SUPPORTING INFORMATION

First Total Synthesis and Stereochemical Definition of Isodomoic Acid G

Yike Ni, Kande K. D. Amarasinghe, Bashar Ksebati, and John
Montgomery*

Department of Chemistry, Wayne State University, Detroit, MI 48202-3489

jwm@chem.wayne.edu

Unless otherwise noted, reagents were commercially available and were used without purification. Tetrahydrofuran (THF) was freshly distilled from sodium/benzophenone ketyl. Dichloromethane was distilled from calcium hydride. Zinc chloride was dried at 150 °C at 0.1 mmHg overnight, thoroughly ground by mortar and pestle in an inert atmosphere glovebox, and then dried again overnight at 150 °C at 0.1 mm Hg. Ni(COD)₂ and anhydrous ZnCl₂ were stored and weighed in an inert atmosphere glovebox. All reactions were conducted in flame-dried glassware under a nitrogen or argon atmosphere.

(R)-2-Oxo-oxazolidine-4-carboxylic acid methyl ester. To a suspension of D-serine methyl ester (5.25 g, 33.6 mmol) in 80 mL THF was added a 40 mL THF solution of triphosgene (10 g, 33.6 mmol). The resulting mixture was refluxed for 4 h. Solvent was removed under reduced pressure. The residue was purified by flash chromatography on silica gel (hexane : ethyl acetate = 1 : 3) to afford the product (5 g, quantitative yield) as a

light oil. $[\alpha]_D^{23} + 18.6^\circ$ (c 2.3, CH_2Cl_2); ^1H NMR (500 MHz, CDCl_3) δ 6.22 (s, br, 1H), 4.61 (t, $J = 8.8$ Hz, 1H), 4.52 (dd, $J = 8.8, 4.0$ Hz, 1H), 4.43 (dd, $J = 8.8, 4.0$ Hz, 1H), 3.81 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 170.8, 157.0, 67.0, 54.0, 53.4; IR (film) 3305, 1751 cm^{-1} .

(R)-3-But-2-ynyl-2-oxo-oxazolidine-4-carboxylic acid methyl ester. To a solution of 2-oxo-oxazolidine-4-carboxylic acid methyl ester (145 mg, 1.0 mmol) in 3 mL THF was added slowly KHMDS (0.5 M in toluene, 2.2 mL, 1.1 mmol) at 0 °C. The resulting mixture was stirred at 0 °C for 30 min. Then a 1 mL THF solution of propyne-1-sulfonic acid methyl ester (296 mg, 2 mmol) was added and the resulting mixture was stirred at rt overnight. Extraction with 5% NaHCO_3 and chromatographic purification (hexane : ethyl acetate, 1 : 1) afforded the product (141 mg, 72 %) as a light oil. ^1H NMR (500 MHz, CDCl_3) δ 4.53 (m, 2H), 4.41 (dq, $J = 17.0, 2.5$ Hz, 1H), 4.32 (dd, $J = 8.0, 4.5$ Hz, 1H), 3.93 (dq, $J = 17.5, 2.5$ Hz, 1H), 3.81 (s, 3H), 1.80 (t, $J = 2.5$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 170.1, 157.2, 82.1, 71.9, 64.6, 56.0, 53.2, 34.0, 3.7; IR (film) 2298, 2241, 1735 cm^{-1} ; HRMS (EI) m/e calcd for $\text{C}_9\text{H}_{11}\text{NO}_4$ 197.0688, found 197.0684 (M^+).

(S)-3-But-2-ynyl-4-hydroxymethyl-oxazolidin-2-one (5). To a solution of substrate (1.40 g, 7.1 mmol) in 40 mL 95% ethyl alcohol was added NaBH₄ (322 mg, 8.52 mmol) portionwise at 0 °C. The resulting mixture was stirred at 0 °C for 1 h, then allowed to warm to rt for another 1 h. Extraction with saturated NH₄Cl and chromatographic purification (hexane : ethyl acetate, 1 : 5) afforded compound **5** (1.0 g, 90 %) as a pale oil. ¹H NMR (500 MHz, CDCl₃) δ 4.36 (t, *J* = 9.5 Hz, 1H), 4.25 (dd, *J* = 8.5, 6.5 Hz, 1H), 4.14 (dq, *J* = 17.5, 2.5 Hz, 1H), 3.97 (m, 2H), 3.90 (dd, *J* = 12.0, 3.5 Hz, 1H), 3.64 (dd, *J* = 12.0, 3.0 Hz, 1H), 2.80 (s, br, 1H), 1.80 (t, *J* = 2.5 Hz, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 158.6, 81.2, 72.9, 64.8, 60.6, 56.6, 32.9, 3.7; IR (film) 3412, 2296, 2223, 1717 cm⁻¹; HRMS (EI) *m/e* calcd for C₈H₁₁NO₃ 169.0739, found 169.0737 (M⁺).

3-But-2-ynyl-4(S)-[3-(4(R)-benzyl-2-oxo-oxazolidin-3-yl)-3-oxo-propenyl]-

oxazolidin-2-one (12). To a solution of (COCl)₂ (412 μL, 4.73 mmol) in 12 mL CH₂Cl₂ was added DMSO (0.67 mL, 9.47 mmol) dropwise at -78 °C. The resulting solution was stirred at -78 °C for 20 min, and a 4 mL CH₂Cl₂ solution of substrate **5** (400 mg, 2.36 mmol) was added. The resulting solution was stirred at -78 °C for 1 h, then Et₃N (2.0 mL, 14.2 mmol) was added dropwise and the mixture was warmed to -20 °C over 1.5 h. A 12 mL CH₂Cl₂ solution of **A** (1.98 g, 3.55 mmol) and DMAP (433 mg, 3.55 mmol) was

stirred at 0°C for 20 min and then added to the reaction mixture at –20 °C. The final solution was stirred at –20 °C for 5 min, then warmed to 0°C over 1.5 h. Extraction with pH 8 buffered NH₄Cl + NH₄OH and chromatographic purification (hexane : ethyl acetate, 1 : 1) afforded compound **12** (661 mg, 76 % over 2 steps) as an 18 : 1 mixture of trans + cis isomers (dr = 6.3 : 1) as a thick oil. Major isomer (S,R)-**12**: ¹H NMR (500 MHz, CDCl₃) δ 7.54 (d, *J* = 15.5 Hz, 1H), 7.35 – 7.20 (m, 5H), 6.94 (dd, *J* = 15.5, 8.5 Hz, 1H), 4.74 (m, 1H), 4.67 (q, *J* = 8.5 Hz, 1H), 4.51 (t, *J* = 8.5 Hz, 1H), 4.32 (dq, *J* = 18.0, 2.0 Hz, H), 4.26 – 4.19 (m, 2H), 4.04 (t, *J* = 8.0 Hz, 1H), 3.71 (dq, *J* = 18.0, 2.0 Hz, 1H), 3.34 (dd, *J* = 14.0, 3.0 Hz, 1H), 2.81 (dd, *J* = 14.0, 9.5 Hz, 1H), 1.82 (t, *J* = 2.0 Hz, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 163.8, 157.6, 153.5, 143.7, 135.2, 129.6, 129.3, 127.7, 125.5, 82.1, 71.9, 66.6, 66.5, 56.5, 55.4, 37.9, 33.2, 3.7; IR (film) 2359, 2342, 1766, 1684, 1645 cm^{–1}; HRMS (EI) *m/e* calcd for C₂₀H₂₀N₂O₅ 368.1372, found 368.1369 (M⁺);

(R)-4-Isopropyl-3-pent-4-ynoyl-oxazolidin-2-one (7). To a solution of (R)-4-isopropyl-2-oxazolidinone (129 mg, 1 mmol) in 5 mL THF was added n-BuLi (2.5 M in hexane, 0.48 mL, 1.2 mmol) dropwise at 0 °C. The resulting mixture was stirred at 0 °C for 0.5 h and a 3 mL THF solution of pent-4-ynoyl chloride (128 mg, 1.1 mmol) was added. The resulting mixture was stirred at 0 °C for 3 h. Extraction with saturated NH₄Cl and chromatographic purification (hexane : ethyl acetate, 3 : 1) afforded compound **7** (184 mg, 88 %) as a pale oil. ¹H NMR (500 MHz, CDCl₃) δ 4.45 (dt, *J* = 8.5, 3.5 Hz, 1H), 4.29

(t, $J = 8.5$ Hz, 1H), 4.22 (dd, $J = 9.5, 3.0$ Hz, 1H), 3.11 - 3.26 (m, 2H), 2.56 (td, $J = 7.5, 2.5$ Hz, 2H), 2.35 - 2.42 (m, 1H), 1.97 (t, $J = 2.5$ Hz, 1H), 0.91 (d, $J = 7.0$ Hz, 3H), 0.88 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 171.4, 154.3, 82.8, 69.2, 63.8, 58.7, 34.9, 28.6, 18.2, 14.9, 13.9; IR (film) 3268, 2113, 1776, 1690 cm^{-1} ; HRMS (EI) m/e calcd for $\text{C}_{11}\text{H}_{15}\text{NO}_3$ 209.1052, found 209.1049 (M^+).

(4R, 2'R)-4-Isopropyl-3-(2-methyl-pent-4-ynoyl)-oxazolidin-2-one. To a solution of NaHMDS (2.0 M in THF, 7.8 mL, 15.5 mmol) in 60 mL THF was added a 25 mL THF solution of **7** (2.95 g, 14.0 mmol) by syringe pump at -78 °C over 20 min. The resulting solution was stirred at -78 °C for 30 min, and MeI (4.4 mL, 70.0 mmol) was added dropwise. The final solution was stirred at -78 °C for 5 min and allowed to warm to -40 °C over 4 h. Extraction with saturated NH_4Cl and chromatographic purification (hexane : ethyl acetate, 3 : 1) afforded the product (2.0 g, 78 % based on recovered starting material) as a yellow solid (d.r. = 10 : 1). Recrystallization from hexane afforded white crystals (d.r. > 98 : 2). ^1H NMR (500 MHz, CDCl_3) δ 4.47 (dt, $J = 8.5, 3.5$ Hz, 1H), 4.28 (t, $J = 9.0$ Hz, 1H), 4.21 (dd, $J = 9.0, 2.5$ Hz, 1H), 3.94 (sextet, $J = 7.0$ Hz, 1H), 2.54 (ddd, $J = 16.5, 7.0, 2.5$ Hz, 1H), 2.31 - 2.39 (m, 2H), 1.97 (t, $J = 2.5$ Hz, 1H), 1.31 (d, $J = 6.5$ Hz, 3H), 0.91 (d, $J = 7.5$ Hz, 3H), 0.87 (d, $J = 6.5$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 175.5, 153.8, 82.0, 69.8, 63.7, 58.6, 37.8, 28.6, 22.2, 18.1, 17.7, 14.9; IR (film)

3284, 2119, 1773, 1698 cm^{-1} ; HRMS (EI) m/e calcd for $\text{C}_{12}\text{H}_{17}\text{NO}_3$ 223.1208, found 223.1210 (M^+).

(R)-Triisopropyl-(2-methyl-pent-4-ynyloxy)-silane (8). To a solution of substrate (758 mg, 3.4 mmol) in 40 mL dry ethyl ether was added 0.22 mL anhydrous ethyl alcohol and LiBH_4 (2.0 M in THF, 1.9 mL, 3.8 mmol) at 0 $^{\circ}\text{C}$. The resulting solution was stirred at 0 $^{\circ}\text{C}$ for 1.5 h. The reaction was quenched at 0 $^{\circ}\text{C}$ with 1 M NaOH and extracted with ethyl ether. The organic layers were combined and dried over MgSO_4 . Filtration and condensation gave the crude alcohol. To a solution of the crude alcohol, imidazole (324 mg, 4.5 mmol), and DMAP (41 mg, 0.34 mmol) in 40 mL CH_2Cl_2 was added TIPSCl (0.88 mL, 4.1 mmol) dropwise at rt. The resulting mixture was stirred at rt overnight. Extraction with pH 8 buffer and chromatographic purification (pure hexane) afforded compound **8** (700 mg, 81 % over 2 steps) as a colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 3.61 (dd, $J = 9.6, 4.8$ Hz, 1H), 3.55 (dd, $J = 10.0, 6.4$ Hz, 1H), 2.34 (ddd, $J = 16.4, 6.4, 2.4$ Hz, 1H), 2.14 (ddd, $J = 16.4, 7.2, 2.4$ Hz, 1H), 1.93 (t, $J = 2.4$ Hz, 1H), 1.85 (m, 1H), 1.06 (m, 21H), 0.99 (d, $J = 6.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 83.5, 69.2, 67.2, 35.7, 22.3, 18.2, 16.2, 12.2; IR (film) 3314, 2866, 2119, 1463, 1101 cm^{-1} ; HRMS (EI) m/e calcd for $\text{C}_{12}\text{H}_{23}\text{OSi}$ 211.1518, found 211.1519 ($\text{M}-\text{C}_3\text{H}_7$) $^+$.

7-[2-(4(R)-Benzyl-2-oxo-oxazolidin-3-yl)-2-oxo-ethyl]-6-[1,5(R)-dimethyl-6-(triisopropyl-silanyloxy)-hex-2-enylidene]-2(S),3(S)-tetrahydro-pyrrolo[1,2-*c*]oxazol-3-one (13). To a suspension of Cp_2ZrHCl (645 mg, 2.5 mmol) in THF (6 mL) was added a 2 mL THF solution of alkyne **8** (635 mg, 1.5 mmol) at rt. The resulting suspension was stirred at rt for 30 min, until bright yellow and homogeneous, and the solution was then cooled to 0 °C. A 1 mL THF solution of ZnCl_2 (28 mg, 0.2 mmol) was added slowly to the vinyl zirconium, and the resulting solution was stirred for 20 min. The mixture was then added quickly to a 1 mL THF solution of $\text{Ni}(\text{COD})_2$ (28 mg, 0.1 mmol), followed by the addition of substrate **12** (368 mg, 1.0 mmol) in 2 mL THF. The final solution became dark brown and was stirred at 0 °C for 2 h. Extraction with pH 8 buffer and chromatographic purification (hexane : ethyl acetate, 3 : 2) afforded compound **13** (440 mg, 70 %) as a *single diastereomer* as a pale oil. ^1H NMR (500 MHz, CDCl_3) δ 7.36 – 7.20 (m, 5H), 6.09 (d, $J = 15.5$ Hz, 1H), 5.77 (dt, $J = 15.0, 7.5$ Hz, 1H), 4.69 – 4.64 (m, 2H), 4.51 (dd, $J = 9.5, 4.5$ Hz, 1H), 4.46 (d, $J = 15.5$ Hz, 1H), 4.25 – 4.18 (m, 2H), 3.78 (dt, $J = 9.5, 5.0$ Hz, 1H), 3.71 (d, $J = 15.5$ Hz, 1H), 3.54 (dd, $J = 9.5, 6.0$ Hz, 1H), 3.49 (dd, $J = 9.5, 6.5$ Hz, 1H), 3.40 (dd, $J = 19.0, 2.8$ Hz, 1H), 3.31 (dd, $J = 13.5, 3.0$ Hz, 1H), 3.26 (m, 1H), 3.05 (dd, $J = 19.0, 11.5$ Hz, 1H), 2.79 (dd, $J = 13.5, 10.0$ Hz, 1H), 2.37 (dt, $J = 12.5, 6.5$ Hz, 1H), 1.97 (dt, $J = 14.0, 8.5$ Hz, 1H), 1.77 (s, 3H), 1.75 (m, 1H), 1.07 –

12.0 Hz, 1H), 2.29 (m, 1H), 1.94 (dt, $J = 14.0, 7.0$ Hz, 1H), 1.73 (s, 3H), 1.69 (m, 1H), 1.03 (m, 21H), 0.86 (d, $J = 6.5$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 172.9, 161.4, 134.4, 131.1, 129.2, 127.5, 70.3, 68.2, 65.9, 52.1, 49.9, 43.9, 39.0, 37.2, 36.7, 18.2, 16.7, 12.2; IR (film) 2865, 1759, 1736, 1220, 1092 cm^{-1} ; HRMS (EI) m/e calcd for $\text{C}_{23}\text{H}_{38}\text{NO}_5\text{Si}$ 436.2519, found 436.2524 ($\text{M}-\text{C}_3\text{H}_7$) $^+$.

(2S, 3S, 5'R)-4-(6-Hydroxy-1,5-dimethyl-hex-2-enylidene)-2-hydroxymethyl

-3-methoxycarbonylmethyl-pyrrolidine-1-carboxylic acid methyl ester (14). Sodium (420 mg, 18.2 mmol) was dissolved in 12 mL anhydrous methanol at 0 °C. After 10 min, a 4 mL methanol solution of substrate (109 mg, 0.22 mol) was added dropwise at 0 °C to the MeONa solution. The final solution was allowed to warm to rt over 4 h. Extraction with saturated NH_4Cl gave the crude product. To a solution of the crude product in 5 mL THF was added TBAF (1.0 M in THF, 0.45 mL, 0.45 mmol) at 0 °C, and the resulting mixture was allowed to warm to rt overnight. Extraction with pH 8 buffer and chromatographic purification (hexane : ethyl acetate, 1 : 1 first, then pure ethyl acetate) afforded compound **14** (63 mg, 81% over two steps) as a light oil. Two carbamate rotamers were evident in the NMR spectra at rt: ^1H NMR (500 MHz, CDCl_3) δ 6.23 (m, 1H), 5.69 (dt, $J = 15.0, 7.5$ Hz, 1H), 4.00 - 4.23 (m, 3H), 3.88 (t, $J = 6.0$ Hz, 0.5H), 3.66 -

3.72 (m, 6H), 3.58 (m, 2H), 3.38 - 3.49 (m, 3H), 3.28 (m, 0.5H), 2.40 (m, 2H), 2.24 (dt, J = 13.5, 7.0 Hz, 2H), 2.00 (m, 1H), 1.69 - 1.74 (m, 4H), 0.91 (d, J = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 172.9, 172.5, 156.9, 155.8, 134.1, 133.6, 129.7, 129.5, 129.4, 129.2, 127.6, 127.4, 67.8, 65.2, 65.1, 64.3, 64.0, 53.0, 52.9, 52.2, 52.1, 49.5, 48.9, 39.9, 39.2, 38.7, 38.5, 37.1, 36.3, 16.8, 16.7, 15.7, 15.6; IR (film) 3400, 1734, 1682, 1452, 1192, 1042 cm^{-1} ; HRMS (EI) m/e calcd for $\text{C}_{18}\text{H}_{29}\text{NO}_6$ 355.1995, found 355.1993 (M^+).

(2S, 3S, 5'R)-3-Carboxymethyl-4-(5-carboxy-1-methyl-hex-2-enylidene)-pyrrolidine-2-carboxylic acid (4) [5'R-Isodomoic acid G]. To a solution of Dess-Martin periodinane (72 mg, 0.169 mmol) in 2 mL CH_2Cl_2 was added a 1 mL CH_2Cl_2 solution of substrate **14** (15 mg, 0.042 mmol) at 0 °C. The resulting solution was allowed to warm to rt over 2 h. The reaction was diluted with ethyl ether and washed with saturated $\text{Na}_2\text{S}_2\text{O}_3$, saturated $\text{Na}_2\text{S}_2\text{O}_3$ / saturated NaHCO_3 (1 : 1) and brine, respectively, then dried over MgSO_4 . Filtration and condensation afforded the crude aldehyde. To a solution of the crude aldehyde in 1.6 mL *tert*-butanol was added 2-methyl-2-butene (1.6 mL, 15.5 mmol), water (0.8 mL, 40.0 mmol), and NaH_2PO_4 (23 mg, 0.168 mmol) at rt. After 5 min, NaClO_2 (23 mg, 0.252 mmol) was added, and the resulting mixture was stirred at rt for 6 h. The reaction mixture was quenched with methylene chloride / water (2 : 1) and

slightly acidified with 4 drop of trifluoroacetic acid, extracted with CH_2Cl_2 , washed with brine, and dried over MgSO_4 . Filtration and concentration afforded the crude acid. To a solution of the crude acid in 2.5 mL methanol was added 1.5 mL of a 38% aqueous NaOH solution at 0 °C. The resulting mixture was allowed to warm to rt over 18 h. The reaction mixture was diluted with water and extracted with methylene chloride to remove organic impurities. The aqueous phase was concentrated under reduced pressure, and the crude sample was dissolved in a minimum amount of water, loaded onto a strongly acidic (DOWEX 50WX8-200) ion-exchange column, washed with water until the eluant pH was neutral. Further elution with 1N NH_4OH afforded basic fractions which were concentrated to dryness. The residue was dissolved in a minimum of water and was loaded onto a weakly acidic (Amberlite CG-50) ion-exchange column. Elution with water afforded synthetic **4** as a white powder after lyophilization. $[\alpha]_D^{23} + 17^\circ$ (c 0.2, H_2O); mp 181-183 °C (decomp.); ^1H NMR (500 MHz, 4% CD_3COOD in D_2O) δ 6.35 (d, $J = 15.5$ Hz, 1H), 5.86 (dt, $J = 15.5, 7.5$ Hz, 1H), 4.30 (s, 1H), 4.21 (d, $J = 15.5$ Hz, 1H), 4.13 (d, $J = 15.5$ Hz, 1H), 3.8 (m, 1H), 2.72 (d, $J = 6.0$ Hz, 2H), 2.65 (q, $J = 7.0$ Hz, 1H), 2.44 (dt, $J = 14.0, 7.5$ Hz, 1H), 2.37 (dt, $J = 13.5, 7.0$ Hz, 1H), 1.75 (s, 3H), 1.16 (d, $J = 6.5$ Hz, 3H); ^{13}C NMR (125 MHz, D_2O) δ 185.9, 179.5, 173.7, 130.4, 130.1, 129.4, 128.9, 66.4, 47.7, 42.9, 41.8, 41.6, 37.6, 17.2, 15.1; ESI/MS m/e 311.88 ($\text{M}+1$) $^+$, 333.85 ($\text{M}+\text{Na}$) $^+$, 349.83 ($\text{M}+\text{K}$) $^+$.

***Note:** All experimental procedures and yields for the synthesis of 5'(S)-isodomoic acid G were analogous to that reported for 5'(R)-isodomoic acid G.

(2S, 3S, 5'S)-3-Carboxymethyl-4-(5-carboxy-1-methyl-hex-2-enylidene)-pyrrolidine-2-carboxylic acid (4) [5'S-Isodomoic acid G]. ^1H NMR (500 MHz, 4% CD_3COOD in

D₂O) δ 6.35 (d, $J = 16.0$ Hz, 1H), 5.87 (dt, $J = 15.5, 7.5$ Hz, 1H), 4.31 (s, 1H), 4.21 (d, $J = 16.0$ Hz, 1H), 4.13 (d, $J = 15.5$ Hz, 1H), 3.81 (m, 1H), 2.71 (d, $J = 7.0$ Hz, 2H), 2.65 (q, $J = 7.0$ Hz, 1H), 2.44 (dt, $J = 15.0, 7.2$ Hz, 1H), 2.37 (dt, $J = 14.0, 7.0$ Hz, 1H), 1.75 (s, 3H), 1.16 (d, $J = 7.0$ Hz, 3H); ¹³C NMR (125 MHz, D₂O) δ 186.2, 179.6, 173.8, 130.6, 130.2, 129.3, 128.9, 66.5, 47.7, 43.1, 41.9, 41.5, 37.6, 17.3, 15.1; ESI/MS m/e 311.92 (M+1)⁺, 333.88 (M+Na)⁺, 349.88 (M+K)⁺.

YN-I-253

Pulse Sequence: s2pul

Mercury 400 spectrometer

YN-I-99B

Pulse Sequence: zgpg30

Unity 500 spectrometer

YN-I-111

Pulse Sequence: s2pul

Unity 500 spectrometer

YN-III-75

Pulse Sequence: zgpg30

Unity 500 spectrometer

YN-III-132B

Pulse Sequence: s2pul

Unity 500 spectrometer

YN-I-291

Pulse Sequence: s2pul

Unity 500 spectrometer

YW-I-184

Pulse Sequence: zgpg30

Unity 500 spectrometer

12

M-1-149

Pulse Sequence: zgpg30

Unity 500 spectrometer

YN-I-187

Pulse Sequence: zgpg30

Unity 500 spectrometer

YN-II-45

Mercury 400 spectrometer

Pulse Sequence: s2pu1

YN-IT-43

Pulse Sequence: zgpg30

Unit: 500 spectrometer

YN-I-129

Pulse Sequence: zgpg30

Unity 500 spectrometer

YN-II-46

Mercury 400 spectrometer

Pulse Sequence: zgpg30

YN-III-169

Pulse Sequence: s2pul

Unity 500 spectrometer

YN-III-153

Pulse Sequence: zgpg30

Unity 500 spectrometer

YN-I-273

Pulse Sequence: s2pul

Mercury 400 spectrometer

YN-II-21

Pulse Sequence: s2pul

Unity 500 spectrometer

YN-I-281

Pulse Sequence: s2pul

Unity 500 spectrometer

XM-117-175-5RING-presat-normaltube-081803

Pulse Sequence: presat

Unity 500 spectrometer

5R-Isodomoic acid G-d2o+4%acetic acid-shigamitube

Pulse Sequence: presat

Unity 500 spectrometer

5'(R)-isodomoic acid G (4)
(in D₂O+4%CD₃COOD)

5S-Isodomoic acid G-d2c+4%acetic acid-shigamituge

Pulse Sequence: presat

Unity 500 spectrometer

5(S)-isodomoic acid G (4)
(in D₂O+4%CD₃COOD)

Natural Prod-Isodomonic acid G-d2o+4%acetic acid-shigemilube

Unity 500 spectrometer

Pulse Sequence: presat

Natural sample-isodomonic acid G
(in D₂O+4%CD₃COOD)

^1H NMR (500MHz, 4% CD_3COOD in D_2O)

Isodomoic Acid G
5S

Isodomoic Acid G
5R

Isodomoic Acid G
5S+Nat

Isodomoic Acid G
5R+Nat

Isodomoic Acid G
5R+5S

5.91 5.89 5.87 5.85 5.83 5.81 ppm

CD Spectra of Isodomoic Acid G 0.1~0.2 mg/mL, H₂O, 1 mm path