SEQUENTIAL Pd-CATALYZED ASYMMETRIC ALLENE DIBORATION/ α -AMINOALLYLATION

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Supplementary Material

General. Melting points were determined using a Mel-Temp II melting point apparatus and are uncorrected. ^1H NMR spectra were recorded on Bruker DRX 300 or 400 MHz spectrometers. Chemical shifts are reported in ppm from tetramethylsilane with the solvent resonance as the internal standard (CDCl_3 : 7.24 ppm). Data are reported as follows: chemical shift, integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, p = pentet, h = hextet, br = broad, m = multiplet), coupling constants (Hz) and assignment. ^{13}C NMR was recorded on a Bruker 400 MHz (100 MHz) spectrometer with complete proton decoupling. Chemical shifts are reported in ppm from tetramethylsilane with the solvent as the internal standard (CDCl_3 : 77.0 ppm). Low-resolution mass spectrometry was performed by the University of North Carolina, Department of Chemistry Mass Spectrometry Facility. Infrared (IR) spectra were obtained on a Nicolet 560 Magna-FTIR.

Liquid chromatography was performed using forced flow (flash chromatography) on silica gel (SiO_2 , 230 X 450 mesh) purchased from Sorbent Technologies. Thin layer chromatography was performed on 250 μm silica gel plates from EMD Chemicals Inc. Visualization was achieved using UV light, phosphomolybdic acid in ethanol, potassium permanganate in water, or cerium sulfate and ammonium molybdate in sulfuric acid, each followed by heating.

Analytical high performance liquid chromatography (HPLC) was performed on a Hewlett-Packard Series 1100 liquid chromatograph equipped with a UV detector and a Daicel Chiralcel OD-H column. Analytical supercritical fluid chromatography (SFC) was performed on a Berger Instruments supercritical chromatograph equipped with an Alcott autosampler and a Knauer UV detector.

All reactions were conducted in oven or flame dried glassware under an inert atmosphere of nitrogen or argon. Toluene was distilled over CaH_2 and degassed by freeze-pump-thaw cycles prior to use. $\text{Pd}_2(\text{dba})_3$ and tricyclohexylphosphine were purchased from Strem Chemical Company. Aldehydes were purchased from Aldrich Chemical Company and distilled prior to use. *N*-(trimethylsilyl)aldimines were synthesized via the known literature procedure (Colvin, E. W.; McGarry, D.; Nugent, M. J. *Tetrahedron* **1988**, *44*, 4157.) and distilled prior to use. Bis(pinacolato)diboron was purchased from BASF. [(*R,R*)-xylyl]TADDOL]PNMe₂ was synthesized according to the literature (Woodward, A. R.; Burks, H. E.; Chan, L., K., M.; Morken, J. P. *Org. Lett.* **2005**, ASAP.). Allenes were synthesized according to the literature (Vermeer, P.; Meijer, J.; Brandsma, L. *Recl. Trav. Chim. Pays-Bas* **1975**, *94*, 112.). Acetic anhydride was purchased from Fisher Chemical and distilled under N_2 . All other reagents were purchased from either Fisher or Aldrich Chemical Companies and used directly.

Representative procedure for the preparation of β -amidoketones from allenenes (Table 1).

Method A: An oven-dried 2-dram vial equipped with a magnetic stir-bar was charged with 9.9 mg (0.011 mmol) of tris(dibenzylideneacetone)dipalladium(0), 16.8 mg (0.0258 mmol) [(*R,R*)-xylyl]TADDOL]PNMe₂, and 0.86 mL of toluene in a dry-box under an argon atmosphere. The vial was capped and stirred for 45 minutes and then 131 mg (0.516 mmol) of bis(pinacolato)diboron was added followed by 50.0

mg (0.430 mmol) of phenyl allene. The vial was capped, taped with electrical tape, removed from the dry-box, and allowed to stir at ambient temperature for 14 h. After this time period, the vial was taken back into the dry-box, and 114 mg (0.645 mmol) of *N*-(trimethylsilyl)benzaldimine was weighed in. Next 28.7 μ L (0.710 mmol) of MeOH was added dropwise while stirring the reaction. The vial was capped, removed from the dry-box, and stirred at ambient temperature for 1 h. Next, 0.12 mL (1.3 mmol) of acetic anhydride was added, followed by 0.5 mL of CHCl_3 . The vial was purged with N_2 , sealed with a cap, and stirred for an additional 1 h. Volatiles were then removed under reduced pressure, and the resulting residue was diluted with 1.6 mL of THF and 1.3 mL of pH 7 buffer. To this solution was added 0.48 mL of 30% $\text{H}_2\text{O}_{2(\text{aq})}$, and the mixture was stirred for 13 h at ambient temperature under N_2 . The final mixture was then transferred to a separatory funnel with 25 mL of CH_2Cl_2 and 10 mL of pH 7 buffer. 2 mL of saturated $\text{Na}_2\text{S}_2\text{O}_3$ solution was added, and the organic layer was collected. The aqueous phase was extracted with CH_2Cl_2 (2 x 20 mL), and the combined organics were washed with 15 mL of brine, dried with anhydrous Na_2SO_4 , filtered, and concentrated using reduced pressure. Silica gel chromatography (hexanes/EtOAc) of the crude mixture afforded 80.7 mg (67 %) of (*R*)-*N*-(3-oxo-1,4-diphenylbutyl)acetamide as a cream-colored solid.

Method B: An oven-dried 2-dram vial equipped with a magnetic stir-bar was charged with 9.9 mg (0.011 mmol) of tris(dibenzylideneacetone)dipalladium(0), 16.8 mg (0.0258 mmol) [(*R,R*)-xylyl]TADDOL]P NMe_2 , and 0.86 mL of toluene in a dry-box under an argon atmosphere. The vial was capped and stirred for 45 minutes. After complexation was complete, 131 mg (0.516 mmol) of bis(pinacolato)diboron was added, followed by 50.0 mg (0.430 mmol) of phenyl allene. The vial was capped, taped with electrical tape, removed from the dry-box, and allowed to stir at ambient temperature for 14 h. After this time period, the mixture was taken up in a syringe and added to a solution of benzaldehyde and NH_4OAc in anhydrous MeOH with activated 4 Å molecular sieves that had been stirring under N_2 at room temperature for 2 h (see below for the preparation of this solution). The residue in the vial was then rinsed into the aldehyde solution using 0.5 mL of MeOH. This mixture was then stirred for 1 h. Next, 0.41 mL (4.3 mmol) of acetic anhydride was added by syringe, and the reaction was stirred for an additional 1 h. The crude reaction was then poured into 20 mL of Et_2O , and filtered over celite until a homogeneous solution was obtained. Volatiles were removed under reduced pressure, and the resulting mixture was then diluted with 1.2 mL of THF and 1.1 mL of pH 7 buffer. To this solution was added 0.37 mL of 30 % $\text{H}_2\text{O}_{2(\text{aq})}$, and it was then allowed to stir at ambient temperature under N_2 for 13 h. The final mixture was then transferred to a separatory funnel with 25 mL of CH_2Cl_2 and 10 mL of pH 7 buffer. 2 mL of saturated $\text{Na}_2\text{S}_2\text{O}_3$ solution was added, and the organic layer was collected. The aqueous phase was extracted with CH_2Cl_2 (2 x 20 mL), and the combined organics were washed with 15 mL of brine, dried with anhydrous Na_2SO_4 , filtered, and concentrated using reduced pressure.. Silica gel chromatography (hexanes/EtOAc) of the crude mixture afforded 74.5 mg (62 %) of (*R*)-*N*-(3-oxo-1,4-diphenylbutyl)acetamide as a cream-colored solid.

Preparation of the aldehyde solution: To a 5-mL RBF with a magnetic stir-bar and activated 4 Å molecular sieves, in a dry-box under an argon atmosphere, was added 249 mg (3.23 mmol) of NH_4OAc . Next, 0.81 mL of anhydrous MeOH was added, followed by 65.5 μ L (0.645 mmol) of benzaldehyde. The flask was capped with a septum, removed from the dry-box, and allowed to stir, under N_2 , for 2 h before addition of the crude diboration mixture.

Representative procedure for the preparation of vinylboronate ester intermediates (Scheme 1).

The diboration-allylation sequence was carried out as described in method A using phenyl allene and *N*-(trimethylsilyl)benzaldimine. After this was complete, 3.0 eq of the appropriate anhydride was added, followed by 0.5 mL of CHCl_3 . After stirring the mixture for 1 h at ambient temperature, volatiles were removed under reduced pressure, and the product was isolated from the crude mixture using silica gel chromatography.

Procedure for the protonation of the vinylboronate ester intermediate in Scheme 1.

To 85.2 mg (0.210 mmol) of (*R,Z*)-*N*-(1,4-diphenyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)but-3-enyl)propionamide, in a 20-mL scintillation vial with a magnetic stir-bar, was added 2.1 mL of propionic acid. The vial was purged with N₂, sealed with a polypropylene cap, taped with electrical tape, and heated at 140°C for 28 h. After this time period, the reaction was transferred to a separatory funnel with 20 mL of Et₂O and extracted with saturated aqueous NaHCO₃ (3 x 15 mL). The combined aqueous layers were then extracted with Et₂O (1 x 20 mL). The combined organics were washed with 10 mL of brine, dried over anhydrous Na₂SO₄, filtered, and concentrated using reduced pressure to afford a brown solid. Purification by silica gel chromatography (20:1 CH₂Cl₂:Et₂O) afforded 54.2 mg (92 %) of (*R,Z*)-*N*-(1,4-diphenylbut-3-enyl)propionamide as a white solid.

Procedure for the Suzuki cross-coupling reaction in Scheme 1.

To 66.9 mg (0.149 mmol) of (*R,Z*)-*tert*-butyl-1,4-diphenyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)but-3-enylcarbamate, in a 20-mL scintillation vial with a magnetic stir-bar, was weighed 6.1 mg (0.0075 mmol) of [1,1'-bis(diphenylphosphino)ferrocene]dichloropalladium(II) dichloromethane complex. The vial was capped with a septum and put under N₂. To the vial was added 1.06 mL of dioxane, followed by 25.1 μL (0.224 mmol) of iodobenzene, and lastly 0.11 mL of 4 M KOH_(aq). The septum was then quickly replaced with a polypropylene cap; the cap was taped on with electrical tape, and then the vial was heated at 80°C for 14 h. After this time period, the reaction was transferred to a separatory funnel with 20 mL of CH₂Cl₂ and 10 mL of H₂O. The organic layer was collected, and the aqueous layer was washed with CH₂Cl₂ (1 x 20 mL). The combined organics were washed with 15 mL of H₂O, 10 mL of brine, dried over anhydrous Na₂SO₄, filtered, and concentrated using reduced pressure. Purification by silica gel chromatography (9:1 hexanes:EtOAc) afforded 37.2 mg (63 %) of (*R,E*)-*tert*-butyl-1,3,4-triphenylbut-3-enylcarbamate as a waxy solid.

Procedure for the preparation of the compound shown in Figure 2.

The diboration to phenyl allene was performed as described in Method A, with tricyclohexylphosphine in place of the chiral ligand and at 0.16 M concentration of toluene relative to the allene. After 14h at room temperature, 1.0 eq of *N*-(trimethylsilyl)benzalimine was added in the dry-box, followed by 1.1 eq of MeOH. A white precipitate formed after stirring for 1h at ambient temperature, and after this time period, the reaction was poured into 15 mL of hexanes. The mixture was filtered using vacuum filtration, and an off-white solid was collected. Recrystallization from a mixture of hexanes and EtOAc afforded X-ray quality crystals.

Representative procedure for absolute stereochemistry determinations using X-ray analysis.

The same procedure described in Method A was employed using cyclohexyl allene and (1*E*, 2*E*)-*N*-(trimethylsilyl)hex-2-en-1-imine with one modification. After the allylation step was complete, 0.24 mL (1.2 mmol) of (*S*)-(+)-2-methylbutyric anhydride (>98% *ee* and commercially available from Aldrich Chemical Co.) was added in place of Ac₂O. After oxidation and the aqueous workup, silica gel column chromatography (30:1 DCM:Et₂O) afforded 81.7 mg (62 %) of (*S*)-*N*-((*R,E*)-1-cyclohexyl-2-oxonon-5-en-4-yl)-2-methylbutanamide as a white solid. Recrystallization using a mixture of hexanes and EtOAc afforded X-ray quality crystals.

(1E, 2E)-N-(trimethylsilyl)hex-2-en-1-imine. ^1H NMR: δ 8.48 (1H, d, $J = 8.4$ Hz, $\text{N}=\text{CH}$), 6.34 (1H, dt, $J = 16$ Hz, $J = 6.8$ Hz, $\text{CH}=\text{CH}=\text{N}$), 6.14 (1H, dd, $J = 16$ Hz, $J = 8.4$ Hz, $\text{CH}=\text{CH}=\text{N}$), 2.20 (2H, m, allylic CH_2), 1.47 (2H, h, $J = 7.6$ Hz, $\text{CH}_2\text{CH}_2\text{CH}_3$), 0.92 (3H, t, $J = 7.6$ Hz, CH_3), 0.15 (9H, s, $\text{Si}(\text{CH}_3)_3$).

(R)-N-(3-oxo-1,4-diphenylbutyl)acetamide. A cream-colored solid. mp 123-126°C. $R_f = 0.17$ (1:2 Hexanes:EtOAc); IR (KBr): 3312 (br, s), 3060 (br, m), 1953 (w), 1871 (w), 1716 (s), 1650 (s), 1534 (s), 1406 (s), 1367 (s) cm^{-1} ; ^1H NMR: δ 7.10-7.40 (8H, m, ArH), 7.04 (2H, d, $J = 6.4$ Hz, ArH), 6.63 (1H, br d, $J = 7.6$ Hz, NH), 5.37 (1H, q, $J = 7.6$ Hz, ArCHN), 3.61 (1H, d, $J = 16$ Hz, $\text{ArCH}_a\text{H}_b\text{C}(\text{O})$), 3.55 (1H, d, $J = 16$ Hz, $\text{ArCH}_a\text{H}_b\text{C}(\text{O})$), 3.08 (1H, dd, $J = 16$ Hz, $J = 6.0$ Hz, $\text{C}(\text{O})\text{CH}_a\text{H}_b\text{CN}$), 2.89 (1H, dd, $J = 16$ Hz, $J = 5.4$ Hz, $\text{C}(\text{O})\text{CH}_a\text{H}_b\text{CN}$), 1.93 (3H, s, $\text{NC}(\text{O})\text{CH}_3$); ^{13}C NMR: δ 207.1, 169.3, 140.6, 133.2, 129.4, 128.7, 128.6, 127.4, 127.1, 126.2, 50.7, 49.5, 46.4, 23.3. LRMS (APCI+) Calc'd for $\text{C}_{18}\text{H}_{19}\text{NO}_2$ ($\text{M} + \text{Na}$) $^+$: 304.1. Found ($\text{M} + \text{Na}$) $^+$: 304.3.

Proof of stereochemistry. Stereochemical ratios were determined in comparison with authentic racemic material prepared using Method A with tricyclohexylphosphine as the achiral ligand in the diboration step. Absolute stereochemistry was determined by X-ray analysis of (*S*)-2-methyl-*N*-((*R*)-3-oxo-1,4-diphenylbutyl)butanamide (characterization data shown below), which was prepared as described above using phenyl allene and *N*-(trimethylsilyl)benzalimine.

Chiral SFC (AD-H, Chiralpak, 150 psi, 50°C, flow = 5.0 mL/min, 5.0 % MeOH) analysis of β -amidoketone product:

(S)-2-methyl-N-((R)-3-oxo-1,4-diphenylbutyl)butanamide. A white solid. mp 115-118°C. $R_f = 0.19$ (2:1 hexanes:EtOAc); IR (KBr): 3328 (br, m), 3029 (br, m), 2928 (br, m), 1945 (w), 1871 (w), 1706 (s), 1639 (s), 1526 (s), 1491 (s), 1449 (s), 1238 (s), 1172 (s) cm^{-1} ; ^1H NMR: δ 7.20-7.32 (6H, m, ArH), 7.16 (2H, d, $J = 7.2$ Hz, ArH), 7.04 (2H, d, $J = 6.8$ Hz, ArH), 6.57 (1H, d, $J = 7.6$ Hz, NH), 5.40 (1H, m, ArCHN), 3.62 (1H, d, $J = 16$ Hz, ArCH_aH_bC(O)), 3.56 (1H, d, $J = 16$ Hz, ArCH_aH_bC(O)), 3.10 (1H, dd, $J = 16$ Hz, $J = 5.6$ Hz, C(O)CH_aH_bCN), 2.89 (1H, dd, $J = 16$ Hz, $J = 6.0$ Hz, C(O)CH_aH_bCN), 2.09 (1H, h, $J = 7.2$ Hz, NC(O)CH), 1.63 (1H, m, NC(O)CHCH_aH_bCH₃), 1.40 (1H, m, NC(O)CHCH_aH_bCH₃), 1.08 (3H, d, $J = 6.8$ Hz, CHCH₃), 0.86 (3H, t, $J = 7.6$ Hz, CH₂CH₃); ^{13}C NMR: δ 207.2, 175.7, 140.9, 133.3, 129.4, 128.7, 128.6, 127.3, 127.1, 126.1, 50.69, 49.13, 46.47, 43.07, 27.18, 17.30, 11.82. LRMS (ES⁺) Calc'd for C₂₁H₂₅NO₂ (M + Na)⁺: 346.2. Found (M + Na)⁺: 346.3.

(*R*)-*N*-(1-(furan-2-yl)-3-oxo-4-phenylbutyl)acetamide. A cream-colored solid. mp 125-127°C. R_f = 0.20 (4:1 CH₂Cl₂:Et₂O); IR (KBr): 3293 (br, s), 3064 (br, m), 2901 (m), 2803 (w), 1953 (w), 1712 (s), 1631 (s), 1542 (s), 1363 (s), 1289 (s) cm⁻¹; ¹H NMR: δ 7.20-7.45 (4H, m, ArH), 7.15 (2H, d, J = 9.6 Hz, ArH), 6.49 (1H, d, J = 11 Hz, NH), 6.28 (1H, m, ArH), 6.09 (1H, m, ArH), 5.49 (1H, m, ArCHN), 3.69 (2H, s, ArCH₂C(O)), 3.16 (1H, dd, J = 22 Hz, J = 6.4 Hz, C(O)CH_aH_bCN), 2.90 (1H, dd, J = 22 Hz, J = 8.0 Hz, C(O)CH_aH_bCN), 1.97 (3H, s, NC(O)CH₃); ¹³C NMR: δ 206.7, 169.2, 153.1, 141.7, 133.3, 129.4, 128.8, 127.2, 110.4, 106.2, 50.49, 43.98, 43.83, 23.26. LRMS (APCI+) Calc'd for C₁₆H₁₇NO₃ (M + Na)⁺: 294.3. Found (M + Na)⁺: 294.3.

Proof of stereochemistry. Stereochemical ratios were determined in comparison with authentic racemic material prepared using Method A with tricyclohexylphosphine as the achiral ligand in the diboration step. Absolute stereochemistry was assumed to be analogous to that obtained for (*R*)-*N*-(3-oxo-1,4-diphenylbutyl)acetamide.

Chiral SFC (AD-H, Chiralpak, 150 psi, 50°C, flow = 5.0 mL/min, 5.0 % MeOH) analysis of β -amidoketone product:

(*R,E*)-*N*-(2-oxo-1-phenylnon-5-en-4-yl)acetamide. An off-white solid. mp 63-67°C. $R_f = 0.23$ (4:1 $\text{CH}_2\text{Cl}_2:\text{Et}_2\text{O}$); IR (KBr): 3293 (br, s), 2963 (br, m), 1712 (s), 1642 (s), 1541 (s), 1413 (m), 1366 (m) cm^{-1} ; ^1H NMR: δ 7.23-7.45 (3H, m, ArH), 7.18 (2H, d, $J = 9.6$ Hz, ArH), 6.19 (1H, d, $J = 10$ Hz, NH), 5.47 (1H, dt, $J = 15$ Hz, $J = 6.4$ Hz, vinyl), 5.35 (1H, dd, $J = 15$ Hz, $J = 6.4$ Hz, vinyl), 4.73 (1H, m, $\text{CH}=\text{CHCHN}$), 3.69 (2H, s, $\text{ArCH}_2\text{C(O)}$), 2.77 (1H, dd, $J = 17$ Hz, $J = 4.8$ Hz, $\text{C(O)CH}_a\text{H}_b\text{CN}$), 2.69 (1H, dd, $J = 17$ Hz, $J = 5.2$ Hz, $\text{C(O)CH}_a\text{H}_b\text{CN}$), 1.75-2.15 (5H, m, allylic $\text{CH}_2 + \text{NC(O)CH}_3$), 1.29 (2H, h, $J = 7.4$ Hz, CH_2CH_3), 0.81 (3H, t, $J = 7.2$ Hz, CH_2CH_3); ^{13}C NMR: δ 207.4, 169.1, 133.4, 132.3, 129.37, 128.7, 128.4, 127.1, 50.54, 47.43, 45.88, 34.08, 23.34, 22.03, 13.51. LRMS (APCI+) Calc'd for $\text{C}_{17}\text{H}_{23}\text{NO}_2$ ($\text{M} + \text{Na}$) $^+$: 296.2. Found ($\text{M} + \text{Na}$) $^+$: 296.3.

Proof of stereochemistry. Stereochemical ratios were determined in comparison with authentic racemic material prepared using Method A with tricyclohexylphosphine as the achiral ligand in the diboration step. Absolute stereochemistry was assumed to be analogous to that obtained for (*R*)-*N*-(3-oxo-1,4-diphenylbutyl)acetamide.

Chiral SFC (AD-H, Chiralpak, 150 psi, 50°C, flow = 5.0 mL/min, 4.0 % MeOH) analysis of β -amidoketone product:

(R)-N-(3-oxo-1,6-diphenylhexyl)acetamide. A white solid. mp 111-113°C. R_f = 0.16 (1:2 Hexanes:EtOAc); IR (KBr): 3351 (br, s), 3025 (br, m), 2932 (br, m), 1949 (w), 1872 (w), 1712 (s), 1646 (s), 1530 (s), 1367 (s), 1281 (s) cm^{-1} ; ^1H NMR: δ 7.10-7.35 (8H, m, ArH), 7.07 (2H, d, J = 7.6 Hz, ArH), 6.61 (1H, d, J = 7.6 Hz, NH), 5.36 (1H, q, J = 6.0 Hz, ArCHN), 3.05 (1H, dd, J = 16 Hz, J = 5.2 Hz, C(O)CH_aH_bCN), 2.83 (1H, dd, J = 16 Hz, J = 5.6 Hz, C(O)CH_aH_bCN), 2.50 (2H, t, J = 7.2 Hz, ArCH₂), 2.37 (1H, dt, J = 17 Hz, J = 10 Hz, -CH₂CH_aH_bC(O)), 2.26 (1H, dt, J = 17 Hz, J = 7.2 Hz, -CH₂CH_aH_bC(O)), 2.23 (3H, s, NC(O)CH₃), 1.79 (2H, p, J = 7.2 Hz, ArCH₂CH₂); ^{13}C NMR: δ 209.6, 169.3, 141.3, 140.8, 128.6, 128.4, 128.3, 127.4, 126.3, 125.9, 49.54, 47.29, 42.57, 34.74, 24.66, 23.36. LRMS (APCI+) Calc'd for C₂₀H₂₃NO₂ (M + Na)⁺: 332.2. Found (M + Na)⁺: 332.4.

Proof of stereochemistry. Stereochemical ratios were determined in comparison with authentic racemic material prepared using Method A with tricyclohexylphosphine as the achiral ligand in the diboration step. Absolute stereochemistry was determined by comparison to authentic material prepared by the following sequence. First, the β -amido methyl ester, (*R*)-methyl 3-acetamido-3-phenyl-propanoate, was prepared using asymmetric hydrogenation as described in the literature (Zhou, Y. G.; Tang, W.; Wang, W., B.; Li, W.; Zhang, X. *J. Am. Chem. Soc.* **2002**, 124, 4952.). This compound was then converted to the Weinreb's amide using a procedure employed by Toyooka and Nemoto (Toyooka, N.; Okumura, M.; Nemoto, H. *J. Org. Chem.* **2002**, 67, 6078.). Lastly, the amide was converted to the ketone by the addition of (3-phenylpropyl)magnesium bromide using Weinreb's conditions (Nahm, S.; Weinreb, S., M. *Tetrahedron Lett.* **1981**, 22, 3815.).

Chiral SFC (AS-H, Chiralpak, 150 psi, 50°C, flow = 5.0 mL/min, 4.0 % MeOH) analysis of β -amidoketone product:

(*R*)-*N*-(1-(furan-2-yl)-3-oxo-6-phenylhexyl)acetamide. A cream-colored solid. mp 86-90°C. R_f = 0.22 (4:1 CH₂Cl₂:Et₂O); IR (KBr): 3304 (br, s), 3025 (br, m), 2932 (br, m), 1950 (w), 1872 (w), 1713 (s), 1646 (s), 1522 (s), 1405 (s), 1367 (s), 1297 (s) cm⁻¹; ¹H NMR: δ 7.05-7.30 (6H, m, ArH), 6.54 (1H, d, J = 8.4 Hz, NH), 6.27 (1H, m, ArH), 6.12 (1H, m ArH), 5.44 (1H, m ArCHN), 3.06 (1H, dd, J = 17 Hz, J = 4.8 Hz, C(O)CH_aH_bCN), 2.81 (1H, dd, J = 17 Hz, J = 6.0 Hz, C(O)CH_aH_bCN), 2.56 (2H, t, J = 7.6 Hz, ArCH₂), 2.38 (2H, m, -CH₂CH₂C(O)), 1.97 (3H, s, NC(O)CH₃), 1.85 (2H, p, J = 6.8 Hz, ArCH₂CH₂); ¹³C NMR: δ 209.0, 169.2, 153.3, 141.6, 141.3, 128.34, 128.30, 125.9, 110.4, 106.2, 44.78, 43.76, 42.24, 34.78, 24.76, 23.18. LRMS (APCI+) Calc'd for C₁₈H₂₁NO₃ (M + Na)⁺: 322.1. Found (M + Na)⁺: 322.3.

Proof of stereochemistry. Stereochemical ratios were determined in comparison with authentic racemic material prepared using Method A with tricyclohexylphosphine as the achiral ligand in the diboration step. Absolute stereochemistry was assumed to be analogous to that obtained for (*R*)-*N*-(3-oxo-1,6-diphenylhexyl)acetamide.

Chiral SFC (AD-H, Chiralpak, 150 psi, 50°C, flow = 5.0 mL/min, 5.0 % MeOH) analysis of β -amidoketone product:

(*R,E*)-*N*-(4-oxo-1-phenylundec-7-en-6-yl)acetamide. A white solid. mp 62-64°C. $R_f = 0.25$ (4:1 $\text{CH}_2\text{Cl}_2\text{:Et}_2\text{O}$); IR (KBr): 3293 (br, m), 2947 (br, m), 1708 (s), 1650 (s), 1530 (s), 1370 (s) cm^{-1} ; ^1H NMR: δ 7.10-7.30 (5H, m, ArH), 6.28 (1H, d, $J = 8.4$ Hz, NH), 5.54 (1H, dt, $J = 15$ Hz, $J = 6.8$ Hz, vinyl), 5.42 (1H, dd, $J = 15$ Hz, $J = 6.4$ Hz, vinyl), 4.69 (1H, m, $\text{CH}=\text{CHCHN}$); 2.71 (1H, dd, $J = 17$ Hz, $J = 4.8$ Hz, $\text{C(O)CH}_a\text{H}_b\text{CN}$), 2.62 (1H, dd, $J = 17$ Hz, $J = 5.6$ Hz, $\text{C(O)CH}_a\text{H}_b\text{CN}$), 2.58 (2H, t, $J = 7.6$ Hz, ArCH_2), 2.39 (2H, m, $-\text{CH}_2\text{CH}_2\text{C(O)}$), 1.80-2.0 (7H, m, allylic CH_2 + ArCH_2CH_2 + NC(O)CH_3), 1.32 (2H, h, $J = 7.2$ Hz, CH_2CH_3), 0.85 (3H, t, $J = 7.6$ Hz, CH_2CH_3); ^{13}C NMR: δ 210.2, 169.3, 141.5, 133.6, 128.7, 128.6, 128.5, 126.1, 47.78, 433.6, 128.7, 128.6, 128.5, 126.1, 47.78, 46.86, 42.73, 35.09, 34.32, 25.03, 23.62, 22.30, 13.74. LHRMS (APCI+) Calc'd for $\text{C}_{19}\text{H}_{27}\text{NO}_2$ ($\text{M} + \text{Na}$) $^+$: 324.2. Found ($\text{M} + \text{Na}$) $^+$: 324.4.

Proof of stereochemistry. Stereochemical ratios were determined in comparison with authentic racemic material prepared using Method A with tricyclohexylphosphine as the achiral ligand in the diboration step. Absolute stereochemistry was assumed to be analogous to that obtained for (*R*)-*N*-(3-oxo-1,6-diphenylhexyl)acetamide.

Chiral SFC (AD-H, Chiralpak, 150 psi, 50°C, flow = 4.0 mL/min, 4.0 % MeOH) analysis of β -amidoketone product:

Diboration-
allylation-oxidation
product

Racemic

Diboration-allylation-
oxidation product +
racemic coinjection

(R)-N-(4-cyclohexyl-3-oxo-1-phenylbutyl)acetamide. A white solid. mp 90-94°C. $R_f = 0.26$ (4:1 CH_2Cl_2 : Et_2O); IR (KBr): 3324 (br, s), 3064 (br, m), 2912 (br, s), 1949 (w), 1704 (s), 1642 (s), 1537 (s), 1413 (m), 1378 (m) cm^{-1} ; ^1H NMR: δ 7.15-7.40 (5H, m, ArH), 6.74 (1H, d, $J = 7.6$ Hz, NH), 5.36 (1H, m, CHN), 3.04 (1H, dd, $J = 16$ Hz, $J = 5.2$ Hz, $\text{C(O)CH}_a\text{H}_b\text{CN}$), 2.82 (1H, dd, $J = 16$ Hz, $J = 6.0$ Hz, $\text{C(O)CH}_a\text{H}_b\text{CN}$), 2.16 (2H, m, $\text{CyCH}_2\text{C(O)}$), 1.97 (3H, s, NC(O)CH_3), 1.42-1.75 (6H, m, C_6H_{11}), 1.0-1.35 (3H, m, C_6H_{11}), 0.70-0.90 (2H, m, C_6H_{11}); ^{13}C NMR: δ 209.9, 169.3, 140.9, 128.6, 127.4, 126.3, 51.28, 49.59, 47.63, 33.52, 33.04, 32.94, 26.04, 25.98, 23.37. LRMS (APCI+) Calc'd for $\text{C}_{18}\text{H}_{25}\text{NO}_2$ ($\text{M} + \text{Na}$) $^+$: 310.2. Found ($\text{M} + \text{Na}$) $^+$: 310.4.

Proof of stereochemistry. Stereochemical ratios were determined in comparison with authentic racemic material prepared using Method A with tricyclohexylphosphine as the achiral ligand in the diboration step. Absolute stereochemistry was determined by comparison to authentic material prepared by the following sequence. First, the β -amido methyl ester, (*R*)-methyl 3-acetamido-3-phenyl-propanoate, was prepared using asymmetric hydrogenation as described in the literature (Zhou, Y. G.; Tang, W.; Wang, W., B.; Li, W.; Zhang, X. *J. Am. Chem. Soc.* **2002**, 124, 4952.). This compound was then converted to the Weinreb's amide using a procedure employed by Toyooka and Nemoto (Toyooka, N.; Okumura, M.; Nemoto, H. *J. Org. Chem.* **2002**, 67, 6078.). Lastly, the amide was converted to the ketone by the addition of (cyclohexylmethyl)magnesium bromide using Weinreb's conditions (Nahm, S.; Weinreb, S., M. *Tetrahedron Lett.* **1981**, 22, 3815.).

Chiral SFC (AD-H, Chiralpak, 150 psi, 50°C, flow = 5.0 mL/min, 3.0 % MeOH) analysis of β -amidoketone product:

(R)-N-(4-cyclohexyl-1-(furan-2-yl)-3-oxobutyl)acetamide. A white solid. mp 118-120°C. R_f = 0.26 (4:1 CH₂Cl₂:Et₂O); IR (KBr): 3304 (br, s), 3064 (br, m), 2912 (br, s), 1693 (s), 1642 (s), 1537 (s), 1370 (s), 1285 (s) cm⁻¹; ¹H NMR: δ 7.25 (1H, s, ArH), 6.68 (1H, d, J = 8.4 Hz, NH), 6.27 (1H, dd, J = 4.0 Hz, J = 2.6 Hz, ArH), 6.12 (1H, d, J = 4.0 Hz, ArH), 5.44 (1H, m, CHN), 3.06 (1H, dd, J = 17 Hz, J = 4.8 Hz, C(O)CH_aH_bCN), 2.80 (1H, dd, J = 17 Hz, J = 6.0 Hz, C(O)CH_aH_bCN), 2.23 (2H, d, J = 6.9 Hz, CyCH₂C(O)), 1.96 (3H, s, NC(O)CH₃), 1.50-1.85 (6H, m, C₆H₁₁), 1.0-1.35 (3H, m, C₆H₁₁), 0.75-1.0 (2H, m, C₆H₁₁); ¹³C NMR: δ 209.3, 169.2, 153.4, 141.5, 110.4, 106.2, 50.95, 45.12, 43.83, 33.57, 33.02, 32.95, 26.04, 25.97, 23.22. LRMS (APCI+) Calc'd for C₁₆H₂₃NO₃ (M + Na)⁺: 300.2. Found (M + Na)⁺: 300.3.

Proof of stereochemistry. Stereochemical ratios were determined in comparison with authentic racemic material prepared using Method A with tricyclohexylphosphine as the achiral ligand in the diboration step. Absolute stereochemistry was assumed to be analogous to that obtained for (*R*)-*N*-(4-cyclohexyl-3-oxo-1-phenylbutyl)acetamide and (*R,E*)-*N*-(1-cyclohexyl-2-oxonon-5-en-4-yl)acetamide.

Chiral SFC (AS-H, Chiralpak, 150 psi, 50°C, flow = 3.0 mL/min, 3.0 % MeOH) analysis of β -amidoketone product:

(*R,E*)-*N*-(1-cyclohexyl-2-oxonon-5-en-4-yl)acetamide. A cream-colored solid. mp 61-63°C. R_f = 0.19 (4:1 CH_2Cl_2 : Et_2O); IR (KBr): 3324 (br, m), 2920 (br, m), 1704 (s), 1627 (s), 1546 (s), 1370 (s), 1293 (s) cm^{-1} ; ^1H NMR: δ 6.32 (1H, d, J = 8.4 Hz, NH), 5.53 (1H, dt, J = 16 Hz, J = 6.4 Hz, vinyl), 5.42 (1H, dd, J = 16 Hz, J = 6.4 Hz, vinyl), 4.68 (1H, m, $\text{CH}=\text{CHCHN}$), 2.72 (1H, dd, J = 17 Hz, J = 4.8 Hz, $\text{C}(\text{O})\text{CH}_a\text{H}_b\text{CN}$), 2.64 (1H, dd, J = 17 Hz, J = 5.2 Hz, $\text{C}(\text{O})\text{CH}_a\text{H}_b\text{CN}$), 2.23 (2H, d, J = 6.8 Hz, $\text{CyCH}_2\text{C}(\text{O})$), 1.85-1.97 (5H, m, allylic CH_2 + $\text{NC}(\text{O})\text{CH}_3$), 1.76 (1H, m, C_6H_{11}), 1.53-1.69 (5H, m, C_6H_{11}), 1.32 (2H, h, J = 7.2 Hz, CH_2CH_3) 1.02-1.26 (3H, m, C_6H_{11}), 0.82 (3H, t, J = 7.2 Hz, CH_2CH_3), 0.75-0.97 (2H, m, C_6H_{11}); ^{13}C NMR: δ 210.3, 169.1, 132.3, 128.6, 51.17, 47.63, 47.12, 34.14, 33.74, 33.15, 33.04, 26.08, 25.99, 23.45, 22.11, 13.56. LRMS (APCI+) Calc'd for $\text{C}_{17}\text{H}_{29}\text{NO}_2$ ($\text{M} + \text{Na}$) $^+$: 302.2. Found ($\text{M} + \text{Na}$) $^+$: 302.4.

Proof of stereochemistry. Stereochemical ratios were determined in comparison with authentic racemic material prepared using Method A with tricyclohexylphosphine as the achiral ligand in the diboration step. Absolute stereochemistry was determined by X-ray analysis of (*S*)-*N*-((*R*)-1-(furan-2-yl)-3-oxo-6-phenylhexyl)-2-methylbutanamide (characterization data shown below), which was prepared as described above.

Chiral HPLC (Chiralcel-OD-H, Daicel, 3.0 % iPrOH in hexanes, 0.9 mL/min) analysis of β -amidoketone product:

(S)-N-((R, E)-1-cyclohexyl-2-oxonon-5-en-4-yl)-2-methylbutanamide. A white solid. mp 92-96°C. $R_f = 0.19$ (30:1 CH_2Cl_2 : Et_2O); IR (KBr): 3393 (br, s), 2916 (br, s), 1709 (s), 1631 (s), 1538 (s), 1383 (m), 1281 (m) cm^{-1} ; ^1H NMR: δ 6.26 (1H, d, $J = 8.4$ Hz, NH), 5.52 (1H, dt, $J = 16$ Hz, $J = 6.4$ Hz, vinyl), 5.43 (1H, dd, $J = 16$ Hz, $J = 6.0$ Hz, vinyl), 4.70 (1H, m, $\text{CH}=\text{CHCHN}$), 2.73 (1H, dd, $J = 17$ Hz, $J = 4.8$ Hz, $\text{C}(\text{O})\text{CH}_a\text{H}_b\text{CN}$), 2.63 (1H, dd, $J = 17$ Hz, $J = 5.6$ Hz, $\text{C}(\text{O})\text{CH}_a\text{H}_b\text{CN}$), 2.23 (2H, d, $J = 6.8$ Hz, $\text{CyCH}_2\text{C}(\text{O})$), 2.04 (1H, h, $J = 6.8$ Hz, $\text{NC}(\text{O})\text{CH}(\text{CH}_3)\text{CH}_2-$), 1.93 (2H, q, $J = 7.2$ Hz, allylic CH_2), 1.54-1.86 (7H, m, aliphatic), 1.03-1.45 (7H, m, aliphatic), 1.07 (3H, d, $J = 6.8$ Hz, $\text{NC}(\text{O})\text{CH}(\text{CH}_3)\text{CH}_2-$), 0.77-0.96 (7H, m, aliphatic); ^{13}C NMR: δ 210.3, 175.4, 132.2, 128.8, 51.15, 47.30, 47.26, 43.19, 34.16, 33.78, 33.15, 33.06, 27.25, 26.10, 26.00, 21.15, 17.35, 13.54, 11.81. LRMS (ES+) Calc'd for $\text{C}_{20}\text{H}_{35}\text{NO}_2$ ($\text{M} + \text{Na}$) $^+$: 344.3. Found ($\text{M} + \text{Na}$) $^+$: 344.2.

(*R,Z*)-*N*-(1,4-diphenyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)but-3-enyl)propionamide. A waxy solid. $R_f = 0.27$ (10:1 $\text{CH}_2\text{Cl}_2:\text{Et}_2\text{O}$); IR (KBr): 3293 (br, s), 3060 (br, m), 2970 (br, m), 1949 (w), 1642 (s), 1600 (s), 1537 (s), 1355 (s), 1142 (s) cm^{-1} ; ^1H NMR: δ 7.10-7.48 (11H, m, ArH + vinyl CH), 6.36 (1H, d, $J = 6.8$ Hz, NH), 5.05 (1H, m, ArCHN), 2.82 (1H, m, C(B)CH_aH_bCN), 2.73 (1H, m, C(B)CH_aH_bCN), 2.12 (2H, q, $J = 7.6$ Hz, NC(O)CH₂CH₃), 1.32 (6H, s, C(CH₃)₂), 1.30 (6H, s, C(CH₃)₂), 1.08 (3H, t, $J = 7.6$ Hz, NC(O)CH₂CH₃); ^{13}C NMR: δ 172.9, 145.9, 143.2, 137.3, 128.9, 128.33, 128.31, 127.5, 126.8, 126.0, 83.94, 54.33, 35.87, 29.63, 25.18, 24.51, 9.78. LRMS (APCI+) Calc'd for $\text{C}_{25}\text{H}_{32}\text{BNO}_3$ ($\text{M} + \text{Na}$)⁺: 428.2. Found ($\text{M} + \text{Na}$)⁺: 428.5.

(*R,Z*)-*tert*-butyl 1,4-diphenyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)but-3-enylcarbamate. A white foam. $R_f = 0.24$ (CH_2Cl_2); IR (KBr): 3390 (br, m), 2975 (br, m), 1950 (w), 1717 (s), 1611 (m), 1514 (s), 1382 (s), 1344 (s), 1262 (s), 1165 (s) cm^{-1} ; ^1H NMR: δ 7.41 (1H, s, vinyl CH), 7.12-7.38 (10H, m, ArH), 5.81 (1H, br d, $J = 5.2$ Hz, NH), 4.71 (1H, br m, ArCHN), 2.77 (1H, m, C(B)CH_aH_bCN), 2.54 (1H, m, C(B)CH_aH_bCN), 1.37 (9H, s, OC(CH₃)₃), 1.35 (6H, s, C(CH₃)₂), 1.33 (6H, s, C(CH₃)₂); ^{13}C NMR: δ 155.3, 145.7, 144.2, 137.3, 128.9, 128.3, 128.2, 127.5, 126.7, 125.8, 84.01, 78.86, 56.33, 36.48, 28.40, 24.98, 24.68. LRMS (ES+) Calc'd for $\text{C}_{27}\text{H}_{36}\text{BNO}_4$ ($\text{M} + \text{Na}$)⁺: 472.3. Found ($\text{M} + \text{Na}$)⁺: 472.3.

(*R,E*)-tert-butyl-1,3,4-triphenylbut-3-enylcarbamate. A waxy solid. $R_f = 0.18$ (9:1 Hexanes:EtOAc); IR (CH₂Cl₂ solution): 3437 (br, m), 2966 (br, m), 1953 (w), 1705 (s), 1495 (s), 1363 (s), 1246 (m), 1165 (s) cm⁻¹; ¹H NMR: δ 7.45 (2H, d, $J = 7.2$ Hz, ArH), 7.40 (2H, t, $J = 7.2$ Hz, ArH), 7.33 (3H, t, $J = 7.6$ Hz, ArH), 7.17-7.29 (4H, m, ArH), 7.11 (2H, d, $J = 7.6$ Hz, ArH), 7.00 (2H, dd, $J = 7.6$ Hz, $J = 2.0$ Hz, ArH), 6.75 (1H, s, vinyl CH), 4.65 (2H, br s, NH + ArCHN), 3.19 (1H, m, ArCHNCH_aH_b), 3.07 (1H, m, ArCHNCH_aH_b), 1.37 (9H, s, OC(CH₃)₃); ¹³C NMR: δ 154.8, 142.1, 139.4, 137.7, 131.2, 128.8, 128.5, 128.3, 128.2, 127.4, 127.1, 126.8, 126.7, 126.2, 53.85, 37.26, 28.28. LRMS (ES+) Calc'd for C₂₇H₂₉NO₂ (M + Na)⁺: 422.2. Found (M + Na)⁺: 422.4.

Proof of stereochemistry. Stereochemical ratios were determined in comparison with authentic racemic material, which was prepared using racemic starting material that had been synthesized as described above with tricyclohexylphosphine in place of the chiral ligand in the diboration step. Absolute stereochemistry was determined by comparison to that obtained for (*R*)-*N*-(3-oxo-1,4-diphenylbutyl)acetamide.

Chiral SFC (AD-H, Chiralpak, 150 psi, 50°C, flow = 0.5 mL/min, 5.0 % MeOH) analysis of the Suzuki-coupling product:

Suzuki
product

Racemic

Suzuki product +
racemic coinjection

(*R,Z*)-*N*-(1,4-diphenylbut-3-enyl)propionamide. A white solid. $R_f = 0.22$ (20:1 $\text{CH}_2\text{Cl}_2\text{:Et}_2\text{O}$); IR (KBr): 3328 (br, m), 3017 (br, m), 2940 (br, m), 1953 (w), 1884 (w), 1802 (w), 1642 (s), 1530 (s), 1452 (s), 1383 (s), 1274 (s), 1102 (s) cm^{-1} ; ^1H NMR: δ 7.15–7.39 (10H, m, ArH), 6.51 (1H, d, $J = 11.6$ Hz, ArCH=CH), 5.71 (1H, d, $J = 8.0$ Hz, NH), 5.58 (1H, m, ArCH=CHCH₂), 5.13 (1H, q, $J = 7.6$ Hz, ArCHN), 2.84 (2H, t, $J = 6.8$ Hz, ArCH=CHCH₂), 2.15 (2H, q, $J = 7.6$ Hz, CH₂CH₃), 1.10 (3H, t, $J = 7.6$ Hz, CH₂CH₃); ^{13}C NMR: δ 173.0, 141.63, 137.0, 131.5, 128.7, 128.6, 128.3, 127.7, 127.4, 126.9, 126.5, 52.97, 34.93, 29.69, 9.76. LRMS (ES⁺) Calc'd for $\text{C}_{19}\text{H}_{21}\text{NO}$ ($\text{M} + \text{Na}$)⁺: 302.2. Found ($\text{M} + \text{Na}$)⁺: 302.3.

Proof of stereochemistry. Stereochemical ratios were determined in comparison with authentic racemic material, which was prepared using racemic starting material that had been synthesized as described above with tricyclohexylphosphine in place of the chiral ligand in the diboration step. Absolute stereochemistry was determined by comparison to that obtained for (*R*)-*N*-(3-oxo-1,4-diphenylbutyl)acetamide.

Chiral SFC (AD-H, Chiralpak, 150 psi, 50°C, flow = 4.0 mL/min, 3.0 % MeOH) analysis of the protonation product:

(Z)-1,4-diphenyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)but-3-en-1-amine. A white solid. mp 170-173°C. IR (KBr): 3309 (br, m), 3196 (br, m), 3067 (br, s), 2971 (br, s), 1945 (w), 1884 (w), 1806 (w), 1596 (s), 1495 (s), 1460 (s), 1383 (s), 1212 (s), 1150 (s) cm^{-1} ; ^1H NMR: δ 7.24-7.50 (5H, m, ArH), 7.11 (5H, s, ArCH), 6.85 (1H, s, vinyl CH), 3.5-5.0 (2H, br s, NH_2), 4.09 (1H, m, ArCHN), 2.93 (1H, m, ArCH(N) CH_aH_b), 2.83 (1H, m, ArCH(N) CH_aH_b), 1.20 (6H, s, $\text{C}(\text{CH}_3)_2$), 1.17 (6H, s, $\text{C}(\text{CH}_3)_2$); ^{13}C NMR: δ 140.9, 139.3, 131.6, 128.7, 128.6, 128.1, 127.9, 126.9, 125.9, 80.25, 57.26, 38.84, 25.49. LRMS (ES+) Calc'd for $\text{C}_{22}\text{H}_{28}\text{BNO}_2$ ($\text{M} + \text{H}$) $^+$: 350.2. Found ($\text{M} + \text{H}$) $^+$: 350.4.

(R)-3-acetamido-N-methoxy-N-methyl-3-phenylpropanamide. An off-white solid. R_f = 0.19 (5% MeOH in EtOAc); IR (KBr): 3352 (br, s), 3033 (br, m), 2940 (br, m), 1957 (w), 1876 (w), 1670 (s), 1631 (s), 1534 (s), 1460 (s), 1367 (s), 1293 (s), 1204 (s) cm^{-1} ; ^1H NMR: δ 7.41 (1H, d, J = 6.8 Hz, NH), 7.15-7.40 (5H, m, ArH), 5.39 (1H, m, ArCHN), 3.44 (3H, s, CH_3), 3.12 (1H, dd, J = 16 Hz, J = 5.0 Hz, ArCH(N) CH_aH_b), 3.06 (3H, s, CH_3), 2.77 (1H, dd, J = 16 Hz, J = 5.0 Hz, ArCH(N) CH_aH_b), 1.99 (3H, s, $\text{NC}(\text{O})\text{CH}_3$); ^{13}C NMR: δ 171.9, 169.3, 141.3, 128.5, 127.2, 126.2, 61.16, 49.66, 36.65, 31.73, 23.36. LRMS (ES+) Calc'd for $\text{C}_{13}\text{H}_{18}\text{N}_2\text{O}_3$ ($\text{M} + \text{Na}$) $^+$: 273.1. Found ($\text{M} + \text{Na}$) $^+$: 273.2.

Js2-236 column

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 PROCNO 1

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 SOLVENT CDCl3
 NS 13
 DS 2
 SMH 6410.256 Hz
 FIDRES 0.195625 Hz
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 RG 181
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 DE 6.00 usec
 TE 293.0 K
 DI 1.00000000 sec

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 PL1 0.00 dB
 SFO1 400.0524003 MHz

F2 - Processing parameters
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 SF 400.0500200 MHz
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 SSB 0
 LB 0.30 Hz
 GB 0

JS2-236 column

Current Data Parameters
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 PROCNO 1

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 SMH 26246.719 Hz
 FIDRES 0.400493 Hz
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 TE 300.0 K
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 d11 0.03000000 sec
 d12 0.00002000 sec

===== CHANNEL f1 =====
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 PL1 0.00 dB
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===== CHANNEL f2 =====
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 NUC2 1H
 PCPO2 80.00 usec
 PL2 -6.00 dB
 PL12 13.80 dB
 PL13 14.50 dB
 SF02 400.0516002 MHz

F2 - Processing parameters
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 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
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 CY 7.00 cm
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 F1 22330.39 Hz
 F2P -10.000 ppm
 F2 -1005.93 Hz
 PPMCM 11.50000 ppm/cm
 HZCM 1156.81580 Hz/cm

Js2-244 column

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PROCNO 1
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PULPROG zg30
TD 32768
FIDRES 0.125112 Hz
SFO1 300.1300041 MHz
AQ 3.864096 sec
RG 118.000 usec
DE 168.57 usec
TE 293.0 K
D1 1.00000000 sec
SFO2 300.130000 MHz
NUC12H5 1H
F2 - Processing parameters
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SF 300.1300041 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

JS2-244 column

Current Data Parameters

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PROCNO	1

F2 - Acquisition Parameters

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DS	2
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FIDRES	0.400493 Hz
AQ	1.2485298 sec
RG	2298.8
DM	19.050 usec
DE	6.00 usec
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D1	0.80000001 sec
d11	0.03000000 sec
d12	0.00002000 sec

***** CHANNEL f1 *****

NUC1	13C
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PL1	0.00 dB
SFO1	100.6237964 MHz

***** CHANNEL f2 *****

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	13.80 dB
PL13	14.50 dB
SFO2	400.1322200 MHz

F2 - Processing parameters

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WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	12.50 cm
F1P	220.000 ppm
F1	22134.80 Hz
F2P	-10.000 ppm
F2	-1006.13 Hz
PPMCM	11.50000 ppm/cm
HZCM	1157.04639 Hz/cm

Js2-248 column

Current Data Parameters

NAME Js2-248c01
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters

Date_ 20050806
Time 16.08
INSTRUM spect
PROBHD 5mm QNP 1H/13C
PULPROG zgpg30
TD 29930
FIDRES 0.400493 Hz
AQ 1.2485298 sec
RG 3649.1
DM 19.050 usec
DE 5.00 usec
TE 300.0 K
D1 0.80000001 sec
d11 0.03000000 sec
d12 0.00002000 sec

***** CHANNEL f1 *****

NUC1 13C
P1 5.70 usec
PL1 0.00 dB
SFO1 100.6237964 MHz

***** CHANNEL f2 *****

CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -6.00 dB
PL12 13.80 dB
PL13 14.50 dB
SFO2 400.1322200 MHz

F2 - Processing parameters

SI 32768
SF 100.6127290 MHz
MDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
CY 8.00 cm
F1P 220.000 ppm
F1 22334.80 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PNUCM 11.50000 ppm/cm
HZCM 1157.04639 Hz/cm

Js2-275 column

ppm

209.520

169.177

141.171

140.703

128.496

128.239

128.201

127.308

126.146

125.796

77.199

76.881

76.563

49.422

47.167

42.448

34.625

24.542

23.237

Current Data Parameters

NAME Js2-275col1
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters

Date_ 20050903
Time 17.32

INSTRUM spect

PROBHD 5mm QNP 1H/13C

PULPROG zgpg30

TD 65536

SOLVENT CDCl3

NS 122

DS 2

SMH 26246.719 Hz

FIDRES 0.400493 Hz

AQ 1.2485298 sec

RG 5160.6

DW 19.050 usec

DE 6.00 usec

TE 300.0 K

D1 0.80000001 sec

d11 0.03000000 sec

d12 0.0002000 sec

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

NUC1 13C

P1 6.00 usec

PL1 0.00 dB

SFO1 100.6036782 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16

NUC2 1H

PCPD2 80.00 usec

PL2 -6.00 dB

PL12 13.80 dB

PL13 14.50 dB

SFO2 400.0516002 MHz

F2 - Processing parameters

SI 32768

SF 100.5926741 MHz

MDW EM

SSB 0

LB 1.00 Hz

GB 0

PC 1.40

10 NMR plot parameters

CX 20.00 cm

CY 8.00 cm

F1P 220.000 ppm

F1 22130.39 Hz

F2P -10.000 ppm

F2 -1005.93 Hz

PPMCH 11.50000 ppm/cm

HZCH 1156.81580 Hz/cm

Js2-245 column

Current Data Parameters
 NAME: Js2-245col
 METHOD: 1
 PROCNO: 1

F2 - Acquisition Parameters
 Date_: 20050803
 Time: 11.43
 INSTRUM: spect
 PROCNO: 5mm QNP 1H/13C
 PULPROG: zgpg30
 TOU: 32768
 SFO: 300.135
 SOLVENT: CDCl3
 NS: 16
 DS: 2
 SWH: 6410.256 Hz
 FIDRES: 0.155945 Hz
 AQ: 2.355945 sec
 RG: 362
 TM: 78.000 sec
 DE: 5.00 usec
 TE: 300.2 K
 D1: 1.00000000 sec

===== CHANNEL f1 =====
 NUC1: 13C
 P1: 22.00 usec
 PL1: 0.00 dB
 SFO1: 400.112608 MHz

F2 - Processing parameters
 SI: 32768
 SF: 400.130172 MHz
 WDW: EM
 SSB: 0
 LB: 0.30 Hz
 GB: 0
 PC: 1.00

Js2-245 column

Current Data Parameters
 NAME Js2-245col
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050802

Time 18.12
 INSTRUM spect
 PROBRD 5mm QNP 1H/13C
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 122
 DS 2
 SMH 26246.719 Hz
 FIDRES 0.400493 Hz
 AQ 1.2465298 sec
 RG 1290.2
 DM 19.050 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.80000001 sec
 d11 0.03000000 sec
 d12 0.0002000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 5.70 usec
 PL1 0.00 dB
 SF01 100.6237964 MHz

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -6.00 dB
 PL12 13.80 dB
 PL13 14.50 dB
 SF02 400.1322200 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127290 MHz
 MVM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

10 NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 220.000 ppm
 F1 22134.80 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PPMQCM 11.50000 ppm/cm
 HZCM 1157.04639 Hz/cm

Js2-274 column

Js2-274 column

Current Data Parameters
NAME Js2-274c01
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050904
Time 16.53

INSTRUM spect
PROBHD 5mm QNP 1H/13C
PULPROG zgpg30

TD 65536
SOLVENT CDCl3
NS 191

DS 2
SMH 26246.719 Hz
FIDRES 0.400493 Hz

AD 1.2485298 sec
RG 14596.5

DM 19.050 usec
DE 6.00 usec

TE 300.0 K
D1 0.80000001 sec
d11 0.03000000 sec
d12 0.00002000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 6.00 usec
PL1 0.00 dB
SF01 100.6036782 MHz

***** CHANNEL f2 *****
CPOPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -6.00 dB
PL12 13.80 dB
PL13 14.50 dB
SF02 400.0516002 MHz

F2 - Processing Parameters
SI 32768
SF 100.5926741 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

10 NMR plot parameters
CX 20.00 cm
CY 8.00 cm
F1P 220.000 ppm
F1 22130.39 Hz
F2P -10.000 ppm
F2 -1005.93 Hz
PPMCM 11.50000 ppm/cm
HZCM 1156.81580 Hz/cm

Js2-250 column

7.300
7.281
7.263
7.242
7.225
7.205
6.747
6.728

5.362
5.348
5.342

3.064
3.051
3.023
3.010
2.848
2.833
2.807
2.792
2.188
2.170
2.160
2.144
2.019
1.969
1.692
1.684
1.675
1.666
1.657
1.596
1.573
1.500
1.469
1.238
1.231
1.183
1.177
1.167
1.157
1.152
1.127
1.113
1.100
1.075
1.068

Current Data Parameters
NAME Js2-250col
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050807
Time 16.03
INSTRUM spect
PROBHD 5mm QNP 1H/13C
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5560319 sec
RG 203.2
DM 78.000 usec
DE 6.00 usec
TE 293.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 22.50 usec
PL1 0.00 dB
SFO1 400.1326008 MHz

F2 - Processing parameters
SI 32768
SF 400.1300172 MHz
WDW EM
SSB 0
LB 0
GB 0.30 Hz

Current Data Parameters
NAME Js2-250col
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 20050807
Time 16.11
INSTRUM spect
PROBHD 5mm QNP 1H/13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 202
DS 2
SWH 26246.719 Hz
FIDRES 0.400493 Hz
AQ 1.2485298 sec
RG 1625.5
DM 19.050 usec
DE 6.00 usec
TE 300.0 K
D1 0.80000001 sec
d11 0.03000000 sec
d12 0.00002000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 5.70 usec
PL1 0.00 dB
SFO1 100.6237964 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -6.00 dB
PL12 13.80 dB
PL13 14.50 dB
SFO2 400.1322200 MHz

F2 - Processing parameters
SI 32768
SF 100.6127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 220.000 ppm
F1 22134.80 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCH 11.50000 ppm/cm
HZCH 1157.04639 Hz/cm

JS2-270 column

Current Data Parameters

NAME JS2-270c01

EXPNO 12

PROCNO 1

F2 - Acquisition Parameters

Date_ 20050826

Time 18.20

INSTRUM spect

PROBHD 5mm QNP 1H/13C

PULPROG zgpg30

TD 65536

SOLVENT CDCl3

NS 72

DS 2

SWH 26245.719 Hz

FIDRES 0.400493 Hz

AQ 1.2485298 sec

RG 4597.6

DM 19.050 usec

DE 6.00 usec

TE 300.0 K

D1 0.80000001 sec

d11 0.03000000 sec

d12 0.00002000 sec

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

NUC1 13C

P1 5.70 usec

PL1 0.00 dB

SFO1 100.6237964 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16

NUC2 1H

PCPD2 80.00 usec

PL2 -6.00 dB

PL12 13.80 dB

PL13 14.50 dB

SFO2 400.1322200 MHz

F2 - Processing parameters

SI 32768

SF 100.6127290 MHz

WDW EM

SSB 0

LB 1.00 Hz

GB 0

PC 1.40

1D NMR plot parameters

CX 20.00 cm

CY 8.00 cm

F1P 220.000 ppm

F1 22134.80 Hz

F2P -10.00 ppm

F2 -1005.13 Hz

PPHMC 11.50000 ppm/cm

HZCM 1157.04639 Hz/cm

J52-277 column

Current Data Parameters
 NAME J52-277c01
 EXPNO 12
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050902
 Time 18.32
 INSTRUM spect
 PROBNM 5mm QNP 1H/13C
 PULPROG zgpg30
 TO 65536
 SOLVENT CDCl3
 NS 158
 DS 2
 SMH 26246.719 Hz
 FIDRES 0.400493 Hz
 AQ 1.2465298 sec
 RG 5160.6
 DM 19.050 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.8000001 sec
 d11 0.0300000 sec
 d12 0.0002000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 5.00 usec
 PL1 0.00 dB
 SF01 100.6036782 MHz

===== CHANNEL f2 =====
 CPOPRG2 waltz16
 NUC2 1H
 PCPO2 80.00 usec
 PL2 -6.00 dB
 PL12 13.80 dB
 PL13 14.50 dB
 SF02 400.0516002 MHz

F2 - Processing parameters
 SI 32768
 SF 100.5926741 MHz
 MDN EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 8.00 cm
 F1P 220.000 ppm
 F1 22130.39 Hz
 F2P -10.000 ppm
 F2 -1005.93 Hz
 PPMCM 11.50000 ppm/cm
 HZCM 1156.81580 Hz/cm

js2-281 column2

Js2-281 column2

Current Data Parameters
 NAME Js2-281c012
 EXPNO 12
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050904
 Time 15.08
 INSTRUM spect
 PROBRD 5mm QNP 1H/13C
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 255
 DS 2
 SMH 26246.719 Hz
 FIDRES 0.400493 Hz
 AQ 1.2485298 sec
 RG 4597.6
 DW 19.050 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.80000001 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 6.00 usec
 PL1 0.00 dB
 SF01 100.6036782 MHz

***** CHANNEL f2 *****
 CPOPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -6.00 dB
 PL12 13.80 dB
 PL13 14.50 dB
 SF02 400.0516002 MHz

F2 - Processing parameters
 SI 32768
 SF 100.5926741 MHz
 MDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 8.00 cm
 F1P 220.000 ppm
 F1 22130.39 Hz
 F2P -10.000 ppm
 F2 -1005.93 Hz
 PPMCM 11 50000 ppm/cm
 HZCM 1156.81580 Hz/cm

Js2-291 column2

Js2-291 column2

Current Data Parameters

NAME	Js2-291c012
EXPNO	101
PROCNO	1

F2 - Acquisition Parameters

Date_	20050919
Time	18.14
INSTRUM	spect
PROBHD	5mm QNP 1H/13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	992
DS	2
SWH	26246.719 Hz
FIDRES	0.400493 Hz
AQ	1.2465298 sec
RG	3649.1
DM	19.050 usec
DE	6.00 usec
TE	300.0 K
D1	0.80000001 sec
d11	0.03000000 sec
d12	0.00002000 sec

CHANNEL f1

NUC1	13C
P1	6.00 usec
PL1	0.00 dB
SFO1	100.6036782 MHz

CHANNEL f2

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-6.00 dB
PL12	-13.80 dB
PL13	14.50 dB
SFO2	400.0516002 MHz

F2 - Processing parameters

SI	32768
SF	100.5926741 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	12.50 cm
F1P	220.000 ppm
F1	22130.39 Hz
F2P	-1.000 ppm
F2	-100.59 Hz
PPMCH	11.05000 ppm/cm
HZCH	1111.94907 Hz/cm

JS3-4 column

7.332
7.313
7.272
7.253
7.247
7.241
7.235
7.221
7.202
7.189
7.173
7.169
7.118
7.099
7.011
7.006
6.992
6.988
6.750

4.648

3.187
3.172
3.091
3.083
3.063
3.049

1.371
1.269
1.240

Current Data Parameters
NAME JS3-4col
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050921
Time 18.27
INSTRUM spect
PROBHD 5mm QNP 1H/13C
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 14
DS 2
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5560319 sec
RG 143.7
DW 78.000 usec
DE 6.00 usec
TE 293.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 24.20 usec
PL1 0.00 dB
SFO1 400.0524003 MHz

F2 - Processing parameters
SI 32768
SF 400.0500200 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0

JS3-4 column

Current Data Parameters
NAME JS3-4c01
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050921
Time 18.34
INSTRUM spect
PROBHD 5mm QNP 1H/13C
PULPROG zgpg30
TO 65536
SOLVENT CDCl3
NS 1000
DS 2
SWH 26246.719 Hz
FIDRES 0.400493 Hz
AQ 1.2485298 sec
RG 9195.2
DM 19.050 usec
DE 6.00 usec
TE 300.0 K
D1 0.80000001 sec
d11 0.03000000 sec
d12 0.0002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 6.00 usec
PL1 0.00 dB
SF01 100.6036782 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -6.00 dB
PL12 13.80 dB
PL13 14.50 dB
SF02 400.0516002 MHz

F2 - Processing parameters
SI 32768
SF 100.5926741 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 11.00 cm
F1P 180.000 ppm
F1 18105.88 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCH 9.00000 ppm/cm
HZCH 905.33405 Hz/cm

Js2-283 column

7.341
7.321
7.303
7.285
7.243
7.230
7.211

6.526
6.497

5.697
5.598
5.587
5.581
5.569

5.143
5.124

2.858
2.841
2.825
2.822

2.181
2.179
2.162
2.160
2.143
2.123

1.120
1.101
1.082

Current Data Parameters
NAME Js2-283col
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050910
Time 14.00
INSTRUM spect
PROBHD 5mm QNP 1H/13C
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5560319 sec
RG 256
DW 78.000 usec
DE 6.00 usec
TE 293.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 24.20 usec
PL1 0.00 dB
SFO1 400.0524003 MHz

F2 - Processing parameters
SI 32768
SF 400.050200 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0

J52-283 column

Current Data Parameters
 NAME J52-283c01
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050910
 Time 14.03
 INSTRUM spect
 PROBHD 5mm QNP 1H/13C
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 96
 DS 2
 SMH 26246.719 Hz
 FIDRES 0.400493 Hz
 AQ 1.2485298 sec
 RG 6502
 DM 19.050 usec
 DE 5.00 usec
 TE 300.0 K
 D1 0.80000001 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 6.00 usec
 PL1 0.00 dB
 SF01 100.6036782 MHz

***** CHANNEL f2 *****
 CPOPRG2 waltz16
 NUC2 1H
 PCPRG2 80.00 usec
 PL2 -6.00 dB
 PL12 13.80 dB
 PL13 14.50 dB
 SF02 400.0516002 MHz

F2 - Processing parameters
 SI 32768
 SF 100.5926741 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 8.00 cm
 F1P 220.000 ppm
 F1 22130.39 Hz
 F2P -10.000 ppm
 F2 -1005.93 Hz
 PPM0H 11.50000 ppm/cm
 HZ0H 1156.81580 Hz/cm