Facile Preparation of New Optically Pure 7,7'-disubstituted BINOLs and Their Application in Asymmetric Catalysis

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(Supplementary Materials)

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General procedure for the synthesis of 7,7'-dialkoxy-2,2'-dihydroxy-1,1'-dinaphthyl (7a-7f):

5 (121.8 mg, 0.3 mmol), CsOH•H₂O (101 mg, 0.6mmol) and alkyl bromide (0.6 mmol) were dissolved in DMF (50 mL). After the solution was stirred at 80 °C for 2 hours, water (100 ml) was added. The aqueous solution was extracted with ethyl acetate (100 mL), the combined organic layer was washed with brine and dried over anhydrous Na₂SO₄. After removal of solvent, the residue was dissolved in a mixture of chloroform (25 mL), 6N HCl (25 mL) and ethanol (10 mL), and then refluxed for 2 hours. The chloroform layer was separated, and the aqueous layer was extracted with chloroform (25 mL). The combined organic layer was concentrated and purified by column chromatography on silica gel to give **7a-7f**.

7a, R_f = 0.43 (ethyl acetate/petroleum = 1:3), yield: 91%, white solid, mp 136-137°C, [α]_D²⁰ = -146.7°, (c = 0.36, CHCl₃)(lit^{5j}: [α]_D²⁰ = -126.4°, (c = 1.0, CH₃OH). ¹HNMR (300 MHz, CDCl₃) δ (ppm) 3.58 (s, 6H) 5.08 (s, 2H), 6.49 (d, *J* = 2.4 Hz, 2H), 7.03 (dd, *J* = 9.0 Hz, 2.4 Hz, 2H), 7.22 (dd, *J* = 8.8 Hz, 2H), 7.78 (d, *J* = 9.0 Hz, 2H), 7.87 (d, *J* = 8.8 Hz, 2H); ¹³CNMR (75 MHz, CDCl₃) δ (ppm) 55.1, 103.1, 110.0, 115.1, 116.0, 124.8, 130.0, 131.1, 134.7, 153.3, 159.1; MS (EI) *m/z* 346 (M⁺); HRESI-MS(positive ion) C₂₂H₁₉O₄ ([M+H]⁺) requires 347.1283, found 347.1278. IR (KBr) ν 3444, 3062, 2937, 2835, 1621, 1513, 1463, 1221, 1032 cm⁻¹. Anal. Calca for C₂₂H₂₁₈O₄: C, 76.29; H, 5.24. Found: C, 76.56; H, 5.26.

7b, R_f = 0.58 (ethyl acetate/petroleum = 1:3), yield: 91%, white solid, mp 69.0-70.3°C; [α]_D²⁰ = -196.3°, (c = 0.108, CHCl₃); ¹HNMR (300 MHz, CDCl₃) δ (ppm) 1.27 (t, *J* = 7.0 Hz, 6H), 3.67-3.86 (m, 4H), 5.06 (brs, 2H), 6.48 (d, *J* = 1.9 Hz, 2H), 7.03 (dd, *J* = 8.9, 2.3 Hz, 2H), 7.20 (d, *J* = 8.8 Hz, 2H), 7.77 (d, *J* = 8.9 Hz, 2H), 7.87 (d, *J* = 8.8 Hz, 2H); ¹³CNMR (75 MHz, CDCl₃) δ (ppm) 14.5, 63.3, 103.9, 109.9, 115.0, 116.2, 124.7, 129.9, 131.1, 134.7, 153.2, 158.4; MS (EI) *m/z* 374 (M⁺); HRESI-MS(positive ion) C₂₄H₂₃O₄ ([M+H]⁺) requires 375.1596, found 375.1591. IR (KBr) ν 3427, 3061, 2977, 2931, 1620, 1513, 1216, 1042 cm⁻¹. Anal. Calca for C₂₄H₂₂O₄: C, 76.99; H, 5.92. Found: C, 76.80; H, 5.79.

7c, R_f = 0.29 (ethyl acetate/petroleum = 1:5), yield: 93%, colourless sticky oil, [α]_D²⁰ = -192.7°, (c = 0.178, CHCl₃); ¹HNMR (300MHz, CDCl₃) δ (ppm) 0.87 (t, *J* = 7.0 Hz, 6H), 1.29-1.41 (m, 4H), 1.57-1.66 (m, 4H), 3.65-3.77 (m, 4H), 5.04 (brs, 2H), 6.49 (d, *J* = 2.4 Hz, 2H), 7.03 (dd, *J* = 8.9, 2.4 Hz, 2H), 7.18 (d, *J* = 8.8 Hz, 2H), 7.76 (d, *J* = 8.9 Hz, 2H), 7.83 (d, *J* = 8.8 Hz, 2H); ¹³CNMR (75 MHz, CDCl₃) δ (ppm) 13.6, 19.0, 30.9, 67.3, 103.9, 110.0, 114.9, 116.2, 124.6, 129.8, 130.9, 134.7, 153.2, 158.5; MS (EI) *m/z* 430 (M⁺); HRESI-MS(positive ion) C₂₈H₃₁O₄ ([M+H]⁺) requires 431.2222, found 431.2217. IR (KBr) ν 3477, 3061, 2958, 2872, 1620, 1513, 1454, 1216, 1027 cm⁻¹. Anal. Calcd for C₂₈H₃₀O₄: C, 76.11; H, 7.02. Found: C, 76.30; H, 7.18.

7d, $R_f = 0.30$ (ethyl acetate/petroleum = 1:5), Yield: 92%, colourless sticky oil, $[\alpha]_D^{20} = -224.5^\circ$, ($c = 0.144$, CHCl_3); $^1\text{H NMR}$ (300 MHz, CDCl_3) δ (ppm) 0.86 (t, $J = 6.6$ Hz, 6H), 1.21-1.34 (m, 12H), 1.59-1.64 (m, 4H), 3.66-3.76 (m, 4H), 5.08 (brs, 2H), 6.48 (d, $J = 2.4$ Hz, 2H), 7.03 (dd, $J = 8.8$ Hz, 2.4 Hz, 2H), 7.19 (d, $J = 8.8$ Hz, 2H), 7.77 (d, $J = 8.8$ Hz, 2H), 7.85 (d, $J = 8.8$ Hz, 2H), $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ (ppm) 14.0, 22.5, 25.6, 28.9, 31.5, 67.8, 103.9, 110.0, 114.9, 116.3, 124.6, 129.8, 131.0, 134.7, 153.2, 158.5; MS (EI) m/z 486 (M^+); HRESI-MS(positive ion) $\text{C}_{32}\text{H}_{39}\text{O}_4$ ($[\text{M}+\text{H}]^+$) requires 487.2848, found 487.4823. IR (KBr) ν 3474, 3060, 2954, 2860, 1620, 1513, 1454, 1216, 1024 cm^{-1} . Anal. Calcd. for $\text{C}_{32}\text{H}_{38}\text{O}_4$: C, 78.98; H, 7.87. Found: C, 79.06; H, 7.80.

7e, $R_f = 0.30$ (ethyl acetate/petroleum = 1:10), yield: 85%, colourless sticky oil, $[\alpha]_D^{20} = -165.1^\circ$, ($c = 0.146$, CHCl_3); $^1\text{H NMR}$ (300 MHz, CDCl_3) δ (ppm) 0.91 (t, $J = 6.9$ Hz, 6H), 1.27-1.32 (m, 20H), 1.59-1.69 (m, 4H), 3.65-3.79 (m, 4H), 4.88 (brs, 2H), 6.50 (d, $J = 2.4$ Hz, 2H), 7.04 (dd, $J = 8.9$ Hz, 2.4 Hz, 2H), 7.17 (d, $J = 8.8$ Hz, 2H), 7.76 (d, $J = 8.9$ Hz, 2H), 7.82 (d, $J = 8.8$ Hz, 2H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ (ppm) 14.0, 22.6, 25.9, 28.9, 29.1, 29.3, 31.7, 67.7, 103.9, 110.1, 114.9, 116.2, 124.6, 129.8, 130.8, 134.7, 153.2, 158.5; MS (EI) m/z 542 (M^+); HRESI-MS(positive ion) $\text{C}_{36}\text{H}_{47}\text{O}_4$ ($[\text{M}+\text{H}]^+$) requires 543.3474, found 543.3469. IR (KBr) ν 3475, 3061, 2927, 2854, 1620, 1513, 1455, 1271, 1028 cm^{-1} . Anal. Calcd. for $\text{C}_{36}\text{H}_{46}\text{O}_4$: C, 79.67; H, 8.54. Found: C, 79.65; H, 8.50.

7f, $R_f = 0.24$ (ethyl acetate/petroleum = 1:10), yield $\square$ 85%, colourless sticky oil, $[\alpha]_D^{20} = -150.0^\circ$ ($c = 0.414$, CHCl_3). $^1\text{H NMR}$ (300 MHz, CDCl_3) δ (ppm) 0.88 (tr, $J = 6.9$ Hz, 6H), 1.23-1.30 (m, 36H), 1.59-1.64 (m, 4H), 3.65-3.76 (m, 4H), 5.05 (s, 2H), 6.48 (d, $J = 2.4$ Hz, 2H), 7.03 (dd, $J = 8.9$ Hz, 2.4 Hz, 2H), 7.21 (d, $J = 8.9$ Hz, 2H), 7.78 (d, $J = 8.9$ Hz, 2H), 7.87 (d, $J = 8.9$ Hz, 2H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ (ppm) 14.10, 22.67, 25.94, 28.95, 29.33, 29.50, 29.53, 29.61, 29.62, 31.90, 67.78, 103.91, 109.95, 114.93, 116.33, 124.65, 129.87, 131.03, 134.71, 153.22, 158.56; MS (EI) m/z 654 (M^+); HRESI-MS(positive ion) $\text{C}_{44}\text{H}_{63}\text{O}_4$ ($[\text{M}+\text{H}]^+$) requires 655.4726, found 655.4721. IR (KBr) ν 3529, 3061, 2924, 2853, 1621, 1513, 1456, 1271, 1028 cm^{-1} . Anal. Calcd. for $\text{C}_{44}\text{H}_{52}\text{O}_4$: C, 80.69; H, 9.54. Found: C, 80.41; H, 9.27.

General procedure for the asymmetric addition of phenylacetylene to aromatic aldehyde¹

Under nitrogen, phenylacetylene (1.1 mmol, 121 μL) and diethylzinc (1.0 mmol, 1.0 mL, 1M in hexane) were added to a flask (25 mL) containing toluene (2.0 mL). This solution was refluxed for 5 h and a white precipitate appeared. To the above reaction mixture was added 7,7'-disubstituted-BINOLs (>99% *ee*, 0.10 mmol) and CH_2Cl_2 (8 mL). After the mixture was stirred at room temperature for 15 min, $\text{Ti}(\text{O}^i\text{Pr})_4$ (0.25 mmol, 74 μL) was added. After the reaction solution was stirred for another hour,

aromatic aldehyde (0.5 mmol) was then added, and the reaction mixture was stirred for 4 h. The reaction was quenched with aqueous ammonium chloride. The organic layer was separated. The aqueous layer was extracted with ethyl acetate (15 mL×3). The combined organic layer was washed with brine and dried over anhydrous Na₂SO₄. After removal of the solvent, the crude product was purified by chromatography on silical gel (ethyl acetate: petroleum = 1:10) to give the optically active propargylic alcohols.

General procedure for the asymmetric addition of phenylacetylene to aliphatic aldehyde^{1b}

A flask (25 mL) was charged with solution of phenylacetylene (2.0 mmol, 220 µL) and diethyl zinc (2.0 mmol, 2 mL, 1 M in hexane) in toluene (1 mL). After refluxed for 1 hour under nitrogen, the solution was cooled down to room temperature, and then 7,7'-dialloxy-BINOL (>99% *ee*, 80 mg, 0.2 mmol), diethyl ether (8 mL) and Ti(O^{*i*}Pr)₄ (0.5 mmol, 150 µL) were added sequentially. The solution was stirred for another hour, and aliphatic aldehyde (0.5 mmol) was added. After stirred for additional 4 h, the reaction was quenched with saturated ammonium chloride, the resulting mixture was extracted with ethyl acetate (15 mL×3). The combined organic layer was washed with brine and dried over anhydrous Na₂SO₄. Purification of the crude product by column chromatography on silical gel (ethyl acetate: petroleum = 1:10) afforded the product.¹

1,3-Diphenyl-prop-2-yn-1-ol: 91% yield, 95% *ee* determined by HPLC analysis (Chiralcel OD column, 10% IPA in hexane, rate: 1 mL/min, 254 nm). Retention time: *t_R*(minor)= 12.20 and *t_R*(major)= 20.76 min. [α]_D²⁰ = -9.1° (c= 1.68, CHCl₃). ¹HNMR (300 MHz, CDCl₃) δ (ppm) 2.21 (d, *J*= 6.0 Hz, 1H), 5.63 (d, *J*= 6.0 Hz, 1H), 7.24-7.35 (m, 6H), 7.40-7.43 (m, 2H), 7.55 (m, 2H).

1-(4-Methylphenyl)-3-phenyl-prop-2-yn-1-ol: 71% yield, 92% *ee* determined by HPLC analysis (Chiralcel OD column, 10% IPA in hexane, rate: 1 mL/min, 254 nm). Retention time: *t_R*(minor)= 9.14 and *t_R*(major)= 18.71 min. [α]_D²⁰ = -3.6° (c= 1.17, CHCl₃). ¹HNMR(300 MHz, CDCl₃) δ (ppm) 2.26 (s, 1H), 2.29 (s, 3H), 5.58 (s, 1H), 7.12-7.18 (m, 2H), 7.23-7.26 (m, 3H), 7.38-7.44 (m, 4H).

1-(1-Naphthyl)-3-phenyl-prop-2-yn-1-ol: 83% yield, 94% *ee* determined by HPLC analysis (Chiralcel OD column, 15% IPA in hexane, rate: 1 mL/min, 254 nm). Retention time: *t_R*(minor)= 10.14 and *t_R*(major)= 18.68 min. [α]_D²⁰ = +24.1° (c = 2.56, CHCl₃). ¹HNMR (300 MHz, CDCl₃) δ (ppm) 2.40 (s, 1H), 6.28 (s, 1H), 7.23-7.26 (m, 3H), 7.40-7.44 (m, 5H), 7.51-7.84 (m, 3H), 8.31 (d, *J*= 8.4 Hz, 1H).

1-piperonyl-3-phenyl-prop-2-yn-1-ol: 87% yield, 92% *ee* determined by HPLC analysis (Chiralcel OD column, 15% IPA in hexane, rate: 1 mL/min, 254 nm). Retention time: *t_R*(minor)= 9.44 and *t_R*(major)= 20.07 min. [α]_D²⁰ = -33.1° (c = 1.42, CHCl₃). ¹HNMR (300 MHz, CDCl₃) δ (ppm) 2.30 (s,

1H), 5.59 (s, 1H), 5.98 (s, 2H), 6.82 (d, J = 8.0 Hz, 1H), 7.07-7.13 (m, 2H), 7.30-7.33 (m, 3H), 7.46-7.49 (m, 2H).

1-(4-chlorophenyl)-3-phenyl-prop-2-yn-1-ol: 79% yield, 93% *ee* determined by HPLC analysis (Chiralcel OD column, 10% IPA in hexane, rate: 1 mL/min, 254 nm). Retention time: t_R (minor) = 8.69 and t_R (major) = 28.84 min. $[\alpha]_D^{20}$ = -8.8° (c = 0.672, CHCl₃), ¹HNMR (300 MHz, CDCl₃) δ (ppm) 2.38 (d, J = 6.0 Hz, 1H), 5.67 (d, J = 6.0 Hz, 1H), 7.32-7.39 (m, 5H), 7.45-7.49 (m, 2H), 7.54-7.57 (m, 2H).

1,5-Diphenyl-pent-1-en-4-yne-3-ol: 69% yield, 93% *ee* determined by HPLC analysis (Chiralcel OD column, 20% IPA in hexane, rate: 1 mL/min, 254 nm). Retention time: t_R (minor) = 9.19 and t_R (major) = 21.27 min. $[\alpha]_D^{20}$ = -1.9° (c = 0.984, CHCl₃), ¹HNMR (300 MHz, CDCl₃) δ (ppm) 2.12 (s, 1H), 5.24 (s, 1H), 6.33 (dd, J = 16.0 Hz, 7.5 Hz, 1H), 6.79 (d, J = 16.0 Hz, 1H), 6.77-7.44 (m, 10H).

1-(3-Methyl-phenyl)-3-phenyl-prop-2-yn-1-ol: 80% yield, 92% *ee* determined by HPLC analysis (Chiralcel OD column, 10% IPA in hexane, rate: 1 mL/min, 254 nm). Retention time: t_R (minor) = 9.74 and t_R (major) = 23.59 min. $[\alpha]_D^{20}$ = -6.5° (c = 2.52, CHCl₃), ¹HNMR (300 MHz, CDCl₃) δ (ppm) 2.46 (s, 3H), 2.54 (s, 2H), 5.67 (s, 1H), 7.19 (m, 1H), 7.31-7.34 (m, 4H), 7.44 (m, 2H), 7.48-7.51 (m, 2H).

1-(4-Methoxy-phenyl)-3-phenyl-prop-2-yn-1-ol: 93% yield, 91% *ee* determined by HPLC analysis (Chiralcel OD column, 10% IPA in hexane, rate: 1 mL/min, 254 nm). Retention time: t_R (minor) = 13.54 and t_R (major) = 27.42 min. $[\alpha]_D^{20}$ = -6.2° (c = 1.45, CHCl₃), ¹HNMR (300 MHz, CDCl₃) δ (ppm) 2.23 (s, 1H), 3.76 (s, 3H), 5.71 (s, 1H), 6.84-6.87 (m, 2H), 7.24-7.27 (m, 3H), 7.39-7.42 (m, 2H), 7.46-7.49 (m, 2H).

4-Methyl-1-phenyl-pent-1-yn-ol. 64% yield, 92% *ee* determined by HPLC analysis (Chiralcel OD column, 10% IPA in hexane, rate: 1 mL/min, 254 nm). Retention time: t_R (minor) = 5.28 and t_R (major) = 9.52 min. $[\alpha]_D^{20}$ = +2.7° (c = 0.946, CHCl₃), ¹HNMR (300 MHz, CDCl₃) δ (ppm) 1.02 (tr, J = 6.6 Hz, 3H), 1.79 (s, 1H), 1.87-1.95 (m, 1H), 4.34 (m, 1H), 7.23-7.28 (m, 3H), 7.35-7.40 (m, 2H).

1-phenyl-hept-1-yn-3-ol. 73% yield, 93% *ee* determined by HPLC analysis (Chiralcel OD column, 20% IPA in hexane, rate: 1 mL/min, 254 nm). Retention time: t_R (minor) = 5.60 and t_R (major) = 14.37 min. $[\alpha]_D^{20}$ = +1.5° (c = 1.11, CHCl₃), ¹HNMR (300 MHz, CDCl₃) δ (ppm) 0.88 (tr, J = 7.2 Hz, 3H), 1.32-1.48 (m, 4H), 1.72-1.77 (m, 2H), 2.11 (br, 1H), 4.54 (tr, J = 6.6 Hz, 1H), 7.20-7.27 (m, 3H), 7.36-7.39 (m, 2H).

References:

- (a) Moore, D.; Pu, L. *Org. Lett.* **2002**, *4*, 1855. (b) Gao, G.; Moore, D.; Xie, R. G.; Pu, L. *Org. Lett.* **2002**, *4*, 4143. (c) Lu, G.; Li, X.; Chan, A. S. C. *Chem. Commun.* **2002**, 172. (d) Li, X.; Lu, G;

Kwok, W. H.; Chan, A. S. C. *J. Am. Chem. Soc.* **2002**, *124*, 12636.

¹HNMR for compound 3

Current Data Parameters
NAME 1567-167-117
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030223
Time 15.02
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 16
DS 0
SWH 5895.204 Hz
FIDRES 0.182259 Hz
AQ 2.7329611 sec
RG 64
DM 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.1361009 MHz

F2 - Processing parameters
SI 32768
SF 300.130123 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 10.00 cm
F1 10.000 ppm
F2 300.130 Hz
F3 -0.500 ppm
F4 -150.06 Hz
PPH4 0.52500 ppm/cm
HZCM 157.56825 Hz/cm

¹³CNMR for compound 3

15# 13C NMR bbl probe

Current Data Parameters
NAME 1307-13C-001
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030603
Time 9.04
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgpg30
TO 101830
SOLVENT CDCl3
NS 800
DS 2
SWH 20664.361 Hz
FIDRES 0.228315 Hz
AQ 1.7980751 sec
RG 10241.3
DM 23.856 uSAC
DE 6.00 uSAC
TE 300.2 K
D1 3.0000000 sec
d11 0.0300000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 11.30 uSAC
PL1 2.00 dB
SFO1 75.477954 MHz

===== CHANNEL f2 =====
CPDPRG2 MUlti2
NUC2 1H
PCPD2 80.00 uSAC
PL2 120.00 dB
PL12 27.00 dB
SFO2 300.135507 MHz

F2 - Processing parameters
SI 65536
SF 75.4677570 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.00

10 NMR PLOT parameters
CX 20.00 cm
F1P 107.200 MHz
F1 14154.61 Hz
F2P -10.344 MHz
F2 -780.64 Hz
PNUC1 9.89512 MHz/cm
HZCM 746.75282 Hz/Hz

HPLC for racemic **3**

HPLC for Optically Pure **3**

¹HNMR for compound 5

¹³CNMR for compound **5**

¹HNMR for compound **7a**

Current Data Parameters
NAME 1367-107-142
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 2003/31
Time 9.02
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT MeOD
NS 16
DS 0
SWH 5989.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 64
RW 83.400 usec
DE 5.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.1320092 MHz

F2 - Processing parameters
SI 32768
SF 300.1300111 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

10 MHz plot parameters
CX 20.00 cm
CY 30.00 cm
C1P 10.000 ppm
F1 3001.30 Hz
F2P 0.000 ppm
FZ 0.00 Hz
FREQM 0.50000 ppm/cm
HZCM 150.06500 Hz/cm

¹HNMR for compound **7b**

¹³CNMR for compound **7b**

Current Data Parameters
NAME 1567-101-50
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030220
Time 20.18
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
RG 1.024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 8192
DM 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00020000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 5.50 usec
PL1 -8.00 dB
SFO1 75.475653 MHz

===== CHANNEL f2 =====
CROSSP2 90.00 usec
NUC2 1H
P2 12.00 usec
PL2 -3.00 dB
PL12 17.20 dB
PL13 17.71 dB
SFO2 200.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.467525 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

10 MHz plot parameters
CX 20.00 cm
CY 12.00 cm
F1P 180.422 GHz
F1 13616.03 MHz
F2P -750.25 MHz
F2 9.53810 GHz/cm
PITCH 719.06396 Hz/cm

¹HNMR for compound 7c

Current Data Parameters
NAME 1507-107-115
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030730
Time 15.41
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 2620
TO 3.2768
SOLVENT H₂O
NS 28
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 64
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
***** CHANNEL f1 *****
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.1320082 MHz
F2 - Processing parameters
SI 32768
SF 300.130121 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
TD 65536
FID 15.00 usec
F1 10.000 usec
F2 3001.30 Hz
F3 0.652 usec
F4 193.08 Hz
PC 0.32620 sec/can
HDCN 159.81563 Hz/can

¹³CNMR for compound 7c

Current Data Parameters
NAME 1297-13C-001
EXPNO 51
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020602
Time 10:22
INSTRUM spect
PROBHD 5 mm Nalima
PULPROG zgpg30
TO 201830
SOLVENT DMSO
NS 900
DS 2
SWH 20964.361 Hz
FIDRES 0.276315 Hz
AQ 1.7960751 sec
RG 10321.3
DM 23.890 usec
DE 5.00 usec
TE 300.2 K
D1 3.0000000 sec
d11 0.0300000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 11.30 usec
PL1 2.00 dB
SF01 75.47564 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 140.00 dB
PL12 27.00 dB
SF02 300.135007 MHz

F2 - Processing parameters
SI 65536
SF 75.467570 MHz
WDW EN
SSB 0
LB 0.50 Hz
GB 0
PC 1.00

10 user parameters
CX 20.00 cm
FAP 105.315 GHz
F1 13985.36 MHz
F2 300.134 GHz
F3 -786.04 MHz
FREQN 9.76800 GHz/cm
WCH 730.30056 MHz/cm

¹HNMR for compound 7d

¹³CNMR for compound **7d**

¹HNMR for compound 7e

¹³CNMR for compound **7e**

Current Data Parameters
 Name: 13C-10f-128
 Date_: 20030730
 Time: 14.59
 Instrument: AV-300
 Probe: 5 mm QNP 1H/13
 PULPROG: zgpg30
 TO: 32.000
 SOLVENT: CDCl3
 NS: 64
 DS: 4
 SWH: 18812.363 Hz
 FIDRES: 0.20730 Hz
 AQ: 1.7400308 sec
 RG: 6192
 CW: 26.590 uWHz
 DE: 6.00 uWHz
 TE: 300.2 K
 O1: 2.0000000 sec
 O11: 0.0000000 sec
 O12: 0.0000000 sec

===== CHANNEL f1 =====
 NUC1: 13C
 P1: 5.50 uWHz
 PL1: -6.00 dB
 SFO1: 75.4763278 MHz

===== CHANNEL f2 =====
 CPDPRG2: zgpg30
 NUC2: 1H
 PCPD2: 80.00 uWHz
 PL2: -2.00 dB
 PL12: 17.70 dB
 PL13: 17.71 dB
 SFO2: 300.1342653 MHz

F2 - Processing parameters
 SI: 32768
 SF: 75.4677581 MHz
 WCN: EM
 SFO: 0
 LB: 1.00 Hz
 GB: 0
 PC: 1.40

1D NMR plot parameters
 CY: 16.80 cm
 FID: 241.188 cm
 F1: 10200.40 Hz
 F2: -8.375 cm
 F2: -632.02 Hz
 FREQM: 12.47713 MHz
 KCM: 541.82007 Hz/cm

¹HNMR for compound 7f

¹³CNMR for compound **7f**

¹HNMR for compound **8a**

¹³CNMR for compound **8a**

¹HNMR for compound **8b**

¹³CNMR for compound **8b**

¹HNMR for compound 8c

¹³CNMR for compound **8c**

Current Data Parameters
NAME 13c7-12-22
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20021111
Time 12:44
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 19694
DS 4
SWH 17505.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219008 sec
RG 3152
GB 0
HM 27.400 MHz
DE 6.500 MHz
TE 300.2 K
F0 100.626130 MHz
B1 2.00000000 sec
B11 0.00000000 sec
B12 0.00000000 sec
===== CHANNEL f1 =====
NUC1 13C
P1 0.50 USEC
PL1 -6.00 DB
SFO1 75.4702532 MHz
===== CHANNEL f2 =====
PROBHD spect
NUC2 1H
PCPDZ 90.00 USEC
PL2 -2.00 DB
PL12 17.70 DB
PL13 17.71 DB
SFO2 500.135065 MHz
F2 - Processing parameters
SI 32768
SF 75.468756 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
ID - NMR file parameters
CX 20.00 cm
CY 6.00 cm
FZP 160.492 cm
F1 136.05.03 Hz
F2 -10.1402 MHz
F3 120.918 MHz
PQNCN 8.55815 Hz
HCHN 719.85205 Hz/cm

¹HNMR for compound **9a**

¹³CNMR for compound **9a**

¹HNMR for compound **9b**

¹³CNMR for compound **9b**

¹HNMR for compound 11

Current Data Parameters
NAME 1507-107-153
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030730
Time 15.28
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 32768
SOLVENT H2O
NS 32
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.732911 sec
RG 64
DM 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
***** CHANNEL f1 *****
NUC1 1H
P1 3.00 usec
PL1 -2.00 dB
SFO1 300.132082 MHz
F2 - Processing parameters
SI 32768
SF 300.130121 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
10 web plot parameters
CX 20.00 cm
CY 15.00 cm
F1P 10.000 cm
F1 3001.30 Hz
F2P -0.650 ppm
F2 -180.08 Hz
P0PCHE 0.53250 ppm/cm
KICK 159.81853 Hz/cm

¹³CNMR for compound 11

Current Data Parameters
NAME 13C7-107-68
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030304
Time 21.06
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
F4DELTD 0
SOLVENT DMSO
NS 1221
DS 0
SWH 25675.735 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 4095
DM 22.050 usec
DE 5.00 usec
TE 300.2 K
D1 3.00000000 sec
d11 0.00000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 5.50 usec
PL1 -6.00 dB
SFO1 75.4750598 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P2 80.00 usec
PL2 -2.00 dB
PL12 17.70 dB
SFO2 300.135007 MHz

F2 - Processing parameters
SI 32768
SF 75.4677536 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.40

ID parameters
EX 20.00 cm
ET 12.00 cm
FID 220000000
F1 15502.90 Hz
F2 -1005.26 Hz
ORIGIN 12.00000000
H2O 905.61286 Hz/cm