Supporting Information

Asymmetric Multicomponent Reactions: Diastereoselective Synthesis of Substituted Pyrrolidines and Prolines

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1. Experimental Section for compounds **3a-5** (S2-S7)
2. References (S-7)
3. ¹H and ¹³C-NMR (S-8-

General Information

All melting points were recorded on a Thomas-Hoover melting point apparatus and are uncorrected. ¹H NMR and ¹³C NMR spectra were recorded on Varian Oxford 300, Bruker Avance DRX-500 spectrometers. IR spectra were recorded on a Matteson Genesis II FT-IR spectrometer. Optical rotations were recorded on a Perkin Elmer 341 polarimeter. Anhydrous solvents were obtained as follows: THF and diethyl ether by distillation from sodium and benzophenone; pyridine and dichloromethane from CaH₂. All other solvents were reagent grade. All moisture sensitive reactions were carried out in flame dried flask under nitrogen atmosphere. Column chromatography was performed with Whatman 240-400 mesh silica gel under low pressure of 3-5 psi. TLC was carried out with E. Merck silica gel 60-F-254 plates. The title α -*N*-tosyl imino ester was made from ethyl glyoxylate and *N*-toluenesulfonylisocyanate (Aldrich) by Weinreb's procedure¹. Compounds **6a-b**², **6c**³, **6d**⁴ were prepared according to the reported procedures.

(2R,3R,5S)-ethyl 3-((R)-1-hydroxybut-3-en-1-yl)-5-phenyl-1-tosylpyrrolidine-2-carboxylate (3a) : To a mixture of freshly distilled α -N-tosyl imino ester **1** (350 mg, 2.4 mmol) and (*R*)-2,3-dihydro-2-phenylfuran **2** (510 mg, 2 mmol) in dry CH₂Cl₂ (20 mL) at -78°C was added a solution of TiCl₄ (1 M in CH₂Cl₂, 2.4 mmol) and the resulting orange solution was stirred at -78°C for 1 h. Allyltrimethylsilane (686 mg, 6 mmol) was added and the mixture was allowed to warm to 23°C over 1 h. The reaction was then quenched with saturated aqueous NaHCO₃. The organic layer was separated and the aqueous layer was extracted with CH₂Cl₂ (3 x). The combined organic extracts were dried over anhydrous Na₂SO₄ and concentrated under reduced pressure. The residue was further purified by flash column chromatography on silica gel to afford pyrrolidine **3a** (623 mg, 70% yield) as a clear oil, R_f = 0.3 (hex : EtOAc = 3:1), $[\alpha]_D^{23}$ = -29.7 (*c* 2.3, CHCl₃); ¹H NMR (500 MHz, CDCl₃): δ 1.33 (t, *J* = 7.3 Hz, 3H), 1.87-1.93 (m, 2H), 2.04-2.09 (m, 1H), 2.16-2.27 (m, 2H), 2.33-2.38 (m, 4H), 3.61-3.63 (m, 1H), 4.27 (q, *J* = 7.3 Hz, 2H), 4.39 (d, *J* = 7 Hz, 1H), 4.89 (dd, *J* = 4.5, 8 Hz, 1H), 5.09-5.20 (m, 2H), 5.65-5.78 (m, 1H), 7.18-7.27 (m, 5H), 7.52 (d, *J* = 7.5 Hz, 2H), 7.62 (d, *J* = 8 Hz, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 14.2, 21.5, 35.0, 40.1, 47.4, 61.7, 63.6, 64.1, 68.3, 119.1, 126.8, 127.2, 127.8, 128.2, 129.4, 133.9, 134.9, 141.9, 143.7, 172.2; IR (film, NaCl) 3523, 2357, 1742 cm⁻¹; HRMS (ESI) [M+H]⁺ calcd for C₂₄H₃₀NO₅S 444.1845, found 444.1850.

Same procedure as above was used when allyltributylstannane was used as a nucleophile. Thus, α -N-tosyl imino ester **1** (80 mg, 0.31 mmol), (*R*)-2,3-dihydro-2-phenylfuran **2** (55 mg, 0.37 mmol), TiCl₄ (1 M in CH₂Cl₂, 0.38 mmol) and allyltributylstannane (312 mg, 0.94 mmol) afford pyrrolidine **3a** (92 mg, 68% yield).

(R)-1-((2R,3R,5S)-2-(hydroxymethyl)-5-phenyl-1-tosylpyrrolidin-3-yl)but-3-en-1-ol(4) : To a stirring solution of pyrrolidine **3a** (443 mg, 1 mmol) and calcium chloride (233 mg, 2.1 mmol) in dry ethanol : THF mixture (3 mL : 2 mL) at 0°C was added NaBH₄ (160 mg, 4.2 mmol) and the resulting solution was allowed to warm to 23°C over

a period of 3 h. After this period 10% aqueous citric acid solution (10 mL) was added to the reaction mixture and concentrated under vacuum. The aqueous layer was extracted with EtOAc (3 x). The combined organic extracts were dried over anhydrous Na₂SO₄ and concentrated under reduced pressure. The residue was further purified by flash column chromatography on silica gel to afford pyrrolidine **4** (371 mg, 93% yield) as a white solid, Mp = 139-142°C ; ¹H NMR (500 MHz, CDCl₃): δ 1.90-2.13 (m, 4H), 2.47 (s, 3H), 3.35 (quint, *J* = 4 Hz, 1H), 3.77-3.85 (m, 3H), 4.92 (dd, *J* = 4, 7.5 Hz, 1H), 5.07-5.15 (m, 2H), 5.63-5.66 (m, 1H), 7.27-7.30 (m, 1H), 7.36 (t, *J* = 8 Hz, 3H), 7.46 (d, *J* = 7.5 Hz, 2H), 7.80 (d, *J* = 8Hz, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 21.7, 32.8, 40.1, 46.2, 63.8, 64.7, 65.8, 68.6, 118.7, 126.2, 127.3, 127.9, 128.6, 129.9, 134.1, 134.2, 141.9, 144.1; IR (film, NaCl) 3530, 1349, 1190, 1020 cm⁻¹; HRMS (ESI) [M+H]⁺ calcd for C₂₂H₂₇NO₄S 401.1661, found 401.1670.

(2R,3R,5S)-ethyl 3-(hydroxymethyl)-5-phenyl-1-tosylpyrrolidine-2-carboxylate (3b) :

To a mixture of freshly distilled α-*N*-tosyl imino ester **1** (73 mg, 0.28 mmol) and (*R*)-2,3-dihydro-2-phenylfuran **2** (50 mg, 0.34 mmol) in dry CH₂Cl₂ (3 mL) at -78°C was added a solution of TiCl₄ (1 M in CH₂Cl₂, 0.31 mmol) and the resulting orange solution was stirred at -78°C for 1 h. Triethylsilane (101 mg, 0.87 mmol) was added and the mixture was allowed to warm to 23°C over 1 h. The reaction was then quenched with saturated aqueous NaHCO₃. The organic layer was separated and the aqueous layer was extracted with CH₂Cl₂ (3 x). The combined organic extracts were dried over anhydrous Na₂SO₄ and concentrated under reduced pressure. The residue was further purified by flash column chromatography on silica gel to afford pyrrolidine **3b** as a mixture of inseparable diastereomers in ratio of 90:10, (79 mg, 69% yield) as a clear oil, *R_f* = 0.23 (hex : EtOAc = 3:2), [α]_D²³ = -15.7 (*c* 2.0, CHCl₃); ¹H NMR (500 MHz, CDCl₃): δ 1.34 (t, *J* = 7.5 Hz, 3H, major), 1.34 (t, *J* = 7.5 Hz, 0.34 H, minor), 1.73(s, 1H), 1.77-1.82 (m, 0.23H, minor), 1.98-2.05 (m, 3H, major), 2.34 (s, 0.42H, minor), 2.38 (s, 3H, major), 2.50-2.56 (m, 1H, major), 2.61-2.67 (m, 0.13H, minor), 3.50-3.56 (m, 2H, major), 3.64-3.70 (m, 0.14H, minor), 3.75-3.80 (m, 0.13H, minor), 4.24-4.29 (m, 2H, major), 4.38 (d, *J* = 6 Hz, 1H, major), 4.59 (d, *J* = 6 Hz, 0.12H, minor), 4.86 (t, *J* = 6.5 Hz, 1H, major), 5.08 (t, *J* = 6 Hz, 0.12H, minor), 7.0-7.1 (m, 0.79 H, minor) 7.17-7.26 (m, 5H, major), 7.48 (d, *J* = 7 Hz, 2H, major), 7.59 (d, *J* = 8.5 Hz, 2H, major); ¹³C NMR (125 MHz, CDCl₃): δ 14.2 (major), 21.6 (major), 30.0 (minor) 37.8 (major), 37.8 (minor), 44.7 (major), 61.6 (minor), 61.7 (major), 62.5 (major), 63.6 (major), 64.0 (minor), 64.2 (major), 65.9 (minor), 126.9 (major), 127 (minor) 127.3 (major), 127.8 (major), 128.0 (minor), 128.2 (major), 128.9 (minor), 129.4 (major), 133.2 (major), 138.2 (minor), 139.2 (minor), 141.5 (major), 142.9

(minor), 143.7 (major), 172.3 (major), 173.0 (minor); IR (film, NaCl) 3527, 3363, 2912, 1737, 1591 cm^{-1} ; HRMS (ESI) $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{21}\text{H}_{25}\text{NO}_5\text{S}$ 404.1532, found 404.1545.

Same procedure as above was used when tributyltin hydride was used as a nucleophile. Thus, α -*N*-tosyl imino ester **1** (84 mg, 0.33 mmol), (*R*)-2,3-dihydro-2-phenylfuran **2** (58 mg, 0.39 mmol), TiCl_4 (1 M in CH_2Cl_2 , 0.39 mmol) and tributyltin hydride (287 mg, 0.99 mmol) afford pyrrolidine **3b** (71 mg, 53% yield) as a mixture of inseparable diastereomers in ratio of 85:15.

(2*R*,3*R*,5*S*)-ethyl 3-((*R*)-1-hydroxy-3-oxo-3-phenylpropyl)-5-phenyl-1-tosylpyrrolidine-2-carboxylate (3c**)** : To a mixture of freshly distilled α -*N*-tosyl imino ester **1** (230 mg, 0.9 mmol) and (*R*)-2,3-dihydro-2-phenylfuran **2** (300 mg, 1.2 mmol) in dry CH_2Cl_2 (10 mL) at -78°C was added a solution of TiCl_4 (1 M in CH_2Cl_2 , 1.2 mmol) and the resulting orange solution was stirred at -78°C for 1 h. After this period *tert*-butyl(1-phenylvinyl)oxy)silane (708 mg, 3 mmol) was added, which was followed by addition of TiCl_4 (1 M in CH_2Cl_2 , 3 mmol) and stirred at 78°C for 1 h. Then mixture was allowed to warm to 23°C over 1 h. The reaction was then quenched with saturated aqueous NaHCO_3 . The organic layer was separated and the aqueous layer was extracted with CH_2Cl_2 (3 x). The combined organic extracts were dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was further purified by flash column chromatography on silica gel to afford pyrrolidine **3c** (295 mg, 63% yield) as a clear oil, $R_f = 0.21$ (hex : EtOAc = 3:1), $[\alpha]_D^{23} = -14.5$ (c 1.2, CHCl_3); ^1H NMR (500 MHz, CDCl_3): δ 1.34 (t, $J = 7.5$ Hz, 3H), 1.66 (s, 1H), 1.94-1.98 (m, 1H), 2.32-2.37 (m, 4H), 2.41-2.45 (m, 1H), 2.92 (dd, $J = 10, 18$ Hz, 1H), 3.10 (dd, $J = 2, 18$ Hz, 1H), 3.42 (d, $J = 3$ Hz, 1H), 4.14 (m, 1H), 4.25-4.32 (m, 2H), 4.42 (d, $J = 7$ Hz, 1H), 4.92 (dd, $J = 4, 7.5$ Hz, 1H), 7.18-7.30 (m, 6H), 7.50-7.60 (m, 5H), 7.65 (d, $J = 8.0$ Hz, 2H), 7.89 (d, $J = 8.5$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3): δ 14.2, 21.5, 43.0, 47.3, 61.7, 63.6, 63.8, 65.6, 126.8, 127.0, 127.2, 127.8, 128.0, 128.1, 128.3, 128.8, 129.5, 133.9, 134.8, 136.3, 141.9, 143.8, 172.2, 200.1; IR (film, NaCl) 3519, 1740, 1671 cm^{-1} ; HRMS (ESI) $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{29}\text{H}_{31}\text{NO}_6\text{SNa}$ 544.1770, found 544.1769.

(2R,3R,5S)-ethyl 3-((R)-1-hydroxy-4,4-dimethyl-3-oxopentyl)-5-phenyl-1-tosylpyrrolidine-2-carboxylate (3d) : The procedure described for **3c** was used for **3d**. α -*N*-tosyl imino ester **1** (82 mg, 0.32 mmol), (*R*)-2,3-dihydro-2-phenylfuran **2** (56 mg, 0.38 mmol), TiCl_4 (1 M in CH_2Cl_2 , 0.38 + 1 mmol) and *tert*-butyl(3,3-dimethylbut-1-en-2-yloxy)dimethylsilane (205 mg, 0.96 mmol) afford pyrrolidine **3d** (134 mg, 83% yield) as a clear oil, R_f = 0.21 (hex : EtOAc = 3:1), $[\alpha]_D^{23}$ = -15.3 (*c* 1.2, CHCl_3); ^1H NMR (500 MHz, CDCl_3): δ 1.09 (s, 9H), 1.34 (t, J = 7.5 Hz, 3H), 1.86 (quint, J = 5 Hz, 1H), 2.23-2.36 (m, 2H), 2.37 (s, 3H), 2.44 (dd, J = 10, 18 Hz, 1H), 2.58 (d, J = 18 Hz, 1H), 3.30 (s, 1H), 3.98-4.01 (m, 1H), 4.23-4.32 (m, 2H), 4.37 (d, J = 7 Hz, 1H), 4.89 (dd, J = 4, 7.5 Hz, 1H), 7.20 (d, J = 7.5 Hz, 2H), 7.25-7.28 (m, 2H), 7.54 (d, J = 7.5 Hz, 2H), 7.62 (d, J = 8. Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 14.3, 21.7, 26.4, 35.3, 41.3, 44.5, 47.3, 61.8, 63.6, 63.9, 65.6, 126.9, 127.3, 127.2, 127.9, 128.3, 129.5, 135.0, 143.8, 172.3, 217.1; IR (film, NaCl) 3471, 3453, 1736, 1658 cm^{-1} ; m/z (ESI) 524.05 ($\text{M} + \text{Na}$) $^+$.

(2R,3R,5S)-ethyl 3-((S)-1-hydroxy-2,2-dimethyl-3-oxo-3-phenoxypropyl)-5-phenyl-1-tosylpyrrolidine-2-carboxylate (3e) : The procedure described for **3c** was used for **3e**. α -*N*-tosyl imino ester **1** (77 mg, 0.3 mmol), (*R*)-2,3-dihydro-2-phenylfuran **2** (53 mg, 0.36 mmol), TiCl_4 (1 M in CH_2Cl_2 , 0.36 + 0.96 mmol) and *tert*-butyldimethyl(2-methyl-1-phenoxyprop-1-enyloxy)silane (250 mg, 0.9 mmol) afforded pyrrolidine **3e** (138 mg, 76% yield) as a clear oil, R_f = 0.21 (hex : EtOAc = 3:1), $[\alpha]_D^{23}$ = -13.8 (*c* 1.2, CHCl_3); ^1H NMR (500 MHz, CDCl_3): δ 1.21 (s, 3H), 1.32 (s, 3H), 1.34 (t, J = 7 Hz, 3H), 1.77 (ddd, J = 2.5, 6.3, 12.3 Hz, 1H), 2.14-2.20 (m, 1H), 2.38 (s, 3H), 2.56-2.60 (m, 1H), 2.94 (s, 1H), 3.91 (s, 1H), 4.26-4.31 (m, 2H), 4.42 (d, J = 9 Hz, 1H), 4.93-4.95 (m, 1H), 6.86-6.88 (m, 2H), 7.21-7.37 (m, 9H), 7.55 (d, J = 7.5 Hz, 2H), 7.69 (d, J = 8.5 Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 14.1, 21.3, 21.5, 23.3, 29.6, 34.3, 43.8, 46.8, 61.6, 63.7, 65.6,

73.7, 121.1, 126.1, 126.4, 127.0, 127.8, 128.2, 129.4, 134.5, 141.9, 143.7, 150.1, 171.9, 176.0; *IR* (film, NaCl) 3508, 2843, 1736 cm^{-1} ; *m/z* (ESI) 588.14 ($\text{M} + \text{Na}$)⁺.

(2*R*,3*R*,5*S*)-ethyl 3-((*R*)-1-hydroxy-3-methoxy-3-oxopropyl)-5-phenyl-1-tosylpyrrolidine-2-carboxylate (3f) : The procedure described for **3c** was used for **3f**. α -*N*-tosyl imino ester **1** (82 mg, 0.32 mmol), (*R*)-2,3-dihydro-2-phenylfuran **2** (57 mg, 0.39 mmol), TiCl_4 (1 M in CH_2Cl_2 , 0.38 + 1 mmol) and *tert*-butyldimethyl(2-methyl-1-phenoxyprop-1-enyloxy)silane (250 mg, 0.9 mmol) afforded pyrrolidine **3f** (93 mg, 61% yield) as a clear oil, R_f = 0.27 (hex : EtOAc = 3:1), $[\alpha]_D^{23}$ = -15.9 (*c* 2.0, CHCl_3); ^1H NMR (500 MHz, CDCl_3): δ 1.34 (t, J = 7.0 Hz, 3H), 1.75 (s, 1H), 1.85-1.90 (m, 1H), 2.23-2.88 (m, 1H), 2.30-2.35 (m, 2H), 2.38-2.43 (m, 4H), 3.18 (s, 1H), 3.68 (s, 3H), 4.00 (s, 1H), 4.24-4.31 (m, 2H), 4.37 (d, J = 7 Hz, 1H), 4.90 (dd, J = 4.5, 7.5 Hz, 1H), 7.18-7.27 (m, 5H), 7.52 (d, J = 7.5 Hz, 2H), 7.62 (d, J = 8.5 Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3): δ 14.2, 21.5, 35.0, 39.3, 47.2, 52.0, 61.7, 63.5, 63.8, 65.8, 126.8, 127.2, 127.8, 128.2, 129.4, 134.9, 141.7, 143.7, 172.0, 172.9; *IR* (film, NaCl) 3507, 2919, 1731 cm^{-1} ; HRMS (ESI) $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{24}\text{H}_{29}\text{NO}_7$ 498.1557, found 498.1552 ($\text{M} + \text{H}$)⁺.

(2*R*,3*R*,5*S*)-3-((*S*)-1-hydroxybut-3-enyl)-5-phenylpyrrolidine-2-carboxylic acid (5):

To a stirring solution of **3a** (44 mg, 0.1 mmol) in THF (3 mL) was added 1N LiOH (2 mL, 2 mmol) the reaction mixture was stirred at room temperature for 1h. After this time the reaction mixture was acidified to pH = 2 with 1N HCl. The aqueous layer was extracted with ethyl acetate (3 x). The combined organic extracts were dried over anhydrous Na_2SO_4 and the solvent was removed in vacuum. The crude product was used for the next reaction without purification. Thus, to a flask equipped with a condenser were added the crude product from last step, sodium phosphate dibasic (78 mg, 0.5 mmol), methanol (2.5 mL) and 3% Na/Hg (1.5 g, 1.9 mmol). The mixture was refluxed

for one day, after this period sodium phosphate dibasic (78 mg, 0.5 mmol) and 3% Na/Hg (1.5 g, 1.9 mmol) were added. The mixture was refluxed for another day. Water (2 mL) was used to quench the reaction. Mercury was removed and washed with methanol. The solution was concentrated under vacuum and resulting solid was dissolved in 10 mL methanol and sodium phosphate dibasic was removed by filtration. The crude product was purified by column chromatography on silica gel (eluent: CHCl₃ : CH₃OH = 9 : 1 to 8 : 2) to afford the desired product **5** (19 mg, 72% yield, two steps) as a white solid. ¹H NMR(500 MHz, CD₃OD): δ 2.13-2.18 (m, 1H), 2.30-2.36 (m, 1H), 2.50 (ddd, *J* = 2.4, 7, 13.6 Hz, 1H) 2.70-2.72 (m, 1H), 3.95-3.99 (m, 1H), 4.02 (d, *J* = 4.6 Hz, 1H), 4.81-4.86 (m, 1H), 5.04-5.13 (m, 2H), 5.84-5.88 (m, 2H), 7.38-7.44 (m, 3H), 7.50 (d, *J* = 7.5 Hz, 2H); ¹³C NMR (125 MHz, CD₃OD), δ 31.2, 39.9, 48.2, 63.5, 64.7, 71.5, 116.4, 127.1, 128.8, 128.9, 134.6, 135.7, 173.0; IR (film, NaCl) 3390, 2925, 1631, 1454 cm⁻¹.

References

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2. Cazeau, P.; Duboudin, F.; Moulines, F.; Babot, O.; Dunogues. J. *Tetrahedron* **1987**, *43*, 2075-2088.
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4. Oisaki, K.; Suto, Y.; Kanai, M.; Shibasaki, M. *J. Amer. Chem. Soc.* **2003**, *125*, 5644-5645.

H1 standard parameters, BBO probe.

Current Data Parameters
 NAME Ssk-77
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
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 Time 16.12
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 NS 8
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 SWH 6009.615 Hz
 FIDRES 0.183399 Hz
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 RG 101.6
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 DE 6.00 use
 TE 293.4 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

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 PL1 -2.00 dB
 SFO1 500.6427535 MHz

F2 - Processing parameters
 SI 32768
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 WDW EM
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 LB 0.30 Hz
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 PC 1.00

C13 standard parameters, BBO probe.

C13 standard parameters, BBO probe.

Current Data Parameters
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EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
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Time 19.12
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PROBHD 5 mm BBO BB-1H
PULPROG zgpg
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SOLVENT CDCl3
NS 256
DS 2
SWH 30303.031 Hz
FIDRES 0.924775 Hz
AQ 0.5407385 sec
RG 3649.1
DW 16.500 use
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TE 292.3 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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NUC1 13C
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SFO1 125.8998755 MHz

==== CHANNEL f2 =====
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NUC2 1H
PCPD2 80.00 use
PL2 -2.00 dB
PL12 17.00 dB
PL13 20.00 dB
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F2 - Processing parameters
SI 16384
SF 125.8860280 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

H1 standard parameters, BBO probe.

Current Data Parameters
NAME 6SK-4
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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Time 20.04
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 128
DS 2
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7264309 sec
RG 128
DW 83.200 use
DE 6.00 use
TE 294.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
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PL1 -2.00 dB
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F2 - Processing parameters
SI 32768
SF 500.6400128 MHz
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LB 0.30 Hz
GB 0
PC 1.00

C13 standard parameters, BBO probe.

Current Data Parameters
NAME 6SK-4
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
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Time 21.23
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PROBHD 5 mm BBO BB-1H
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 1442
DS 2
SWH 30303.031 Hz
FIDRES 0.924775 Hz
AQ 0.5407385 sec
RG 3251
DW 16.500 use
DE 6.00 use
TE 294.6 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

==== CHANNEL f1 =====
NUC1 13C
P1 2.70 use
PL1 1.00 dB
SFO1 125.8998755 MHz

==== CHANNEL f2 =====
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NUC2 1H
PCPD2 80.00 use
PL2 -2.00 dB
PL12 17.00 dB
PL13 20.00 dB
SFO2 500.6422529 MHz

F2 - Processing parameters
SI 16384
SF 125.8860280 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

H1 standard parameters, BBO probe.

C13 standard parameters, BBO probe.

C13 standard parameters, BBO probe.

Current Data Parameters
NAME 6SK-4
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050627
Time 15.18
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PULPROG zgpg
TD 32768
SOLVENT CDCl3
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DS 2
SWH 30303.031 Hz
FIDRES 0.924775 Hz
AQ 0.5407385 sec
RG 3649.1
DW 16.500 use
DE 6.00 use
TE 294.4 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

==== CHANNEL f1 =====
NUC1 13C
P1 2.70 use
PL1 1.00 dB
SFO1 125.8998755 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 use
PL2 -2.00 dB
PL12 17.00 dB
PL13 20.00 dB
SFO2 500.6422529 MHz

F2 - Processing parameters
SI 16384
SF 125.8860280 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

5

Current Data Parameters
NAME CXU-E-59a3-5
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20060614
Time 1.14
INSTRUM spect
PROBHD 5 mm CPTXI 1H-
PULPROG zg30
TD 32768
SOLVENT MeOD
NS 111
DS 2
SWH 6510.400 Hz
FIDRES 0.198682 Hz
AQ 2.5167158 sec
RG 16
DW 76.800 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====

NUC1 1H
P1 8.00 usec
PL1 1.50 dB
SFO1 499.8925000 MHz

F2 - Processing parameters

SI 16384
SF 499.8900278 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
CY 10.00 cm
FIP 7.997 ppm
F1 3997.50 Hz
F2P 0.185 ppm
F2 92.40 Hz
PPMCM 0.39060 ppm/cm
HZCM 195.25494 Hz/cm

C13 standard parameters, cryoprobe (#02).

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Current Data Parameters
 NAME CXU-E-59a3-5
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060614
 Time 1.27
 INSTRUM spect
 PROBD 5 mm CPTXI 1H-
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 12281
 DS 8
 SMH 30303.031 Hz
 FIDRES 0.924775 Hz
 AQ 0.5407385 sec
 RG 16384
 DW 16.500 usec
 DE 6.00 usec
 TE 298.0 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 DELTA 1.8999998 sec
 MCREST 0.0000000 sec
 MCWRK 0.0150000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 15.00 usec
 PL1 0.00 dB
 SFO1 125.7112400 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCD2 100.00 usec
 PL2 1.50 dB
 PL12 23.44 dB
 PL13 26.10 dB
 SFO2 499.8920000 MHz

F2 - Processing parameters
 SI 16384
 SF 125.6974442 MHz
 WDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 80.00 cm
 F1P 184.169 ppm
 F1 23149.63 Hz
 F2P -4.350 ppm
 F2 -546.73 Hz
 PPMCM 9.42595 ppm/cm
 HZCM 1184.81799 Hz/cm