**Title:** Synthesis and Absolute Configuration of (+)-Pseudodeflectusin: Structural Revision of Aspergione B  
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**Ref. No.:** O200600702
**Experimental**

**General:** All nonaqueous reactions were carried out with the use of freshly distilled solvents and under an argon atmosphere. THF was distilled from sodium/benzophenone. Dichloromethane was distilled from P₂O₅. N,N-Dimethylformamide and propylene oxide were distilled from calcium hydride. All other reagents were obtained from commercial sources and used without further purification. Analytical thin layer chromatography was performed with Silica Gel 60 F₂₅₄ plates (Merck, Germany). Flash chromatography was carried out with PSQ 100B (Fuji Silysia Co., Ltd., Japan) silica gel. ¹H- and ¹³C NMR spectra were recorded with a Bruker 600 MHz spectrometer (Avance DRX-600), a Bruker 400 MHz spectrometer (Avance DRX-400) or a JEOL 400 MHz spectrometer (JNM-LD400). Chemical shifts are expressed in δ (ppm) relative to Me₄Si or the residual solvent resonance, and coupling constants (J) are expressed in Hz. Melting points were determined with a Yanaco MP-3S instrument and are uncorrected. Optical rotations were recorded as CHCl₃ or MeOH solutions with a JASCO P-1030 digital polarimeter. Infrared (IR) spectra were recorded with a Jasco FTIR-410 spectrometer as neat samples (oil) on NaCl plates or as KBr pellets (solid). Mass spectra (MS) were obtained with an Applied Biosystems mass spectrometer (API QSTAR pulsar i) at high resolution. HPLC were recorded with a GL Science DG660, GL Science PU614, GL Science CO630, GL Science UV660, and Hitachi D-2500 Chromato-Integrator.
Figure S1. $^1$H-NMR data of natural 1 and synthetic (+)-1.

Natural 1

Synthetic (–)-1.
**Figure S2.** The chiral HPLC analysis of synthetic (+)-1, (±)-1 and natural pseudodeflectusin.
**Table S1. Crystal data and measurement conditions of (+)-1.**

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(2R)-N-tert-Butyl-2-(2-hydroxypropyl)-6-methoxybenzamide, (−)-4

\[
\text{CH}_3\text{O} \quad \text{N} \quad \text{t-Bu} \quad \text{OH} \\
\text{(-)-4}
\]

To a solution of 3¹ (7.17 g, 34.6 mmol) in dry THF (104.0 mL) was added TMEDA (11.3 mL, 76.1 mmol) at -78 °C. To the resulting mixture was added dropwise a solution n-BuLi in hexane (2.67M, 28.5 mL, 76.1 mmol) at -78 °C. After 2hrs, to the resulting mixture was added dropwose (R)-(+) propylene oxide (2.9 mL, 41.5 mmol) at -78 °C. The reaction mixture was stirred at -78 °C for 7 hrs. The resulting mixture was quenched with saturated aqueous NH₄Cl and 1M HCl aq., and extracted with EtOAc and CH₂Cl₂. The combined extracts were washed with brine, dried over Na₂SO₄, and concentrated in vacuo. The residue was purified by silica gel chromatography (hexane: EtOAc = 5 : 1 to 3: 1 to 1: 1) to afford (−)-4 (4.60 g, 17.3 mmol, 61%) as a white crystal. Mp = 120-121 °C. [α]D²⁴ = -102.2 (c 0.93, CHCl₃). IR (film) 3284, 2965, 2931, 1637, 1598, 1551, 1469, 1261, 1083, 755 cm⁻¹. ¹H-NMR (600MHz, CDCl₃) δ 1.28 (d, 3H, J = 6.2Hz), 2.65 (dd, 1H, J = 9.1Hz, 13.6Hz), 2.81 (dd, 1H, J = 3.7, 13.6Hz), 3.82 (s, 3H), 3.99 (m, 1H), 4.11 (br s, 1H), 5.92 (br s, 1H), 6.78 (d, 1H, J = 8.3Hz), 6.85 (d, 1H, J = 7.6Hz), 7.27 (t, 1H, J = 8.3Hz). ¹³C-NMR (100MHz, CDCl₃) δ 24.3, 28.7 (×3), 42.4, 52.0, 55.9, 68.6, 109.2, 122.7, 127.6, 138.8, 156.0, 167.6. ESIMS calcd for C₁₅H₂₃NO₃Na ([M+Na]⁺) 288.1570, found 288.1565.
(R)-(−)-Mellein methyl ester, (−)-9

To a solution of (−)-4 (326.5 mg, 1.23 mmol) in toluene (18.0 mL) was added p-TsOH· H2O (264.1 mg, 1.39 mmol). The reaction mixture was refluxed and stirred for 2 hrs. The resulting mixture was concentrated in vacuo. The residue was purified by silica gel chromatography (hexane: AcOEt = 1: 1) to afford (−)-9 (169.0 mg, 0.87 mmol, 72%) as a white solid. $^1$H-NMR (600MHz, CDCl₃) δ 1.45 (d, 1H, $J = 6.2$Hz), 2.85 (dd, 1H, $J = 3.1$, 16.0Hz), 2.92 (dd, 1H, $J = 11.2$Hz, 16.0Hz), 3.95 (s, 3H), 4.56 (m, 1H), 6.80 (d, 1H, $J = 7.4$Hz), 6.92 (d, 1H, $J = 8.5$Hz), 7.45 (dd, 1H, $J = 7.4$, 8.5Hz).
(R)-(−)-Mellein, (−)-5

To a solution of (−)-9 (1.37 g, 7.12 mmol) in dry CH₂Cl₂ (71.0 mL) was added dropwise a solution of boron trichloride in CH₂Cl₂ (1.0M, 15.0 mL, 15.0 mmol) at -78 °C. After 1 hrs, the reaction mixture was raised at 0 °C and stirred for 1 hrs. The resulting mixture was quenched with saturated aqueous NH₄Cl, and extracted with CH₂Cl₂. The combined extracts were washed with brine, dried over Na₂SO₄, and concentrated in vacuo. The residue was purified by silica gel chromatography (hexane: AcOEt = 3: 1) to afford (−)-5 (1.21 g, 6.80 mmol, 95%) as white solid. Mp = 44 °C. [α]D²³ = −118.5 (c 0.02, CHCl₃).¹H-NMR (600MHz, CDCl₃) δ 1.54 (d, 3H, J = 6.4Hz), 2.94 (d, 2H, J = 7.3Hz), 4.74 (m, 1H), 6.70 (d, 1H, J = 7.3Hz), 6.90 (d, 1H, J = 8.5Hz), 7.41 (t, 1H, J = 7.32Hz), 11.0 (s, 1H). ¹³C-NMR (100MHz, CDCl₃) δ 20.7, 34.5, 76.0, 108.2, 116.1, 117.8, 136.1, 139.3, 162.1, 169.9.
(3R)-8-Hydroxy-3-methyl-1-oxo-3,4-dihydro-1H-isochromene-7-carbaldehyde, (−)-10

To a solution (−)-5 (206.7 mg, 1.16 mmol) in dry CH₂Cl₂ (5.0 mL) was added Cl₂CHOCH₃ (0.52 mL, 5.80 mmol) at −10 °C. To the resulting mixture was added dropwise TiCl₄ (5.8 mL, 5.80 mmol) at −10 °C. The reaction mixture was stirred at −10 °C for overnight. The resulting mixture was quenched with cold 1 M HCl aq., and extracted with CH₂Cl₂. The combined extracts were washed with brine, dried over Na₂SO₄, and concentrated in vacuo. The residue was purified by silica gel chromatography (hexane: AcOEt = 1: 1) to afford (−)-10 (209.4 mg, 0.99 mmol, 88%) as a white solid and ortho-formyl isomer (19.7 mg, 0.096 mmol, 8%) as a white solid. Mp = 117-118 °C. [α][D]₂₂ = -114.3 (c 0.78, CHCl₃): IR (film) 3020, 1685, 1617, 1433, 1272, 1216, 1129, 757, 669 cm⁻¹. ¹H-NMR (600MHz, CDCl₃) δ 1.57 (d, 3H, J = 6.4Hz), 3.00 (d, 1H, J = 2.6Hz), 3.01 (s, 1H), 4.79 (d, 1H, J = 7.9Hz), 8.00 (d, 1H, J = 7.9Hz), 10.49 (s, 1H), 11.80 (s, 1H). ¹³C-NMR (100MHz, CDCl₃) δ 20.5, 34.7, 75.9, 109.4, 118.2, 123.0, 134.6, 146.5, 164.1, 169.2, 187.9. EMIMS calcd for C₁₁H₁₀O₄Na ([M+Na]+) 229.0471, found 229.0487. ortho-Formyl isomer: ¹H-NMR (400MHz, CDCl₃) δ 1.59 (d, 3H, J = 8.0Hz), 3.07 (dd, 1H, J = 11.6Hz, 17.6Hz), 3.96 (dd, 1H, J = 3.0Hz, 17.6Hz), 4.72 (m, 1H), 7.06 (d, 1H, J = 8.4Hz), 7.94 (d, 1H, J = 8.4Hz), 10.02 (s, 1H), 11.95 (s, 1H).
Methyl (3R)-8-hydroxy-3-methyl-1-oxo-3,4-dihydro-1H-isochromene-7-carboxylate, (–)-11

To a solution of (–)-10 (179.6 mg, 0.87 mmol) in CH₃OH (14.5 mL) and CH₂Cl₂ (1.0 mL) was added NaCN (213.4 mg, 4.36 mmol). After 20 min, to resulting mixture was added MnO₂ (757.3 mg, 8.71 mmol). The reaction mixture was stirred at room temperature overnight. The resulting mixture was quenched with saturated aqueous sodium hydrogen sulfite, and stirred at room temperature for 30 min. The resultant mixture was extracted with CHCl₃. The combined extracts were washed with brine, dried over Na₂SO₄, and concentrated in vacuo. The residue was purified by silica gel chromatography (hexane: AcOEt = 3: 1 to 2 :1) to afford (–)-11 (186.3 mg, 0.79 mmol, 91%) as a white solid. Mp = 133-134 °C. [α]D²⁴ = -97.9 (c 0.75, CHCl₃) IR (film) 3021, 1724, 1670, 1619, 1431, 1216, 1138, 756, 668 cm⁻¹. ¹H-NMR (600MHz, CDCl₃) δ 1.54 (d, 3H, J = 6.3Hz), 2.97 (d, 2H, J=6.9Hz), 3.95 (s, 3H), 4.73 (m, 1H), 6.76 (d, 1H, J = 7.9Hz), 8.05 (d, 1H, J = 7.9Hz), 12.20 (s, 1H). ¹³C-NMR (100MHz, CDCl₃) δ 20.6, 35.1, 52.3, 75.4, 110.1, 117.0, 171.5, 137.9, 145.2, 162.5, 166.3, 168.1. EMIMS calcd for C₁₂H₁₂O₅Na ([M+Na]⁺) 259.0576, found 259.0577.
Methyl (3R)-8-(2-methoxy-2-oxoethoxy)-3-methyl-1-oxo-3,4-dihydro-1H-isochromene-7-carboxylate, (–)-12

To a solution of (–)-11 (472.1 mg, 2.00 mmol) in dry DMF (20.0 mL) was added K2CO3 (412.8 mg, 2.99 mmol). After 10 min, to the resulting mixture was added ethyl bromoacetate (0.28 mL, 3.00 mmol). The reaction mixture was stirred at 50 °C for 1 hrs 30 min. The resulting mixture was quenched with saturated aqueous NH4Cl, and extracted with CH2Cl2. The combined extracts were washed with brine, dried over Na2SO4, and concentrated in vacuo. The residue was purified by silica gel chromatography (hexane: AcOEt = 2: 1 to 1: 1) to afford (–)-12 (605.9 mg, 1.97 mmol, 98%) as a white solid. Mp = 95 °C. [α]D19 = –72.7 (c 0.57, CHCl3), IR (film) 3020, 1735, 1603, 1439, 1312, 1216, 1140, 1089, 756, 668 cm⁻¹. ¹H-NMR (600MHz, CDCl3) δ 1.50 (d, 3H, J = 6.3Hz), 2.94 (s, 1H), 2.95 (d, 1H, J = 3.9Hz), 3.84 (s, 3H), 3.90 (s, 3H), 4.57 (m, 1H), 4.80 (d, 1H, J = 14.9Hz), 4.93 (d, 1H, J = 14.9Hz), 7.11 (d, 1H, J = 7.9Hz), 7.97 (d, 1H, J = 7.9Hz). ¹³C-NMR (100MHz, CDCl3) δ 20.4, 35.9, 51.9, 52.5, 71.9, 74.1, 120.2, 123.5, 126.4, 135.8, 145.5, 159.2, 161.4, 165.4, 169.2. EMIMS calcd for C15H16O7Na ([M+Na⁺]⁺) 331.0788, found 331.0779.
A solution of (–)-12 (100.4 mg, 0.33 mmol) in CH$_3$OH (2.5 mL) was added to a solution of Na (9.0 mg, 0.391 mmol) in CH$_3$OH (4.0 mL). The reaction mixture was refluxed and stirred for 3 hrs. The resulting mixture was cooled to 0 °C. The residue was added CHCl$_3$ and quenched with saturated aqueous NH$_4$Cl, and extracted with CHCl$_3$. The combined extracts were washed with brine, dried over Na$_2$SO$_4$, and concentrated in vacuo. The residue was purified by silica gel chromatography (CHCl$_3$: CH$_3$OH = 50: 1) to afford (–)-13 (82.5 mg, 0.30 mmol, 93%) as a white solid. 

Mp = 160-163 °C. [α]$_D^{23}$ = -68.0 (c 0.44, CHCl$_3$). IR (film) 3019, 1727, 1614, 1281, 1216, 1122, 755, 669 cm$^{-1}$. $^1$H-NMR (600MHz, CDCl$_3$) $\delta$ 1.57 (d, 3H, $J = 6.3$Hz), 3.05 (dd, 1H, $J = 3.5$Hz, 16.6Hz), 3.11 (dd, 1H, $J = 11.0$Hz, 16.6Hz), 4.01 (s, 3H), 4.74 (m, 1H), 7.20 (d, 1H, $J = 8.0$Hz), 7.91 (d, 1H, $J = 8.0$Hz), 8.19 (br s, 1H). $^{13}$C-NMR (100MHz, CDCl$_3$) $\delta$ 20.7, 35.3, 52.1, 75.0, 111.2, 121.0, 122.6, 126.0, 127.3, 141.8, 150.2, 152.2, 161.5, 162.9. EMIMS calcd for C$_{14}$H$_{12}$O$_6$Na ([M+Na]$^+$) 299.0526, found 299.0543.
(7R)-7-Methyl-7H-furo[3,2-h]isochromene-3,9(2H,6H)-dione, (–)-6

To a cloudy solution (–)-13 (53.2 mg, 0.20 mmol) in DMSO (1.3 mL) and H₂O (1.9 mL) was LiOH· H₂O (38.5 mg, 0.92 mmol). The reaction mixture was stirred at 75°C for 3 hrs. The resulting mixture was quenched with 10% HCl aq., and extracted with CHCl₃. The combined extracts were washed with brine, dried over Na₂SO₄, and concentrated in vacuo. The residue was purified by silica gel chromatography (hexane: AcOEt = 1: 1 to 1: 2) to afford (–)-6 (34.0 mg, 0.16 mmol, 80%) as a white solid. Mp = 236 °C. [α]D²⁴ = –126.5 (c 0.64, CHCl₃), IR (film) 3019, 1720, 1611, 1442, 1216, 1124, 756, 669 cm⁻¹. ¹H-NMR (600MHz, CDCl₃) δ 1.56 (d, 3H, J = 6.3Hz), 3.03 (s, 1H), 3.04 (d, 1H, J = 5.5Hz), 4.71 (m, 1H), 4.81 (d, 1H, J = 18.4Hz), 4.87 (d, 1H, J = 18.4Hz), 7.07(d, 1H, J = 7.7Hz), 7.85(d, 1H, J = 7.7Hz). ¹³C-NMR (100MHz, CDCl₃) δ 20.7, 35.8, 74.5, 75.9, 111.8, 121.4, 122.3, 129.3, 150.1, 161.4, 173.8, 197.8. EMIMS calcd for C₁₂H₁₀O₄Na ([M+Na⁺]²⁻) 241.0471, found 241.0466.
To a solution of (−)-6 (105.1 mg, 0.48 mmol) in dry acetone (10.0 mL) was added TsOH $\cdot$ H$_2$O (109.9 mg, 0.58 mmol). The reaction mixture was refluxed and stirred for 3 hrs 20 min. The resulting mixture was quenched with saturated aqueous sodium hydrogen carbonate, and extracted with CHCl$_3$. The combined extracts were washed with brine, dried over Na$_2$SO$_4$, and concentrated in vacuo. The residue was purified by silica gel chromatography (hexane: AcOEt = 1: 1 to 1: 2) to afford (−)-7 (60.9 mg, 0.24 mmol, 49%) as a white needle crystal. Mp = 217-218 °C. $[\alpha]_D^{23} = -102.8$ (c 0.76, CHCl$_3$) IR (film) 3019, 1727, 1699, 1651, 1607, 1435, 1286, 1216, 1178, 1117, 755, 688 cm$^{-1}$. $^1$H-NMR (600MHz, CDCl$_3$) $\delta$ 1.56 (d, 3H, $J$ = 6.3Hz), 2.33 (s, 3H), 2.40 (s, 3H), 3.02 (s, 1H), 3.03 (d, 1H, $J$ = 8.8Hz), 4.71 (m, 1H), 7.01 (d, 1H, $J$ = 7.7Hz), 7.89 (d, 1H, $J$ = 7.7Hz). $^{13}$C-NMR (100MHz, CDCl$_3$) $\delta$ 17.5, 20.7, 35.8, 74.5, 110.8, 121.3, 124.3, 129.2, 135.1, 145.1, 148.2, 161.2, 164.1, 182.1, 182.1. EMIMS calcd for C$_{15}$H$_{14}$O$_4$Na ([M+Na]$^+$) 281.0784, found 281.0780.
To a solution of (–)-7 (12.9 mg, 49.9 µmol) in dry THF (2.5 mL) was added dropwise a solution of DIBAL (0.4M in THF/ n-hexane, 0.15 mL, 59.9 µmol) at −78 °C. The reaction mixture was stirred at −78 °C for 2 hrs, a solution of DIBAL (0.4M in THF/ n-hexane, 0.20 mL, 79.9 µmol) was added dropwise to the reaction mixture. The reaction mixture was stirred at −78 °C for 5 hrs. The resulting mixture was quenched with saturated aqueous NH₄Cl and 1 M HCl aq., and stirred vigorously. The resulting mixture was extracted with with CHCl₃. The combined extracts were washed with brine, dried over Na₂SO₄, and concentrated in vacuo. The residue was purified by silica gel chromatography (hexane: AcOEt = 2: 1 to 1: 1) to afford (+)-1 (3.6 mg, 13.8 µmol, 28%) as a white crystal. Mp = 170 °C. [α]D²³ = +63.7 (c 0.08, CH₃OH). IR (film) 3399, 3019, 1697, 1652, 1614, 1438, 1119, 1079, 1027, 854, 669 cm⁻¹. ¹H-NMR (600MHz, CDCl₃) δ 1.40 (d, 3H, J = 6.2Hz), 2.13 (s, 3H), 2.37 (s, 3H), 2.72 (dd, 1H, J = 11.0Hz, 17.2Hz), 2.79 (dd, 1H, J = 3.1Hz, 17.2Hz), 2.98 (d, 1H, J = 4.2Hz), 4.46 (m, 1H), 6.28 (d, 1H, J = 4.2Hz), 6.89 (d, 1H, J = 7.9Hz), 7.62 (d, 1H, J = 7.9Hz). ¹³C-NMR (100MHz, CDCl₃) δ 17.4, 20.2, 21.0, 35.9, 62.8, 87.6, 119.5, 121.9, 122.6, 123.6, 132.3, 143.7, 145.2, 161.9, 183.2. EMIMS calcd for C₁₅H₁₆O₄Na ([M+Na⁺]²⁺) 283.0940, found 283.0959. Enantiomeric excess was determined by HPLC using a chiral colume (DAICEL CHIRALPAK OD; hexane: isopropanol = 15: 1; flow rate 0.5 mL/min; (R)-(+)−1, RT = 53 min; (S)-(−)−1, RT = 37 min; monitoring 254nm; 99% e.e.).
8-Hydroxy-3-methyl-7-propionyl-3,4-dihydro-1H-isochromen-1-one, (±)-15

To a solution of (±)-10 (20.6 mg, 0.10 mmol) in THF (2.0 mL) was added EtMgBr (220 µL, a 1.0 M solution in Et₂O, 0.22 mmol) at –5 °C. The mixture was stirred at rt for 40 min. Then the mixture was quenched by the addition of 1N HCl aqueous solution and diluted with EtOAc. After the layers were separated, the organic layer was wash with H₂O, brine, dried (Na₂SO₄), and evaporated.

To a solution of the residue in CH₂Cl₂ (2.5 mL) was added MnO₂ (159 mg, 85% purity, 1.56 mmol), and the mixture was stirred for 3 hr. Then MnO₂ (239 mg, 85% purity, 2.34 mmol) was added to the mixture, and the mixture was stirred for 13 hr. The mixture was filtrated through Celite and washed with CH₂Cl₂. The solvent was removed under a reduced pressure. The residue was purified by silica gel chromatography (hexane : EtOAc = 2 : 1) to give (±)-15 (12.2 mg, 52%) as a white solid. Mp = 103~104 °C. IR (KBr) 2978, 2930, 1667, 1614, 1424, 1389, 1274, 1224, 1125, 960, 816, 632 cm⁻¹. ¹H NMR (600 MHz, CDCl₃) δ 1.20 (t, 3H, J = 7.2 Hz), 1.55 (d, 3H, J = 6.3 Hz), 2.97 (m, 2H), 3.11 (m, 2H), 4.74 (m, 1H), 6.77 (d, 1H, J = 7.9 Hz), 8.00 (d, 1H, J = 7.9 Hz), 12.3 (s, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 8.2, 20.7, 34.9, 36.6, 75.7, 109.6, 117.9, 125.2, 137.2, 144.7, 162.3, 169.2, 201.5. HRMS: calcd for C₁₃H₁₄O₄Na ([M+Na]⁺) 257.0784, found 257.0785.
2,6,7-Trimethyl-7,8-dihydro-4H,10H-pyrano[4,3-h]chromene-4,10-dione, (±)-16

A solution of (±)-15 (18.6 mg, 0.079 mmol), Ac₂O (15 µL, 0.16 mmol) and DBU (50 µL, 0.33 mmol) in pyridine (1.5 mL) was stirred at 60 ºC for 6 hr. Then the mixture was evaporated in vacuo. The residue was purified by PTLC (hexane : EtOAc = 1.5 : 1) to give (±)-16 (8.0 mg, 40%) as a white solid. Mp = 191~193 ºC. IR (KBr) 2971, 2925, 2858, 1725, 1637, 1437, 1400, 1273, 1172, 1114, 963, 882, 795, 693, 621. ¹H NMR (600 MHz, CDCl₃) δ 1.55 (d, 3H, J = 6.2 Hz), 2.07 (s, 3H), 2.51 (s, 3H), 3.02 (dd, 1H, J = 16.6 Hz, 3.3 Hz), 3.07 (dd, 1H, J = 16.6 Hz, 10.9 Hz), 4.67 (m, 1H), 7.23 (d, 1H, J = 8.1 Hz), 8.39 (d, 1H, J = 8.1 Hz). ¹³C NMR (100 MHz, CDCl₃) δ 9.9, 18.7, 20.6, 36.1, 74.3, 114.1, 117.6, 122.7, 123.4, 131.6, 146.2, 155.4, 160.9, 162.8, 176.9. HRMS: calcd for C₁₃H₁₅O₄ ([M+H]+) 259.0964, found 259.0953.
10-Hydroxy-2,3,8-trimethyl-7,10-dihydro-4\(H\),8\(H\)-pyrano[4,3-\(h\)]chromen-4one, the proposed structure of aspergione B, (±)-2

To a solution of (±)-16 (1.5 mg, 0.0059 mmol) was added DIBAL-H (18 µL, a 0.97 M solution in hexanes, 0.018 mmol) at –78 ºC and the mixture was stirred at –78 ºC for 20 min. Then the mixture was quenched by the addition of saturated aqueous solution of Na\(_2\)SO\(_4\) and Celite. After the mixture was stirred at rt for 1 hr, the mixture was filtrated through Celite and washed with CH\(_2\)Cl\(_2\). The solvent was removed under a reduced pressure. The residue was purified by PTLC (hexane : EtOAc = 1 : 2) to give (±)-2 (1.4 mg, 92%) as a white solid. Mp = 219~224 ºC. IR (KBr) 3311, 2927, 2859, 1605, 1568, 1492, 1433, 1082, 1018, 849, 786, 771, 713, 619. \(^1\)H NMR (600 MHz, CDCl\(_3\)) \(\delta\) 1.41 (d, 3H, \(J = 6.2\) Hz) 2.06 (s, 3H), 2.45 (s, 3H), 2.74 (dd, 1H, \(J = 17.0\) Hz, 11.2 Hz), 2.82 (dd, 1H, \(J = 17.0\) Hz, 3.4 Hz), 3.04 (br s, 1H), 4.51 (ddq, 1H, \(J = 11.2\) Hz, 3.4 Hz, 6.2 Hz), 6.37 (d, 1H, \(J = 4.0\) Hz), 7.10 (d, 1H, \(J = 8.2\) Hz), 8.09 (d, 1H, \(J = 8.2\) Hz). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 10.0, 18.6, 21.2, 35.5, 62.6, 88.1, 117.1, 121.0, 123.1, 124.8, 125.6, 140.3, 153.2, 161.3, 177.7. HRMS: calcd for C\(_{15}\)H\(_{16}\)O\(_4\)Na ([M+Na]+) 283.0940, found 283.0945.

Reference