The First Asymmetric Total Synthesis of (+)-Coriandrone A and B

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Supporting Information

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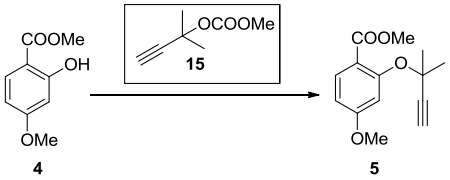
1. Materials and General information

CH_2Cl_2 was dried by distillation over CaH_2, and THF was dried by distillation over Na/K. Other chemicals were used as received, and all reactions conducted under standard conditions were monitored by thin-layer chromatography (TLC) on gel F254 plates. The silica gel (200-300 meshes) was used for column chromatography. ^1H and ^{13}C spectra were recorded on *Bruker* AM-400 MHz instruments, and spectral data were reported in ppm relative to tetramethylsilane (TMS) as internal standard. Mass spectra (MS) were measured on spectrometer by direct inlet at 70 eV, and signals were given in *m*/*z* with relative intensity (%) in brackets. HRMS data were determined on a *Bruker Daltonics* APEXII 47e FT-ICR spectrometer. Optical rotations were measured using sodium D line on a Perkin Elmer341 polarimeter. The enantiomeric excess values were determined by chiral stationary phase HPLC analysis (IB-3 10U 250×4.6 M, hexane: 2-propanol 85:15, flow rate 1 mL/min, detected at 236.0 nm on a Prostar 330 detector).

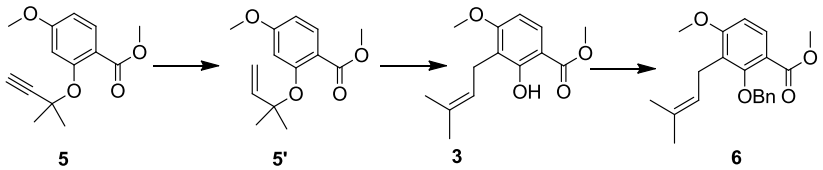
2. Procedures for the synthesis of compounds

Methyl 4-methoxy-2-((2-methylbut-3-yn-2-yl)oxy)benzoate (5)



The phenol **4** (2.40 g, 13.49 mmol) was dissolved in CH_3CN (50 mL) and cooled to 0 ^oC. Commercially available $CuCl_2{\cdot}2H_2O$ (56.7 mg, 0.42 mmol) is then added with 1, 1-dimethyl-prop-2-ynyl methyl carbonate **15** (2.90 g, 20.4 mmol). The reaction mixture was stirred for 15 min before the addition of commercially available DBU (2.6 mL, 21.0 mmol) via syringe. The reaction mixture remains at 0 ^oC for 1 hour and was allowed to warm to 38 ^oC for 6 h. The volatile organics are removed under reduced pressure and the resulting oil was diluted with EtOAc (150 mL). The organic solution was washed sequentially with 1M HCl (50 mL), water (50 mL) and brine (50 mL). The organic layer was dried over Na_2SO_4, filtered and concentrated in vacuo. The resulting brown oil was purified by flash silica gel chromatography (Petroleum ether/EtOAc 16: 1) to afford the desired aryl ether **5** (2.31 g, 68 %) as a colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 7.81 (d, *J* = 8.4 Hz, 1H), 7.16 (d, *J* = 2.4 Hz, 1H), 6.28 (q, *J* = 2.4 Hz, *J* = 8.8 Hz, 1H), 3.84 (s, 3H), 3.82 (s, 3H), 2.59 (s, 1H), 1.68 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 166.5, 163.05, 157.3, 132.9, 117.2, 108.4, 107.7, 86.2., 74.0, 73.8, 55.4, 51.5, 29.2; **MS** (EI) *m/z* (%): 248 (M^+, 1), 233 (1), 217 (2), 189 (4), 122 (29), 150 (100), 107 (21), 79 (17), 67 (39), 51 (26), 41 (36)

The Preparation of Compound 3 and 6



Methyl 2-hydroxy-4-methoxy-3-(3-methylbut-2-en-1-yl)benzoate (3)

A solution of alkyne **5** (1.45 g, 5.86 mmol) in EtOAc (10 mL) was treated with $Pd/BaSO_4$ (60 mg)

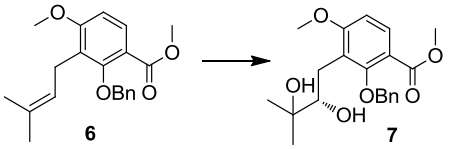
and quinoline (20 μL) and the mixture was hydrogenated (1atm) over 40min (the reaction was monitored every 10min by TLC and stopped once the starting material was consumed). The reaction mixture was filtered through celite, concentrated and submitted directly to the Claisen rearrangement.

The crude alkene **5'** was dissolved in DMF (20 mL) and heated at 120 °C for 5 h, The yellow solution was concentrated and purified by column chromatography on silica gel (Petroleum ether /EtOAc 10: 1) to yield a crude phenol **3** (1.23g, 84% for two steps). **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 11.06 (s, 1H), 7.71 (d, *J* = 8.8 Hz, 1H), 6.45 (d, *J* = 8.8 Hz, 1H), 5.22 (t, *J* = 7.2 Hz, 1H), 3.91 (s, 3H), 3.88 (s, 3H), 3.38 (d, *J* = 7.2 Hz, 2H), 1.80 (s, 3H), 1.69 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 170.7, 162.7, 160.5, 131.7, 128.8, 122.1, 117.0, 105.9, 102.2, 55.6, 51.9, 25.8, 21.9, 17.7; **MS** (EI) *m/z* (%): 250 (M^+, 61), 218 (51), 203 (100), 190 (46), 175 (92), 163 (81), 133 (22), 105 (14), 91 (10), 77 (11), 40 (15).

Methyl 2-(benzyloxy)-4-methoxy-3-(3-methylbut-2-en-1-yl)benzoate (6)

To a solution of phenol **3** (1.23 g, 4.92 mmol) in acetone (40 mL) was added benzyl bromide (1.05 mL, 8.79 mmol) and potassium carbonate (1.20 g, 8.79 mmol). The resulting suspension was rapidly stirred at reflux for 16 h, cooled, and the acetone removed under reduced pressure. The resulting residue was partitioned between ethyl acetate (60 mL) and 1.0N hydrochloric acid (30 mL). The aqueous layer was washed twice with 60 mL portions of ethyl acetate. Combined organic layers were dried over sodium sulfate and concentrated under reduced pressure. The residue was purified by flash chromatography (Petroleum ether/EtOAc 8:1), which afforded **6** (1.59 g, 95%) as a colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 7.83 (d, *J* = 8.8 Hz, 1H), 7.51 (d, *J* = 7.2 Hz, 2H), 7.32-7.42 (m, 3H), 5.16 (t, *J* = 1.6 Hz, 1H), 4.94 (s, 2H), 3.89 (s, 3H), 3.84 (s, 3H), 3.39 (d, *J* = 7.2 Hz 2H), 1.66-1.67 (t, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 166.5, 162.0, 158.1, 137.5, 131.7, 131.0, 128.4, 128.0, 127.9, 125.1, 122.6, 117.0, 106.1, 76.9, 55.8, 51.8, 25.7, 23.0, 17.9; **MS** (EI) *m/z* (%): 340 (M^+, 3), 249 (7), 235 (15), 217 (57), 195 (17), 163 (22), 91 (100), 40 (33).

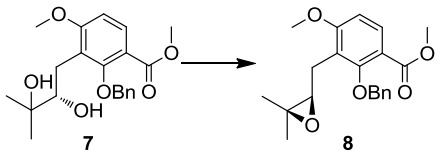
(S)-methyl 2-(benzyloxy)-3-(2,3-dihydroxy-3-methylbutyl)-4-methoxybenzoate (7)



A 50 mL flask, equipped with a magnetic stirrer, was charged with *t*-BuOH (28 mL), water (28 mL), and AD-mix- α (7.80 g). Stirring at room temperature produced two clear phases. Methanesulfonamide (563mg, 6.1 mmol) was added and the mixture was cooled to 0 °C where upon some of the dissolved salts precipitated. Olefin **6** (1.90 g, 5.57 mmol) was added at once, and the heterogeneous slurry was stirred vigorously at 0 °C for 48 h. While the mixture was stirred at 0 °C, solid sodium sulfite (7.5 g) was added and the mixture was allowed to warm to room temperature and stirred for 30 min. Ethyl acetate (50 mL) was added to the mixture. After separation of the layers, the aqueous phase was further extracted with EtOAc (3 x 75 mL). The combined organic layers were washed with 2 N KOH (25 mL) thoroughly, dried over a hydrous Na_2SO_4 and concentrated. This crude product was purified by flash chromatography (Petroleum ether/EtOAc 2:1) to afford **7** (1.63 g, 78%) as a colorless oil. $[\alpha]_D^{20}$ -4.82° (*c* 1.0, $CHCl_3$) **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 7.89 (d, *J* = 8.8 Hz, 1H), 7.50 (d, *J* = 6.8 Hz, 2H), 7.33-7.42 (m, 3H), 6.75 (d, *J* = 8.8 Hz, 2H), 5.09 (d, *J* = 10.4 Hz, 1H), 4.89 (d, *J* = 10.4 Hz, 1H), 3.89 (s, 3H), 3.87 (s, 3H), 3.50-3.54 (m, 1H), 2.90-2.94 (dd, *J* = 2 Hz, *J* = 13.6, 1H), 2.68-2.74 (m, 2H),

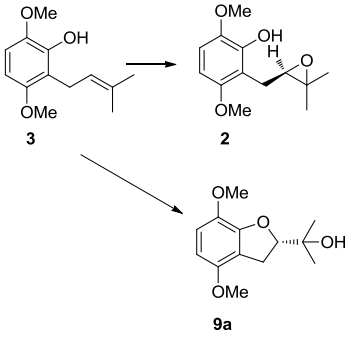
2.35 (s, 1H), 1.18 (s, 3H), 1.14 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 166.0, 161.9, 158.6, 136.8, 131.8, 128.5, 128.4, 128.2, 122.4, 117.0, 106.3, 78.4, 72.8, 55.9, 51.9, 43.2, 26.2, 25.8, 23.6; **MS** (EI) *m/z* (%): 374 (M^+, <1), 283 (5), 225 (6), 195 (20), 193 (90), 163 (50), 91 (100), 43 (22); **HRMS** (ESI): calcd for $C_{21}H_{27}O_6Na$ [M+ Na]$^+$: 397.1622; Found 397.1631

(R)-methyl 2-(benzyloxy)-3-((3,3-dimethyloxiran-2-yl)methyl)-4-methoxybenzoate (8)



To a solution of **7** (2.60 g, 6.95 mmol) in CH_2Cl_2 (60 mL), pyridine (1.13 mL, 13.9 mmol) and MsCl (0.80 mL) was added at 0 °C. Then the reaction mixture was stirred for 12 h at room temperature. After completion of the reaction, the volatiles were evaporated and the residue were dissolved in MeOH (30 mL) and treated with K_2CO_3 (1.82 g, 13.2 mmol) by refluxing for 4 h. The solvent was removed in vacuo and the residue was diluted by Et_2O (70 mL), washed by water (10 mL), dried (Na_2SO_4), and concentrated. The residue was purified by flash chromatography (Petroleum ether/EtOAc 8:1), which afforded **8** (1.22 g, 49%) as a colorless oil. $[\alpha]_D^{20}$ +5.0° (*c* 1.0, $CHCl_3$) **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 7.89 (d, *J* = 8.8 Hz, 1H), 7.52 (d, *J* = 7.2 Hz, 2H), 7.31-7.41 (m, 3H), 6.74 (d, *J* = 8.8 Hz, 2H), 5.00 (s, 2H), 3.90 (s, 3H), 3.86 (s, 3H), 2.95-3.02 (m, 2H), 2.85 (dd, *J* = 4 Hz, *J* = 11.6 Hz, 1H), 1.31 (s, 3H), 1.26 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 166.2, 162.2, 158.7, 137.3, 131.8, 128.4, 128.2, 127.9, 121.6, 117.1, 106.0, 77.1, 63.4, 59.0, 55.7, 51.9, 24.8, 23.8, 19.0; **MS** (EI) *m/z* (%): 356(M^+, <1), 285 (3), 265 (4), 253 (7), 207 (13), 195 (12), 163 (24), 91 (100), 65 (6), 40 (39), **HRMS** (ESI): calcd for $C_{21}H_{25}O_5$ [M+ H]$^+$: 357.1697; Found 357.1699.

Preparation of Compound 2 and 9a

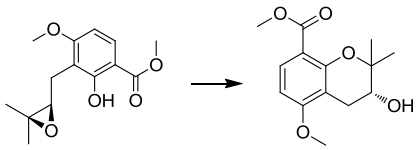


To a solution of olefin **3** (250 mg, 1 mmol) and chiral ketone **18** (91 mg, 0.3 mmol) in 2:1 dimethoxymethane (12 mL) and acetonitrile (6 mL) were added tetrabutylammonium sulfate (14 mg, 0.04 mmol) and an aqueous buffer solution (3.6 mL) containing $Na_2B_4O_7$ (0.05 M) and Na_2EDTA (0.004 M). To this cooled (0 °C), rapidly stirring biphasic mixture were simultaneously added, via two syringe pumps, a solution of Oxone (6.5 mL, 0.25 M) in 0.004 M aqueous Na_2EDTA and an aqueous solution of K_2CO_3 (333 mg, 6.5 mL) over the course of 12 h. The reaction mixture was then stirred for a further 10 h until TLC analysis indicated complete consumption of substrate. The reaction was diluted with EtOAc (300 mL) and washed with water (x 2) and brine, dried over MgSO4 and concentrated in vacuo. The residue was purified by flash chromatography (Petroleum ether/EtOAc 4:1), which afforded **2** as a colorless solid. **(R)-2-((3,3-dimethyloxiran-2-yl)methyl)-3,6-dimethoxyphenol**

(2): yield :95%**;** 74% ee $[\alpha]_D^{20}$ +4.2° (*c* 1.43, $CHCl_3$) **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 11.11 (s,1H), 7.74 (d, *J* = 8.8 Hz, 1H), 6.45 (d, *J* = 8.8 Hz, 1H), 3.90 (s, 3H), 3.87 (s, 3H), 3.04 (dd, *J* = 4.8 Hz, *J* = 13.2 Hz, 1H), 2.96 (dd, *J* = 4.8 Hz, *J* = 6.8 Hz, 1H), 2.82 (dd, *J* = 6.8 Hz, *J* = 13.6), 1.41 (s, 3H), 1.26 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 170.6, 163.1, 160.9, 130.0, 113.2, 105.9, 102.2, 63.2, 59.0, 55.6, 51.9, 24.8, 22.5, 19.0; (EI) *m/z* (%): 266 (M^+, 11), 223 (41), 207 (40),191 (23), 163 (100), 148 (24), 133 (39), 105 (21), 77 (10), 43 (6), **HRMS** (ESI): calcd for $C_{14}H_{19}O_5$ [M+ H]$^+$: 267.1227; Found 267.1221. When the reaction mixture was then stirred for a further 24 h until TLC analysis indicated complete consumption of substrate. follow the same producer, The residue was purified by flash chromatography (Petroleum ether/EtOAc 4:1), which afforded **9a** as a colorless solid.

(S)-2-(4,7-dimethoxy-2,3-dihydrobenzofuran-2-yl)propan-2-ol (9a): yield 92%; 77% ee $[\alpha]_D^{20}$ +22.0° (*c* 1.0, $CHCl_3$) **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 7.67 (d, *J* = 8.8 Hz, 1H), 6.37 (d, *J* = 8.8 Hz, 1H), 4.71 (t, *J* = 8.8 Hz, 1H), 3.81 (s, 3H), 3.80 (s, 3H), 3.20 (br, s, 1H), 3.02 (d, *J* = 8.8 Hz, 2H), 1.29 (s, 3H), 1.19 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 165.5, 161.6, 159.7, 131.7, 115.2, 106.1, 103.1, 90.9, 71.5, 55.4, 51.4, 27.4, 25.3, 23.9; (EI) *m/z* (%): 266 (M^+, 2), 219 (17), 207 (100), 208 (96), 177 (18), 175 (26), 163 (14), 148 (19), 133 (15), 117 (21), 59 (19), 43 (42), **HRMS** (ESI): calcd for $C_{14}H_{19}O_5$ [M+ H]$^+$: 267.1227; Found 267.1222.

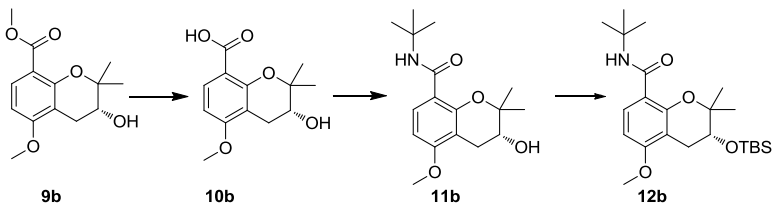
(R)-methyl 3-hydroxy-5-methoxy-2,2-dimethylchroman-8-carboxylate (9b)



2 **9b**

To a solution of **2** (128 mg, 0.48 mmol) in EtOAc (15 mL) was added formic acid (0.1 ml). Then the reaction mixture was stirred for 2 h at room temperature. After completion of the reaction, it was diluted with EtOAc (20 mL). The organic solution was washed sequentially with water (5 mL) and brine (5 mL). The organic layer was dried over Na_2SO_4, filtered and concentrated in vacuo. The resulting brown oil was purified by flash silica gel chromatography (Petroleum ether/EtOAc 1:2) to afford the desired Methyl ester **9b** (115 mg, 90%) as a white solid. $[\alpha]_D^{20}$ 4.0° (*c* 1.0, $CHCl_3$) **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 7.77 (d, *J* = 8.8 Hz, 1H), 6.46 (d, *J* = 8.8 Hz, 2H), 3.87 (s, 6H), 3.86 (s, 6H), 2.91 (dd, *J* = 5.2 Hz, *J* = 17.6 Hz, 1H), 2.69 (dd, *J* = 5.6 Hz, *J* = 17.6 Hz, 1H), 1.40 (s, 3H), 1.36 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 166.4, 161.3, 154.2, 131.3, 112.4, 108.9, 101.5, 77.32, 68.74, 55.6, 51.5, 26.5, 24.6, 21.6; **MS** (EI) *m/z* (%): 266 (M^+, 33), 207 (13), 195 (27), 164 (28), 163 (100), 136 (13), 133 (16), 105 (8), 43 (7); **HRMS** (ESI): calcd for $C_{14}H_{19}O_5$ [M+ H]$^+$: 267.1227; Found 267.1230.

Preparation of compound 10, 11 and 12



9b **10b** **11b** **12b**

(R)-3-hydroxy-5-methoxy-2,2-dimethylchroman-8-carboxylic acid (10b)

Methyl ester **9b** (680 mg, 2.56 mmol) was saponified in THF: MeOH: H_2O (3:1:1, 30 mL) in a 50 mL vial equipped with a stir bar. $LiOH{\cdot}H_2O$ (859 mg, 20.48 mmol) was added to the suspension, and the reaction was allowed to stir until completion (4 h) as determined by TLC. The reaction mixture was diluted with brine (3 mL) and acidified to pH 2 with 1 M HCl (pH paper), resulting in a biphasic solution. The upper layer (THF) was removed, and the aqueous layer was extracted with THF (63 mL). The combined organic layers were dried with Na_2SO_4 and then concentrated under reduced pressure to afford acid **10b** as a white solid. $[\alpha]_D^{20}$-13° (*c*1.0, $CHCl_3$) **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 8.00 (d, *J* = 8.8 Hz, 1H), 6.58 (d, *J* = 8.8 Hz, 1H), 3.92 (t, *J* = 5.6 Hz, 1H), 3.88 (s, 3H), 2.95 (dd, *J* = 5.6 Hz, *J* = 17.6 Hz, 1H), 2.70 (dd, *J* = 6.0 Hz, *J* = 17.6 Hz, 1H), 1.45 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 165.9, 162.2, 152.4, 133.0, 110.0, 108.8, 103.6, 80.8, 68.1, 55.8, 26.1, 24.9, 21.5; **MS** (EI) *m/z* (%): 252 (M^+, 40), 193 (17), 181 (17), 164 (35),163 (100), 136 (23), 105 (13), 57 (22), 43 (31); **HRMS** (ESI): calcd for $C_{13}H_{17}O_5$ $[M+H]^+$: 253.1071; Found 253.1074.

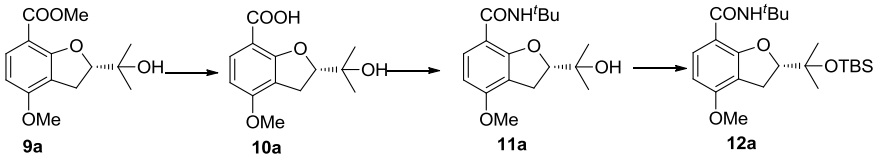
(R)-N-(tert-butyl)-3-hydroxy-5-methoxy-2,2-dimethylchroman-8-carboxamide (11b)

A scintillation vial containing acid **10b** was charged with DMF (10.0 mL) and cooled to 0 °C. Diisopropylethylamine (1.31 mL, 2.56 mmol, 1.0 equiv) was added dropwise to the flask. ^{t}BuNH$_2$ (0.31 mL, 5.12 mmol, 2 equiv) was then added to the flask. 2-(1H-Benzotriazole-1-yl)-1,1,3,3-tetramethylaminiumtetrafluoroborate, TBTU (0.930 g, 2.46 mmol, 0.96 equiv), was then added to the reaction mixture. The reaction mixture was stirred a room temperature for 4 h. then 40 mL EtOAc was added. The organic layer was washed with DI H_2O (2 x 10 mL), 1% phosphoric acid (2 × 10 mL), 2% K_2CO_3 (2 × 10 mL), and brine (2 ×10 mL). Each aqueous layer was back-extracted with EtOAc (2 × 40 mL). The combined organic layers were dried over Na_2SO_4, filtered and concentrated under reduced pressure, the desired product was isolated as white foam and the residue was purified by flash chromatography (Petroleum ether/EtOAc 4:1) to give the desired **11b**. $[\alpha]_D^{20}$-14° (*c* 1.0, $CHCl_3$) **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 8.13 (s, 1H), 8.03 (d, *J* = 8.8 Hz, 1H), 6.52 (d, *J* = 8.8 Hz, 1H), 3.83-3.85 (m, 4H), 2.92 (dd, *J* = 5.6 Hz, *J* = 17.6 Hz, 1H), 2.66 (dd, *J* = 6.4 Hz, *J* = 17.6 Hz, 1H), 1.44 (s, 9H), 1.40 (s, 3H), 1.39 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 164.3, 160.1, 151.4, 131.0, 115.5, 107.9, 102.6, 78.4, 68.6, 55.5, 50.9, 29.0, 26.7, 24.9, 21.8; **MS** (EI) *m/z* (%): 307 (M^+, 22), 235 (66), 217 (33), 163 (56), 132 (30), 91 (100), 57 (23), 43 (22), 40 (53); **HRMS** (ESI): calcd for $C_{17}H_{26}NO_4$ $[M+H]^+$: 308.1856; Found 308.1848.

(R)-N-(tert-butyl)-3-((tert-butyldimethylsilyl)oxy)-5-methoxy-2,2-dimethylchroman-8-carboxamide (12b)

To a solution of the alcohol **11b** and 2,6-lutidine (0.75 mL, 6.4 mmol, 2.5eq) in an hydrous CH_2Cl_2 (15 mL) was added TBSOTf (0.88 mL, 3.84 mmol, 1.5eq) at room temperature and the mixture was stirred at room temperature for 12 h. A saturated aqueous solution of $NaHCO_3$ was added to the reaction mixture and the product was thoroughly extracted with Et_2O. The organic extracts were successively washed with a 3% aqueous solution of $KHSO_4$ twice, a saturated aqueous solution of $NaHCO_3$, and saturated brine, dried over $NaSO_4$, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (Petroleum ether/EtOAc 1:8) to give TBS ether **12b** (1.006 g, 93% from **9b** for three steps). $[\alpha]_D^{20}$-46° (*c* 1.0, $CHCl_3$) **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 8.10 (s, 1H), 8.05 (d, *J* = 8.8 Hz, 2H), 6.52 (d, *J* = 8.8 Hz, 1H), 3.85 (s, 3H), 3.82 (m, 1H), 2.90 (dd, *J* = 6.4 Hz, *J* = 17.2 Hz, 1H), 2.44 (dd, *J* = 9.6 Hz, *J* = 17.2 Hz, 1H), 1.46 (s, 3H), 1.45 (s, 9H), 1.22 (s, 3H), 0.92 (s, 9H), 0.12 (d, *J* = 9.2 Hz); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 164.3, 159.6, 151.4, 130.7, 115.3, 109.5, 102.3, 78.7, 69.9, 55.5, 50.8, 29.01, 27.2, 26.5, 25.7, 18.9, 17.8, -4.1, -5.0; **MS** (ESI) *m/z* (%): $[M+H]^+$: Found 422.3; **HRMS** (ESI): calcd for $C_{23}H_{40}NO_4Si$ $[M+H]^+$: 422.2721;

Found 422.2726.



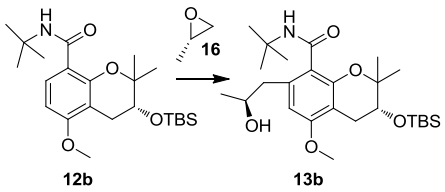
Follow the above operation, **10a**, **11a** and **12a** could be obtained.

(S)-2-(2-hydroxypropan-2-yl)-4-methoxy-2,3-dihydrobenzofuran-7-carboxylic acid (10a) $[\alpha]_D^{20}$ +32° (*c*1.0, $CHCl_3$) **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 7.79 (d, *J* = 8.8 Hz, 1H), 6.47 (d, *J* = 8.8 Hz, 1H), 6.18 (br, s, 1H), 4.81 (t, *J* = 8.8 Hz, 1H), 3.87 (s, 3H), 3.11 (dd, *J* = 9.6 Hz, *J* = 16 Hz, 1H), 3.03 (dd, *J* = 8.8 Hz, *J* = 16 Hz, 1H), 1.35 (s, 3H), 1.21 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 168.1, 161.5, 160.5, 132.8, 115.0, 105.5, 103.9, 91.9, 71.8, 55.6, 27.7, 25.8, 23.5; **MS** (EI) *m/z* (%): 252 (M^+, 3), 223 (26), 207 (64), 191 (21), 175 (32), 163 (100), 149 (48), 133 (37), 106 (45), 71 (33), 57 (45), 42 (76); **HRMS** (ESI): calcd for $C_{13}H_{17}O_5$ [M+ H]$^+$: 253.1071; Found 253.1065.

(S)-N-(tert-butyl)-2-(2-hydroxypropan-2-yl)-4-methoxy-2,3-dihydrobenzofuran-7-carboxamide (11a) $[\alpha]_D^{20}$ -2.0° (*c* 1.0, $CHCl_3$)**^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 7.84 (d, *J* = 8.8 Hz, 1H), 7.41 (s, 1H), 6.47 (d, *J* = 8.8 Hz, 1H), 4.75 (t, *J* = 9.6 Hz, 1H), 3.83 (s, 3H), 3.17 (dd, *J* = 8.8 Hz, *J* = 16 Hz, 1H), 3.07 (dd, *J* = 9.6 Hz, *J* = 16 Hz, 1H), 2.25 (br, s, 1H), 1.43 (s, 9H), 1.36 (s, 3H), 1.26 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 163.5, 158.5, 157.8, 130.8, 113.9, 110.5, 103.9, 91.3, 71.2, 55.4, 50.8, 29.0, 27.3, 26.0, 25.0; **MS** (EI) *m/z* (%): 307 (M^+, 1), 249 (100), 235 (32), 217 (15), 192 (87), 163 (43), 149 (23), 133 (18), 105 (15), 57 (20), 43 (45); **HRMS** (ESI): calcd for $C_{17}H_{26}NO_4$ [M+ H]$^+$: 308.1856; Found 308.1848.

(S)-N-(tert-butyl)-2-(2-((tert-butyldimethylsilyl)oxy)propan-2-yl)-4-methoxy-2,3-dihydrobenzofuran-7-carboxamide (12a) (91% from **9a** for three steps) $[\alpha]_D^{20}$ -7.0° (*c* 1.0, $CHCl_3$)**^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 7.85 (d, *J* = 8.8 Hz, 2H), 7.49 (s, 1H), 6.45 (d, *J* = 8.8 Hz, 1H), 3.85 (dd, *J* = 7.2, 9.6 Hz 1H), 3.82 (s, 1H), 3.18 (dd, *J* = 7.2 Hz, 15.6 Hz, 1H), 2.44 (dd, *J* = 9.6 Hz, 15.6 Hz, 1H), 1.44 (s, 3H), 1.36 (s, 9H), 1.26 (s, 3H), 0.64 (s, 9H), 0.012 (d, *J* = 9.6 Hz); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 163.7, 158.4, 158.3, 130.5, 114.0, 110.2, 103.5, 91.45, 74.13, 55.4, 50.7, 29.0, 27.3, 26.9, 25.6, 25.3, 17.8, -2.3, -2.4. (ESI) *m/z* (%): [M+ H]$^+$: Found 422.4; **HRMS** (ESI): calcd for $C_{23}H_{40}NO_4Si$ [M+ H]$^+$: 422.2721; Found 422.2727.

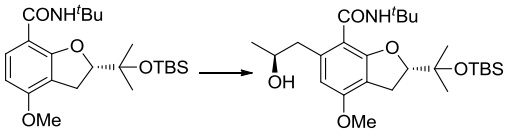
(R)-N-(tert-butyl)-3-((tert-butyldimethylsilyl)oxy)-7-((S)-2-hydroxypropyl)-5-methoxy-2,2-dimethylchroman-8-carboxamide (13b)



To a solution of **12b** (544 mg, 1.30 mmol) in dry THF (10 mL) was added TMEDA (0.78 mL, 5.20 mmol) at -78 °C. To the resulting mixture was added dropwise a solution n-BuLi in hexane (2.5M, 1.77 mL, 5.20 mmol) at -78 °C. After 2 h, to the resulting mixture was added dropwise (s)-(-)-propylene oxide (0.36 mL) at -78 °C. The reaction mixture was stirred at -78 °C for 3 h, and

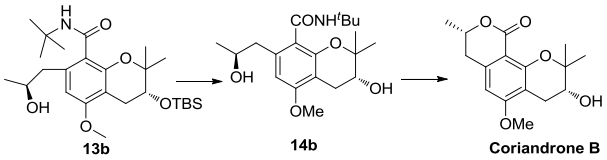
allowed to warm to 25 °C for an additional hour. The resulting mixture was quenched with saturated aqueous NH_4Cl and 1M HCl aqueous, and extracted with EtOAc and CH_2Cl_2. The combined extracts were washed with brine, dried over Na_2SO_4, and concentrated in vacuo. The residue was purified by silica gel chromatography (Petroleum ether/EtOAc 2:1 to 1:1) to afford **13b** (334 mg, 54%) as a white crystal. $[\alpha]_D^{20}$ +34° (*c* 1.0, $CHCl_3$); **^{1}H NMR** ($CDCl_3$, 400MHz): δ 6.26 (s, 1H), 5.98 (s, 1H), 5.23 (br, s, 1H), 3.89-3.96 (m, 1H), 3.79 (s, 3H), 3.74 (dd, 1H, *J* = 6.0, 6.6 Hz), 2.80 (dd, *J* = 6, 16.8 Hz, 1H), 2.64-2.71 (m, 2H), 2.39 (dd, *J* = 8.8, 16.8 Hz, 1H), 1.41 (s, 9H), 1.33 (s, 3H), 1.25 (d, *J* = 6.4 Hz, 3H), 1.17 (s, 3H), 0.88 (s, 9H), 0.08 (d, *J* = 7.2 Hz, 6H). **^{13}C NMR** ($CDCl_3$, 100MHz): δ 167.6, 157.8, 150.0, 138.4, 119.1, 107.3, 103.7, 77.4, 69.6, 68.4, 55.2, 51.4, 42.9, 28.6, 26.7, 26.2, 25.6, 24.4, 19.4, 17.7, -4.3, -5.2. **MS** (ESI) *m/z* (%):[M+ H]$^+$: Found 480.4; **HRMS** (ESI): calcd for $C_{26}H_{46}O_5SiN$ [M+ H]$^+$: 480.3140; Found 480.3147.

(S)-N-(tert-butyl)-2-(2-((tert-butyldimethylsilyl)oxy)propan-2-yl)-6-((S)-2-hydroxypropyl)-4-methoxy-2,3-dihydrobenzofuran-7-carboxamide (13a)



Follow the above operation, **13a** could be obtained. **13a** (61% yield) $[\alpha]_D^{20}$ +48° (*c* 1.0, $CHCl_3$); **^{1}H NMR** ($CDCl_3$, 400MHz): δ 6.74 (s, 1H), 6.25 (s, 1H), 5.48 (br, s, 1H), 4.54 (t, *J* = 8.8 Hz, 1H), 3.93 (br, s, 1H), 3.80 (s, 1H), 3.12 (dd, *J* = 7.2, 15.6 Hz, 1H), 2.96-3.04 (m, 2H), 2.84 (dd, *J* = 3.2, 13.2 Hz, 1H), 1.41 (s, 9H), 1.32 (s, 3H), 1.27 (d, *J* = 7.6 Hz, 3H), 1.26 (s, 3H), 0.67 (s, 9H), 0.04 (d, *J* = 17.6 Hz, 6H). **^{13}C NMR** ($CDCl_3$, 100MHz): δ 166.2, 158.8, 156.6, 142.1, 112.5, 111.5, 106.6, 90.9, 74.2, 69.3, 55.3, 51.3, 43.0, 28.8, 27.5, 26.8, 25.5, 25.3, 24.4, 17.8, -2.3, -2.4. **MS** (ESI) *m/z* (%): [M+ H]$^+$: Found 480.4; **HRMS** (ESI): calcd for $C_{26}H_{46}O_5SiN$ [M+ H]$^+$: 480.3140; Found 480.3142.

Preparation of 14, **Coriandrone B and Coriandrone A**



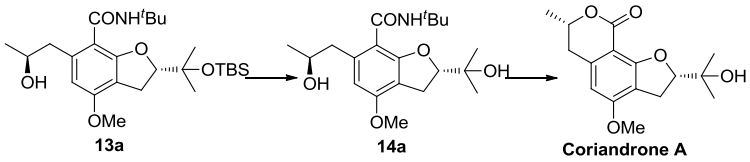
(R)-N-(tert-butyl)-3-hydroxy-7-((S)-2-hydroxypropyl)-5-methoxy-2,2-dimethylchroman-8-carboxamide (14b)

To a solution of **13b** (448 mg, 0.94 mmol) in anhydrous THF (10 mL) was added a 1.0 M solution of TBAF (0.96 mL, 0.96 mmol) in THF at room temperature and the mixture was stirred at room temperature for 2 h. Then THF was removed under reduced pressure, the residue was diluted by Et_2O (30 mL), and the organic solution was washed sequentially with water (5 mL) and brine (5 mL). The organic layer was dried over Na_2SO_4, filtered and concentrated in vacuo, yielded crude diol **14b** (340mg, 99%), then carried on to the next synthetic transformation without purification. $[\alpha]_D^{20}$ +61.0° (*c*2.0, $CHCl_3$) **^{1}H NMR** ($CDCl_3$, 400MHz): δ 6.29 (s, 1H), 6.19 (br, s, 1H), 3.88-3.96 (m, 1H), 3.82 (s, 3H), 2.63-2.77 (m, 3H), 2.31(dd, *J* = 6, 16.8 Hz, 1H), 2.12 (dd, *J* = 9.2, 16.8 Hz, 1H), 1.4 (s, 9H), 1.29 (d, *J* = 6.4 Hz, 3H), 1.21 (s, 3H), 1.09 (s, 3H). **^{13}C NMR** ($CDCl_3$, 100MHz): δ 168.0, 158.3, 150.2, 138.8, 118.4, 108.0, 103.6, 77.80, 68.9, 67.2, 55.3, 51.7, 42.9, 28.6, 25.6, 25.5, 24.6, 19.4. **MS** (ESI)

m/z (%): [M+ H]$^+$: Found 366.3; **HRMS** (ESI): calcd for $C_{20}H_{32}NO_5$ [M+ H]$^+$: 366.2275; Found 366.2267.

(3R,8S)-3-hydroxy-5-methoxy-2,2,8-trimethyl-3,4,7,8-tetrahydropyrano[4,3-h]chromen-10(2H)-one (Coriandrone B)

The crude diol (128 mg, 0.35 mmol) was treated with a mixture of 50% aqueous NaOH (6 mL) and EtOH (6 mL) and the mixture heated under reflux for 12 h. Then EtOH was distilled off and the residue neutralized with concentrated HCl at 0 ^{o}C. The aqueous phase was extracted three times with EtOAc and the collected organic solutions were washed with saturated aqueous $NaHCO_3$ and brine and dried over Na_2SO_4. After evaporation of solvent, the solid residue was purified by flash chromatography (Petroleum ether/EtOAc 1:2) yielding 90 mg (88% yield for two steps) of the **Coriandrone B**. $[\alpha]_D^{20}$ +139.0° (*c*1.1, $CHCl_3$); [lit.$[\alpha]_D^{24}$ +159.1 (*c* 0.87, $CHCl_3$)] **^{1}H NMR** ($CDCl_3$, 400MHz): δ 6.24 (s, 1H), 4.46-4.54 (m, 1H), 3.86 (s, 3H), 3.80 (t, *J* = 5.2 Hz, 1H), 2.79-2.89 (m, 2H), 2.75 (dd, *J* = 3.2 Hz, *J* = 16 Hz, 1H), 2.68 (dd, *J* = 4.8 Hz, *J* = 17.6 Hz), 1.90 (br, s, 1H), 1.46 (s, 3H), 1.46 (d. *J* = 6 Hz, 3H), 1.31 (s, 3H); **^{13}C NMR** ($CDCl_3$, 100MHz): δ 162.7, 161.6, 155.8, 141.4, 107.9, 107.0, 100.7, 77.8, 73.6, 68.4, 55.6, 36.6, 26.4, 24.3, 22.3, 20.7; **MS** (EI) *m/z* (%): 292 (M^+, 40), 233 (23), 221 (100), 206 (38), 188 (17), 176 (21), 149 (24), 91 (18), 75 (57), 57 (21), 43 (50); **HRMS** (ESI): calcd for $C_{16}H_{21}O_5$ [M+ H]$^+$: 293.1384; Found 293.1377.

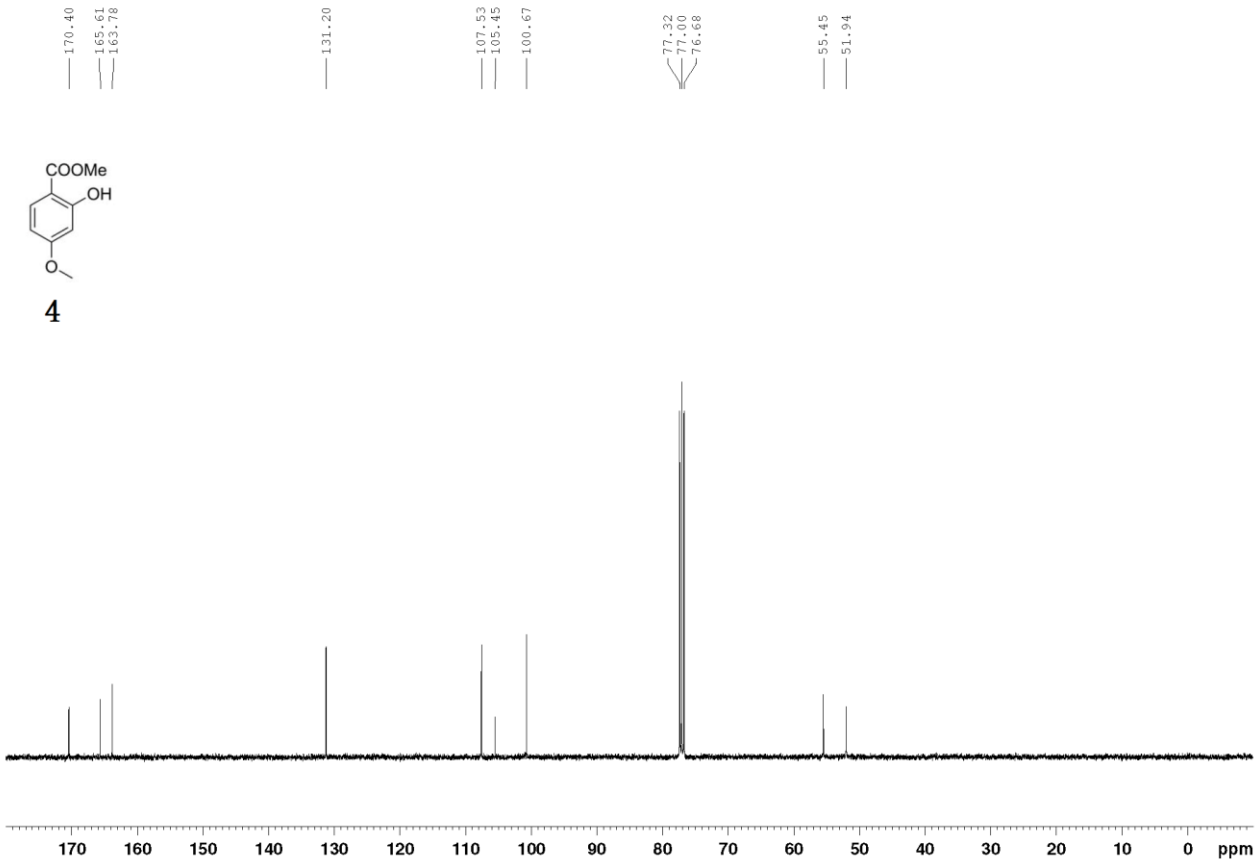


Follow the above operation, **14a** and **Coriandrone A** could be obtained.

(S)-N-(tert-butyl)-2-(2-hydroxypropan-2-yl)-6-((S)-2-hydroxypropyl)-4-methoxy-2,3-dihydrobenzofuran-7-carboxamide (14a) $[\alpha]_D^{20}$ +56.5° (*c*2.0, $CHCl_3$) **^{1}H NMR** ($CDCl_3$, 400MHz): δ7.14 (s, 1H), 6.29 (s, 1H), 6.19 (s, 1H), 5.70 (br, s, 1H), 4.42 (t, *J* = 8.8 Hz, 1H), 3.90 (br, s, 1H), 3.76 (s, 3H), 2.85-2.2.97 (m, 2H), 2.68-2.77 (m, 2H), 1.36 (s, 9H), 1.23 (d, *J* = 6.0 Hz, 3H), 1.18 (s, 3H), 1.12 (s, 3H). **^{13}C NMR** ($CDCl_3$, 100MHz): δ 166.30, 158.5, 156.6, 140.8, 112.6, 112.5, 105.8, 90.5, 70.7, 69.3, 55.1, 51.4, 42.6, 28.7, 27.3, 25.2 (25.0), 24.3. **MS** (ESI) *m/z* (%): [M+ H]$^+$: Found 366.3; **HRMS** (ESI): calcd for $C_{20}H_{32}NO_5$ [M+ H]$^+$: 366.2275; Found 366.2266.

(2S,7S)-2-(2-Hydroxypropan-2-yl)-4-methoxy-7-methyl-6,7-dihydro-2H-furo[3,2-h]isochromen-9(3H)-one (Coriandrone A) (83% yield for two steps) $[\alpha]_D^{20}$ +121.0° (*c*1.08, $CHCl_3$); [lit.$[\alpha]_D^{24}$ +122.5 (*c* 0.99, $CHCl_3$)] **^{1}H NMR** ($CDCl_3$, 400MHz): δ 6.20(s, 1H), 4.76 (t, *J* = 9.2 Hz, 1H), 4.47-4.56 (m, 1H), 3.84 (s, 3H), 2.99 (d, *J* = 9.2 Hz, 2H), 2.79 (dd, *J* = 7.6 Hz, *J* = 11.6 Hz, 2H), 1.44 (d, *J* = 4.8 Hz, 3H), 1.32 (s, 3H), 1.18 (s, 3H); **^{13}C NMR** ($CDCl_3$, 100MHz): δ 163.5, 162.9, 159.8, 141.6, 114.6, 102.0, 101.8, 92.2, 74.4, 71.3, 55.4, 35.8, 27.4, 25.6, 23.5, 20.7. **MS** (EI) *m/z* (%): 292 (M^+, <1), 277 (5), 234 (76), 233 (100), 215 (19), 205 (12), 188 (15), 177 (9), 149 (48), 71 (16), 57 (20), 43 (37); **HRMS** (ESI): calcd for $C_{16}H_{21}O_5$ [M+ H]$^+$: 293.1384; Found 293.1376.

3 ^{1}H and ^{13}C NMR Spectra



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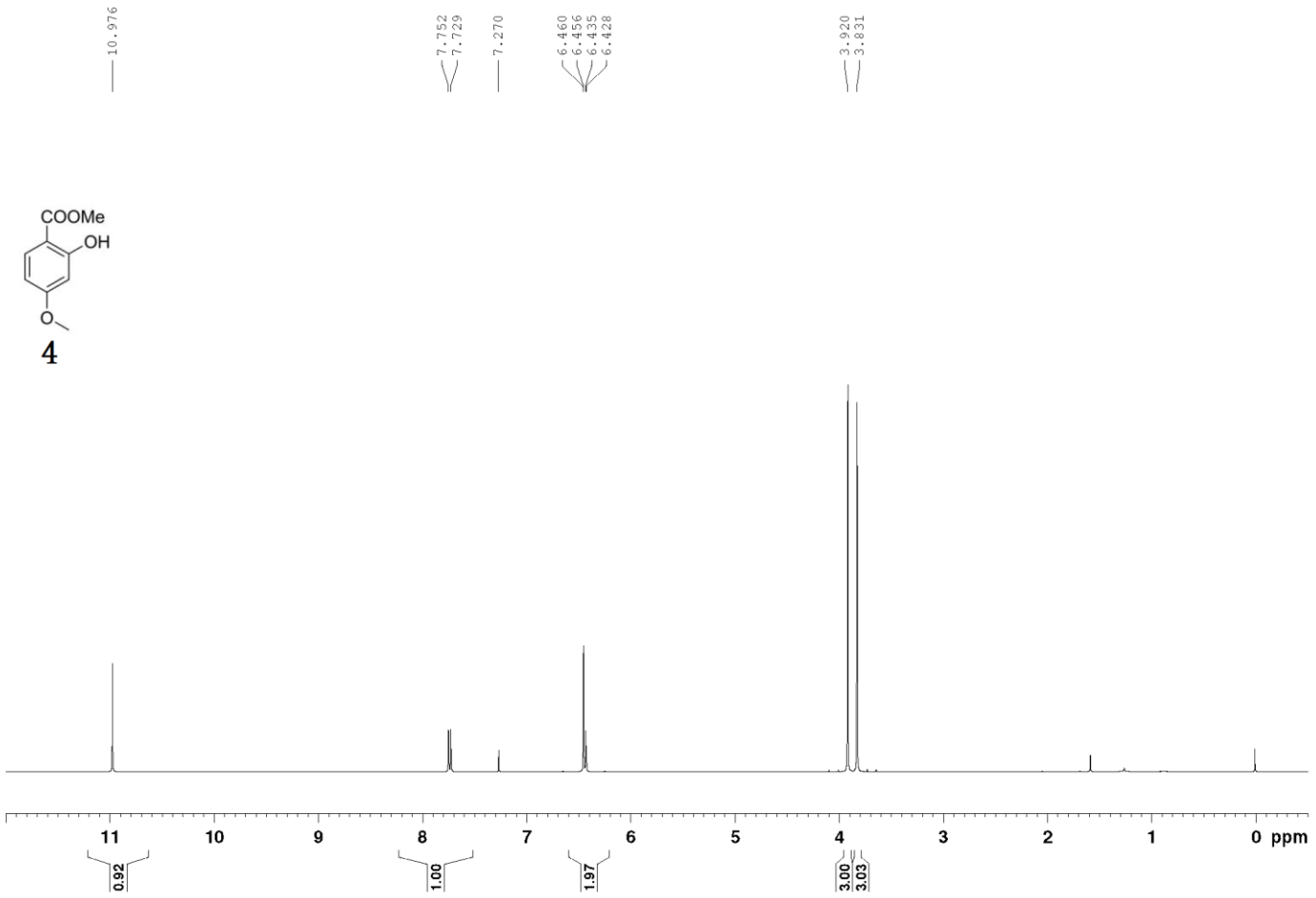

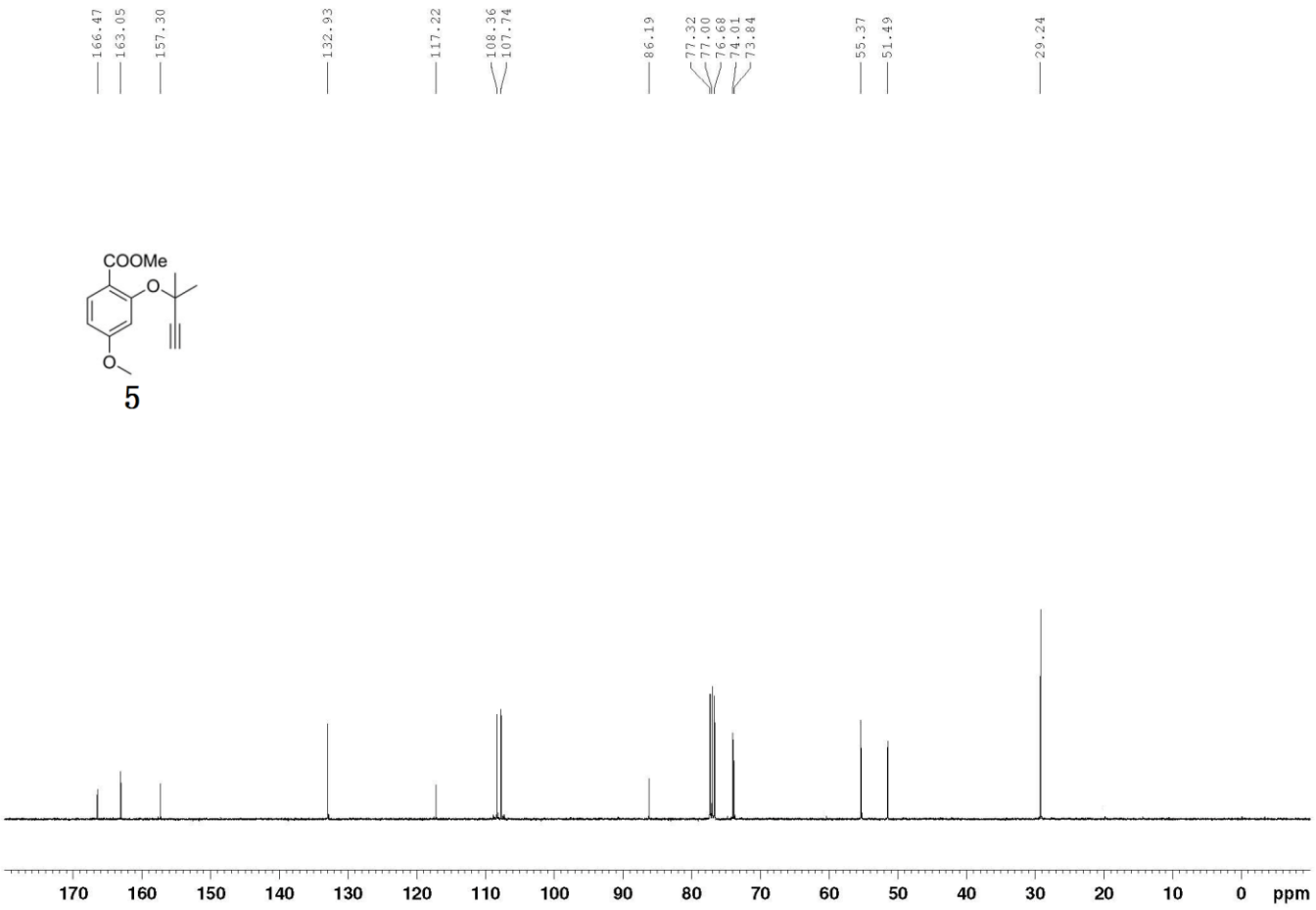

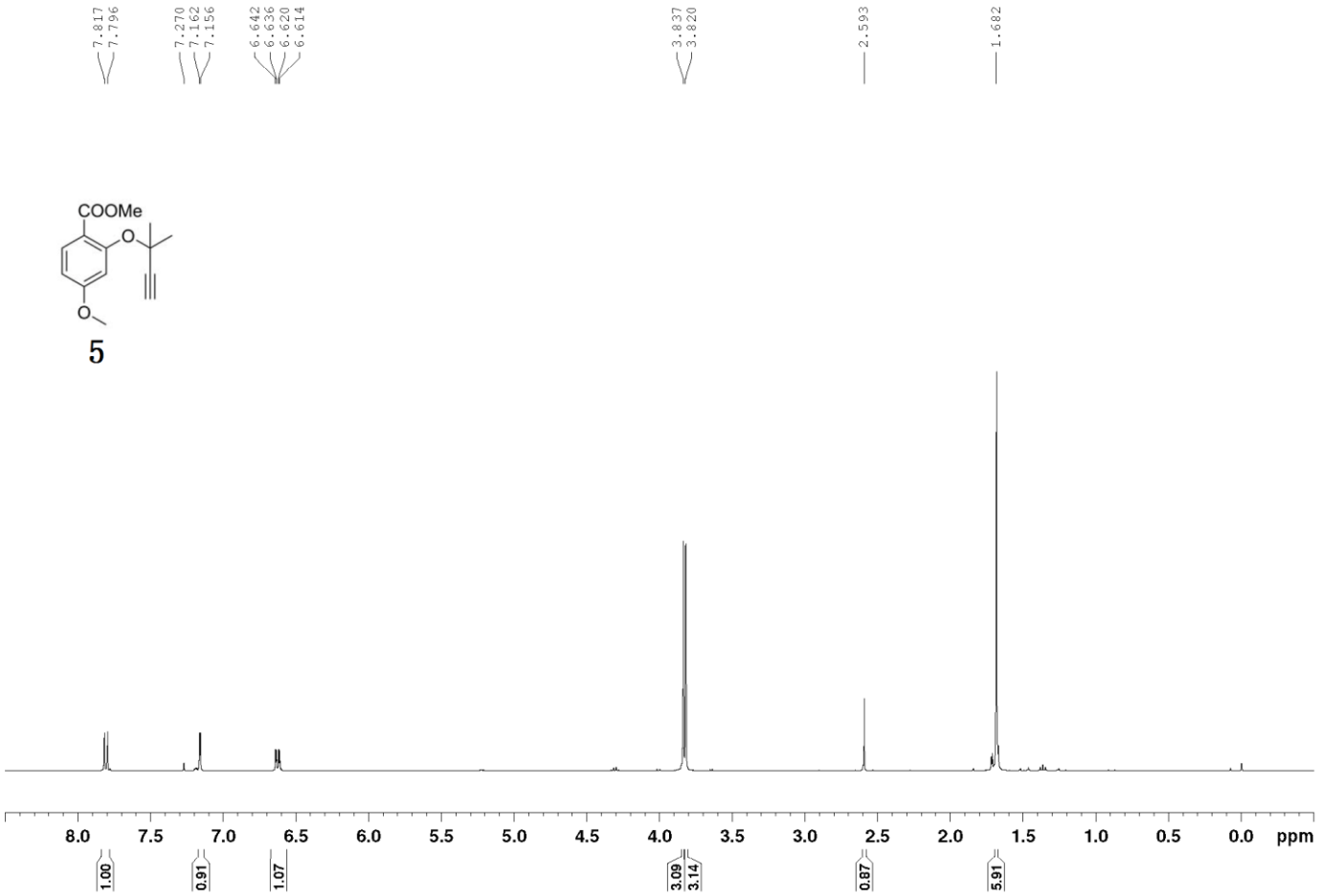

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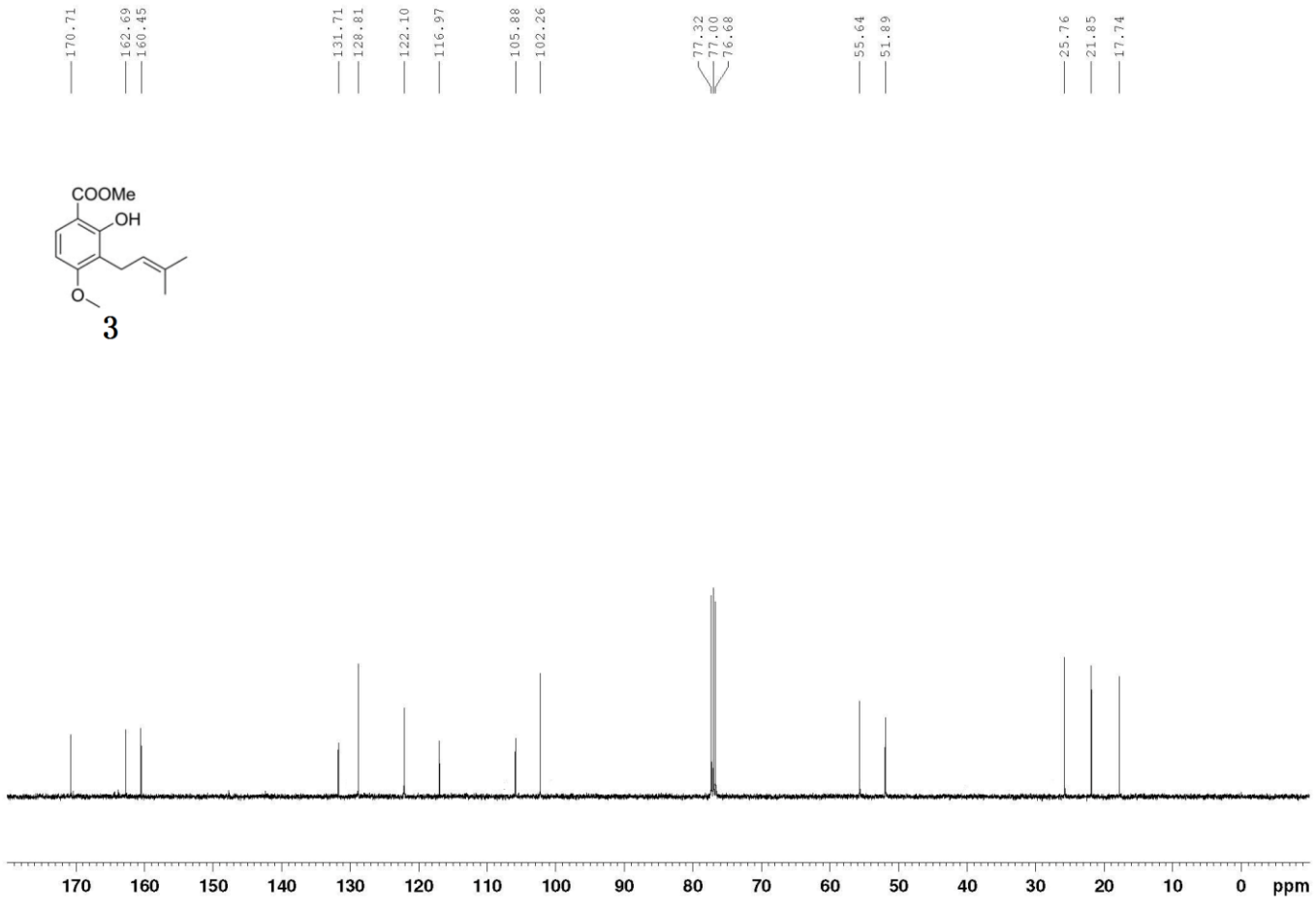

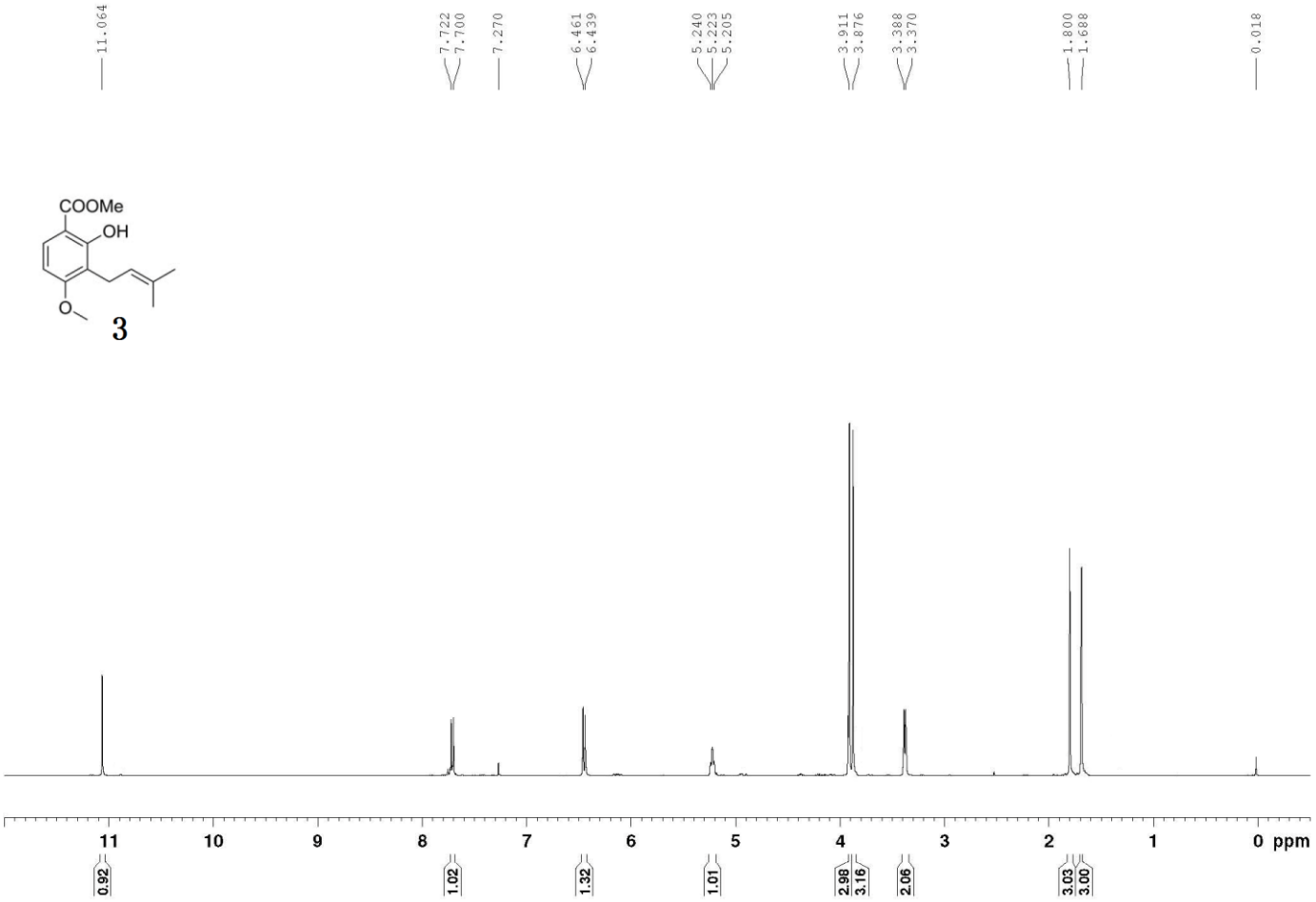

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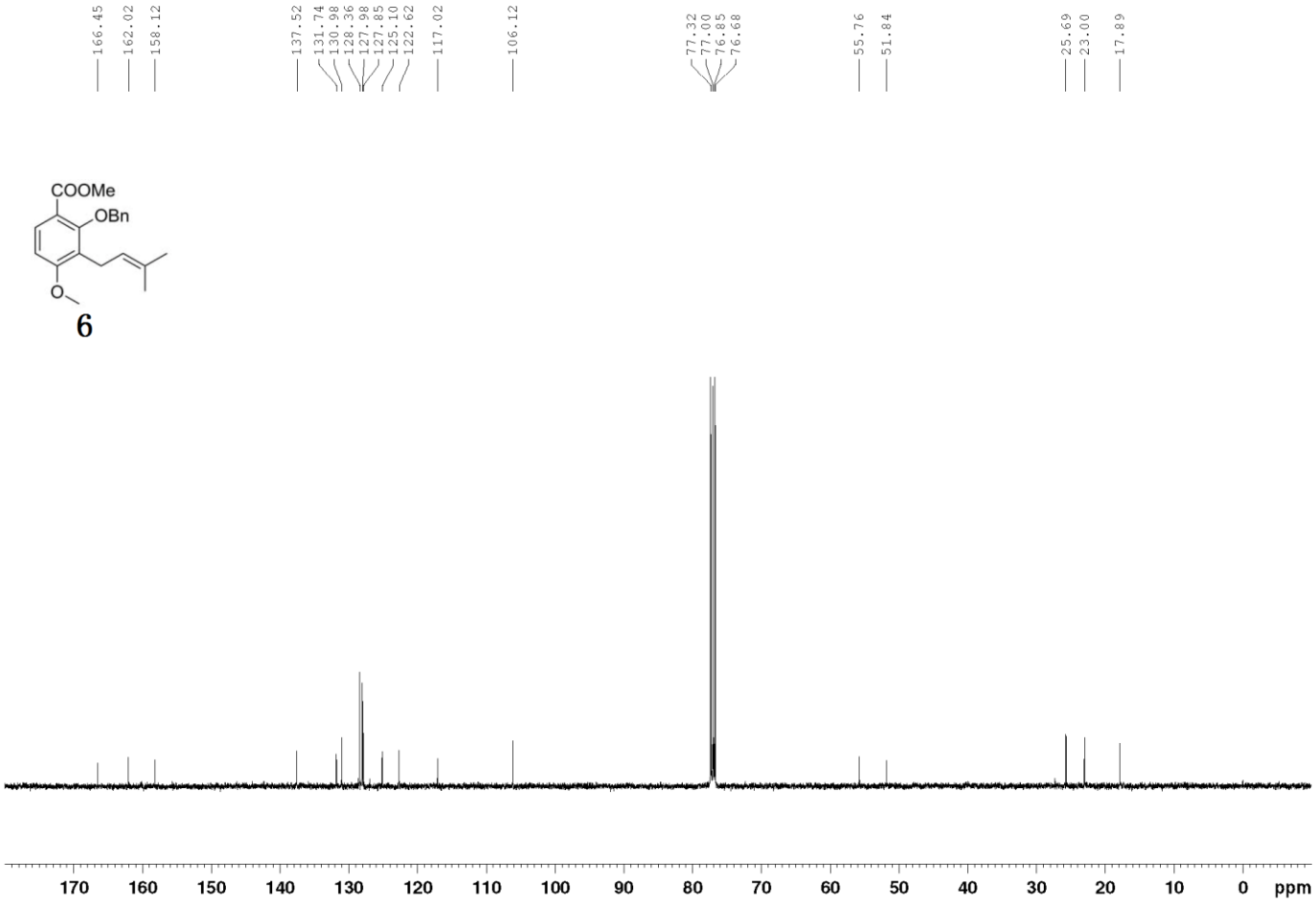

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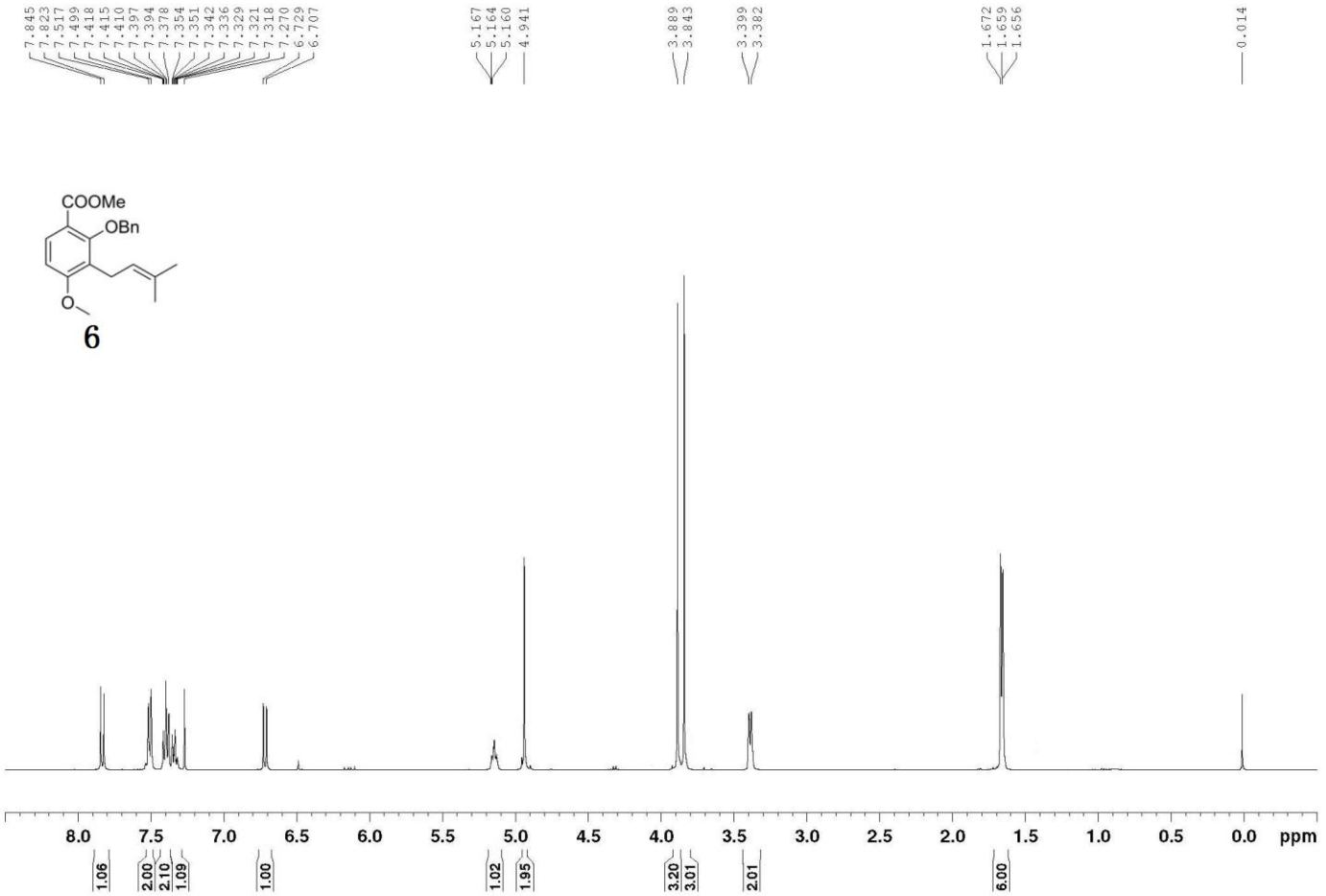

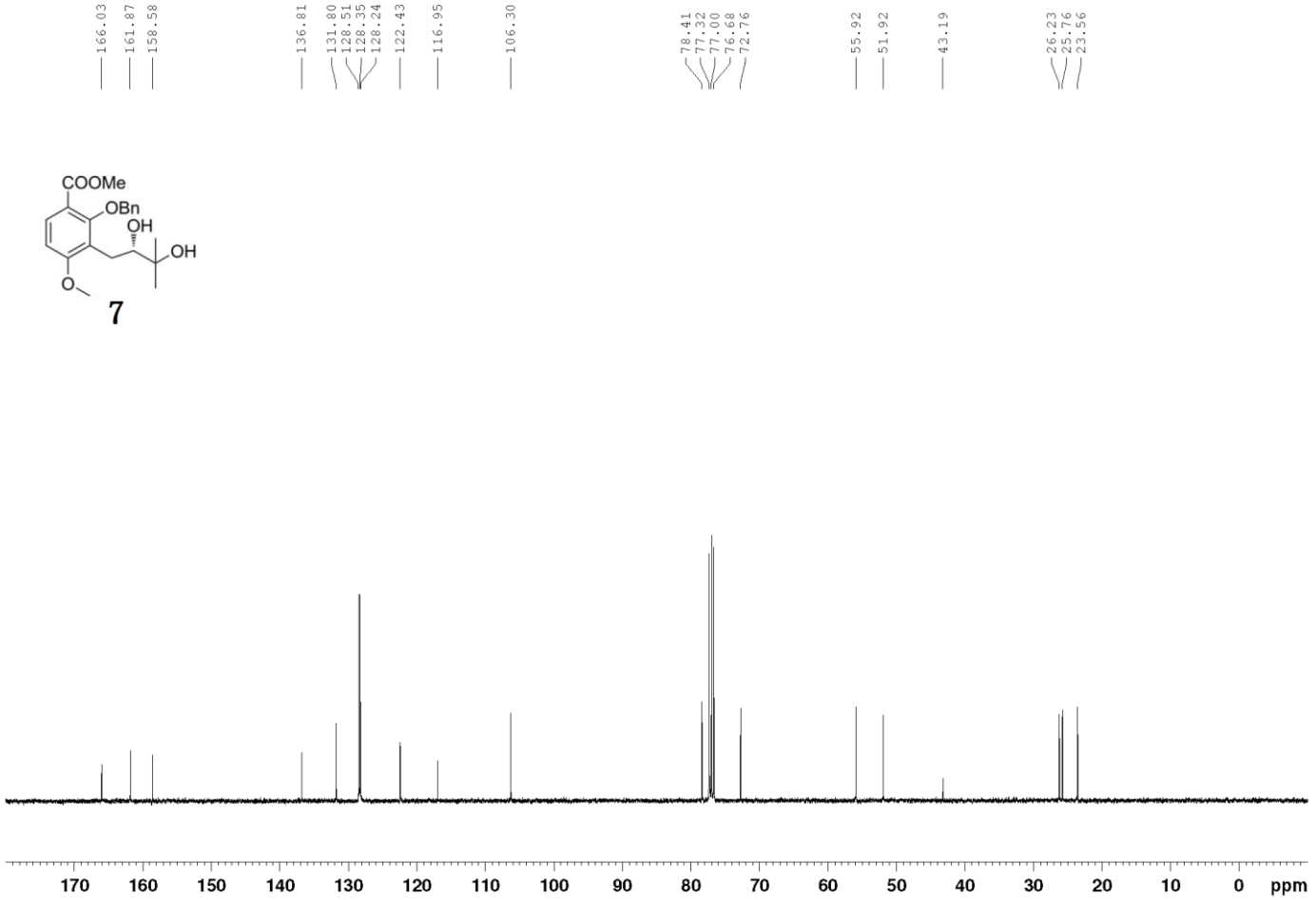

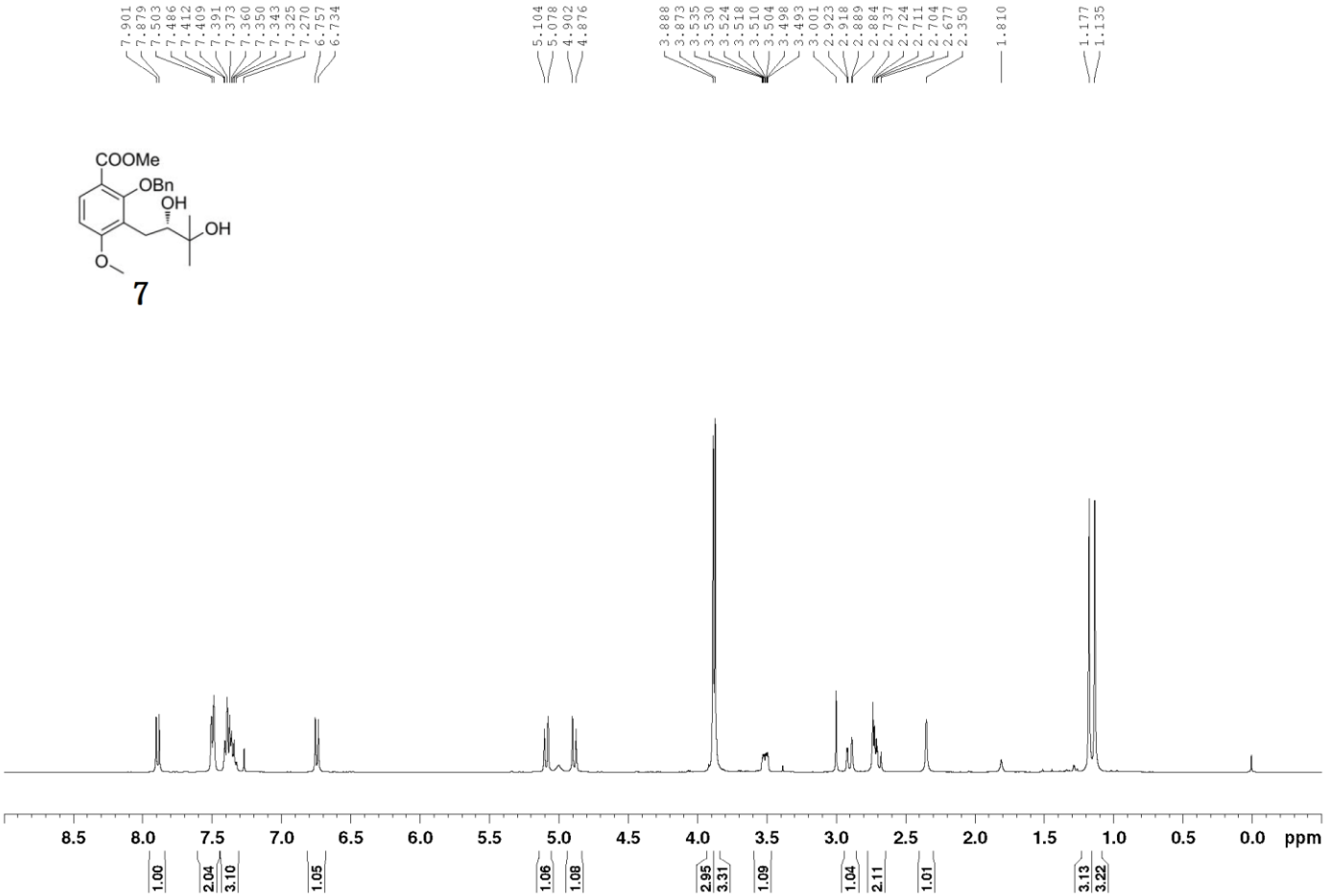

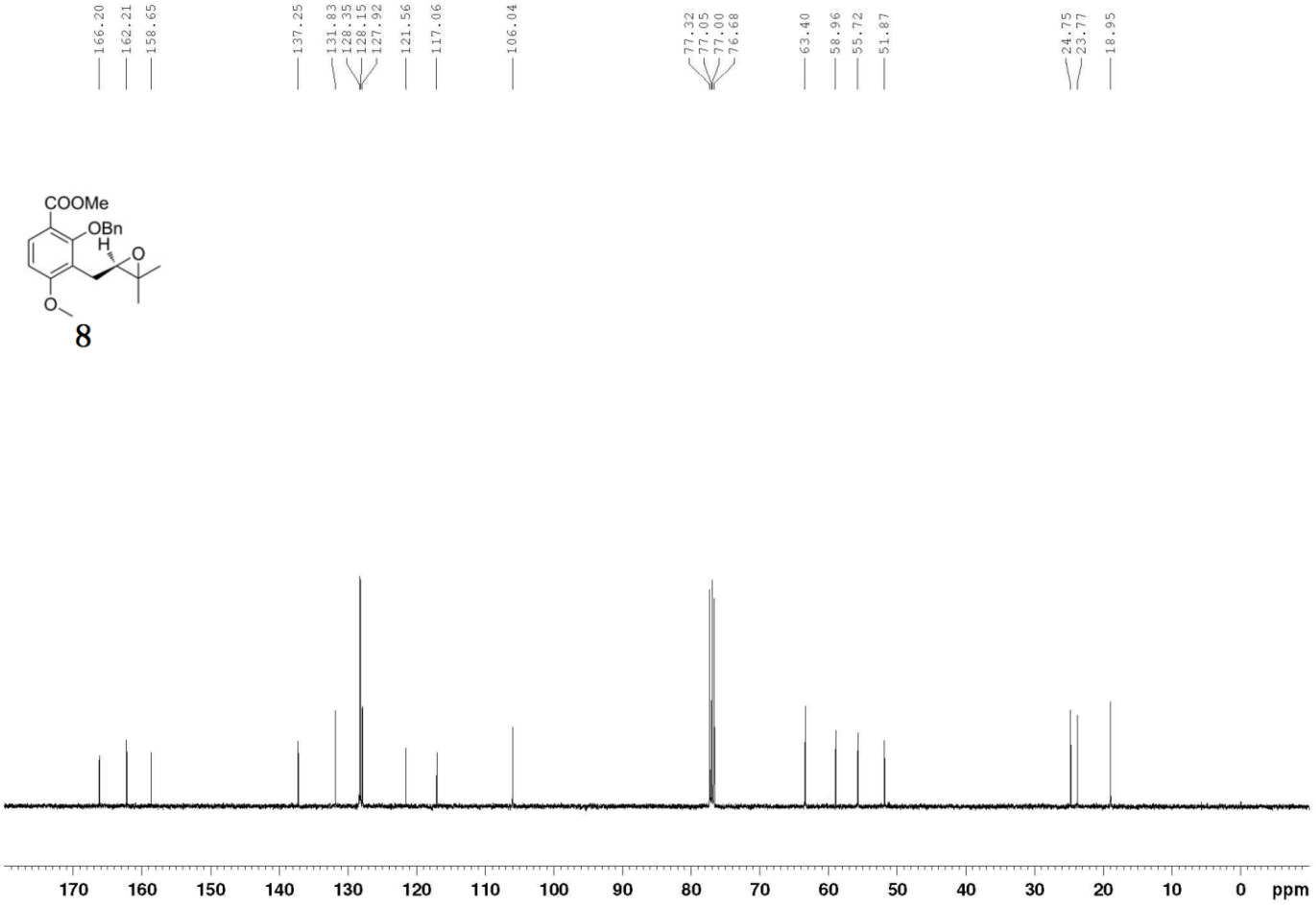

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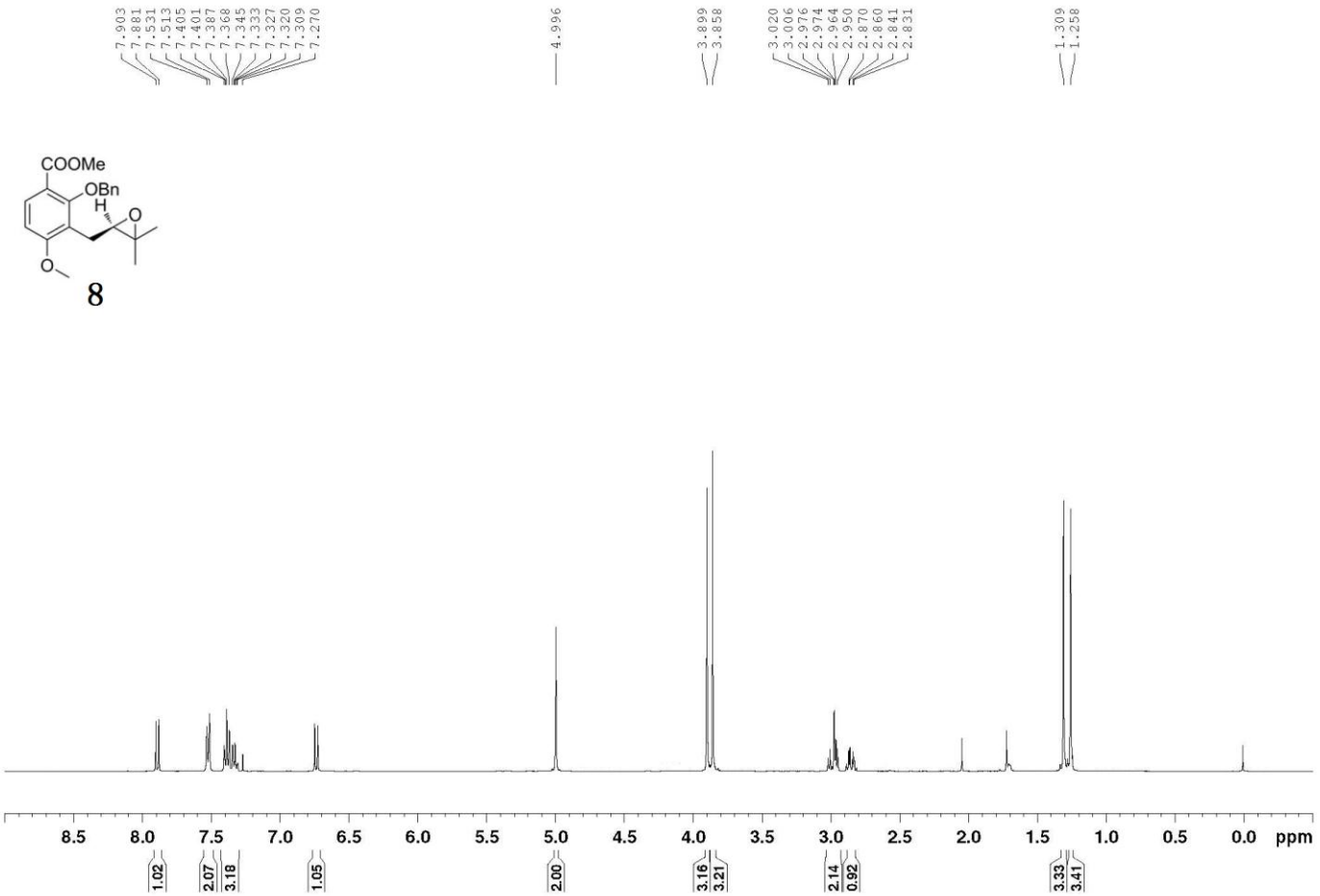
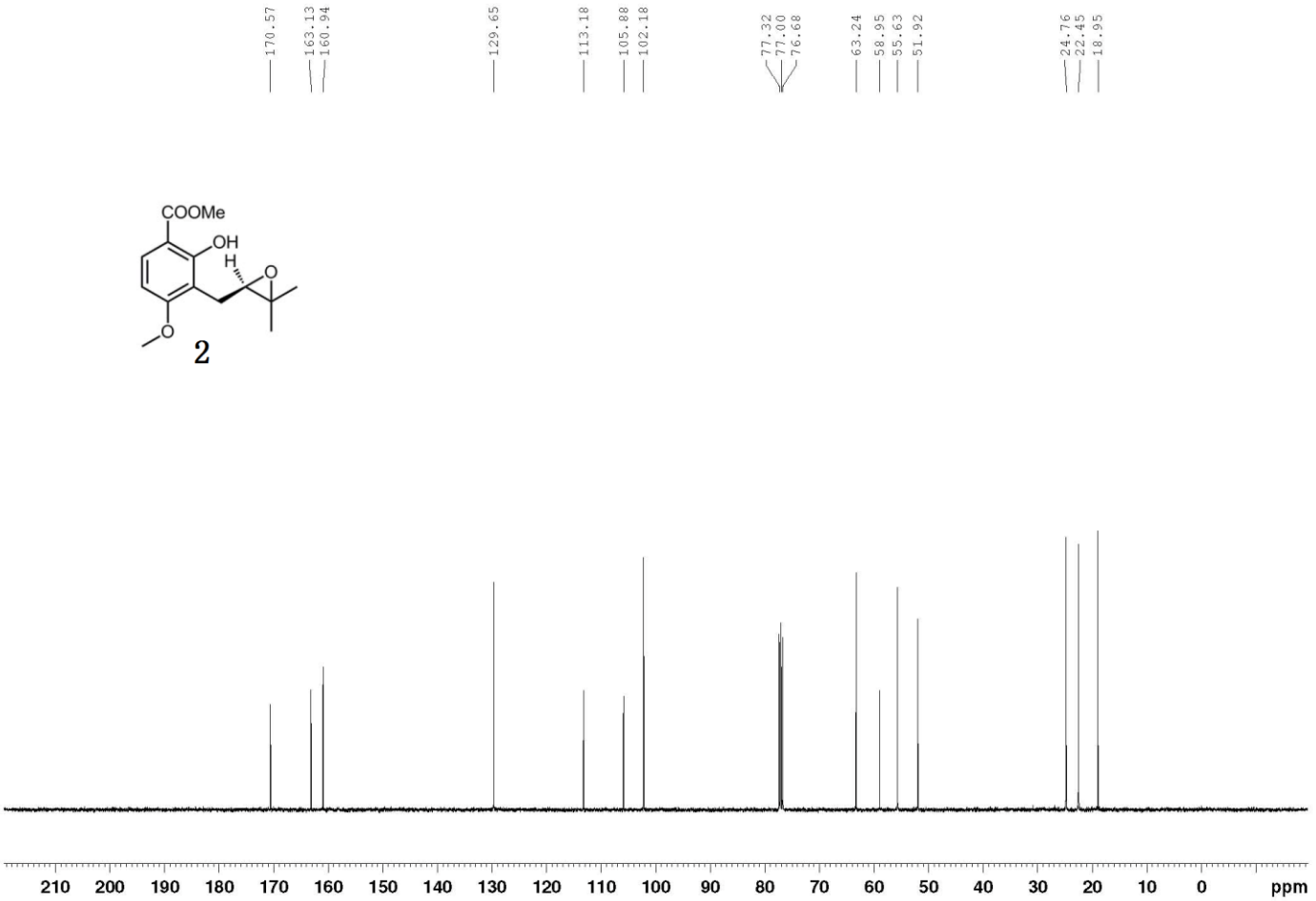
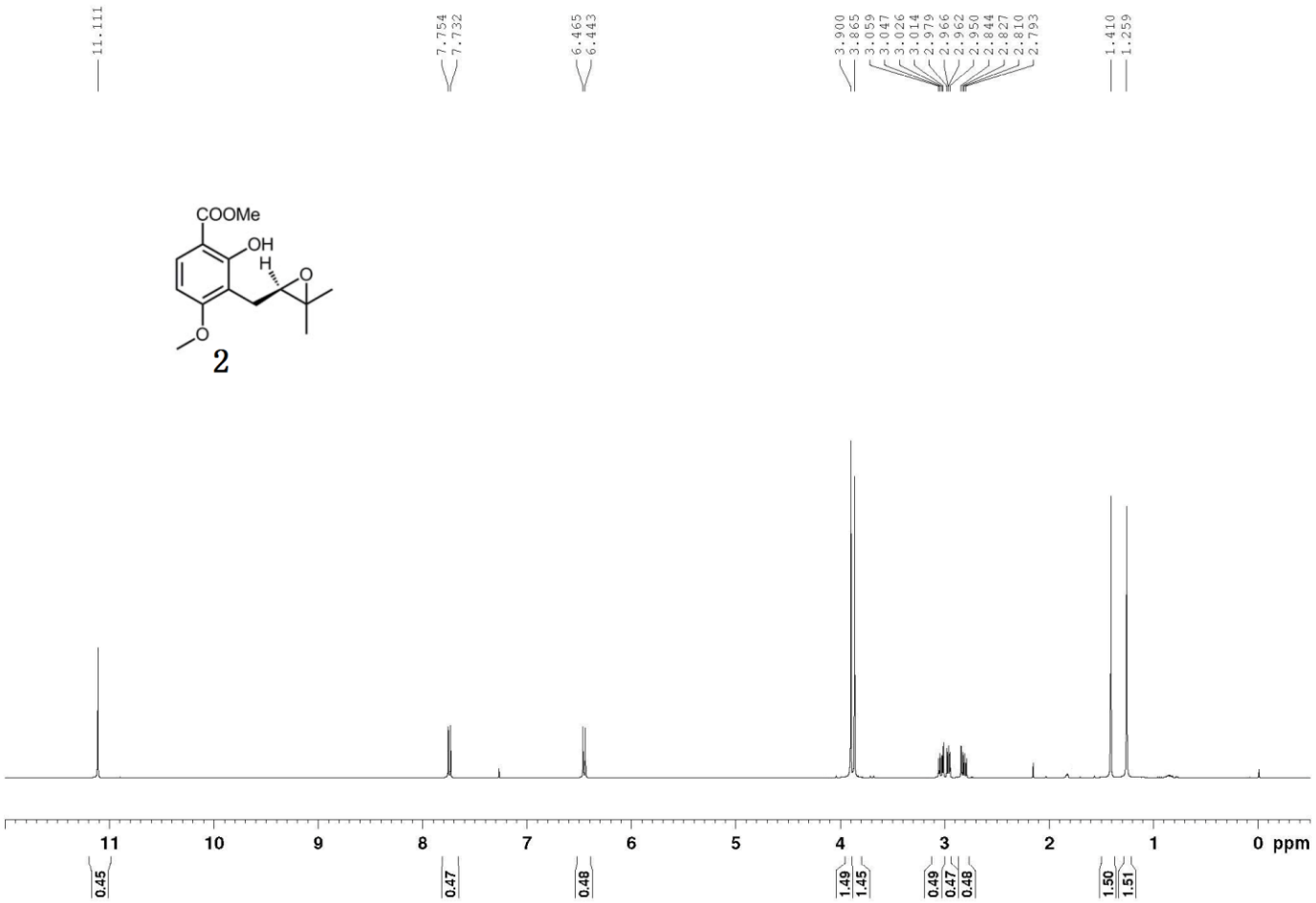
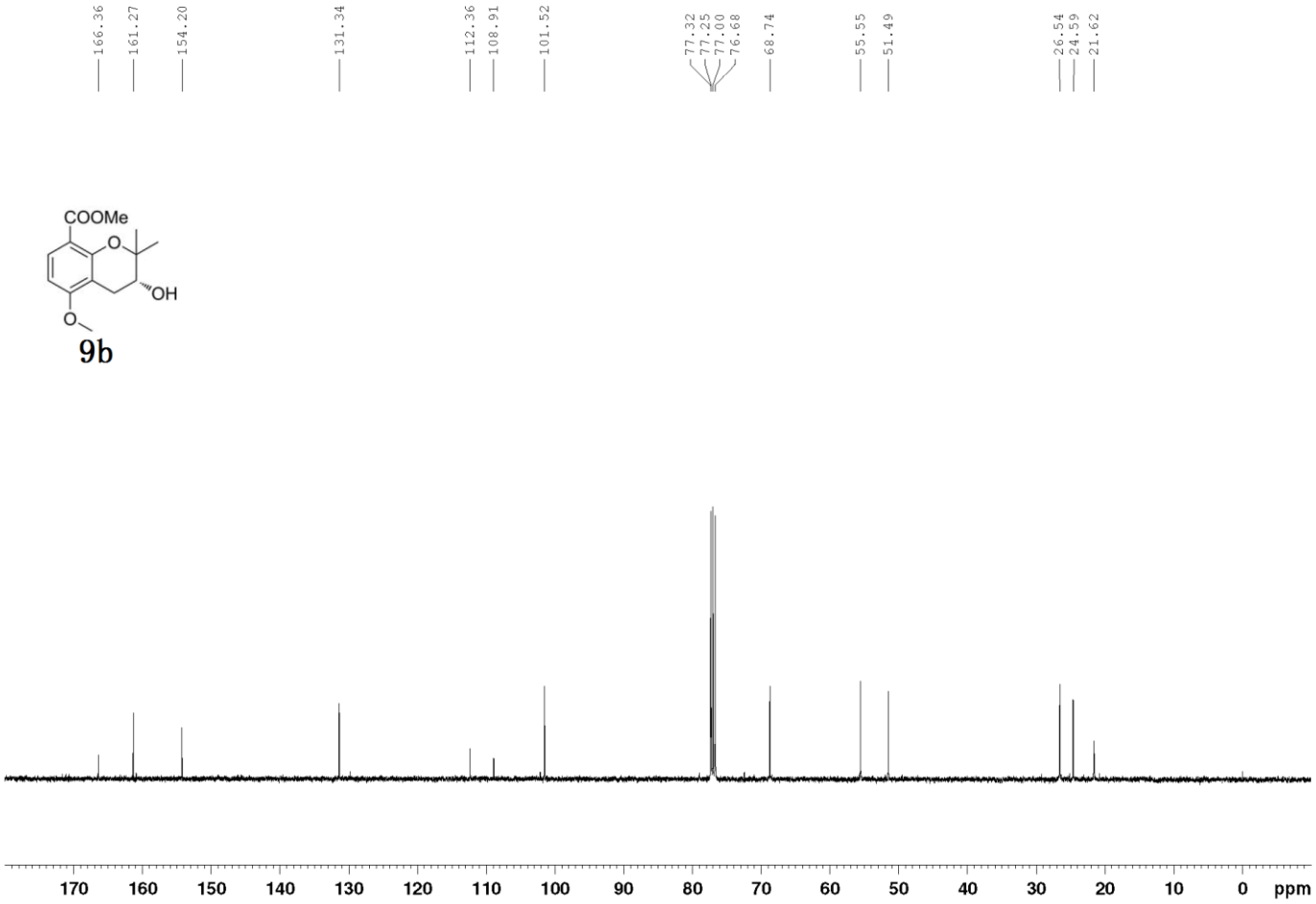
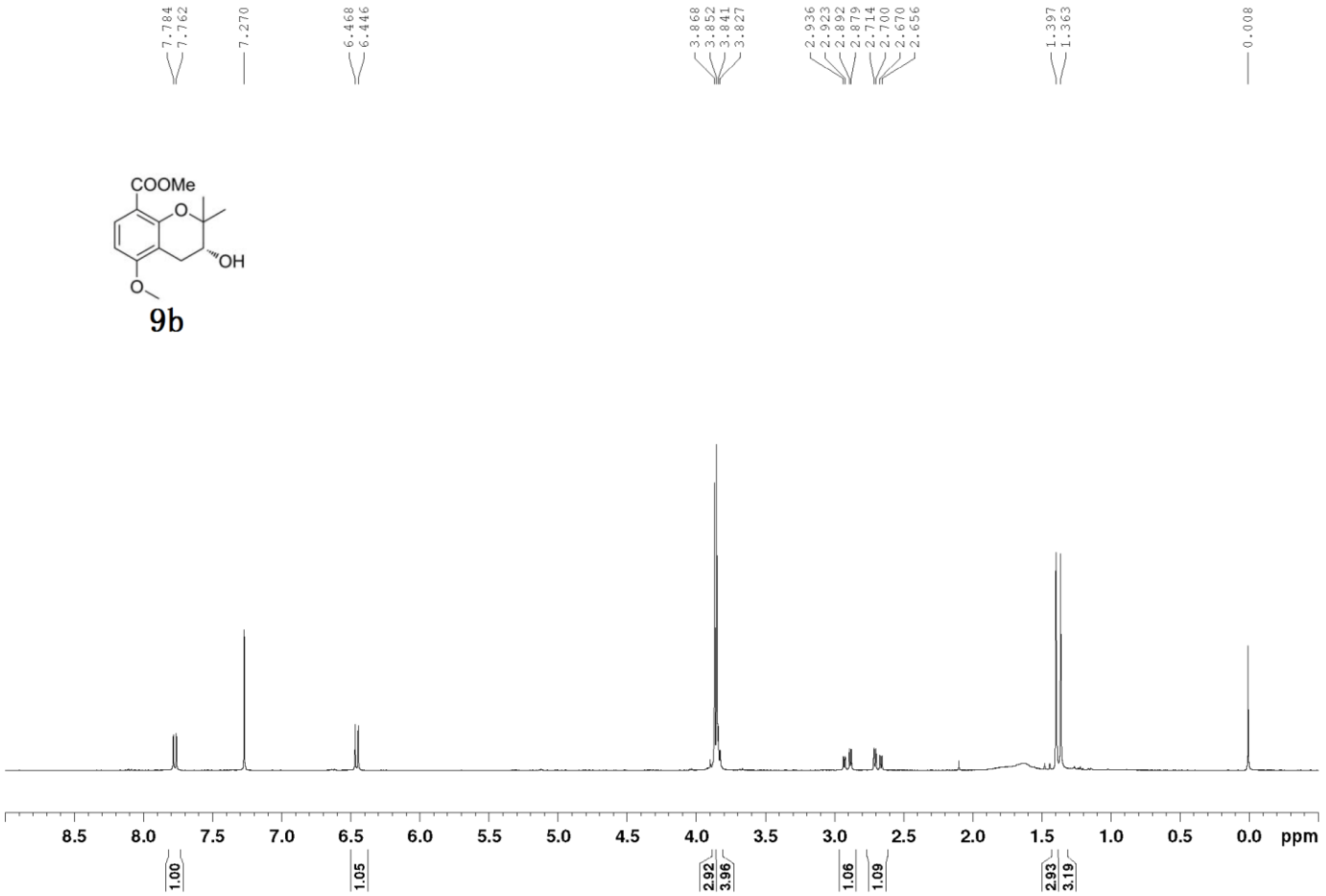


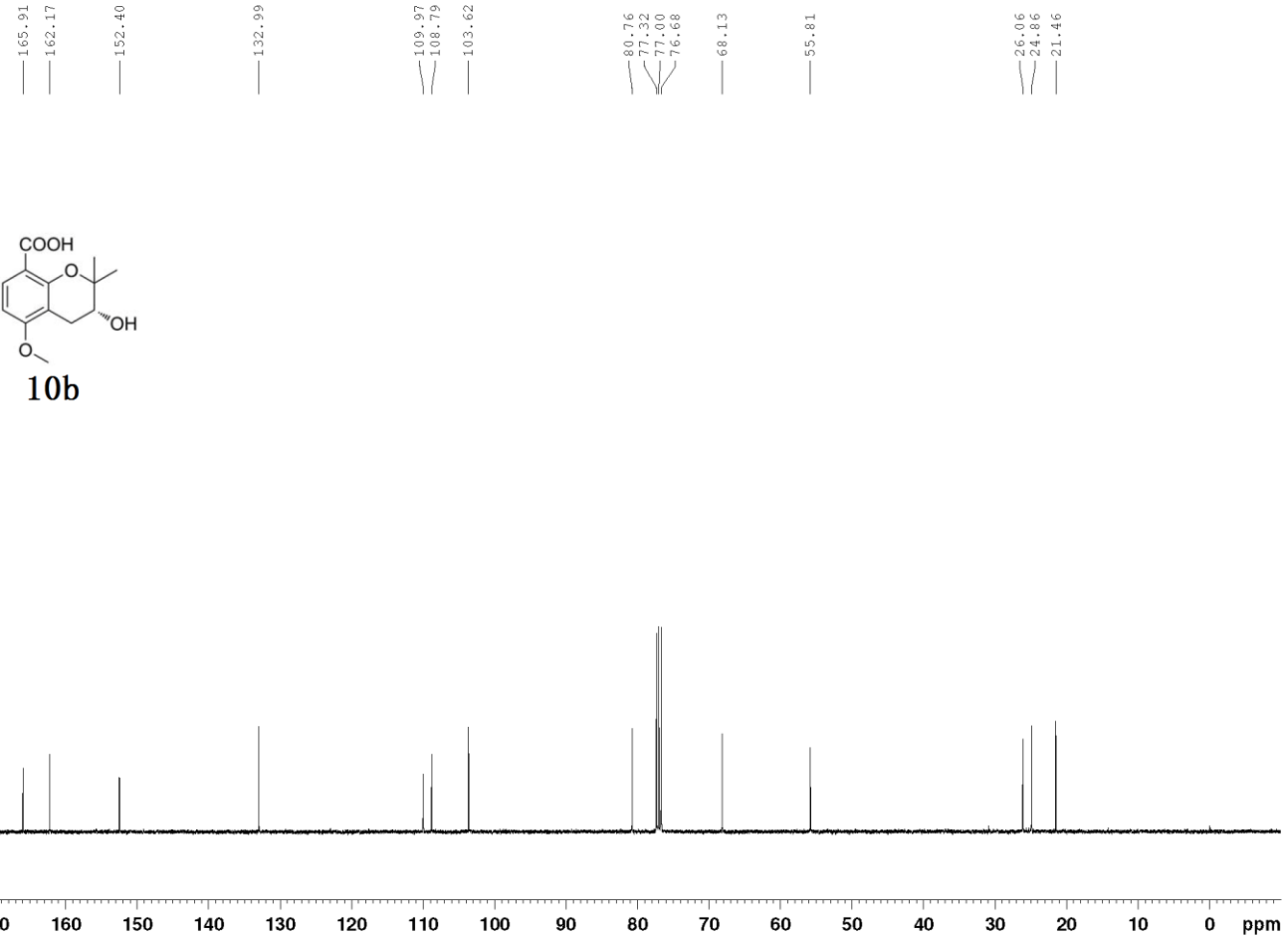




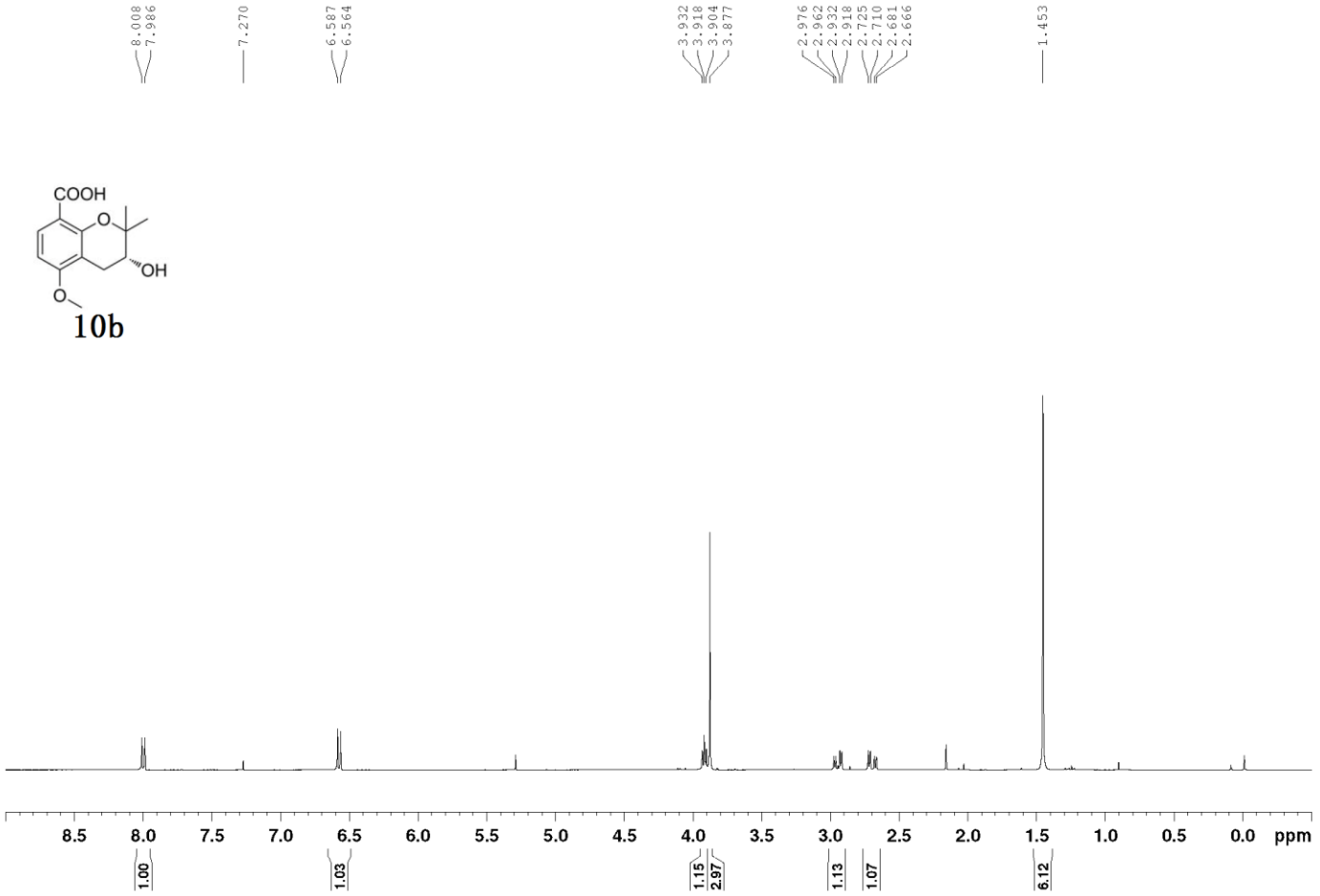


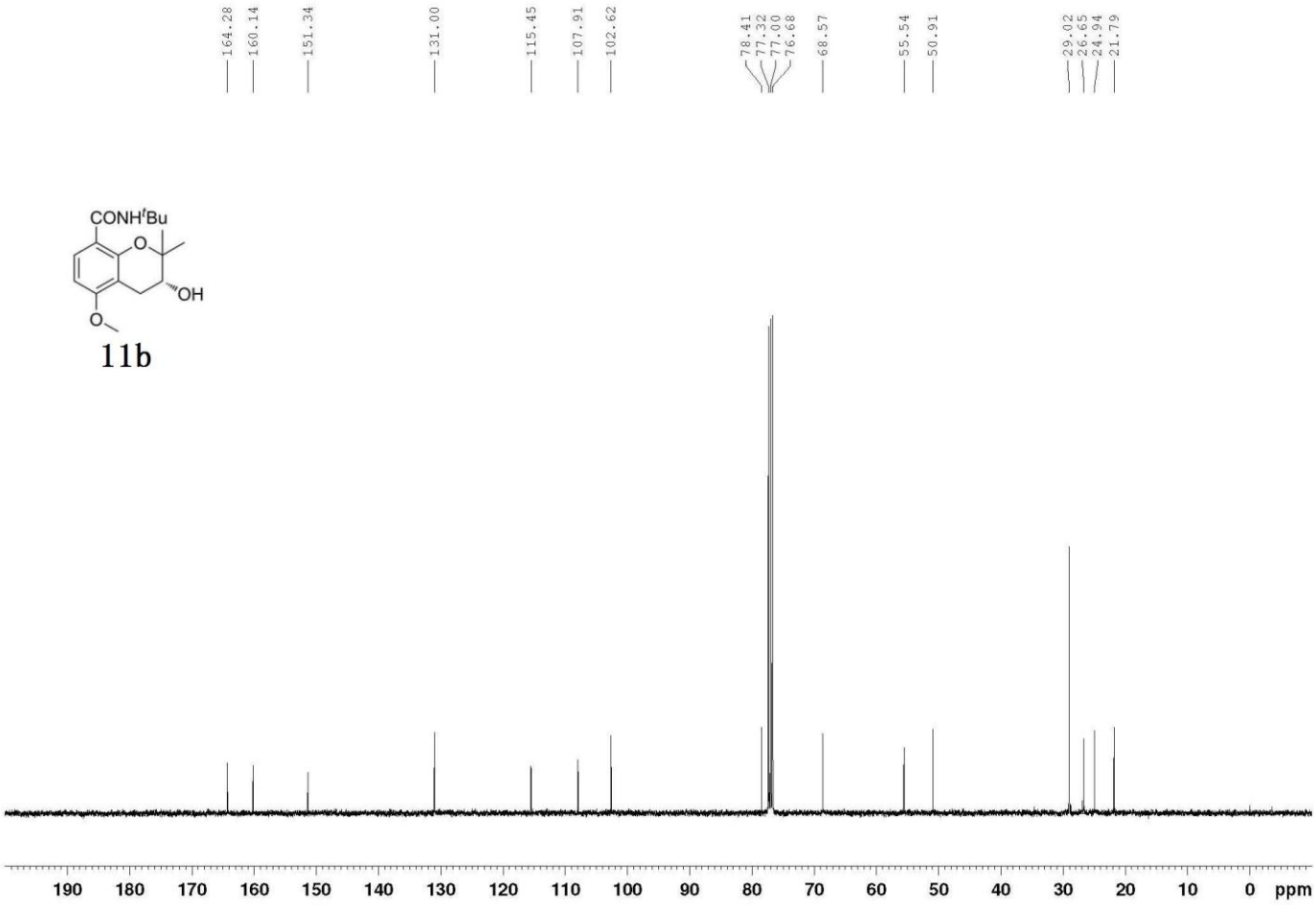
9b



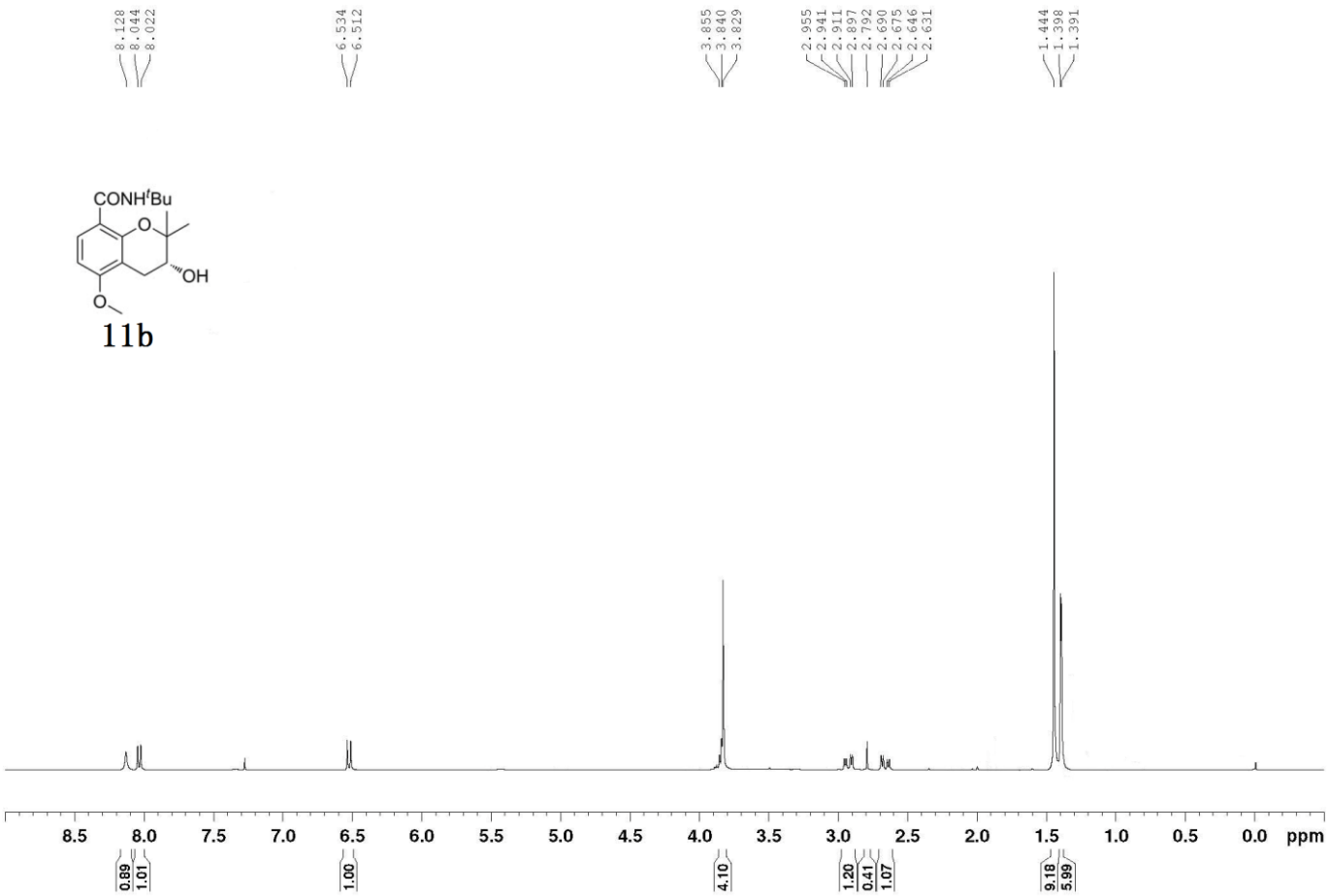


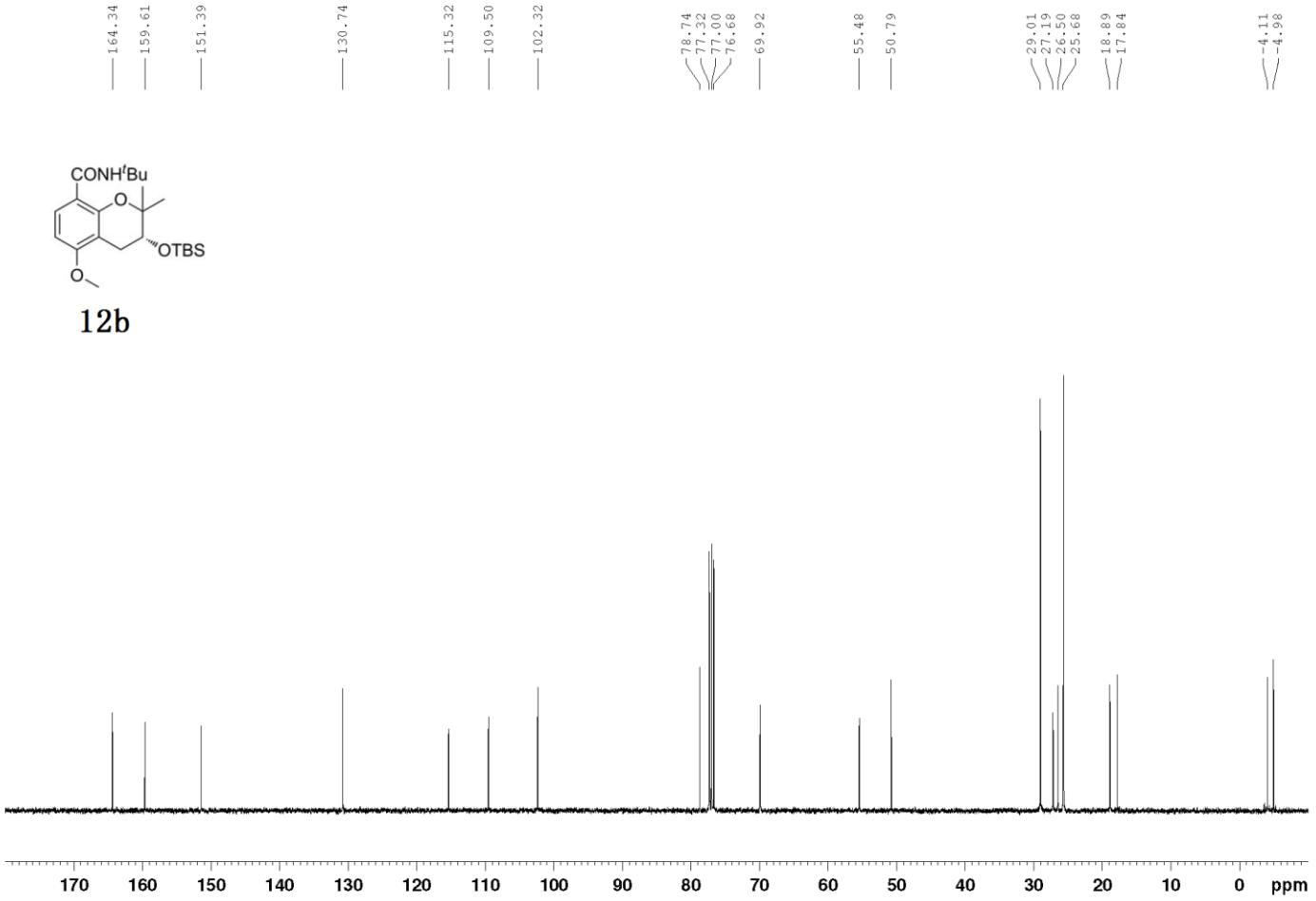
10b



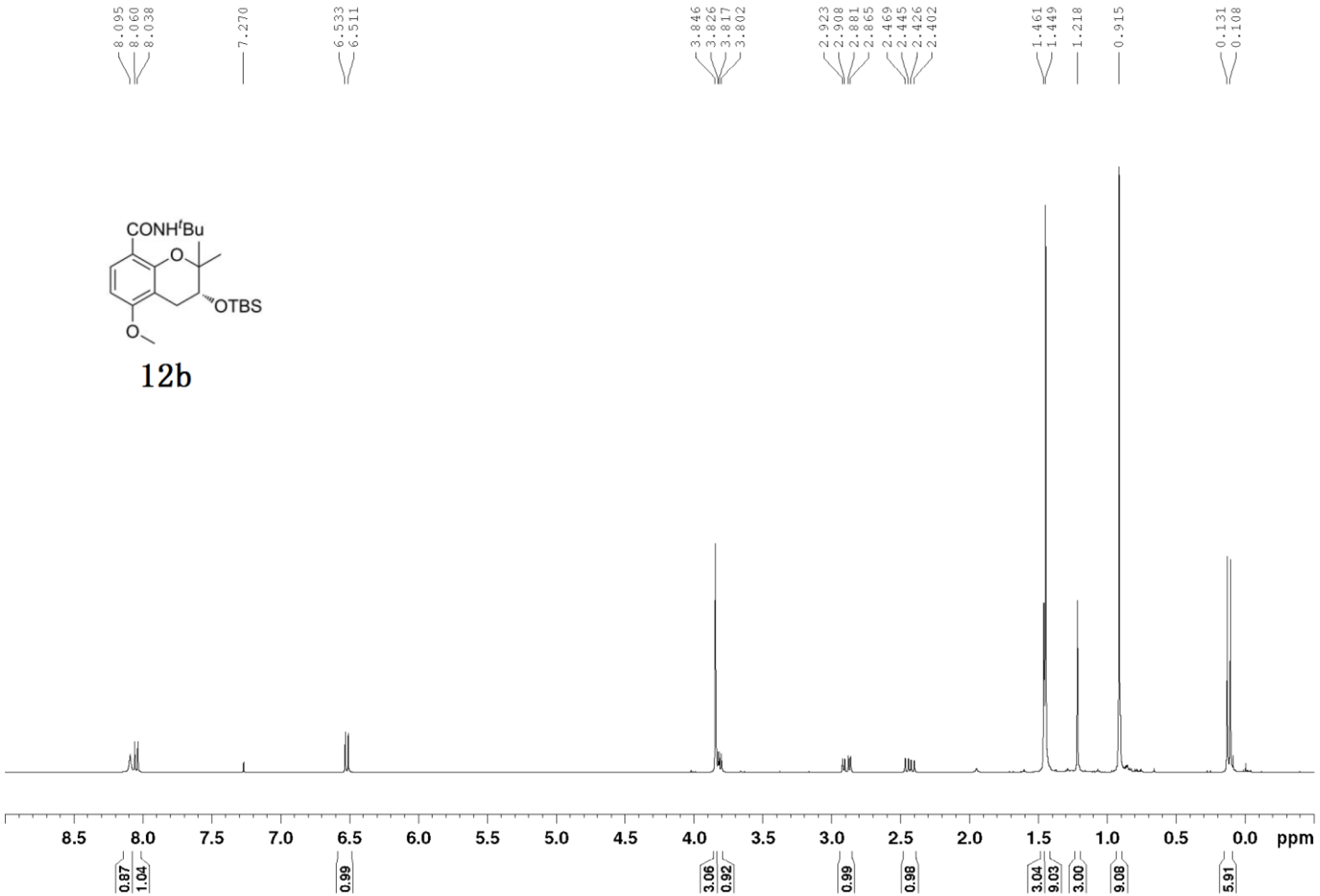


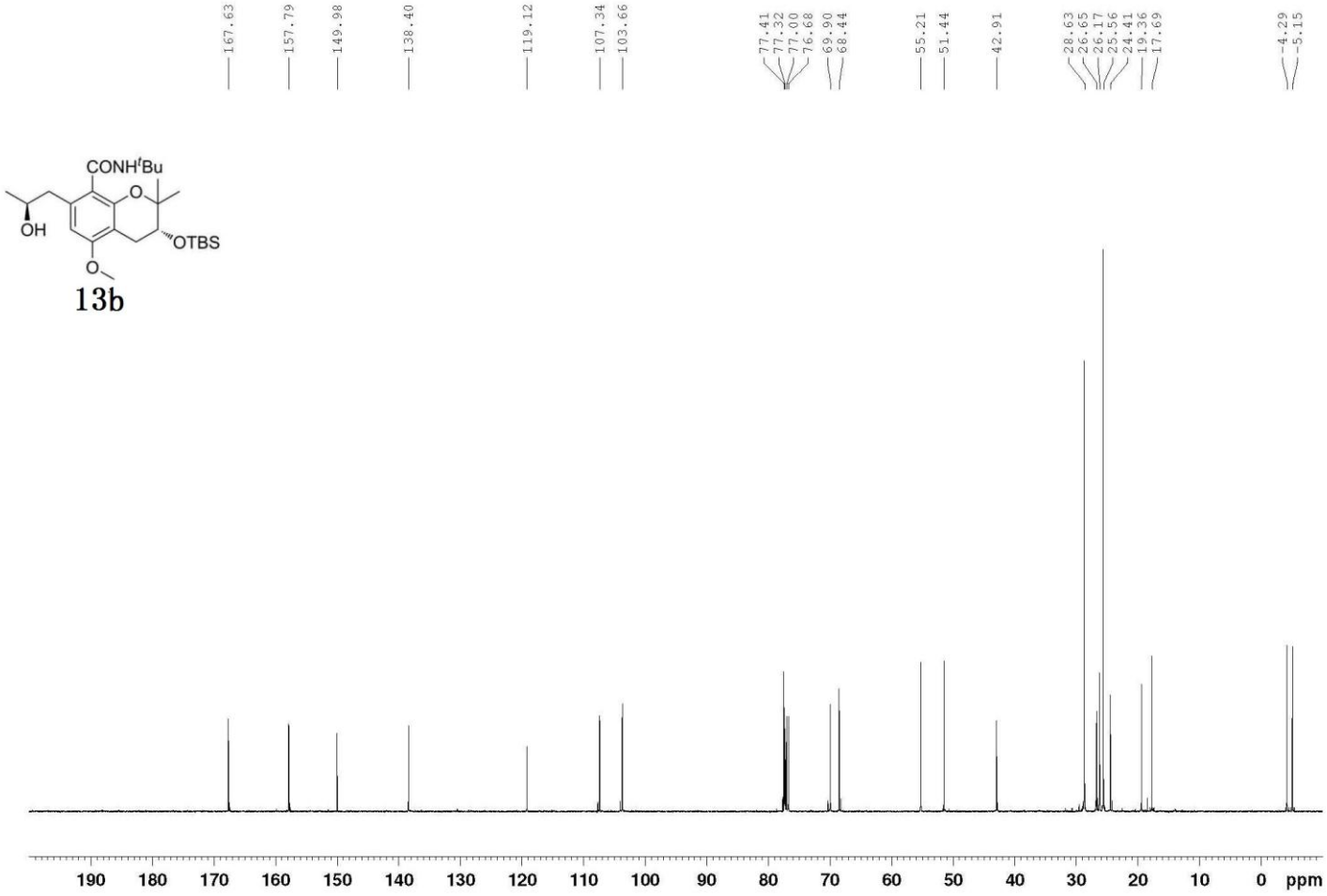
11b

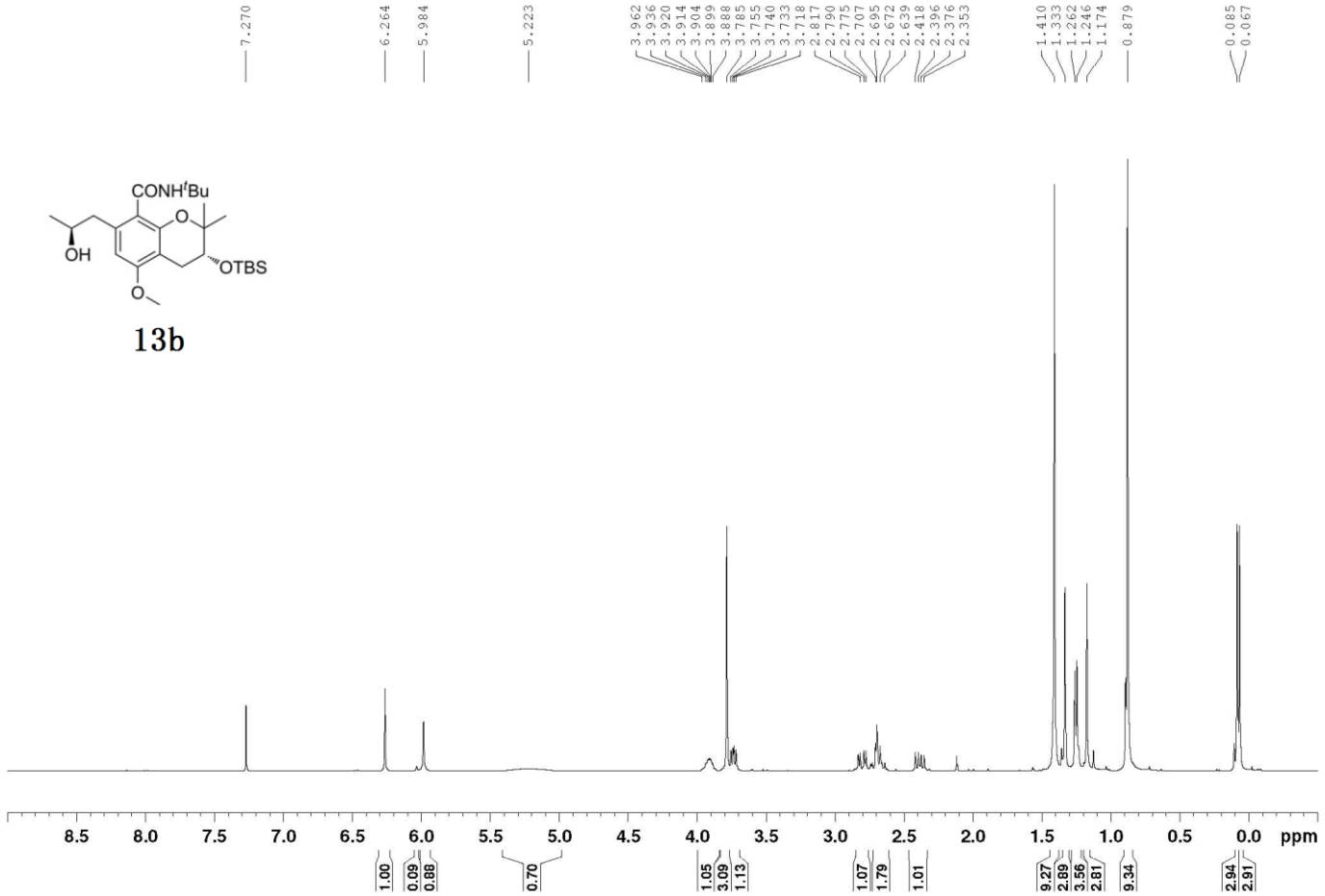


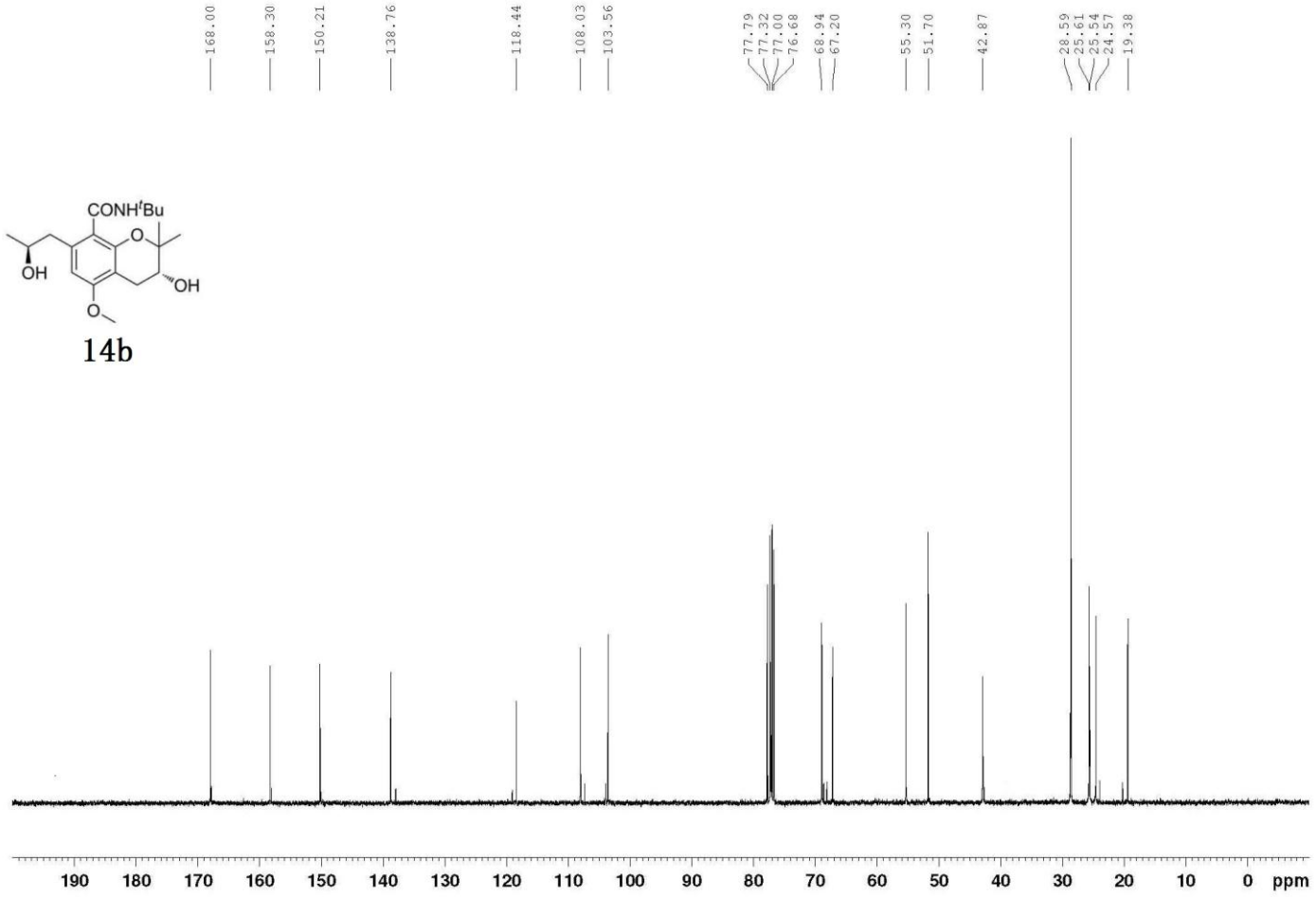


12b

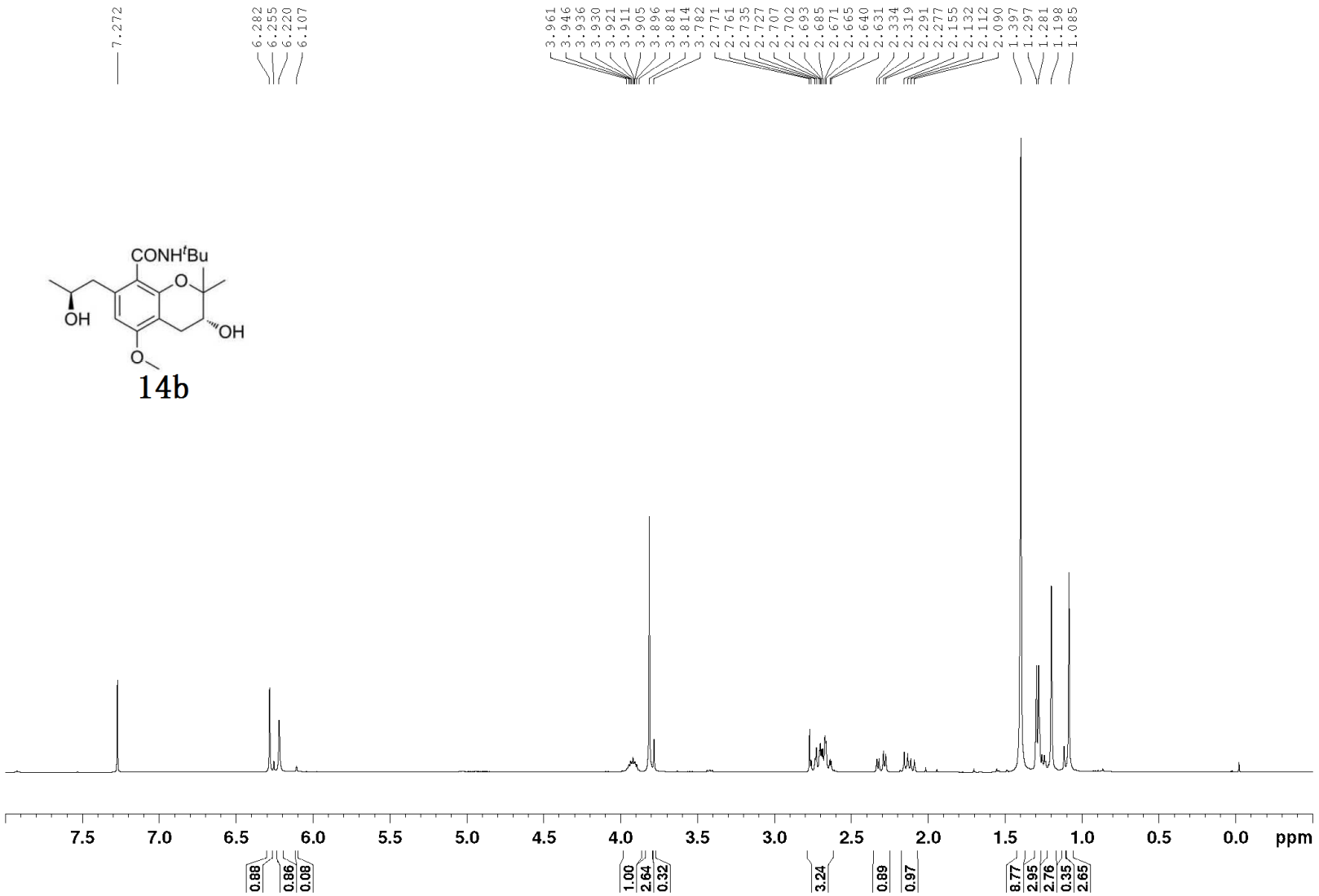


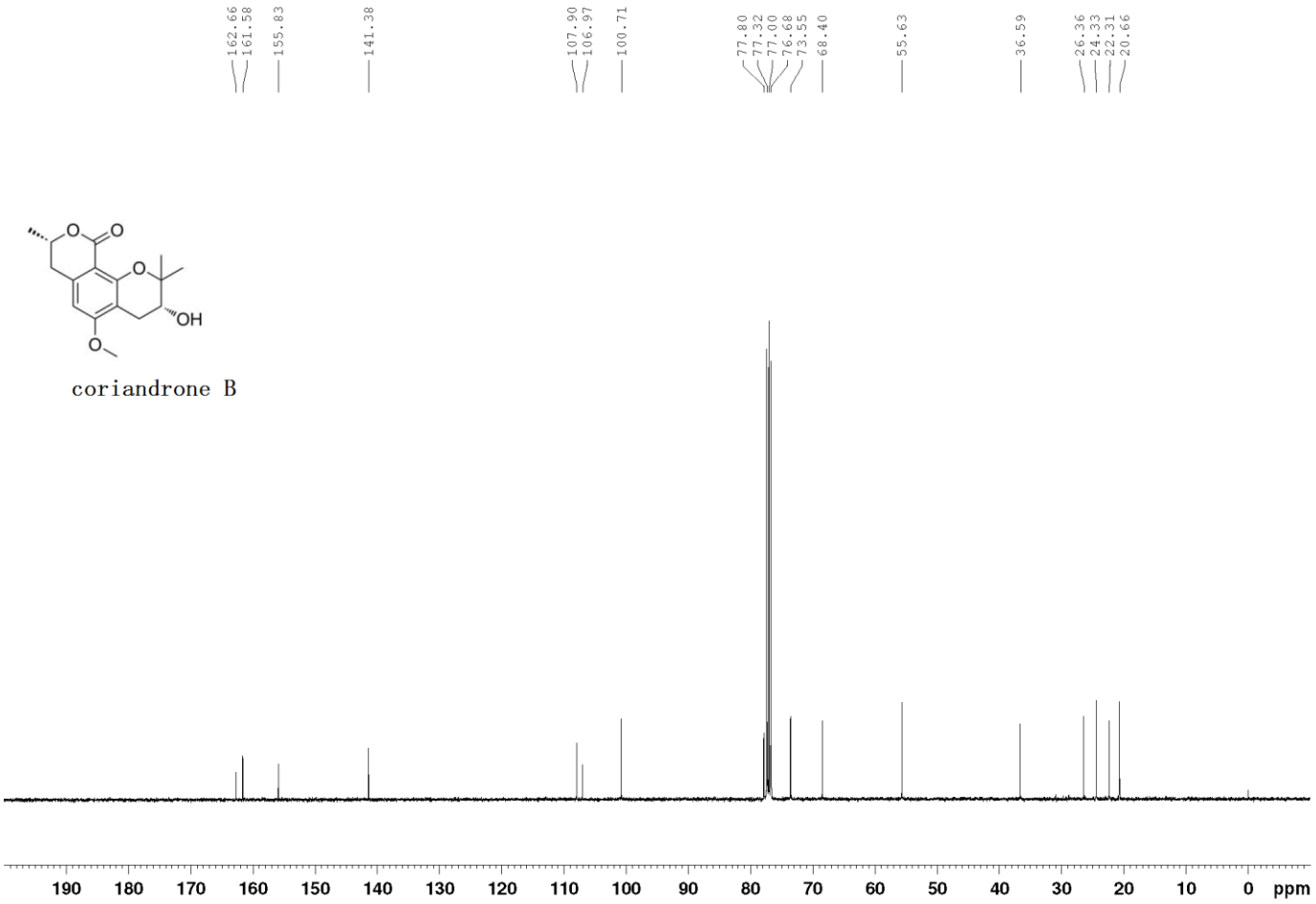




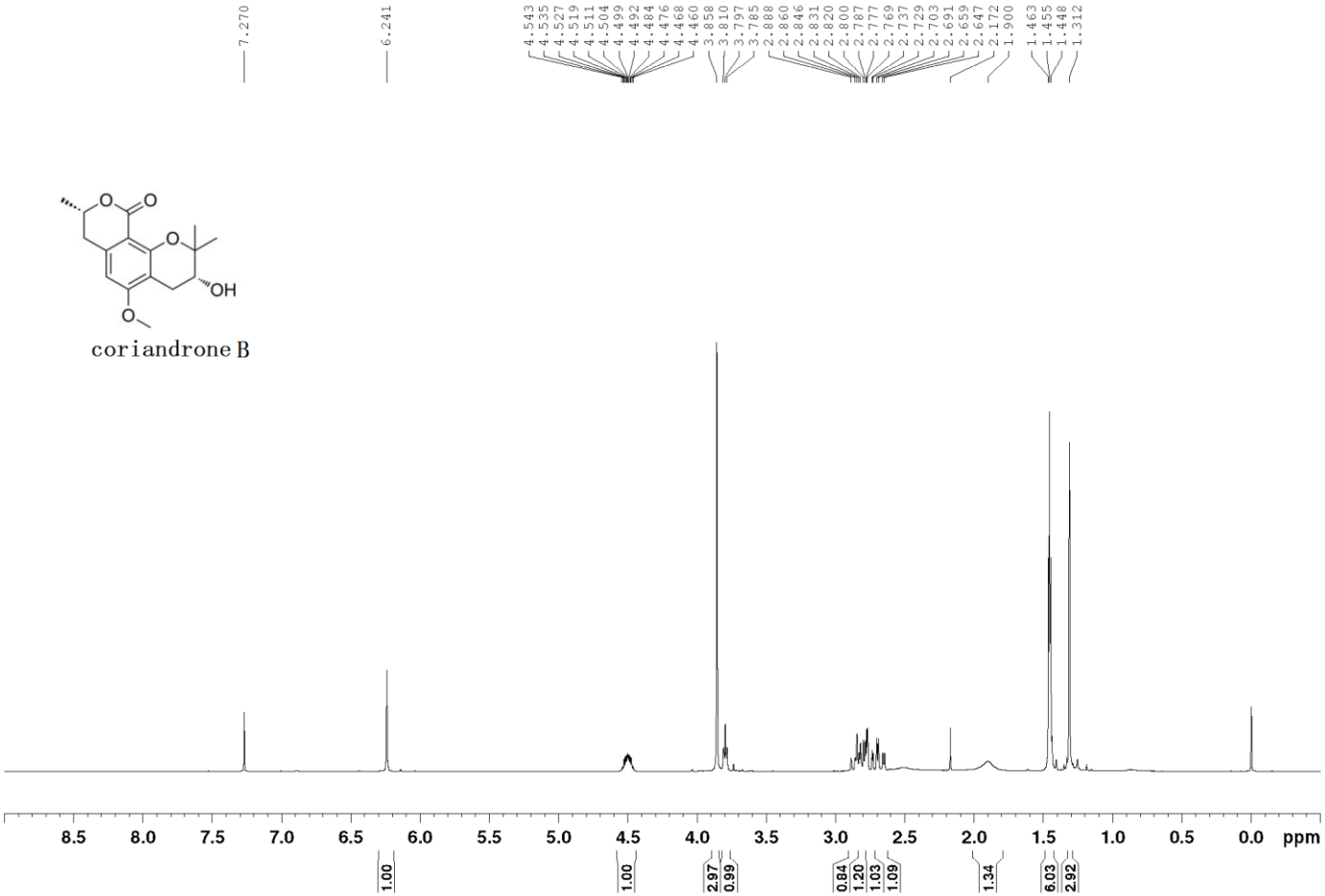


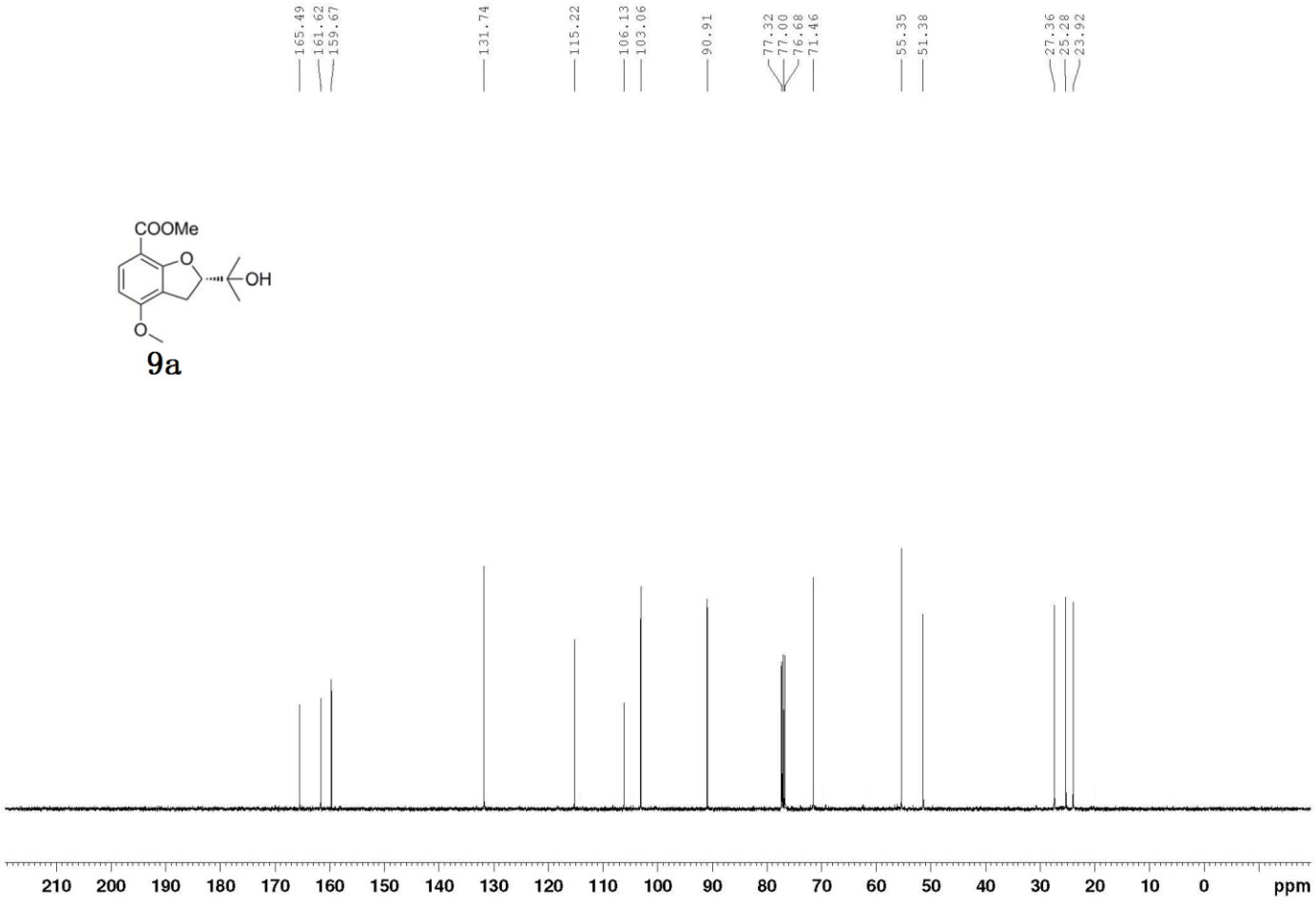
14b



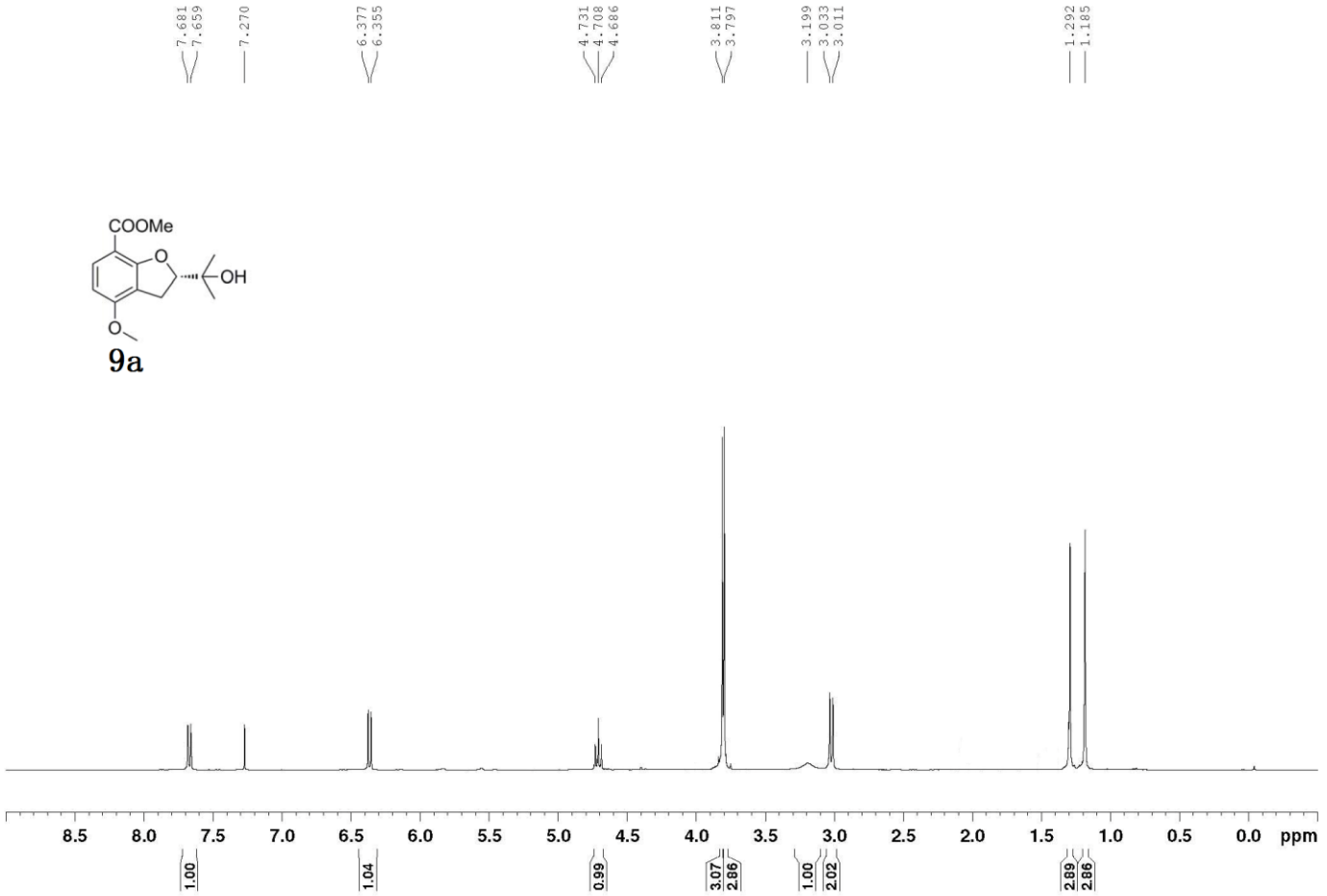


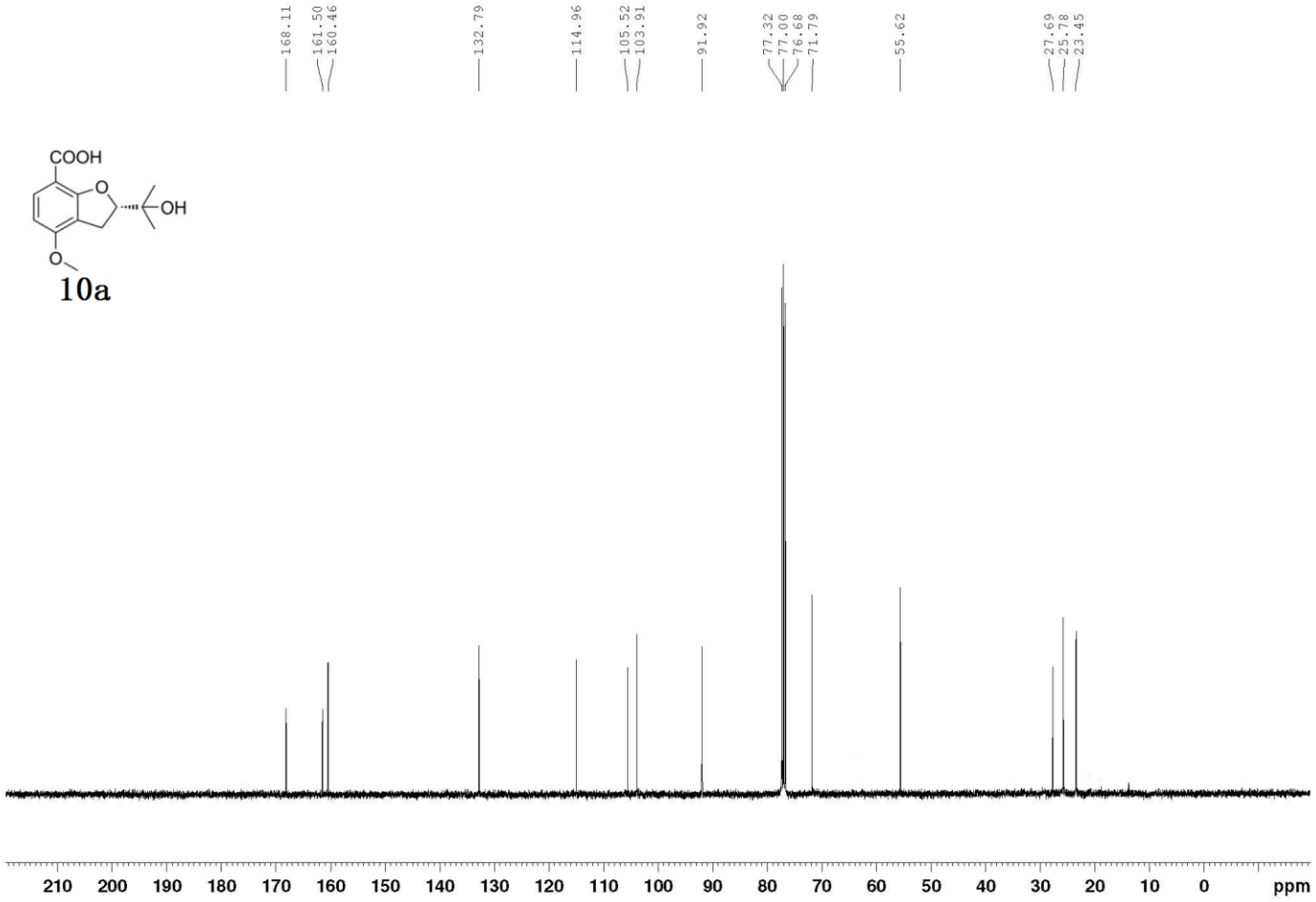
coriandrone B



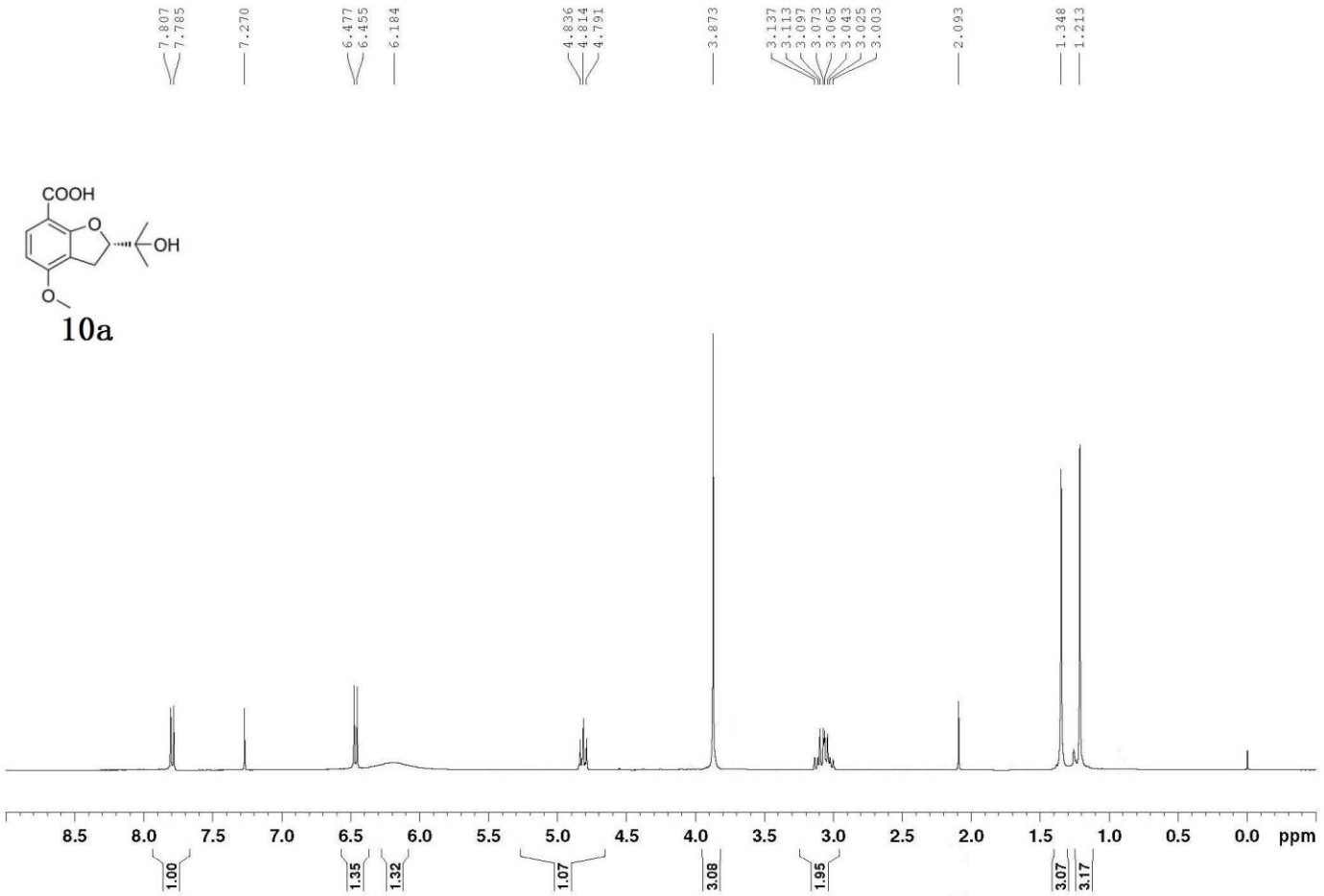


9a

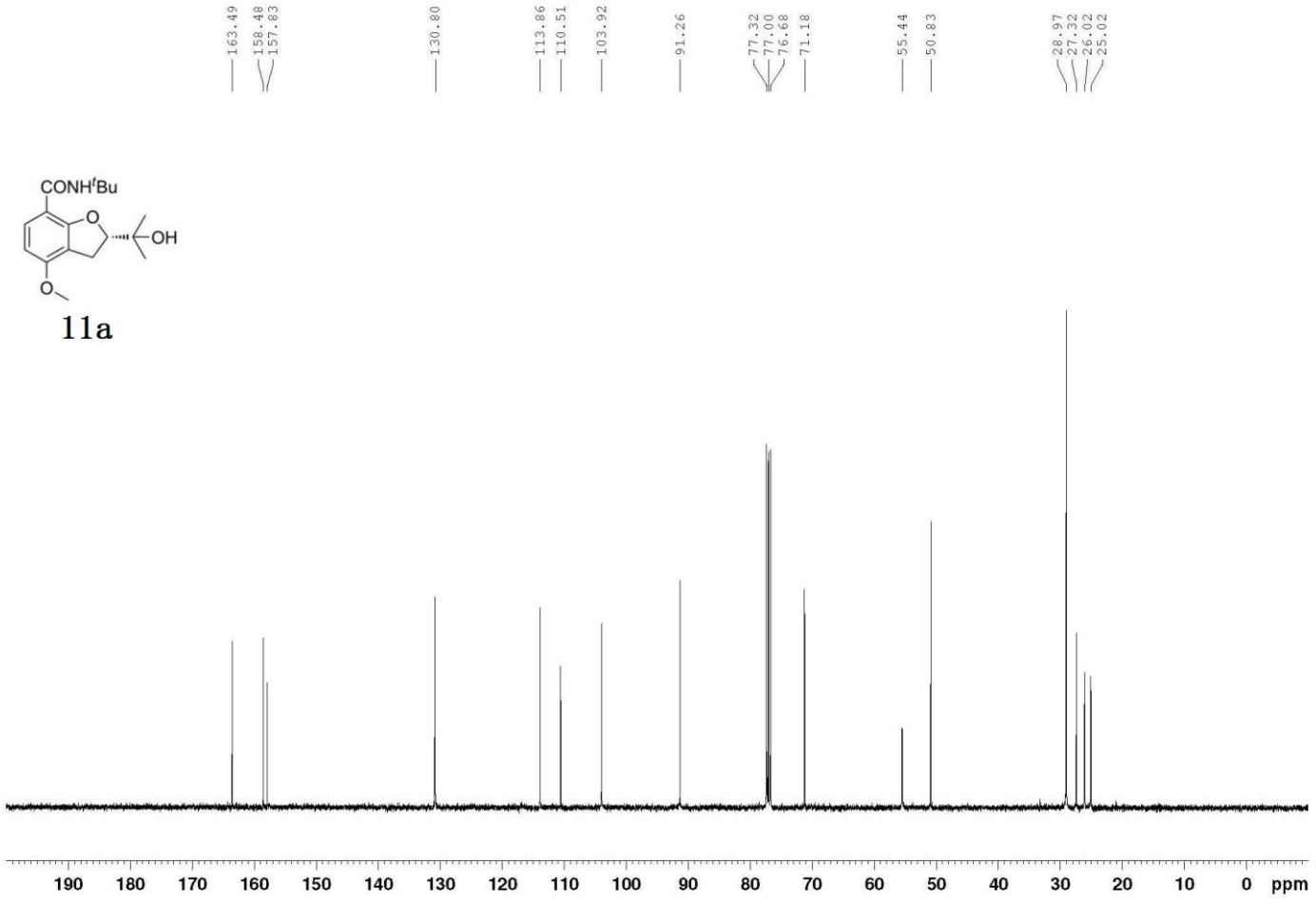




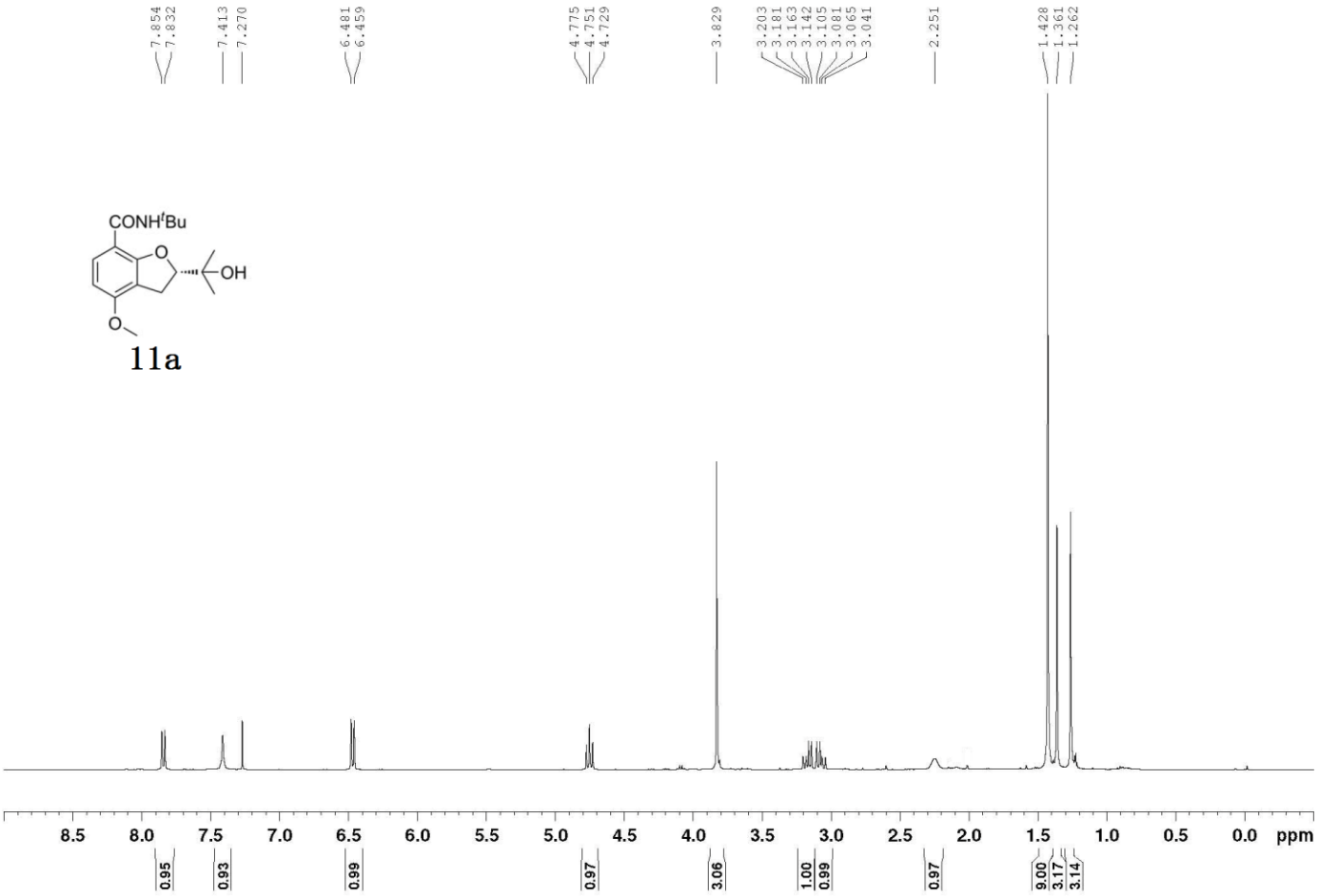
10a



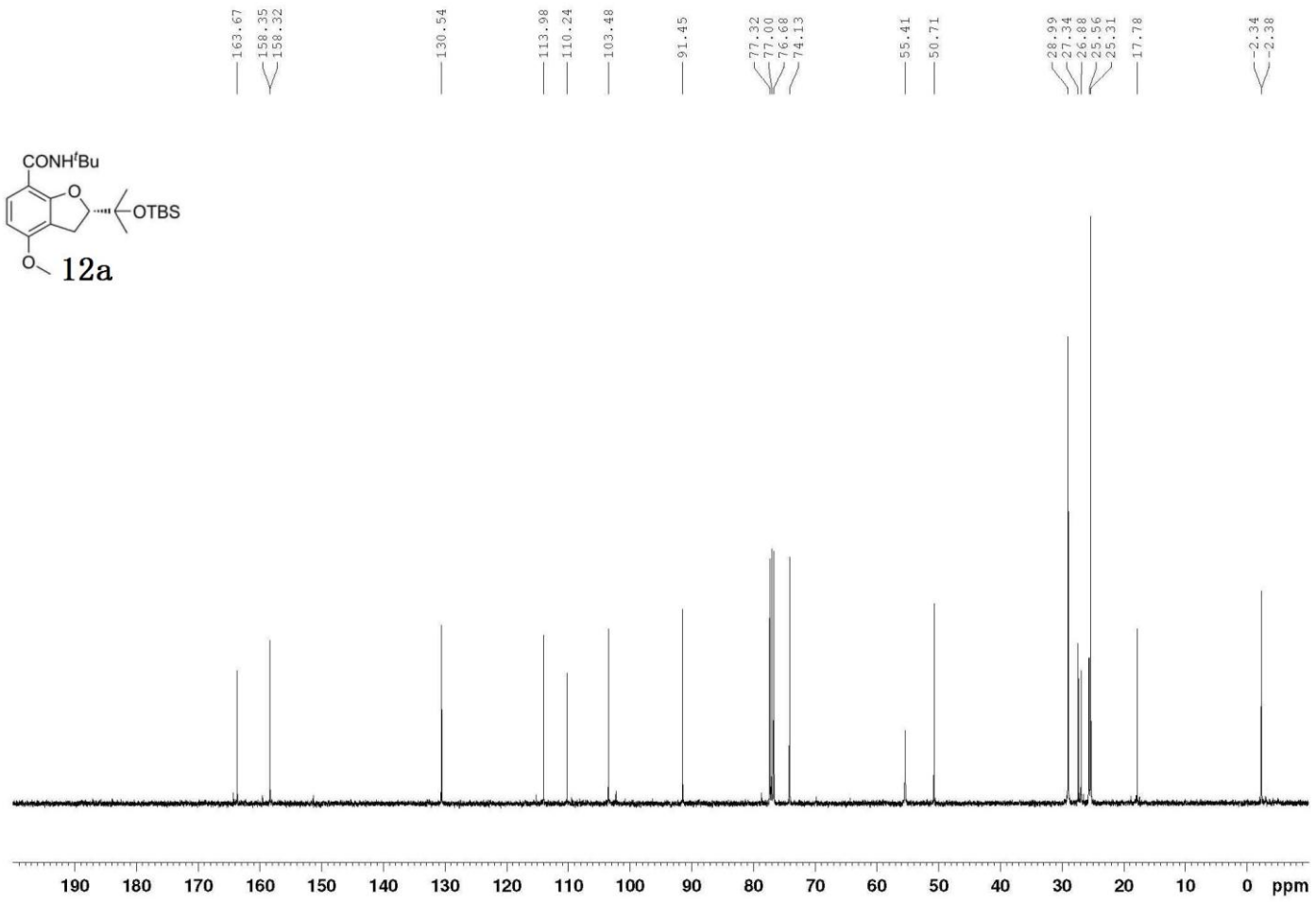
10a

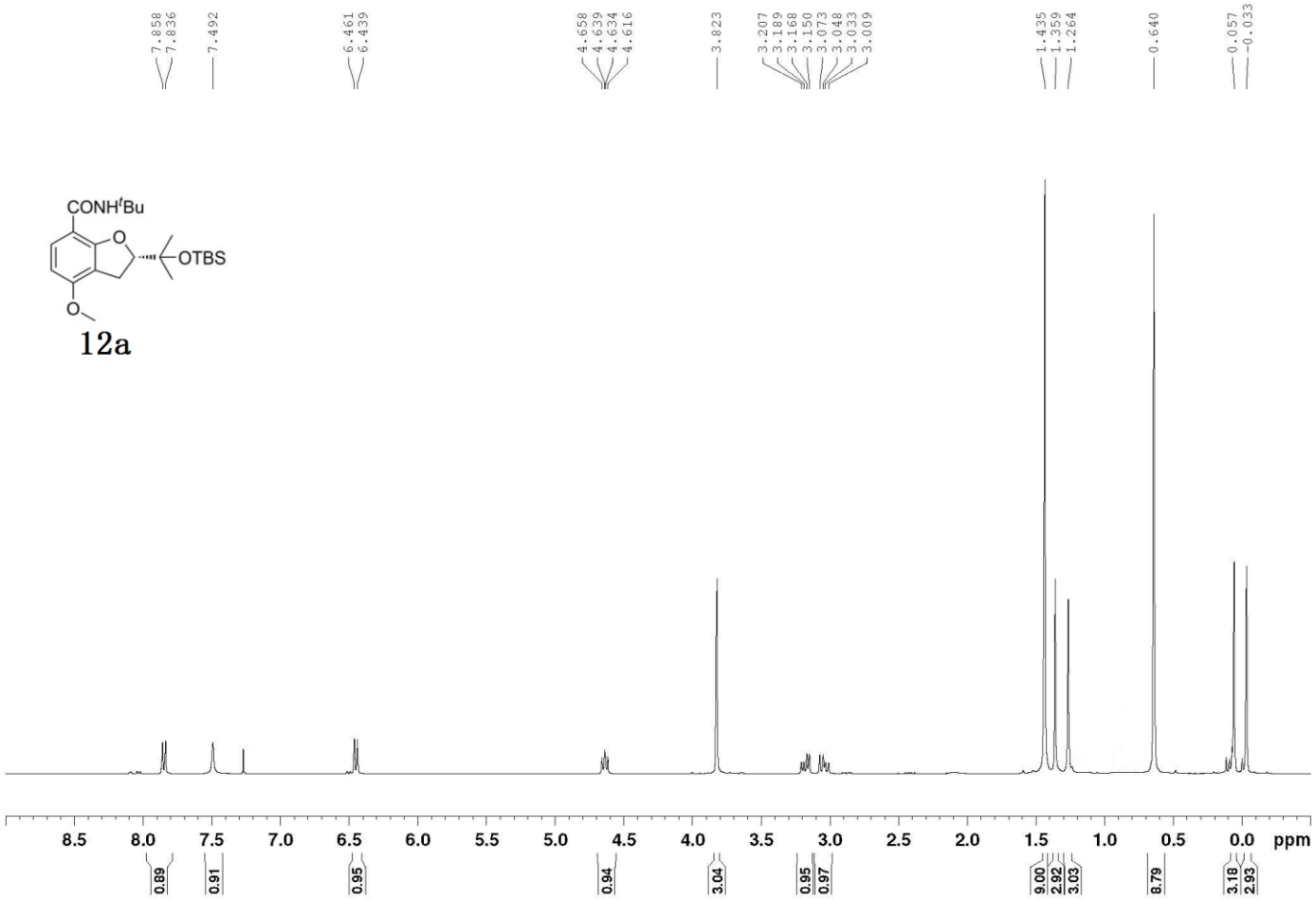


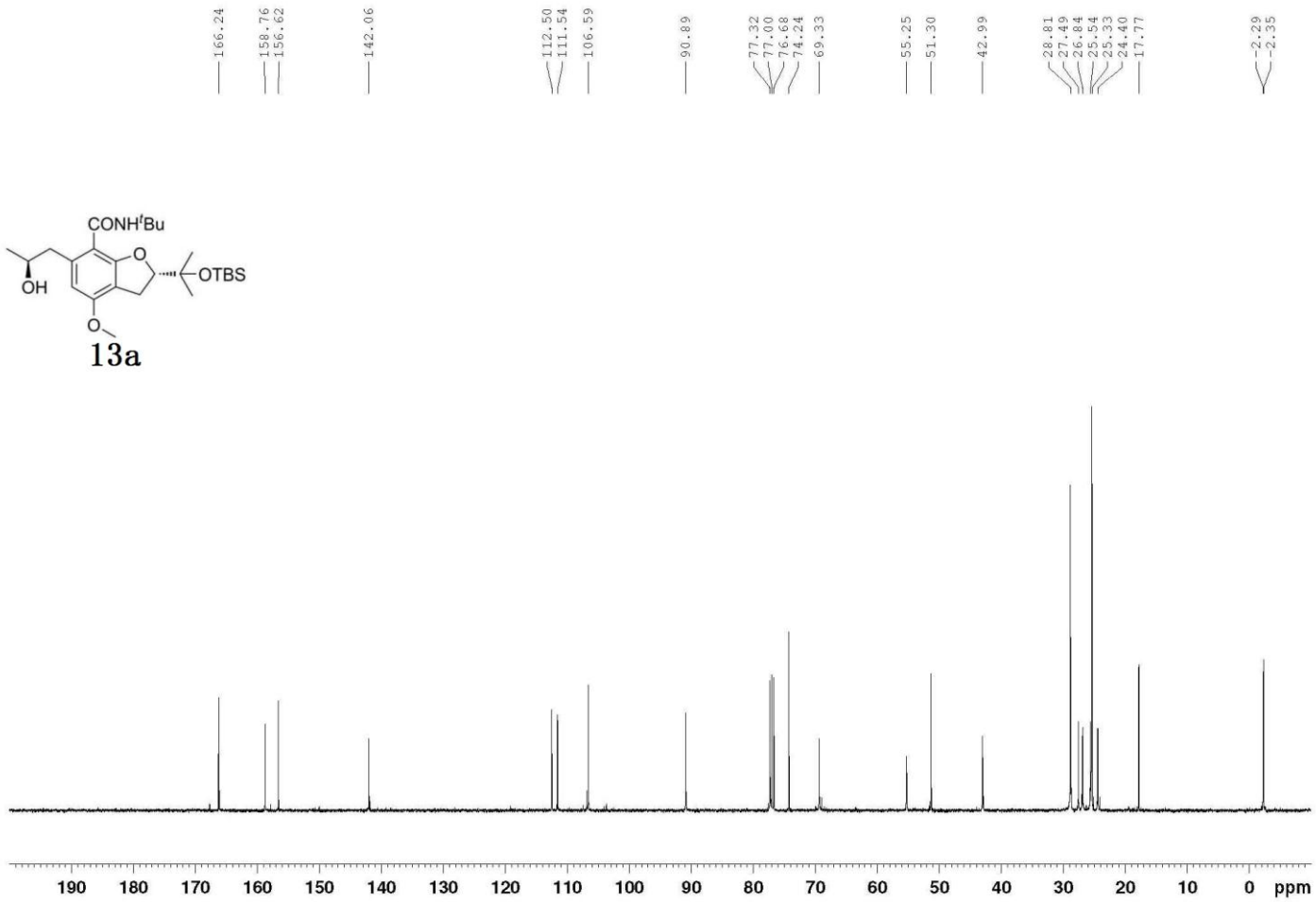
11a

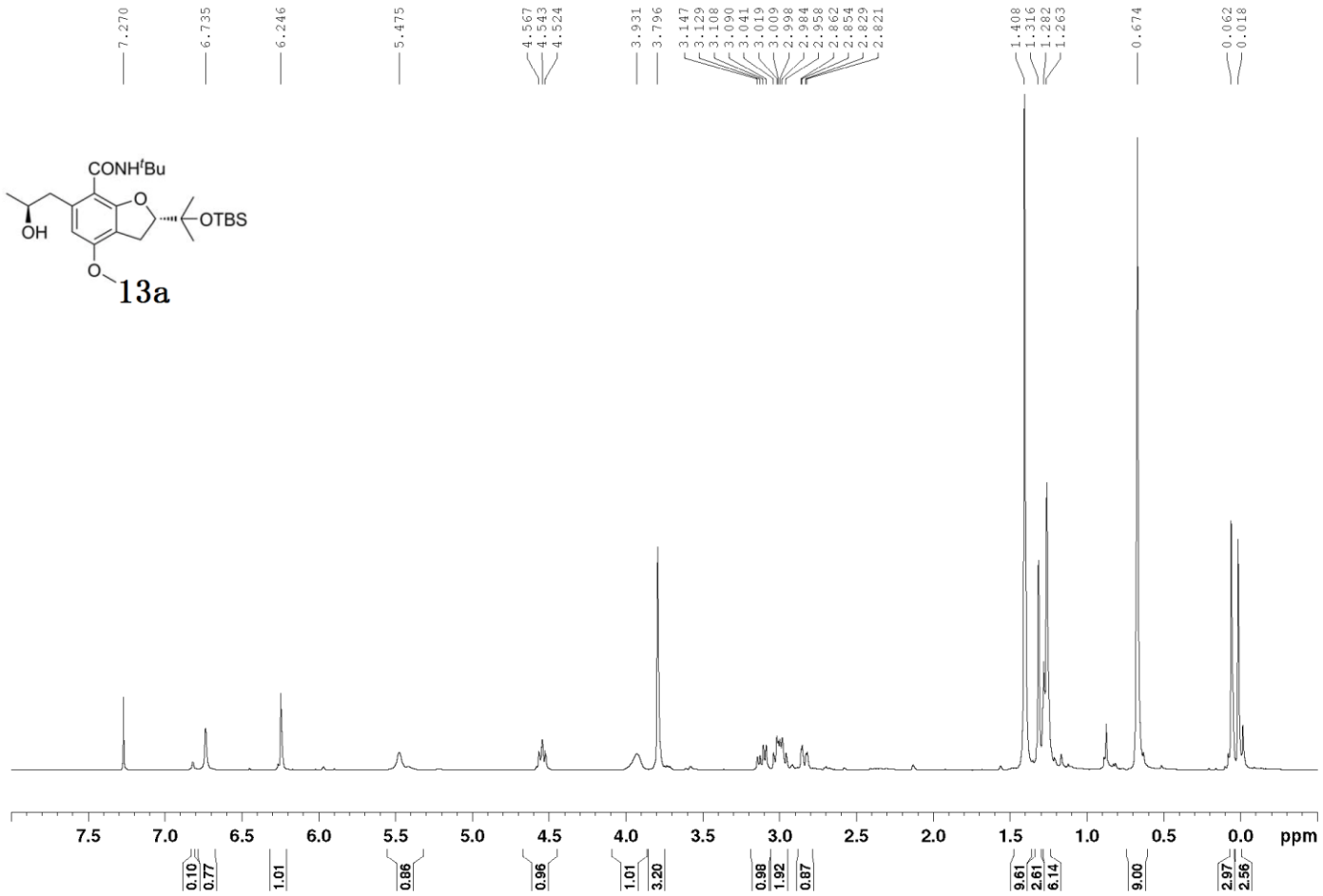


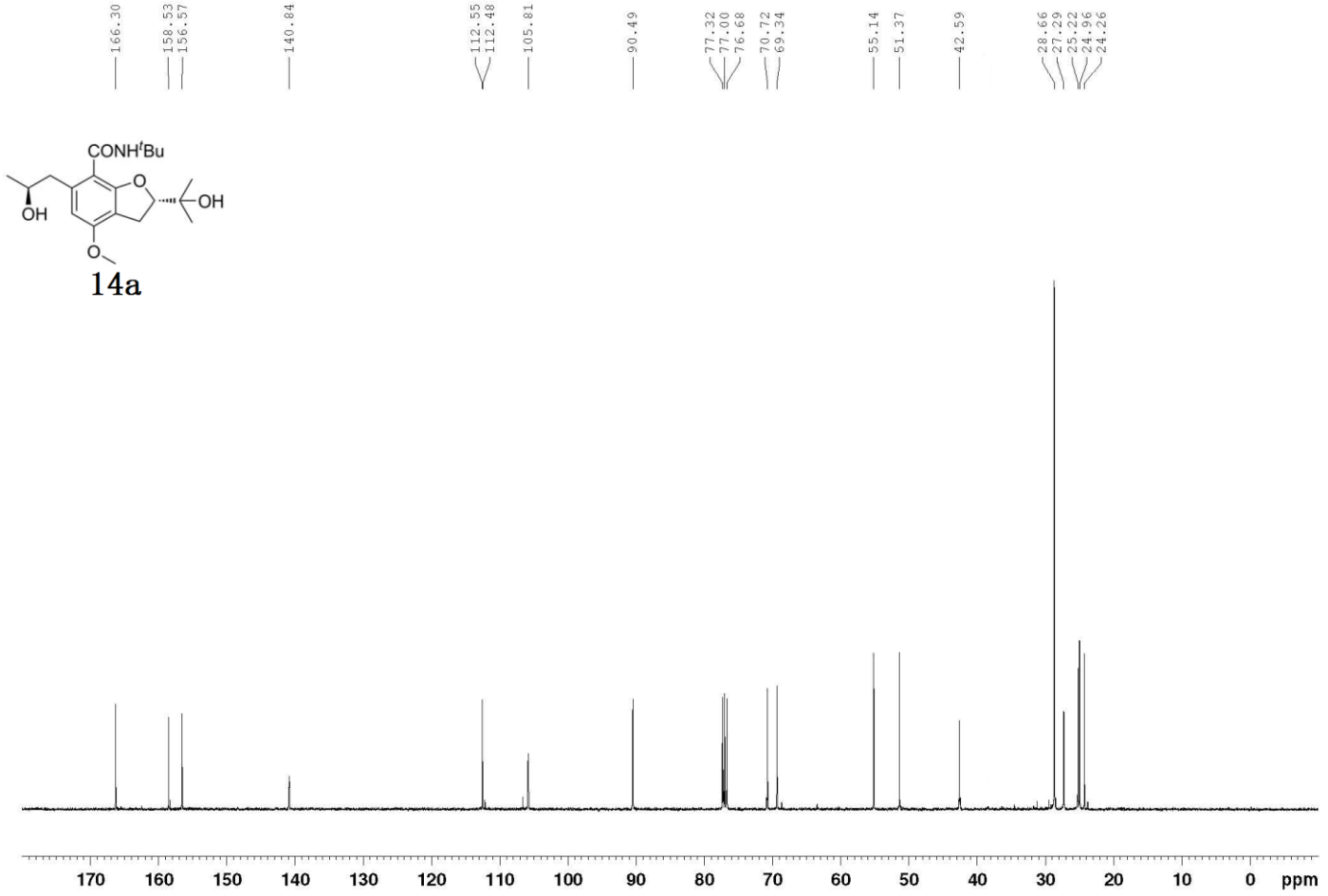
11a



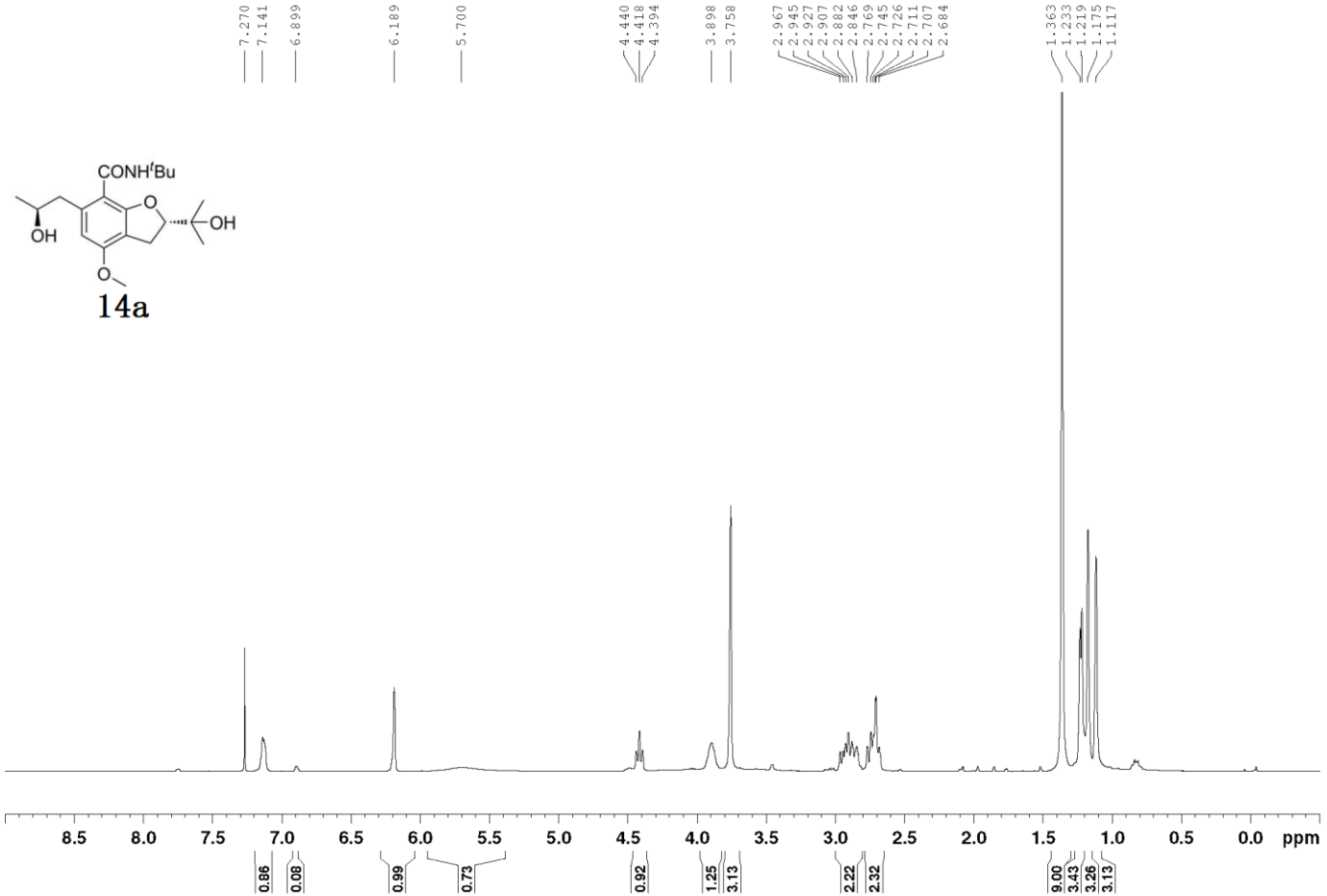


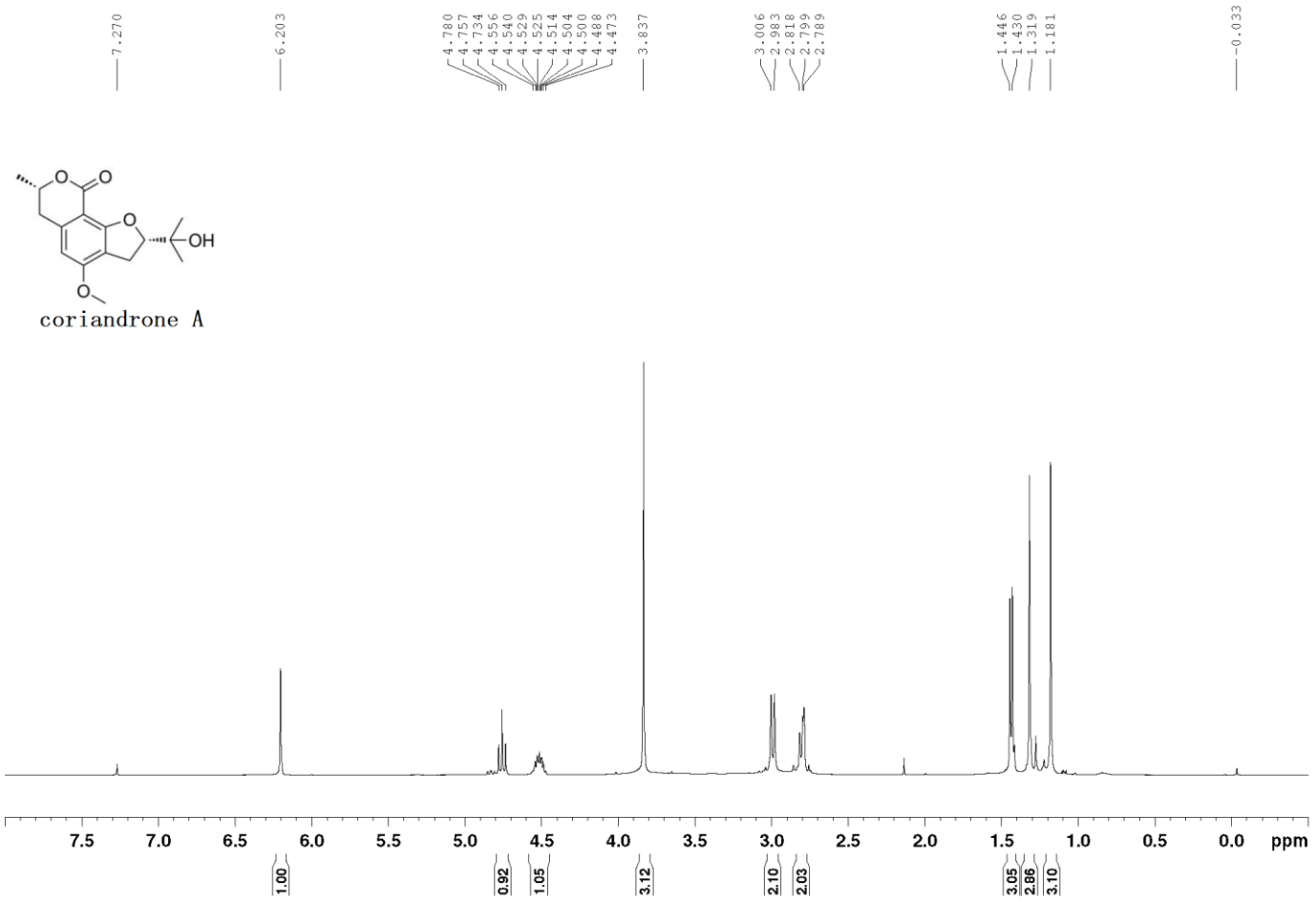

166.24
158.76
156.62
142.06
112.50
111.54
106.59
90.89
77.32
77.00
76.68
74.24
69.33
55.25
51.30
42.99
28.81
27.49
26.84
25.54
25.33
24.40
17.77
-2.29
-2.35
CONH^{t}Bu
OH
OTBS
13a
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

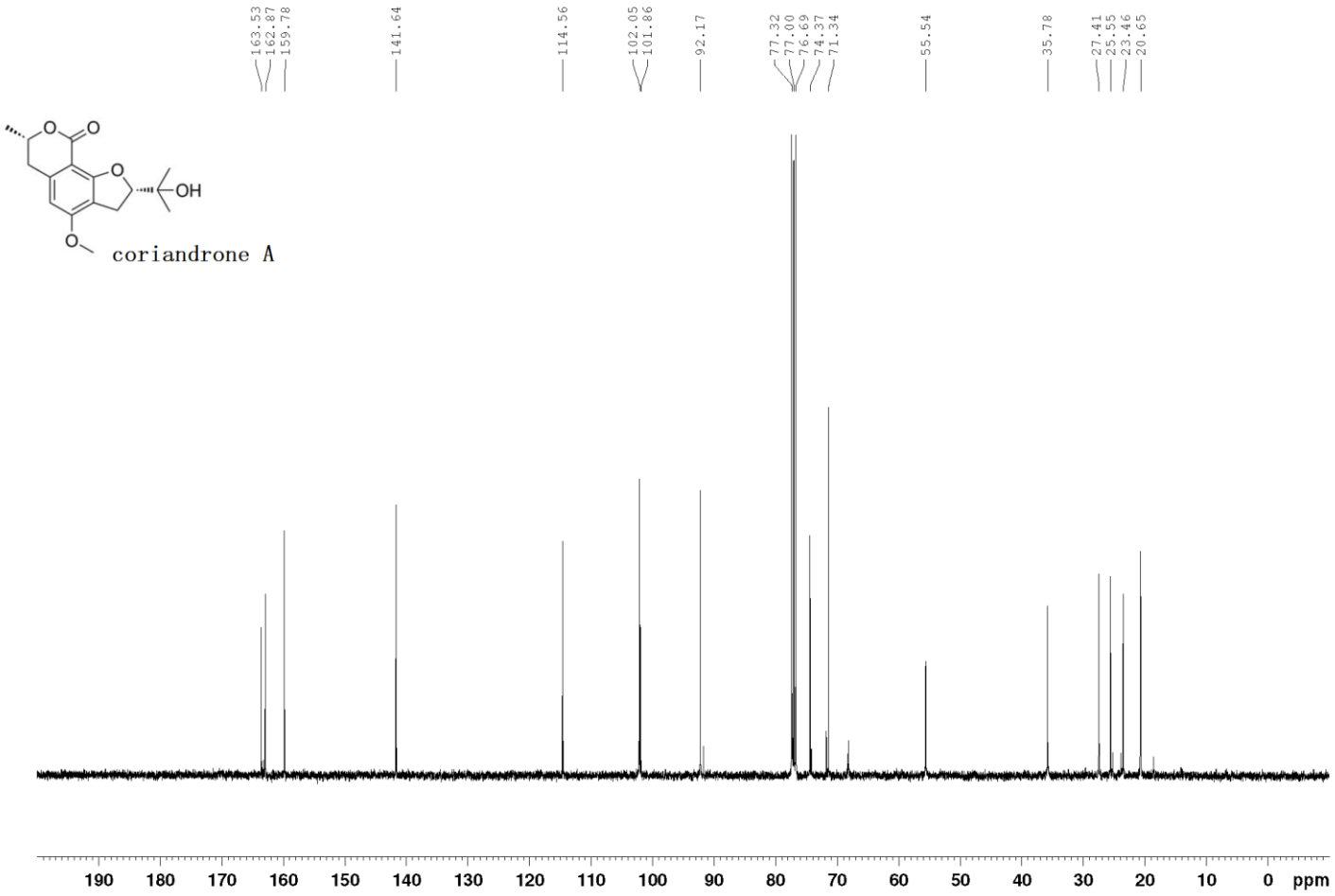




14a





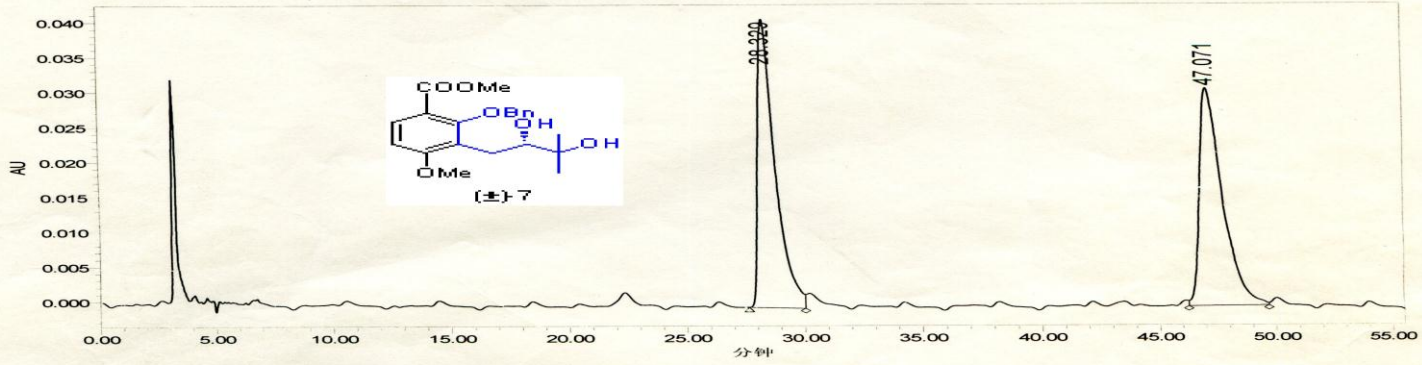


Lanzhou
项目名称: fan
用户名称: FanChunAn

Breeze 2 HPLC System

样品信息

样品名称:	wwj xiaoxuan	采集者:	FanChunAn
样品类型:	未知	样品组名称:	ZGB20100908
瓶号:	2:A,7	采集方法组:	IB3 1ml Hexlpr95v5
进样次数:	1	处理方法:	ZGB05
进样体积:	10.00 ul	通道名称:	236.2 纳米
运行时间:	60.0 Minutes	处理通道注释:	PDA 236.2 纳米
采集时间:	2010/9/8 10:40:37 CST	色谱柱类型:	PDA 236.2 纳米
处理时间:	2010/9/8 11:39:14 CST		



	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	% 高度
1	28.329	2119102	49.94	41250	57.00
2	47.071	2124389	50.06	31125	43.00

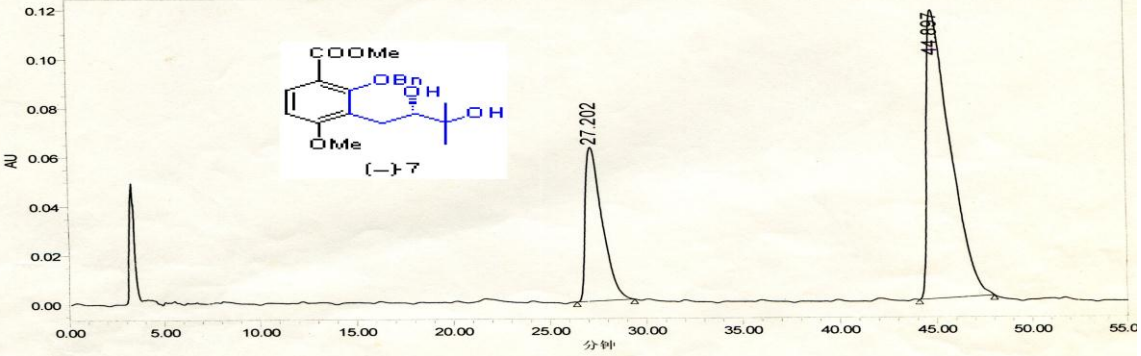
报告方法: 单个报告 ASC 打印于 11:39:53 PM2010/9/8 页码: 1 (共计 1)

Lanzhou
项目名称: fan
用户名称: FanChunAn

Breeze 2 HPLC System

样品信息

样品名称:	wwj shouxing	采集者:	FanChunAn
样品类型:	未知	样品组名称:	ZGB20100908
瓶号:	2:A,8	采集方法组:	IB3 1ml Hexlpr95v5
进样次数:	1	处理方法:	ZGB05
进样体积:	20.00 ul	通道名称:	236.2 纳米
运行时间:	55.0 Minutes	处理通道注释:	PDA 236.2 纳米
采集时间:	2010/9/8 11:38:08 CST	色谱柱类型:	PDA 236.2 纳米
处理时间:	2010/9/8 12:36:05 CST		



	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	% 高度
1	27.202	3800799	27.52	62661	34.79
2	44.897	10008674	72.48	117471	65.21

报告方法: 单个报告 ASC 打印于 12:36:46 PM 2010/9/8 页码: 1 (共计 1)

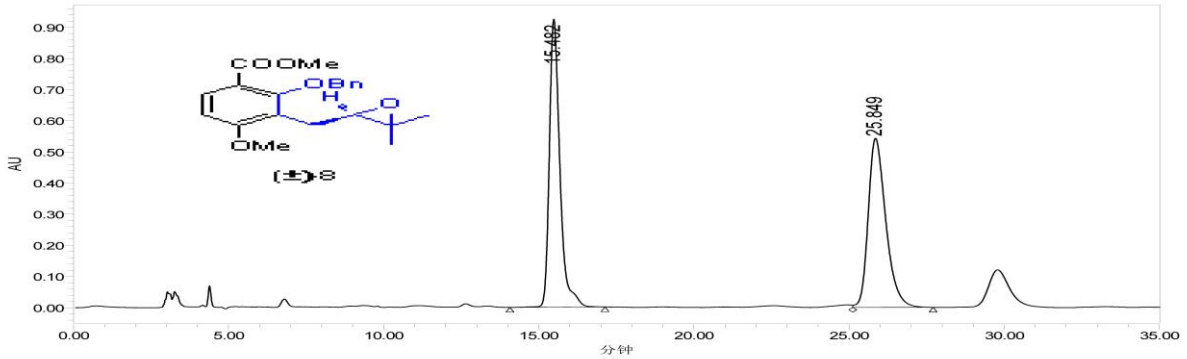
Lanzhou

项目名称: fan
用户名称: FanChunAn

Breeze 2 HPLC System

样品信息

样品名称:	wwj201109131	采集者:	FanChunAn
样品类型:	未知	样品组名称:	wwj
瓶号:	2:E,1	采集方法组:	IC3 1ml Hexlpr90v10
进样次数:	1	处理方法:	chenpeng
进样体积:	15.00 ul	通道名称:	222.1 纳米
运行时间:	35.0 Minutes	处理通道注释:	PDA 222.1 纳米
采集时间:	2011/9/13 15:42:49 CST	色谱柱类型:	PDA 222.1 纳米
处理时间:	2011/9/13 17:44:46 CST		



	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	% 高度
1	15.482	21920300	50.64	924497	63.00
2	25.849	21369376	49.36	542917	37.00

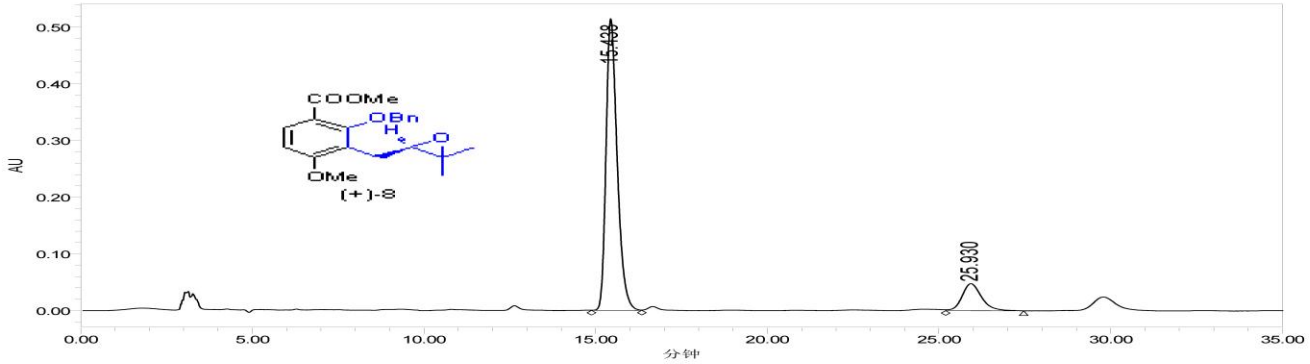
报告方法: 单个报告 ASC 打印于 17:56:23 PM2011/9/13 页码: 1 (共计 1)

Lanzhou
项目名称: fan
用户名称: FanChunAn

Breeze 2 HPLC System

样品信息

样品名称:	wwj201109133	采集者:	FanChunAn
样品类型:	未知	样品组名称:	wwj
瓶号:	2:E,3	采集方法组:	IC3 1ml Hexlpr90v10
进样次数:	1	处理方法:	chenpeng
进样体积:	15.00 ul	通道名称:	230.0 纳米@2
运行时间:	35.0 Minutes	处理通道注释:	PDA 230.0 纳米
采集时间:	2011/9/13 16:53:37 CST	色谱柱类型:	PDA 230.0 纳米
处理时间:	2011/9/13 17:49:03 CST		



	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	% 高度
1	15.438	11619883	86.11	514608	91.53
2	25.930	1874895	13.89	47617	8.47

报告方法: 单个报告 ASC 打印于 17:49:21 PM 2011/9/13 页码: 1 (共计 1)

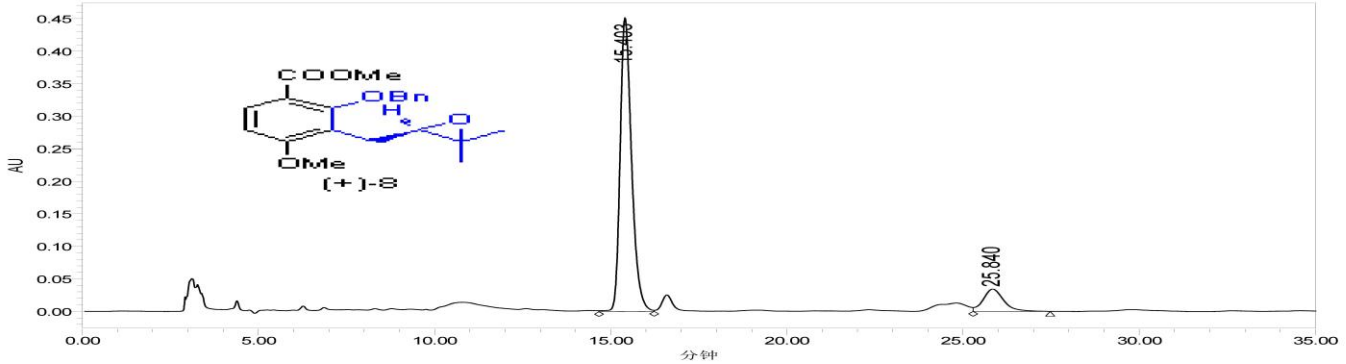
Lanzhou

项目名称: fan
用户名称: FanChunAn

Breeze 2 HPLC System

样品信息

样品名称:	wwj201109132	采集者:	FanChunAn
样品类型:	未知	样品组名称:	wwj
瓶号:	2:E,2	采集方法组:	IC3 1ml Hexlpr90v10
进样次数:	1	处理方法:	chenpeng
进样体积:	15.00 ul	通道名称:	230.0 纳米@2
运行时间:	35.0 Minutes	处理通道注释:	PDA 230.0 纳米
采集时间:	2011/9/13 16:18:13 CST	色谱柱类型:	PDA 230.0 纳米
处理时间:	2011/9/13 17:47:33 CST		



	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	% 高度
1	15.403	10208634	88.04	451693	92.94
2	25.840	1387193	11.96	34296	7.06

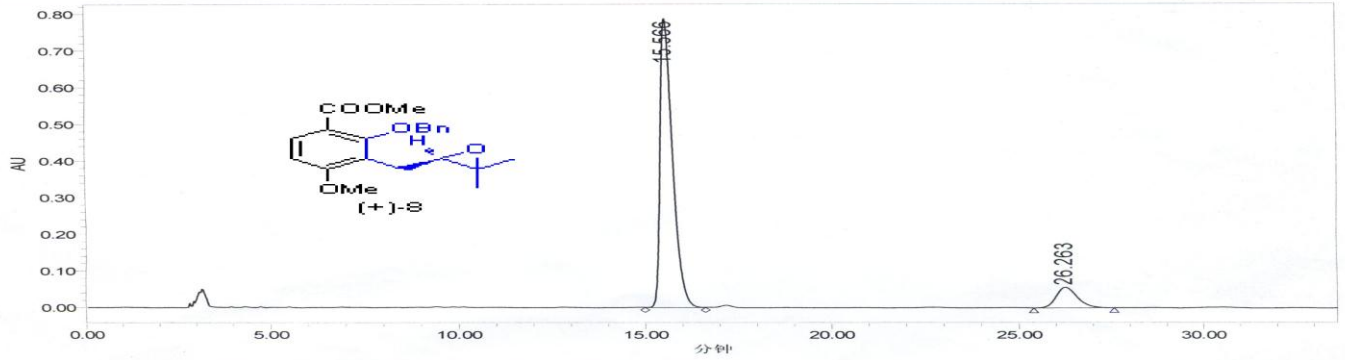
报告方法: 单个报告 ASC 打印于 17:55:49 PM2011/9/13 页码: 1 (共计 1)

Lanzhou
项目名称: fan
用户名称: FanChunAn

Breeze 2 HPLC System

样品信息

样品名称:	wwj20110518	采集者:	FanChunAn
样品类型:	未知	样品组名称:	wwj
瓶号:	1:F,7	采集方法组:	IC3 1ml Hexlpr90v10
进样次数:	1	处理方法:	chenpeng
进样体积:	10.00 ul	通道名称:	230.0 纳米@2
运行时间:	35.0 Minutes	处理通道注释:	PDA 230.0 纳米
采集时间:	2011/5/18 17:23:53 CST	色谱柱类型:	PDA 230.0 纳米
处理时间:	2011/5/18 17:58:18 CST		



	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	% 高度
1	15.566	18352585	89.29	787338	93.36
2	26.263	2200691	10.71	55976	6.64

报告方法: 单个报告 ASC 打印于 17:59:13 PM 2011/5/18 页码: 1 (共计 1)

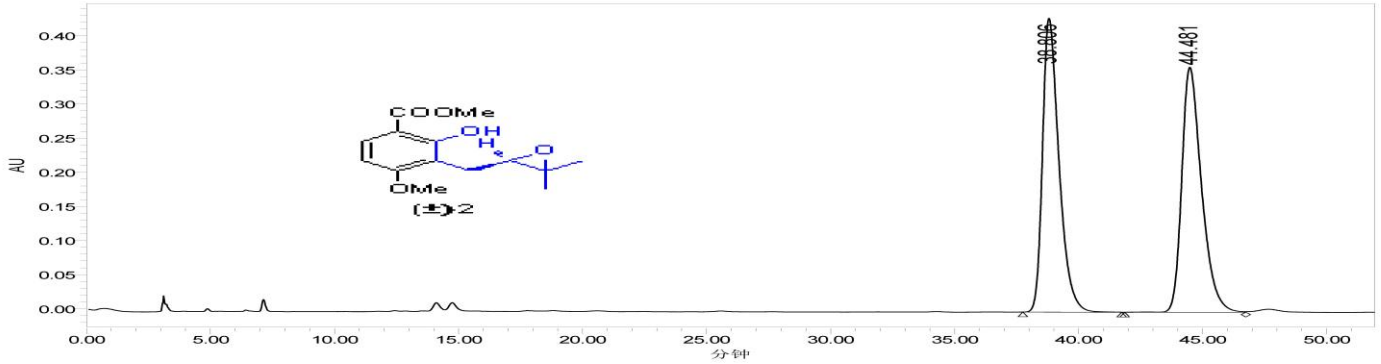
Lanzhou

项目名称: fan
用户名称: FanChunAn

Breeze 2 HPLC System

样品信息

样品名称:	wwj201109061	采集者:	FanChunAn
样品类型:	未知	样品组名称:	wwj
瓶号:	2:D,1	采集方法组:	IC3 1ml Hexlpr97v3
进样次数:	1	处理方法:	wwj
进样体积:	15.00 ul	通道名称:	260.0 纳米
运行时间:	60.0 Minutes	处理通道注释:	PDA 260.0 纳米
采集时间:	2011/9/6 17:26:38 CST	色谱柱类型:	PDA 260.0 纳米
处理时间:	2011/9/6 19:19:06 CST		



	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	% 高度
1	38.806	19855064	49.98	430299	54.54
2	44.481	19873422	50.02	358677	45.46

报告方法: 单个报告 ASC 打印于 17:57:10 PM2011/9/13 页码: 1 (共计 1)

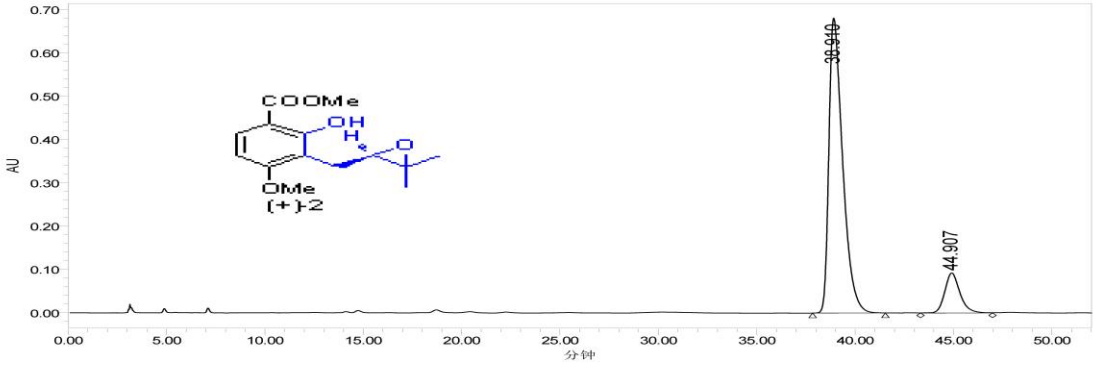
Lanzhou

项目名称: fan
用户名称: FanChunAn

Breeze 2 HPLC System

样品信息

样品名称:	wwj201109062	采集者:	FanChunAn
样品类型:	未知	样品组名称:	wwj
瓶号:	2:D,2	采集方法组:	IC3 1ml Hexlpr97v3
进样次数:	1	处理方法:	wwj
进样体积:	15.00 ul	通道名称:	260.0 纳米
运行时间:	52.0 Minutes	处理通道注释:	PDA 260.0 纳米
采集时间:	2011/9/6 18:19:59 CST	色谱柱类型:	PDA 260.0 纳米
处理时间:	2011/9/6 19:21:10 CST		



	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	% 高度
1	38.910	33016039	87.08	680720	88.07
2	44.907	4897484	12.92	92184	11.93

报告方法: 单个报告 ASC 打印于 17:57:39 P2011/9/13 页码: 1 (共计 1)

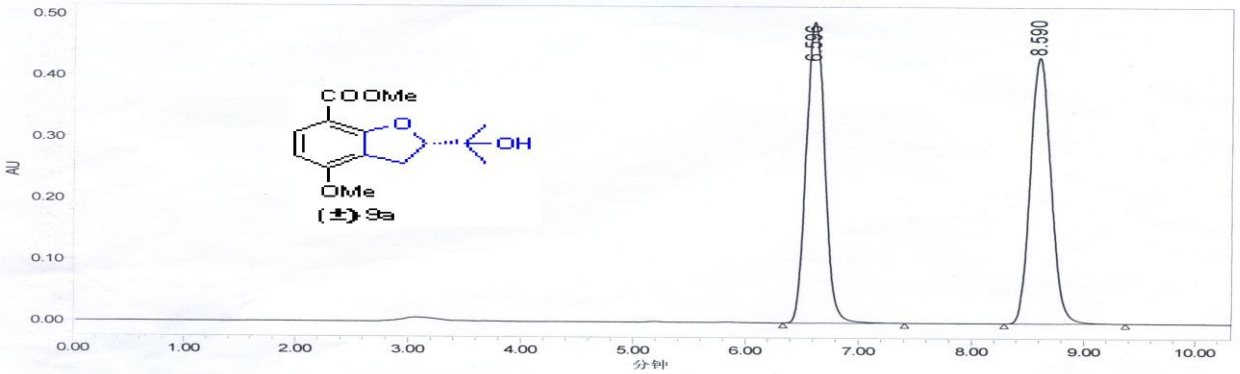
Lanzhou

项目名称: fan
用户名称: FanChunAn

Breeze 2 HPLC System

样品信息

样品名称:	zgb2011.11.19-1	采集者:	FanChunAn
样品类型:	未知	样品组名称:	zgb
瓶号:	1:A,1	采集方法组:	IA3 1ml Hexvlpr85v15
进样次数:	1	处理方法:	zgb
进样体积:	15.00 ul	通道名称:	256.0 纳米
运行时间:	60.0 Minutes	处理通道注释:	PDA 256.0 纳米
采集时间:	2011/11/19 16:24:27 CST	色谱柱类型:	PDA 256.0 纳米
处理时间:	2011/11/19 16:37:04 CST		



	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	% 高度
1	6.596	5622241	50.10	489950	53.10
2	8.590	5600420	49.90	432798	46.90

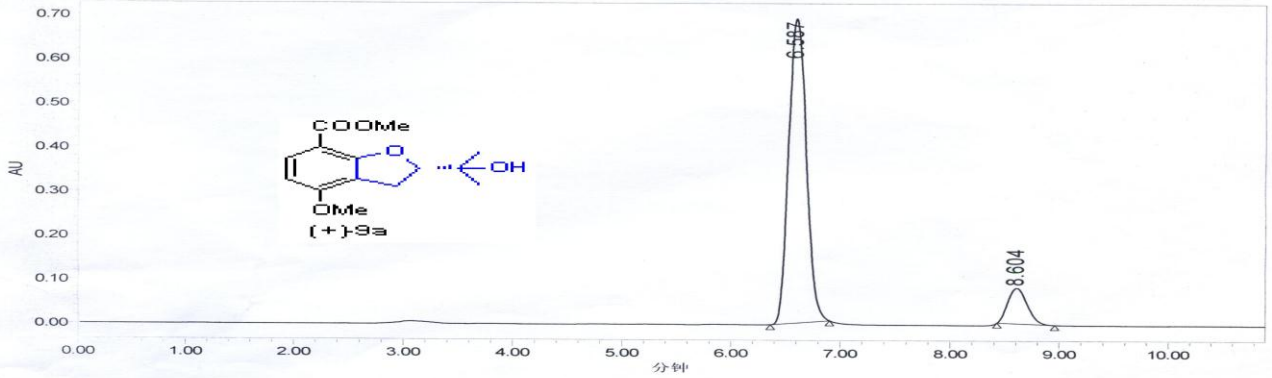
报告方法: 单个报告 ASC 打印于 16:37:48 PM2011/11/19 页码: 1 (共计 1)

Lanzhou
项目名称: fan
用户名称: FanChunAn

Breeze 2 HPLC System

样品信息

样品名称:	zgb2011.11.19-2	采集者:	FanChunAn
样品类型:	未知	样品组名称:	zgb
瓶号:	1:A,2	采集方法组:	IA3 1ml Hexvlpr85v15
进样次数:	1	处理方法:	zgb
进样体积:	15.00 ul	通道名称:	256.0 纳米
运行时间:	13.0 Minutes	处理通道注释:	PDA 256.0 纳米
采集时间:	2011/11/19 16:35:34 CST	色谱柱类型:	PDA 256.0 纳米
处理时间:	2011/11/19 16:47:07 CST		



	保留时间 (分钟)	面积 (微伏*秒)	% 面积	高度 (微伏)	% 高度
1	6.587	7772003	88.69	689354	89.51
2	8.604	990658	11.31	80756	10.49

报告方法: 单个报告 ASC 打印于 16:47:51 PM2011/11/19 页码: 1 (共计 1)