Supporting Information

Enantioselective Total Synthesis of Isishippuric Acid B via Intramolecular Michael Reaction

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Contents

General Information	S2
Experimental Procedures and Characterization for Compounds 7 , 8 , 10 , 11 , 14 , 15 , 16 and 1 ---	S2–S6
¹ H and ¹³ C NMR Spectra for Compounds 7 , 8 , 10 , 11 , 14 , 15 , 16 and 1	S7–S22

General Information

IR spectra were recorded by a Jasco FT/IR-4100 spectrometer. NMR spectra were recorded with TMS as an internal standard in CDCl₃ by a Varian Gemini 2000 spectrometer (300 MHz for ¹H and 75 MHz for ¹³C) or a Varian UNITY plus-500 spectrometer (500 MHz for ¹H and 125 MHz for ¹³C). Optical rotation values were measured with a Jasco DIP-371 polarimeter, and mass spectra were obtained with a Jeol JMS-700 spectrometer operated in the EI or FAB mode. Merck silica gel 60 (70–230 mesh) or Kanto Kagaku silica gel 60N (spherical neutral, particle size 100–210 μm) were used for column chromatography.

Dimethyl (*R*)-4,8-dimethyl-2-oxo-7-nonenylphosphonate (7). To a stirred solution of dimethyl methylphosphonate (12.1 mL, 112 mmol) in THF (150 mL) was added dropwise *n*-BuLi (1.6 M in hexane, 70 mL, 112 mmol) at –78 °C. After 1.5 h, **6** (17.6 mL, 97 mmol) in THF (200 mL) was added dropwise, and the resulting mixture was gradually warmed to 0 °C over 5 h. The reaction mixture was quenched with sat. NH₄Cl aq and concentrated in vacuo. The residue was mixed with acetone (350 mL), and the Jones reagent (2.67 M, 54.5 mL, 146 mmol) was added dropwise to the mixture at 0 °C. After being stirred for 2 h at 0 °C, the reaction mixture was quenched with 2-propanol, and concentrated in vacuo. The residue was diluted with water and extracted with EtOAc. The extract was successively washed with water and brine, dried (MgSO₄) and concentrated in vacuo. The residue was chromatographed over silica gel (hexane/EtOAc = 3:2 to 0:1) to give 19.3 g (72%) of **7** as a colorless oil. $[\alpha]_D^{24} +5.05$ (*c* = 1.00, CHCl₃); IR (film): $\nu_{\max}$ 1713 (s), 1258 (s), 1024 (vs); ¹H NMR (500 MHz): δ 0.91 (3H, d, *J* = 6.3 Hz), 1.16–1.24 (1H, m), 1.28–1.36 (1H, m), 1.60 (3H, br s), 1.68 (3H, br s), 1.91–2.07 (3H, m), 2.44 (1H, dd, *J* = 17.0, 8.3 Hz), 2.60 (1H, dd, *J* = 17.0, 5.4 Hz), 3.05 (1H, dd, *J* = 22.5, 13.7 Hz), 3.09 (1H, dd, *J* = 22.5, 13.7 Hz), 3.79 (6H, d, *J* = 11.2 Hz), 5.08 (1H, t, *J* = 7.1 Hz); ¹³C NMR (125 MHz): δ 17.5, 19.4, 25.3, 25.6, 28.4, 36.6, 41.5 (d, *J* = 127.8 Hz), 51.3, 52.9 (d, *J* = 6.3 Hz), 124.1, 131.4, 201.6 (d, *J* = 6.9 Hz); HRMS (FAB) *m/z*: calcd for C₁₃H₂₆O₄P, 277.1569; found, 277.1567 ([M+H]⁺).

(*R*)-6-Methyl-2-cyclohepten-1-one (8). Ozone was bubbled into a solution of **7** (4.20 g, 15.2 mmol) in CH₂Cl₂ (80 mL) at –78 °C for 25 min. After removal of excess ozone by a stream of O₂ (for about 15 min), Me₂S (excess) was added, and the resulting mixture was gradually warmed to room temperature over 6 h. The mixture was diluted with ether (150 mL), mixed with LiCl (773 mg, 18.2 mmol), and stirred for 15 min. To the mixture was added dropwise DBU (2.6 mL, 18.2 mmol), and the resulting mixture was stirred at room temperature for 2 h. The mixture was quenched with sat. NH₄Cl aq and concentrated in vacuo. The residue was diluted with water and extracted with ether. The extract was dried (MgSO₄) and concentrated in vacuo. The residue was chromatographed over silica gel (hexane/EtOAc = 10:1) to give 1.45 g (77%) of **8** as a pale yellow oil. $[\alpha]_D^{24} +59.8$ (*c* = 1.05, CHCl₃) [lit.^{7a} $[\alpha]_D^{24} +64.6$ (*c* = 1.09, CHCl₃); lit.^{7b} $[\alpha]_D^{20} -59$ (*c* = 1.0, CHCl₃) for the enantiomer of **8**]; IR (film): $\nu_{\max}$ 3027 (w), 1664 (s); ¹H NMR (500 MHz): δ 1.03 (3H, d, *J* = 6.8 Hz), 1.51–1.59 (1H, m), 1.89–1.96 (1H, m), 2.05–2.14 (1H, m), 2.36–2.53 (3H, m), 2.69 (1H, dd, *J* = 14.4, 4.4 Hz), 6.01 (1H, d, *J* = 11.7 Hz), 6.64 (1H, dt, *J* = 11.7, 4.9 Hz); ¹³C NMR (125MHz): δ 22.0, 28.3, 28.4, 34.8, 51.3, 132.7, 147.3, 203.1; HRMS (EI): *m/z* calcd for C₈H₁₂O, 124.0888; found,

124.0886 (M⁺).

Methyl (1*S*,2*R*,5*R*,6*R*)-2-methyl-8-oxobicyclo[3.2.2]nonane-6-carboxylate (10). To a stirred solution of LDA [prepared by stirring (*i*-Pr)₂NH (2.97 mL, 21.2 mmol) and *n*-BuLi (1.6 M in hexane, 12.8 mL, 20.4 mmol) in THF (150 mL) at 0 °C for 20 min] was added dropwise a solution of **8** (1.95 g, 15.7 mmol) in THF (100 mL) at –78 °C. After 1 h, TIPSCl (5.04 mL, 23.6 mmol) and HMPA (5.5 mL) were successively added, and the resulting mixture was gradually warmed to 0 °C over 12 h. The reaction mixture was quenched with sat. NaHCO₃ aq and concentrated in vacuo. The residue was diluted with hexane, and the mixture was successively washed with water and brine. The hexane solution was filtered through a short column of SiO₂, and the column was washed with hexane. The filtrate was concentrated in vacuo to give crude **9** as a pale yellow oil, which was used in the next step without purification. ¹H NMR (300 MHz): δ 1.02 (3H, d, *J* = 6.6 Hz), 1.09 (18H, d, *J* = 5.4 Hz), 1.10–1.20 (3H, m), 1.58–1.72 (1H, m), 1.72–1.82 (1H, m), 2.24–2.32 (2H, m), 2.33–2.47 (1H, m), 5.09 (1H, dd, *J* = 4.8, 1.3 Hz), 5.72 (1H, dm, *J* = 11.8 Hz), 5.83 (1H, dt, *J* = 11.8, 4.9 Hz). To a stirred solution of the crude silyl enol ether (**9**) obtained above in CH₂Cl₂ (50 mL) was added methyl acrylate (7.1 mL, 78.5 mmol) at –78 °C, and the mixture was stirred for 10 min. To the solution was added dropwise EtAlCl₂ (0.96 M in hexane, 8.20 mL, 7.85 mmol) at –78 °C. After 2 h, the reaction mixture was quenched with sat. NaHCO₃, and extracted with EtOAc. The extract was successively washed with water and brine, dried (MgSO₄) and concentrated in vacuo. The residue was chromatographed over SiO₂ (hexane/EtOAc = 7:1) to give 2.31 g (70% from **8**) of **10** as a colorless oil. [α]_D²⁴ +13.2 (*c* = 1.02, CHCl₃); IR (film): ν_{max} 1730 (vs), 1711 (vs), 1169 (s); ¹H NMR (500 MHz): δ 1.02 (3H, d, *J* = 6.8 Hz), 1.12–1.23 (1H, m), 1.63–1.71 (2H, m), 1.78–1.86 (1H, m), 1.94–2.01 (1H, m), 2.10 (1H, ddd, *J* = 14.7, 8.3, 3.9 Hz), 2.19 (1H, dd, *J* = 14.7, 10.7 Hz), 2.23 (1H, d, *J* = 18.6 Hz), 2.31 (1H, br s), 2.54 (1H, dd, *J* = 18.6, 4.9 Hz), 2.58–2.62 (1H, m), 2.83 (1H, br t, *J* = 9.0 Hz), 3.70 (3H, s); ¹³C NMR (125 MHz): δ 22.7, 28.0, 29.9, 31.9, 33.9, 36.7, 39.7, 42.7, 51.9, 53.4, 176.2, 213.5; HRMS (FAB): *m/z* calcd for C₁₂H₁₉O₃, 211.1334; found, 211.1340 ([M+H]⁺).

Methyl (1*S*,2*R*,5*R*,6*R*)-8,8-ethylenedioxy-2-methylbicyclo[3.2.2]nonane-6-carboxylate (11). A mixture of **10** (150 mg, 0.713 mmol), HO(CH₂)₂OH (0.12 mL, 2.1 mmol) and TsOH·H₂O (14 mg, 71.3 μmol) was stirred at reflux for 2 h. After being cooled to room temperature, the reaction mixture was quenched with sat. NaHCO₃ aq, and extracted with EtOAc. The extract was successively washed with water and brine, dried (MgSO₄) and concentrated in vacuo. The residue was chromatographed over SiO₂ (hexane/EtOAc = 7:1) to give 166 mg (91%) of **11** as a colorless oil. [α]_D²⁴ –16.7 (*c* = 2.10, CHCl₃); IR (film): ν_{max} 1736 (s), 1111 (m); ¹H NMR (500 MHz): δ 1.06 (3H, d, *J* = 6.8 Hz), 1.41–1.49 (2H, m), 1.60–1.67 (1H, m), 1.74 (1H, t, *J* = 3.4 Hz), 1.78–1.86 (1H, m), 1.88–1.98 (3H, m), 2.14 (1H, ddd, *J* = 14.2, 7.8, 3.9 Hz), 2.18 (1H, dd, *J* = 15.1, 5.9 Hz), 2.41 (1H, br t, *J* = 6.1 Hz), 2.58 (1H, br t, *J* = 9.3 Hz), 3.67 (3H, s), 3.78–3.83 (1H, m), 3.87–3.93 (2H, m), 3.94–3.99 (1H, m); δ 24.4, 28.5, 28.8, 31.6, 35.2, 36.1, 37.1, 43.2, 44.1, 51.4, 63.0, 64.2, 110.9, 176.4; HRMS (EI): *m/z* calcd for C₁₄H₂₂O₄, 254.1518; found, 254.1515 (M⁺).

(1R,4R,5S)-4,8,8-trimethylbicyclo[3.2.2]nonan-6-one (14). To a stirred solution of LDA [prepared from (*i*-Pr)₂NH (1.10 mL, 7.87 mmol) and *n*-BuLi (1.6 M in hexane, 4.74 mL, 7.59 mmol) in THF (70 mL)] was added dropwise a solution of **11** (1.43 g, 5.62 mmol) in THF (50 mL) at –78 °C. After 50 min, a solution of MeI (1.15 mL, 18.0 mmol) in HMPA (3.2 mL) was added at –78 °C, and the resulting mixture was gradually warmed to 0 °C over 7 h. The reaction mixture was quenched with sat. NH₄Cl aq and concentrated in vacuo. The residue was diluted with water, and extracted with ether. The extract was successively washed with water and brine, dried (MgSO₄) and concentrated in vacuo to give crude **12** as a pale yellow oil, which was used in the next step without purification. ¹H NMR (300 MHz): δ 1.03 (3H, d, *J* = 6.6 Hz), 1.15–1.26 (1H, m), 1.35 (3H, s), 1.32–1.39 (1H, m), 1.55–1.77 (5H, m), 1.97 (1H, d, *J* = 13.8 Hz), 2.16–2.28 (2H, m), 2.61 (1H, dd, *J* = 13.8, 1.8 Hz), 3.69 (3H, s), 3.82–4.00 (4H, m). To a stirred suspension of LAH (427 mg, 11.2 mmol) in ether (70 mL) was added dropwise a solution of the crude ester (**12**) obtained above in ether (40 mL) at 0 °C. After 3 h, the reaction mixture was quenched with sat. NH₄Cl aq and extracted with ether. The extract was successively washed with water and brine, dried (MgSO₄) and concentrated in vacuo to give an alcohol as an colorless oil, which was used in the next step without purification. ¹H NMR (300 MHz): δ 1.03 (3H, d, *J* = 6.9 Hz), 1.12–1.20 (1H, m), 1.15 (3H, s), 1.30–1.36 (1H, m), 1.38–1.46 (1H, m), 1.50–1.82 (6H, m), 1.85 (1H, br t, *J* = 7.3 Hz), 1.96 (1H, dd, *J* = 15.7, 1.6 Hz), 2.22 (1H, dd, *J* = 15.7, 5.8 Hz), 3.33 (1H, dd, *J* = 11.0, 3.6 Hz), 3.69 (1H, dd, *J* = 11.0, 3.6 Hz), 3.81–4.01 (4H, m). To a stirred solution of the crude alcohol obtained above and Et₃N (2.74 mL, 19.7 mmol) in CH₂Cl₂ (50 mL) was added dropwise MsCl (0.87 mL, 11.2 mmol) at 0 °C. After 1 h, the reaction mixture was quenched with sat. NH₄Cl aq and extracted with ether. The extract was successively washed with water and brine, dried (MgSO₄) and concentrated in vacuo to give crude **13** as a pale yellow oil, which was used in the next step without purification. ¹H NMR (300 MHz): δ 1.03 (3H, d, *J* = 6.6 Hz), 1.21 (3H, s), 1.25 (1H, d, *J* = 12.6 Hz), 1.41–1.47 (1H, m), 1.47–1.79 (6H, m), 1.88 (1H, br t, *J* = 6.2 Hz), 1.97 (1H, dd, *J* = 15.7, 1.6 Hz), 2.22 (1H, dd, *J* = 15.7, 6.0 Hz), 3.02 (3H, s), 3.80–4.02 (5H, m), 4.23 (1H, d, *J* = 9.3 Hz). To a stirred solution of the crude mesylate (**13**) obtained above in HMPA (100 mL) was successively added NaI (4.20 g, 28.1 mmol) and Zn powder (3.70 g, 56.2 mmol) at room temperature. The mixture was then stirred at 110 °C for 25 h. After being cooled to room temperature, the mixture was filtered and diluted with hexane. The hexane solution was successively washed with water and brine, dried (MgSO₄) and concentrated in vacuo to give the ethylene acetal of **14** as an oil, which was used in the next step without purification. ¹H NMR (300 MHz): δ 1.02 (3H, d, *J* = 6.6 Hz), 1.05 (3H, s), 1.07 (3H, s), 1.36 (1H, dd, *J* = 14.2, 1.9 Hz), 1.39–1.46 (1H, m), 1.48–1.74 (5H, m), 1.79 (1H, dd, *J* = 14.2, 5.5 Hz), 1.83–1.92 (1H, m), 1.92 (1H, dd, *J* = 15.4, 1.4 Hz), 2.26 (1H, dd, *J* = 15.4, 5.5 Hz), 3.81–4.00 (4H, m). A solution of the crude acetal obtained above in acetone (40 mL) was mixed with 1 M HCl aq (1.5 mL) and stirred at room temperature for 1 h. The reaction mixture was quenched with sat. NaHCO₃ aq, and concentrated in vacuo. The residue was diluted with water and extracted with ether. The extract was successively washed with water and brine, dried (MgSO₄) and concentrated in vacuo. The residue was chromatographed over SiO₂ (hexane/EtOAc = 30:1) to give 851 mg (84% from **11**) of **14** as a colorless oil. [α]_D²⁴ +22.9 (*c* = 1.26, CHCl₃); IR (film): $\nu_{\max}$ 1711 (s); ¹H NMR (500 MHz): δ 0.94 (3H, d, *J* = 6.8 Hz), 1.03 (3H, s),

1.11–1.21 (1H, m), 1.16 (3H, s), 1.64–1.76 (4H, m), 1.77–1.82 (1H, m), 1.82–1.91 (1H, m), 2.02–2.10 (1H, m), 2.17 (1H, dd, $J = 19.5, 1.5$ Hz), 2.18–2.21 (1H, m), 2.58 (1H, dd, $J = 19.5, 5.4$ Hz): δ 22.2, 26.4, 30.0, 30.7, 31.5, 32.4, 38.5, 40.3, 40.6, 41.7, 54.7, 215.8; HRMS (EI): m/z calcd for $C_{12}H_{20}O$, 180.1514; found, 180.1518 (M^+).

Methyl (1*S*,2*R*,5*R*)-5-[2-(*tert*-butoxycarbonyl)vinyl]-2,6,6-trimethylcycloheptane-1-carboxylate (15). To a stirred solution of NaHMDS (1.03 M in THF, 2.83 mL, 2.91 mmol) in DMF/HMPA (5:2, 14 mL) was added dropwise a solution of **14** (420 mg, 2.33 mmol) in DMF (10 mL) at 0 °C. After 20 min, Me_2SO_4 (0.300 mL, 3.14 mmol) was added dropwise, and the mixture was gradually warmed to room temperature over 2 h. The reaction mixture was quenched with sat. $NaHCO_3$ aq and extracted with hexane. The hexane solution was successively washed with water and brine, dried ($MgSO_4$) and concentrated in vacuo to give a methyl enol ether as a pale yellow oil, which was used in the next step without purification. 1H NMR (300 MHz): δ 0.90 (3H, d, $J = 6.9$ Hz), 0.95 (3H, s), 1.07–1.17 (1H, m), 1.10 (3H, s), 1.35–1.56 (3H, m), 1.59–1.73 (3H, m), 1.88–1.95 (1H, m), 2.01–2.07 (1H, m), 3.53 (3H, s), 4.79 (1H, dd, $J = 8.0, 2.2$ Hz). Ozone was bubbled into a solution of the crude enol ether obtained above and pyridine (0.380 mL, 4.66 mmol) in MeOH (25 mL) at –78 °C for 5 min. After removal of excess ozone by a stream of O_2 (for about 15 min), Me_2S (excess) was added, and the resulting mixture was gradually warmed to room temperature over 12 h. The mixture was concentrated in vacuo. The residue was diluted with a mixture of hexane and EtOAc (15:1) and filtered through a pad of SiO_2 . The filtrate was concentrated in vacuo to give a formyl ester, which was used in the next step without purification. 1H NMR (300 MHz): δ 0.89 (3H, d, $J = 6.9$ Hz), 0.92 (3H, s), 1.19–1.28 (1H, m), 1.21 (3H, s), 1.40–1.87 (5H, m), 2.01 (1H, br d, $J = 9.3$ Hz), 2.37–2.51 (1H, m), 2.61 (1H, dd, $J = 9.3, 3.0$ Hz), 3.68 (3H, s), 9.74 (1H, d, $J = 1.9$ Hz). To a stirred solution of $Ph_3P=CHCO_2t-Bu$ (1.05 g, 2.80 mmol) in toluene (10 mL) was added dropwise a solution of the formyl ester obtained above in toluene (10 mL) at room temperature, and the mixture was stirred at 60 °C for 1 day. The mixture was diluted with ether, and the ethereal solution was successively washed with water and brine, dried ($MgSO_4$) and concentrated in vacuo. The residue was chromatographed over SiO_2 (hexane/EtOAc = 40:1) to give 377 mg (50% from **14**) of **15** as a white solid (mp 79–80 °C). $[\alpha]_D^{24} -31.2$ ($c = 0.110, CHCl_3$); IR (film): ν_{max} 1734 (s), 1712 (vs), 1650 (w), 1155 (s), 1140 (vs); 1H NMR (500 MHz): δ 0.83 (3H, s), 0.87 (3H, d, $J = 6.8$ Hz), 0.93 (3H, s), 1.29–1.36 (1H, m), 1.48 (9H, s), 1.58–1.70 (4H, m), 1.75 (1H, dd, $J = 15.1, 9.8$ Hz), 1.93 (1H, t, $J = 8.5$ Hz), 2.37–2.44 (1H, m), 2.64 (1H, dd, $J = 9.8, 2.4$ Hz), 3.67 (3H, s), 5.63 (1H, d, $J = 15.6$ Hz), 6.79 (1H, dd, $J = 15.6, 9.8$ Hz): δ 15.0, 24.1, 26.0, 28.06, 28.14, 32.4, 35.2, 35.9, 36.1, 42.7, 51.6, 52.1, 80.0, 122.0, 150.5, 166.1, 176.5; HRMS (EI): m/z calcd for $C_{19}H_{32}O_4$, 324.2301; found, 324.2301 (M^+).

Methyl (1*R*,2*R*,5*R*,8*R*)-8-[(*tert*-butoxycarbonyl)methyl]-2,6,6-trimethylbicyclo[3.2.1]octane-1-carboxylate (16). To a stirred solution of LHMDs [prepared by stirring $(TMS)_2NH$ (83 μL , 398 μmol) and $n-BuLi$ (1.6 M in hexane, 250 μL , 398 μmol) in THF (3 mL) at 0 °C for 20 min] was added dropwise a solution of **15** (86 mg, 27 μmol) in a mixture of THF (3 mL) and HMPA (0.35 mL) at 0 °C. After being

stirred for 2 h at 0 °C, the reaction mixture was quenched with sat. NH_4Cl aq and extracted with ether. The extract was washed with water and brine, dried (MgSO_4) and concentrated in vacuo. The residue was chromatographed over SiO_2 (hexane/EtOAc = 50:1) to give 47 mg (55%) of **16** as a colorless oil and 20 mg (23%) of its (8*S*)-epimer (**17**) as a colorless oil. **16**: $[\alpha]_D^{24}$ -8.6 (c = 0.80, CHCl_3); IR (film): ν_{max} 1733 (s); ^1H NMR (500 MHz): δ 1.03 (3H, d, J = 7.3 Hz), 1.12 (3H, s), 1.13 (3H, s), 1.24–1.30 (1H, m), 1.47 (9H, s), 1.59–1.65 (2H, m), 1.65–1.71 (1H, m), 1.74 (1H, d, J = 14.2 Hz), 1.99–2.08 (2H, m), 2.05 (1H, d, J = 14.2 Hz), 2.21 (1H, dd, J = 16.1, 6.3 Hz), 2.62 (1H, br t, J = 6.8 Hz), 2.81 (1H, dd, J = 16.1, 7.8 Hz), 3.61 (3H, s); ^{13}C NMR (125 MHz): δ 18.2, 26.3, 26.4, 27.5, 28.2, 34.3, 37.7, 39.7, 40.1, 42.5, 48.8, 51.1, 51.5, 59.4, 79.8, 172.6, 176.1; HRMS (FAB): m/z calcd for $\text{C}_{19}\text{H}_{33}\text{O}_4$, 325.2379; found, 325.2378 ($[\text{M}+\text{H}]^+$).

(1*R*,2*R*,5*R*,8*R*)-8-Carboxymethyl-2,6,6-trimethylbicyclo[3.2.1]octane-1-carboxylic acid (1). To a stirred solution of **16** (21 mg, 65 μmol) in THF (3 mL) was successively added *t*-BuOK (145 mg, 1.29 mmol) and water (10 μL , 520 μmol), and the mixture was stirred at 50 °C for 36 h. The reaction mixture was quenched with 1 M HCl aq, stirred for an additional 10 min, and extracted with CH_2Cl_2 . The extract was successively washed with water and brine, dried (MgSO_4) and concentrated in vacuo. The residue was chromatographed over SiO_2 ($\text{CHCl}_3/\text{MeOH}/\text{AcOH}$ = 100:2:1) to give 14 mg (85%) of **1** as a white solid. $[\alpha]_D^{24}$ -104 (c = 0.075, CHCl_3); IR (film): ν_{max} 1700 (vs), 1409 (w), 1290 (m), 937 (w); ^1H NMR (500 MHz, pyridine- d_5): δ 1.10 (3H, s), 1.23–1.27 (1H, m), 1.27 (3H, s), 1.36 (3H, d, J = 6.8 Hz), 1.58–1.64 (1H, m), 1.74–1.82 (1H, m), 1.85 (1H, br s), 1.96 (1H, d, J = 14.2 Hz), 2.04–2.13 (1H, m), 2.36–2.43 (1H, m), 2.45 (1H, d, J = 14.2 Hz), 2.73 (1H, dd, J = 16.1, 7.3 Hz), 3.16 (1H, br t, J = 7.3 Hz), 3.64 (1H, dd, J = 16.1, 7.8 Hz); ^{13}C NMR (125 MHz, pyridine- d_5): δ 19.2, 27.2, 27.3, 28.5, 35.2, 38.4, 40.3, 40.5, 44.0, 50.1, 52.4, 60.3, 176.4, 178.9; HRMS (FAB): m/z calcd for $\text{C}_{14}\text{H}_{23}\text{O}_4$, 255.1596; found, 255.1604 ($[\text{M}+\text{H}]^+$).

^1H NMR
(500 MHz, CDCl_3)

^{13}C NMR
(125 MHz, CDCl_3)

^1H NMR
(500 MHz, CDCl_3)

^{13}C NMR
(125 MHz, CDCl_3)

¹H NMR
(500 MHz, CDCl₃)

^{13}C NMR
(125 MHz, CDCl_3)

^1H NMR
(500 MHz, CDCl_3)

^{13}C NMR
(125 MHz, CDCl_3)

^1H NMR
(500 MHz, CDCl_3)

^{13}C NMR
(125 MHz, CDCl_3)

^1H NMR
(500 MHz, CDCl_3)

^{13}C NMR
(125 MHz, CDCl_3)

^1H NMR
(500 MHz, CDCl_3)

^{13}C NMR
(125 MHz, CDCl_3)

^1H NMR
(500 MHz, pyridine- d_5)

^{13}C NMR
(125 MHz, pyridine- d_5)