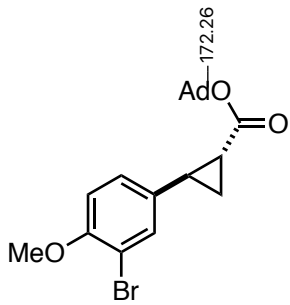


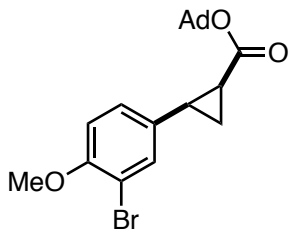




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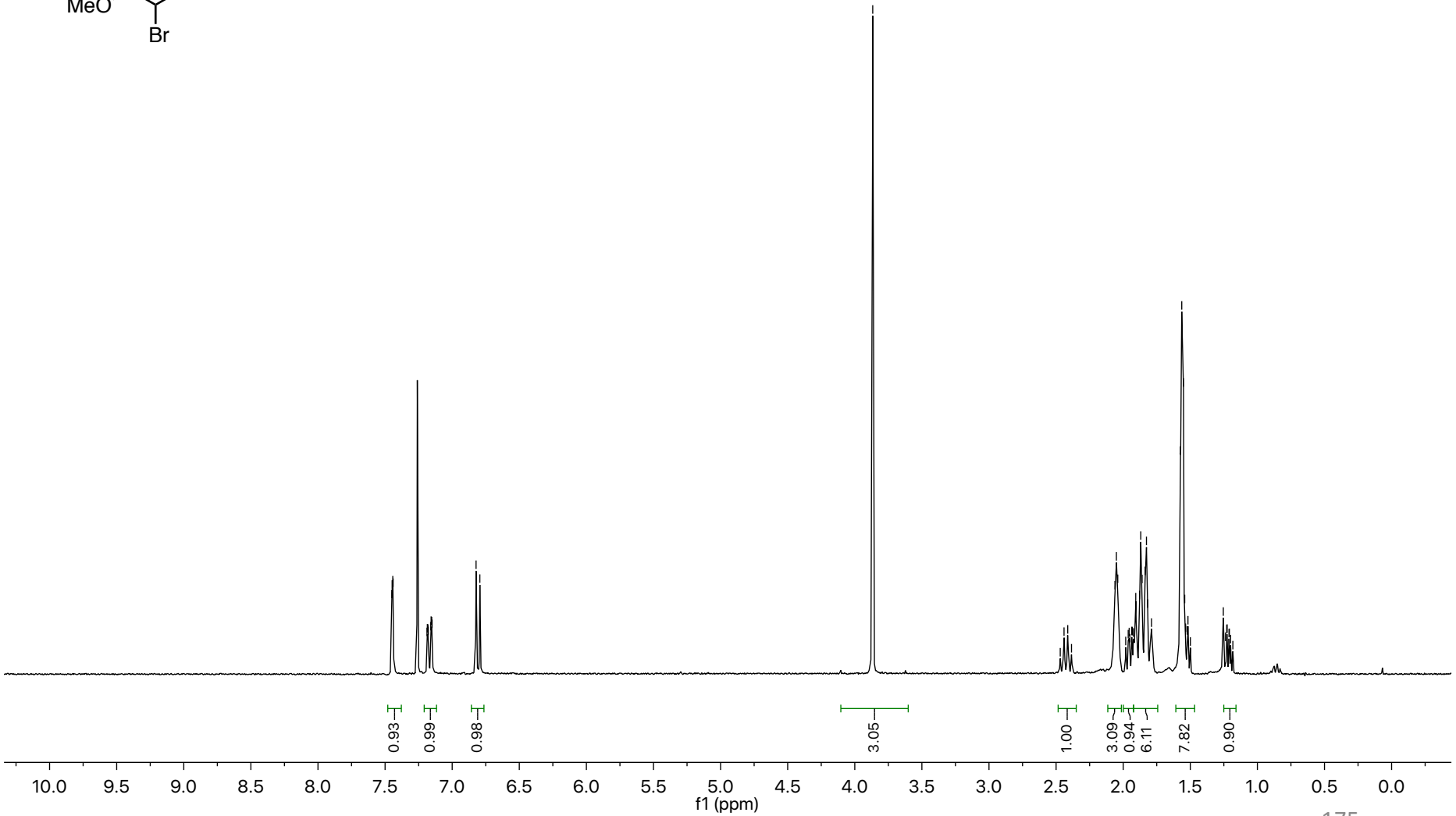


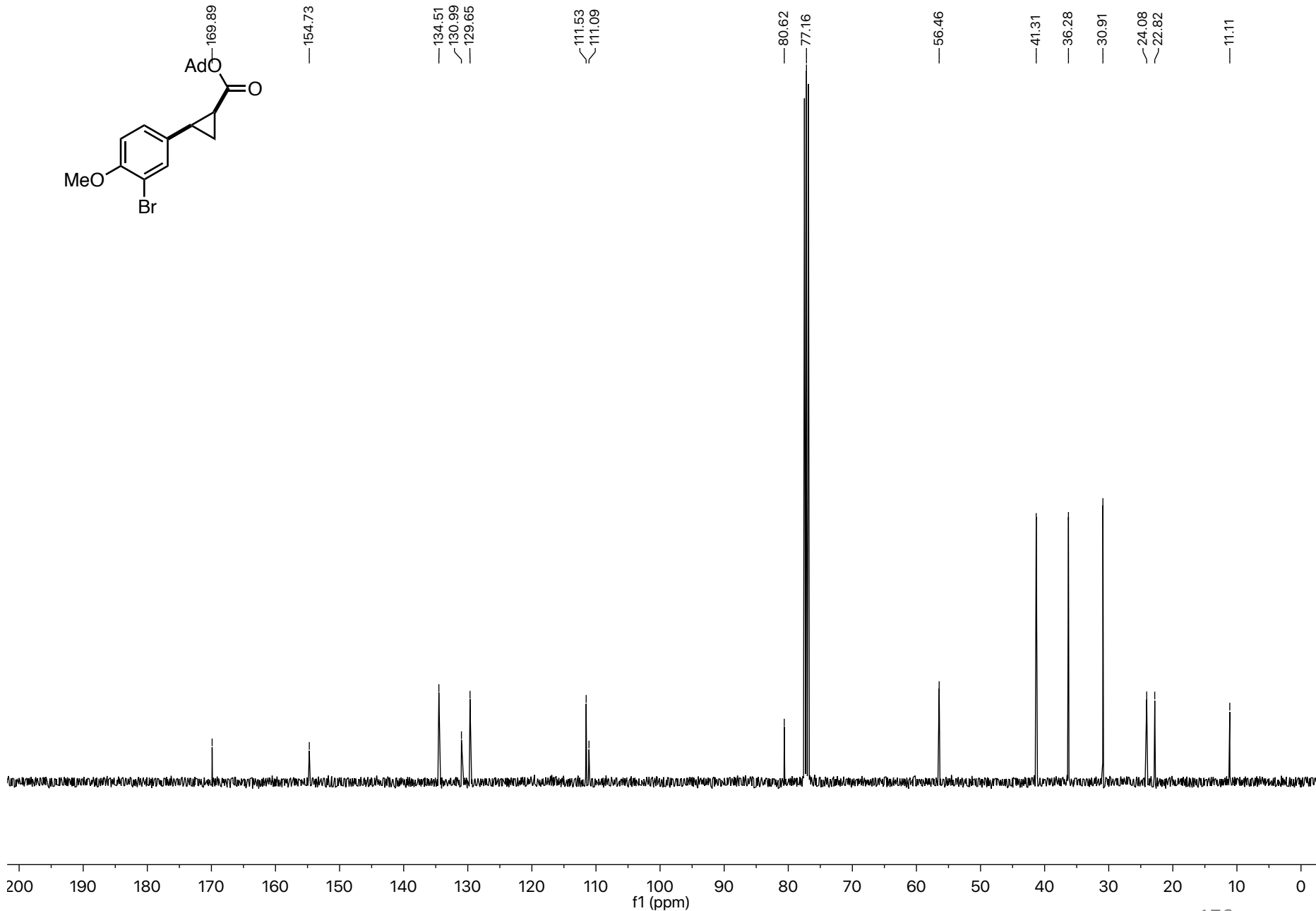
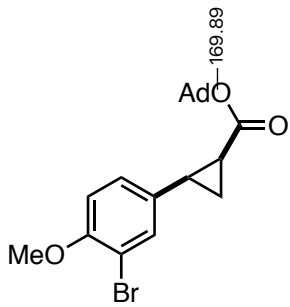




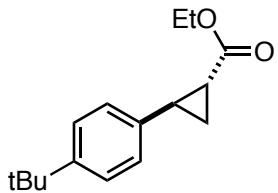
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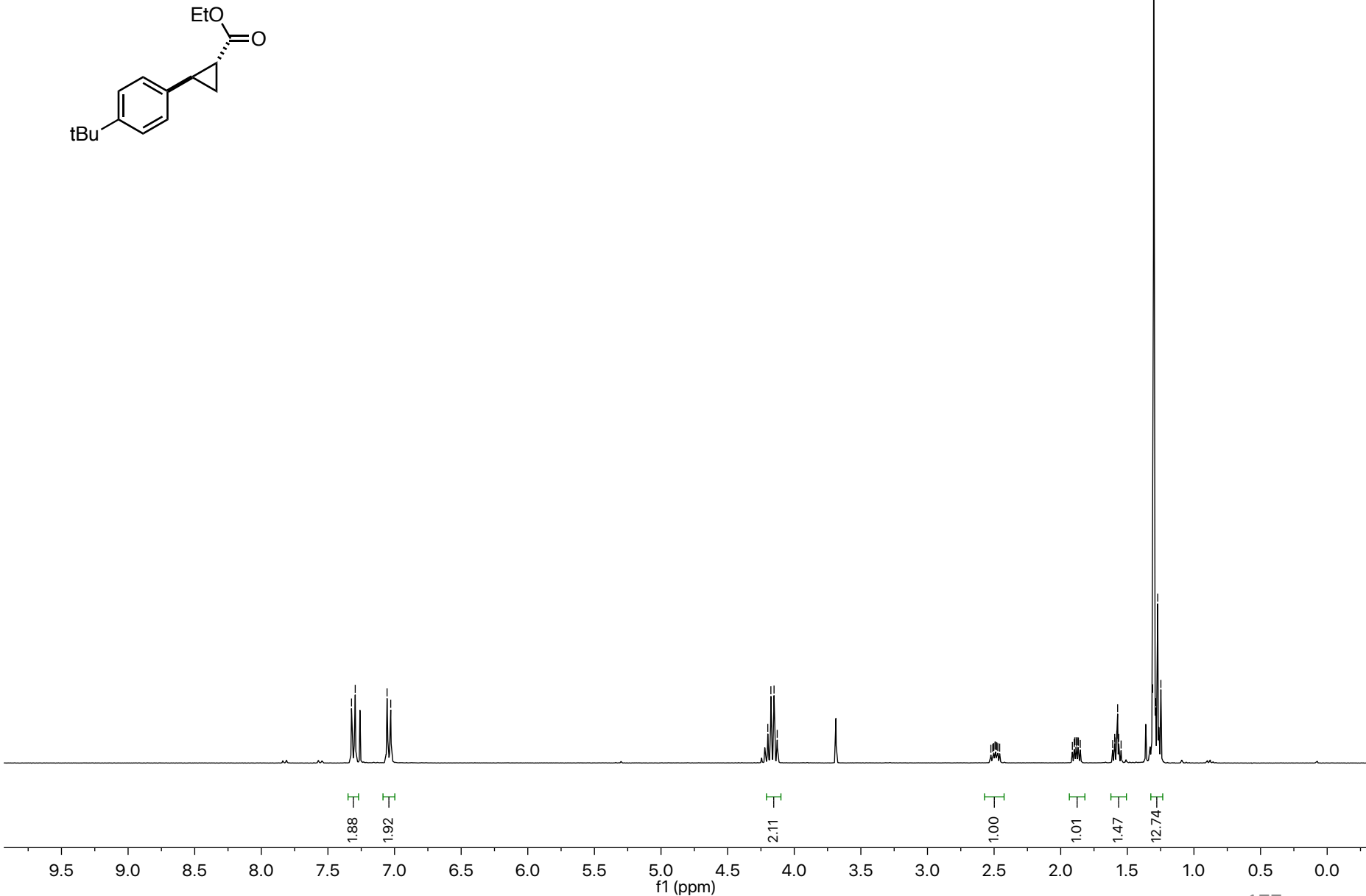


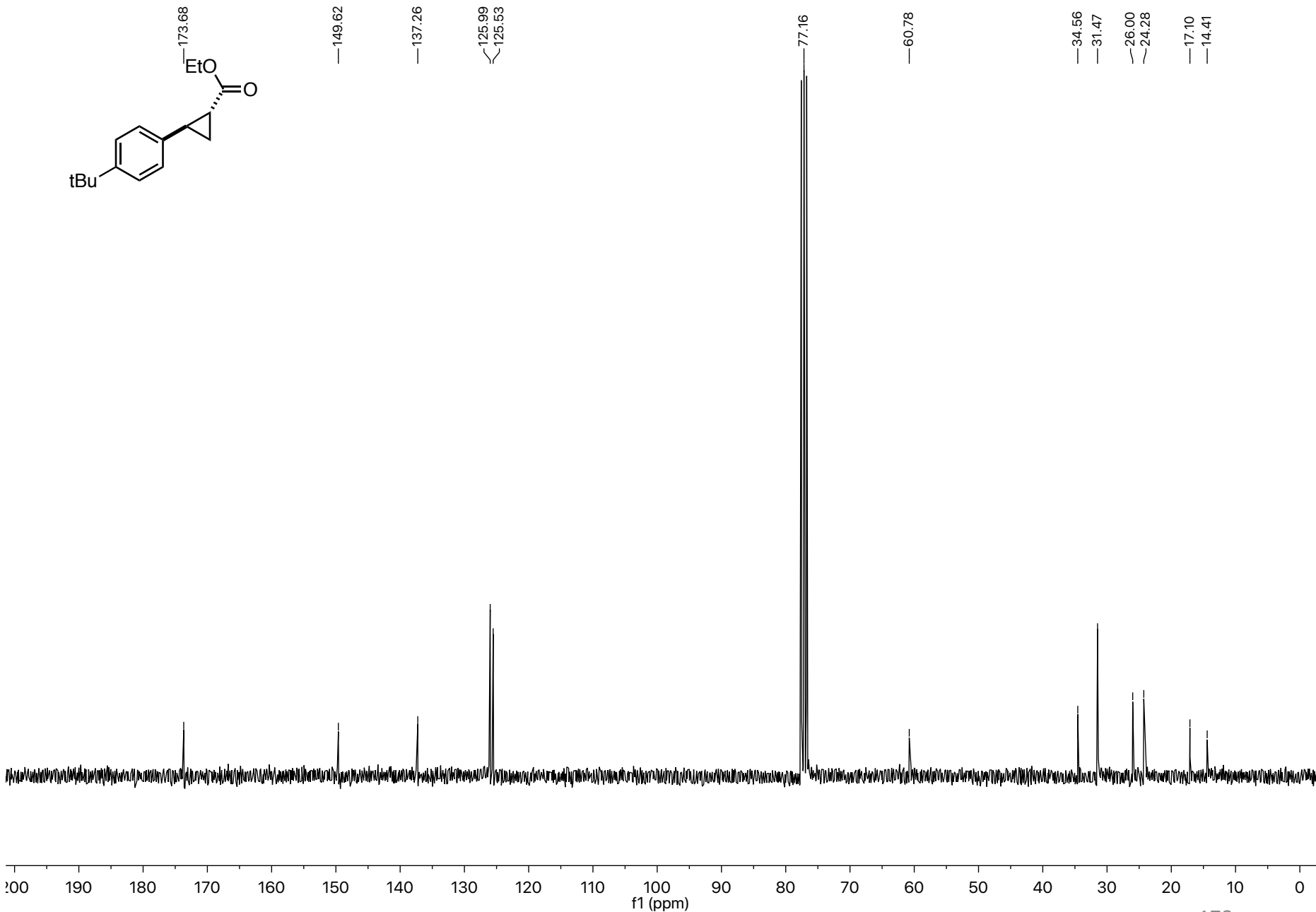
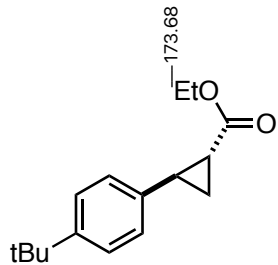


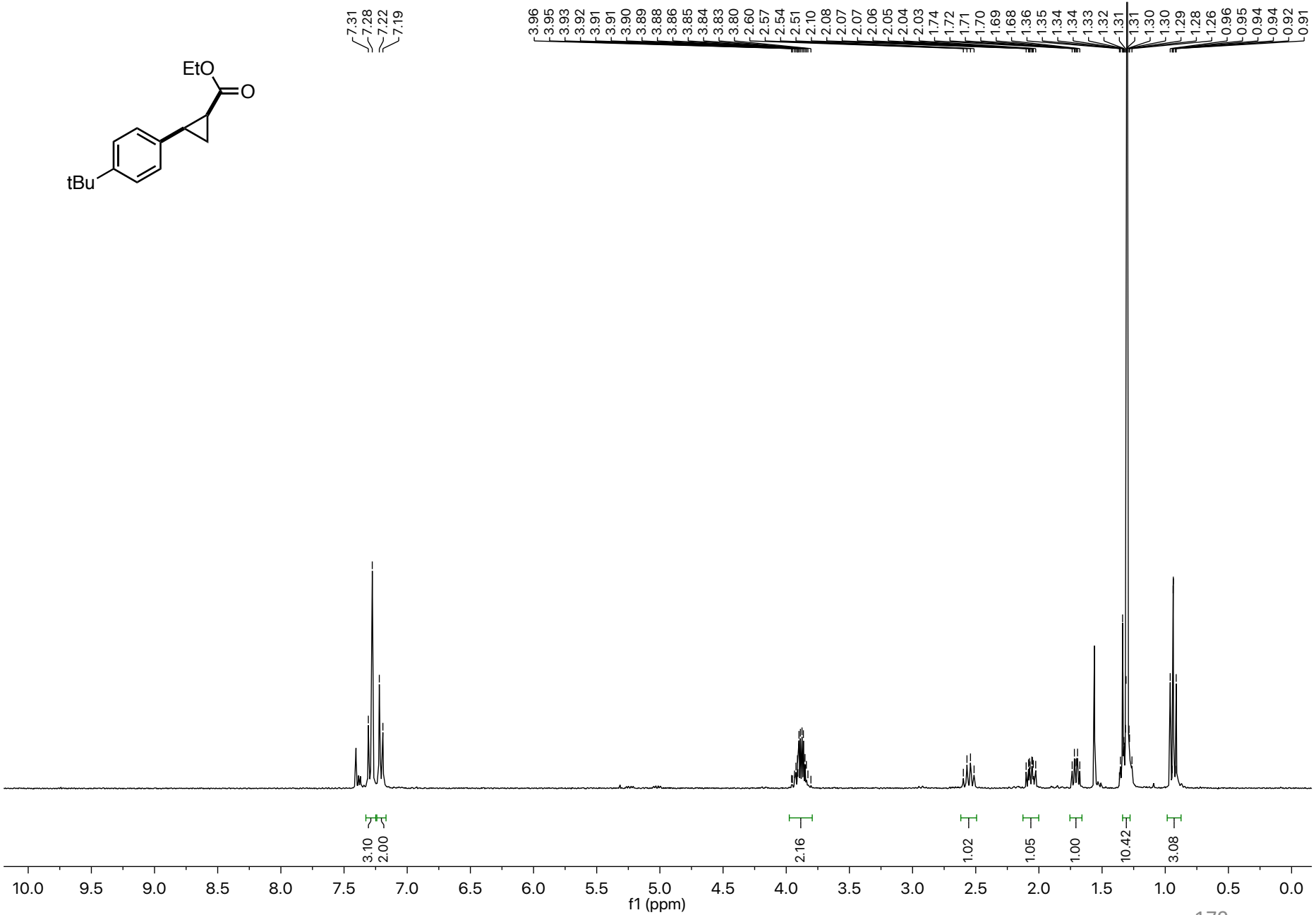
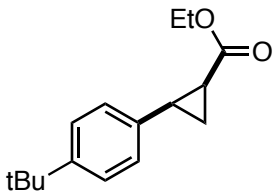
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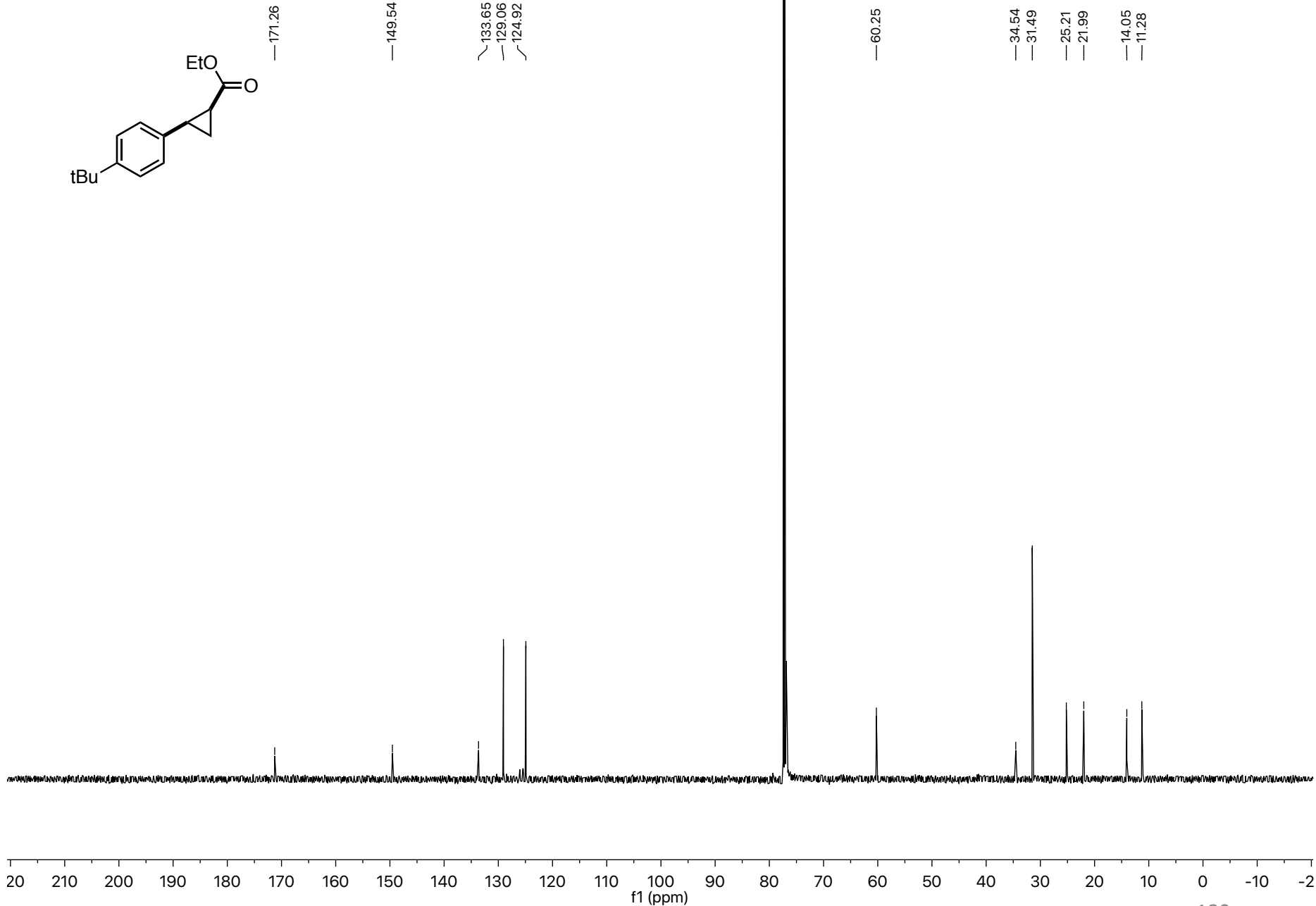
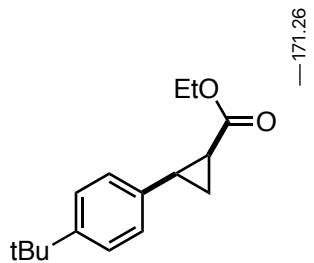
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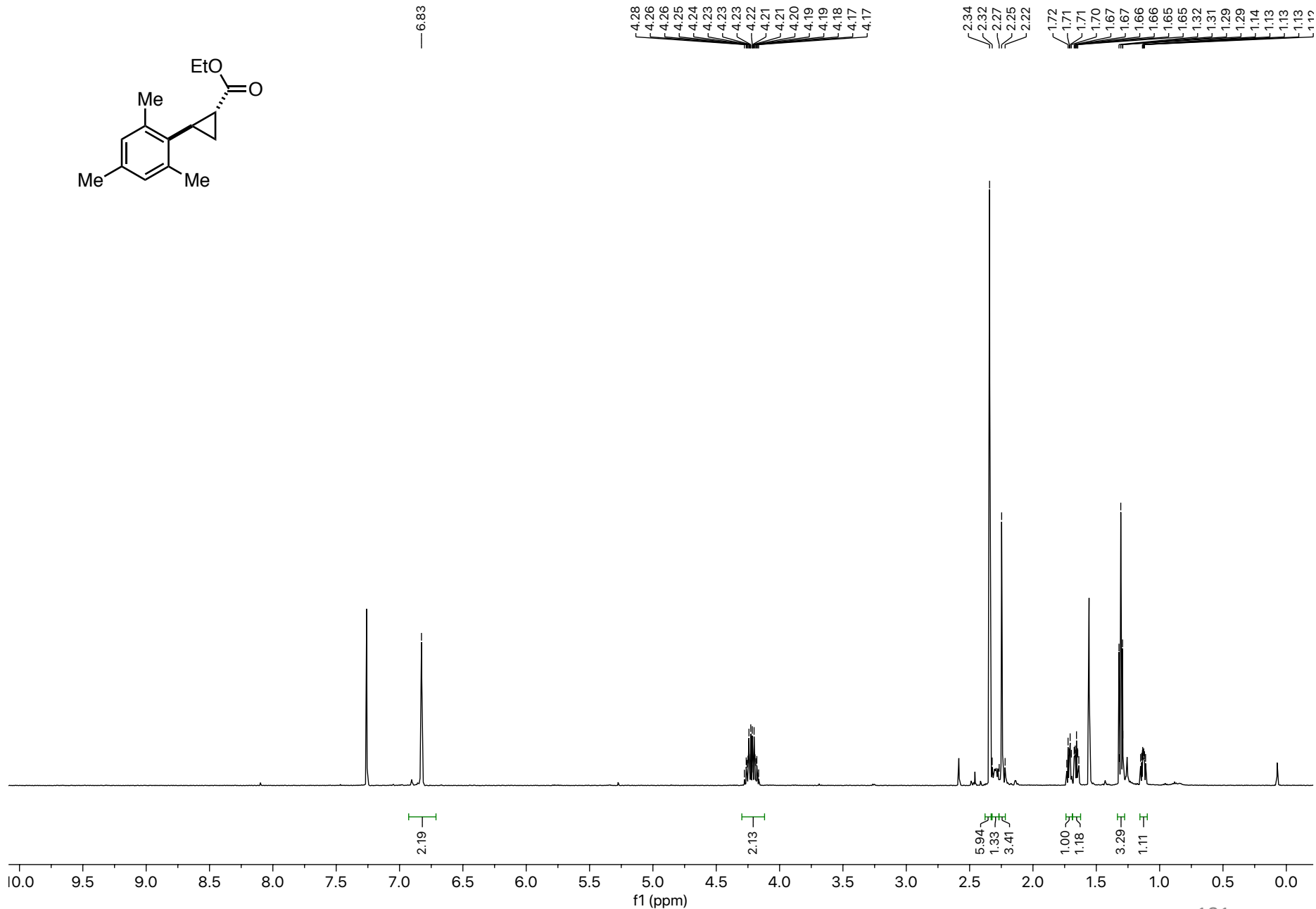
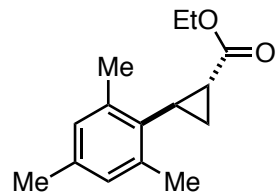
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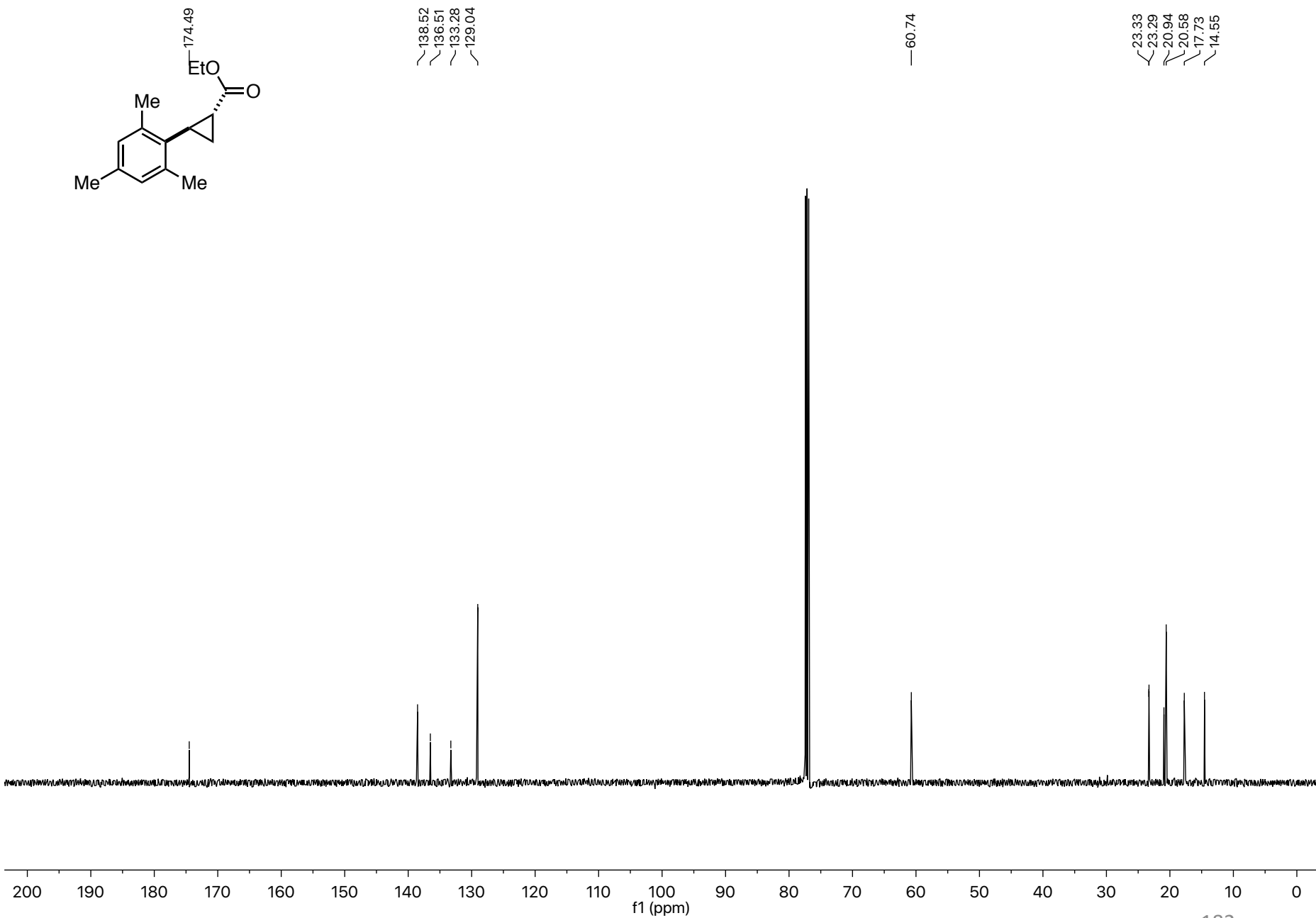
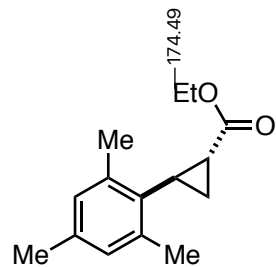


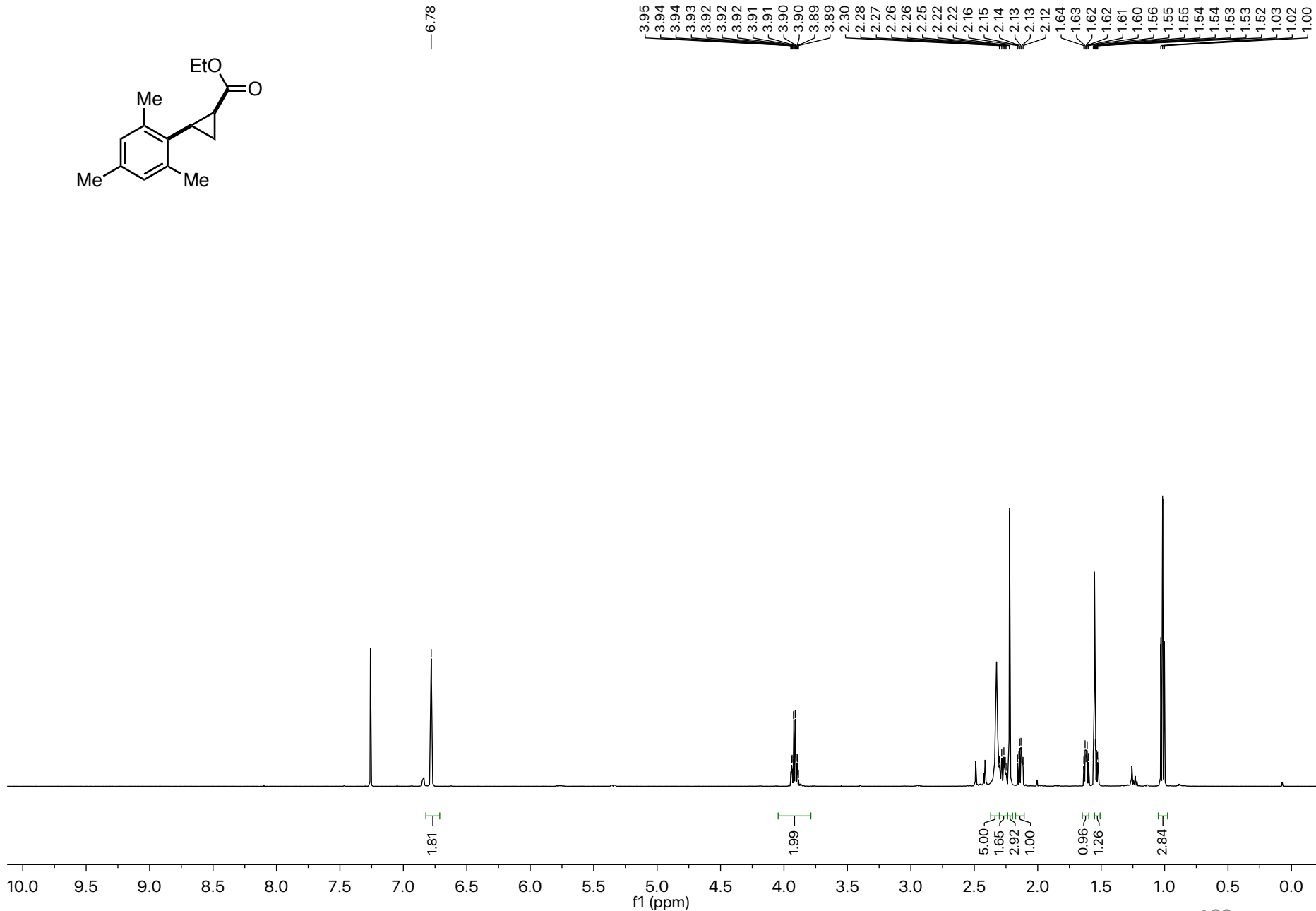
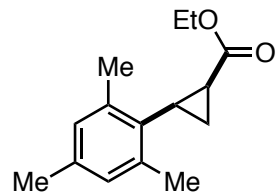


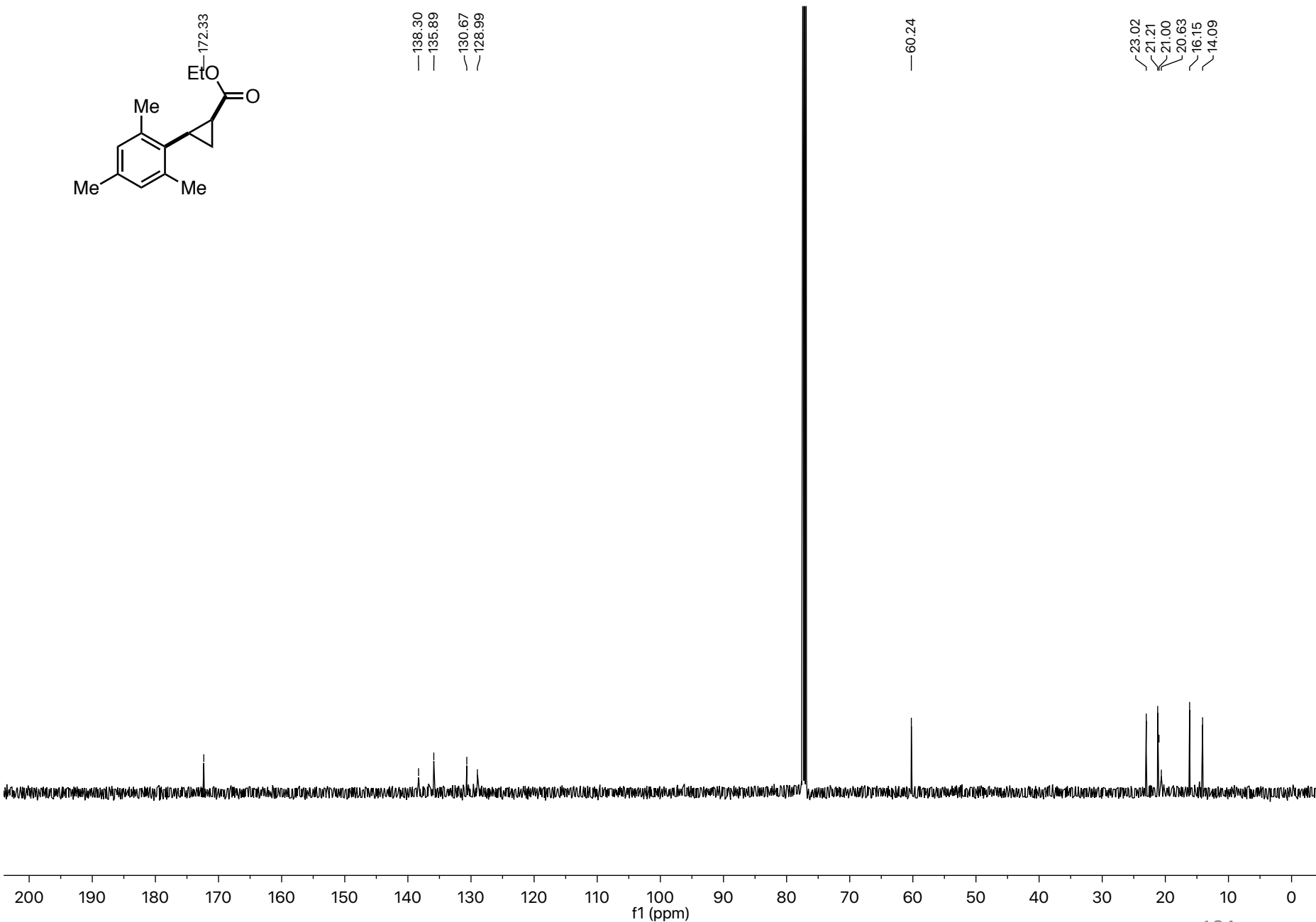
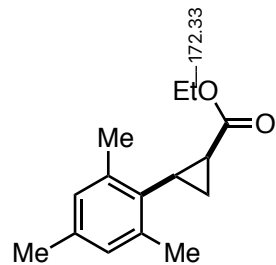






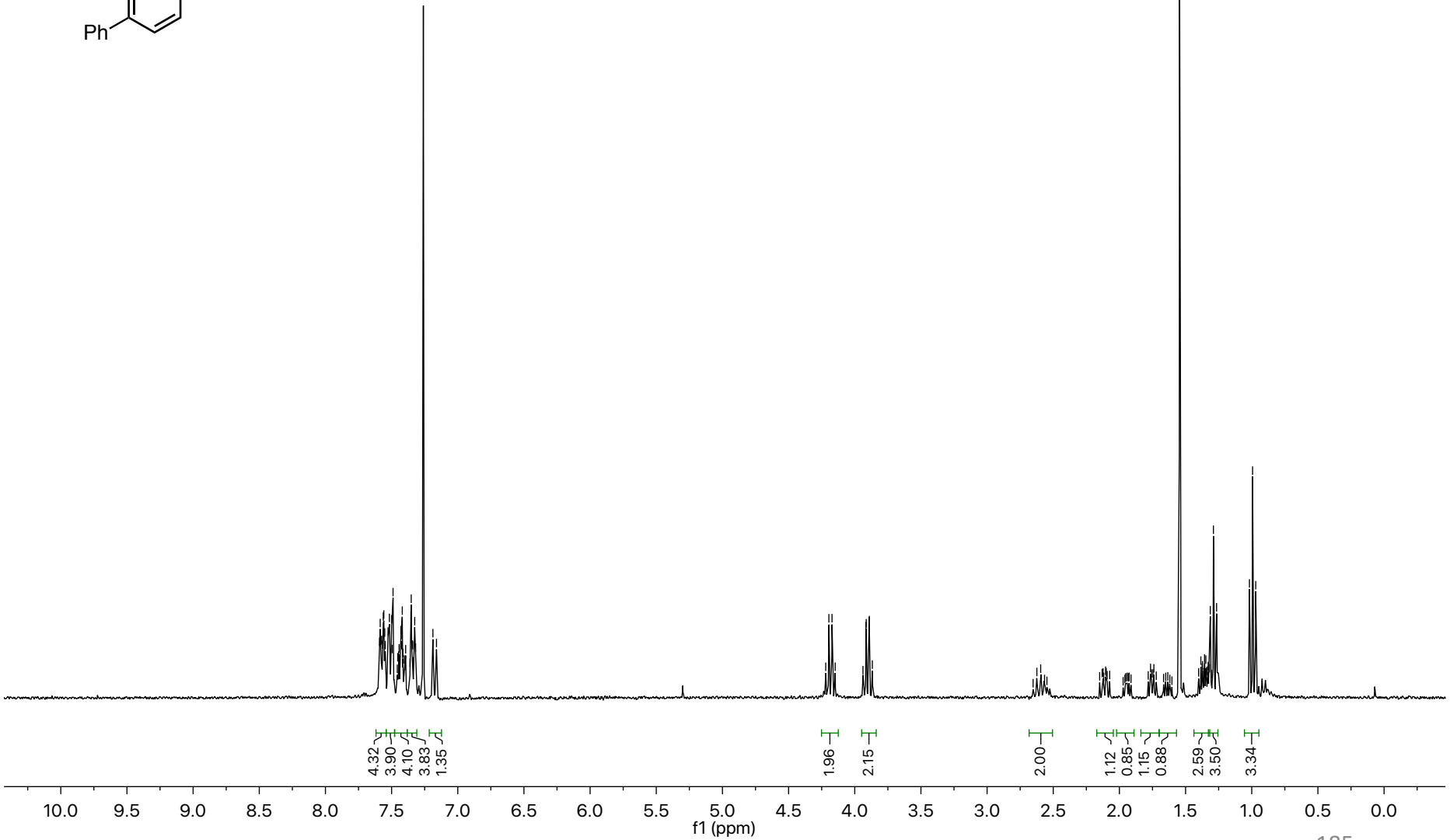
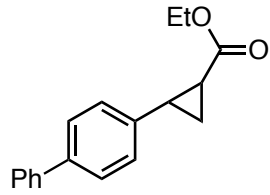


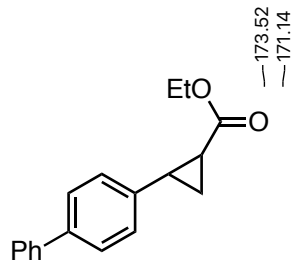






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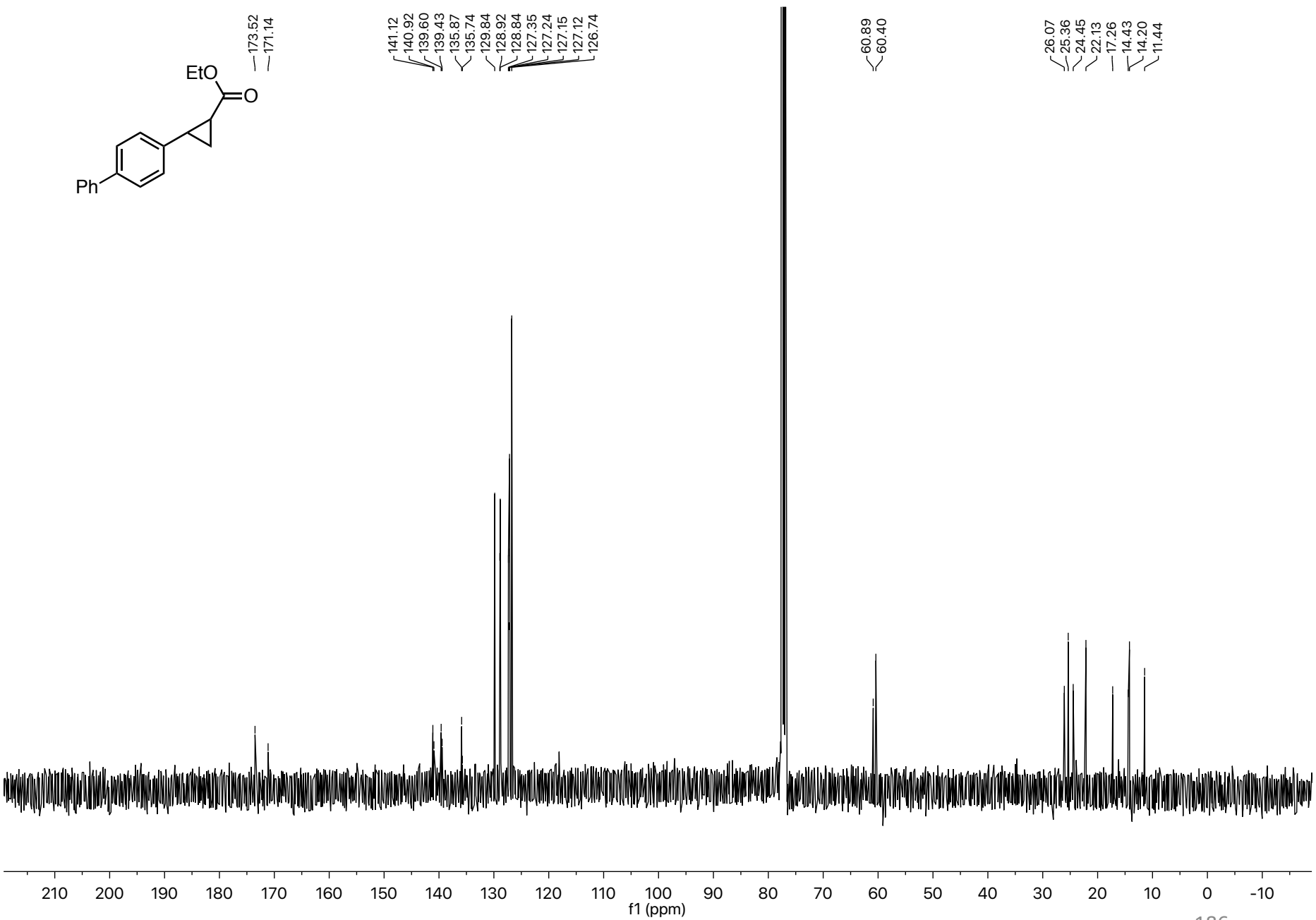


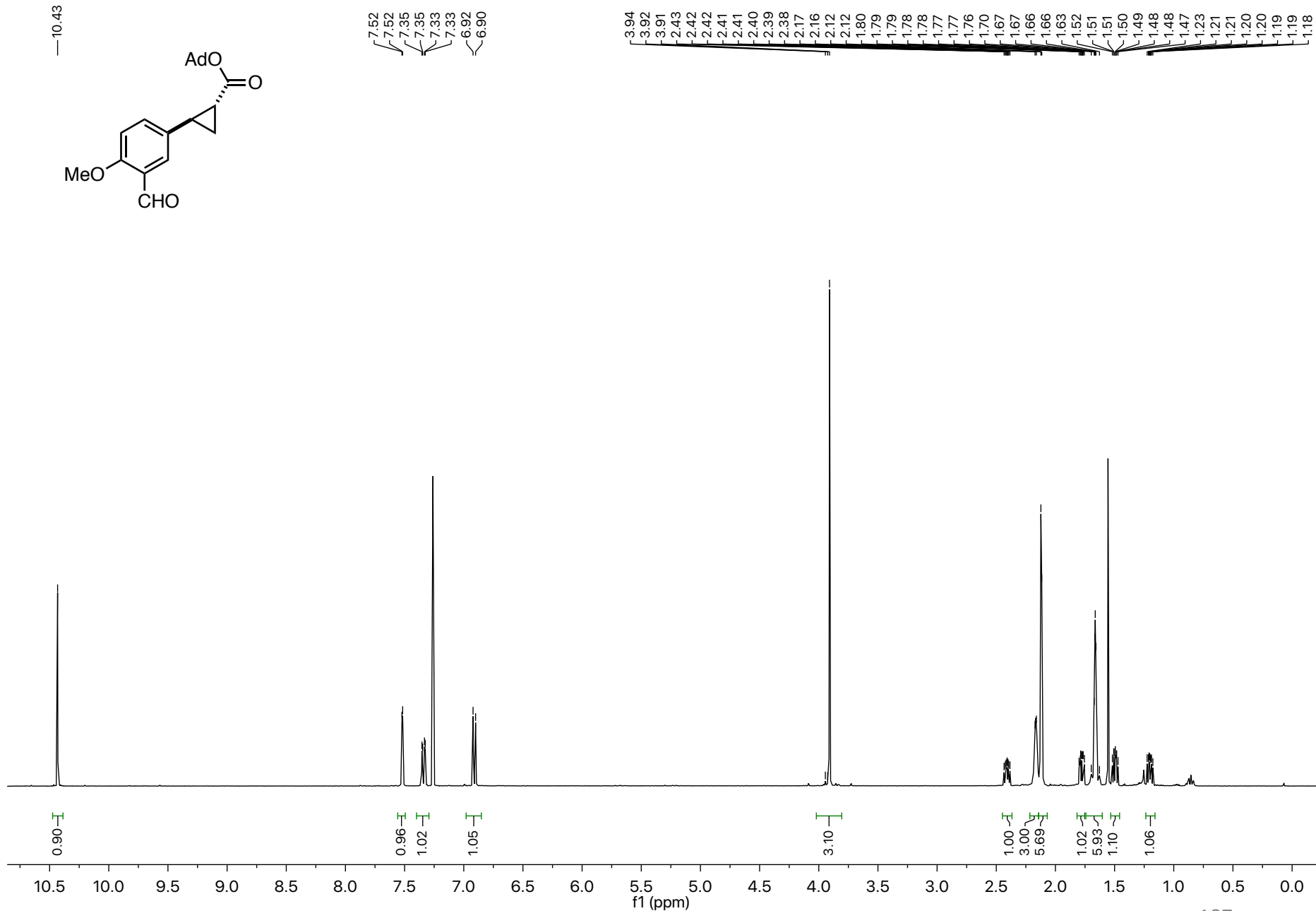
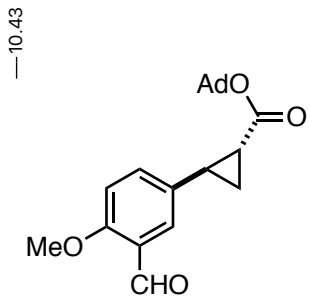
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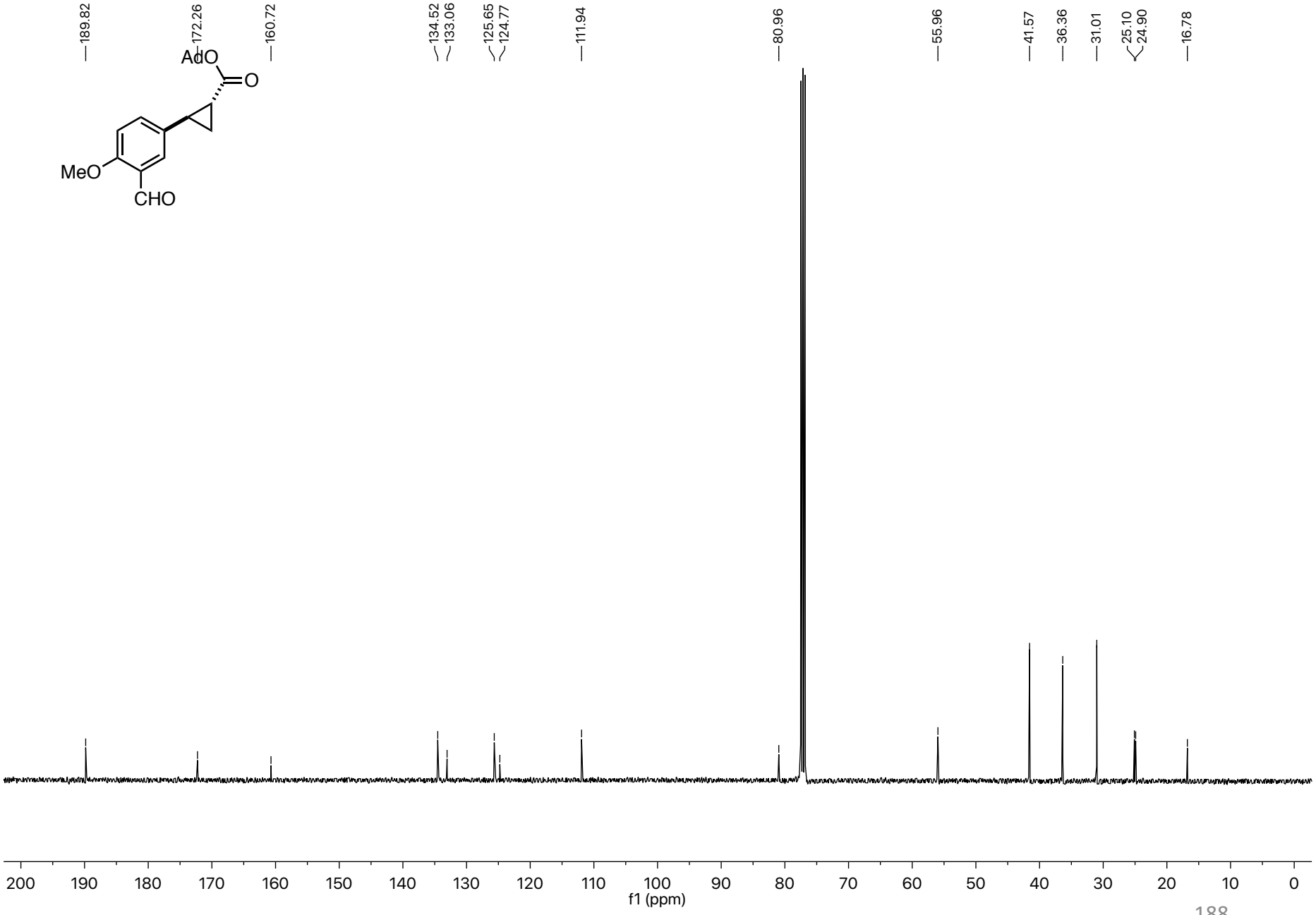
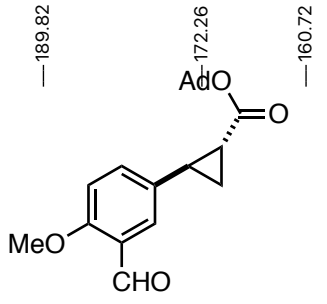
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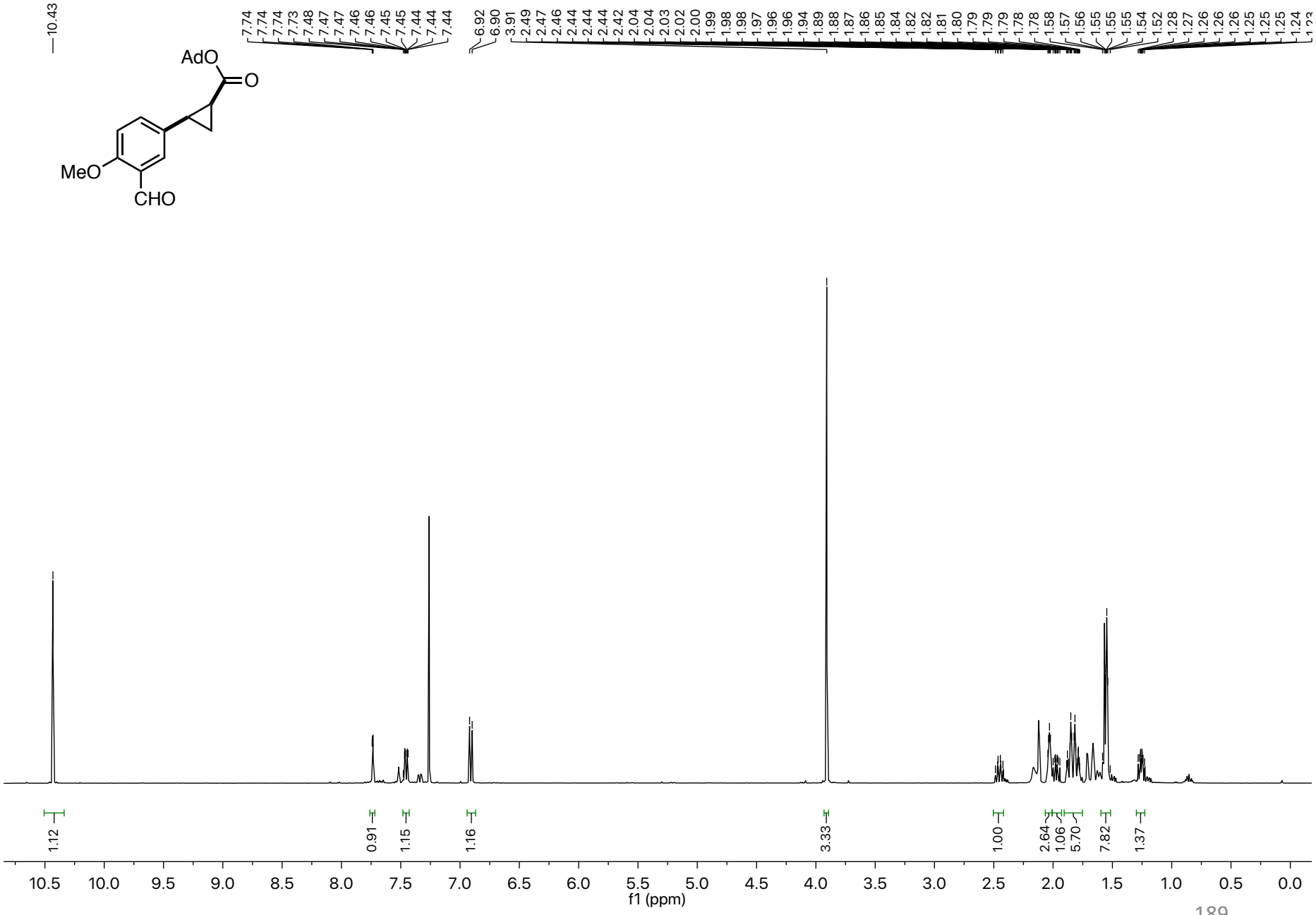
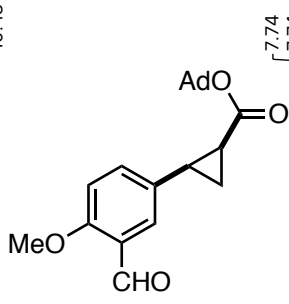
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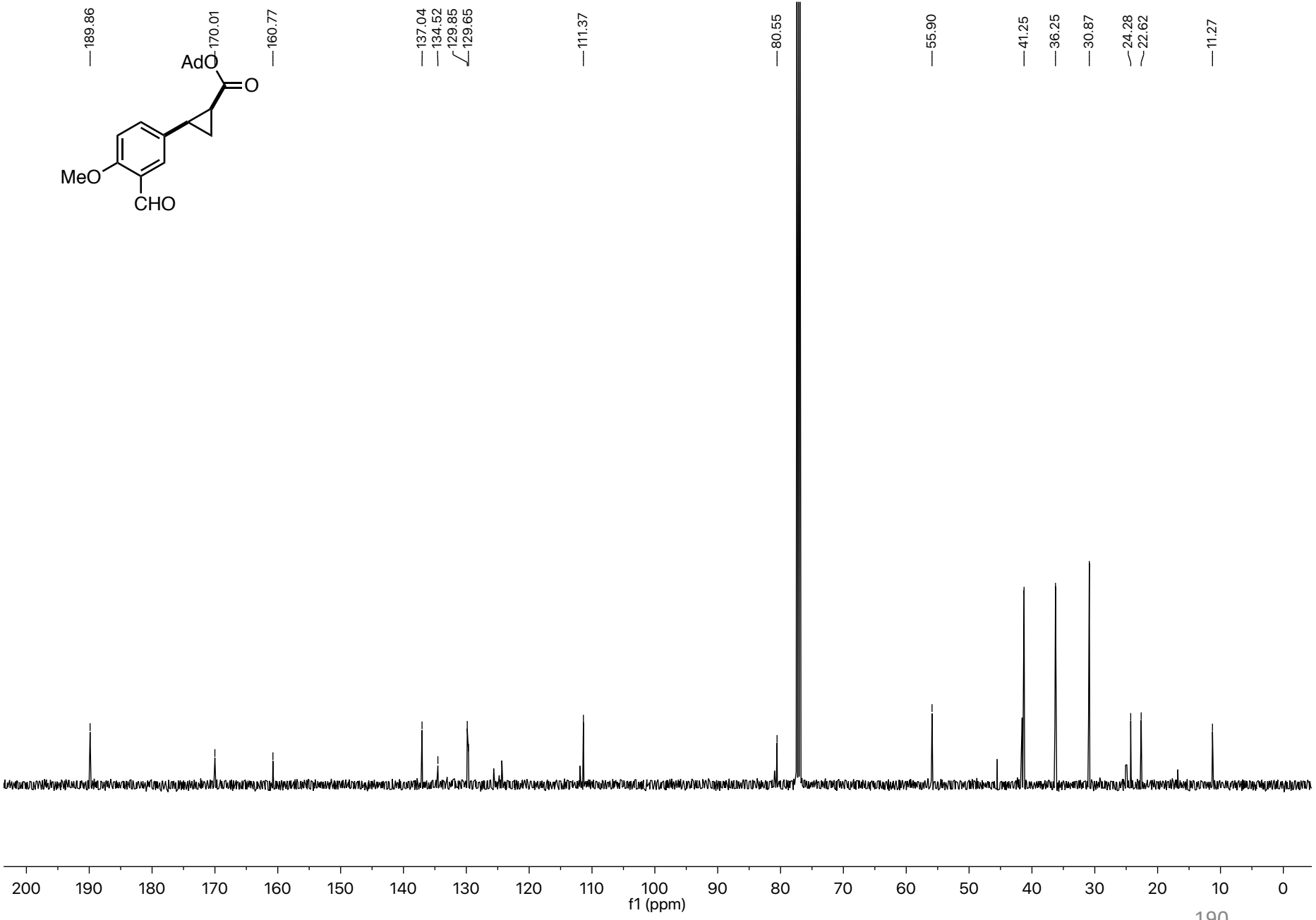
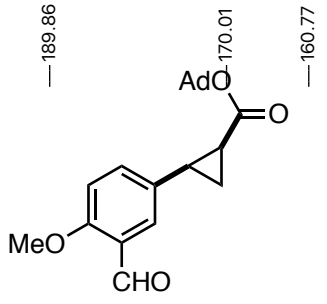
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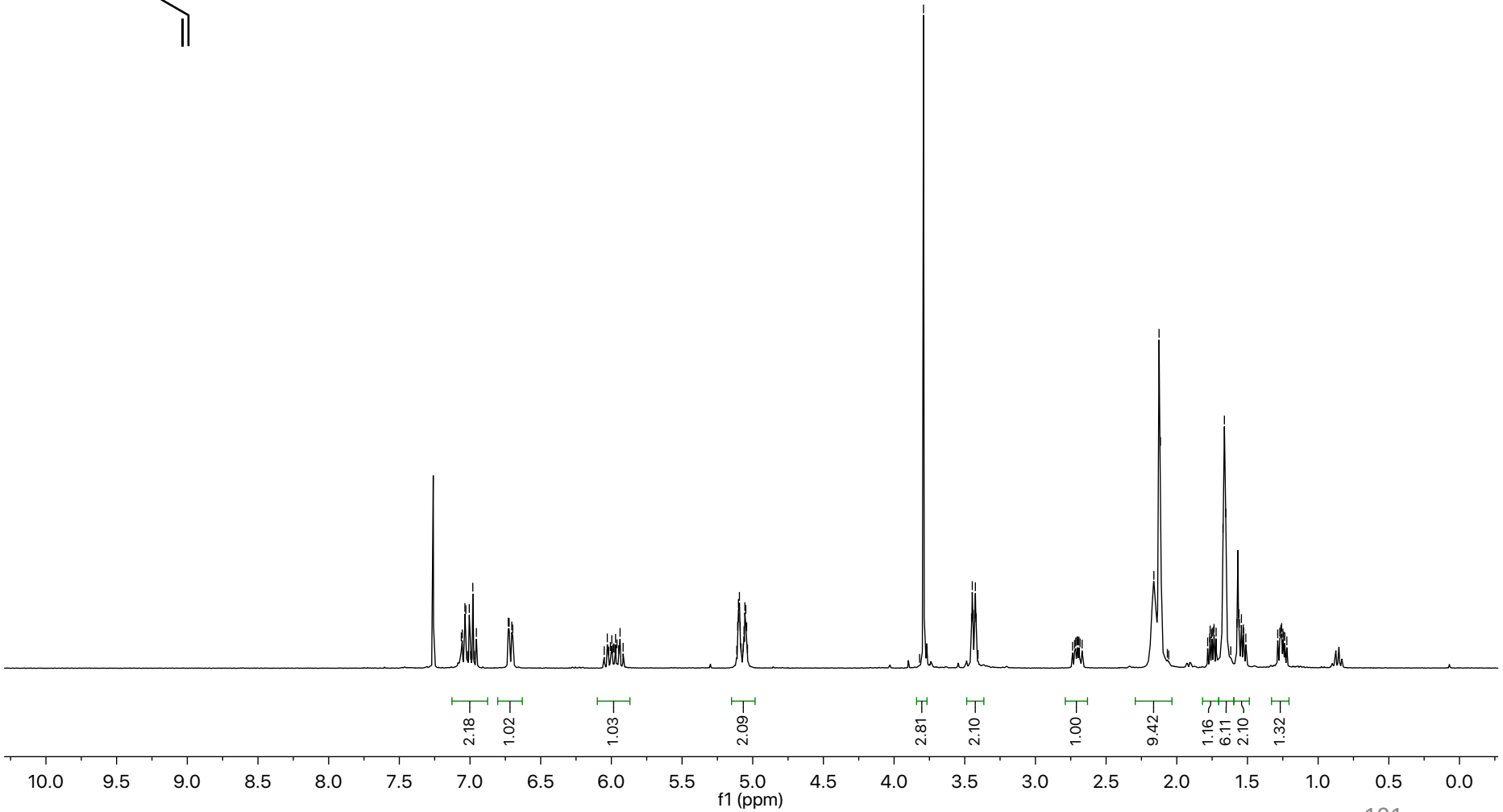
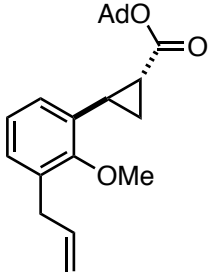


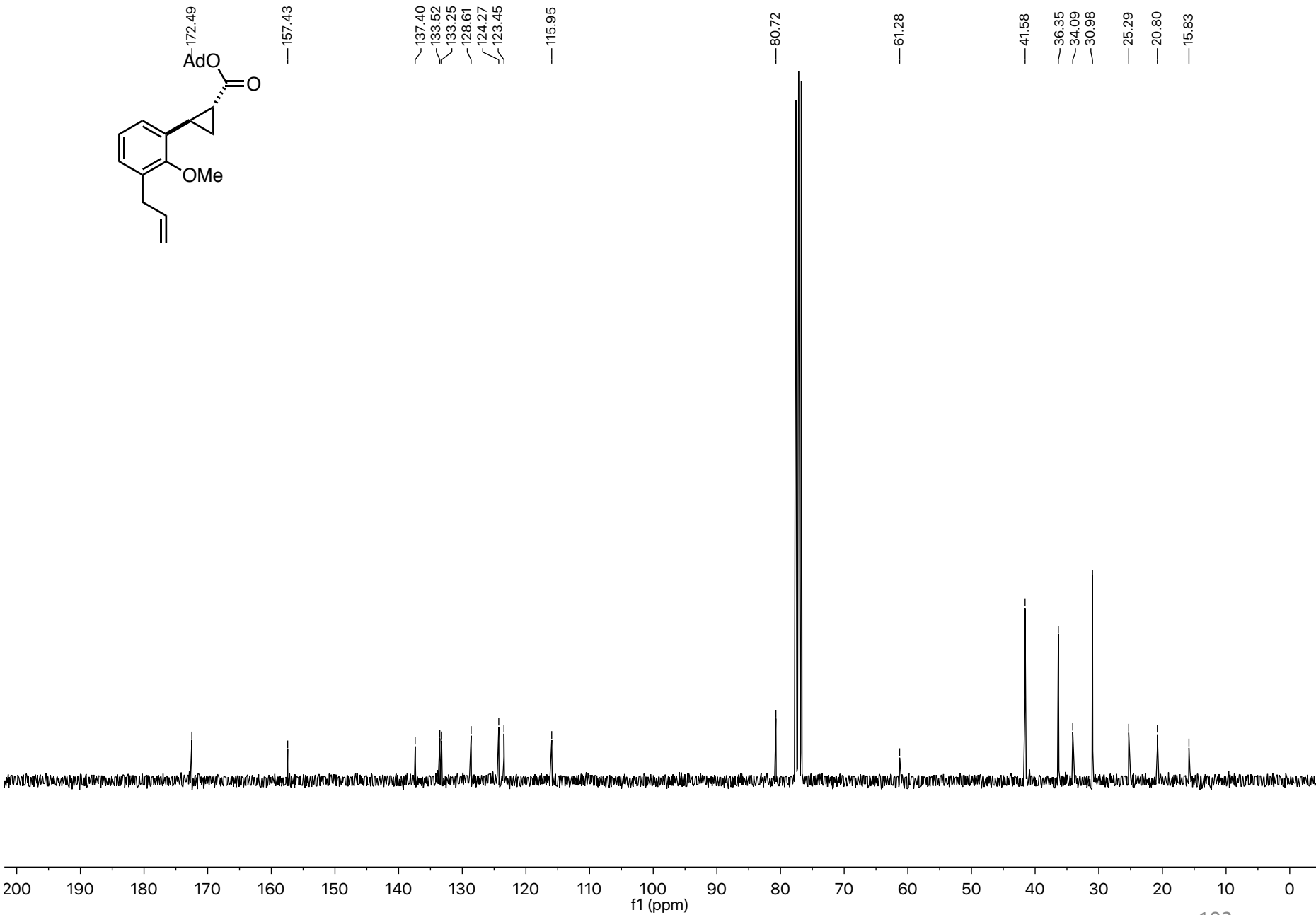
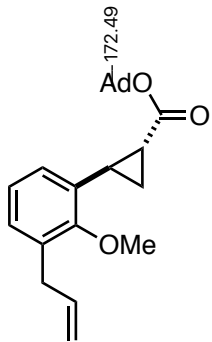






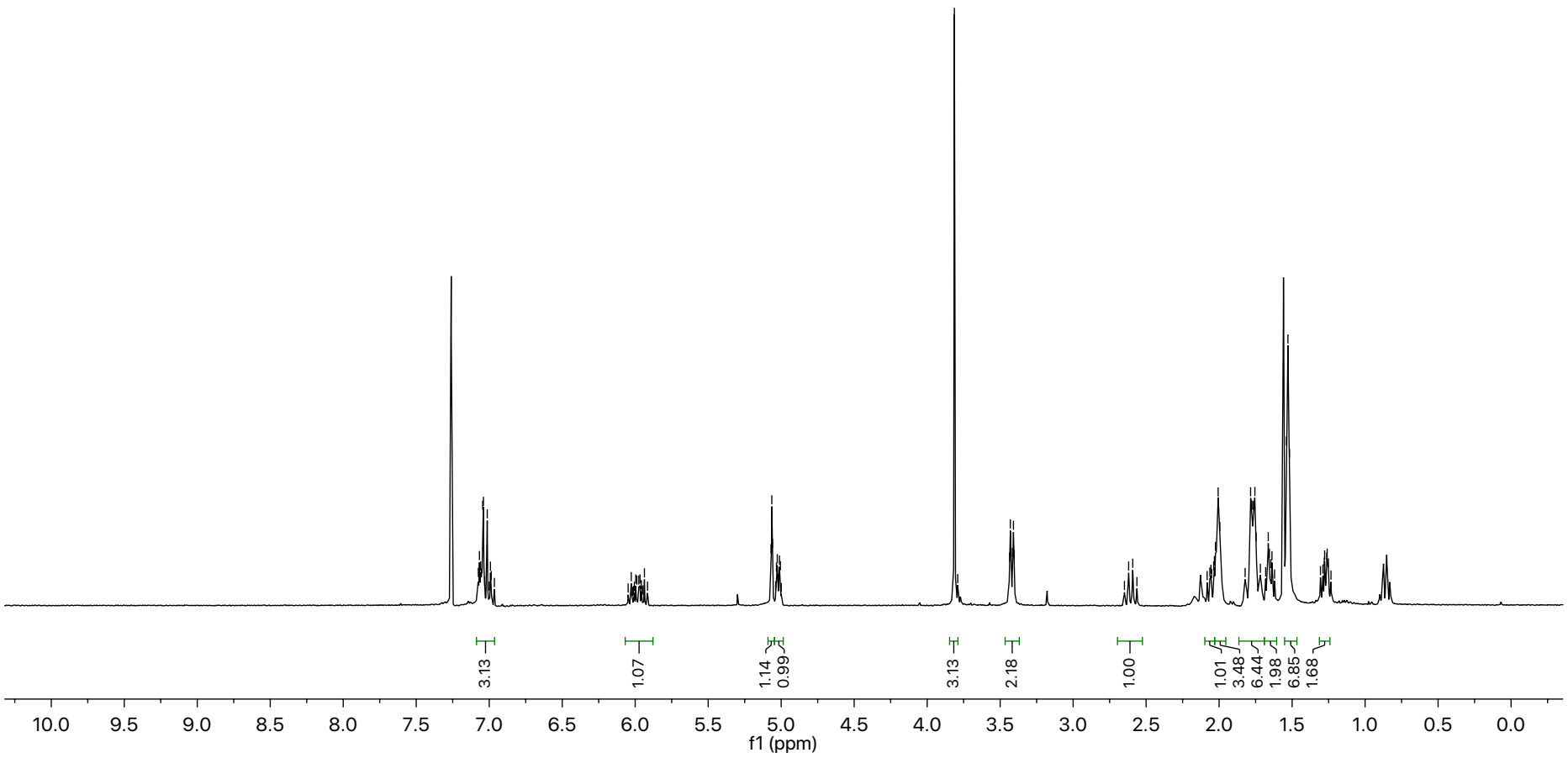
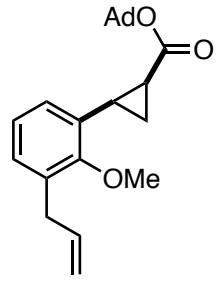
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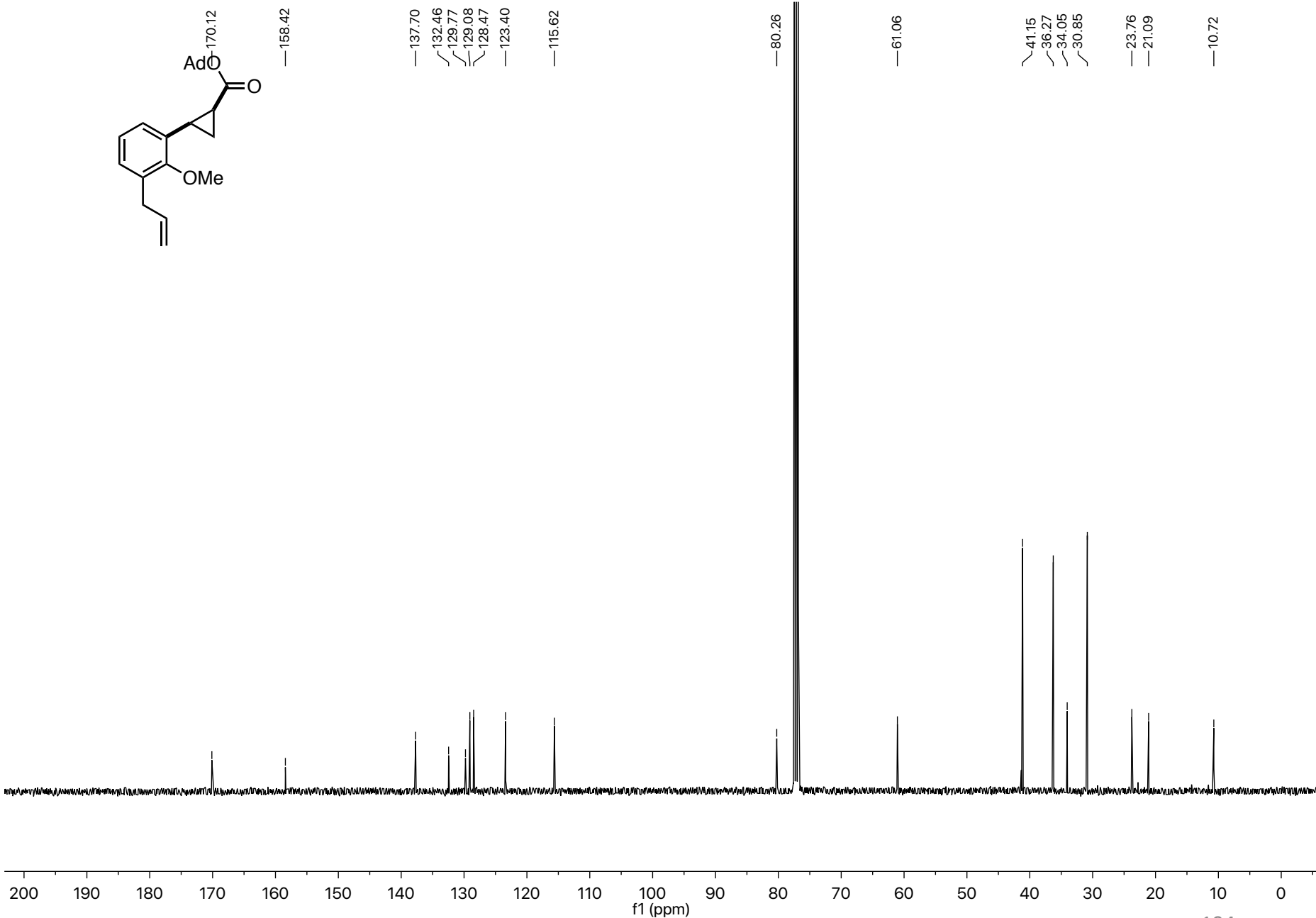
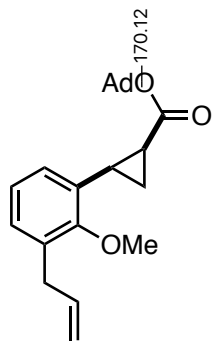




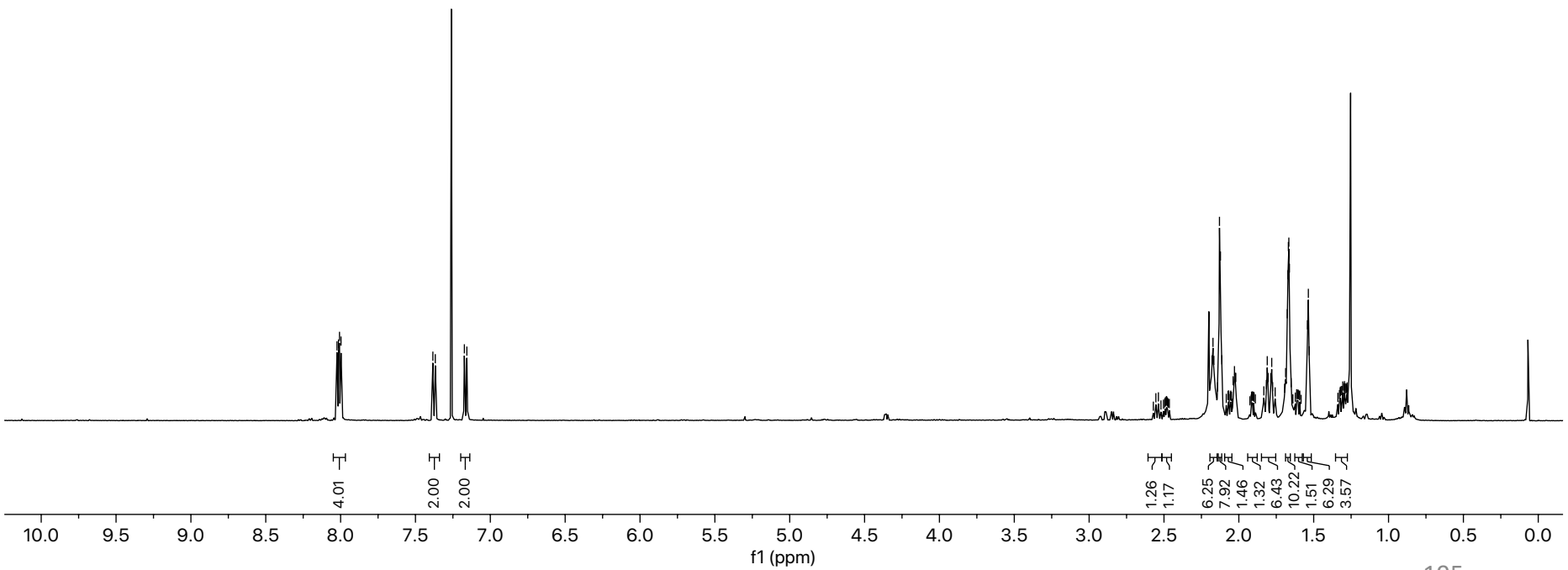
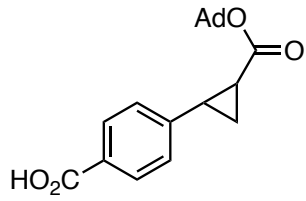


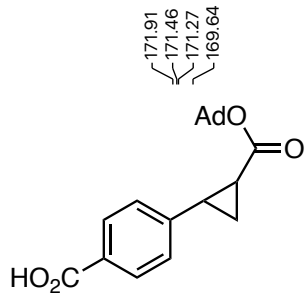
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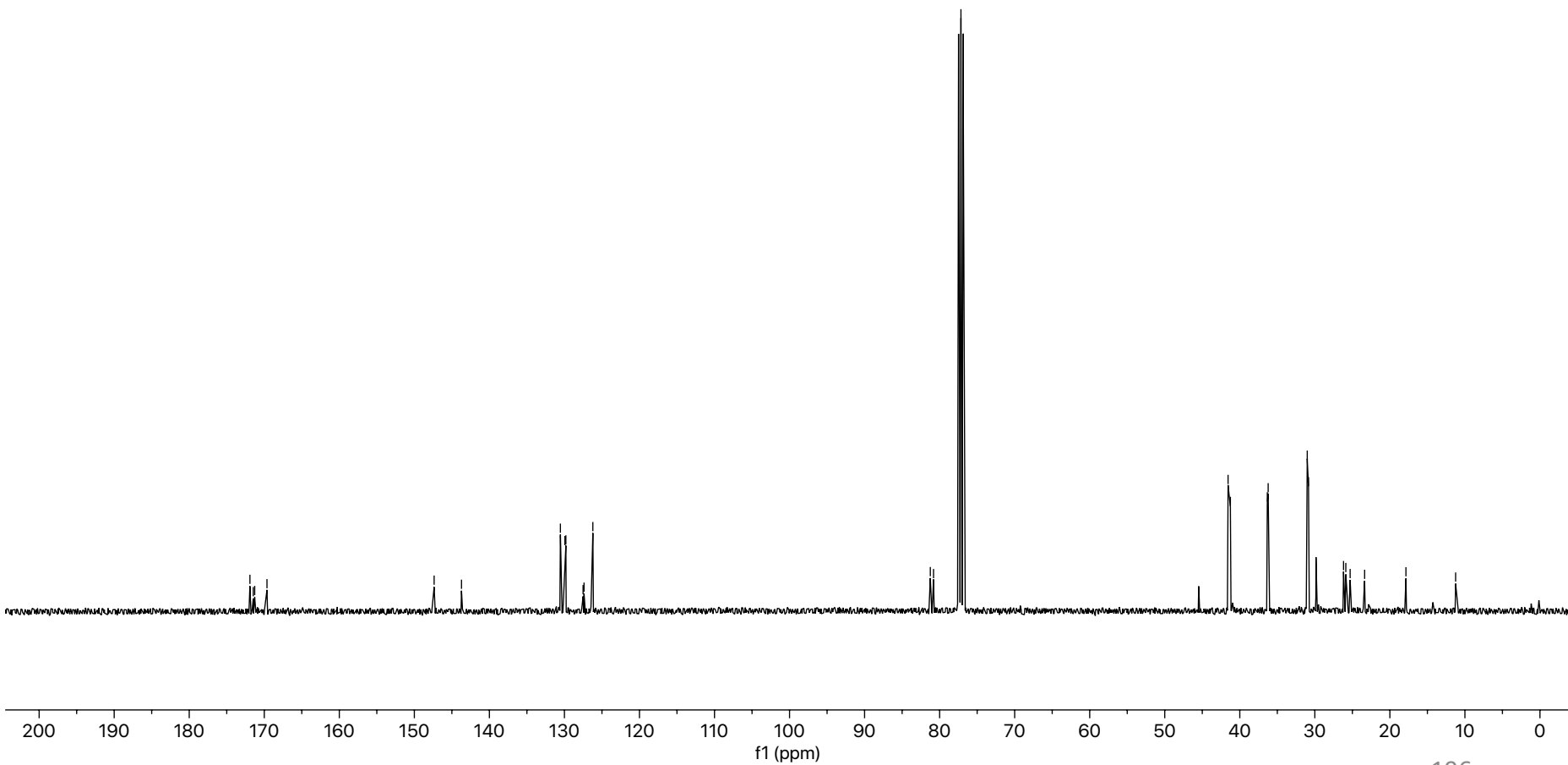
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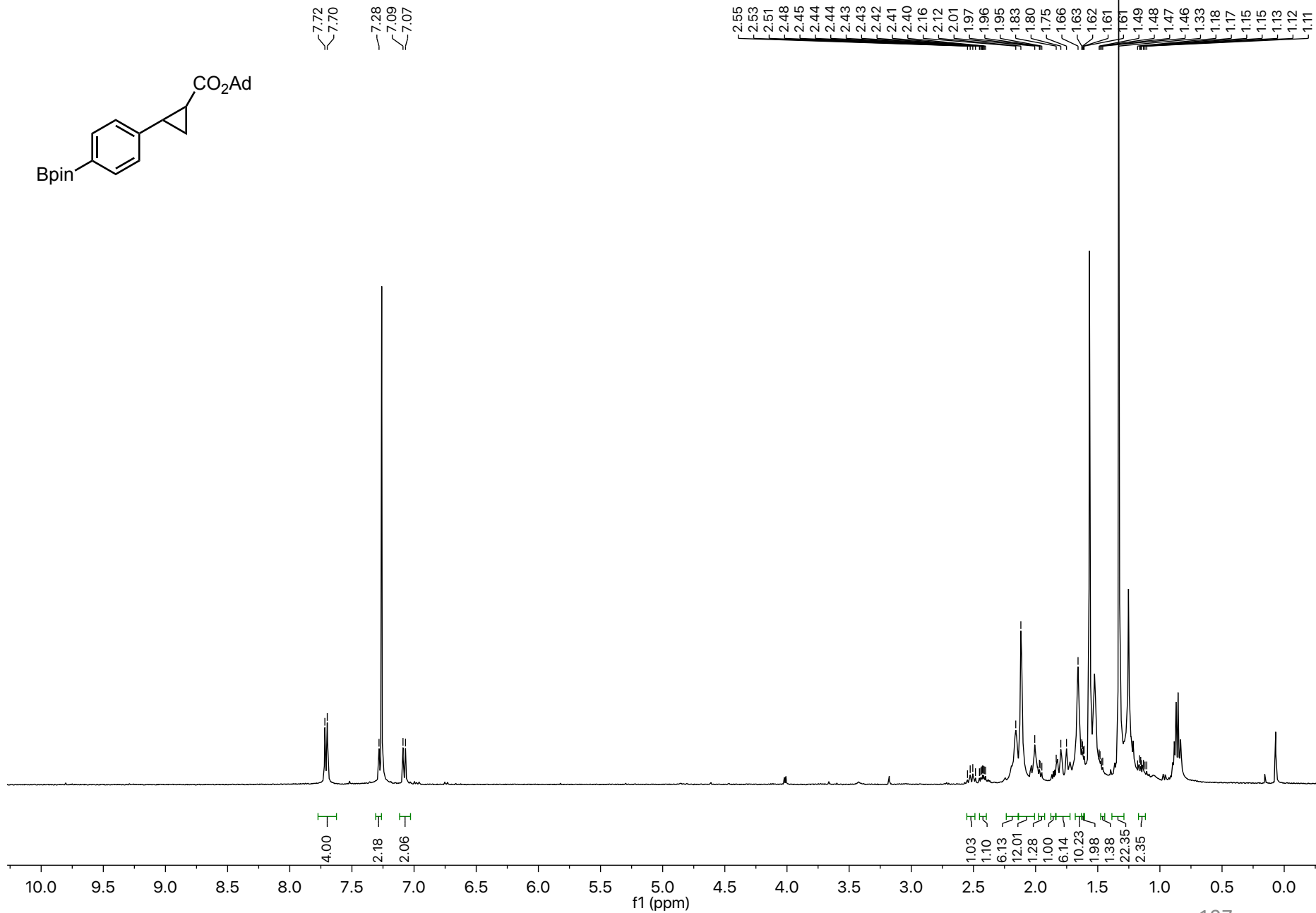
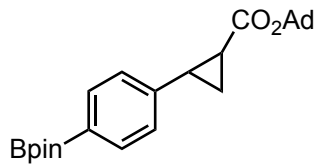
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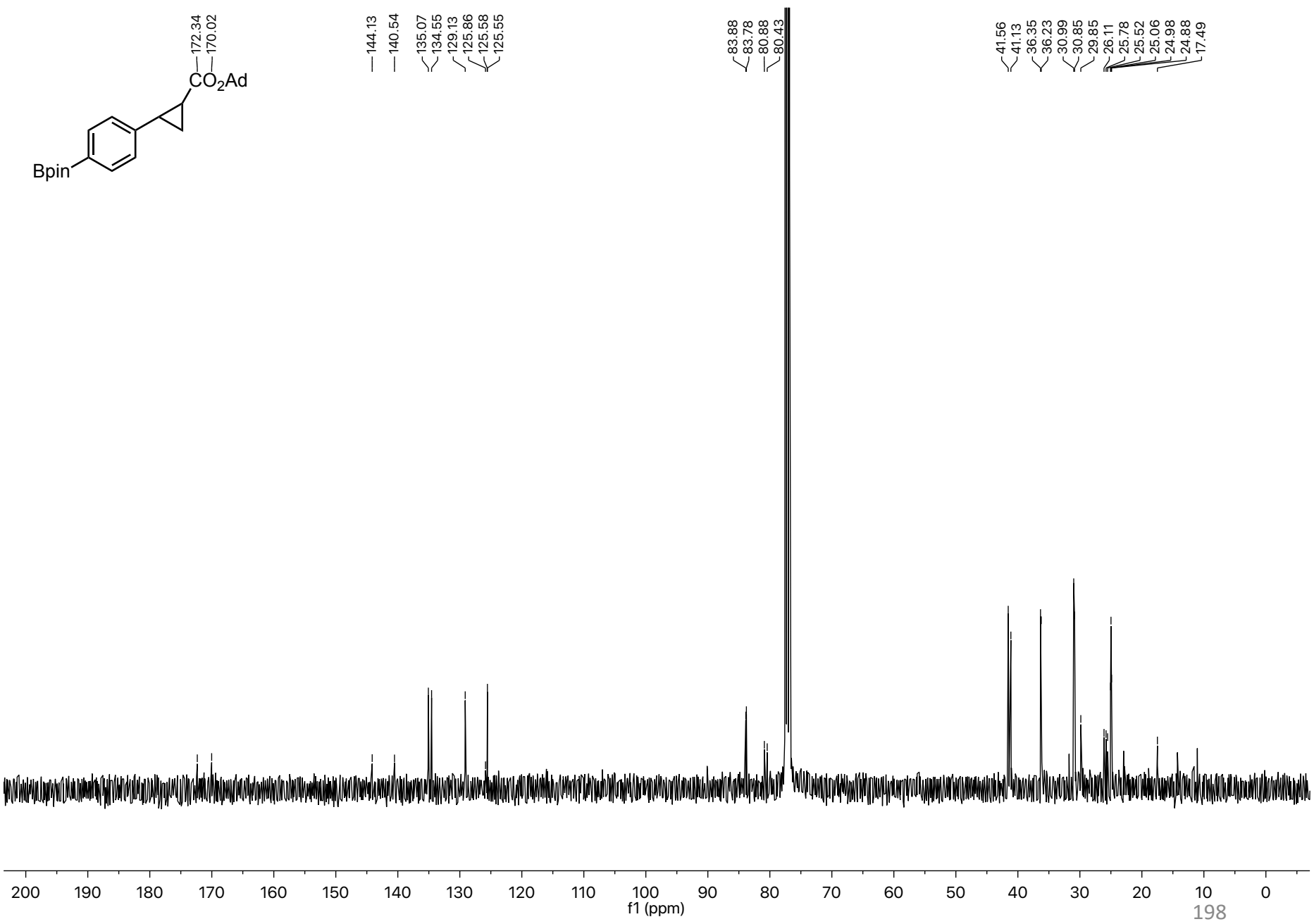
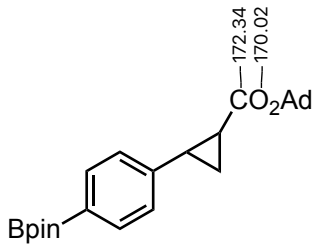
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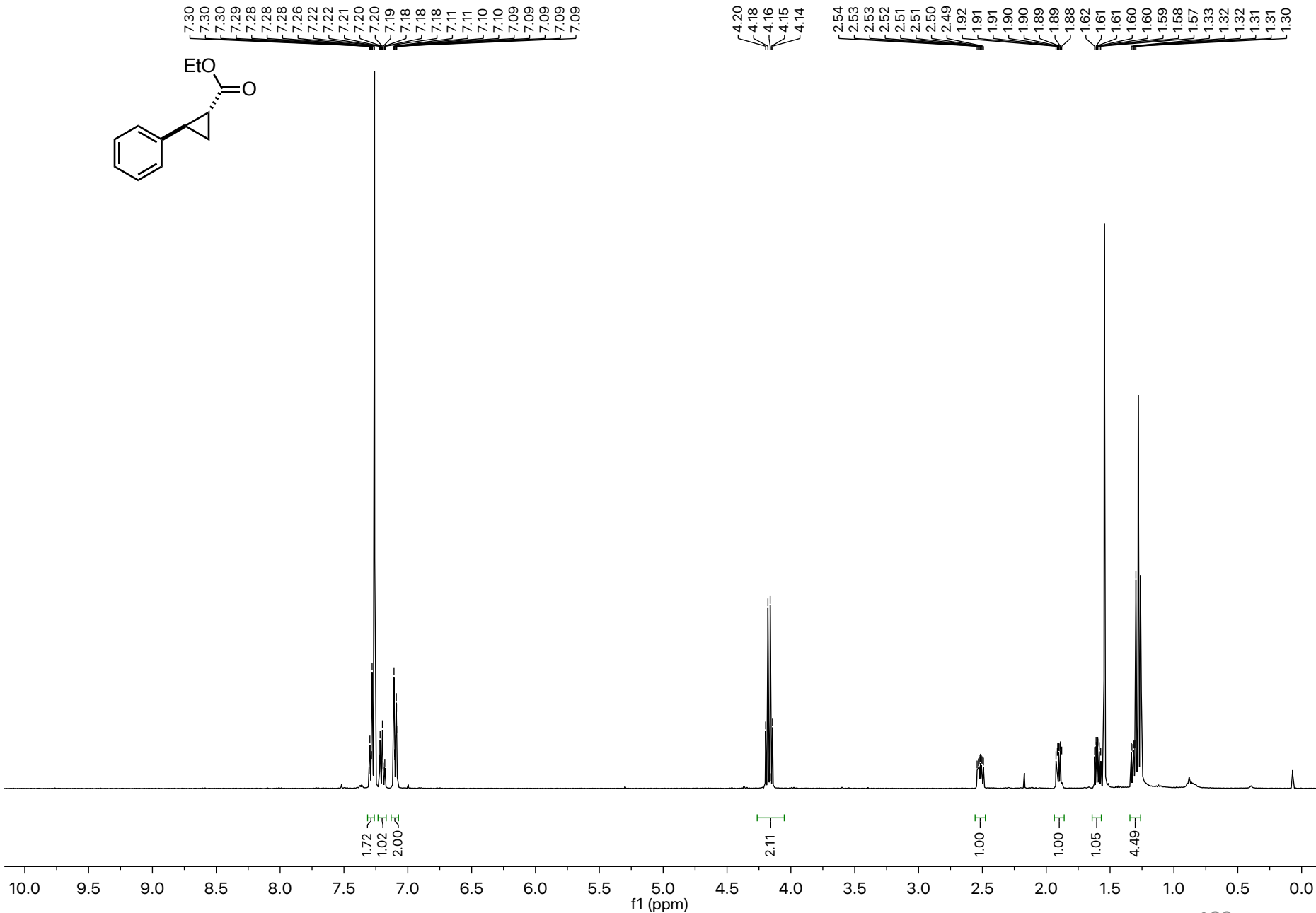
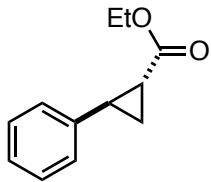
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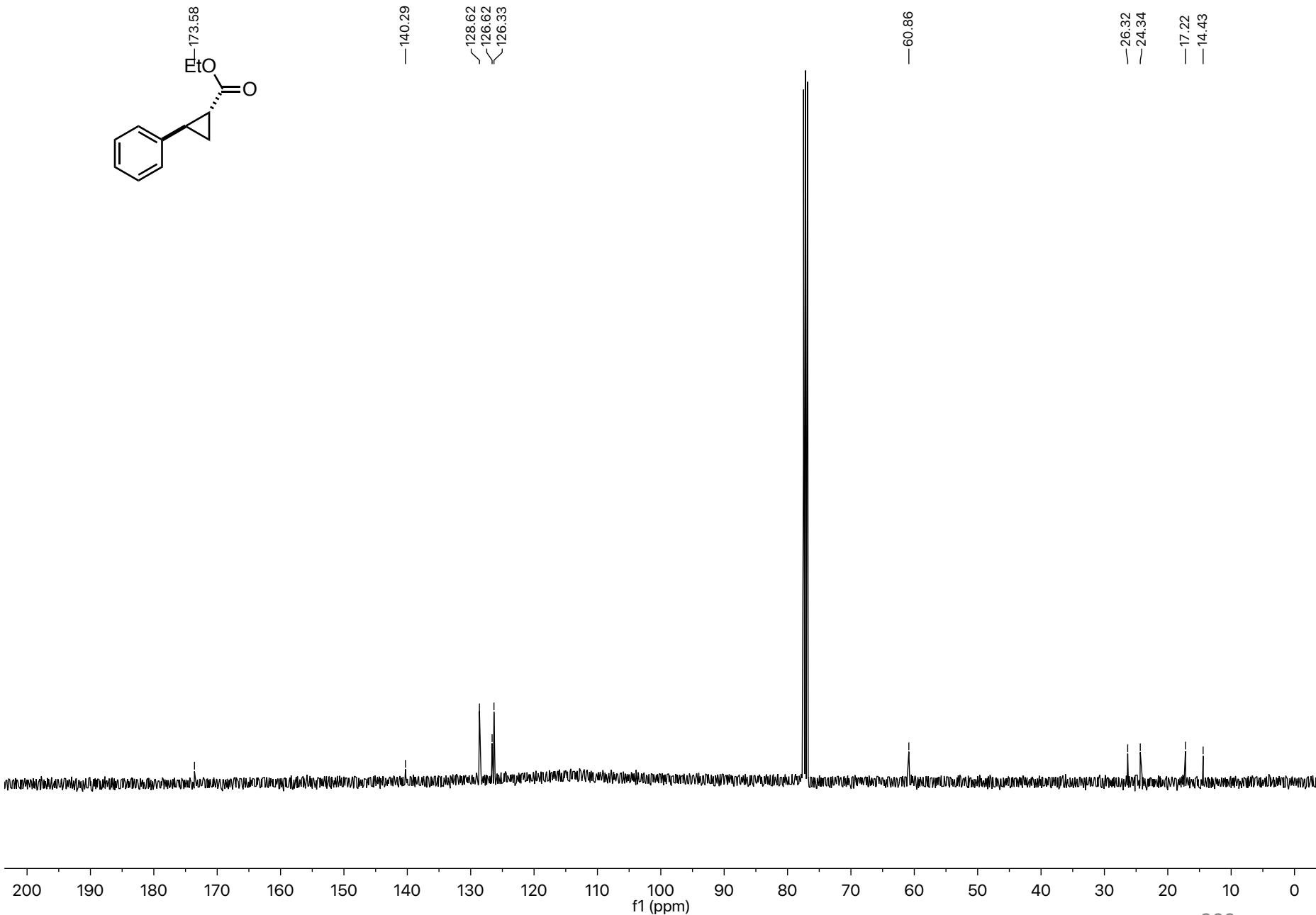
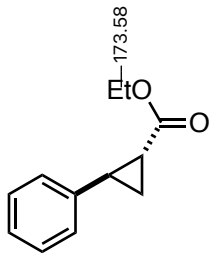
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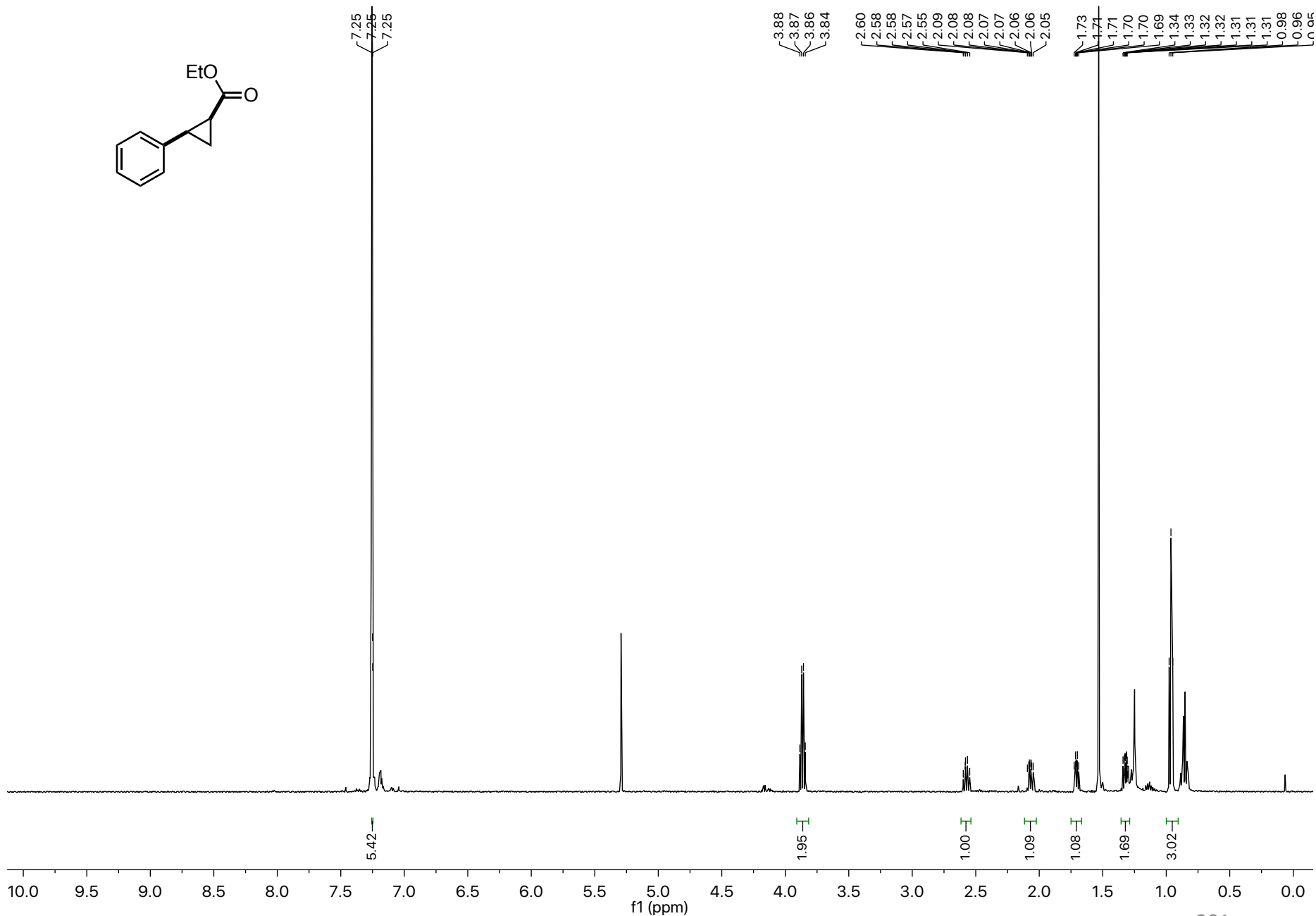
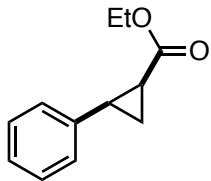


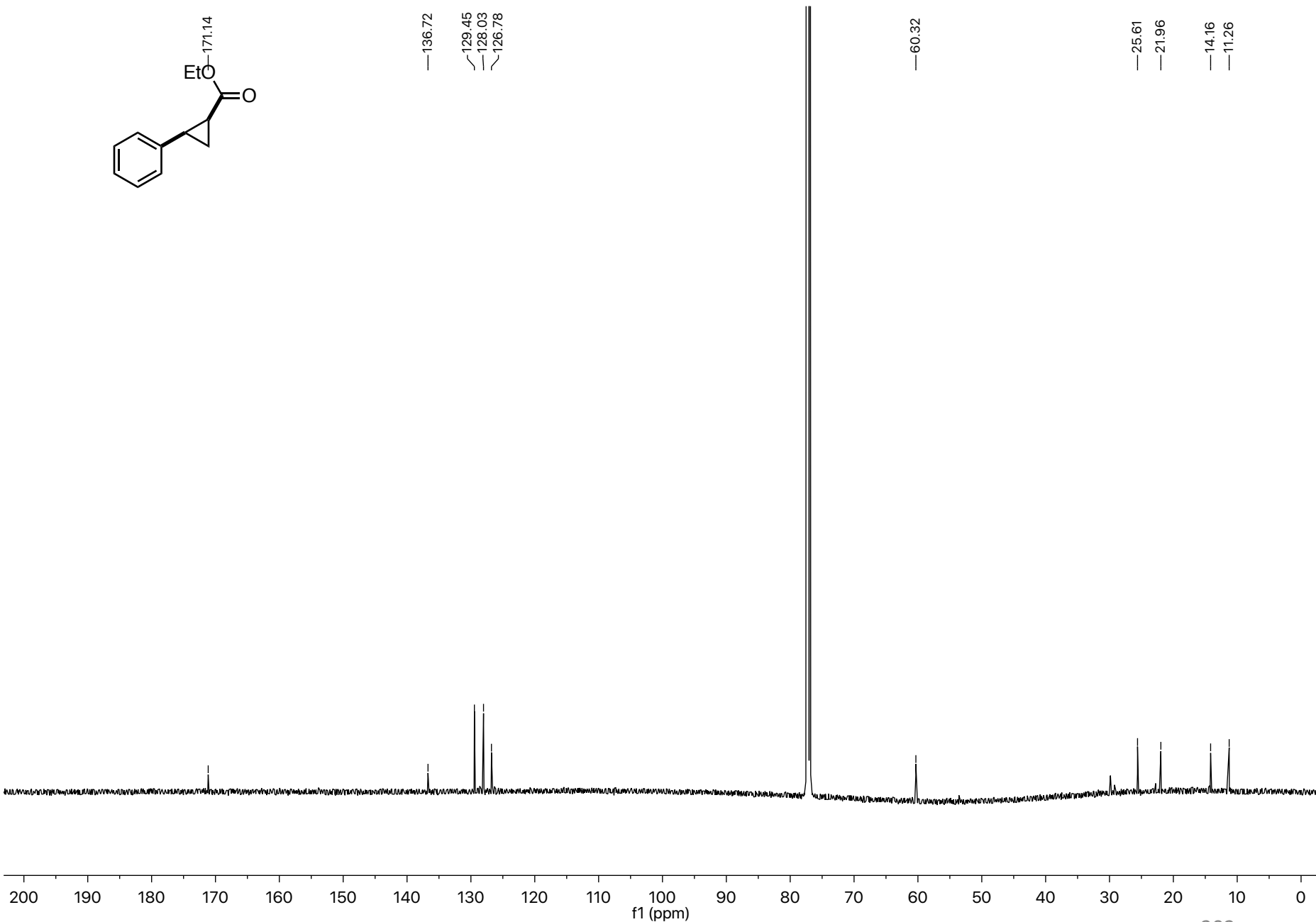
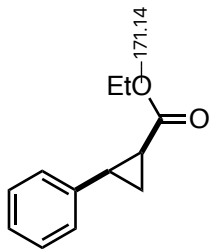


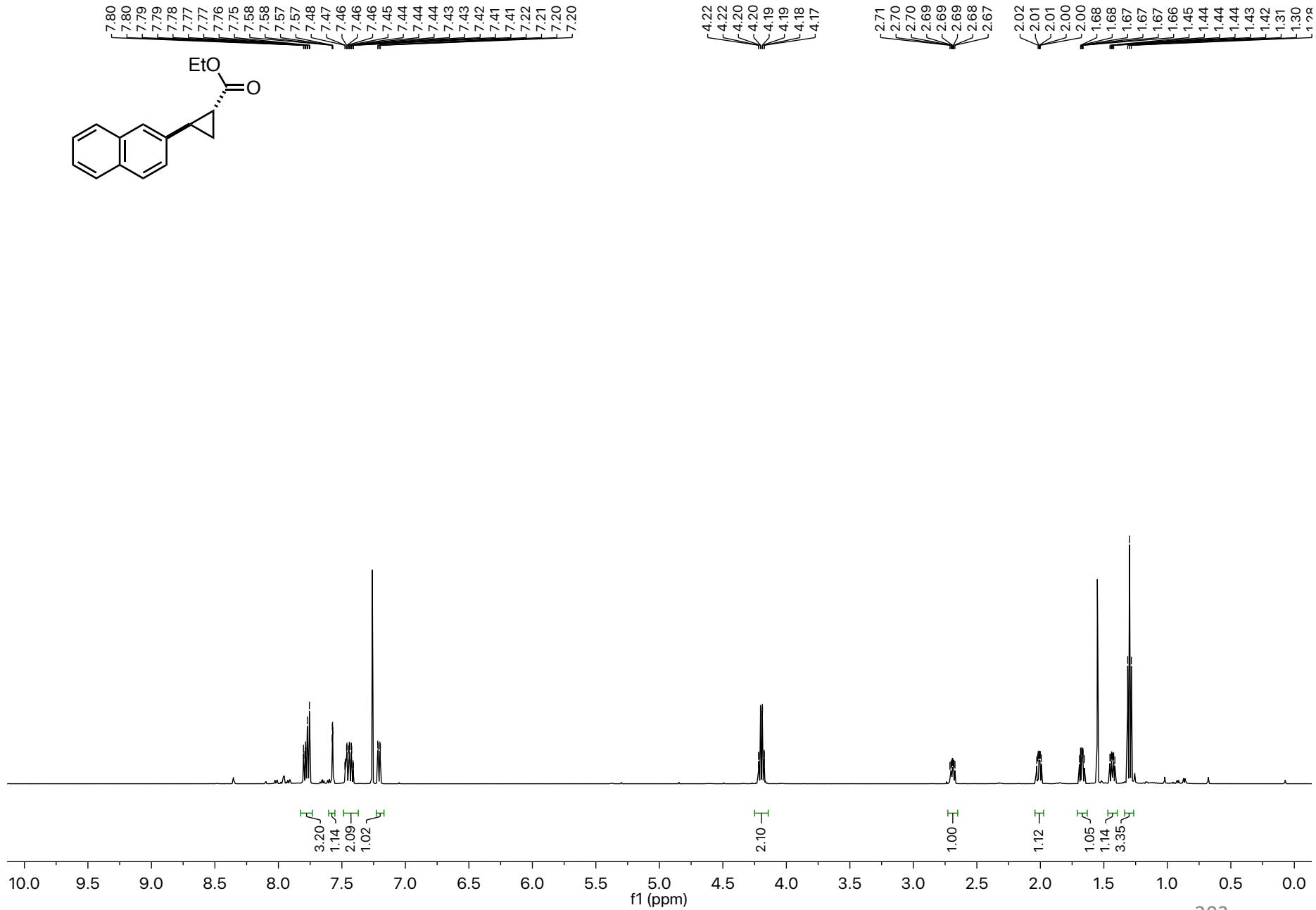
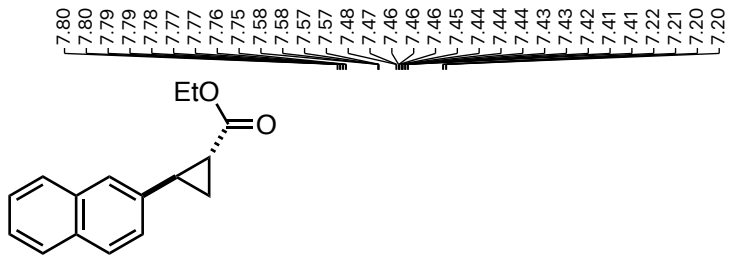


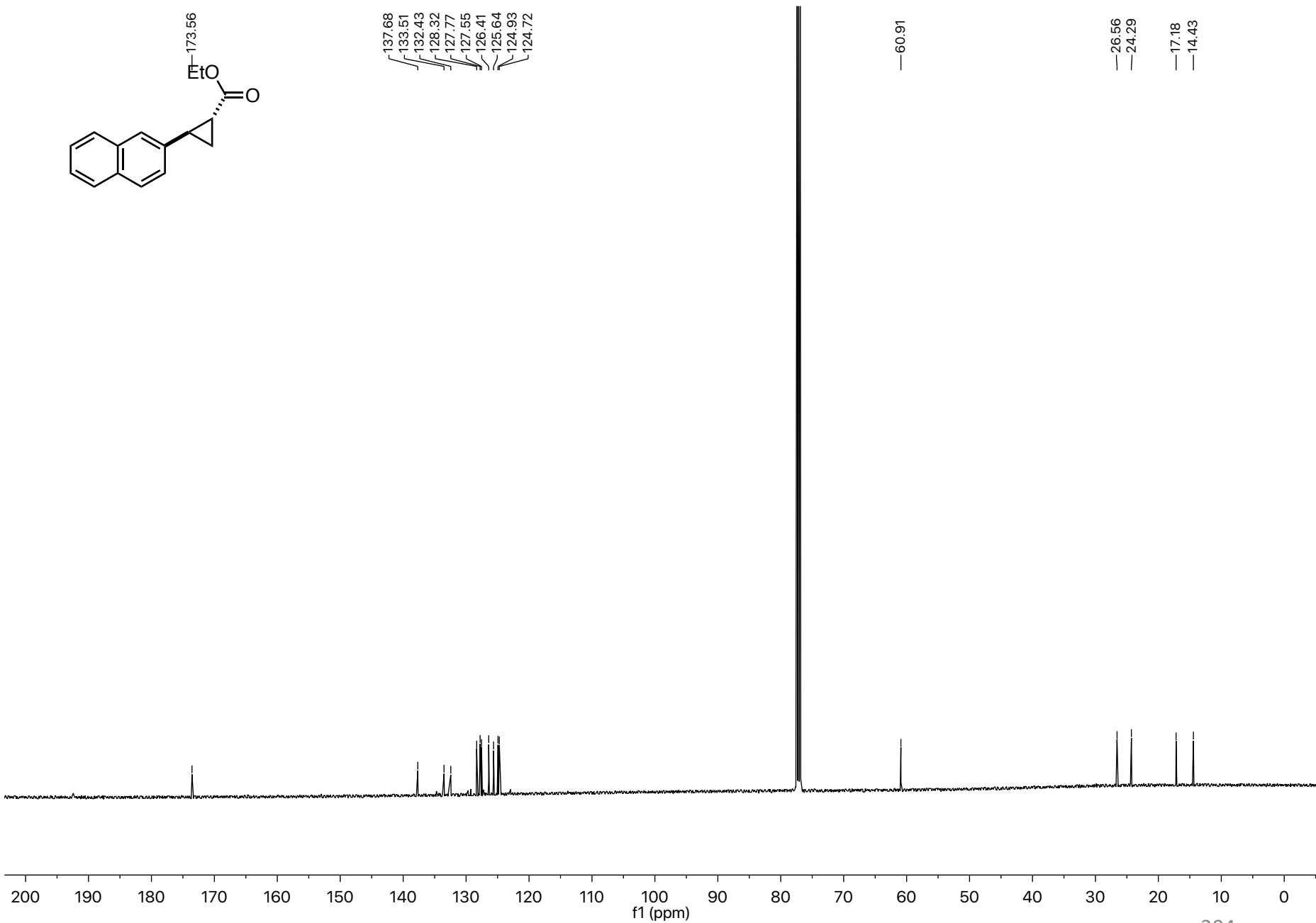
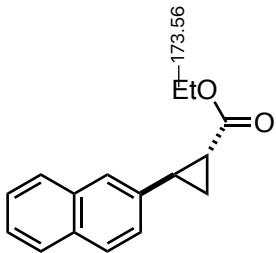


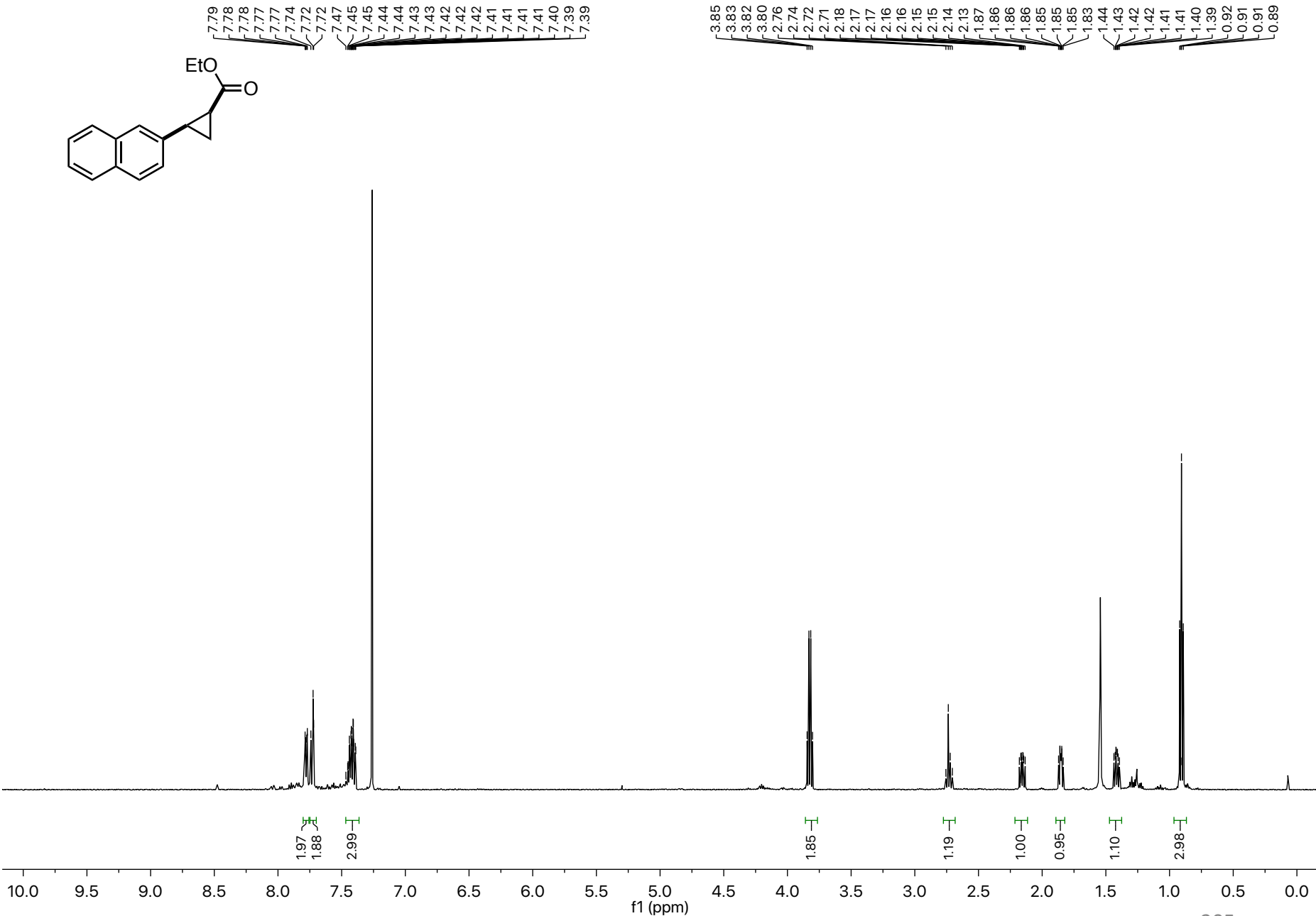
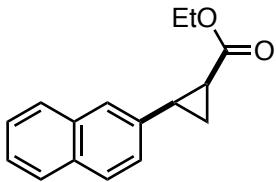


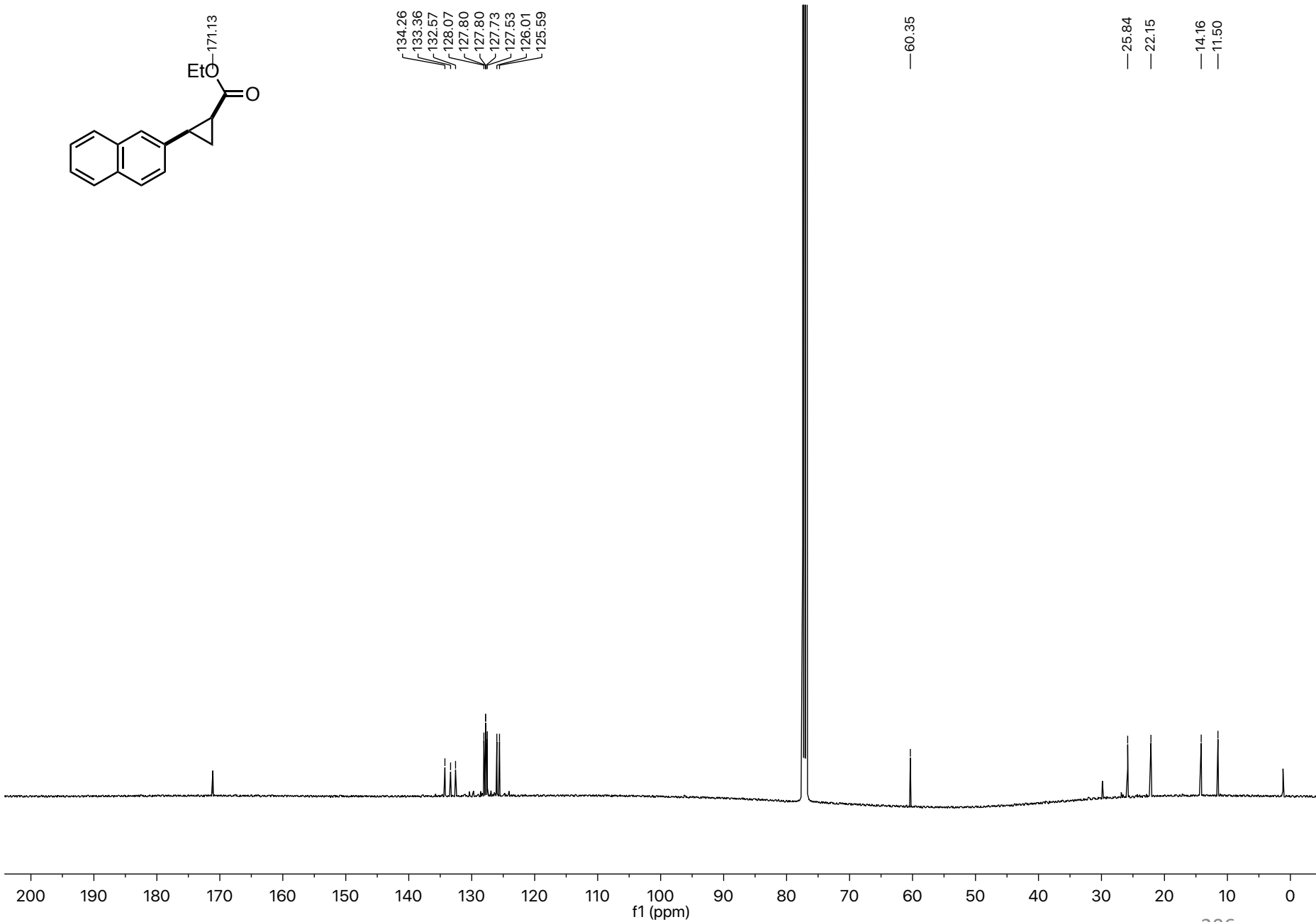
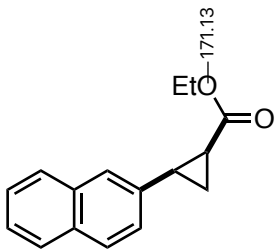


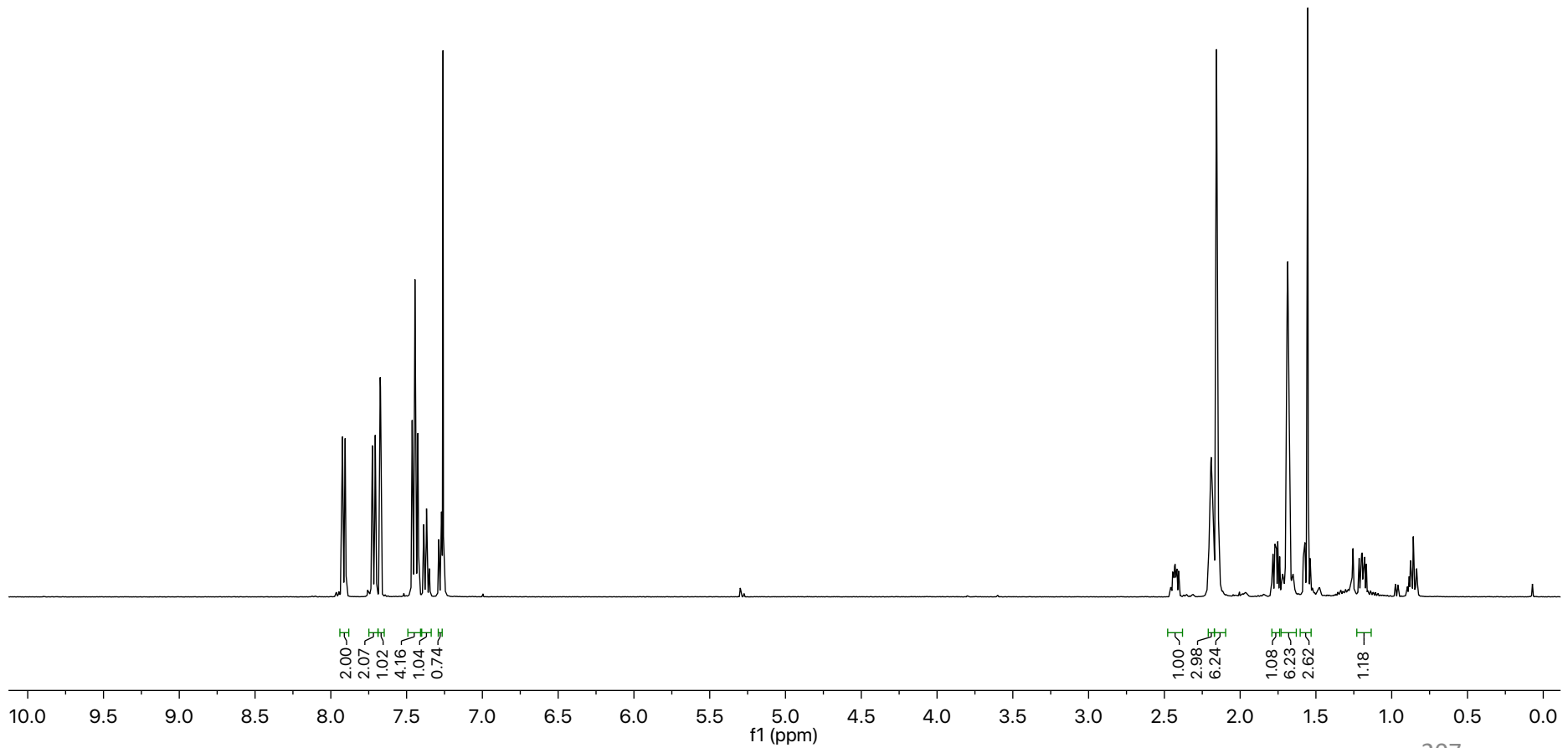
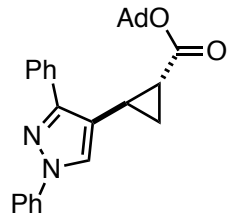


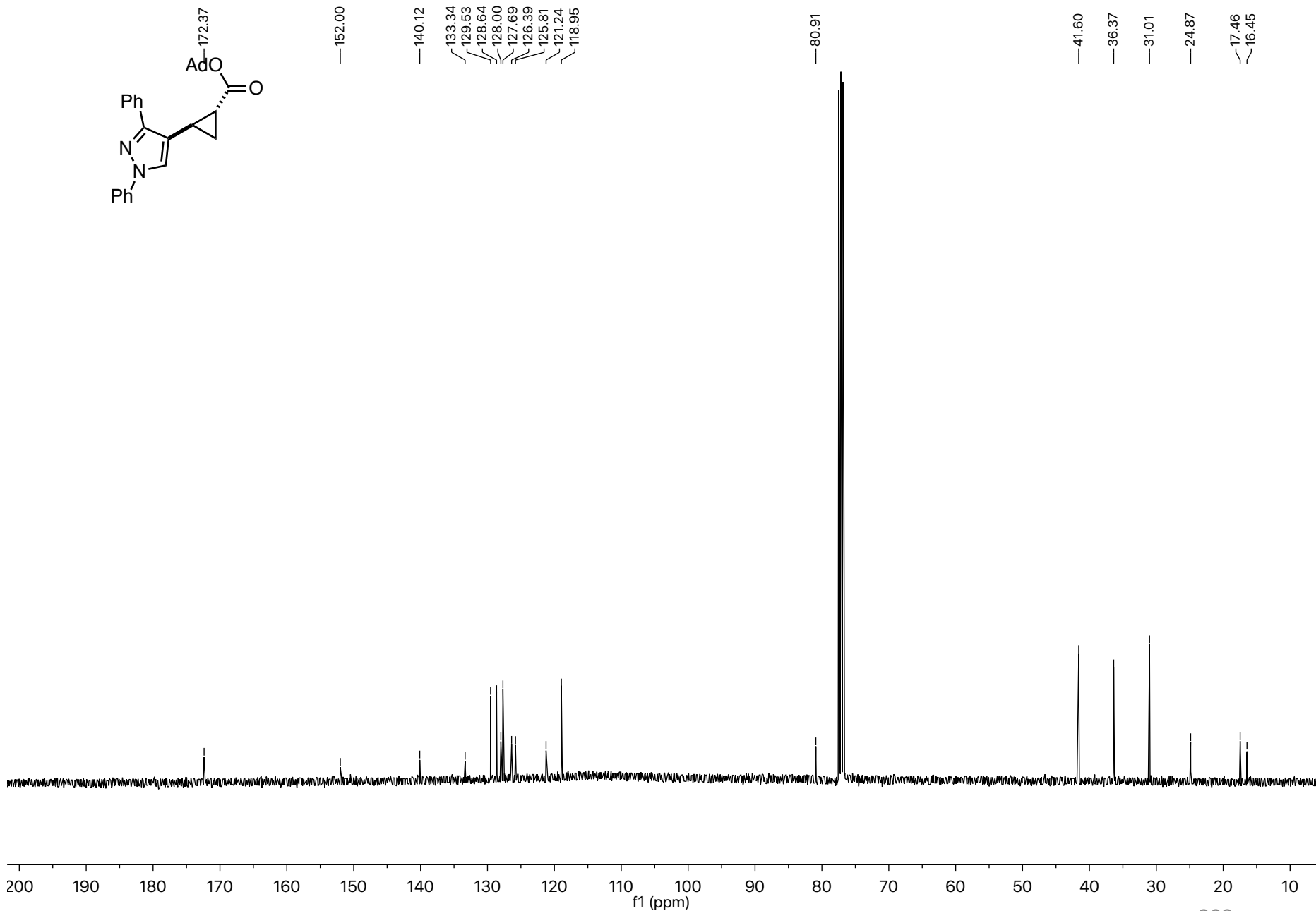
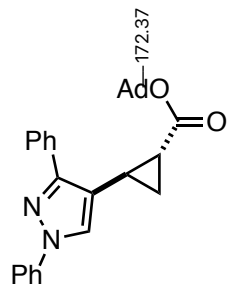






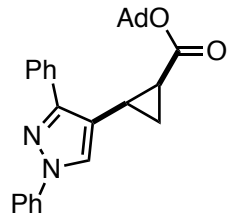




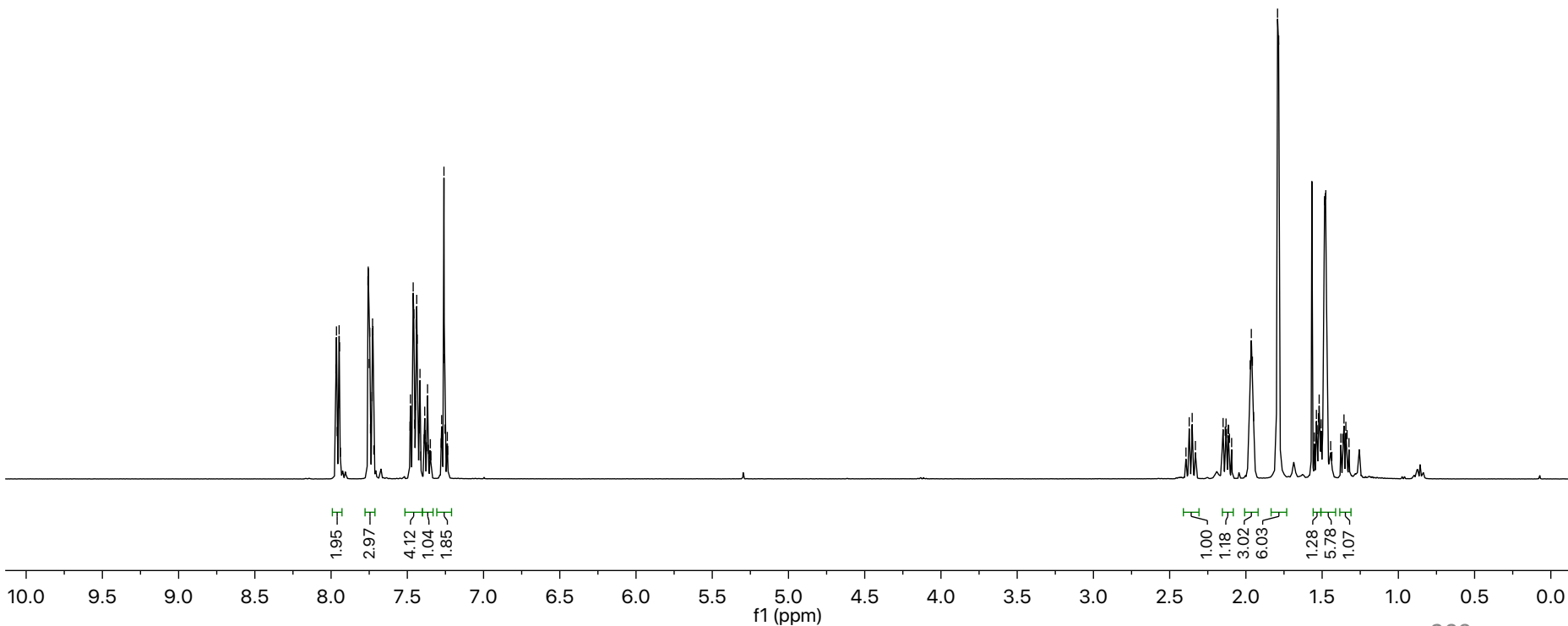


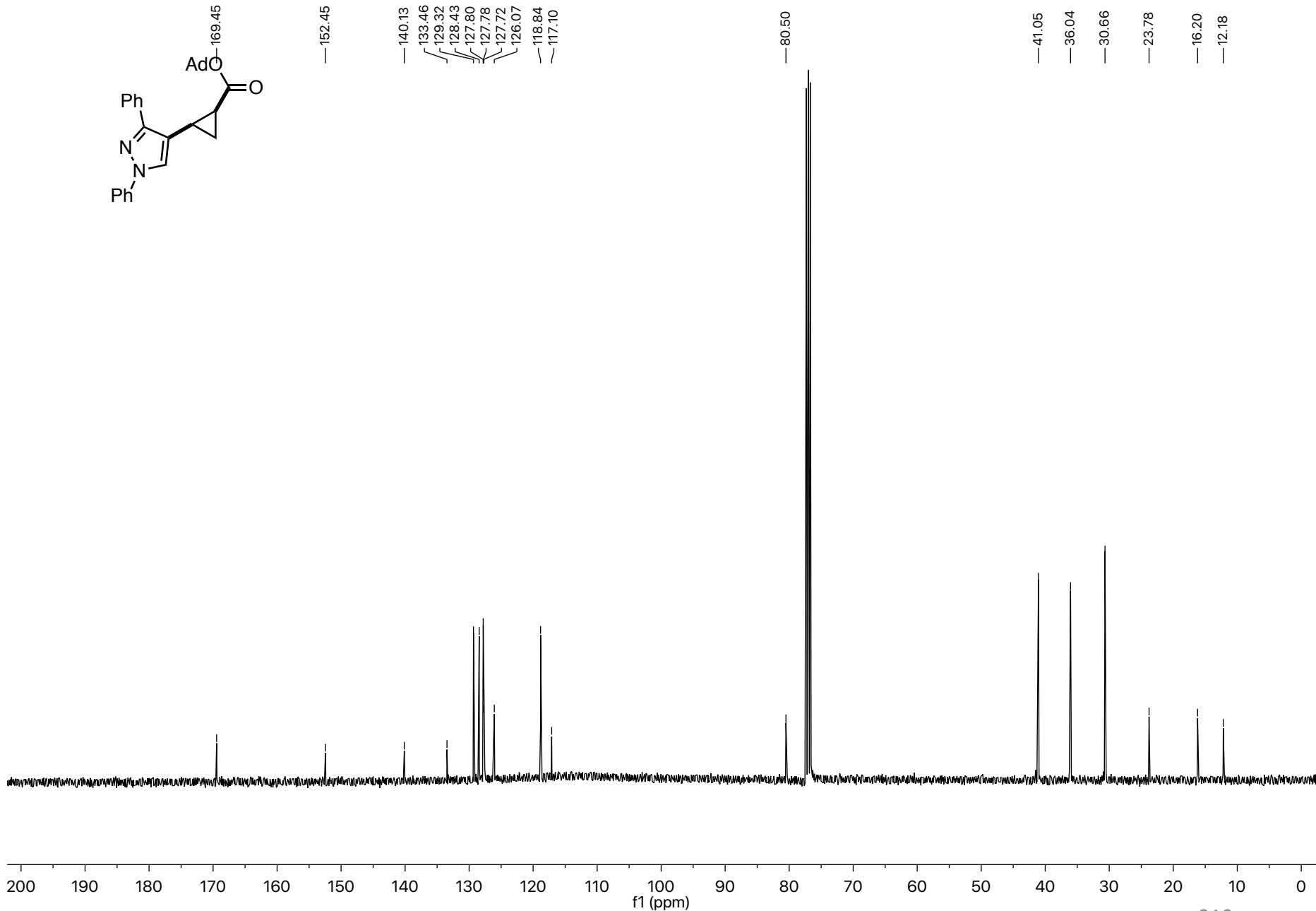
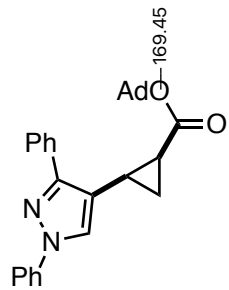


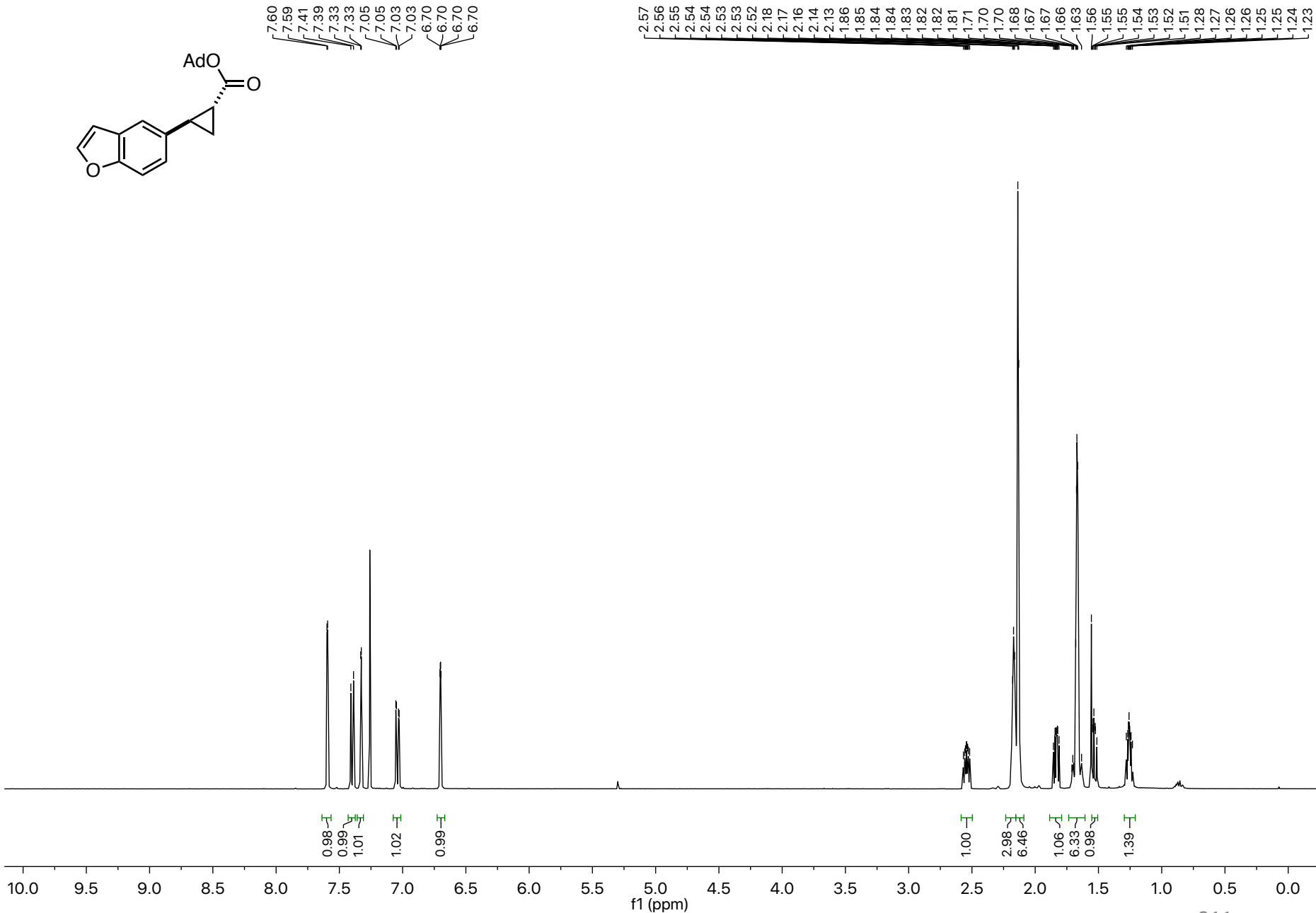
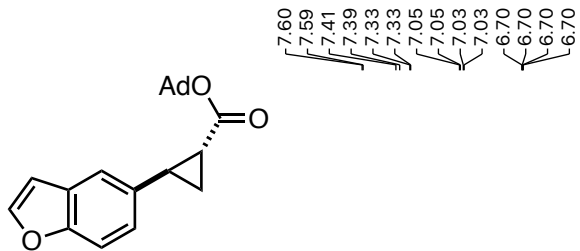
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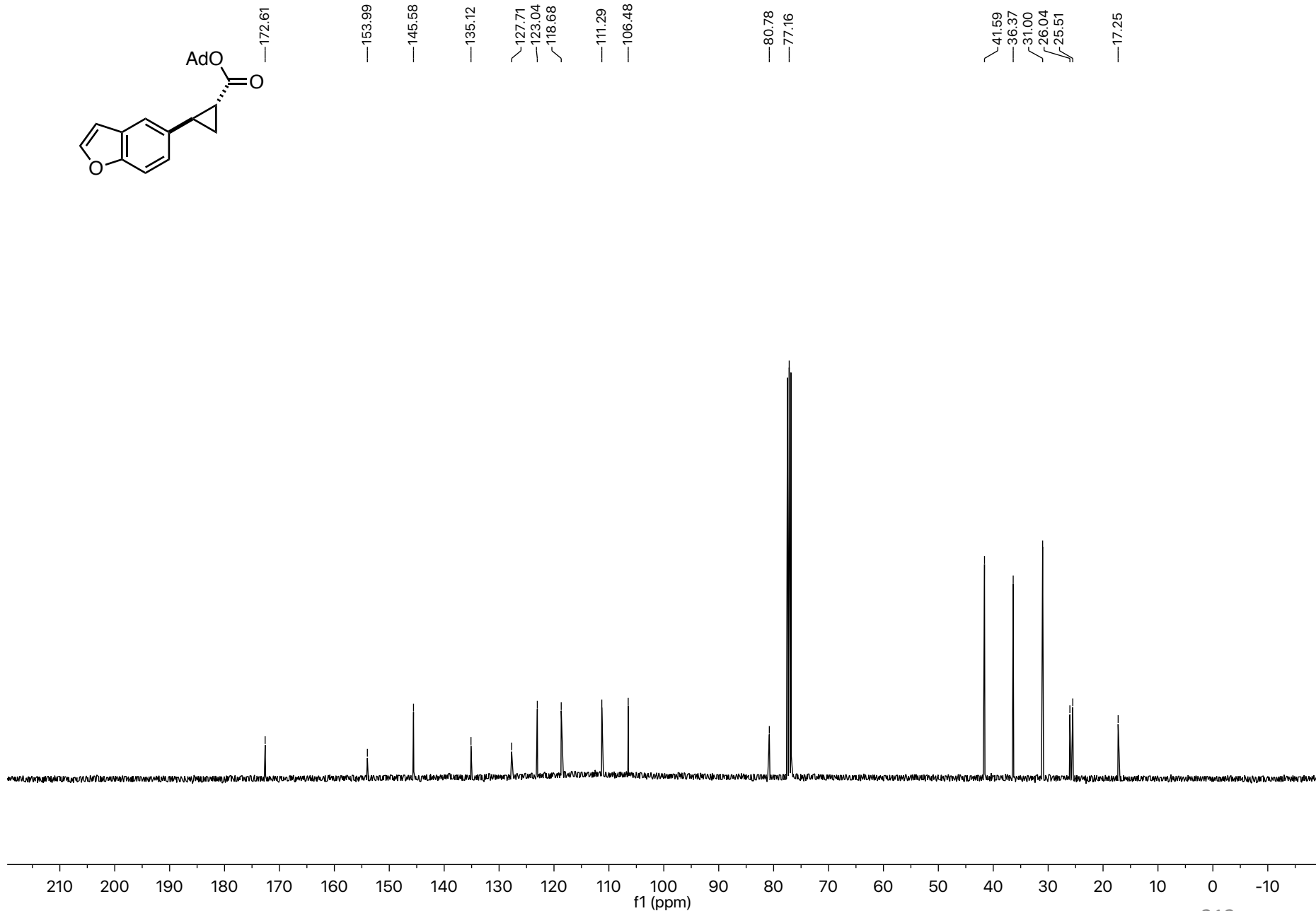
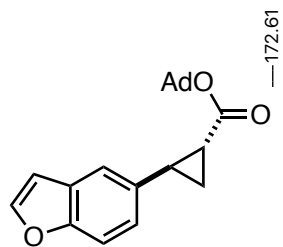


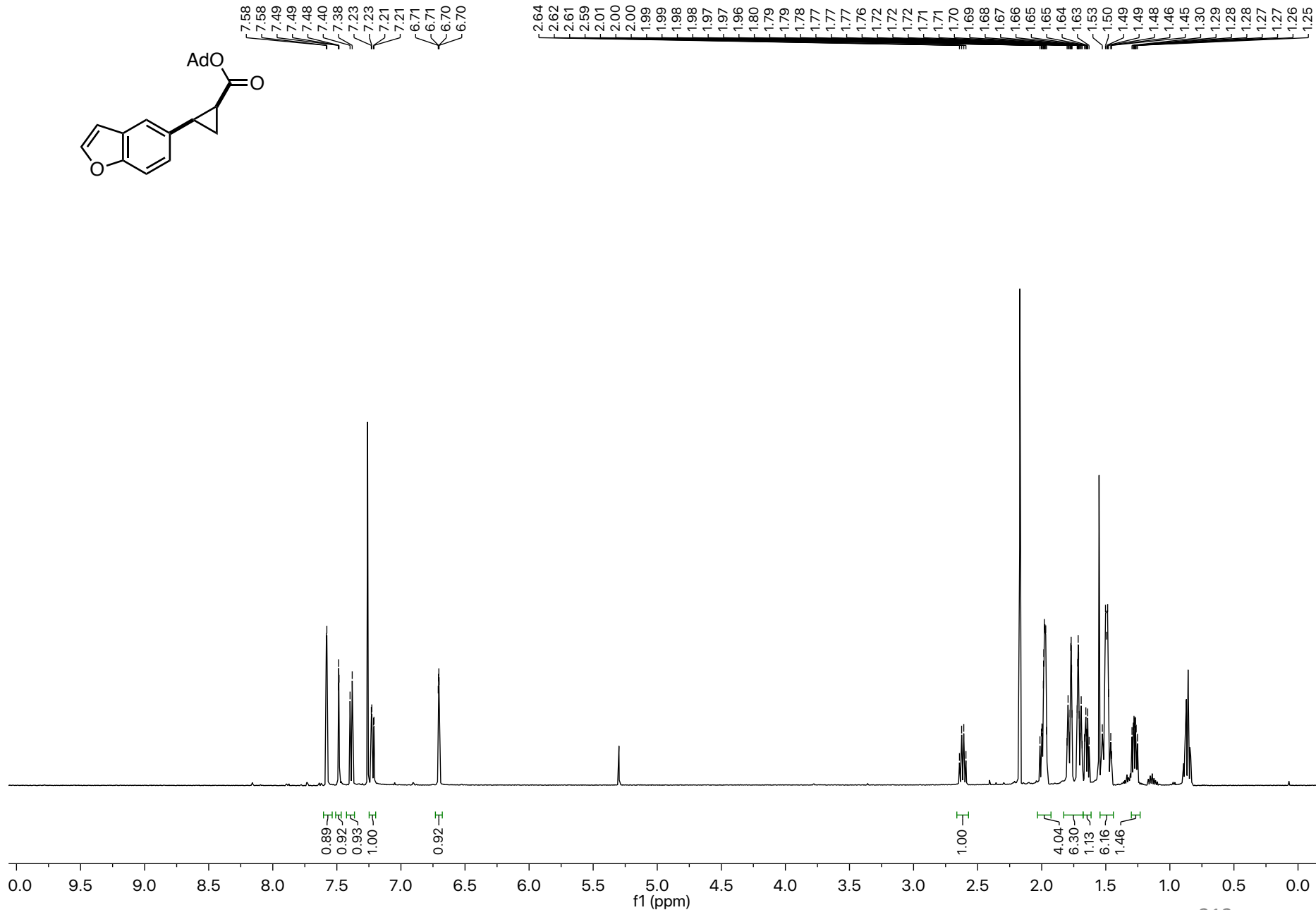
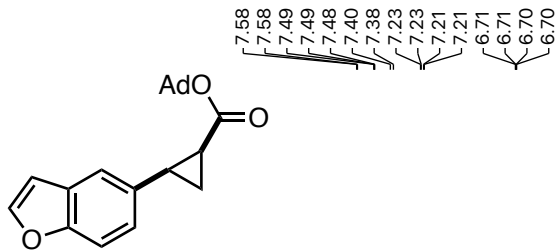
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1.96  
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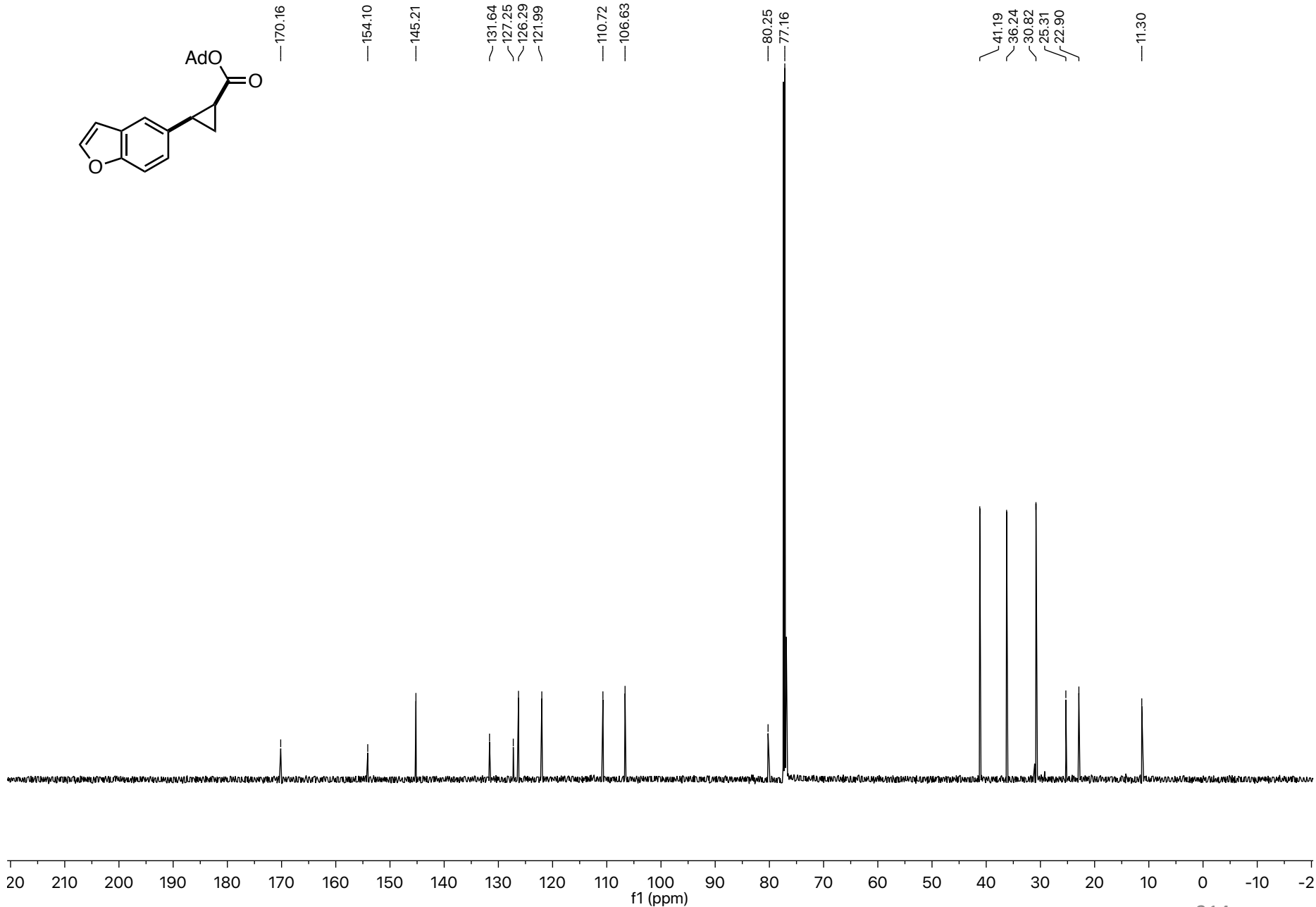
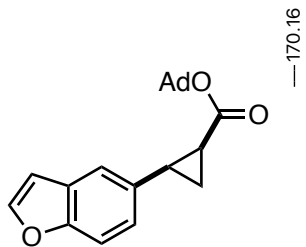


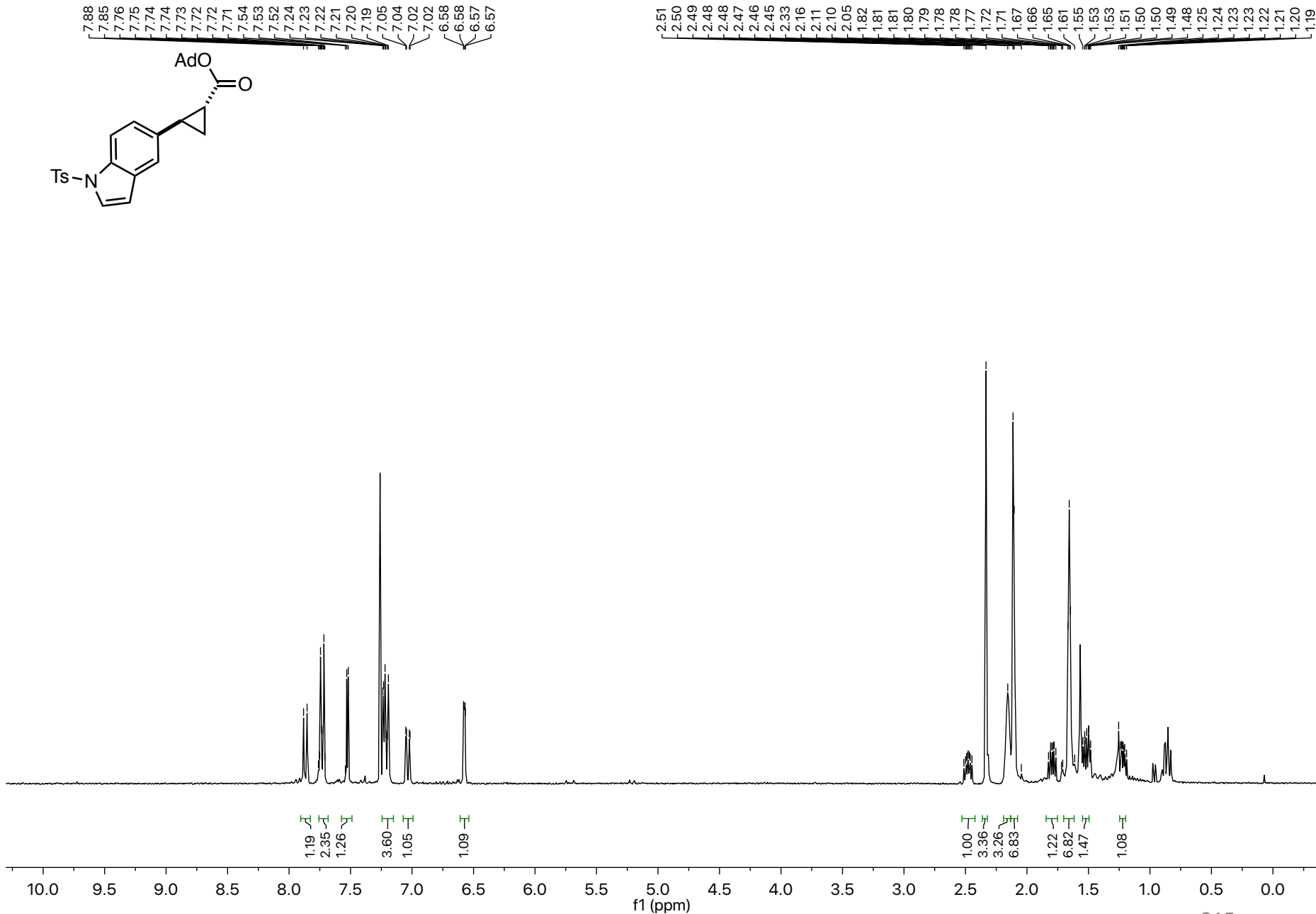
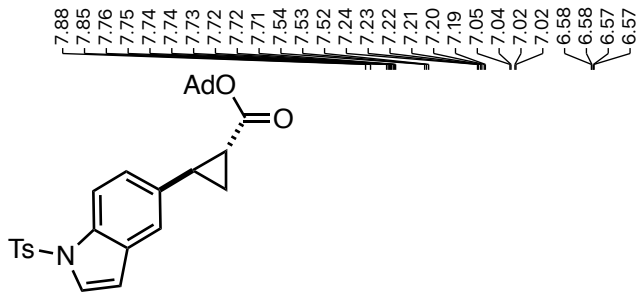


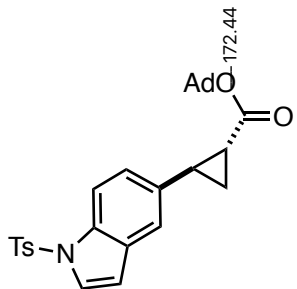






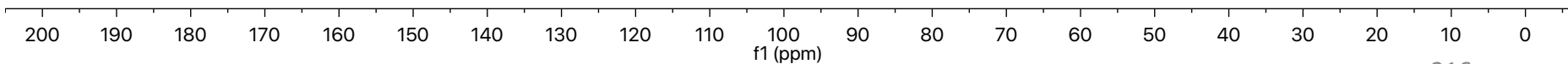




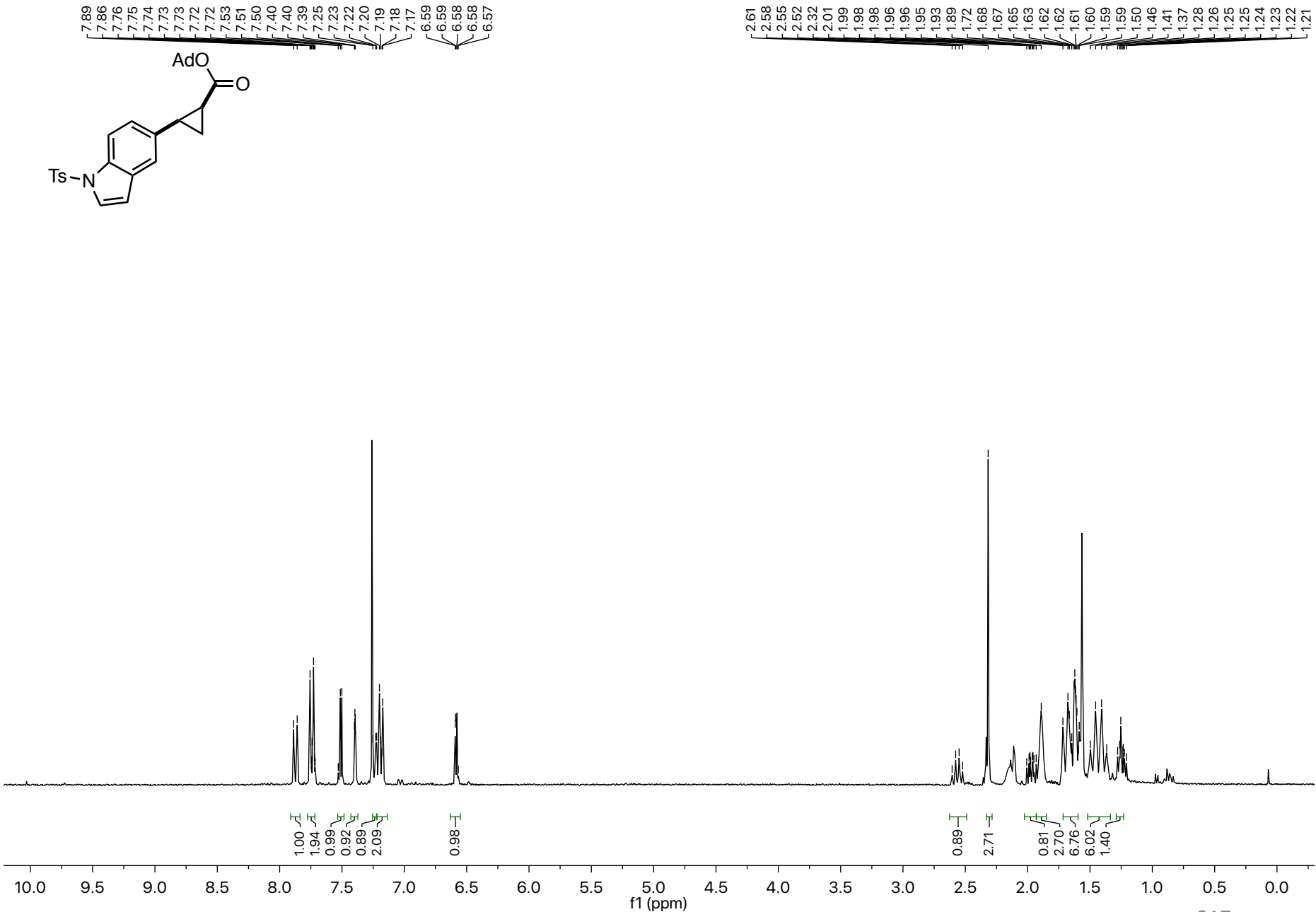
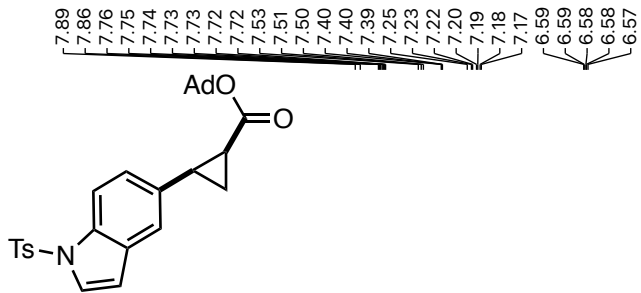


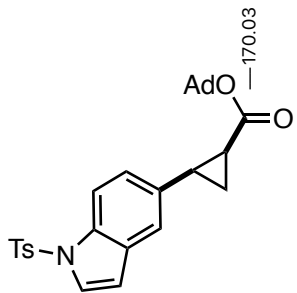
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135.41  
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130.01  
126.99  
126.91  
123.28  
118.80  
113.59  
108.98

80.84  
77.16  
41.57  
36.35  
30.99  
25.93  
25.57  
21.70  
17.23









— 144.91  
— 135.61  
— 133.83  
— 132.28  
— 130.67  
— 129.91  
— 126.90  
— 126.72  
— 126.54  
— 122.12  
— 112.98  
— 109.19

— 80.20  
— 77.16

— 41.08

— 36.18

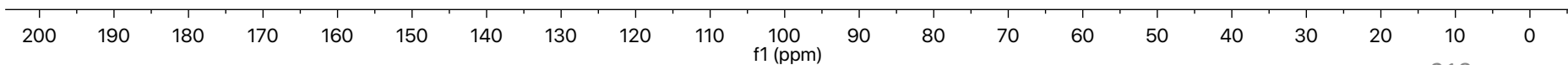
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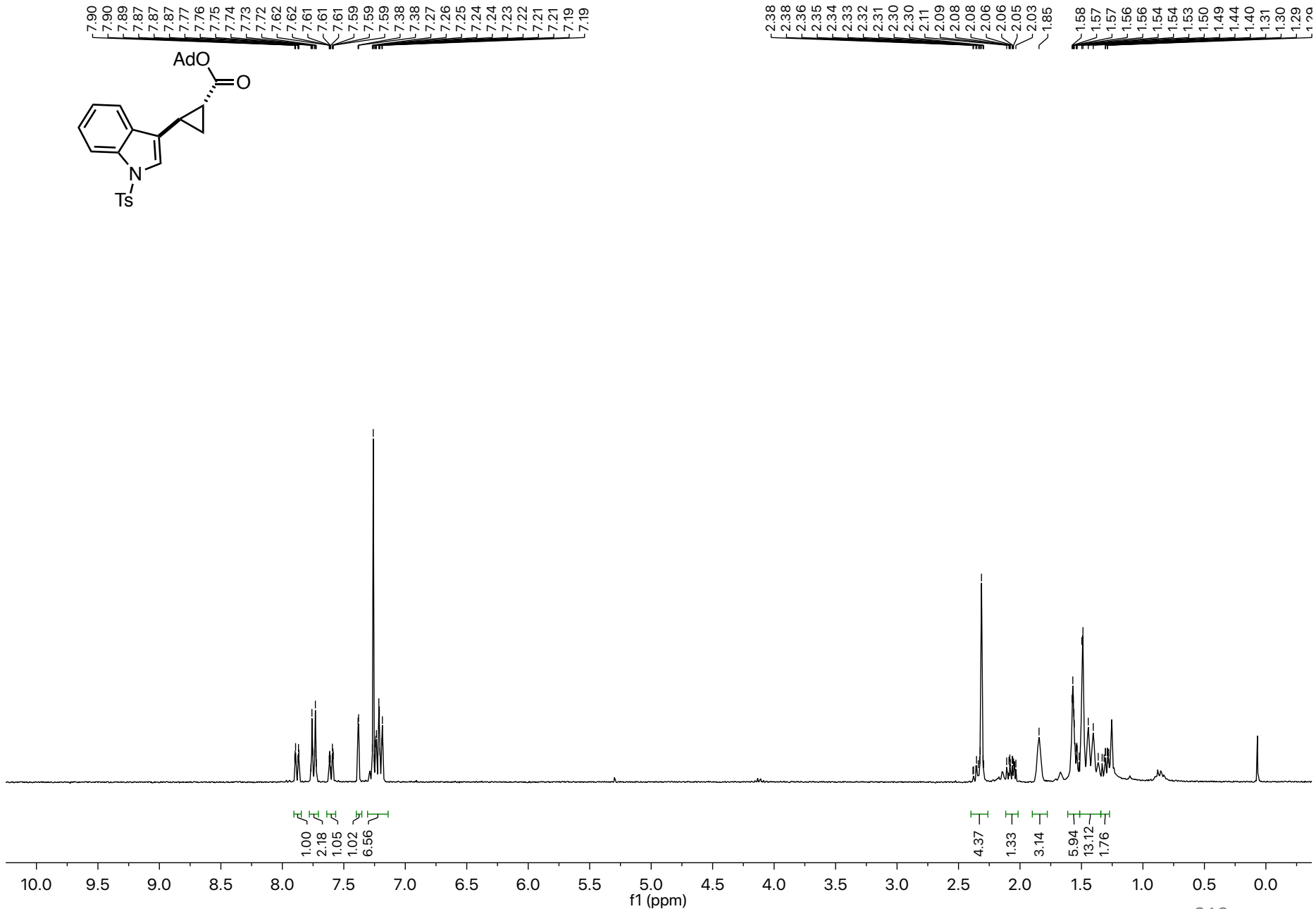
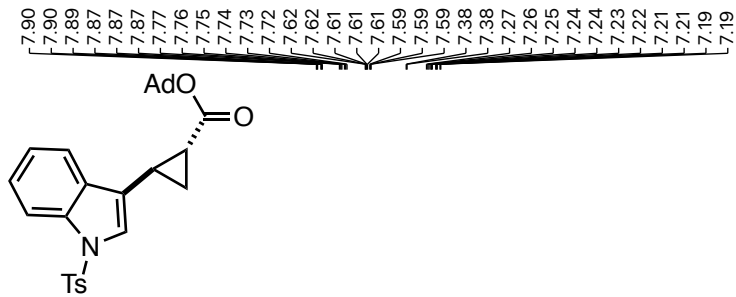
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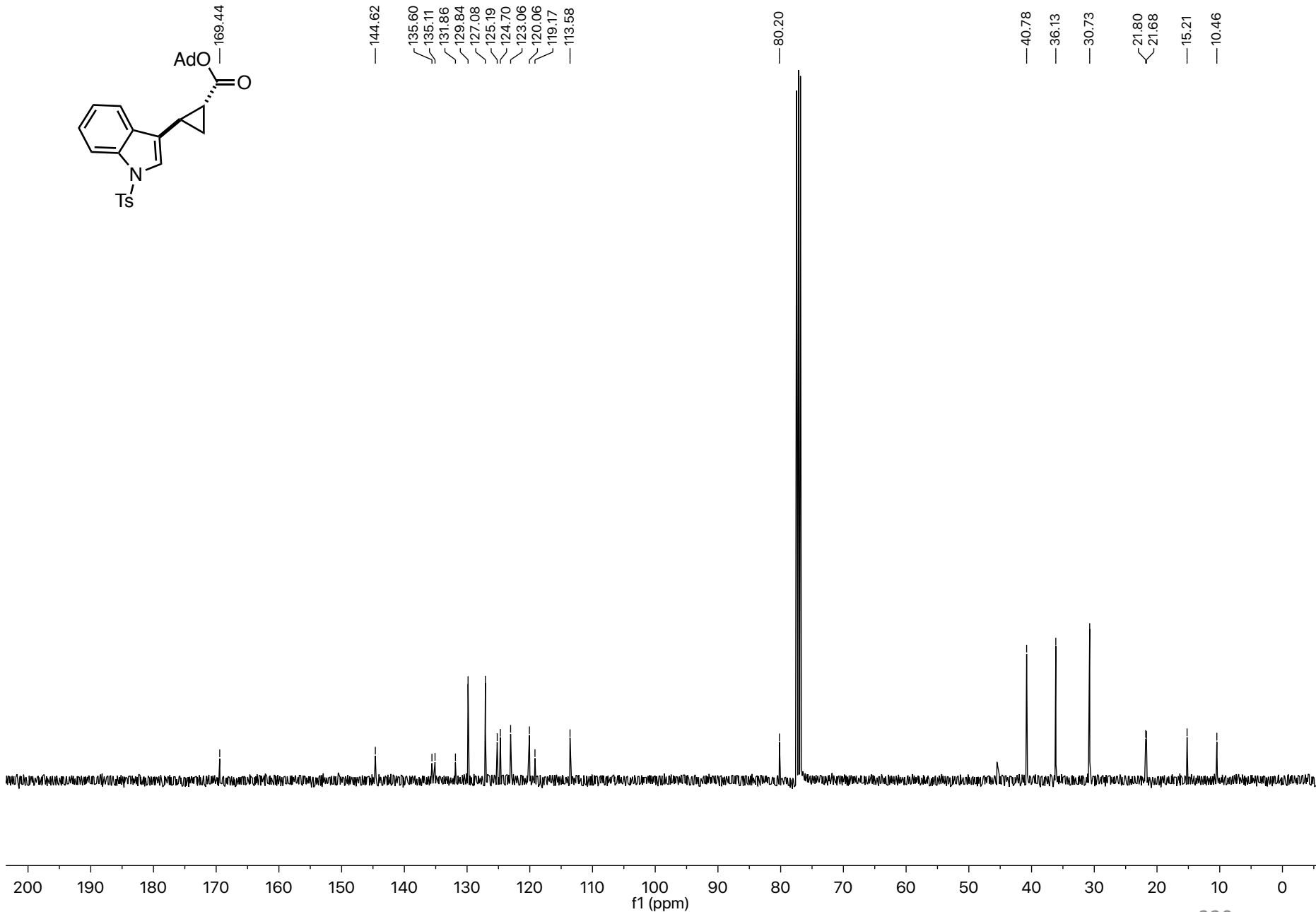
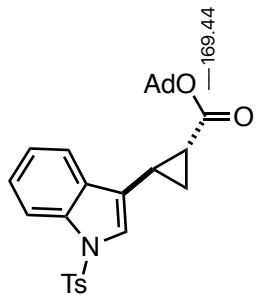
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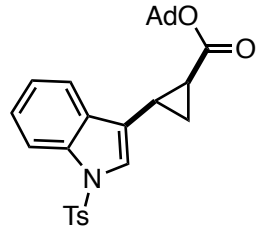
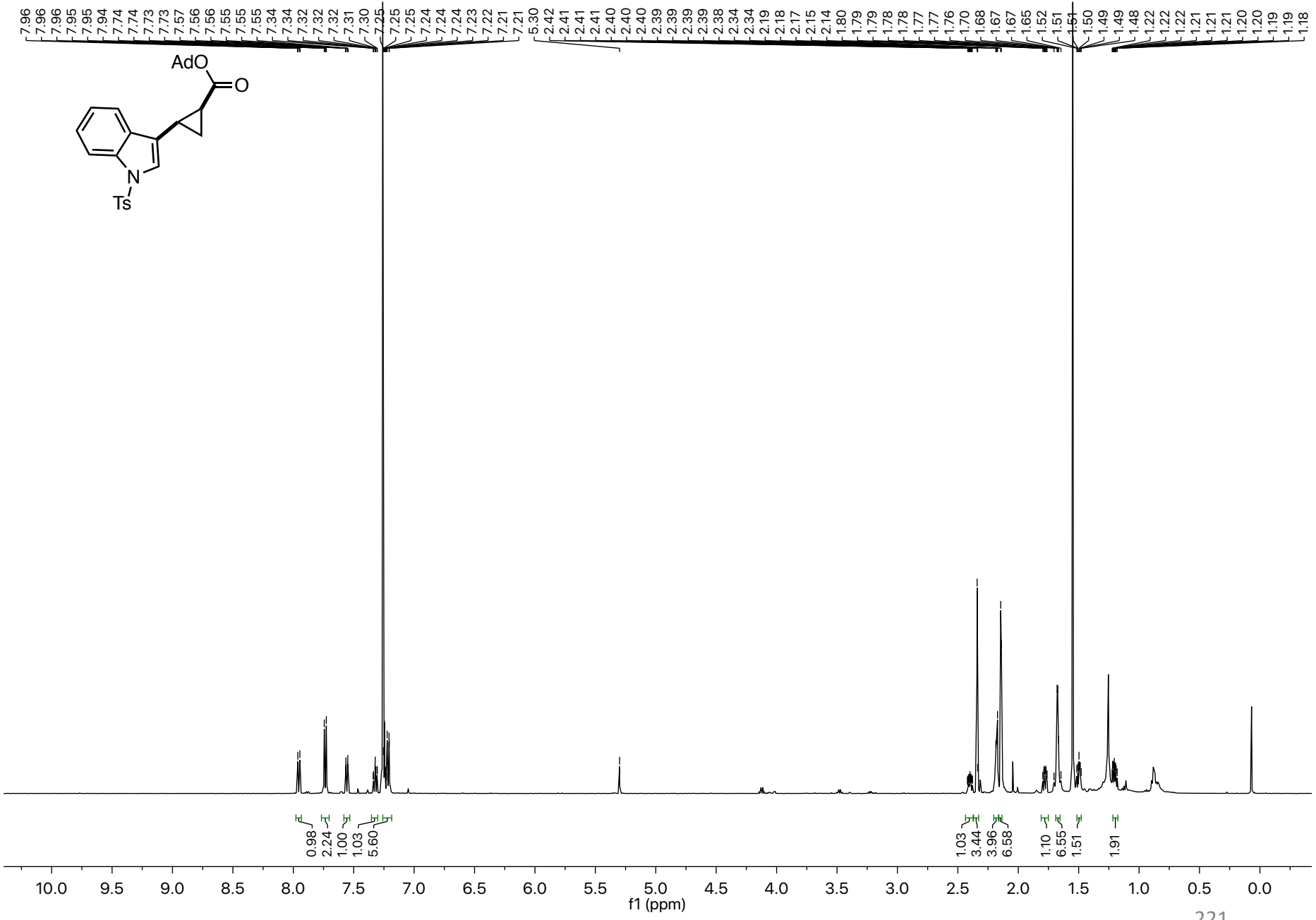
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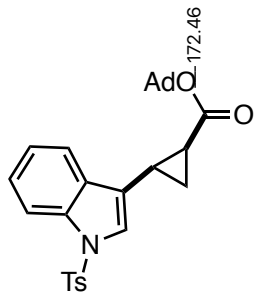
— 11.09











145.06  
135.40  
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130.03  
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125.11  
123.35  
122.82  
122.26  
119.70  
113.87

81.04

41.58

36.35

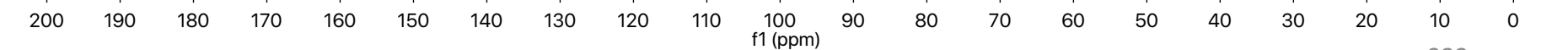
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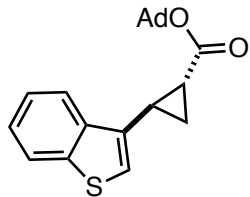
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16.76

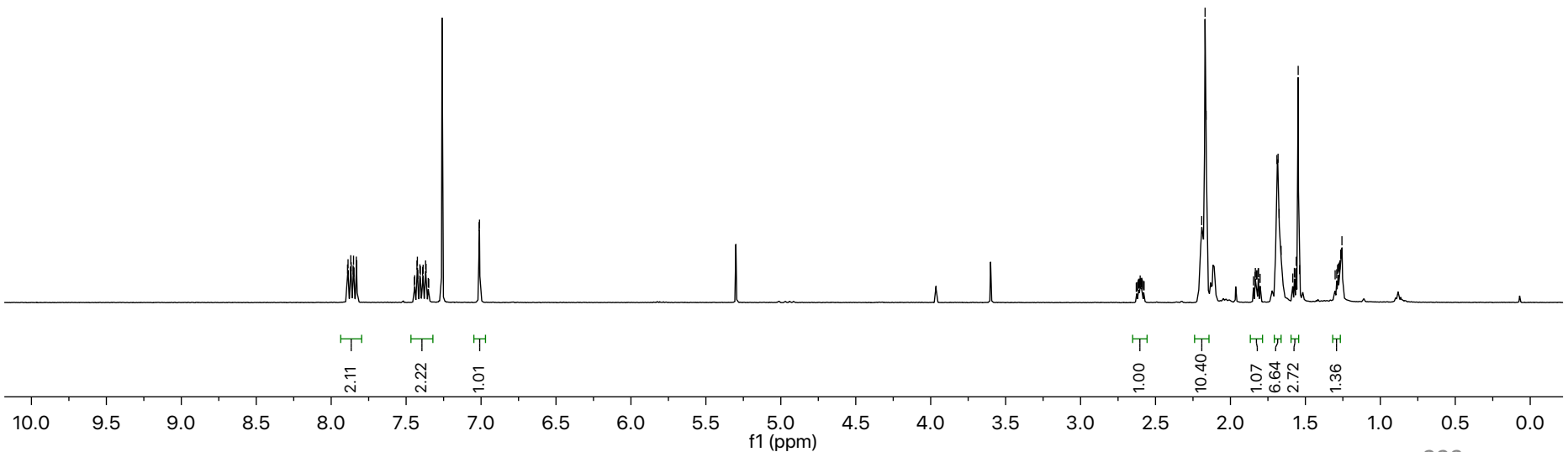
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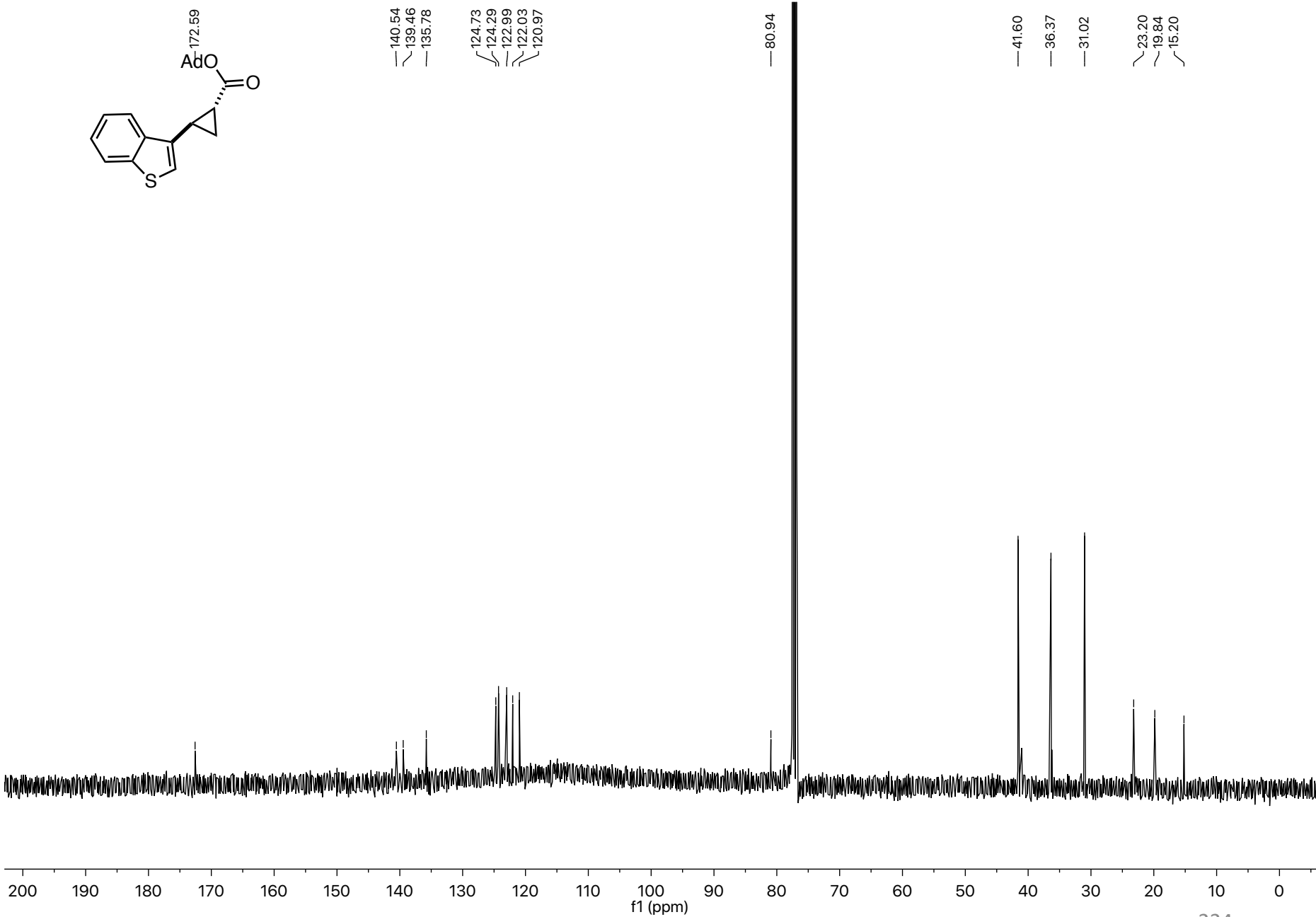
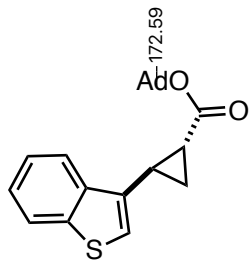


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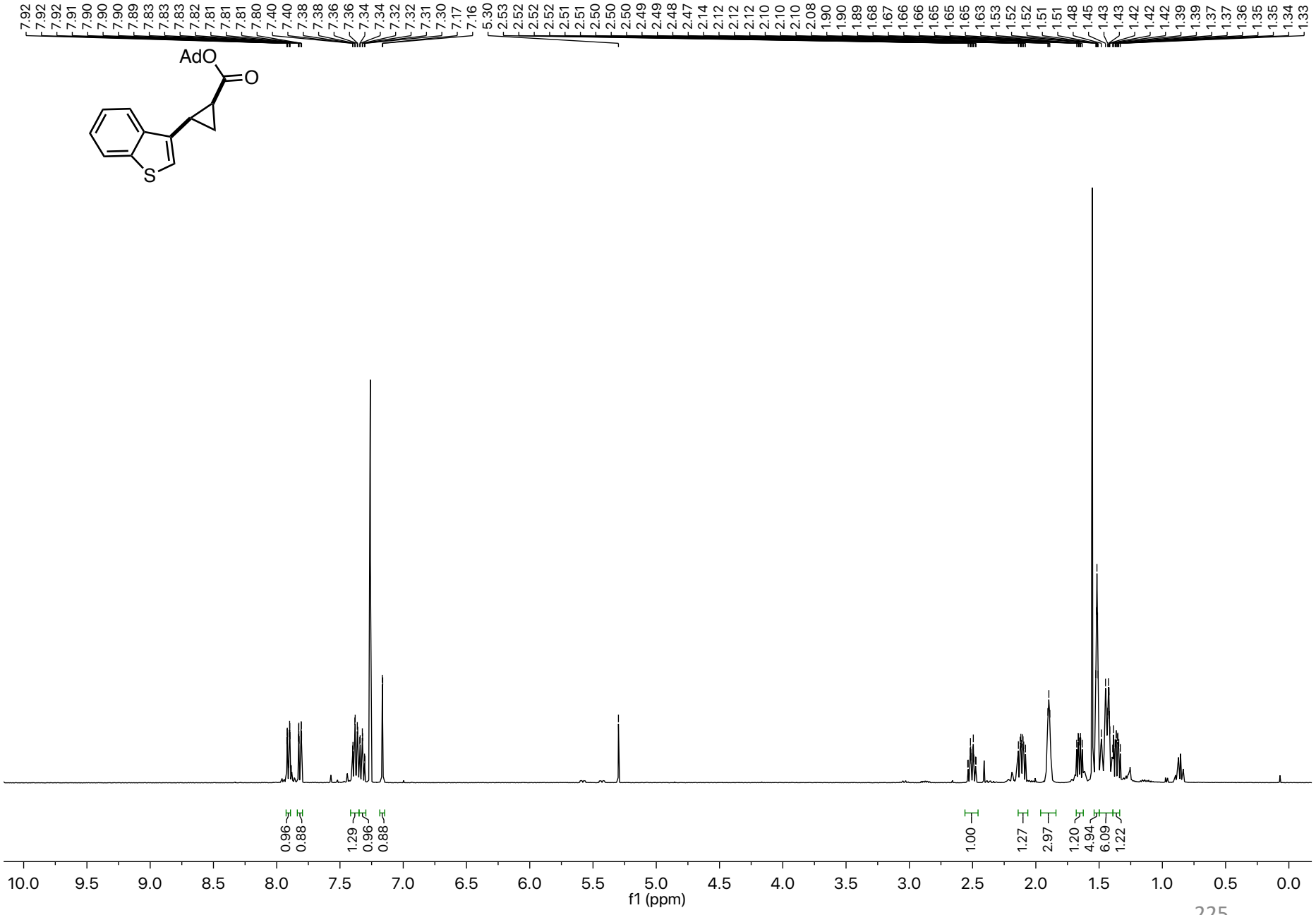


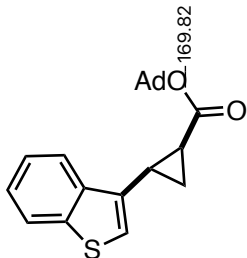
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1.83  
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1.81  
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1.26  
1.26











140.24  
139.96  
— 132.77  
124.39  
124.09  
124.02  
122.77  
122.37

— 80.23

— 40.79

— 36.17

— 30.75

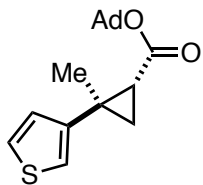
— 21.87

— 18.72

— 10.87

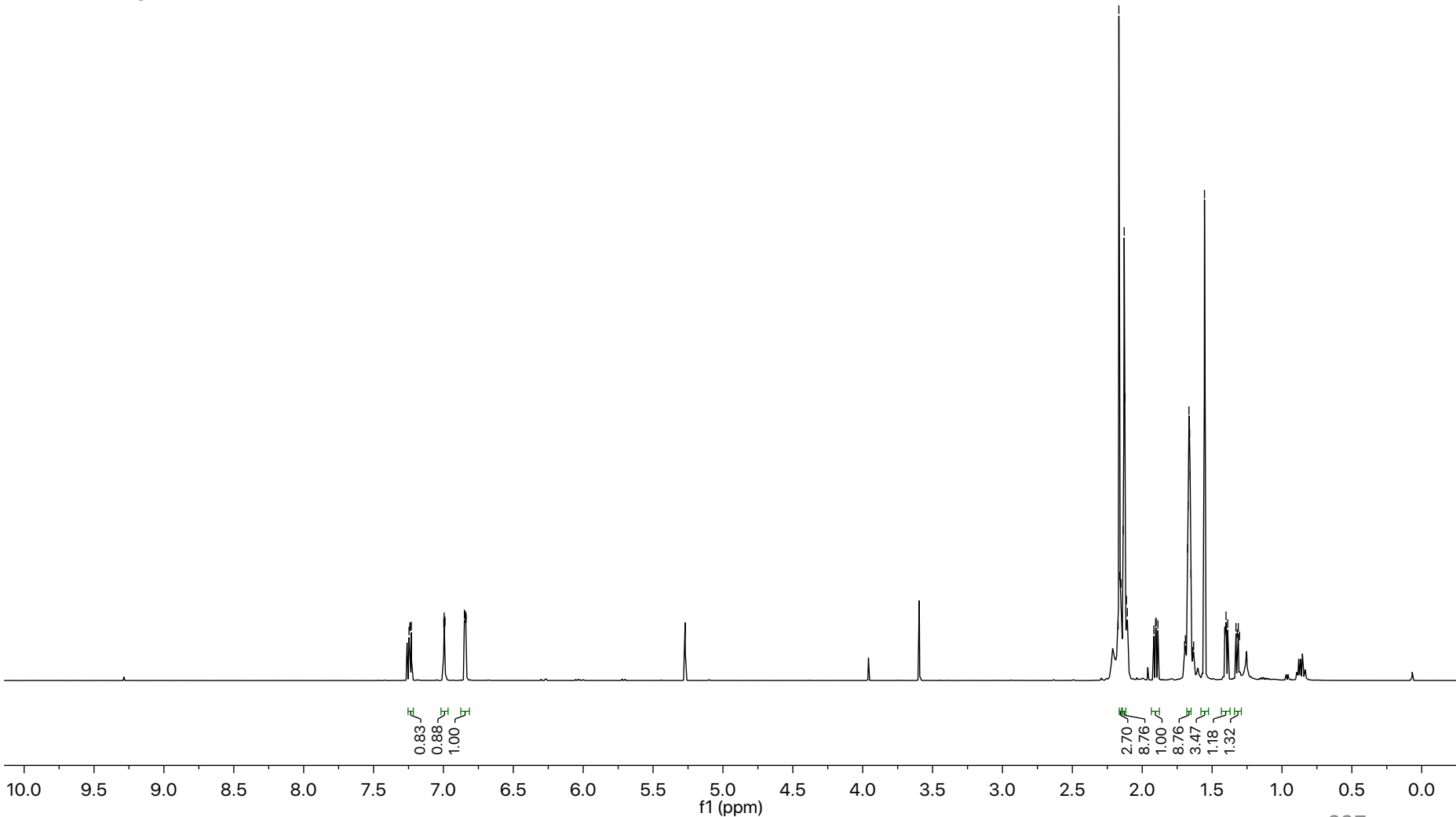
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f1 (ppm)



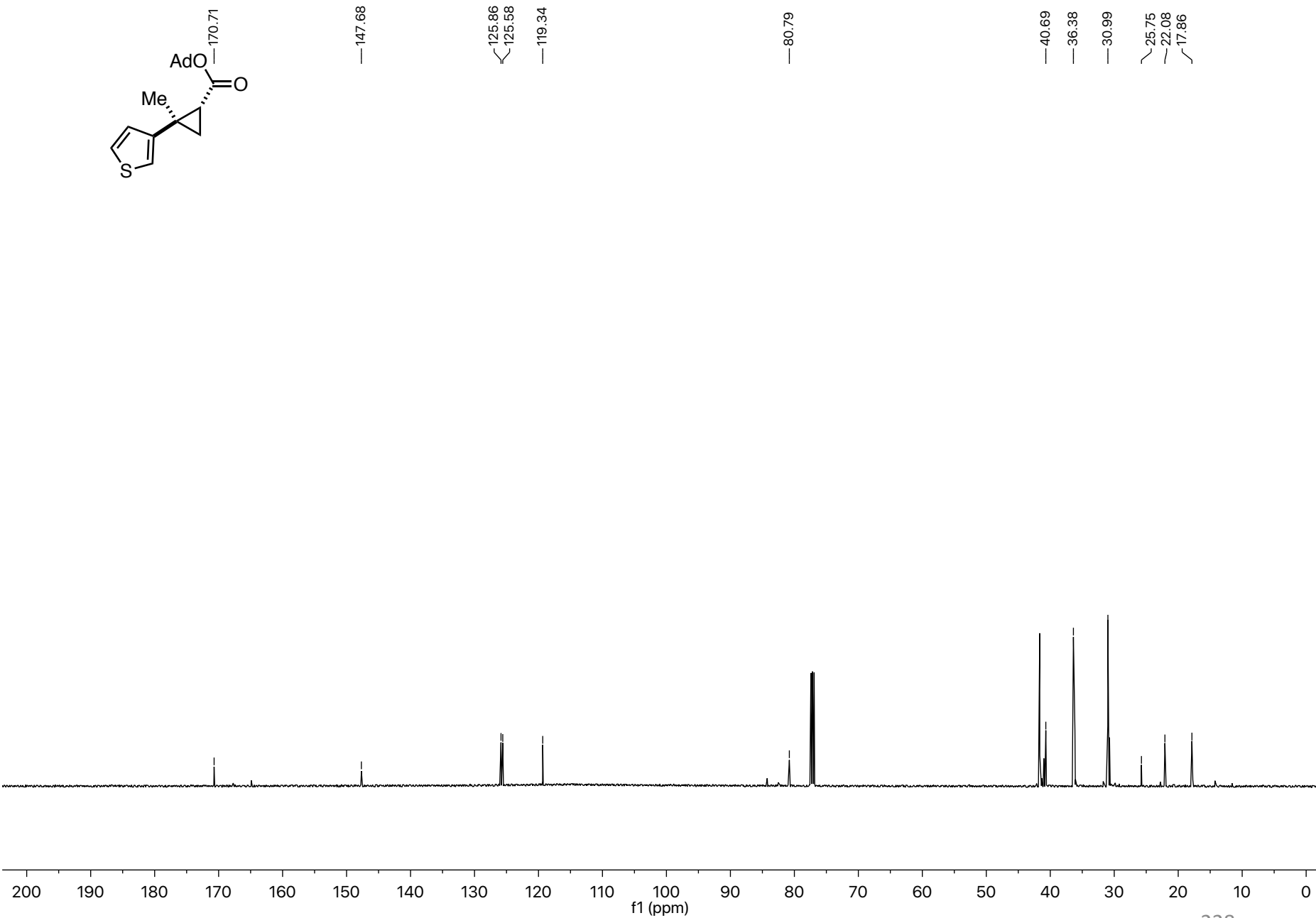
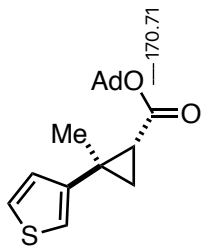
7.25  
7.24  
7.24  
7.23  
7.00  
6.99  
6.99  
6.85  
6.85  
6.84  
6.84

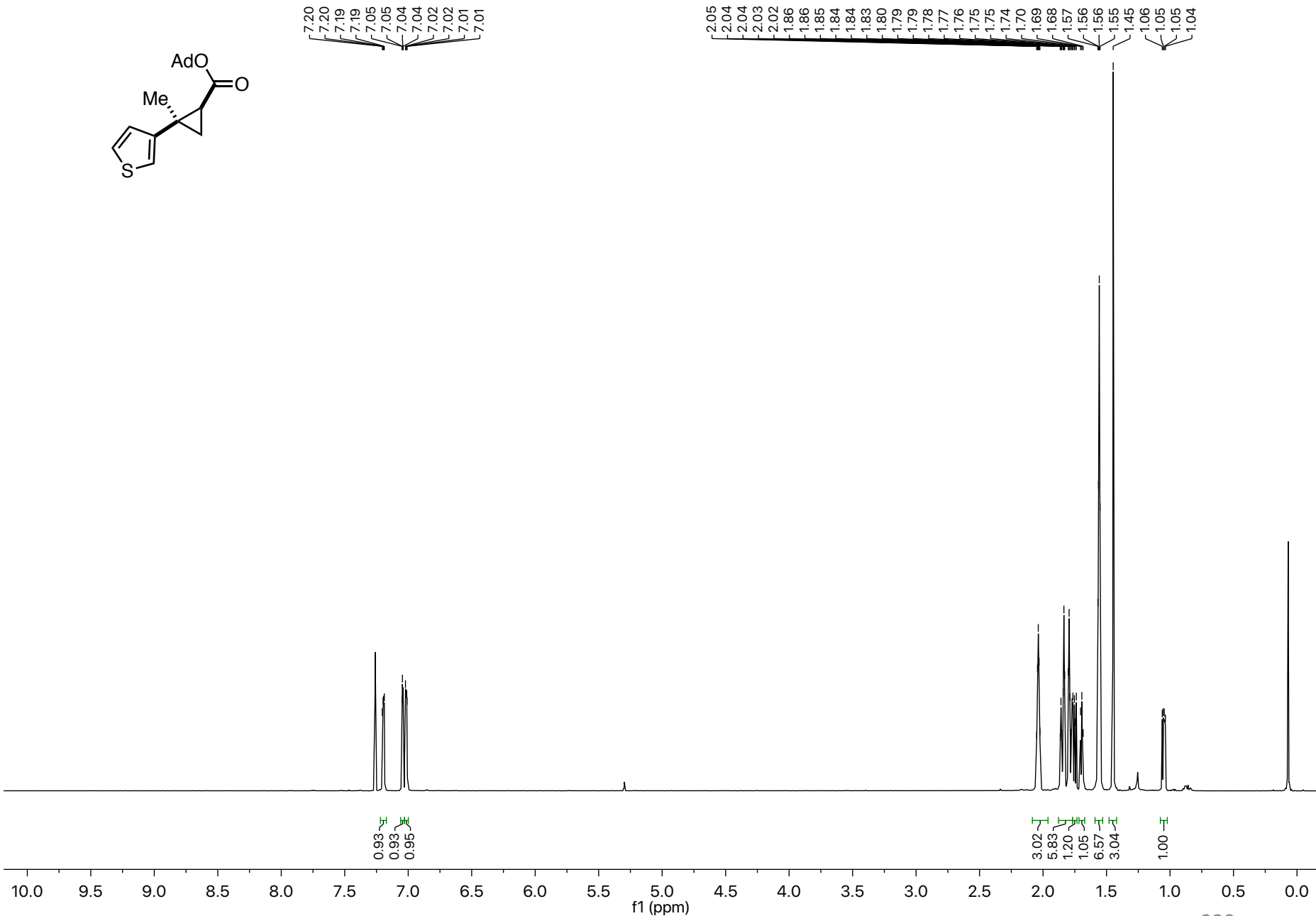
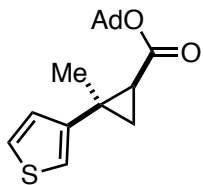
2.17  
2.17  
2.16  
2.15  
2.14  
2.13  
2.12  
2.11  
2.11  
1.92  
1.90  
1.90  
1.89  
1.70  
1.69  
1.67  
1.67  
1.66  
1.66  
1.65  
1.64  
1.63  
1.55  
1.41  
1.40  
1.40  
1.39  
1.33  
1.32  
1.31  
1.30

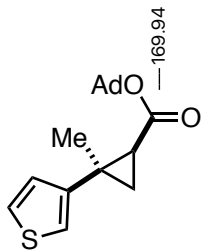


0.83  
0.88  
1.00

2.70  
8.76  
1.00  
8.76  
3.47  
1.18  
1.32







— 142.88

~ 128.48

~ 124.82

~ 122.16

— 80.08

— 77.16

— 41.10

— 36.29

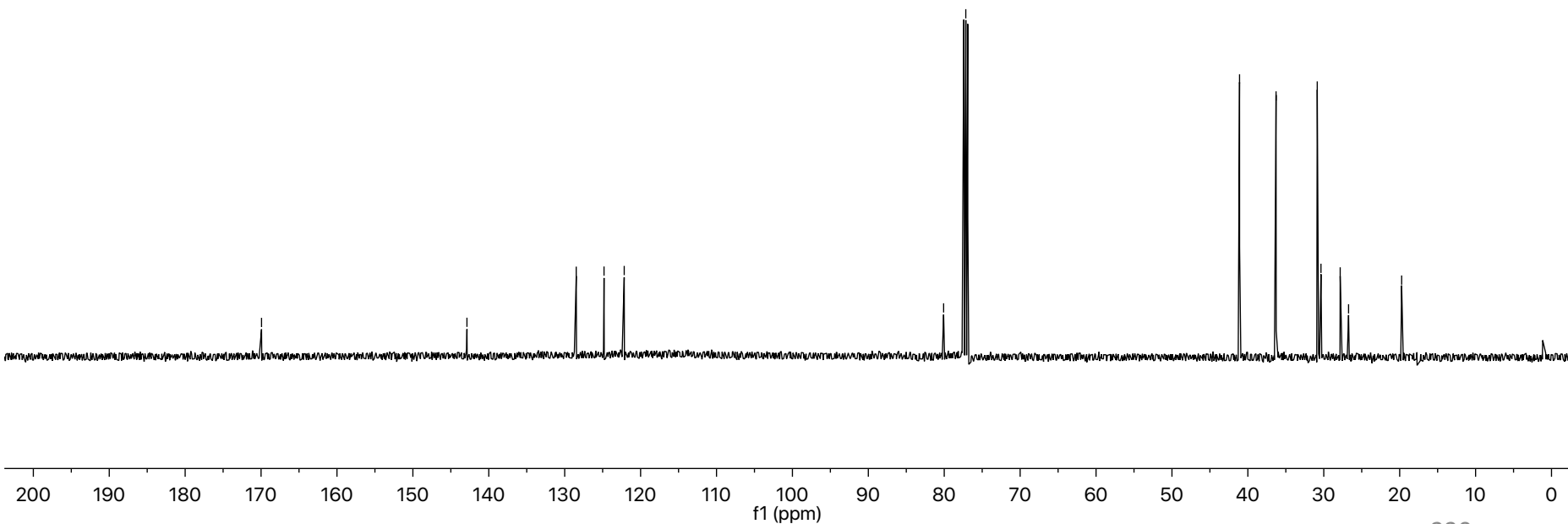
~ 30.86

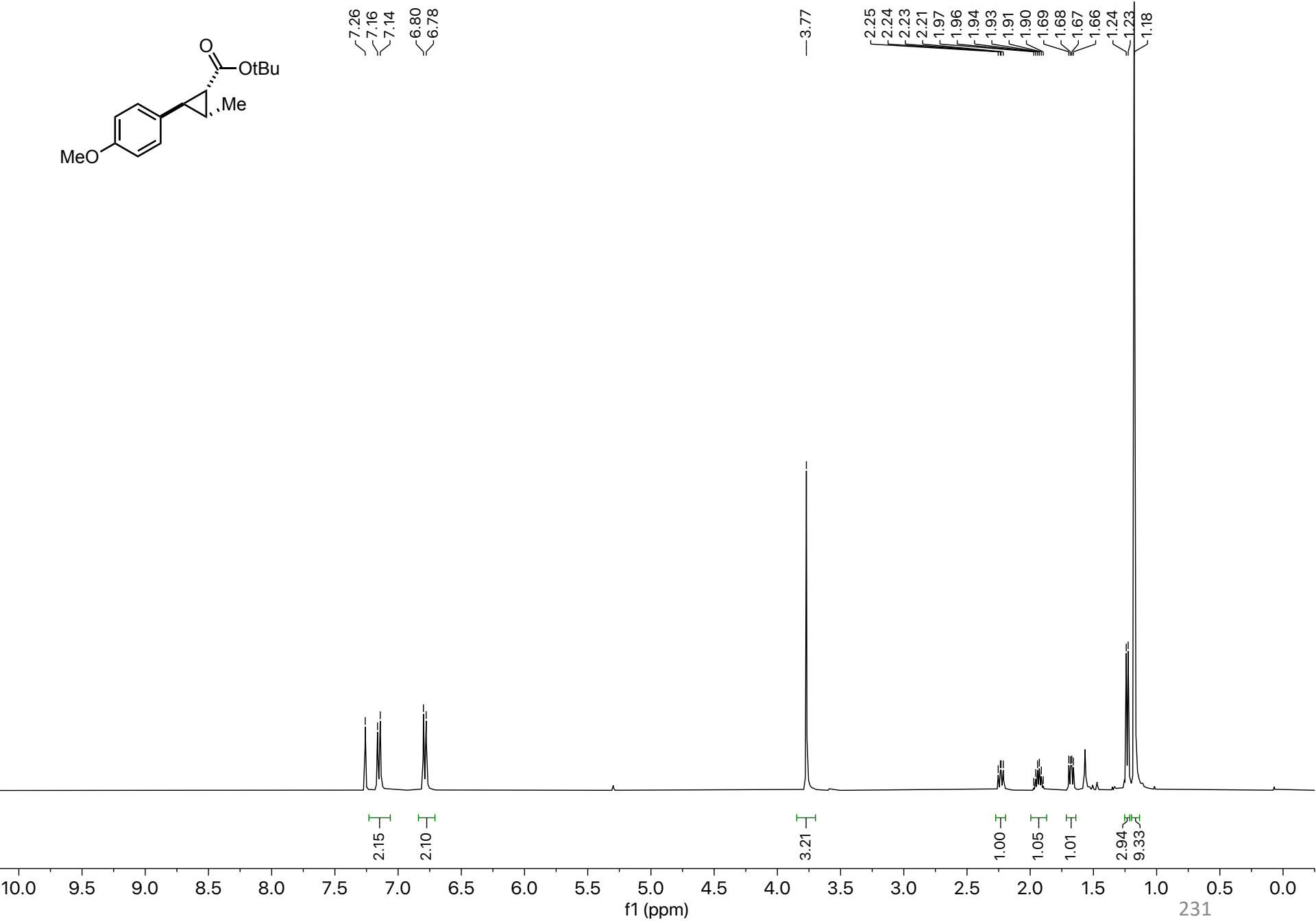
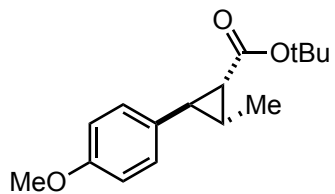
~ 30.37

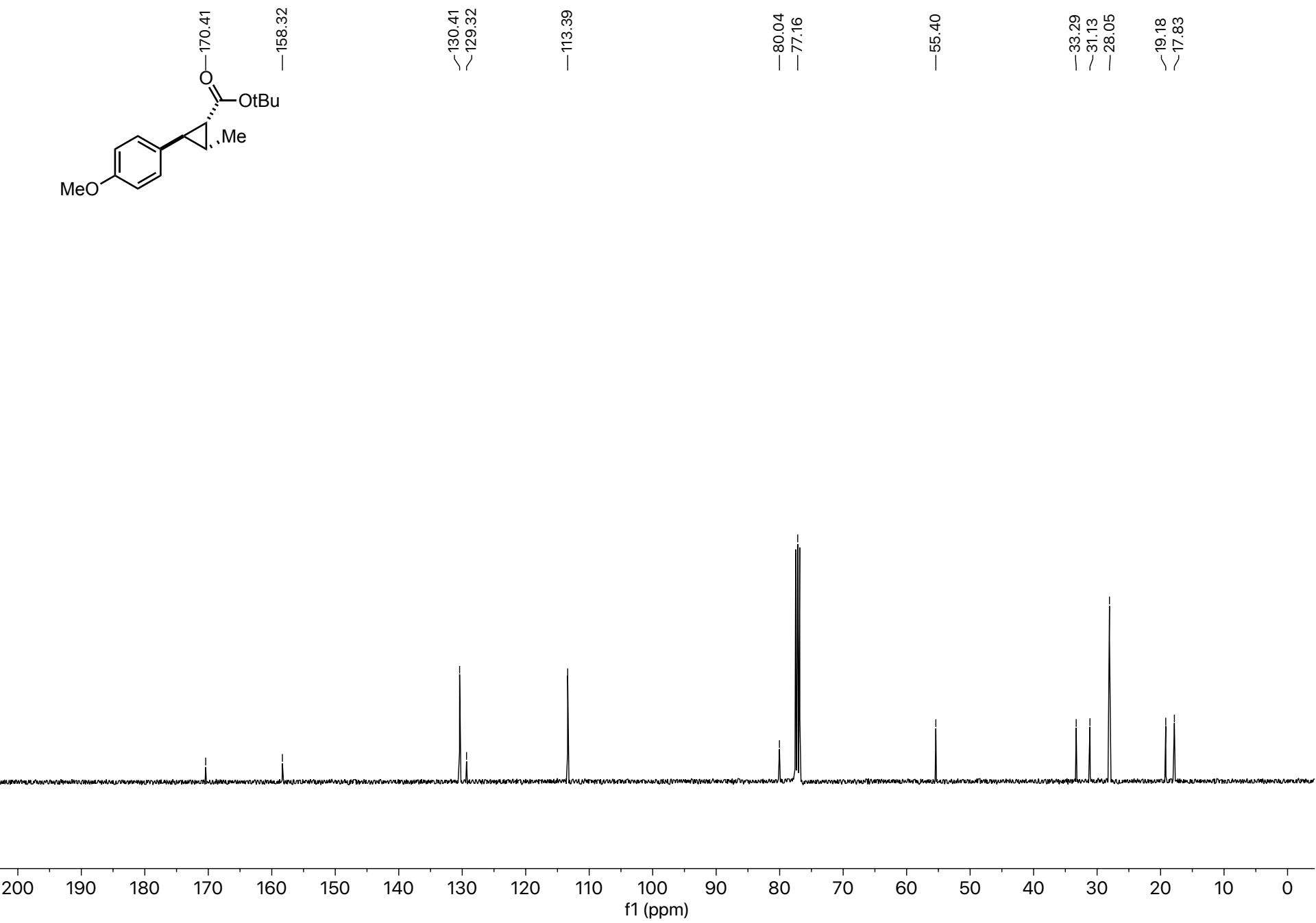
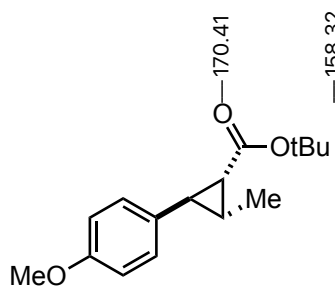
~ 27.84

~ 26.73

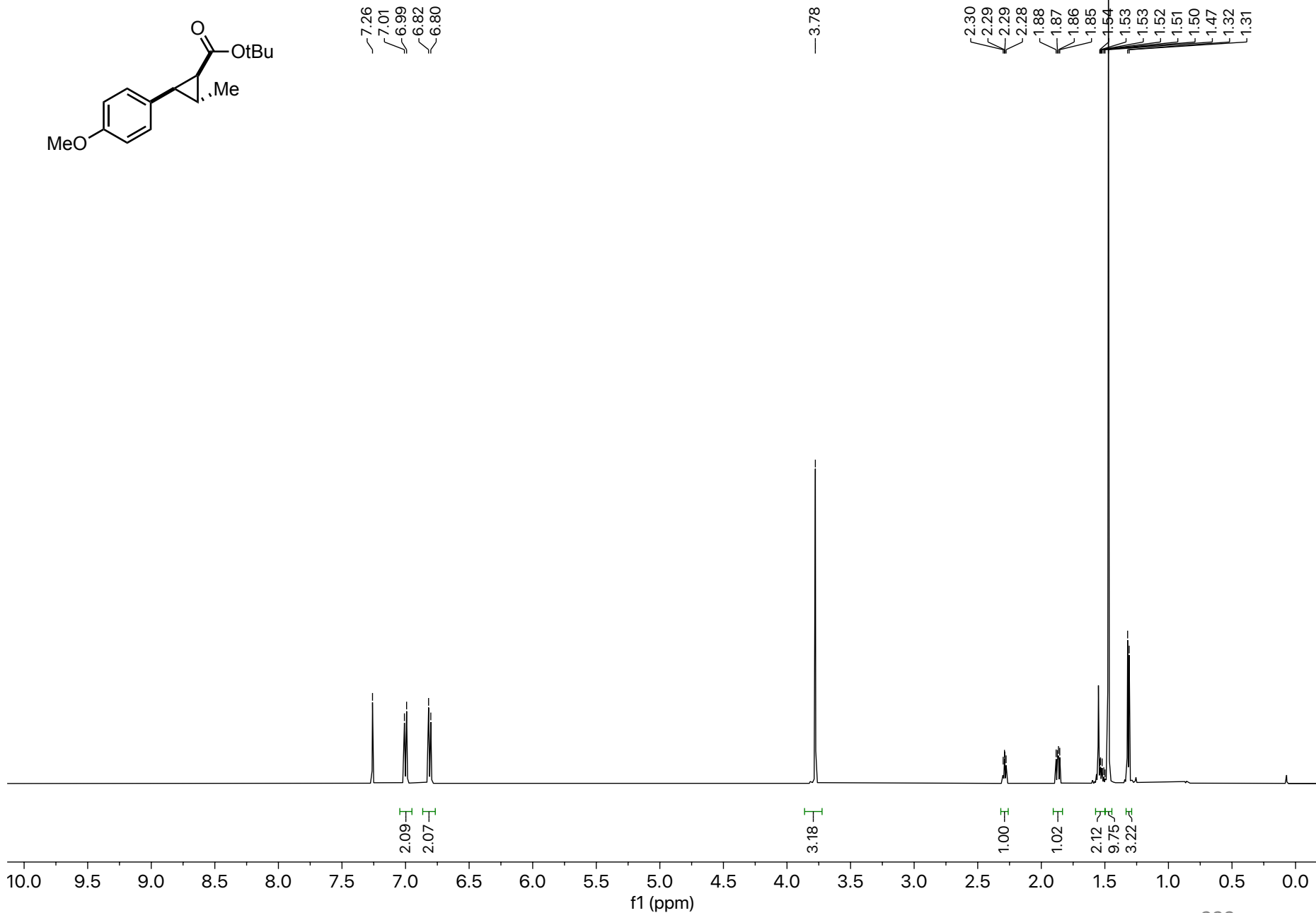
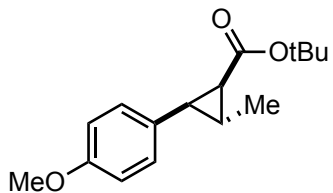
— 19.74

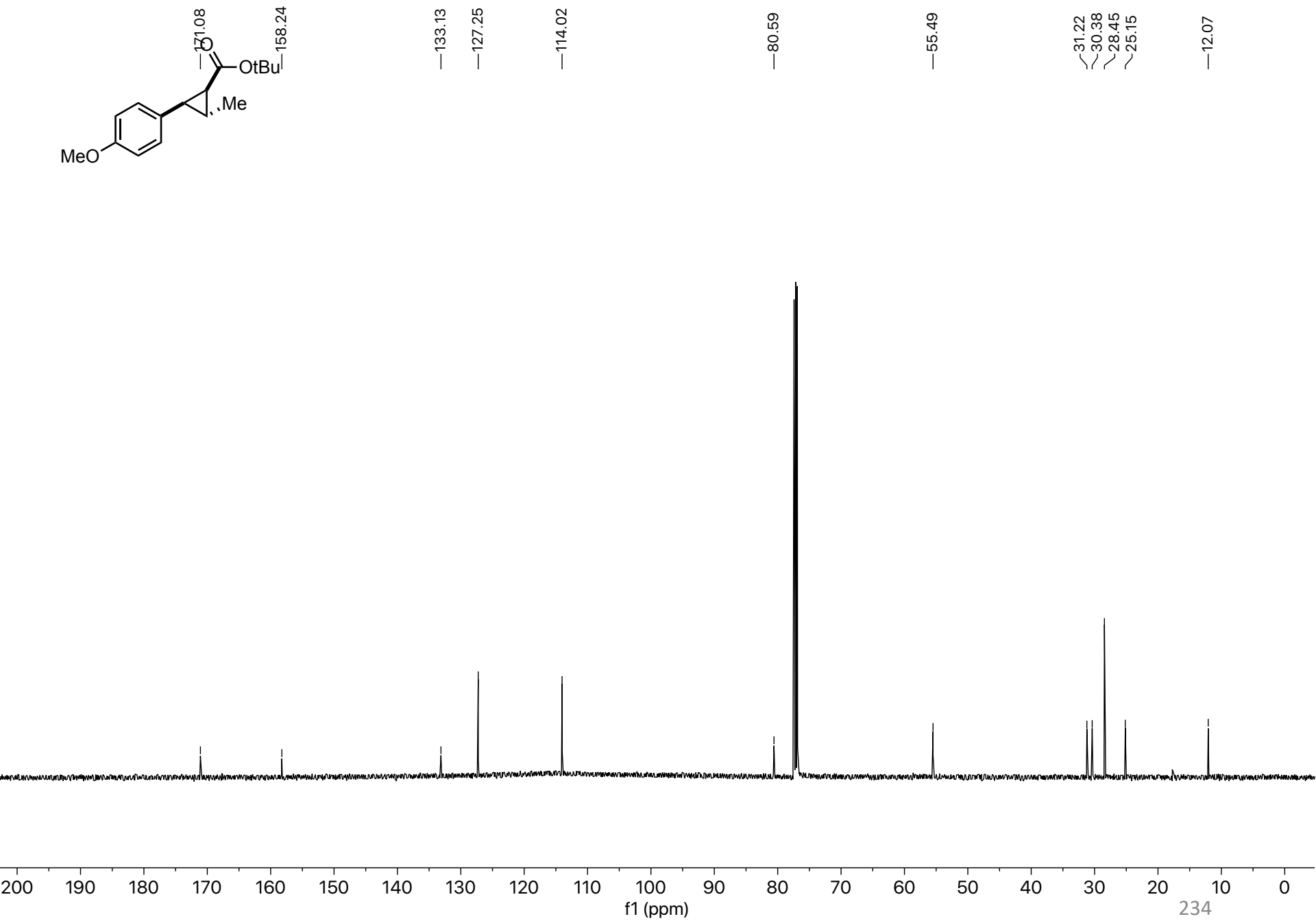
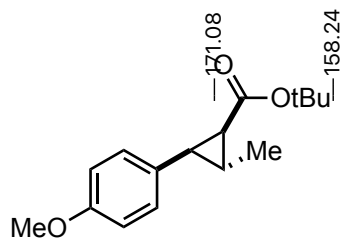


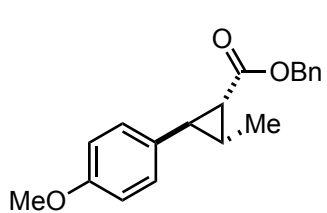










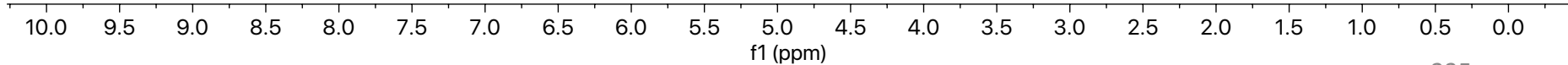


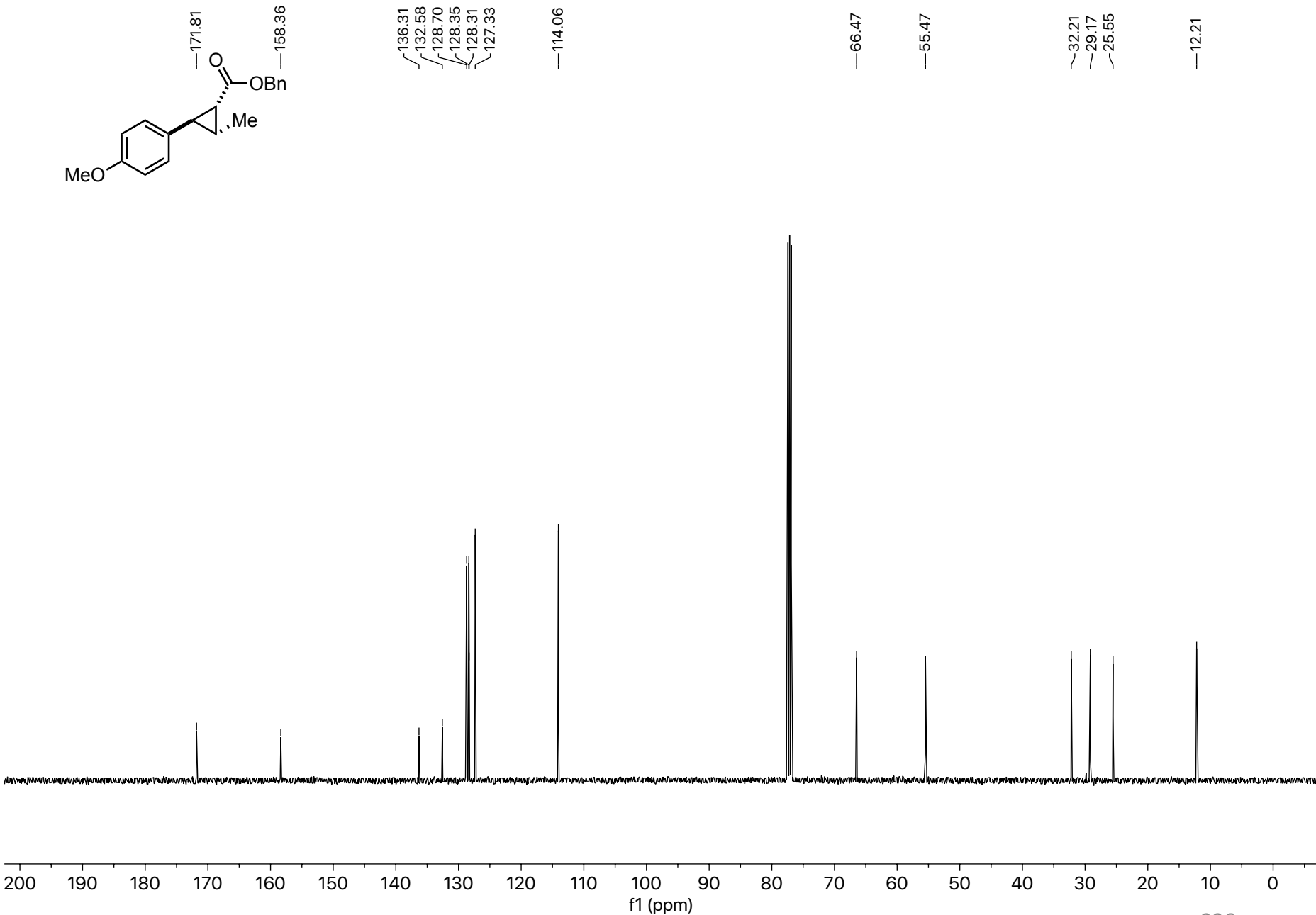
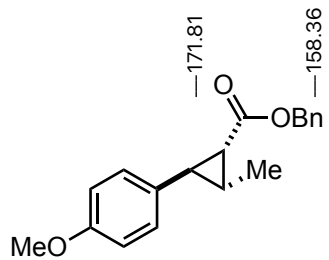
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7.38  
7.37  
7.37  
7.26  
7.01  
7.00  
6.82  
6.80

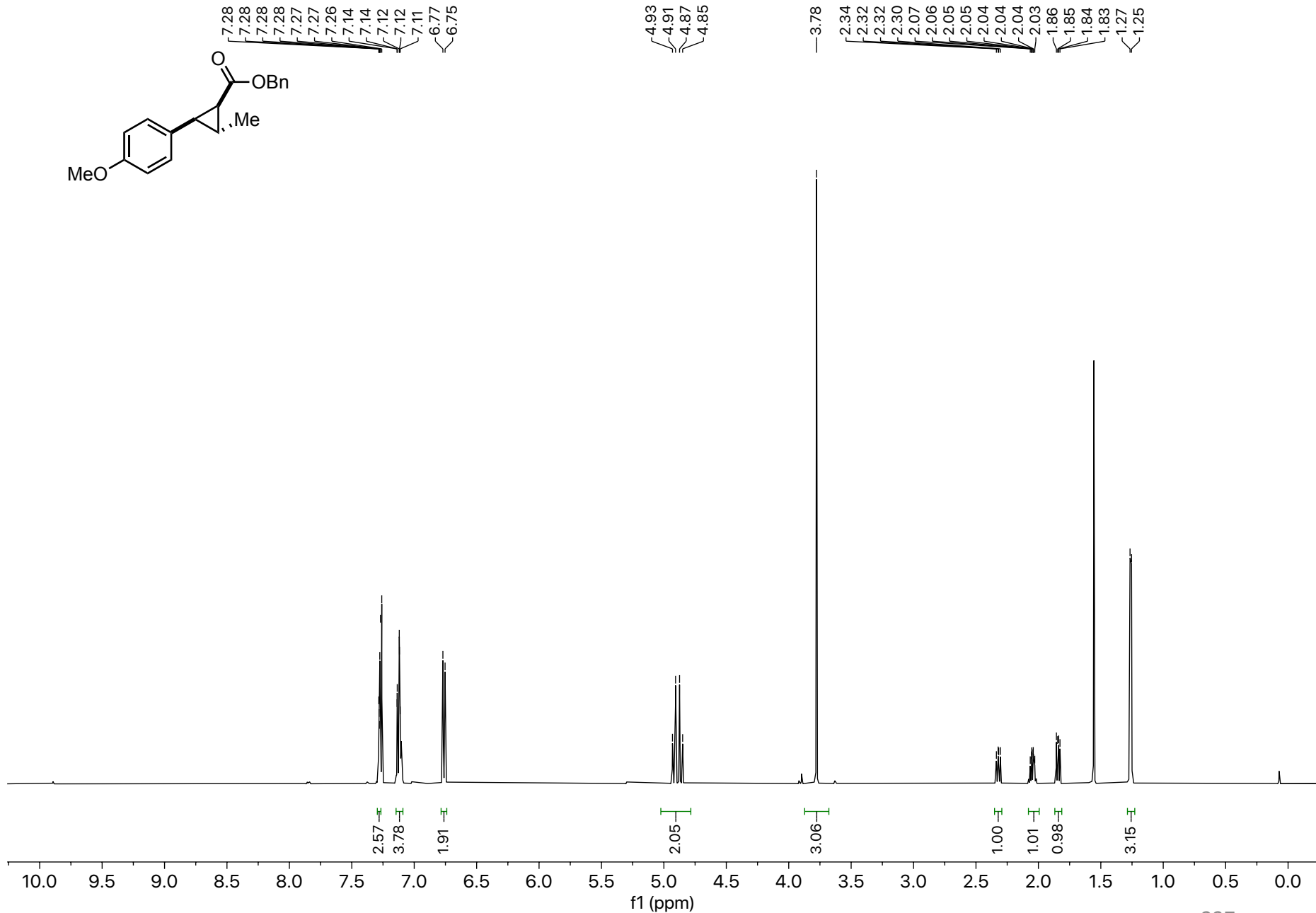
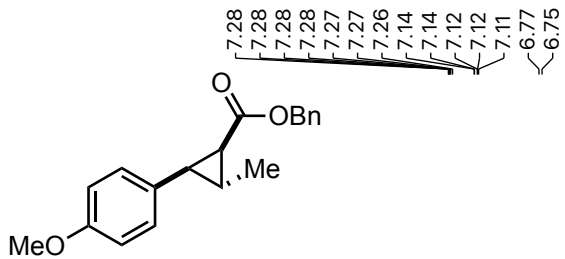
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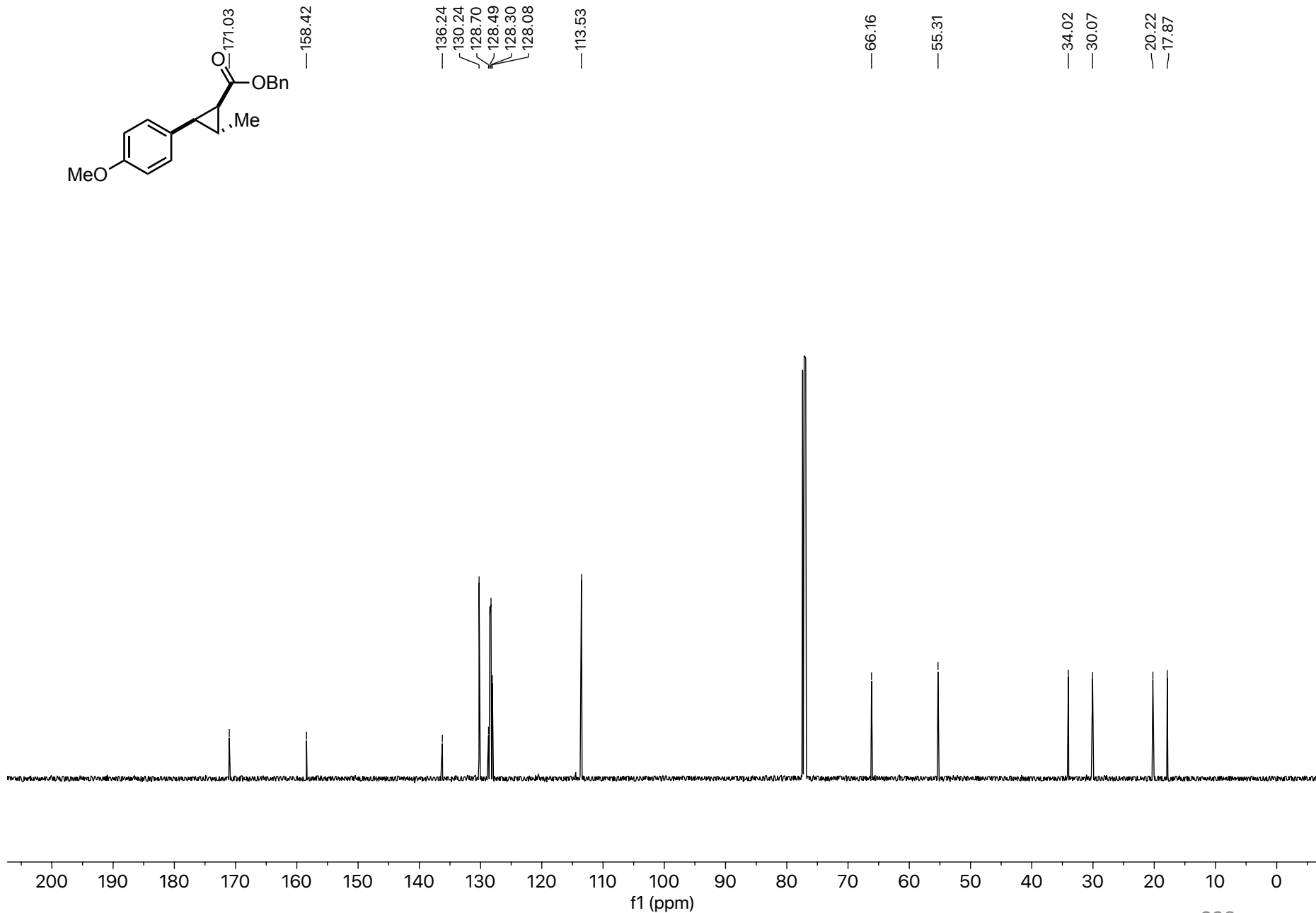
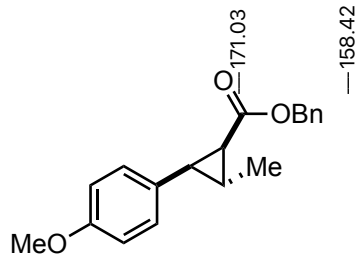
3.78

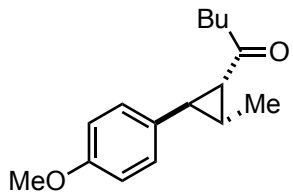
2.42  
2.41  
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2.39  
2.02  
2.01  
2.00  
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1.67  
1.66  
1.65  
1.64  
1.64  
1.63  
1.35  
1.34





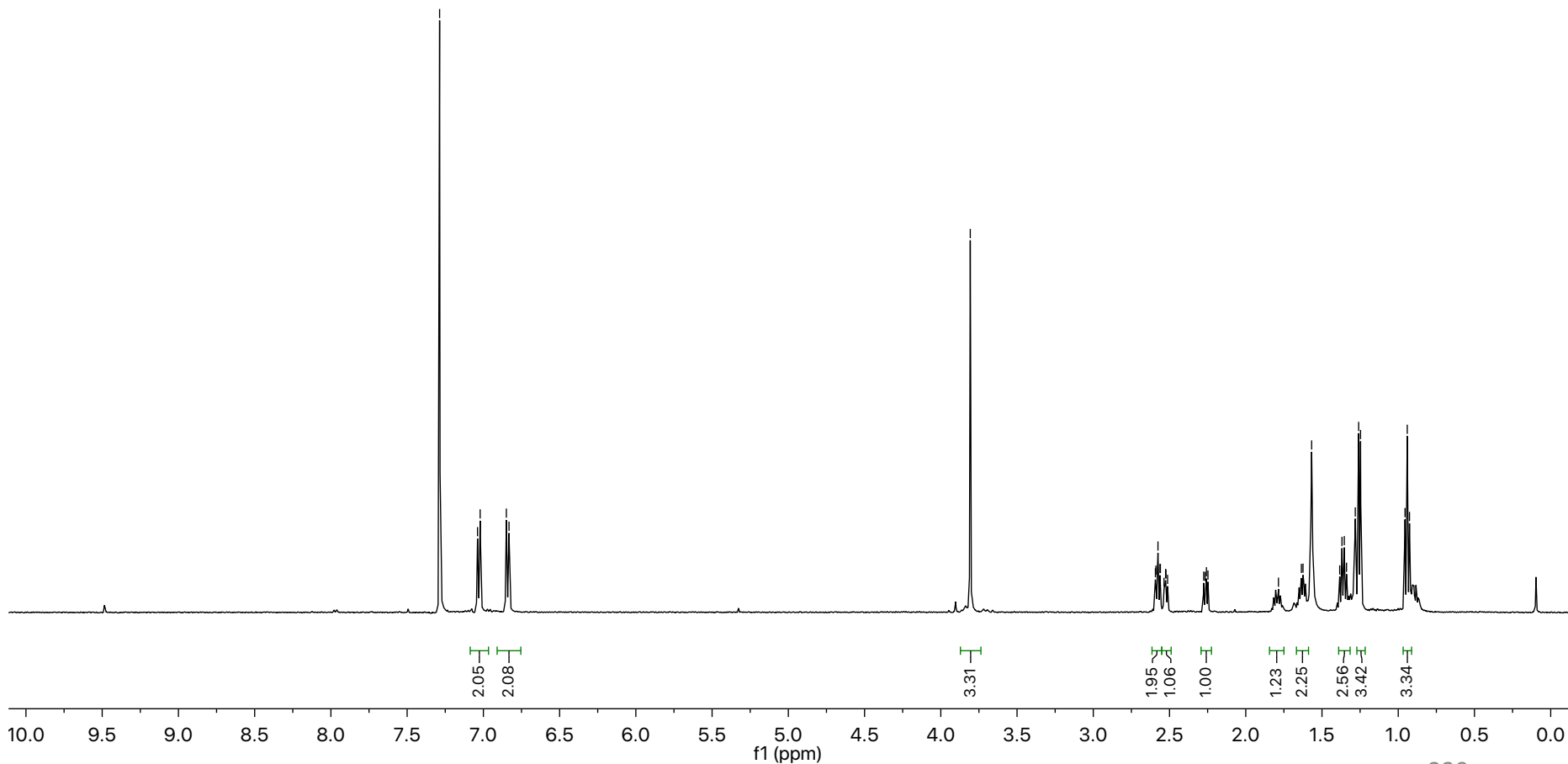


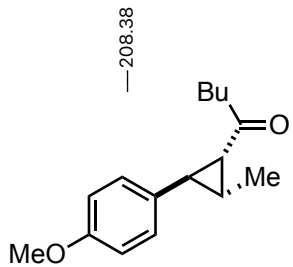




7.29  
7.04  
7.02  
6.85  
6.83

3.81  
2.59  
2.58  
2.56  
2.56  
2.54  
2.53  
2.52  
2.51  
2.28  
2.27  
2.26  
2.25  
1.79  
1.64  
1.62  
1.57  
1.38  
1.37  
1.35  
1.34  
1.28  
1.26  
1.25  
0.96  
0.94  
0.93





— 208.38

— 158.36

— 133.29

— 127.38

— 114.14

— 55.59

— 45.06

— 37.30

— 32.92

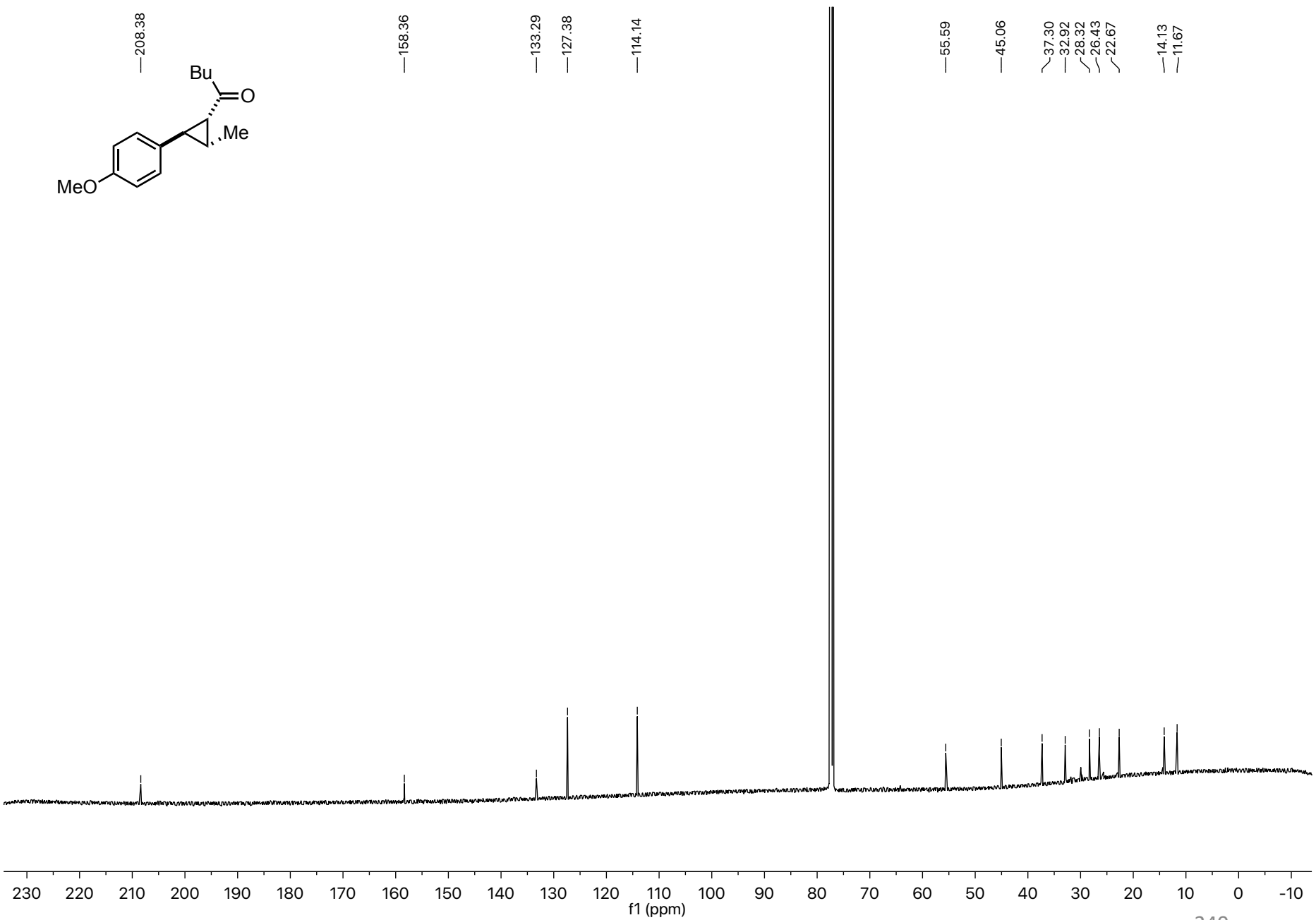
— 28.32

— 26.43

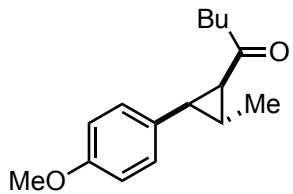
— 22.67

— 14.13

— 11.67





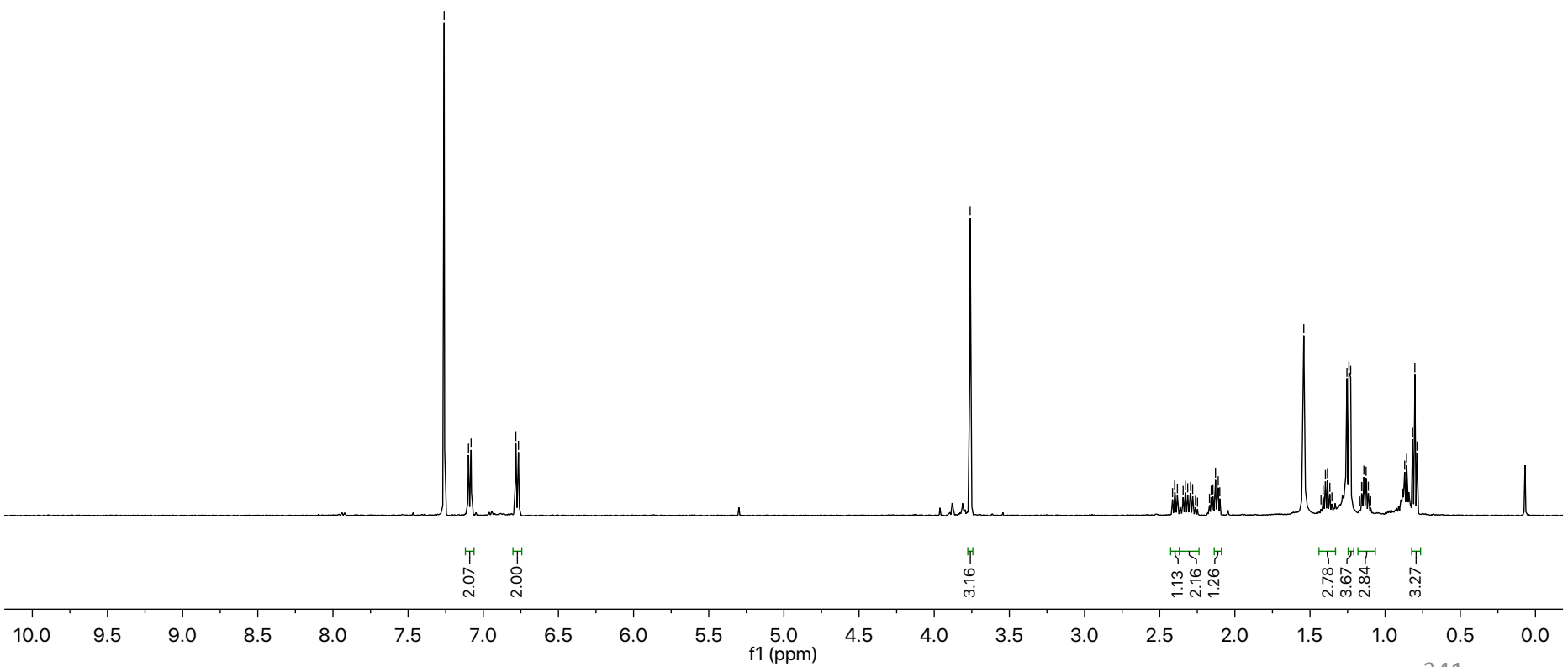


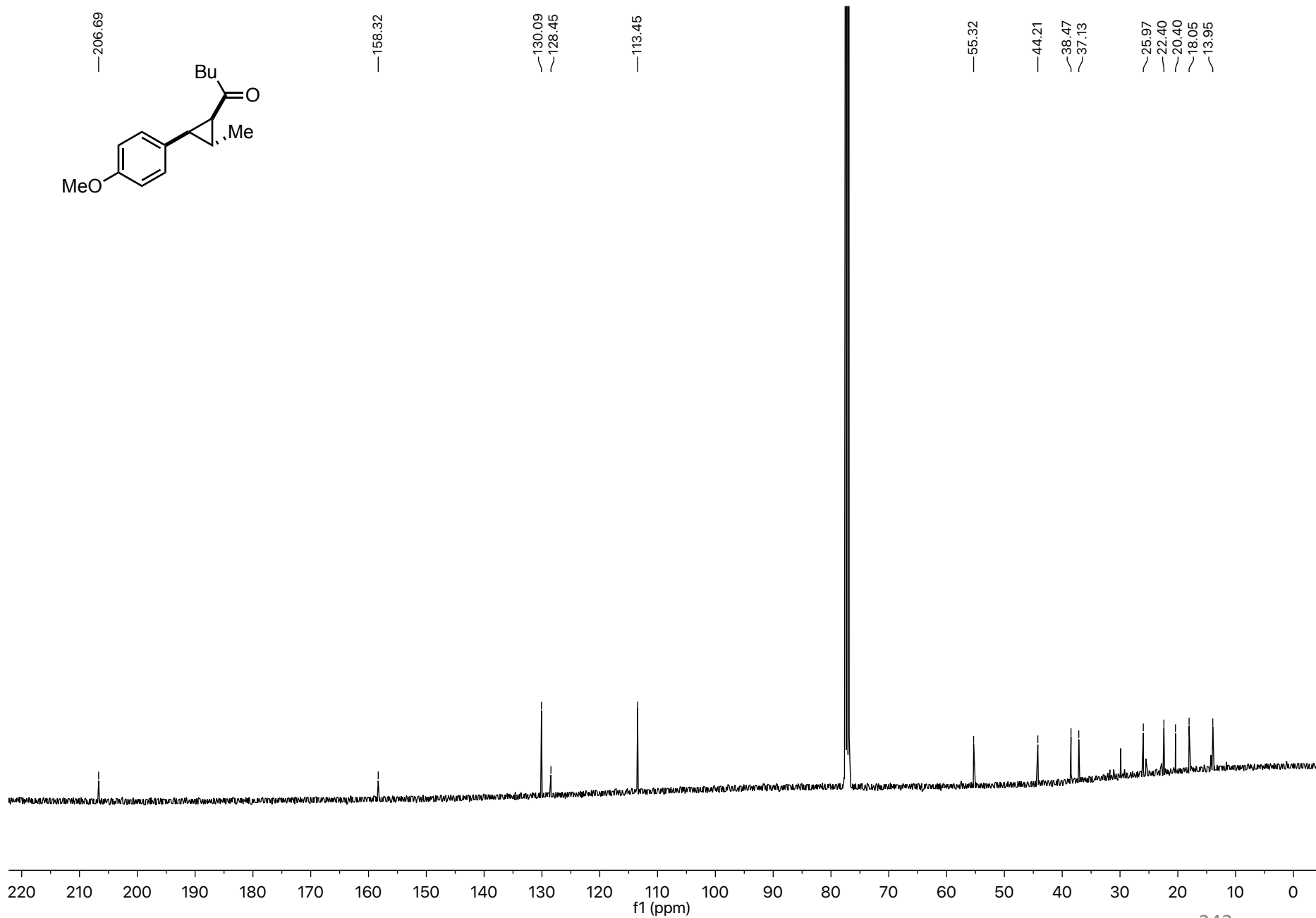
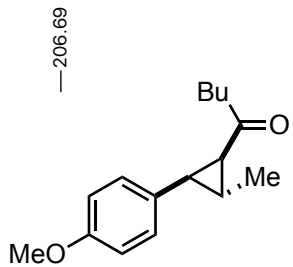
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7.08

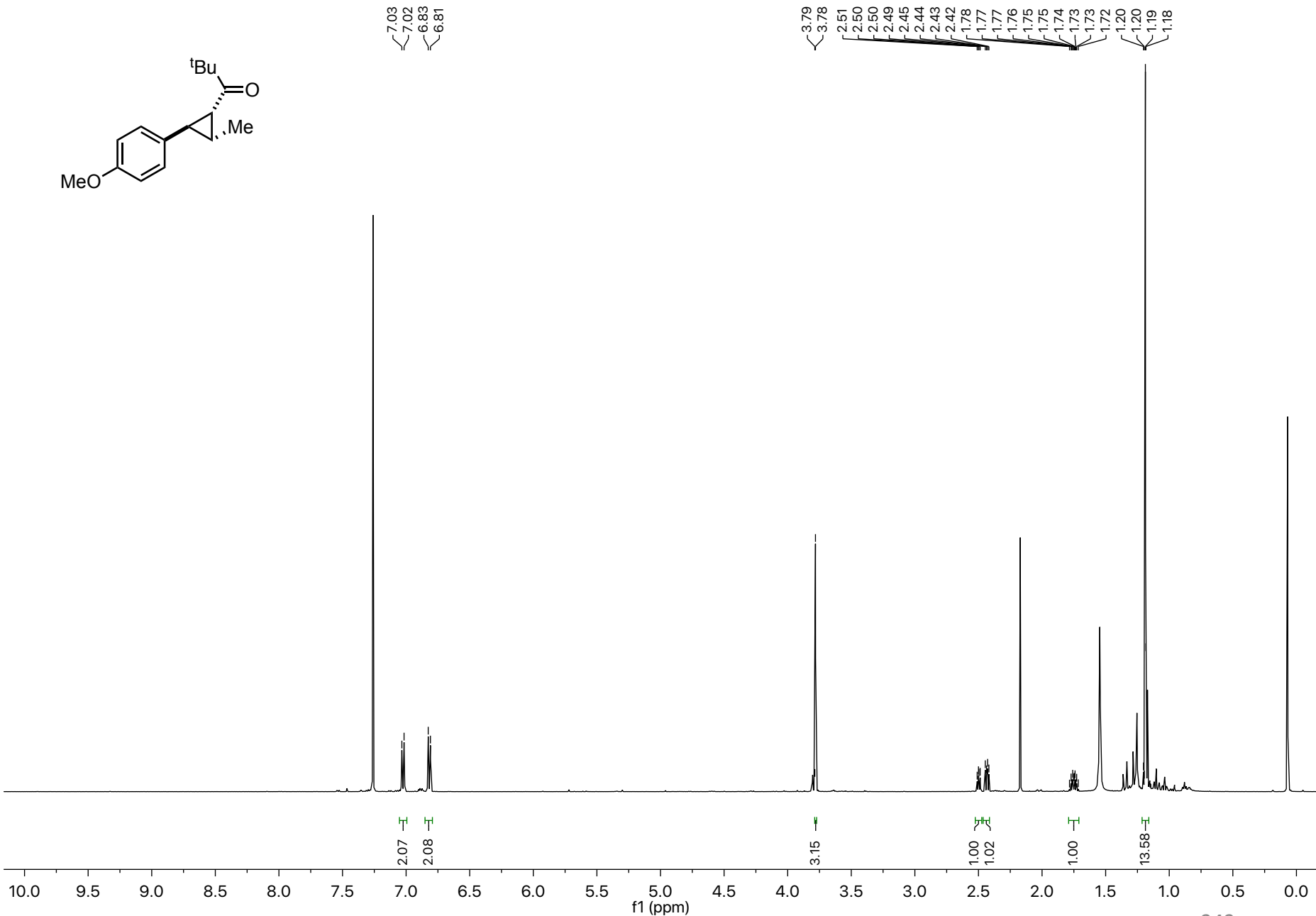
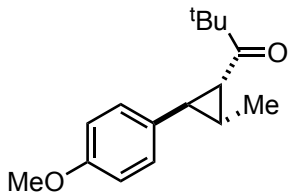
6.78  
6.77

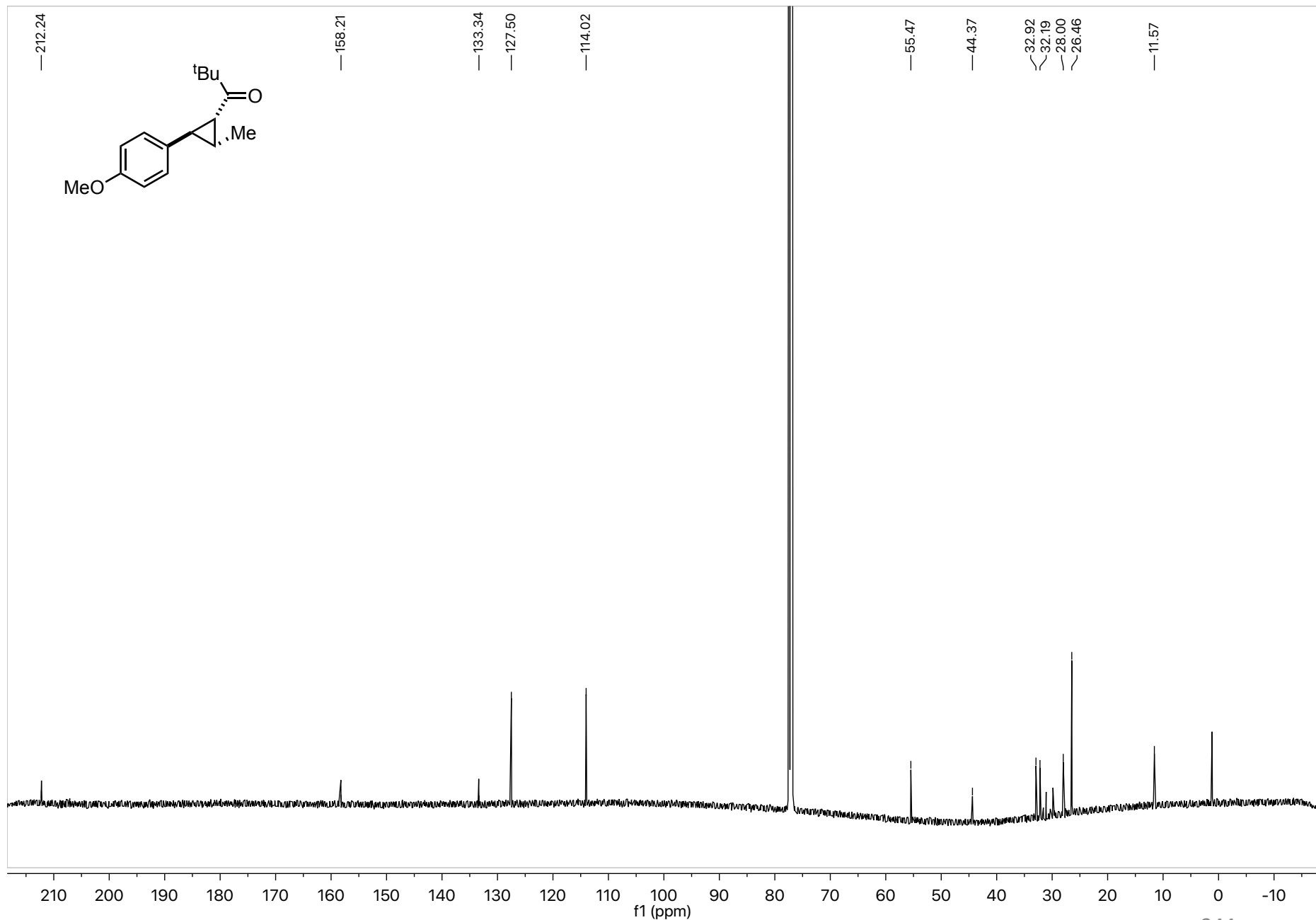
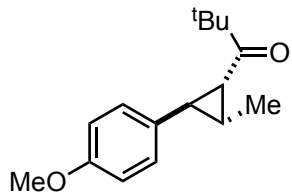
3.76  
2.41  
2.40  
2.40  
2.38  
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2.33  
2.31  
2.29  
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2.17  
2.16  
2.15  
2.13  
2.12  
2.11  
2.10

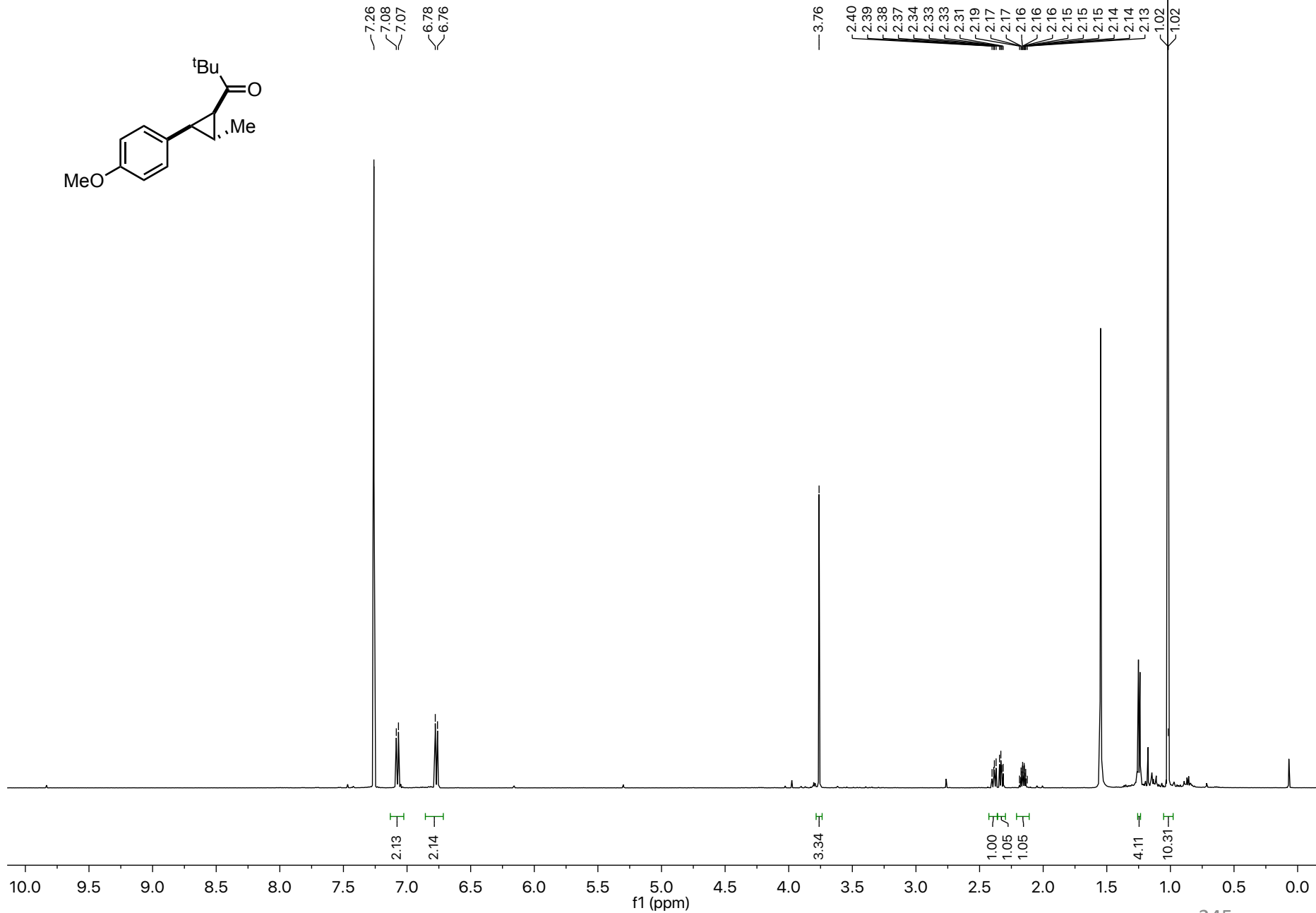
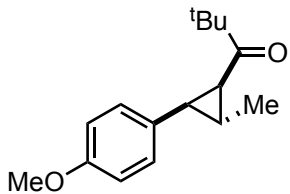
1.54  
1.40  
1.38  
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1.11  
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0.86  
0.82  
0.79

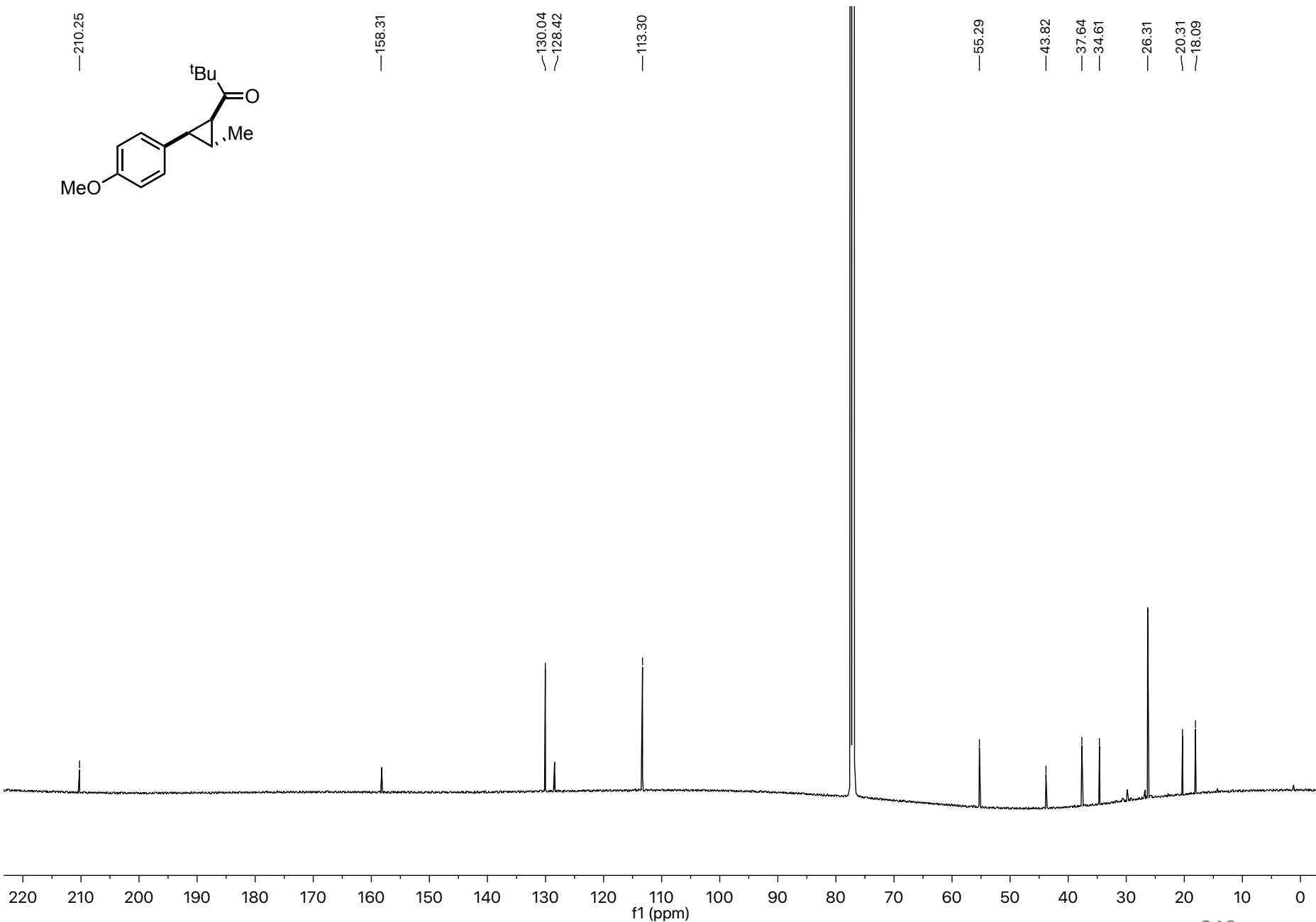
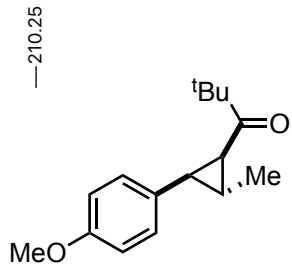


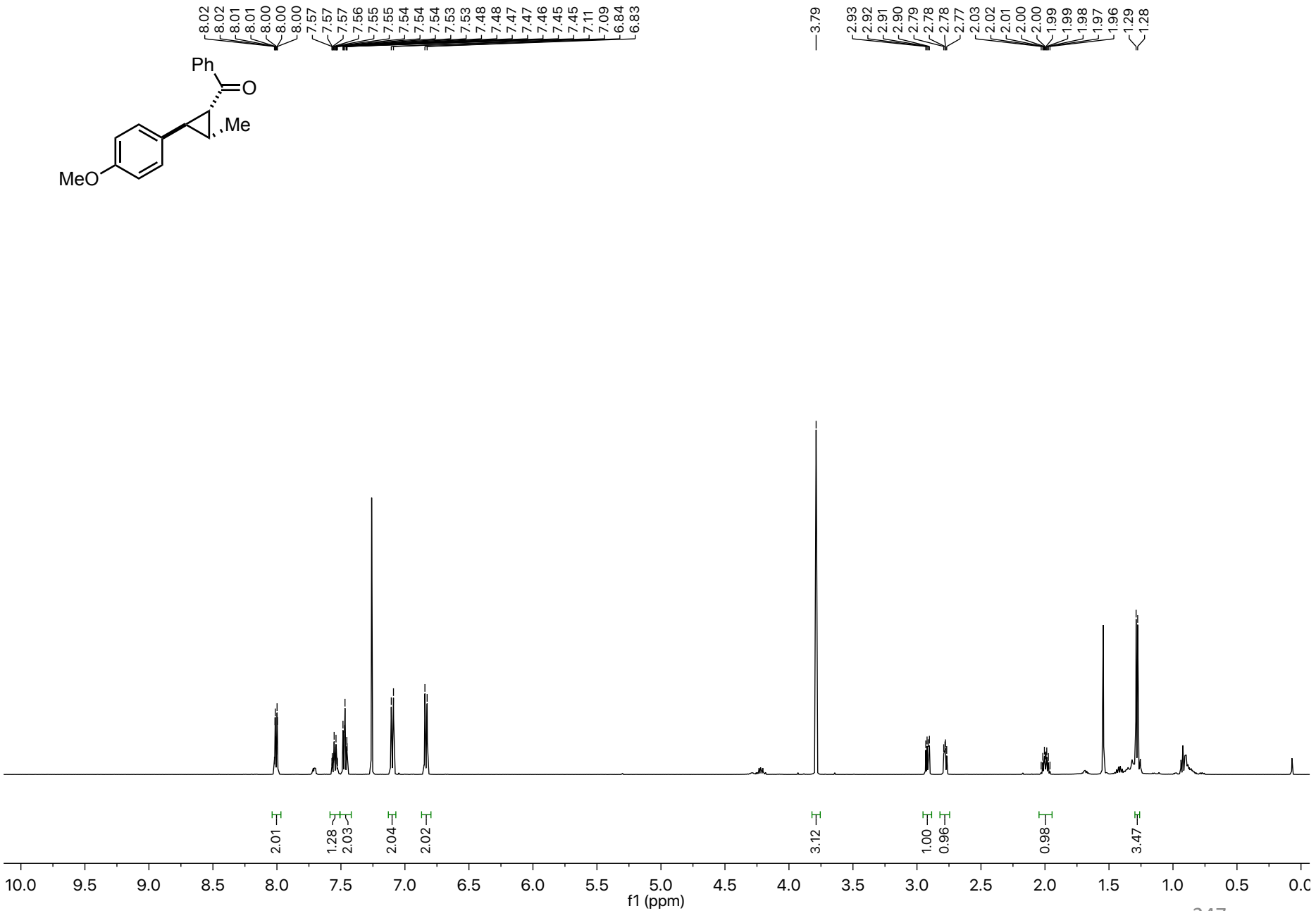
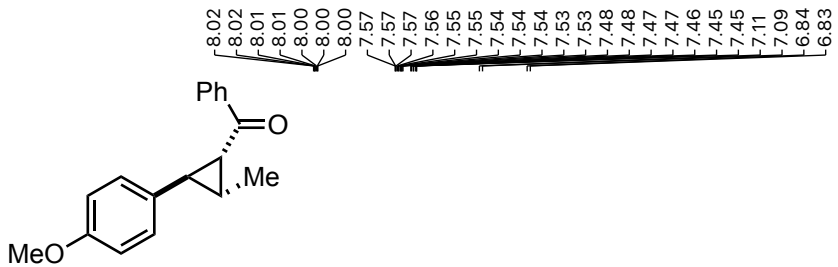


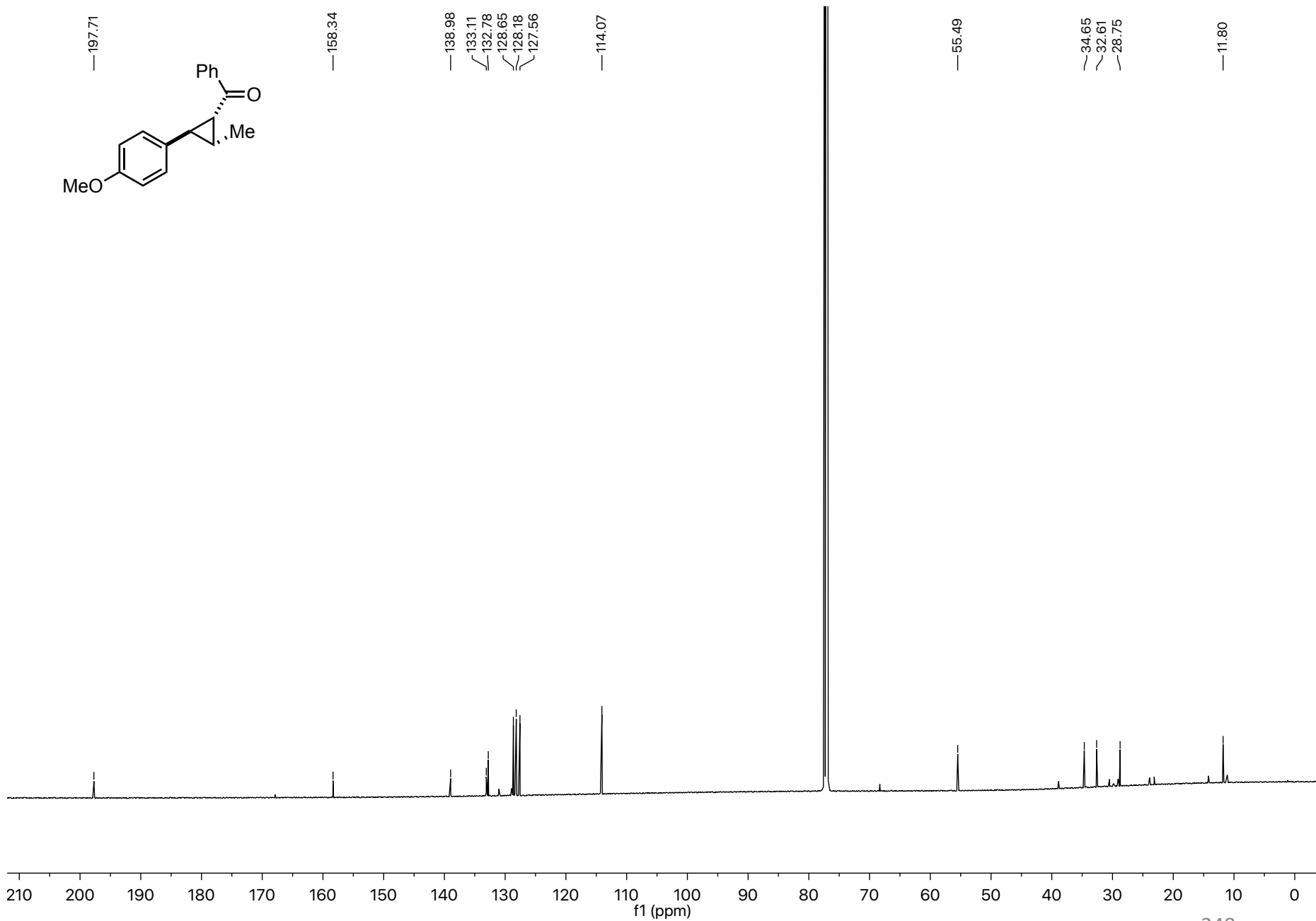
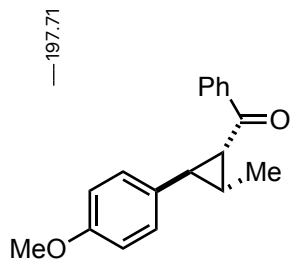




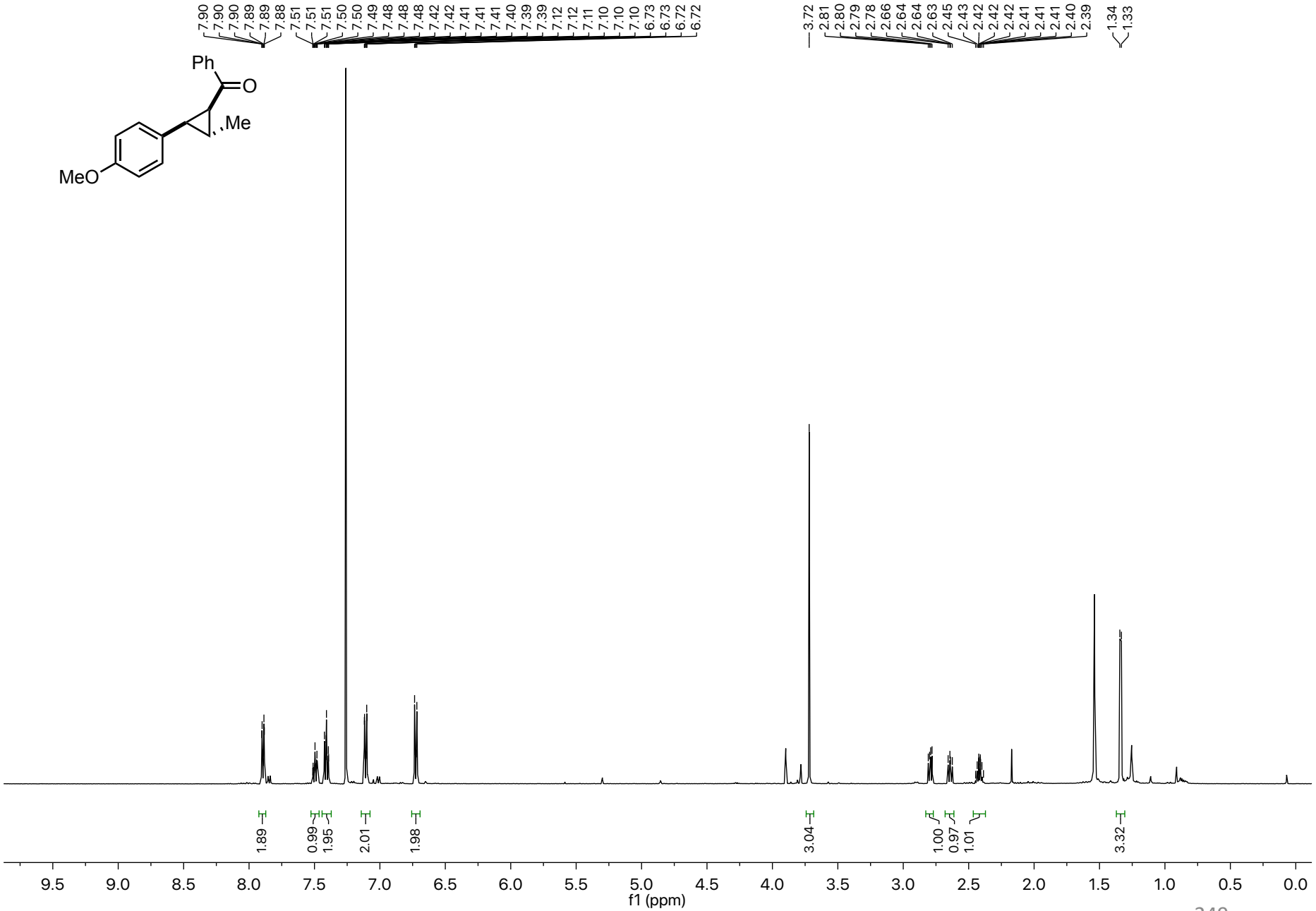
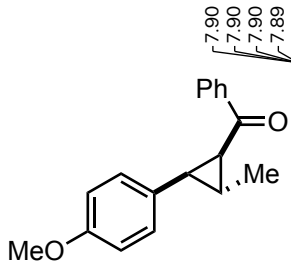


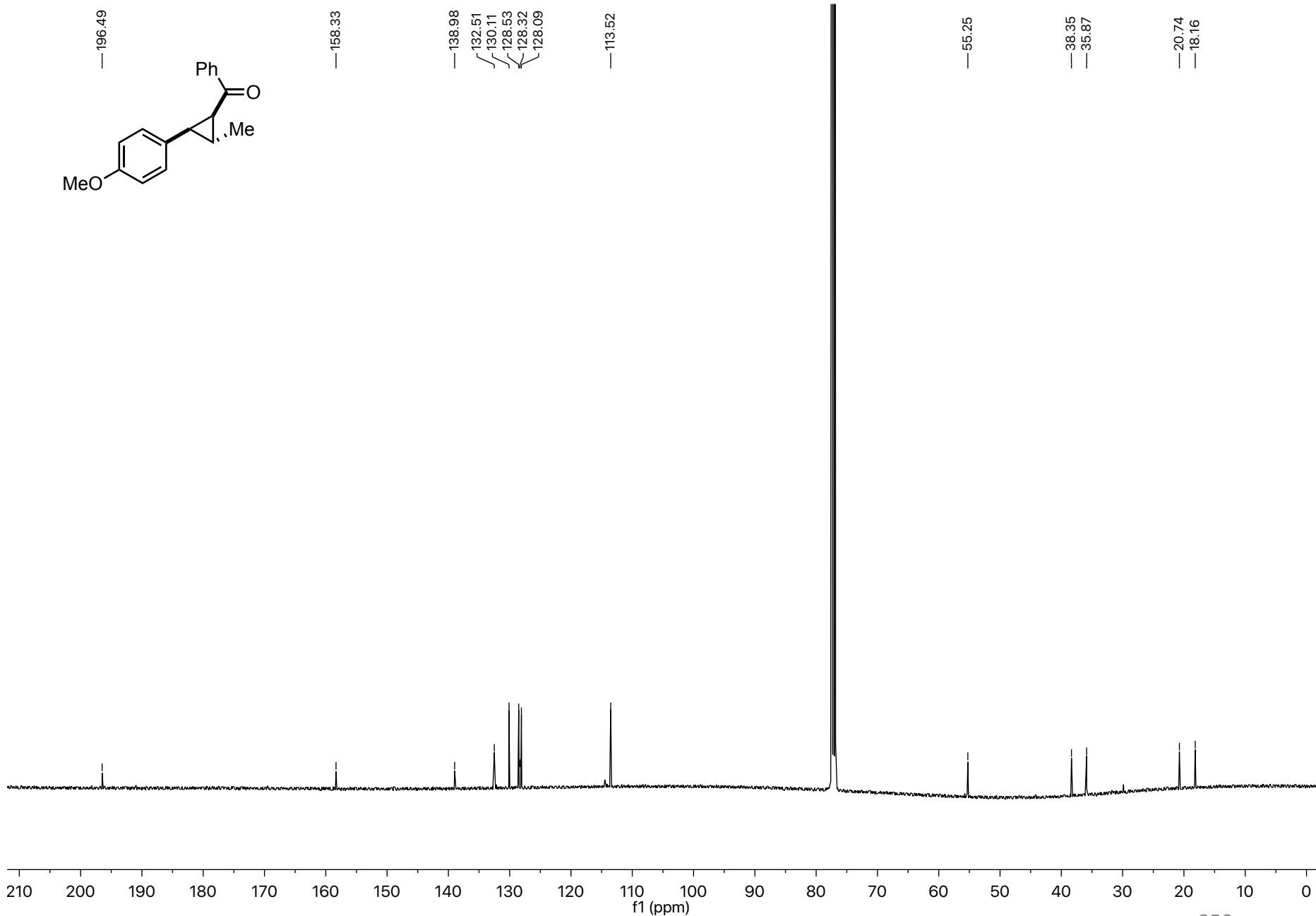
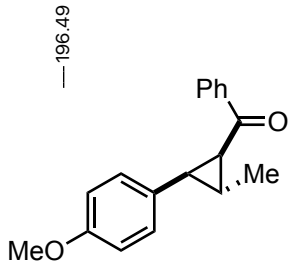


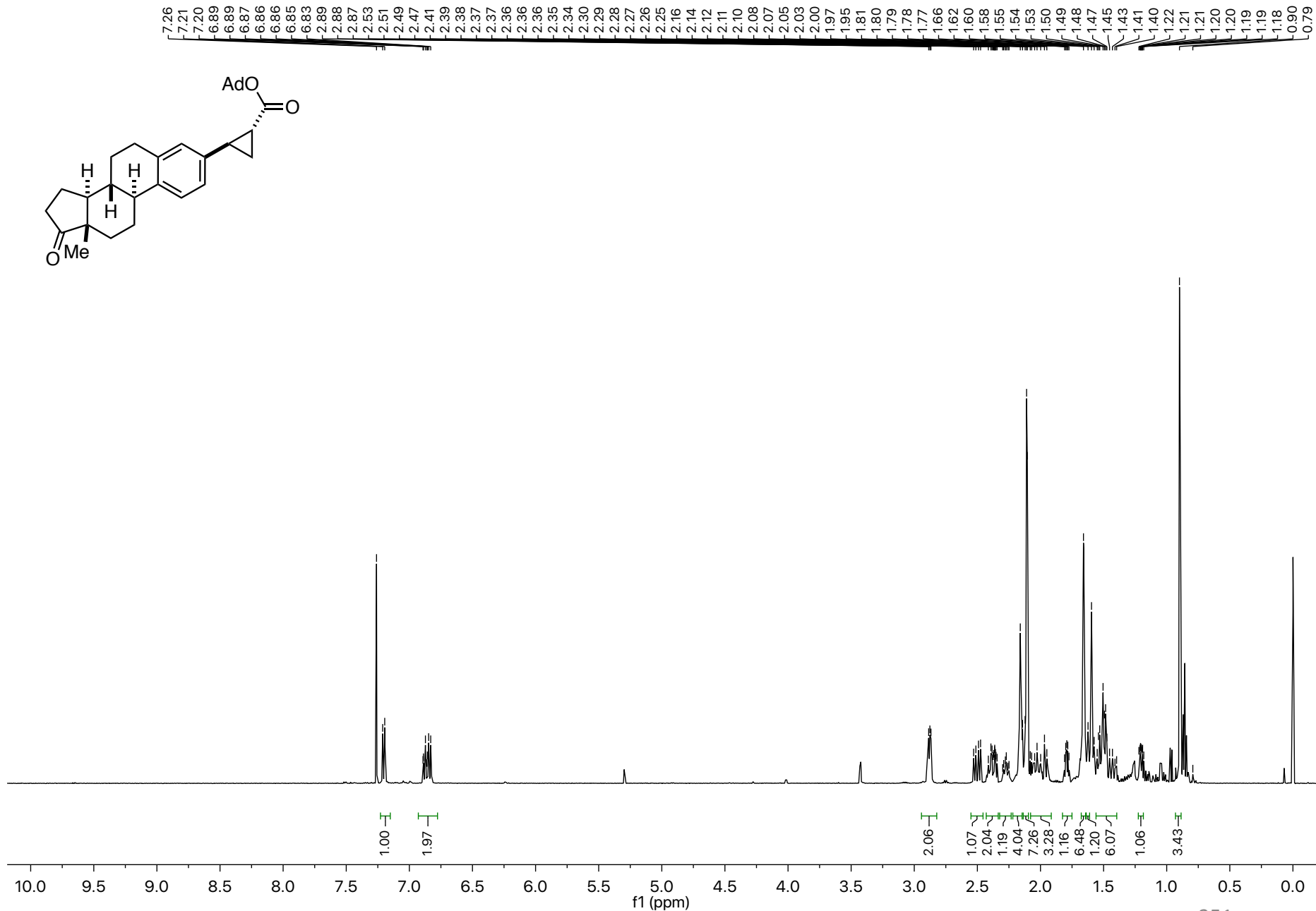
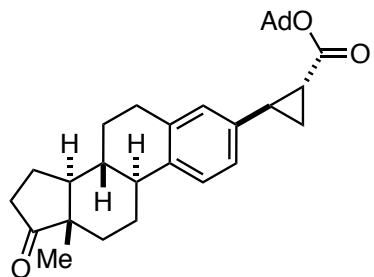












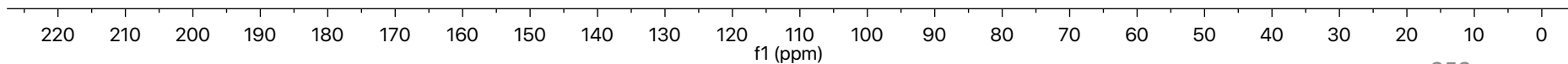
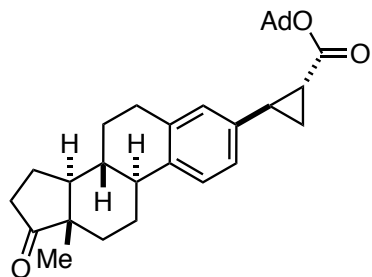
— 221.02

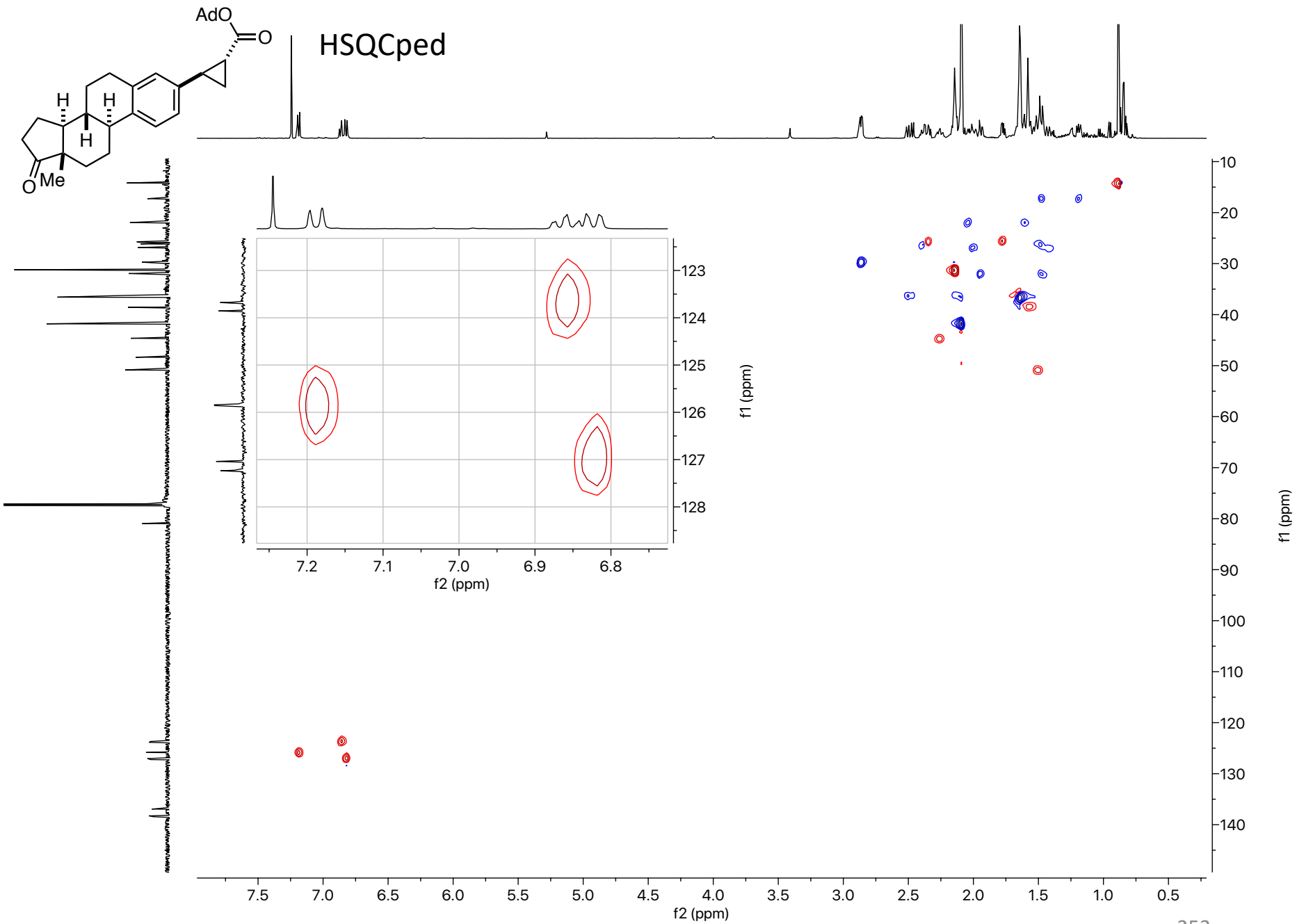
— 172.53

138.23  
138.04  
136.71  
136.71  
127.01  
126.81  
125.63  
125.62  
123.63  
123.45

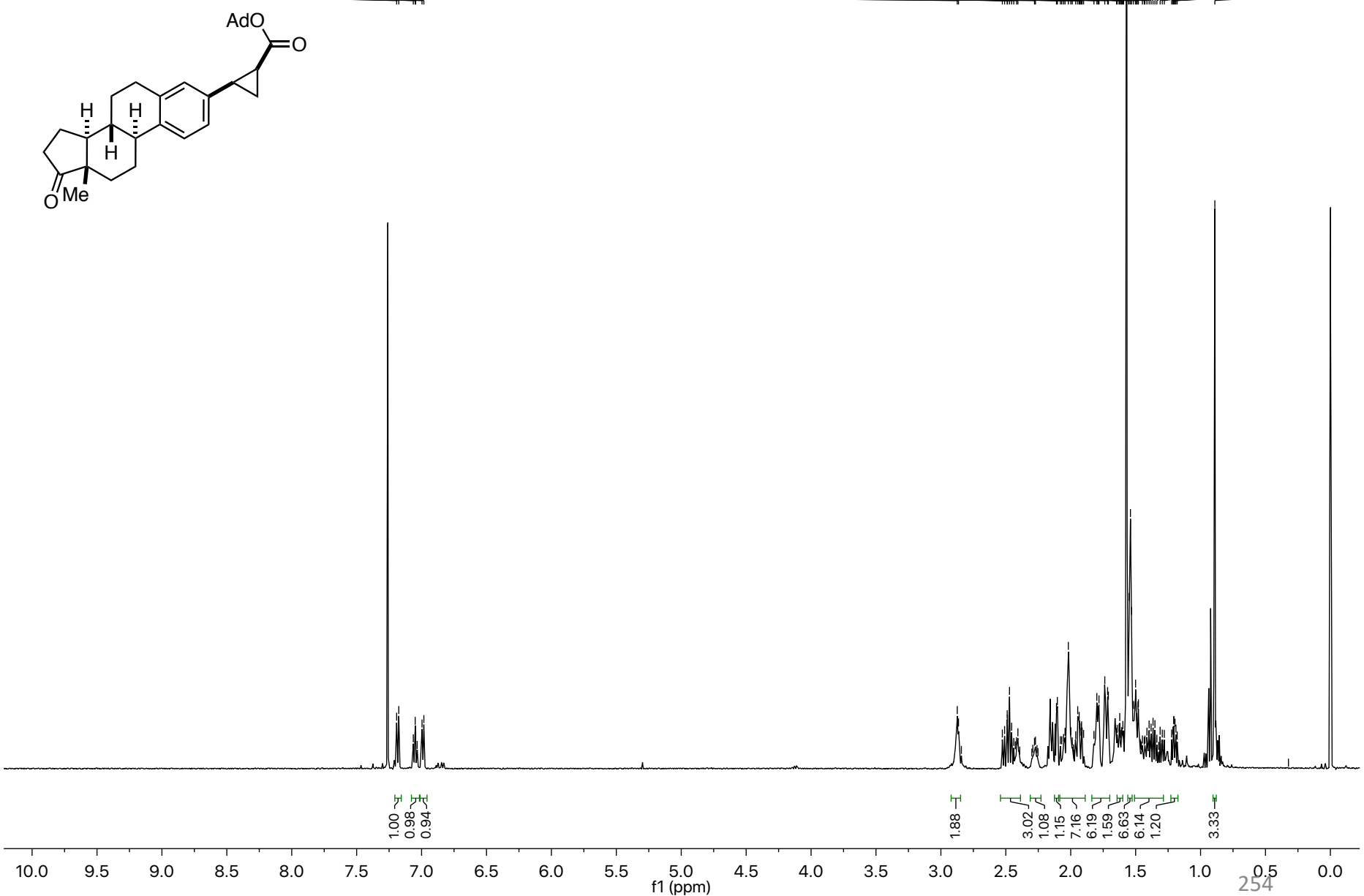
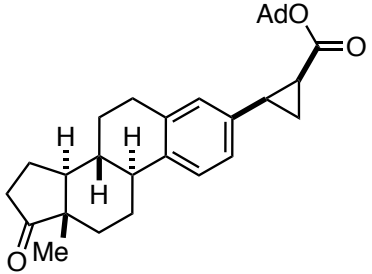
— 80.73  
— 77.16

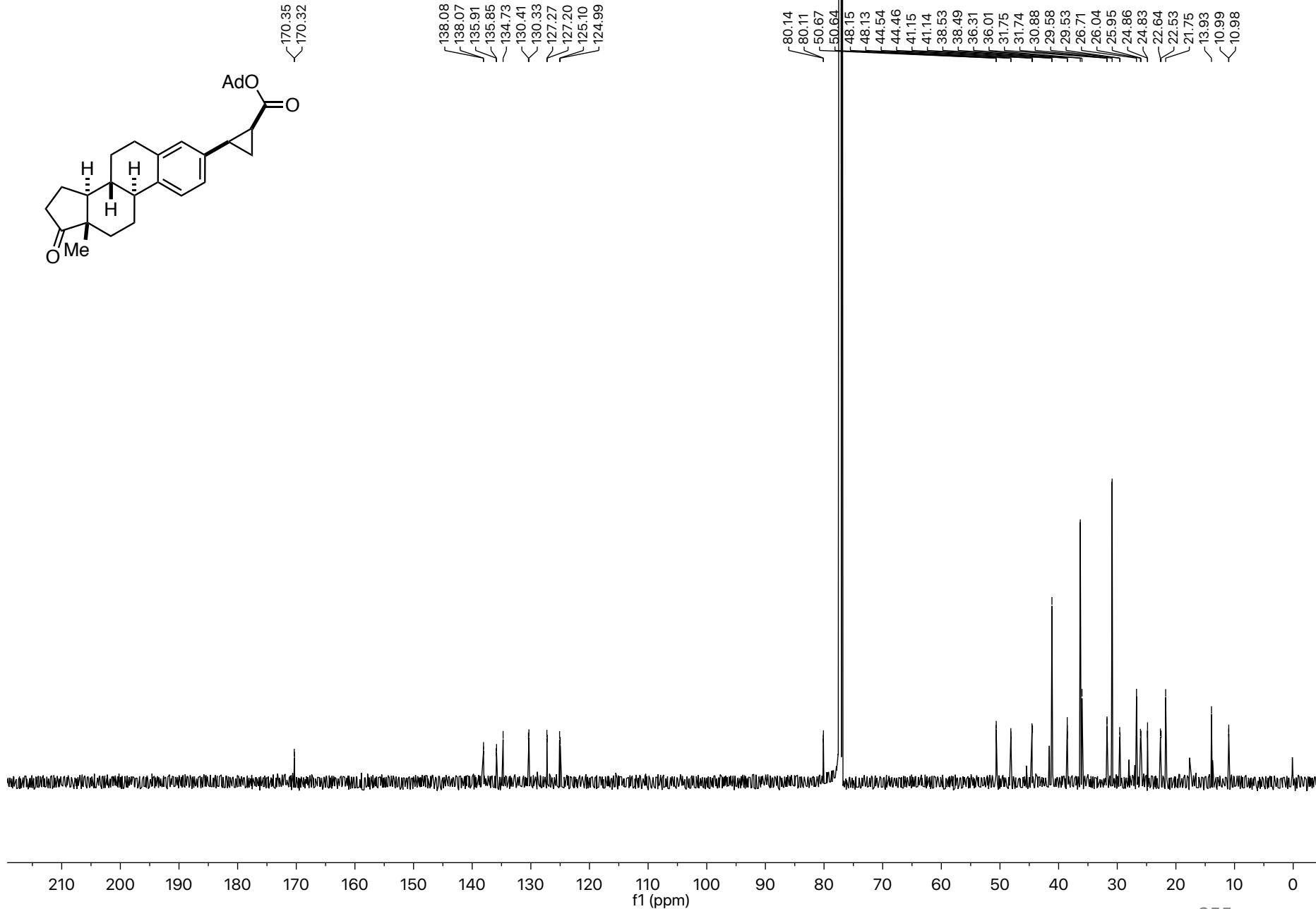
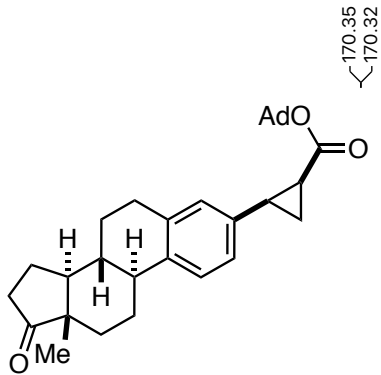
50.61  
48.13  
44.41  
41.58  
38.35  
36.35  
36.00  
31.72  
30.98  
29.51  
26.63  
25.90  
25.59  
25.49  
21.72  
17.07  
17.04  
13.97

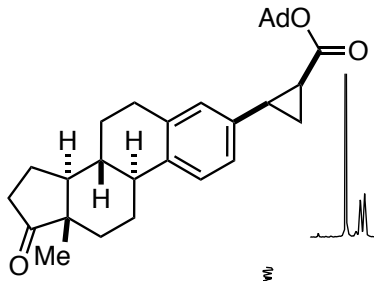




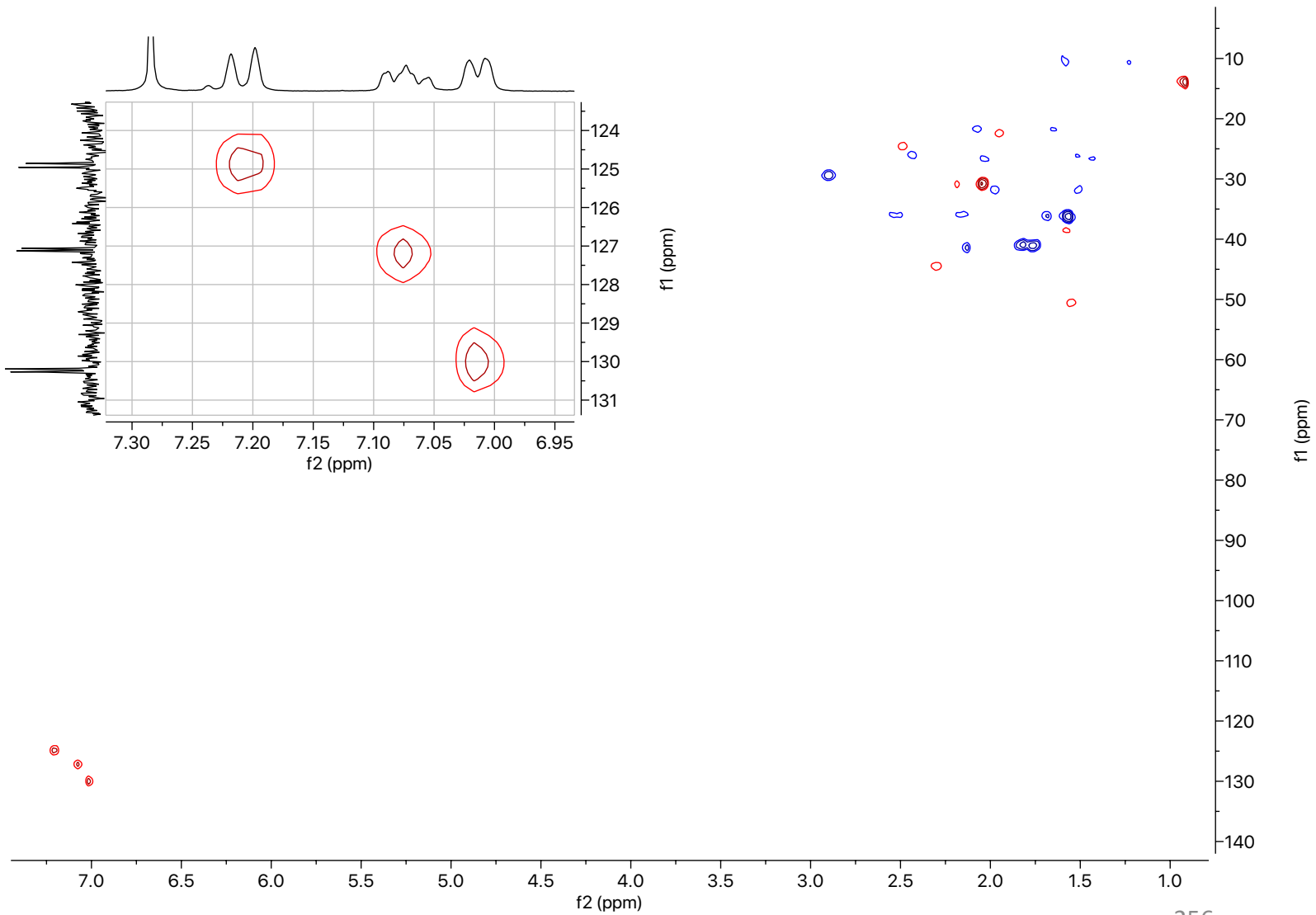
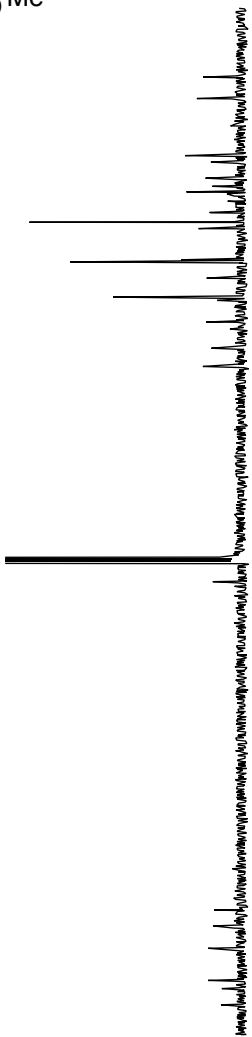
7.19  
7.18  
7.06  
7.05  
7.05  
7.00  
6.98  
2.87  
2.86  
2.53  
2.51  
2.49  
2.49  
2.47  
2.46  
2.44  
2.42  
2.41  
2.28  
2.27  
2.11  
2.10  
2.08  
2.07  
2.05  
2.04  
2.02  
2.00  
1.99  
1.96  
1.95  
1.93  
1.93  
1.93  
1.92  
1.92  
1.91  
1.90  
1.82  
1.80  
1.78  
1.74  
1.72  
1.71  
1.66  
1.64  
1.63  
1.62  
1.62  
1.61  
1.60  
1.60  
1.59  
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1.20  
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1.18  
0.89



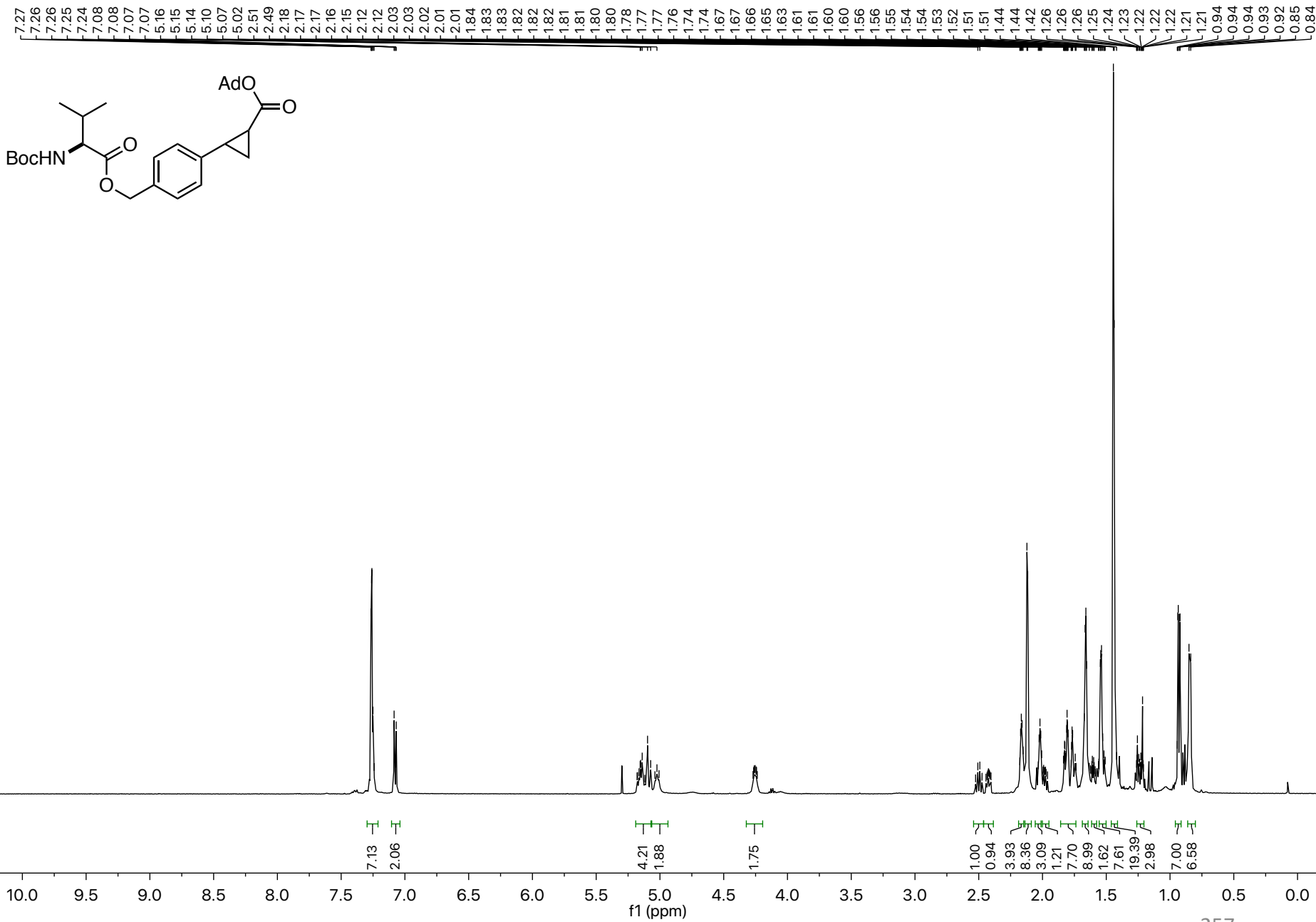


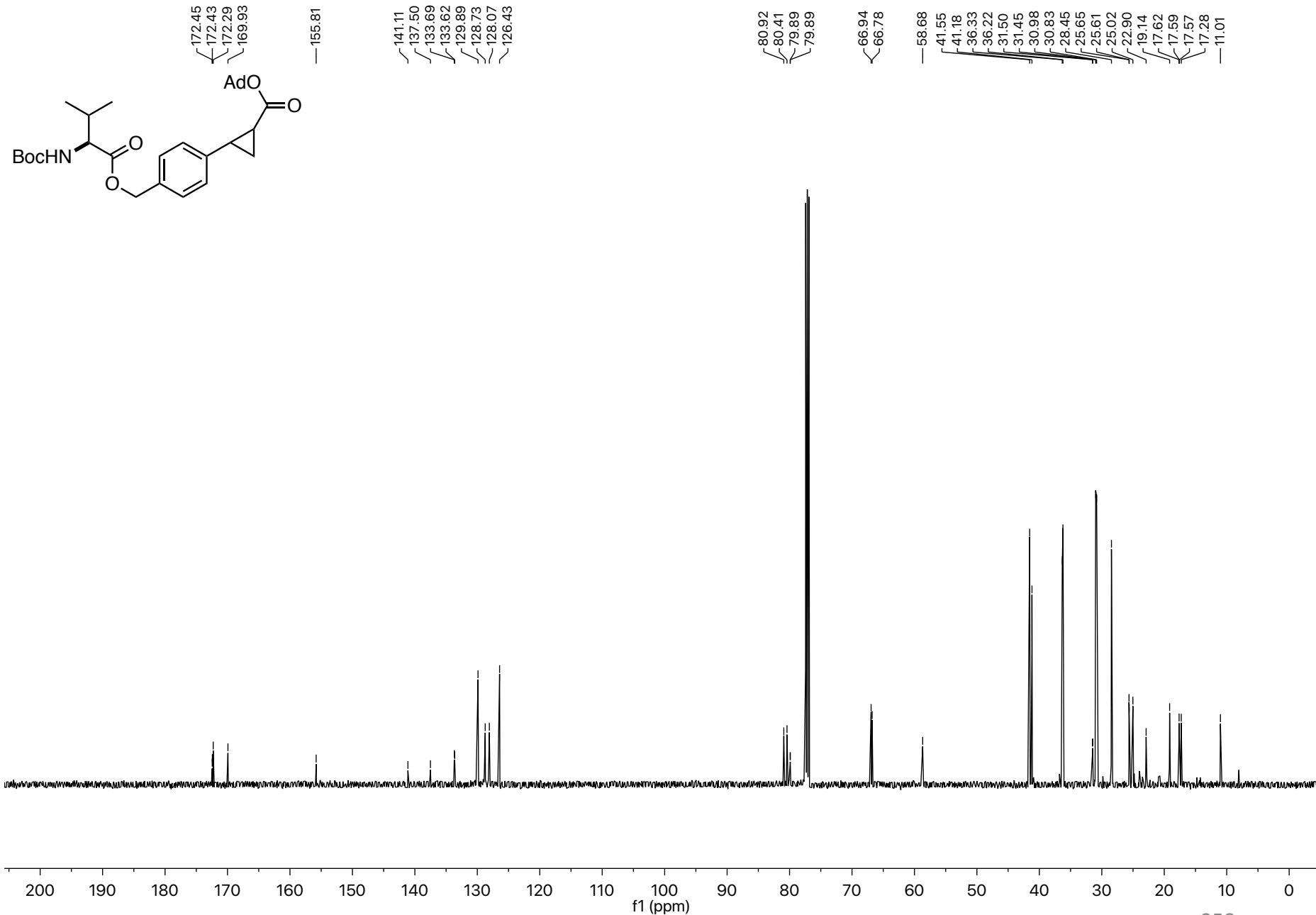
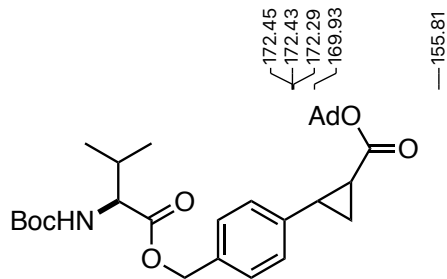


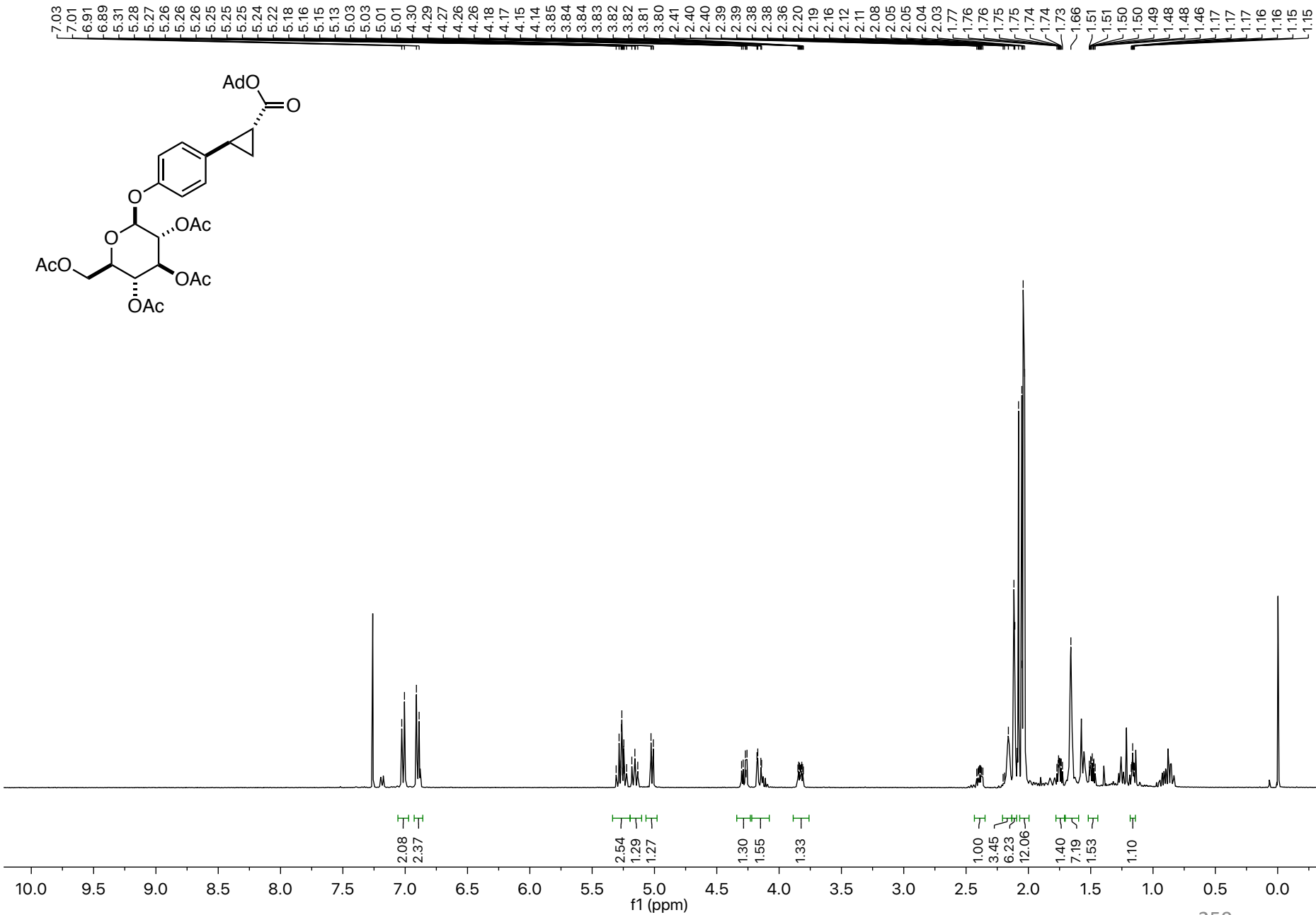
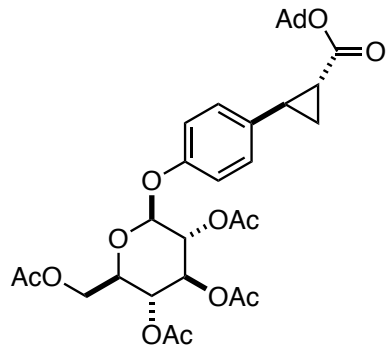
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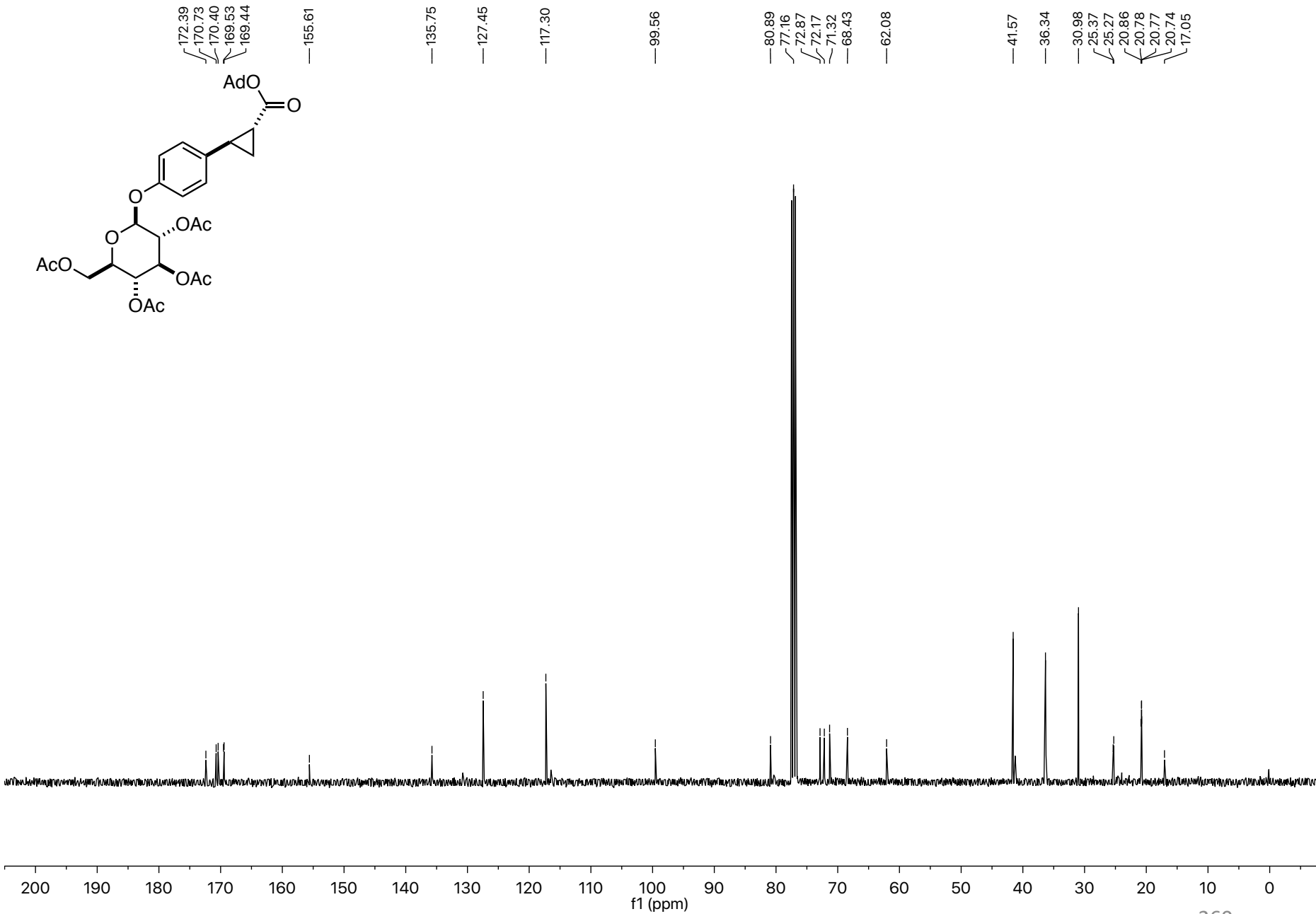
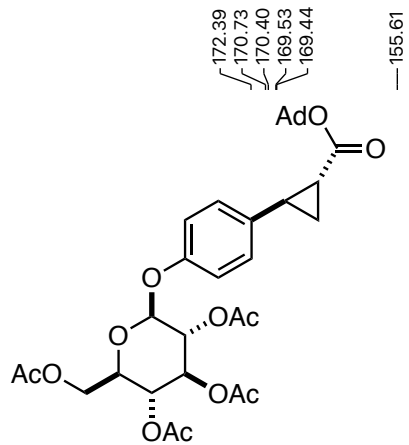




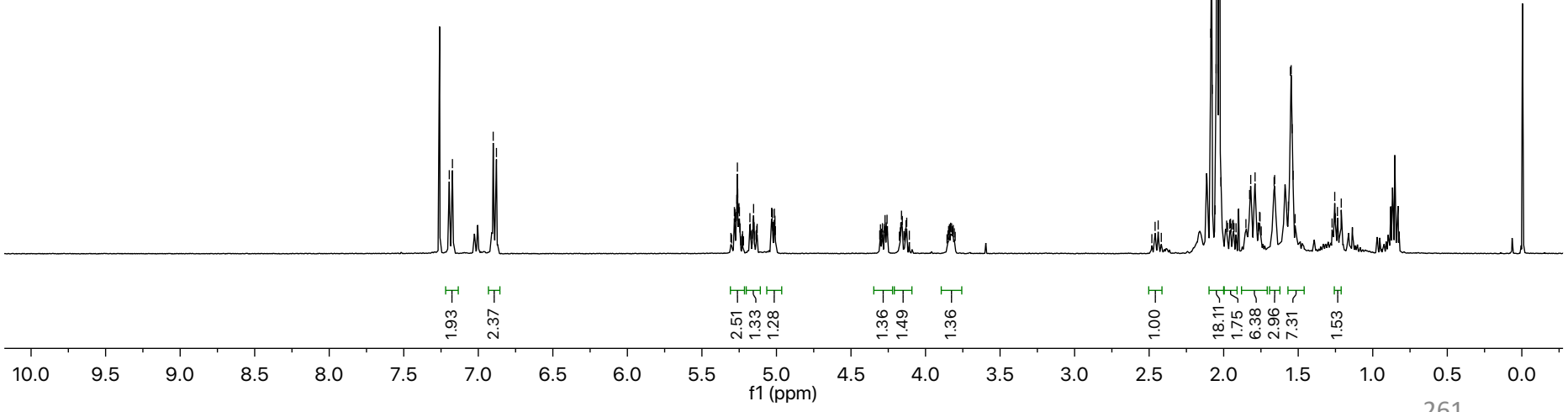
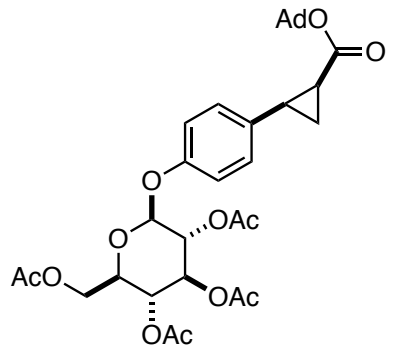


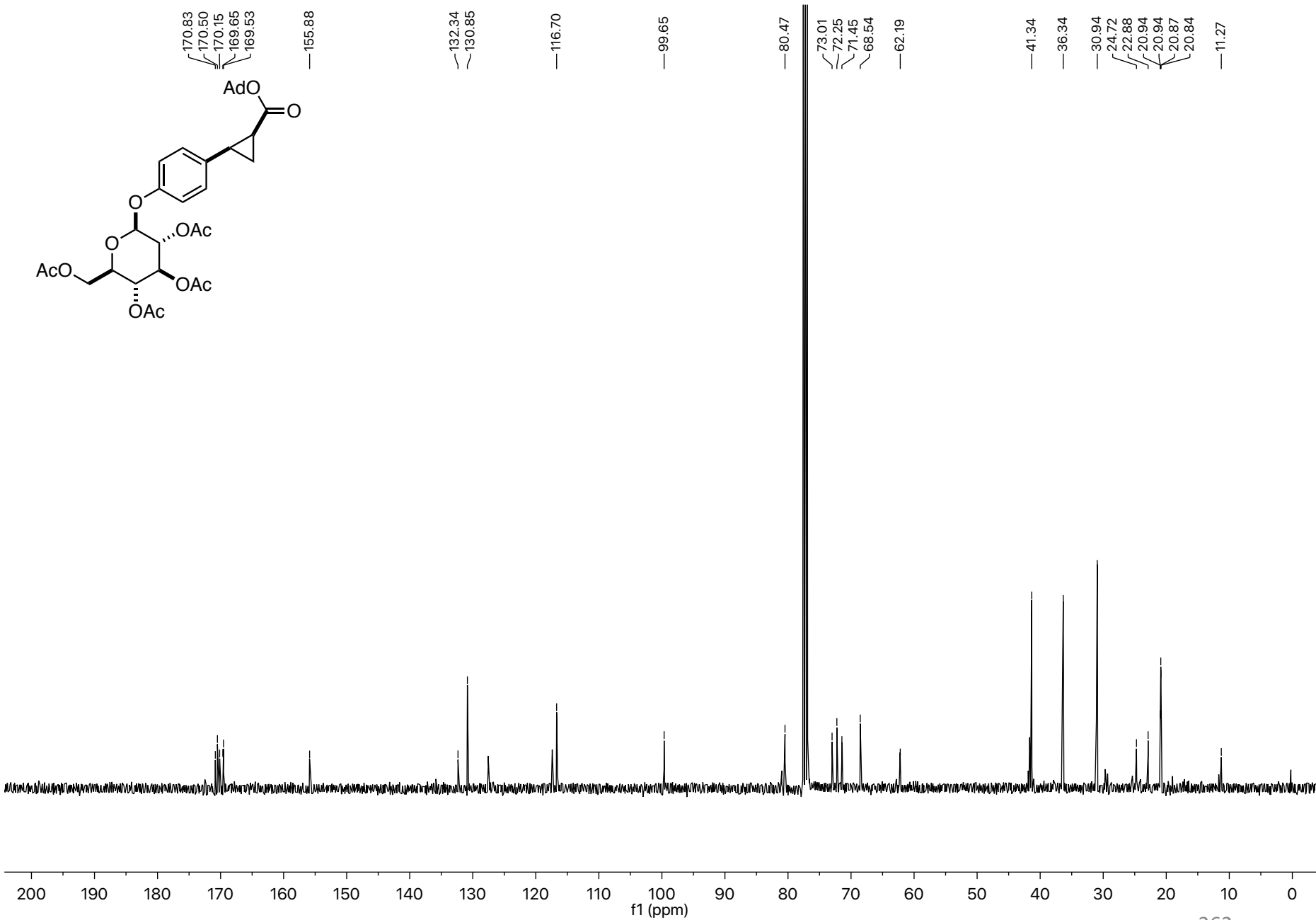
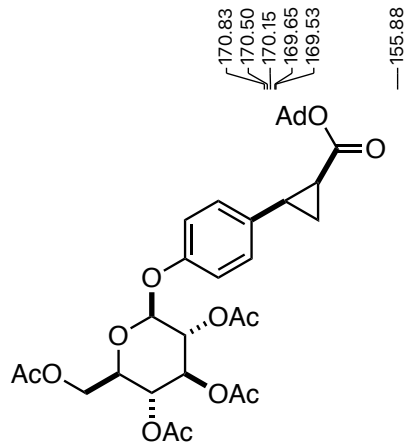




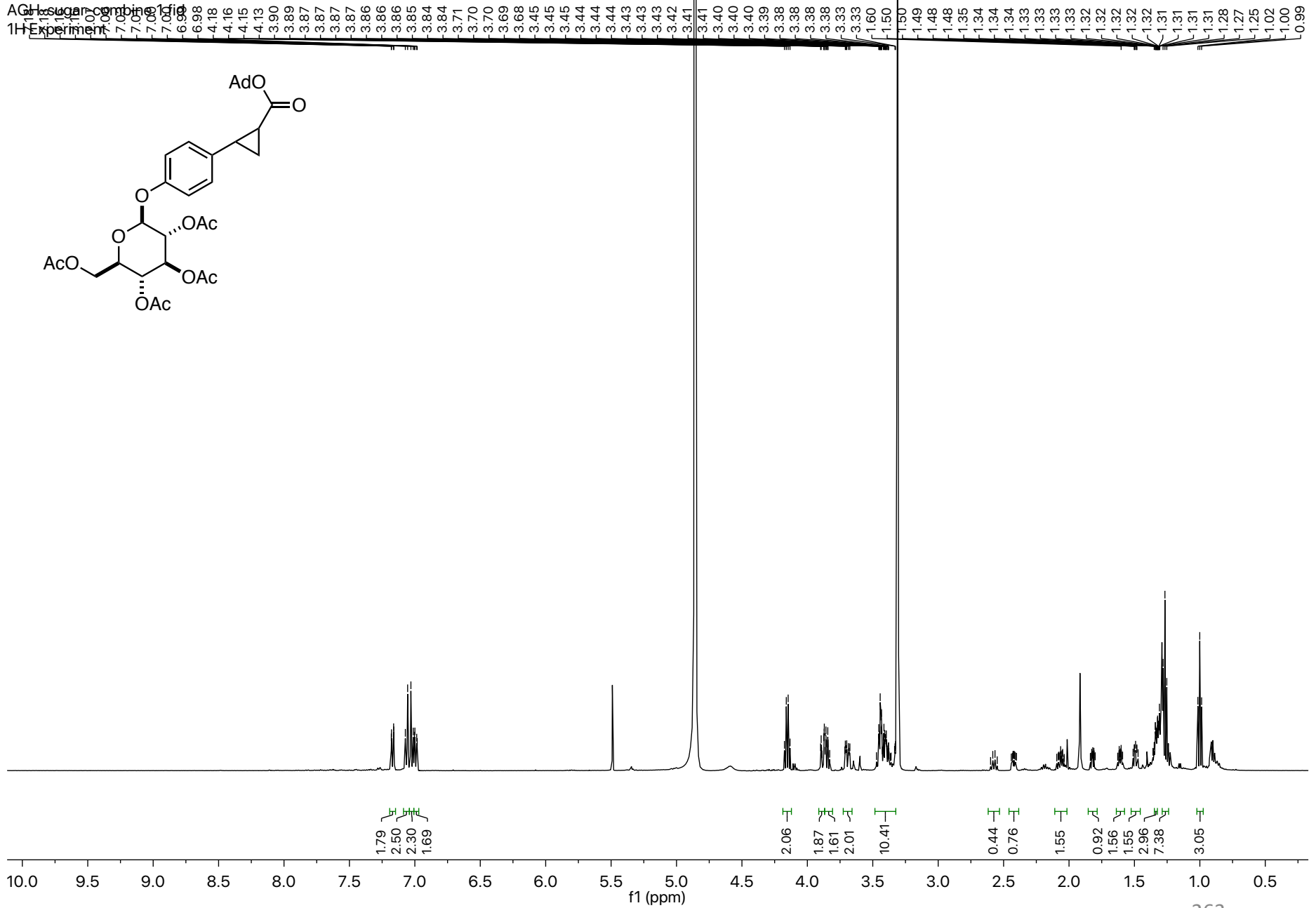
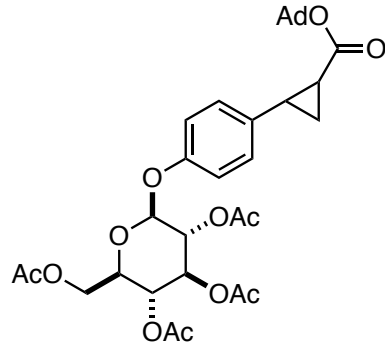


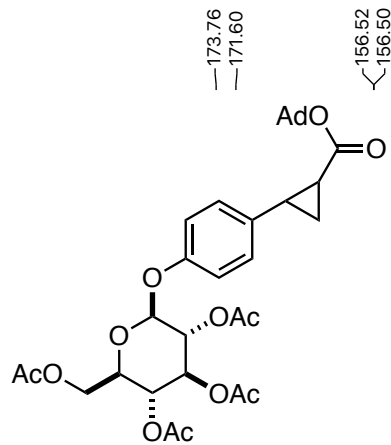
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5.26  
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5.25  
5.25  
5.24  
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5.02  
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5.01  
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2.05  
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1.94  
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1.55  
1.55  
1.54  
1.53  
1.52  
1.27  
1.25  
1.25  
1.24  
1.23  
1.21





AGH Sugar Combine 1  
1H Experiment





173.76  
171.60

156.52  
156.50

133.72  
129.92  
126.79

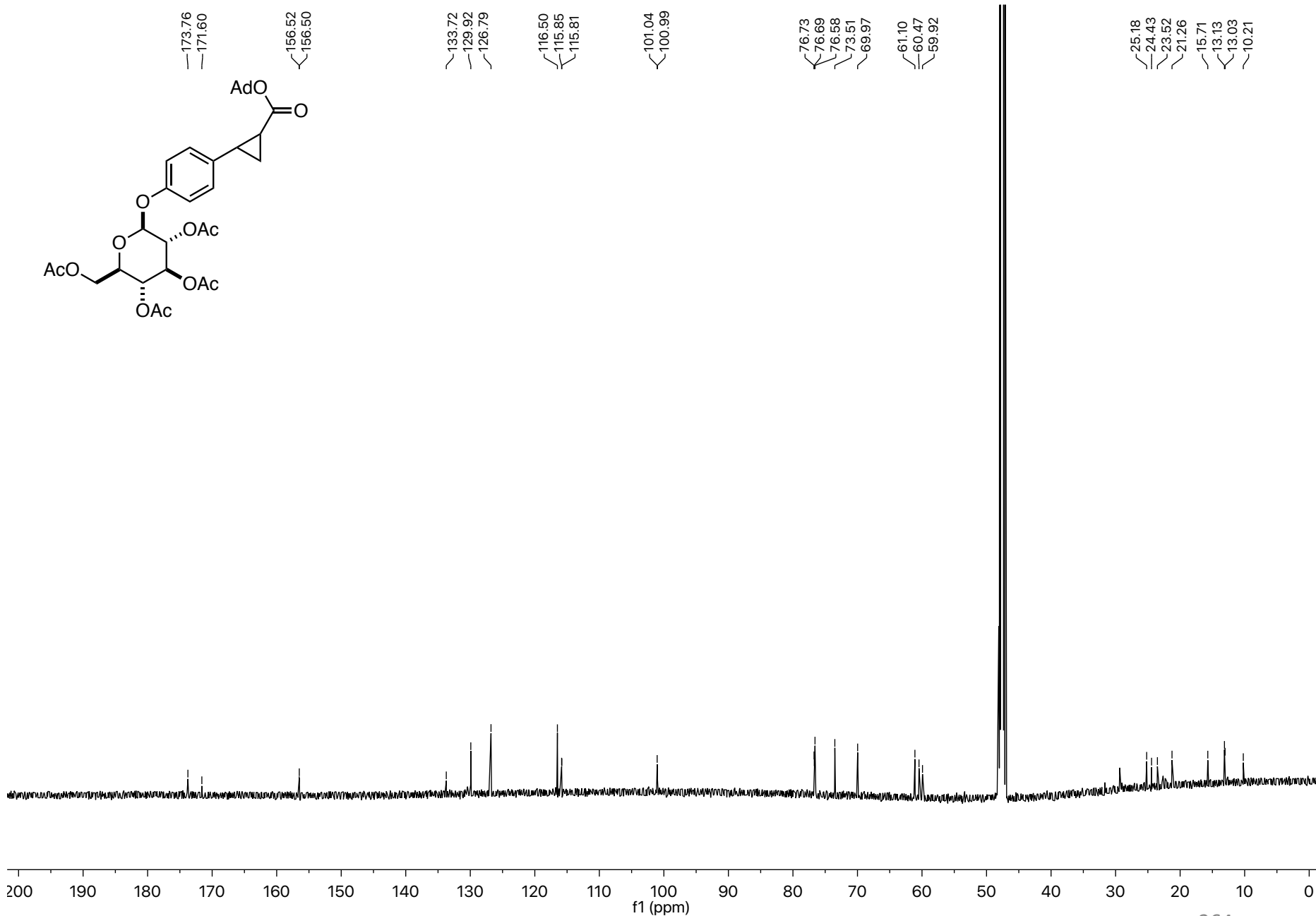
116.50  
115.85  
115.81

101.04  
100.99

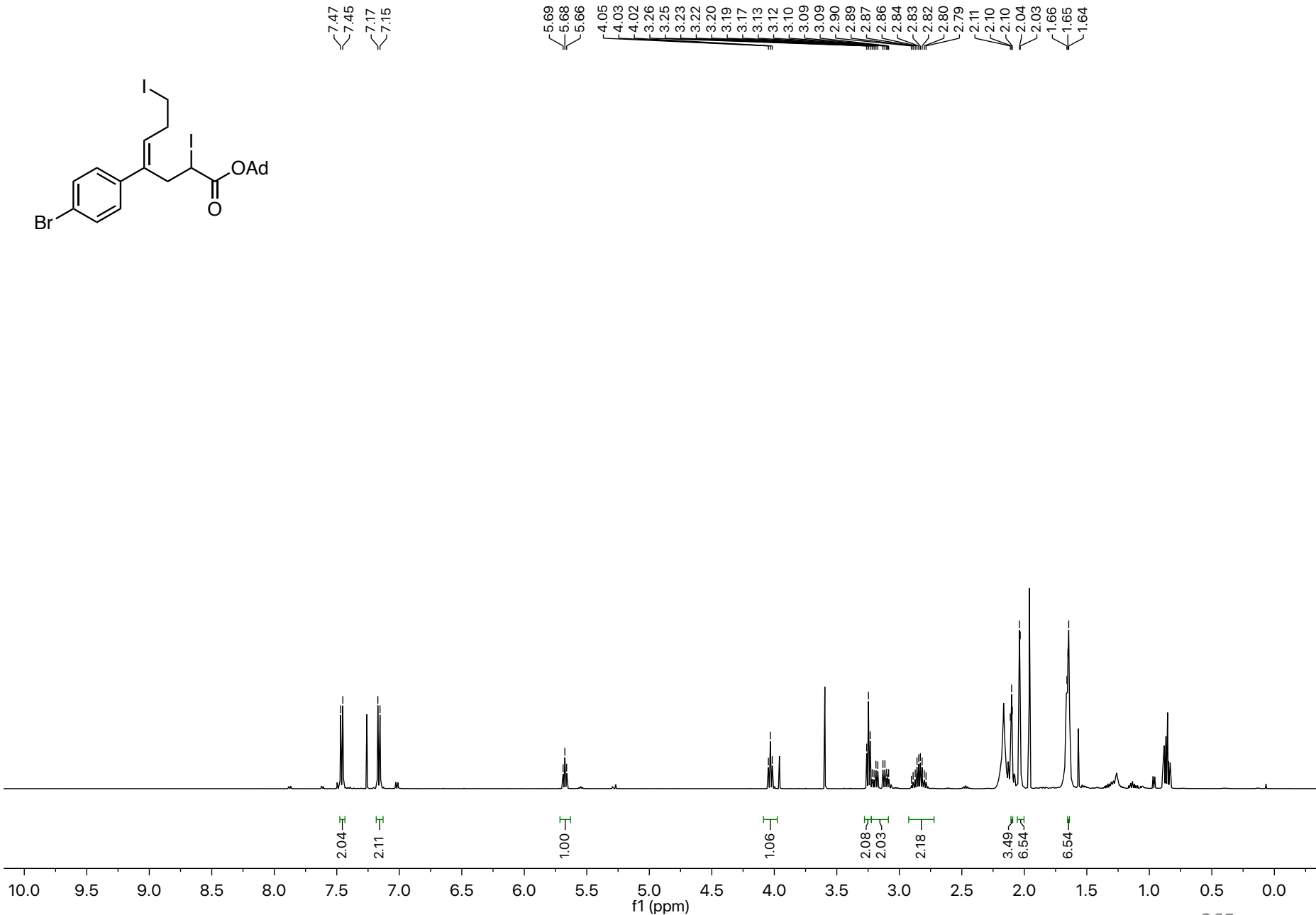
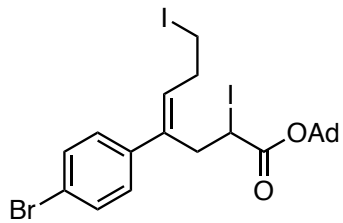
76.73  
76.69  
76.58  
73.51  
69.97

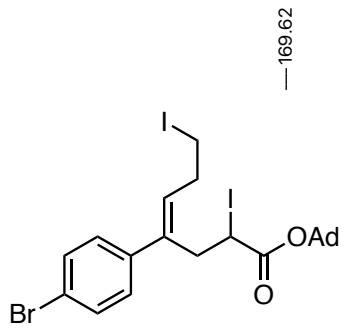
61.10  
60.47  
59.92

25.18  
24.43  
23.52  
21.26  
15.71  
13.13  
13.03  
10.21









—169.62

~139.88

~138.40

~131.74

~131.44

~128.47

—121.56

—82.34

~41.31

~40.75

~36.06

~32.39

~30.79

—21.20

—4.74

