Supporting Information

Synthesis of C₃-Symmetric tris(β -hydroxy amide) ligands and Their Ti(IV) complexes catalyzed enantioselective alkynylation of aldehydes

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Experimental

General remark:

Melting points were measured on an XT-4 melting point apparatus and were uncorrected. ¹H NMR and ¹³C NMR spectra were recorded on Varian 200MHz, Varian 300MHz and Bruker 400MHz spectrometer. Infrared spectra were obtained on a Bruker Vector 22 spectrometer and Nicolet AVATAR 330 FT-IR spectrometer. Mass spectra were obtained on a VG-ZAB-HS (EI) mass spectrometer and APEX II FT-ICRMS (FAB) mass spectrometer. The optical rotations were measured on a Perkin–Elmer 341 LC spectrometer. The enantiomeric excesses of addition products were determined by HPLC analysis on HP 1100 using the Diacel Chiralcel OD column (eluting with hexane-2-propanol 90:10, flow rate 1.0 mL/min, UV detector at 254 nm)

Toluene, diethyl ether and tetrahydrofuran were distilled with sodium under nitrogen. Dichloromethane were distilled with P₂O₅. Other solvents used were purified and dried by standard procedures. Diethylzinc (1.0 M solution in hexane) was purchased from Acros.

General procedure for synthesis of tris(β -hydroxy amide) **4a-d**

A 25 mL flask was charged with 1,3,5-benzenetricarboxylic acid (0.63 g, 3 mmol), SOCl₂ (7.5 g, 63 mmol) and several drops of DMF. The mixture was heated at reflux for 6h forming a clear solution. Complete removal of excess SOCl₂ in vacuo and addition of CH₂Cl₂ (3.0 mL) provided a 1.0 M solution of 1,3,5-benzenetricarboxylic chloride. The solution was added slowly to the mixture of L-(+)- α -phenylglycinol (1.24 g, 9 mmol), Et₃N (4.56 g, 5 mL, 45 mmol) and CH₂Cl₂ (25 mL) at 0°C. The mixture was stirred at reflux for 4h. The reaction mixture was cooled to room temperature, washed with water (5 mL $\times$ 2). The organic layer was dried over anhydrous Na₂SO₄ and concentrated in vacuo to give crude product. Purification by recrystallization from petroleum ether and ethyl acetate to afford pure product **4a** (1.5 g).

4a: 88% yield. Colorless solid. m.p. 278-279 °C. $[\alpha]_D^{20} = +50^\circ$ (c = 0.04, C₂H₅OH); IR (KBr): ν 3300, 3062, 2934, 1643, 1538, 1456, 1295, 1037, 756, 701cm⁻¹; ¹H NMR (400 MHz,

DMSO): δ 89.06 (d, $J = 7.9$ Hz, 3H), 8.49 (s, 3H), 7.41-7.21 (m, 15H), 5.10 (dd, $J = 7.9$, 13.4Hz, 3H), 5.00 (t, $J = 5.4$ Hz, 3H), 3.78-3.64 (m, 6H); ^{13}C NMR (75 MHz, DMSO): δ 165.6, 141.2, 135.1, 129.0, 128.1, 127.1, 126.8, 64.4, 56.3; HRMS (FAB): m/z calcd for $\text{C}_{33}\text{H}_{34}\text{N}_3\text{O}_6$: 568.2442; Found: 568.2447 $[\text{M}+\text{H}^+]$.

4b: 95% yield. Colorless solid, m.p. 240-241 °C. $[\alpha]_{\text{D}}^{20} = -129^\circ$ ($c = 0.16$, $\text{C}_2\text{H}_5\text{OH}$); IR(KBr): ν 3314, 3064, 2931, 1649, 1538, 1452, 1293, 1038, 916, 745, 702 cm^{-1} ; ^1H NMR (400 MHz, DMSO): δ 8.43 (d, $J = 8.2$ Hz, 3H), 8.29 (s, 3H), 7.27-7.24 (m, 12H), 7.19-7.14 (m, 3H), 4.89 (t, $J = 5.4$ Hz, 3H), 4.23-4.17(m, 3H), 3.55-3.44 (m, 6H), 2.96 (d, $J = 5.4$, 13.6Hz, 3H), 2.82 (d, $J = 8.7$, 13.6Hz, 3H); ^{13}C NMR (75 MHz, DMSO): δ 165.5, 139.4, 135.1, 129.1, 128.6, 128.2, 126.0, 62.8, 53.5, 36.5; HRMS (FAB): m/z calcd for $\text{C}_{36}\text{H}_{40}\text{N}_3\text{O}_6$: 610.2911; Found: 610.2911 $[\text{M}+\text{H}^+]$.

4c: 96% yield. Colorless solid, m.p. 275-276 °C. $[\alpha]_{\text{D}}^{20} = -284.7^\circ$ ($c = 0.1$, CHCl_3); IR (KBr): ν 3417, 3032, 2935, 1650, 1520, 1451, 1303, 1057, 915, 702 cm^{-1} ; ^1H NMR (400 MHz, DMSO): δ 8.89 (d, $J = 9.0$ Hz, 3H), 7.99 (s, 3H), 7.44-7.42 (m, 12H), 7.31-7.18 (m, 18H), 5.48 (d, $J = 5.0$ Hz, 3H), 5.12 (t, $J = 8.8$ Hz, 3H), 4.91 (app t, $J = 5.8$, 8.5Hz, 3H); ^{13}C NMR (75 MHz, DMSO): δ 164.5, 143.6, 141.4, 134.7, 128.6, 128.5, 127.7, 127.6, 127.1, 126.8, 126.7, 74.5, 59.4; HRMS (FAB): m/z calcd for $\text{C}_{51}\text{H}_{46}\text{N}_3\text{O}_6$: 818.3200; Found: 818.3200 $[\text{M}+\text{H}^+]$.

4d: 94% yield. Colorless solid, m.p. 275-276 °C. $[\alpha]_{\text{D}}^{20} = +288.2^\circ$ ($c = 0.15$, CHCl_3); IR(KBr): ν 3417, 3032, 2935, 1650, 1520, 1451, 1303, 1057, 915, 702 cm^{-1} ; ^1H NMR (400 MHz, DMSO): δ 8.89 (d, $J = 9.0$ Hz, 3H), 7.99 (s, 3H), 7.44-7.42 (m, 12H), 7.31-7.18 (m, 18H), 5.48 (d, $J = 5.1$ Hz, 3H), 5.11 (t, $J = 8.8$ Hz, 3H), 4.90 (dd, $J = 5.1$, 8.3Hz, 3H); ^{13}C NMR (75 MHz, DMSO): δ 164.4, 143.5, 141.2, 134.7, 128.5, 128.4, 127.6, 127.5, 127.0, 126.8, 126.7, 74.4, 59.3. HRMS (FAB): m/z calcd for $\text{C}_{51}\text{H}_{46}\text{N}_3\text{O}_6$: 818.32003; Found: 818.3200 $[\text{M}+\text{H}^+]$.

Synthesis of bis(β -hydroxy amide) 5: According to the general procedure for synthesis of **4** using 1,3-benzenedicarboxylic acid to afford **5** as colorless solid in 97% yield. m.p. 237-239 °C. $[\alpha]_{\text{D}}^{20} = -119^\circ$ ($c = 0.14$, $\text{CHCl}_3/\text{C}_2\text{H}_5\text{OH}$ 2:1); IR(KBr): ν 3310, 3030, 2930, 1649, 1543, 1498, 1453, 1297, 1043, 756, 700 cm^{-1} ; ^1H NMR (400 MHz, DMSO): δ 8.65 (d, $J = 8.7$ Hz, 2H), 8.04 (s, 1H), 7.73 (d, $J = 7.3$ Hz, 2H), 7.49-7.43 (m, 9H), 7.32-7.21 (m, 12H), 5.33 (d, $J = 4.2$ Hz, 2H), 5.18 (t, $J = 8.4$ Hz, 2H), 5.00 (d, $J = 4.5$ Hz, 2H); ^{13}C NMR (100 MHz, DMSO): δ 164.9, 143.6, 141.3, 134.7, 129.6, 128.4, 128.0, 127.64, 127.61, 127.1, 126.9, 126.7, 126.4, 74.6, 59.2. MS (ESI): m/z 557.7 ($\text{M}+\text{H}$), 556.9 (M^+), 555.7 ($\text{M}-\text{H}$). Anal. Calcd. for $\text{C}_{36}\text{H}_{32}\text{N}_2\text{O}_4$: C, 77.68; H, 5.79; N, 5.03. Found: C, 77.22; H, 5.87; N, 4.99.

Synthesis of β -hydroxy amide 6: According to the general procedure for synthesis of **4** using benzoic acid to afford **6** as colorless solid in 86% yield. m.p. 252-254 °C. $[\alpha]_{\text{D}}^{20} = -107^\circ$ ($c = 0.13$, $\text{CHCl}_3/\text{C}_2\text{H}_5\text{OH}$ 2:1); IR(KBr): ν 3340, 3030, 2930, 1634, 1579, 1527, 1491, 1455, 1322, 1034, 756, 700 cm^{-1} ; ^1H NMR (400 MHz, DMSO): δ 8.67 (d, $J = 9.1$ Hz, 1H), 7.67 (d, $J = 7.3$ Hz, 2H), 7.50-7.19 (m, 13H), 5.50 (d, $J = 5.2$ Hz, 1H), 5.18 (t, $J = 8.8$ Hz, 1H), 4.97 (dd, $J = 5.3$, 8.4Hz, 1H); ^{13}C NMR (100 MHz, DMSO): δ 165.3, 143.7, 141.4, 134.7, 131.0, 128.4,

128.1, 127.64, 127.59, 127.2, 127.0, 126.9, 126.7, 74.6, 59.0. MS (ESI): m/z 318.0 (M+H). Anal. Calcd. for $C_{21}H_{19}NO_2$: C, 79.47; H, 6.03; N, 4.41. Found: C, 79.32; H, 6.07; N, 4.37.

General procedure for the asymmetric addition of alkyne to aldehyde

Under nitrogen, 1.0 M diethylzinc in hexane (1.0 mL, 1.0 mmol) was added to a 10 mL flask containing phenylacetylene (110 μ L, 1.0 mmol) in dichloromethane (1 mL) and stirred at reflux for 2.5h during which a white precipitate was generated. At the same time, titanium tetraisopropoxide (104 μ L, 0.35 mmol) was added to a solution of **4c** (39.8 mg, 0.2 mmol) and dichloromethane (2 mL) and stirred for 2.5h. The titanium catalyst was transferred to alkynylzinc solution via syringe at room temperature. After stirring for 1.5h, the aldehyde (0.25 mmol) in dichloromethane (1 mL) was added and the mixture was stirred for additional 6h. The reaction was quenched with saturated ammonium chloride solution (2 mL), extracted with dichloromethane (3×5 mL) and dried over anhydrous sodium sulfate. After removal of the solvent under reduced pressure, the residue was purified by column chromatography to afford the pure chiral propargylic alcohol.

1,3-Diphenyl-prop-2-yn-1-ol¹

84% yield. 86.5% ee determined by HPLC analysis (Daicel Chiralcel OD column, hexane: 2-propanol 90:10, 1.0 mL/min, 254 nm). Retention time: $t_{\text{major}} = 26.31$ and $t_{\text{minor}} = 13.89$ min. $[\alpha]_D^{20} = -8.83$ ($c = 0.6$, $CHCl_3$). 1H NMR (300 MHz, $CDCl_3$): δ 7.64-7.25 (m, 10H), 5.70 (d, $J = 4.5$ Hz, 1H), 2.33 (d, $J = 4.5$ Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 140.6, 131.7, 128.7, 128.6, 128.4, 128.3, 126.7, 122.4, 88.6, 86.7, 65.1.

1-(2-Methoxyphenyl)-3-phenyl-prop-2-yn-1-ol²

86% yield. 92.4% ee determined by HPLC analysis (Daicel Chiralcel OD column, hexane: 2-propanol 90: 10, 1.0 mL/min, 254 nm). Retention time: $t_{\text{major}} = 20.96$ and $t_{\text{minor}} = 17.31$ min. $[\alpha]_D^{20} = +9.83$ ($c = 0.6$, $CHCl_3$). 1H NMR (300 MHz, $CDCl_3$): δ 7.66-7.63 (m, 1H), 7.50-7.46 (m, 2H), 7.36-7.26 (m, 4H), 7.03-6.92 (m, 2H), 5.93 (d, $J = 6.3$ Hz, 1H), 3.92 (s, 3H), 3.06 (d, $J = 6.3$ Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 156.8, 129.7, 128.8, 128.6, 128.4, 128.2, 128.0, 122.7, 120.9, 110.9, 88.3, 86.1, 61.7, 55.6.

1-(4-Methoxyphenyl)-3-phenyl-prop-2-yn-1-ol

84% yield. 81.5% ee determined by HPLC analysis (Daicel Chiralcel OD column, hexane: 2-propanol 90: 10, 1.0 mL/min, 254 nm). Retention time: $t_{\text{major}} = 33.24$ and $t_{\text{minor}} = 15.17$ min. $[\alpha]_D^{20} = -7.67$ ($c = 0.6$, $CHCl_3$). 1H NMR (300 MHz, $CDCl_3$): δ 7.52-7.43 (m, 5H), 7.30-7.27 (m, 2H), 6.97-6.87 (m, 2H), 5.61(s, 1H), 3.77 (s, 3H), 2.81 (s, 1H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 159.5, 132.9, 131.6, 128.4, 128.2, 128.1, 122.4, 113.9, 89.0, 86.3, 64.5, 55.2.

1-(2-Methylphenyl)-3-phenyl-prop-2-yn-1-ol²

81% yield. 92% ee determined by HPLC analysis (Daicel Chiralcel OD column, hexane: 2-propanol 90:10, 1.0 mL/min, 254 nm). Retention time: $t_{\text{major}} = 23.98$ and $t_{\text{minor}} = 11.45$ min. $[\alpha]_D^{20} = +9.67$ ($c = 0.6$, $CHCl_3$). 1H NMR (300 MHz, $CDCl_3$): δ 7.75-7.70 (m, 1H), 7.49-7.42 (m, 2H), 7.39-7.17 (m, 6H), 5.83 (d, $J = 3.3$ Hz, 1H), 2.47 (s, 3H), 2.27 (d, $J = 3.3$ Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 138.3, 136.0, 131.7, 130.8, 128.52, 128.46, 128.3, 126.6, 126.2,

122.5, 88.5, 86.5, 62.9, 19.0.

1-(4-Methylphenyl)-3-phenyl-prop-2-yn-1-ol

78% yield. 90% ee determined by HPLC analysis (Daicel Chiralcel OD column, hexane: 2-propanol 90:10, 1.0 mL/min, 254 nm). Retention time: $t_{\text{major}} = 22.21$ and $t_{\text{minor}} = 10.80$ min. $[\alpha]_{\text{D}}^{20} = +4.02$ ($c = 0.6$, CHCl_3). ^1H NMR (200 MHz, CDCl_3): δ 7.52-7.18 (m, 9H), 5.64 (d, $J = 6.2\text{Hz}$, 1H), 2.36 (s, 3H), 2.34 (s, 1H). ^{13}C NMR (50 MHz, CDCl_3): δ 138.2, 137.7, 131.7, 129.3, 128.5, 128.2, 126.7, 122.4, 88.9, 86.4, 64.9, 21.1.

1-(2-Fluorophenyl)-3-phenyl-prop-2-yn-1-ol

83% yield. 87.4% ee determined by HPLC analysis (Daicel Chiralcel OD column, hexane: 2-propanol 90: 10, 1.0 mL/min, 254 nm). Retention time: $t_{\text{major}} = 14.76$ and $t_{\text{minor}} = 10.22$ min. $[\alpha]_{\text{D}}^{20} = +5.68$ ($c = 0.6$, CHCl_3). ^1H NMR (200 MHz, CDCl_3): δ 7.74-6.97 (m, 9H), 5.94 (s, 1H), 3.27 (s, 1H). ^{13}C NMR (50 MHz, CDCl_3): δ 160.0 (d, $J = 247\text{Hz}$), 131.6, 130.0 (d, $J = 8.4\text{Hz}$), 128.5, 128.3 (d, $J = 3.4$), 128.1, 127.7 (d, $J = 13.3$), 124.2, (d, $J = 3.8\text{Hz}$), 122.1, 115.4 (d, $J = 20.9$), 87.6, 86.3, 59.1 (d, $J = 5.3\text{Hz}$).

1-(2-Chlorophenyl)-3-phenyl-prop-2-yn-1-ol

79% yield. 87.4% ee determined by HPLC analysis (Daicel Chiralcel OD column, hexane: 2-propanol 90: 10, 1.0 mL/min, 254 nm). Retention time: $t_{\text{major}} = 13.15$ and $t_{\text{minor}} = 11.71$ min. $[\alpha]_{\text{D}}^{20} = +11.3$ ($c = 0.6$, CHCl_3). ^1H NMR (300 MHz, CDCl_3): δ 7.86-7.82 (m, 1H), 7.49-7.25 (m, 8H), 6.05 (d, $J = 4.8\text{Hz}$, 1H), 2.51 (d, $J = 4.8\text{Hz}$, 1H). ^{13}C NMR (75 MHz, CDCl_3): δ 140.6, 131.7, 131.6, 128.4, 128.3, 128.1, 127.2, 126.6, 125.9, 122.3, 88.9, 86.3, 64.7.

1-(4-Chlorophenyl)-3-phenyl-prop-2-yn-1-ol

83% yield. 84.3% ee determined by HPLC analysis (Daicel Chiralcel OD column, hexane: 2-propanol 90:10, 1.0 mL/min, 254 nm). Retention time: $t_{\text{major}} = 24.43$ and $t_{\text{minor}} = 13.66$ min. $[\alpha]_{\text{D}}^{20} = -8.67$ ($c = 0.6$, CHCl_3). ^1H NMR (300 MHz, CDCl_3): δ 7.48-7.15 (m, 9H), 5.59 (s, 1H), 1.99 (s, 1H). ^{13}C NMR (75 MHz, CDCl_3): δ 139.1, 134.0, 128.6, 128.3, 128.2, 128.0, 127.3, 122.1, 88.3, 86.7, 64.0.

1-(1-Naphthyl)-3-phenyl-prop-2-yn-1-ol³

72% yield. 90.3% ee determined by HPLC analysis (Daicel Chiralcel OD column, hexane: 2-propanol 90:10, 1.0 mL/min, 254 nm). Retention time: $t_{\text{major}} = 39.76$ and $t_{\text{minor}} = 18.93$ min. $[\alpha]_{\text{D}}^{20} = +26.2$ ($c = 0.6$, CHCl_3). ^1H NMR (200 MHz, CDCl_3): δ 8.36-8.30 (m, 1H), 7.90-7.79 (m, 3H), 7.58-7.20 (m, 8H), 6.29 (s, 1H), 2.65 (s, 1H). ^{13}C NMR (50 MHz, CDCl_3): δ 135.7, 134.0, 131.7, 130.6, 129.3, 128.7, 128.5, 128.2, 126.4, 125.8, 125.2, 124.6, 124.0, 122.5, 88.6, 87.2, 63.3.

1,5-Diphenyl-pent-1-en-4-yn-3-ol⁴

81% yield. 90.5% ee determined by HPLC analysis (Daicel Chiralcel OD column, hexane: 2-propanol 90: 10, 1.0 mL/min, 254 nm). Retention time: $t_{\text{major}} = 28.17$ and $t_{\text{minor}} = 10.95$ min. $[\alpha]_{\text{D}}^{20} = -9.17$ ($c = 0.6$, CHCl_3). ^1H NMR (200 MHz, CDCl_3): δ 7.50-7.24 (m, 10H), 6.84 (d, $J = 15.8\text{ Hz}$, 1H), 6.44-6.32 (dd, $J = 5.8, 15.8\text{ Hz}$, 1H), 5.28 (d, $J = 5.8\text{ Hz}$, 1H), 2.20 (s, 1H).

¹³C NMR (50 MHz, CDCl₃): δ 144.3, 132.0, 131.7, 131.5, 128.7, 128.6, 128.5, 128.4, 126.8, 126.4, 87.9, 86.4, 63.4.

1,5-Diphenyl-pent-1-yn-3-ol⁴

81% yield. 72% ee determined by HPLC analysis (Daicel Chiralcel OD column, hexane: 2-propanol 90: 10, 1.0 mL/min, 254 nm). Retention time: $t_{\text{major}} = 39.21$ and $t_{\text{minor}} = 17.85$ min. $[\alpha]_{\text{D}}^{20} = +48.0$ ($c = 0.6$, CHCl₃). ¹H NMR (300 MHz, CDCl₃): δ 7.43-7.15 (m, 10H), 4.57 (t, $J = 6.4$ Hz, 1H), 2.84 (t, $J = 7.6$ Hz, 2H), 2.49 (s, 1H), 2.16-2.06 (m, 2H). ¹³C NMR (75 MHz, CDCl₃): δ 141.2, 131.6, 128.42, 128.35, 128.33, 128.2, 125.9, 122.5, 89.8, 85.2, 62.1, 39.2, 31.4.

4-Methyl-1-phenyl-pent-1-yn-3-ol⁵

64% yield. 47.4% ee determined by HPLC analysis (Daicel Chiralcel OD column, hexane: 2-propanol 90: 10, 1.0 mL/min, 254 nm). Retention time: $t_{\text{major}} = 12.62$ and $t_{\text{minor}} = 6.83$ min. $[\alpha]_{\text{D}}^{20} = +1.52$ ($c = 0.6$, CHCl₃). ¹H NMR (300 MHz, CDCl₃): δ 7.43-7.41 (m, 2H), 7.30-7.24 (m, 3H), 4.39 (s, 1H), 2.23 (s, 1H), 2.00-1.94 (m, 1H), 1.06 (d, $J = 7.2$ Hz, 6H). ¹³C NMR (75 MHz, CDCl₃): δ 131.6, 128.24, 128.20, 122.7, 88.9, 85.5, 68.3, 34.6, 18.1, 17.5.

1-Phenyl-3-(4-methylphenyl)-prop-2-yn-1-ol

79% yield. 82% ee determined by HPLC analysis (Daicel Chiralcel OD column, hexane: 2-propanol 90:10, 1.0 mL/min, 254 nm). Retention time: $t_{\text{major}} = 21.07$ and $t_{\text{minor}} = 15.64$ min. $[\alpha]_{\text{D}}^{20} = +4.1$ ($c = 1.8$, CHCl₃). ¹H NMR (300 MHz, CDCl₃): δ 7.63-7.60 (m, 2H), 7.43-7.33 (m, 5H), 7.13-7.10 (m, 2H), 5.68 (d, $J = 6.0$ Hz, 1H), 2.35 (s, 3H), 2.33 (d, $J = 4.5$ Hz, 1H). ¹³C NMR (75MHz, CDCl₃): δ 140.7, 138.7, 131.6, 129.0, 128.6, 128.4, 126.7, 119.3, 87.9, 86.8, 65.1, 21.5. MS (EI): m/z 222 (M^+ , 100), 207 (90), 119(64), 143 (48), 115 (100), 105 (46), 77(75). HRMS (EI): m/z calcd for C₁₆H₁₄O: 222.10447; Found: 222.10476.

1-Phenyl-3-(4-butylphenyl)-prop-2-yn-1-ol

77% yield. 85% ee determined by HPLC analysis (Daicel Chiralcel OD column, hexane: 2-propanol 90:10, 1.0 mL/min, 254 nm). Retention time: $t_{\text{major}} = 23.05$ and $t_{\text{minor}} = 14.30$ min. $[\alpha]_{\text{D}}^{20} = +5.7$ ($c = 0.89$, CHCl₃). ¹H NMR (300 MHz, CDCl₃): δ 7.63-7.61 (m, 2H), 7.43-7.31 (m, 5H), 7.12 (d, $J = 7.8$ Hz, 2H), 5.68 (d, $J = 5.7$ Hz, 1H), 2.60 (t, $J = 7.6$ Hz, 2H), 2.34 (d, $J = 6.0$ Hz, 1H), 1.63-1.52 (m, 2H), 1.40-1.25 (m, 2H), 0.92 (t, $J = 7.4$ Hz, 3H). ¹³C NMR (75 MHz, CDCl₃): δ 143.7, 140.7, 131.6, 128.6, 128.40, 128.37, 126.7, 119.5, 87.9, 86.9, 65.1, 35.5, 33.3, 22.3, 13.9. MS (EI): m/z 264 (M^+ , 40), 221 (50), 207 (55), 179 (30), 115 (100), 105 (28), 91(35), 77(56). HRMS (EI): m/z calcd for C₁₉H₂₀O: 264.15142; Found: 264.15250.

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¹H NMR of 4a

Current Data Parameters
NAME H21024
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20031121
Time 14:36
INSTRUM ARX400
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT DMSO
NS 41
DS 0
SWH 6666.667 Hz
FIDRES 0.203451 Hz
AQ 2.4576499 sec
RG 715
DW 75.000 usec
DE 93.75 usec
TE 300.0 K
D1 2.0000000 sec
P1 3.00 usec
DE 93.75 usec
SF01 400.1326000 MHz
NUCLEUS 1H

F2 - Processing parameters
SI 32768
SF 400.1300016 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 0.30

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 210.06825 Hz/cm

Pulse Sequence: s2put1

F2 - Acquisition Parameters

Date_ 20041111
Time 15.15
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DS 0
SWH 5319.149 Hz
FIDRES 0.162328 Hz
AQ 3.0802419 sec
RG 512
DW 94.000 usec
DE 117.50 usec
TE 300.0 K
D1 2.00000000 sec
P1 3.00 usec
DE 117.50 usec
SF01 400.1321149 MHz
NUCLEUS ¹H

F2 - Processing parameters

SI 16384
SF 400.1299994 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00

1D NMR plot parameters

CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 210.06825 Hz/cm

1.26032
1.19085
1.08888
0.87935
0.86118

2.70217
2.50831
2.00589

3.36802

8.90241
8.87992
7.99935
7.99364
7.65304
7.44062
7.42243
7.30735
7.28640
7.26496
7.24455
7.22635
7.20895
7.19432
7.17599
5.48151
5.47688
5.46897
5.14288
5.12064
5.09899
4.92435
4.90931
4.89477

F2 - Acquisition Parameters

Date_ 20041216
 Time 11.20
 INSTRUM ARX400
 PROBHD 5 mm Multinu
 PULPROG zgdc
 TD 32768
 SOLVENT DMSO
 NS 370
 DS 0
 SWH 29411.766 Hz
 FIDRES 0.897576 Hz
 AQ 0.5571060 sec
 RG 32768
 DW 17.000 usec
 DE 24.29 usec
 TE 300.0 K
 D12 0.0002000 sec
 DL5 24.00 dB
 CPDPRG waltz16
 P31 100.00 usec
 D1 2.00000000 sec
 P1 4.00 usec
 DE 24.29 usec
 SF01 100.6240000 MHz
 NUCLEUS 13C
 D11 0.03000000 sec

F2 - Processing parameters

SI 32768
 SF 100.6128204 MHz
 WDW EM
 SSB 0
 LB 0.50 Hz
 GB 0
 PC 0.30
 1D NMR plot parameters
 CX 20.00 cm
 F1P 220.000 ppm
 F1 22134.82 Hz
 F2P -5.000 ppm
 F2 -503.07 Hz
 PPMCM 11.25000 ppm/cm
 HZCM 1131.89429 Hz/cm