**Ring Expansive Routes to Quinolizidine Alkaloids: Formal Synthesis of (–)-
Lasubine II**

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Supporting Information

Experimental Details

Copies of ^1H and ^{13}C spectra of listed compounds

GC analysis of
a 1 : 2 mixture of **3a** and **3b**
and
the crude nitron photolysis product (**3a**) vs (**3b**)

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Experimental Details

(1R,3S)-(+)-4-Cyclopentene-1,3-diol-1-acetate (A). (1R,3S)-(+)-4-cyclopentene-1,3-diol-1-acetate (**A**) was prepared from the freshly cracked cyclopentadiene according to literature procedures¹⁻³ as white colorless needles. Mp 49-51 °C (lit.³ 49-51°C); [α]_D +71.3 (*c* 1.25, CHCl₃); [α]_D +67.5 (*c* 1.64, CHCl₃); Lit. values [α]_D +73.8 (*c* 1.25, CHCl₃);³ [α]_D +68.0 (*c* 1.64, CHCl₃).⁴

(1R,4S)-(-)-4-Triisopropylsilyloxy-2-cyclopentene-1-ol acetate (B). To (1R,3S)-(+)-4-cyclopentene-1,3-diol-1-acetate (**A**) (3.68 g, 25.9 mmol) in DMF (20 mL) was added imidazole (5.28 g, 77.7 mmol) in one portion. Triisopropylsilylchloride (16.5 mL, 77.7 mmol) was added to the reaction mixture, which was stirred at room temperature under argon for 13 h. The reaction mixture was diluted with CH₂Cl₂ (200 mL) and the organic layer was washed with 2N sulfuric acid (50 mL), H₂O (25 mL), and brine (25 mL). The organic layer was dried (anhyd Na₂SO₄) and concentrated to afford a clear oil. Flash chromatography (25% EtOAc/hexane) gave 5.5 g (72%) of **B** as a clear

oil. $R_f = 0.6$ (10% EtOAc/hexane); ^1H NMR (400 MHz, CDCl_3) δ 1.04-1.14 (m, 21H), 1.64 (dt, $J = 5.1, 13.7$ Hz, 1H), 2.04 (s, 3H), 2.84 (dt, $J = 7.3, 14.5$ Hz, 1H), 4.79 (m, 1H), 5.46 (m, 1H), 5.88 (dt, $J = 1.6, 5.6$ Hz, 1H), 6.02 (dt, $J = 1.6, 5.6$ Hz, 1H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 12.1, 17.8, 17.9, 21.1, 41.5, 74.8, 76.8, 131.0, 139.0, 170.8; IR (neat) 2946, 2866, 1738 cm^{-1} ; MS (CI) m/z 299 ($\text{M}^+ + 1$), 239, 125; HRMS calcd for $\text{C}_{16}\text{H}_{31}\text{O}_3\text{Si}$ ($\text{M}^+ + 1$): 299.2042, found 299.2057; $[\alpha]_D -6.33$ (c 1.53, MeOH).

(1R,4S)-(-)-4-Triisopropylsilyloxy-1-hydroxycyclopent-2-ene (C). According to the procedure of Paquette et al.,⁵ to (1R, 4S)-(-)-4-triisopropylsilyloxy-2-cyclopentenylacetate (**B**) (6.00 g, 20.1 mmol) in CH_3OH (100 mL) was added sodium methoxide (2.17 g, 40.2 mmol) in one portion. The reaction mixture was stirred at room temperature under argon for 15 min, after which time it was concentrated and diluted with CH_2Cl_2 (100 mL). The organic layer was washed with water (10 mL), dried (anhyd Na_2SO_4) and concentrated to give a clear oil. Flash chromatography (10% EtOAc/hexane) afforded 4 g (77%) of **C** as a clear oil. $R_f = 0.4$ (10% EtOAc/hexane). ^1H NMR (400 MHz, CDCl_3) δ 1.04-1.14 (m, 21H), 1.57 (dt, $J = 4.5, 13.7$ Hz, 1H), 1.61 (br s, 1H), 2.71 (dt, $J = 6.9, 13.7$ Hz, 1H), 4.59 (m, 1H), 4.74 (m, 1H), 5.94 (m, 2H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 12.1, 18.0, 45.0, 75.1, 75.2, 135.5, 137.2; IR (neat) 3400 cm^{-1} ; MS (CI) m/z 257 ($\text{M}^+ + 1$), 239; HRMS calcd for $\text{C}_{14}\text{H}_{29}\text{O}_2\text{Si}$ ($\text{M}^+ + 1$): 257.1937, found 257.1923; $[\alpha]_D -19.4$ (c 0.50, MeOH).

(4S)-(-)-4-Triisopropylsilyloxy-2-cyclopenten-1-one (4). According to the procedure of Paquette et al.,⁵ to (1*R*,4*S*)-(-)-4-triisopropylsilyloxy-1-hydroxycyclopent-2-ene (**C**) (4.00 g, 15.6 mmol) in CH₂Cl₂ (20 mL) was added activated MnO₂ (9.40 g, 109.3 mmol). The resulting suspension was stirred under argon. After 3 h, additional manganese dioxide (4.69 g, 54.6 mmol) was added and stirring was continued for another 12 h. The reaction mixture was vacuum filtered through a pad of Celite, and the pad was washed with CH₂Cl₂ (200 mL). The organic layer was concentrated and purified by flash chromatography (10% EtOAc/hexane) to give 3.29 g of **4** (83%) as a clear oil. *R_f* = 0.5 (10% EtOAc/hexane). ¹H NMR (400 MHz, CDCl₃) δ 1.05-1.16 (m, 21H), 2.30 (dd, *J* = 2.3, 18.1 Hz, 1H), 2.74 (dd, *J* = 5.9, 18.1 Hz, 1H), 5.07 (m, 1H), 6.18 (dd, *J* = 1.2, 5.6 Hz, 1H), 7.50 (dd, *J* = 2.2, 5.6 Hz, 1H); ¹³C NMR (100.6 MHz, CDCl₃) δ 12.0, 17.8 (2 carbons), 45.3, 70.9, 134.3, 163.8, 206.4; IR (neat) 2947, 2866, 1716 cm⁻¹; MS (CI) *m/z* 255 (*M*⁺+1), 211; HRMS calcd for C₁₄H₂₇O₂Si (*M*⁺+1): 255.1780, found 255.1804; [α]_D -58.8 (*c* 1.05, MeOH).

(4R)-(+)-1-Triethylsiloxy-4-triisopropylsiloxy-cyclopentene (5). According to the procedure of Johnson et al.,⁶ to triethylsilane (2.14 mL, 13.4 mmol) was added 15

drops of Karstedt's catalyst. The reaction mixture was stirred under argon at room temperature for 15 min. After this time (4*S*)-(-)-4-triisopropylsilyloxy-2-cyclopenten-1-one (**4**) (1.71 g, 6.73 mmol) was added and the reaction mixture was heated on an oil bath at 100 °C for 1 h. The reaction mixture was cooled to room temperature and stirred for an additional 15 h. Flash chromatography (2% EtOAc/hexane) gave 2.4 g (96%) of **5** as a tan oil. R_f = 0.7 (2% EtOAc/hexane). ^1H NMR (400 MHz, CDCl_3) δ 0.67 (q, J = 7.9 Hz, 6H), 0.97 (t, J = 7.9 Hz, 9H), 1.03-1.06 (m, 21H), 2.21-2.34 (m, 2H), 2.50-2.60 (m, 2H), 4.50-4.55 (m, 2H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 4.8, 6.6, 12.1, 17.9, 39.8, 43.9, 70.6, 99.6, 151.9; IR (neat) 2943, 2866, 1644 cm^{-1} ; MS (CI) m/z 371 ($\text{M}^+ + 1$), 327, 132; HRMS calcd for $\text{C}_{20}\text{H}_{43}\text{O}_2\text{Si}$ ($\text{M}^+ + 1$): 371.2812, found 371.2802; $[\alpha]_D$ +5.02 (c 5.33, CHCl_3).

(3*R*)-5-(4'-Benzyloxy-1'-hydroxybutyl)-3-triisopropylsilocyclopentanone (D, mixture of isomers). According to the procedure of Danishefsky et al.,⁷ to the aldehyde **6**⁸ (2.53 g, 14.5 mmol) in CH_2Cl_2 (40 mL) at -78 °C was added freshly distilled TiCl_4 (1.55 mL, 14.5 mmol) over 20 min. The orange reaction mixture was stirred at -78 °C for an additional 15 min. After this time the enol ether **5** (2.69 g, 7.27 mmol) in CH_2Cl_2 (20 mL) was added via syringe pump over 1 h at -78 °C. The reaction mixture is stirred for an additional 20 min and H_2O (20 mL) was added rapidly. The reaction mixture was extracted into CH_2Cl_2 (500 mL), dried (anhyd Na_2SO_4) and concentrated to

afford a pale pink oil. Flash chromatography (10% EtOAc/hexane) gave 2.2 g (70%) of **D** as a pale yellow oil. The product was obtained as mixture of four diastereomers and was used without further purification. IR (neat) 3444, 2941, 1736 cm^{-1} ; MS (CI) m/z 435 ($M^+ + 1$), 257, 243, 179, 108, 91, 71; HRMS calcd for $\text{C}_{25}\text{H}_{43}\text{SiO}_4$ ($M^+ + 1$): 435.2931, found 435.2894.

(3R)-(+)-5-(4'-Benzyloxybutylidene)-3-triisopropylsiloxy-cyclopentanone (7).

To a solution of keto alcohol diastereomers **D** (1.1 g, 2.53 mmol) in CH_2Cl_2 (40 mL) was added MsCl (0.48 mL, 6.33 mmol), followed by the addition of a solution of DMAP (0.61 g, 5.0 mmol) in CH_2Cl_2 (10 mL) over 30 min. After the addition was complete additional DMAP (0.61 g, 5.0 mmol) in CH_2Cl_2 (10 mL) was added and the reaction mixture was heated on an oil bath to 45 $^\circ\text{C}$ until a clear brown solution was formed (1 h). The reaction mixture was cooled and diluted with CH_2Cl_2 (200 mL). The organic layer was washed with water (20 mL), dried (anhyd Na_2SO_4), and concentrated to afford a brown oil. Flash chromatography (25% EtOAc/hexane) gave 0.85 g (81%) of **7** as a clear oil. R_f = 0.6 (25% EtOAc/hexane). ^1H NMR (400 MHz, CDCl_3) δ 0.84-1.11 (m, 21H), 1.77 (m, 2H), 2.24 (m, 2H), 2.40 (dd, J = 8.0, 20.0 Hz, 1H); 2.55-2.62 (overlapping dd and m, J = 6.1, 17.8 Hz, 2H), 2.85 (m, 1H), 3.48 (t, J = 6.3 Hz, 2H), 4.57 (m, 1H); 4.49 (s, 2H), 6.60 (m, 1H), 7.27-7.36 (m, 5H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 12.0, 17.9, 26.3, 28.4, 37.7, 49.0, 67.8, 69.3, 72.9, 127.5, 128.3, 136.0, 136.4, 138.3, 204.2; IR

(neat) 2906, 1725, 1653 cm^{-1} ; MS (CI) m/z 417 ($M^+ + 1$), 273, 243, 91; HRMS calcd for $\text{C}_{25}\text{H}_{41}\text{O}_3\text{Si}$ ($M^+ + 1$): 417.2825, found 417.2805; $[\alpha]_{\text{D}} + 14.7$ (c 0.58, CHCl_3).

(3R, 5S)-(+)-5-(4'-Hydroxybutyl)-3-triisopropylsiloxy-cyclopentanone (8). To the enone **7** (1.2 g, 2.88 mmol) in EtOH (15 mL), 10% Pd/C (0.23 g) was added. The reaction mixture was hydrogenated using a H_2 balloon for 10 h. After this time the reaction mixture was filtered through a pad of Celite and the pad was washed with additional EtOAc (50 mL). The organic layer was concentrated and purified by flash chromatography (25% EtOAc/hexane) to afford 900 mg (95%) of **8** as a clear oil. R_f = 0.2 (25% EtOAc/Hexane); ^1H NMR (400 MHz, CDCl_3) δ 0.97-1.12 (m, 21H), 1.30 (m, 1H), 1.43 (m, 3H), 1.56 (m, 2H), 1.65 (m, 1H), 1.83 (m, 1H), 2.12 (m, 1H), 2.23 (dd, J = 6.7, 18.2 Hz, 1H), 2.41 (m, 1H), 2.59 (dd, J = 6.5, 18.2 Hz, 1H), 3.64 (q, J = 8.0 Hz, 2H), 4.49 (m, 1H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 12.0, 17.9, 23.8, 30.0, 32.5, 39.0, 48.4, 48.6, 62.5, 68.7, 218.3; IR (neat) 3398, 2938, 1738 cm^{-1} ; MS (CI) m/z 329 ($M^+ + 1$), 155; HRMS calcd for $\text{C}_{18}\text{H}_{37}\text{O}_3\text{Si}$ ($M^+ + 1$): 329.2512, found 329.2523; $[\alpha]_{\text{D}} + 61.4$ (c 0.23, CHCl_3).

(3*R*, 5*S*)-(+)-5-(4'-Azidobutyl)-3-triisopropylsiloxy-cyclopentanone (9). The keto alcohol **8** (0.549 g, 1.67 mmol) was dissolved in toluene (50 mL) and cooled to 0 °C. To the reaction mixture was added PPh₃ (1.32 g, 5.0 mmol) followed by Zn(N₃)₂•2pyr (770 mg, 2.5 mmol). After the reaction mixture was stirred for 15 min at 0 °C, diisopropyl azodicarboxylate (DIAD) (1.15 g, 5.0 mmol) was added carefully over 20 min. The reaction mixture was stirred and allowed to warm up to room temperature over 17 h. After this time Et₂O (200 mL) was added, and the organic layer was washed with H₂O (20 mL), dried (anhyd Na₂SO₄), and concentrated to a pale yellow oil. Flash chromatography (10% EtOAc/hexane) gave 490 mg (83%) of **9** as a pale yellow oil. *R_f* = 0.8 (25% EtOAc/Hexane). ¹H NMR (400 MHz, CDCl₃) δ 0.97-1.12 (m, 21H), 1.42 (m, 3H), 1.56 (m, 2H), 1.67 (m, 1H), 1.83 (m, 1H), 2.10 (m, 1H), 2.24 (dd, *J* = 6.5, 18.2 Hz, 1H), 2.41 (m, 1H), 2.59 (dd, *J* = 6.5, 18.2 Hz, 1H), 3.27 (t, *J* = 6.8 Hz, 2H), 4.49 (m, 1H); ¹³C NMR (100.6 MHz, CDCl₃) δ 11.9, 17.8, 24.7, 28.7, 29.8, 38.9, 48.5, 48.1, 51.1, 68.7, 217.9; IR (neat) 2917, 2095, 1743 cm⁻¹; MS (CI) *m/z* 354 (*M*⁺+1), 326, 282, 152; HRMS calcd for C₁₈H₃₆N₃O₂Si (*M*⁺+1): 354.2577, found 354.2552; [α]_D +51.9 (*c* 0.29, CHCl₃).

(2*S*,4*R*)-(+)-4-Triisopropylsiloxy-2-(4'-*N*,*O*-di(benzyloxycarbonyl)hydroxyaminobutyl)cyclopentan-1-one (10). The ketone alcohol **8** (320 mg, 0.97 mmol) was dissolved in THF (15 mL) and cooled in an ice-water bath under argon. To this reaction mixture was added *N*,*O*-di(benzyloxycarbonyl)hydroxylamine (293 mg, 0.97 mmol), followed by the addition of PPh_3 (417 mg, 1.5 mmol). The resulting mixture was stirred in ice-water bath for 15 min. After this time the DIPAD (344 mg, 1.5 mmol) in 2 mL THF was added via a syringe pump over 40 min. After addition, the mixture was stirred for another 1.5 h in ice-water bath. Concentration followed by flash chromatography (2–10% EtOAc/hexane) afforded 530 mg (90%) of compound **10** as an oil. $R_f = 0.42$ (5:1 hexane/EtOAc). ^1H NMR (400 MHz, CDCl_3) δ 1.08 (m, 21H), 1.40 (m, 3H), 1.62 (m, 3H), 1.82 (m, 1H), 2.10 (m, 1H), 2.24 (dd, $J = 6.9, 18.3$ Hz, 1H), 2.41 (m, 1H), 2.57 (dd, $J = 6.5, 18.2$ Hz, 1H), 3.68 (t, $J = 6.9$ Hz, 1H), 4.49 (m, 1H), 5.20 (s, 2H), 5.26 (s, 2H), 7.36 (m, 10H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 12.4, 18.4, 24.9, 27.2, 30.3, 39.4, 48.8, 49.0, 50.9, 68.7, 69.2, 71.6, 128.3, 128.7, 128.8, 128.9, 129.1, 129.3, 134.6, 135.9, 154.7, 156.1, 218.3; IR (neat) 1791, 1740 cm^{-1} ; MS (ESI) m/z 612 ($\text{M}^+ + 1$); HRMS calcd for $\text{C}_{34}\text{H}_{53}\text{N}_2\text{O}_7\text{Si}(\text{M}^+ + \text{NH}_4^+)$: 629.3622, found 629.3646. $[\alpha]_D^{25} +32.02$ (c 1.03, CHCl_3).

(5a*S*,7*R*)-(-)-7-Triisopropylsiloxy-2,3,4,5,5a,6,7,8-octahydrocyclopent[b]-azepine-N-oxide (11). Compound **10** (250 mg, 0.41 mmol) was dissolved in EtOH (3.5 mL) and 10% Pd-C (28 mg) was added. The resulting mixture was hydrogenated at atmosphere (balloon) for 6 h. Concentration followed by chromatography (10% EtOAc/hexane, followed by 1-2% MeOH/CHCl₃) gave 120 mg (90%) of nitron **11** as an oil. R_f = 0.28 (20:1 MeOH/CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 1.05 (m, 21H), 1.57-2.31 (complex, 8H), 2.72 (m, 2H), 2.99 (dd, J = 5.5, 19.6 Hz, 1H), 3.99-4.30 (m, 2H), 4.38 (m, 1H); ¹³C NMR (100.6 MHz, CDCl₃) δ 12.4, 18.4, 25.9, 30.0, 33.0, 41.4, 41.9, 44.0, 66.7, 71.0, 156.3; IR (neat) 1636 cm⁻¹; MS (EI) m/z 326 (M^+ +1); HRMS calcd for C₁₈H₃₆NO₂Si (M^+ +1): 326.2515, found 326.2514. [α]_D -50.75 (c 1.30, CHCl₃). This nitron was used immediately in the next step.

(2*R*, 10*S*)-(+)-2-Triisopropylsilyloxy-octahydro-quinolizin-4-one (3a). *Method A.* To the keto azide **9** (300 mg, 0.85 mmol) in CH₂Cl₂ (15 mL) at 0 °C was added freshly distilled TiCl₄ (0.23 mL, 2.12 mmol) over 5 min. The reaction mixture was

allowed to warm to room temperature over 20 min, and then heated to 45 °C for 11 h. After cooling, 5 mL 15% aqueous NaOH was carefully added and the mixture stirred for an additional 30 min. The pale yellow solution was extracted into CH₂Cl₂ (300 mL), dried (anhyd MgSO₄), and concentrated to a pale yellow oil. Chromatography (25% EtOAc/hexane) gave 218 mg (79%) of lactams **3a** and **3b** as a (1:1.3) mixture of diastereoisomers. A second chromatographic separation (10% EtOAc/hexane) afforded analytical samples of **3a** and **3b**. For lactam **3a**: R_f = 0.73 (50% EtOAc/hexane). ¹H NMR (400 MHz, CDCl₃) δ 1.04-1.10 (m, 21H), 1.26-1.83 (m, 7H), 2.16 (m, 1H), 2.32-2.40 (m, 2H), 2.72 (ddd, J = 2.8, 7.6, 16.5 Hz, 1H), 3.18 (m, 1H), 4.01 (m, 1H), 4.71-4.75 (m, 1H); ¹³C NMR (100.6 MHz, CDCl₃) δ 12.6, 18.4, 24.8, 25.6, 34.9, 40.9, 42.5, 43.4, 54.8, 65.0, 168.7; IR (neat) 2941, 2865, 1650 cm⁻¹; MS (CI) m/z 326 (M^+ +1), 282; HRMS calcd for C₁₈H₃₆NO₂Si (M^+ +1): 326.2515, found 326.2534; [α]_D +14.2° (c 0.13, CHCl₃). For lactam **3b**: R_f = 0.71 (50% EtOAc/hexane). ¹H NMR (400 MHz, CDCl₃) δ 0.99-1.05 (m, 21H), 1.27 (m, 1H), 1.42 (m, 2H), 1.63 (m, 1H), 1.70 (m, 2H), 1.80 (m, 1H), 1.98 (m, 1H), 2.38-2.54 (m, 3H), 3.34 (m, 1H), 4.26 (m, 1H), 4.73 (m, 1H); ¹³C NMR (100.6 MHz, CDCl₃) δ 12.1, 17.9, 24.5, 25.3, 33.9, 38.3, 41.7, 42.0, 52.8, 63.8, 167.8; IR (neat) 2933, 2862, 1648 cm⁻¹; MS (CI) m/z 326 (M^+ +1), 282; HRMS calcd for C₁₈H₃₆NO₂Si (M^+ +1): 326.2515, found 326.2508; [α]_D -9.5 (c 0.09, CHCl₃).

Method B. A degassed mixture of nitron **11** (60 mg, 0.5 mmol) in CH₃CN/toluene (4:1, 20 mL) was photolyzed in a capped quartz tube (254 nm, Merry-Go-Round apparatus) for 2 h. Concentration followed by chromatography (10%

EtOAc/hexane, followed by 1-2% MeOH/CHCl₃) afforded 36 mg (60%) of **3a** isolated as an oil.

4-(1-Aza-4-triisopropylsilyloxy-bicyclo[4.4.0]dec-2-yl)-1,2-dimethoxybenzene

(12). To a dry flask containing anhydrous CeCl₃ (182 mg, 0.740 mmol) was added a solution of lactam **3a** (60 mg, 0.185 mmol) in 2 mL dry THF. After stirring for an hour at room temperature, the reaction mixture was cooled to 0 °C, and a solution of Grignard reagent of 4-bromoveratrole in THF (1.5 mL, 1.48 mmol) was added dropwise. The mixture was allowed to warm to room temperature and stirred overnight. It was then cooled to 0 °C and NaBH₃CN (70 mg, 1.11 mmol) followed by glacial acetic acid (0.25 mL) were added. After stirring for an hour, Et₂O (50 mL) was added and the mixture was washed with 15% aq KOH (2 × 5 mL). The organic layer was dried (Na₂SO₄), concentrated and purified by chromatography (10% ethyl acetate/hexanes) to afford 70 mg (85%) of **12** as a clear oil. ¹H NMR (400 MHz, CDCl₃) δ 1.01 (m, 21H), 1.21 (m, 2H), 1.36-1.69 (m, 7H), 1.83-1.96 (m, 3H), 2.64 (m, 1H), 2.86 (dd, *J* = 4.0, 12.0 Hz, 1H), 3.77 (m, 1H), 3.86 (s, 3H), 3.89 (s, 3H), 6.75-6.80 (m, 3H); ¹³C NMR (100.6 MHz, CDCl₃) δ 12.4, 18.1, 24.7, 26.2, 33.8, 43.6, 45.8, 53.1, 55.9, 56.0, 61.0, 68.4, 69.1, 110.4, 110.5, 119.6, 119.7, 137.2, 147.8; IR (neat) 2935, 2863, 2786, 2747 cm⁻¹; MS (CI) *m/z*

448 ($M^+ + 1$), 282; HRMS calcd for $C_{26}H_{46}NO_3Si$ ($M^+ + 1$): 448.3247, found 448.3263; $[\alpha]_D - 48.6$ (c 0.15, $CHCl_3$).

2-*epi*-Lasubine II (13). To the amine **12** (53 mg, 0.118 mmol) in 5 mL dry THF was added 1.0 M TBAF in THF (0.12 mL, 0.118 mmol) at 0 °C. The mixture was allowed to warm to room temperature and stirred for 3 hours. Concentration and chromatography (10% MeOH/ CH_2Cl_2) afforded 30 mg of **13** (87%) as a clear oil. 1H NMR (400 MHz, $CDCl_3$) δ 1.24-2.02 (complex, 14H), 2.64 (m, 1H), 2.84 (dd, $J = 4.0, 12.0$ Hz, 1H), 3.73 (m, 1H), 3.86 (s, 3H), 3.89 (s, 3H), 6.75-6.80 (m, 3H). ^{13}C NMR (100.6 MHz, 50 °C, $CDCl_3$) δ 24.6, 26.1, 33.8, 42.9, 45.4, 52.8, 55.8, 55.9, 60.8, 68.0, 68.4, 110.7, 111.3, 119.6, 137.1, 148.1, 149.3; IR (neat) 3366, 2934, 2856, 2792 cm^{-1} ; MS (CI) m/z 292 ($M^+ + 1$), 164, 154, 126, 96, 84; HRMS calcd for $C_{17}H_{26}NO_3$ ($M^+ + 1$): 292.1913, found: 292.1934. $[\alpha]_D - 65.5$ (c 1.0, $CHCl_3$); $[\alpha]_D - 56.9$ (c 1.0, MeOH). Lit. $[\alpha]_D - 44.4$ (c 2.1, MeOH).⁹

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S15

F2 - Acquisition Parameters
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 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

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 F1 4001.30 Hz
 F2P -1.000 ppm
 F2 -400.13 Hz
 PPMCM 0.55000 ppm/cm
 HZCM 220.07150 Hz/cm

Currt
 NAME
 EXPN1
 PROCN1

F2 - Acquisition Parameters
 Date_ 970608
 Time 13.46
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 2
 SWH 23148.146 Hz
 FIDRES 0.353213 Hz
 AQ 1.4156276 sec
 RG 4096
 DW 21.600 usec
 DE 4.50 usec
 TE 300.0 K
 d11 0.0300000 sec
 d12 0.0000200 sec
 PL13 18.00 dB
 D1 0.05000000 sec
 CPOPRG2 waltz16
 PCPD2 100.00 usec
 SF02 400.1316005 MHz
 NUC2 13C
 PL2 -6.00 dB
 PL12 18.00 dB
 P1 6.90 usec
 DE 4.50 usec
 SF01 100.6232933 MHz
 NUC1 13C
 PL1 -6.00 dB

F2 - Processing parameters
 SI 32768
 SF 100.6127734 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 220.000 ppm
 F1 22134.61 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PPMCM 11.50000 ppm/cm
 HZCM 1157.04688 Hz/cm

F2 - Acquisition Parameters
Date_ 970619
Time 22.27
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 322.5
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 7.70 usec
DE 4.50 usec
SFO1 400.132007 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 400.1300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

Cur
NAME
EXP
PROC

F2 - Acquisition Parameters
Date_ 970619
Time 22.52
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 694
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 4096
DW 21.600 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
d12 0.0000200 sec
PL13 18.00 dB
D1 0.05000000 sec
PCPDG2 waltz16
PCPD2 100.00 usec
SFO2 400.1316005 MHz
NUC2 13C
PL2 -6.00 dB
PL12 18.00 dB
P1 6.90 usec
DE 4.50 usec
SFO1 100.6232933 MHz
NUC1 13C
PL1 -6.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127720 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04688 Hz/cm

S17

F2 - Acquisition Parameters
Date_ 970630
Time 22.46
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 322.5
DM 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 7.70 usec
DE 4.50 usec
SF01 400.132007 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 400.1300088 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

Current
NAME
EXPNO
PROCNO

F2 - Acquisition Parameters
Date_ 970630
Time 23.18
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 4096
DM 21.600 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
d12 0.0000200 sec
PL13 18.00 dB
D1 0.05000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SF02 400.1316005 MHz
NUC2 13C
PL2 -6.00 dB
PL12 18.00 dB
P1 6.90 usec
DE 4.50 usec
SF01 100.6232933 MHz
NUC1 13C
PL1 -6.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127699 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04688 Hz/cm

S18

4

Date_ 970617
 Time 23.27
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 181
 DW 104.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.0000000 sec
 P1 7.70 usec
 DE 4.50 usec
 SF01 400.1320007 MHz
 NUC1 1H
 PL1 -6.00 dB

F2 - Processing parameters
 SI 16384
 SF 400.1300095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P -1.000 ppm
 F2 -400.13 Hz
 PPMCM 0.55000 ppm/cm
 HZCM 220.07150 Hz/cm

Current
 NAME
 EXPNO
 PROCNO

F2 - Acquisition Parameters
 Date_ 970617
 Time 23.52
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 740
 DS 2
 SWH 23148.148 Hz
 FIDRES 0.353213 Hz
 AQ 1.4156276 sec
 RG 4096
 DW 21.600 usec
 DE 4.50 usec
 TE 300.0 K
 d11 0.0300000 sec
 d12 0.0000200 sec
 PL13 18.00 dB
 D1 0.05000000 sec
 CPDPRG2 waltz16
 PCPD02 100.00 usec
 SF02 400.1316005 MHz
 NUC2 13C
 PL2 -6.00 dB
 PL12 18.00 dB
 P1 6.90 usec
 DE 4.50 usec
 SF01 100.6232933 MHz
 NUC1 13C
 PL1 -6.00 dB

F2 - Processing parameters
 SI 32768
 SF 100.6127741 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 219.587 ppm
 F1 22093.27 Hz
 F2P -10.485 ppm
 F2 -1054.88 Hz
 PPMCM 11.50358 ppm/cm
 HZCM 1157.40747 Hz/cm

F2 - Acquisition Parameters
 Date_ 970618
 Time 20.10
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TO 2930
 SOLVENT CDCl₃
 NS 16
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 161.3
 DW 104.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 7.70 usec
 DE 4.50 usec
 SFO1 400.1320007 MHz
 NUC1 1H
 PL1 -6.00 dB

F2 - Processing parameters
 SI 16384
 SF 400.1300095 MHz
 WDW EK
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P -1.000 ppm
 F2 -400.13 Hz
 PPMCM 0.55000 ppm/cm
 HZCM 220.07150 Hz/cm

F2 - Acquisition Parameters
 Date_ 970618
 Time 20.53
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TO 65536
 SOLVENT CDCl₃
 NS 1024
 DS 2
 SWH 23148.148 Hz
 FIDRES 0.353213 Hz
 AQ 1.4156276 sec
 RG 1625.5
 DW 21.600 usec
 DE 4.50 usec
 TE 300.0 K
 d11 0.0300000 sec
 d12 0.0000200 sec
 PL13 18.00 dB
 Q1 0.05000000 sec
 CPOPRG2 waltz16
 PCPD2 100.00 usec
 SFO2 400.1315005 MHz
 NUC2 1H
 PL2 -6.00 dB
 PL12 18.00 dB
 P1 6.90 usec
 DE 4.50 usec
 SFO1 100.6232933 MHz
 NUC1 13C
 PL1 -6.00 dB

F2 - Processing parameters
 SI 32768
 SF 100.6127699 MHz
 WDW EK
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 220.000 ppm
 F1 22134.81 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PPMCM 11.50000 ppm/cm
 HZCM 1157.04688 Hz/cm

S20

F2 - Acquisition Parameters
Date_ 970731
Time 3.24
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 181
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
P1 7.70 usec
DE 4.50 usec
SFO1 400.132007 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 400.1300089 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07152 Hz/cm

F2 - Acquisition Parameters
Date_ 970731
Time 4.14
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 812.7
DW 21.600 usec
DE 4.50 usec
TE 300.0 K
d11 0.0300000 sec
d12 0.0000200 sec
PL13 18.00 dB
D1 0.05000000 sec
CPDPRG2 waltz16
PCPD2 100.00 usec
SFO2 400.1316005 MHz
NUC2 1H
PL2 -6.00 dB
PL12 18.00 dB
P1 6.50 usec
DE 4.50 usec
SFO1 100.6232933 MHz
NUC1 13C
PL1 -6.00 dB

F2 - Processing parameters
SI 32768
SF 100.6127791 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.000 ppm
F2 -100.613 Hz
PPMCM 11.50000 ppm/cm
HZCM 1157.04700 Hz/cm

S21

F2 - Acquisition Parameters

Date_	970801
Time	17.03
INSTRUM	drx400
PROBHD	5 mm Multinu
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	4789.272 Hz
FIDRES	0.146157 Hz
AQ	3.4210291 sec
RG	256
DW	104.400 usec
DE	4.50 usec
TE	300.0 K
D1	1.00000000 sec
P1	7.70 usec
DE	4.50 usec
SFO1	400.132007 MHz
NUC1	1H
PL1	-6.00 dB

F2 - Processing parameters

SI	16384
SF	400.1300095 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
F1P	10.000 ppm
F1	4001.30 Hz
F2P	-1.000 ppm
F2	-400.13 Hz
PPMCM	0.55000 ppm/cm
HZCM	220.07150 Hz/cm

C
N
E
P

F2 - Acquisition Parameters

Date_	970801
Time	17.33
INSTRUM	drx400
PROBHD	5 mm Multinu
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	2
SWH	23148.148 Hz
FIDRES	0.353213 Hz
AQ	1.4156276 sec
RG	2896.3
DW	21.500 usec
DE	4.50 usec
TE	300.0 K
d11	0.0300000 sec
d12	0.0000200 sec
PL13	18.00 dB
D1	0.05000000 sec
CPDPRG2	waltz16
PCPD2	100.00 usec
SFO2	400.1316005 MHz
NUC2	13C
PL2	-6.00 dB
PL12	18.00 dB
P1	6.90 usec
DE	4.50 usec
SFO1	100.6232933 MHz
NUC1	13C
PL1	-6.00 dB

F2 - Processing parameters

SI	32768
SF	100.6127756 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
F1P	220.000 ppm
F1	22134.81 Hz
F2P	-10.000 ppm
F2	-1006.13 Hz
PPMCM	11.50000 ppm/cm
HZCM	1157.04700 Hz/cm

S22

F2 - Acquisition Parameters
 Date_ 970806
 Time 21.37
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 161.3
 DW 104.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 7.70 usec
 DE 4.50 usec
 SFO1 400.1320007 MHz
 NUC1 1H
 PL1 -6.00 dB

F2 - Processing parameters
 SI 16384
 SF 400.1300098 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P -1.000 ppm
 F2 -400.13 Hz
 PPMCM 0.55000 ppm/cm
 HZCM 220.07152 Hz/cm

F2 - Acquisition Parameters
 Date_ 970806
 Time 21.48
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 976
 DS 2
 SWH 23148.148 Hz
 FIDRES 0.353213 Hz
 AQ 1.4156276 sec
 RG 16384
 DW 21.600 usec
 DE 4.50 usec
 TE 300.0 K
 d11 0.0300000 sec
 d12 0.0000200 sec
 PL13 18.00 dB
 D1 0.05000000 sec
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 SFO2 400.1316005 MHz
 NUC2 1H
 PL2 -6.00 dB
 PL12 18.00 dB
 P1 6.90 usec
 DE 4.50 usec
 SFO1 100.6232933 MHz
 NUC1 13C
 PL1 -6.00 dB

F2 - Processing parameters
 SI 32768
 SF 100.6127748 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 220.000 ppm
 F1 22134.81 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PPMCM 11.50000 ppm/cm
 HZCM 1157.04700 Hz/cm

S23

Date_ 20030708
 Time 14.23
 INSTRUM drx400
 PROBHD 5 mm Multinucl
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 64
 DW 104.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.70 usec
 PL1 -6.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 11.000 ppm
 F1 4401.43 Hz
 F2P -1.000 ppm
 F2 -400.13 Hz
 PPMCM 0.60000 ppm/cm
 HZCM 240.07800 Hz/cm

Current Data Parameters
 NAME II-251
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030708
 Time 14.28
 INSTRUM drx400
 PROBHD 5 mm Multinucl
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 202
 DS 2
 SWH 24154.590 Hz
 FIDRES 0.368570 Hz
 AQ 1.3566452 sec
 RG 32768
 DW 20.700 usec
 DE 4.50 usec
 TE 300.0 K
 D1 0.05000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 12.30 usec
 PL1 2.00 dB
 SFO1 100.6248025 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 RCPD2 100.00 usec
 PL2 0.00 dB
 PL12 18.00 dB
 PL13 18.00 dB
 SFO2 400.1316005 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 240.037 ppm
 F1 24150.82 Hz
 F2P -0.037 ppm
 F2 -3.77 Hz
 PPMCM 12.00374 ppm/cm
 HZCM 1207.72949 Hz/cm

S24

===== CHANNEL f1 =====
 Date_ 20030709
 Time 19.26
 INSTRUM drx400
 PROBHD 5 mm Multinucl
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 64
 DW 104.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.70 usec
 PL1 -6.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 11.000 ppm
 F1 4401.43 Hz
 F2P -1.000 ppm
 F2 -400.13 Hz
 PPMCM 0.60000 ppm/cm
 HZCM 240.07800 Hz/cm

Current Data Parameters
 NAME II-252
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030709
 Time 19.31
 INSTRUM drx400
 PROBHD 5 mm Multinucl
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 920
 DS 2
 SWH 23148.148 Hz
 FIDRES 0.353213 Hz
 AQ 1.4156276 sec
 RG 32768
 DW 21.600 usec
 DE 4.50 usec
 TE 300.0 K
 D1 0.05000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 12.30 usec
 PL1 2.00 dB
 SFO1 100.6232933 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 0.00 dB
 PL12 18.00 dB
 PL13 18.00 dB
 SFO2 400.1316005 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 215.000 ppm
 F1 21631.74 Hz
 F2P -5.000 ppm
 F2 -503.06 Hz
 PPMCM 11.00000 ppm/cm
 HZCM 1106.73999 Hz/cm

S25

Date 20030722
Time 9.20
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 64
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.70 usec
PL1 -6.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 11.000 ppm
F1 4401.43 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.60000 ppm/cm
HZCM 240.07800 Hz/cm

Current Data Parameters
NAME II-258
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date 20030722
Time 9.25
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 392
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 32768
DW 21.600 usec
DE 4.50 usec
TE 300.0 K
D1 0.05000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 12.30 usec
PL1 2.00 dB
SFO1 100.6233933 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 400.1316005 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 220.036 ppm
F1 -10.036 ppm
F2P -1009.73 Hz
F2 11.50359 ppm/cm
PPMCM 1157.40747 Hz/cm

S26

3b

Time 14.11
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zg30
 TD 32768
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 64
 DW 104.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 7.70 usec
 DE 4.50 usec
 SFO1 400.1320007 MHz
 NUC1 1H
 PL1 -6.00 dB

F2 - Processing parameters
 SI 16384
 SF 400.1300095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P -1.000 ppm
 F2 -400.13 Hz
 PPMCM 0.55000 ppm/cm
 HZCM 220.07150 Hz/cm

Current Data Parameters
 NAME vlg-5-37-spt2
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 970331
 Time 14.02
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 1024
 DS 2
 SWH 23148.148 Hz
 FIDRES 0.353213 Hz
 AQ 1.4156276 sec
 RG 2048
 DW 21.500 usec
 DE 4.50 usec
 TE 300.0 K
 d11 0.0300000 sec
 d12 0.0002000 sec
 PL13 18.00 dB
 D1 0.05000000 sec
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 SFO2 400.1316005 MHz
 NUC2 1H
 PL2 -6.00 dB
 PL12 18.00 dB
 P1 6.90 usec
 DE 4.50 usec
 SFO1 100.6232933 MHz
 NUC1 13C
 PL1 -6.00 dB

F2 - Processing parameters
 SI 32768
 SF 100.6127713 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 220.000 ppm
 F1 22134.81 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PPMCM 11.50000 ppm/cm
 HZCM 1157.04688 Hz/cm

S27

INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 4789.272 Hz
 FIDRES 0.146157 Hz
 AQ 3.4210291 sec
 RG 181
 DW 104.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 7.70 usec
 DE 4.50 usec
 SF01 400.1320007 MHz
 NUC1 1H
 PL1 -6.00 dB

F2 - Processing parameters
 S1 16384
 SF 400.1300092 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.000 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P -1.000 ppm
 F2 -400.13 Hz
 PPMCM 0.55000 ppm/cm
 HZCM 220.07150 Hz/cm

F2 - Acquisition Parameters
 Date_ 971019
 Time 20.40
 INSTRUM drx400
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 4619
 DS 2
 SWH 23148.148 Hz
 FIDRES 0.353213 Hz
 AQ 1.4156276 sec
 RG 2048
 DW 21.600 usec
 DE 4.50 usec
 TE 300.0 K
 d11 0.0300000 sec
 d12 0.0000200 sec
 PL13 18.00 dB
 D1 0.05000000 sec
 CPDPRG2 waltz16
 PCPD02 100.00 usec
 SF02 400.1316005 MHz
 NUC2 1H
 PL2 -6.00 dB
 PL12 18.00 dB
 P1 6.90 usec
 DE 4.50 usec
 SF01 100.6232933 MHz
 NUC1 13C
 PL1 -6.00 dB

F2 - Processing parameters
 S1 32768
 SF 100.6127692 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.000 cm
 F1P 220.000 ppm
 F1 22134.81 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PPMCM 11.50000 ppm/cm
 HZCM 1157.04688 Hz/cm

S28

13

F: 1H
 D: 1H
 T: 1H
 INSTRUM: spect
 PROBHD: 5 mm Multinu
 PULPROG: zg30
 TD: 32768
 SOLVENT: CDCl3
 NS: 8
 DS: 2
 SWH: 4789.272 Hz
 FIDRES: 0.146157 Hz
 AQ: 3.4210291 sec
 RG: 101.6
 DW: 104.400 usec
 DE: 4.50 usec
 TE: 300.0 K
 D1: 1.0000000 sec

===== CHANNEL f1 =====
 NUC1: 1H
 P1: 7.70 usec
 PL1: -6.00 dB
 SFO1: 400.1320007 MHz
 F2 - Processing parameters
 SI: 16384
 SF: 400.1300041 MHz
 WDW: EM
 SSB: 0
 LB: 0.30 Hz
 GB: 0
 PC: 1.00

1D NMR plot parameters
 CX: 20.00 cm
 FIP: 10.974 ppm
 F1P: 4391.14 Hz
 F2P: -0.995 ppm
 F2: -398.13 Hz
 PPMCM: 0.59846 ppm/cm
 HZCM: 239.46359 Hz/cm

F2 - Acquisition Parameters
 Date_: 20020221
 Time: 17.08
 INSTRUM: spect
 PROBHD: 5 mm BBO BB-1H
 PULPROG: zgpg30
 TD: 65536
 SOLVENT: CDCl3
 NS: 12486
 DS: 4
 SWH: 31445.541 Hz
 FIDRES: 0.479836 Hz
 AQ: 1.0420883 sec
 RG: 2048
 DW: 15.900 usec
 DE: 6.00 usec
 TE: 300.0 K
 D1: 0.10000000 sec
 d11: 0.03000000 sec
 d12: 0.00002000 sec

===== CHANNEL f1 =====
 NUC1: 13C
 P1: 6.50 usec
 PL1: 5.00 dB
 SFO1: 125.7719472 MHz
 ===== CHANNEL f2 =====
 CPDPRG2: waltz16
 NUC2: 1H
 P2PRG2: 95.00 usec
 PL2: -4.00 dB
 PL12: 19.00 dB
 PL13: 30.00 dB
 SFO2: 500.1325000 MHz

F1 - Acquisition parameters
 MD: 2
 TO: 256
 SFO1: 500.1325 MHz
 FIDRES: 23.475061 Hz
 SW: 12.016 ppm

F2 - Processing parameters
 SI: 65536
 SF: 125.7578008 MHz
 WDW: EM
 SSB: 0
 LB: 1.00 Hz
 GB: 0
 PC: 1.40

F1 - Processing parameters
 SI: 1024
 MC2: 0F
 SF: 500.1300000 MHz
 WDW: nm
 SSB: 0
 LB: 0.30 Hz
 GB: 0

1D NMR plot parameters
 CX: 20.00 cm
 CY: 12.23 cm
 FIP: 237.517 ppm
 F1P: 29869.63 Hz
 F2P: -12.539 ppm
 F2: -1376.91 Hz
 PPMCM: 12.50282 ppm/cm
 HZCM: 1572.32703 Hz/cm

START

1.986 2.034 1.740

8.919

9.414

GC analysis of
the crude nitrene photolysis product (3a) vs (3b)

RUN# 41

JAN 9, 1901 18:02:38

METHOD NAME: M:YZ1.MET

SAMPLE# 18

AREA%

RT	AREA	TYPE	WIDTH	AREA%
1.740	9929875	PB	.060	99.88045
1.986	449	PV	.021	.00452
2.034	3049	UB	.030	.03067
8.919	588	PV	.044	.00591
9.414	7804	PB	.036	.07850

TOTAL AREA=9.9418E+06
MUL FACTOR=1.0000E+00

STOP

S30

START

1.739

8.912

9.415

GC analysis of
a 1 : 2 mixture of 3a and 3b

STOP

RUN# 42 JAN 9, 1901 18:31:59

SAMPLE# 16

METHOD NAME: M:YZ1.MET

AREAX

RT	AREA	TYPE	WIDTH	AREAX
1.739	9853306	PB	.057	99.95184
8.912	3139	PB	.036	.03184
9.415	1609	PB	.040	.01632

TOTAL AREA=9.8580E+06

MUL FACTOR=1.0000E+00