Stereocontrolled Synthesis of Angularly-Substituted 1-Azabicyclic Rings by Cationic 2-Aza-Cope Rearrangements Driven by Methylene Transfer

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Supporting Information

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Part A. Schemes Showing Synthetic Routes, Reagents and Yields for the Synthesis of Compounds 16 and 20.**Scheme 1** **Scheme 2**

Part B. Experimental Procedures and Characterization Data for Key Compounds.

General Methods. All reactions were carried out a N₂ atmosphere. Tetrahydrofuran (THF), diethyl ether (Et₂O), dichloromethane (CH₂Cl₂), toluene (PhMe), dimethoxyethane (DME), hexanes, diisopropylethylamine (DIEA), benzene (PhH), dimethylformamide (DMF), pyridine (pyr), acetonitrile (MeCN) methanol (MeOH), and triethylamine (Et₃N) were degassed with argon and then passed through appropriate column(s) made by Glass Contours, Inc. to remove water and/or oxygen.¹ Other reagents and solvents were used as received from the manufacturer unless otherwise indicated. Concentrations were performed under reduced pressure (*ca.* 15 mmHg) using a Buchi rotary evaporator. Thin Layer Chromatography (TLC) was performed using EM Science Silica Gel 60 PF₂₅₄. Flash Chromatography was performed using Merck silica gel (0.040–0.063 mesh) unless otherwise indicated.

Instrumentation. ¹H and ¹³C NMR spectra were measured at 500 or 400 and 125 or 100 MHz, respectively, with either a Bruker Avance 500 or Bruker Avance 400 FT NMR spectrometer. Chemical shifts are reported as δ values in ppm. Coupling constants are reported in Hz and refer to apparent multiplicities and not true coupling constants. Multiplicity is indicated as follows: s (singlet); d (doublet); t (triplet); q (quartet); quint (quintet); m (multiplet); br s (broad singlet); br d (broad doublet); br t (broad triplet); br q (broad quartet); br quint (br quint). Infrared spectra (IR) were obtained on an ASI Applied Systems ReactIR 1000 and are reported in wavenumbers (cm⁻¹). Mass spectra and high-resolution mass spectra (HRMS) were obtained on a Waters-MicroMass Analytical 7070E (CI) spectrometer, a Thermo-Finnigan TraceMS (EI) spectrometer, a Waters-MicroMass AutoSpec E (FAB) spectrometer, or a Waters-MicroMass LCT (ES) spectrometer. Microanalyses were performed by Atlantic Microlabs, Atlanta, GA. In cases where appropriate, nOe signals (from nOesy spectra) are indicated in compound drawings.

(11-Methyl-1,5-dioxaspiro[5.5]undec-7-yl)acetaldehyde (s2). A solution of ketone **s1** (4.4 g, 29 mmol), 1,3-propanediol (7.1 g, 120 mmol), *p*-TsOH (0.55 g, 2.9 mmol) and PhH (57

¹Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, 15, 1518–1520.

mL) was placed in a 100 mL round bottom flask fitted with a Dean Stark trap and heated to reflux. After 16 h, the reaction mixture was cooled to 25 °C; washed with saturated aqueous sodium bicarbonate (3 X 25 mL), deionized water (3 X 25 mL) and brine (1 X 15 mL), dried (MgSO_4); filtered; and concentrated *in vacuo* to give a colorless oil. Purification of this residue on silica gel (3:17 Et_2O :pentane) provided 3.2 g (~14 mmol, ~49%) of a 1:7 mixture of unreacted **s2** and the desired ketal, which was used without further purification.

Ozone was bubbled through a -78 °C solution of the above mixture (1.7 g, 7.2 mmol), sodium bicarbonate (~0.5 g), MeOH (7.2 mL) and methylene chloride (36 mL) until a blue color persisted (15 minutes). This mixture was purged with oxygen until the reaction was colorless at which point triphenylphosphine (2.8 g, 11 mmol) and MeOH (36 mL) were added. The reaction was maintained at 25 °C for 1 h, filtered, concentrated *in vacuo* and the residue was purified on silica gel (3:40 Et_2O :pentane) to give 1.3 g (6.1 mmol, 85%; 41% over 2 steps) of a colorless oil: ^1H NMR (500 MHz, C_6D_6 , 348 K) δ 9.63 (s, 1H), 3.41–3.55 (m, 4H), 2.67 (ddd, J = 16.3, 5.7, 2.2 Hz, 1H), 2.39–2.46 (m, 1H), 2.24–2.32 (m, 1H), 2.06 (dd, J = 16.3, 6.8, 1.7 Hz, 1H), 1.22–1.55 (m, 7H), 0.90–1.00 (m, 4H); ^{13}C NMR (125 MHz, C_6D_6 , 348 K) 200.9, 100.6, 59.0, 58.9, 44.6, 35.1, 30.1, 29.8, 29.2, 25.9, 20.4, 14.0 ppm; IR (film) 2966, 2933, 2863, 2719, 1721 cm^{-1} ; HRMS (ES) calculated for $\text{C}_{12}\text{H}_{20}\text{O}_3$ ($\text{M} + \text{Na}^+$): 235.1310, found: 235.1315.

But-3-enyl-[2-(11-methyl-1,5-dioxaspiro[5.5]undec-7-yl)ethyl]amine (20). To a solution of aldehyde **s2** (0.54 g, 2.5 mmol), but-3-enylamine hydrochloride (0.55 g, 5.1 mmol) and 3 Å molecular sieves (1.3 g) was added sodium cyanoborohydride (0.40 g, 6.4 mmol) and the reaction was maintained at 25 °C. After 22 h, the reaction was quenched by the addition of aqueous sodium hydroxide (2 mL, 3 M), added to a slurry of Celite and PhH , concentrated to dryness *in vacuo* and the residue was purified on silica gel (deactivated with NH_4OH ; 0:100-1:99 MeOH:methylene chloride saturated with NH_4OH) to provide 0.50 g (1.9 mmol, 74%) of a colorless oil: ^1H NMR (500 MHz, DMSO, 428 K) δ 5.80–5.91 (m, 1H), 5.05 (d, J = 17.2 Hz, 1H), 4.99 (d, J = 10.3 Hz, 1H), 2.55–2.70 (m, 4H), 2.24–2.31 (m, 1H), 2.21 (q, J = 6.9 Hz, 2H), 2.11–2.17 (m, 1H), 1.71–2.10 (m, 3H), 1.60–1.66 (m, 2H), 1.48–1.58 (m, 2H), 1.27–1.44 (m, 7H), 0.92 (d, J = 6.7 Hz, 3H); ^{13}C NMR (125 MHz, DMSO, 428 K) 136.2, 114.0, 99.4, 57.2,

57.0, 47.7, 47.1, 34.0, 33.0, 30.4, 28.7, 27.1, 26.0, 24.2, 18.5, 12.6 ppm; IR (film) 3312, 2934, 2864, 2818 cm^{-1} ; HRMS (ES) calculated for $\text{C}_{16}\text{H}_{29}\text{NO}_2$ ($\text{M} + \text{H}^+$): 268.2277, found: 268.2279.

7-Allyl-1,5-dioxaspiro[5.5]undecane-7-carboxylic acid ethyl ester (s4). A mixture of keto-ester **s3** (5.0 g, 24 mmol), 1,3-propanediol (5.9 g, 95 mmol), *p*-toluenesulfonic acid (*p*-TsOH) (0.45 g, 2.4 mmol) and PhH (50 mL) was placed in a 250 mL round bottom flask fitted with a Dean Stark trap and heated to reflux. After 16 h, the reaction mixture was cooled to 25 °C and washed with saturated aqueous sodium bicarbonate (3 X 25 mL), deionized water (3 X 25 mL) and brine (1 X 15 mL), dried (MgSO_4), filtered and concentrated *in vacuo* to give a 6:4 mixture of the desired **s4** and unreacted **s3**. Purification of this residue on silica gel (1:19 Ethyl acetate (EtOAc):hexanes) provided 2.5 g (9.0 mmol, 38%) of a colorless oil: ^1H NMR (500 MHz, CDCl_3) δ 5.54–5.63 (m, 1H), 4.99 (d, $J = 17.0$ Hz, 1H), 4.94 (dd, $J = 10.1, 1.0$ Hz, 1H), 4.04–4.18 (m, 2H), 3.86–3.97 (m, 2H), 3.71–3.79 (m, 2H), 2.84 (dd, $J = 13.9, 5.8$ Hz, 1H), 2.29–2.36 (m, 1H), 2.25 (dd, $J = 13.9, 8.5$ Hz, 1H), 1.82–1.94 (m, 2H), 1.55–1.69 (m, 2H), 1.42–1.51 (m, 2H), 1.19–1.40 (m, 6H); ^{13}C NMR (125 MHz, CDCl_3) 173.3, 134.6, 117.2, 99.1, 59.9, 59.0, 58.9, 54.9, 34.5, 28.2, 25.1, 23.5, 21.5, 20.7, 14.2 ppm; IR (film) 3076, 2941, 2868, 1722 cm^{-1} ; analysis calculated for $\text{C}_{15}\text{H}_{24}\text{O}_4$: C, 67.14; H, 9.01; found: C, 67.13; H, 8.97. HRMS (ES) calculated for $\text{C}_{15}\text{H}_{24}\text{O}_4$ ($\text{M} + \text{Na}^+$): 291.1572, found: 291.1562.

(7-Allyl-1,5-dioxaspiro[5.5]undec-7-yl)methanol (s5). Solid lithium aluminum hydride (0.37 g, 9.7 mmol) was added in a single portion to a 0 °C solution of ester **s4** (1.5 g, 5.7 mmol) in THF (22 mL) and the reaction was allowed to warm to 25 °C. After 16 h, the reaction was cooled to 0 °C and water (0.37 mL), 2.5 M sodium hydroxide (0.37 mL) and water (0.37 mL) were added dropwise in a sequential manner with vigorous stirring. The resulting slurry was stirred for 2 h, filtered through a pad of Celite and concentrated *in vacuo* to provide 1.1 g (5.0 mmol, 89%) of a colorless oil: ^1H NMR (500 MHz, C_6D_6) δ 5.98–6.09 (m, 1H), 5.19 (dq, $J =$

17.0, 1.1 Hz, 1H), 5.11 (dd, $J = 10.0, 0.7$ Hz, 1H), 4.17 (br d, $J = 11.1$ Hz, 1H), 3.61 (td, $J = 8.9, 3.4$ Hz, 1H), 3.45 (t, $J = 11.9$ Hz, 1H), 3.40 (t, $J = 12.5$ Hz, 1H), 3.25–3.34 (m, 3H), 2.90 (dd, $J = 13.7, 6.3$ Hz, 1H), 2.28 (dd, $J = 13.8, 8.5$ Hz, 1H), 1.87–1.95 (m, 1H), 1.47–1.64 (m, 2H), 1.19–1.45 (m, 6H), 0.56 (br d, $J = 13.2$ Hz, 1H); ^{13}C NMR (125 MHz, C_6D_6) 136.1, 118.0, 103.1, 65.7, 59.09, 59.07, 46.2, 33.8, 28.3, 26.0, 22.9, 22.5, 21.0 ppm; IR (film) 3524 (br), 3073, 2937, 2868 cm^{-1} ; analysis calculated for $\text{C}_{13}\text{H}_{22}\text{O}_3$: C, 68.99; H, 9.80; found: C, 68.75; H, 9.82. EIMS calculated for $\text{C}_{13}\text{H}_{22}\text{O}_3$ (M^+) 226.16, found: 226.12.

7-Allyl-7-benzyloxymethyl-1,5-dioxaspiro[5.5]undecane (s6). A solution of alcohol **s5** (1.0 g, 4.6 mmol) in DMF (1.7 mL) was added dropwise to a 0 °C stirred suspension of sodium hydride (NaH) (0.37 g, 9.2 mmol) in DMF (6.6 mL) and the resulting mixture was maintained at 0 °C for 5 min. At this point, a solution of benzyl bromide (3.2 g, 18 mmol), tetrabutylammonium iodide (0.051 g, 0.14 mmol) in DMF (3.5 mL) was added dropwise and the reaction mixture was allowed to warm to 25 °C for 2 h at which point complete conversion was observed by thin layer chromatography (TLC). The reaction was poured into 0 °C half-saturated aqueous ammonium chloride (100 mL), extracted with Et_2O (3 X 50 mL), and the combined organic layers were washed with water (1 X 50 mL) and brine (1 X 25 mL), dried (MgSO_4), filtered, concentrated *in vacuo* and the residue was purified on silica gel (3:97 Et_2O :pentane) to provide 1.1 g (3.5 mmol, 76%) of a colorless oil: ^1H NMR (500 MHz, C_6D_6) δ 7.31–7.36 (m, 2H), 7.13–7.24 (m, 2H), 7.06–7.11 (m, 1H), 6.42–6.54 (m, 1H), 5.22 (br d, $J = 17.1$ Hz, 1H), 5.14 (br d, $J = 9.1$ Hz, 1H), 4.48 (d, $J = 12.1$, 1H), 4.37 (d, $J = 12.1$ Hz, 1H), 3.84 (d, $J = 9.2$ Hz, 1H), 3.78 (d, $J = 9.1$ Hz, 1H), 3.40–3.56 (m, 4H), 2.92 (dd, $J = 14.0, 5.4$ Hz, 1H), 2.65 (dd, $J = 13.2, 8.5$ Hz, 1H), 1.93–2.04 (m, 2H), 1.64–1.77 (m, 2H), 1.51–1.59 (m, 1H), 1.43–1.50 (m, 2H), 1.32–1.41 (m, 2H), 0.63 (d, $J = 13.0$ Hz, 1H); ^{13}C NMR (125 MHz, C_6D_6) 140.3, 139.0, 128.8, 128.0, 127.8, 115.4, 100.9, 73.9, 72.6, 59.0, 58.9, 47.1, 38.9, 30.5, 26.1, 23.6, 22.8, 22.0 ppm; IR (film) 2934, 2864 cm^{-1} ; analysis calculated for $\text{C}_{20}\text{H}_{28}\text{O}_3$: C, 75.91; H, 8.92; found: C, 75.80; H, 8.94. HRMS (ES) calculated for $\text{C}_{20}\text{H}_{28}\text{O}_3$ ($\text{M} + \text{Na}^+$): 339.1936, found: 339.1926.

(7-Benzyloxymethyl-1,5-dioxaspiro[5.5]undec-7-yl)acetaldehyde (s7). Ozone was bubbled through a $-78\text{ }^{\circ}\text{C}$ solution of **s6** (1.0 g, 3.2 mmol), sodium bicarbonate (~ 0.5 g), MeOH (3.2 mL) and methylene chloride (16 mL) until a faint blue color was observed (3.5 minutes). This mixture was purged with oxygen until the reaction was colorless, at which point triphenylphosphine (1.3 g, 4.8 mmol) and MeOH (16 mL) were added. The reaction was maintained at $25\text{ }^{\circ}\text{C}$ for 1 h, filtered, concentrated *in vacuo* and the residue was purified on silica gel (2:25 Et₂O:pentane) to give 0.80 g (2.5 mmol, 78%) of a colorless oil: ¹H NMR (500 MHz, CDCl₃) δ 9.80 (dd, $J = 3.9, 2.8$ Hz, 1H), 7.25–7.36 (m, 5H), 4.54 (d, $J = 12.2$ Hz, 1H), 4.48 (d, $J = 12.2$ Hz, 1H), 3.92 (br t, $J = 12.6$ Hz, 2H), 3.69–3.79 (m, 4H), 2.49–2.58 (m, 2H), 2.27 (br d, $J = 13.9$ Hz, 1H), 1.84–1.99 (m, 2H), 1.38–1.69 (m, 6H), 1.19–1.33 (m, 1H); ¹³C NMR (125 MHz, CDCl₃) 203.3, 138.7, 128.3, 127.42, 127.38, 99.2, 73.3, 70.2, 59.0, 58.7, 47.8, 46.4, 29.8, 25.2, 23.2, 21.8, 20.6 ppm; IR (film) 2934, 2864, 1702 cm⁻¹; HRMS (ES) calculated for C₁₉H₂₆O₄ (M + Na⁺): 341.1729, found: 341.1742.

[2-(7-Benzyloxymethyl-1,5-dioxaspiro[5.5]undec-7-yl)ethyl]but-3-enylamine (16). To a solution of aldehyde **s7** (0.78 g, 2.4 mmol), but-3-enylamine hydrochloride (0.52 g, 4.9 mmol), 3 Å molecular sieves (1.2 g) was added sodium cyanoborohydride (0.38 g, 6.1 mmol) and the reaction was maintained at $25\text{ }^{\circ}\text{C}$. After 22 h, the reaction was quenched by the addition of aqueous sodium hydroxide (2.0 mL, 3 M), added to a slurry of Celite and PhH, concentrated to dryness *in vacuo* and the residue was purified on silica gel (deactivated with NH₄OH; 0:100-1:99 MeOH:methylene chloride saturated with NH₄OH) to provide 0.80 g (2.1 mmol, 88%) of a colorless oil: ¹H NMR (500 MHz, CDCl₃) δ 7.21–7.35 (m, 5H), 5.76 (ddt, $J = 17.1, 10.2, 6.8$ Hz, 1H), 5.06 (dd, $J = 17.1, 1.3$ Hz, 1H), 5.01 (d, $J = 10.2$ Hz, 1H), 4.48 (q, $J = 12.2$ Hz, 2H), 3.89–3.98 (m, 2H), 3.66–3.76 (m, 2H), 3.56–3.61 (m, 2H), 2.85 (td, $J = 10.2, 5.4$ Hz, 1H), 2.61–2.69 (m, 3H), 2.23 (q, $J = 6.9$ Hz, 2H), 2.07–2.16 (m, 1H), 1.85–2.01 (m, 2H), 1.74–1.83 (m, 1H), 1.55–1.72 (m, 3H), 1.34–1.48 (m, 5H), 1.26 (d, $J = 13.3$ Hz, 1H); ¹³C NMR (125 MHz, CDCl₃)

139.2, 136.7, 128.1, 127.2, 127.1, 116.0, 100.4, 73.2, 71.6, 58.6, 58.5, 48.9, 45.9, 45.3, 34.4, 33.1, 29.9, 25.4, 22.8, 21.9, 20.8 ppm; IR (film) 3320, 3069, 3030, 2934, 2864 cm^{-1} ; HRMS (ES) calculated for $\text{C}_{23}\text{H}_{35}\text{NO}_3$ ($\text{M} + \text{Na}^+$): 396.2515, found: 396.2522.

$\text{C}_{18}\text{H}_{24}\text{O}_4$
Mol. Wt.: 304.38

(1,5-Dioxaspiro[5.5]undec-7-yl)acetic acid benzyl ester (8). To a 0 °C solution of ketone **7** (3.85 g, 15.6 mmol), 1,3 propanediol (21.8 mL, 391 mmol) and trimethylorthoformate (8.60 mL, 78.2 mmol) in acetonitrile (160 mL) was added scandium triflate (77.0 mg, 0.156 mmol). This was maintained at 0 °C for 30 min at which point the reaction was quenched with saturated NaHCO_3 (~5 mL), extracted with Et_2O (3 X 75 mL) and the combined organic extracts washed with water (3 X 50 mL) and brine (25 mL), dried (MgSO_4), filtered, concentrated *in vacuo* and purified on silica gel (1:9 Et_2O :Pentane) to provide 4.20 g (13.7 mmol, 88 %) of a colorless oil: ^1H NMR (500 MHz, CDCl_3) δ 7.25–7.36 (m, 5H), 5.24 (d, J = 12.4 Hz, 1H), 5.06 (d, J = 12.4 Hz, 1H), 3.97 (td, J = 11.8, 2.8 Hz, 1H), 3.85 (td, J = 11.7, 2.7 Hz, 1H), 3.71–3.77 (m, 1H), 3.61–3.67 (m, 1H), 2.87–2.94 (m, 1H), 2.61 (br d, J = 13.4 Hz, 1H), 2.08–2.18 (m, 2H), 1.82–1.92 (m, 1H), 1.53–1.64 (m, 3H), 1.23–1.41 (m, 4H), 1.16 (td, J = 12.6, 3 Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) 173.8, 136.3, 128.3, 128.0, 127.8, 98.0, 65.7, 59.0, 58.8, 42.5, 33.9, 28.3, 27.7, 25.4, 24.7, 22.1 ppm; IR (film) 2935, 2861, 1733 cm^{-1} ; analysis calculated for $\text{C}_{18}\text{H}_{24}\text{O}_4$: C, 71.03; H, 7.95. Found: C, 70.99; H, 8.05. MS (EI) calculated for $\text{C}_{18}\text{H}_{24}\text{O}_4$ (M^+): 304.1, found: 304.1.

$\text{C}_{15}\text{H}_{25}\text{NO}_3$
Mol. Wt.: 267.36

N-But-3-enyl-2-(1,5-dioxaspiro[5.5]undec-7-yl)acetamide (9). A solution of benzyl ester **8** (1.00 g, 3.29 mmol) and palladium hydroxide (0.100 g, 30 wt% on carbon) in EtOAc (33.0 mL) was evacuated and back filled three times with nitrogen, then repeated three times with hydrogen. The reaction was maintained under a 1 atm hydrogen atmosphere with vigorous stirring with vigorous stirring then was filtered through a 4.5 μ polyethylene frit, rinsed with

EtOAc (40 mL), concentrated *in vacuo* to a viscous oil and the unpurified acid was used directly in the subsequent reaction.

To a flask containing this sample of the crude acid was added diisopropylethylamine (0.574 mL, 3.29 mmol), 1-but-3-enyl amine hydrochloride (0.354 g, 3.29 mmol), a catalytic amount of 4-dimethylaminopyridine (DMAP, ~0.005 g), 1-(3-dimethylaminopropyl)-3-ethylcarbodiimide hydrochloride (EDCI) (0.694 g, 3.62 mmol) and methylene chloride (6.6 mL). After 20 h at 25 °C, the reaction was concentrated directly onto celite (*in vacuo*) and was purified on silica gel (EtOAc) to provide 0.703 g (2.63 mmol, 79.9 % over two steps) of a colorless oil: ¹H NMR (500 MHz, CDCl₃) δ 5.94 (br s, 1H), 5.73 (ddt, *J* = 17.1, 10.2, 6.8 Hz, 1H), 5.00–5.09 (m, 2H), 4.01 (td, *J* = 11.8, 2.8 Hz, 1H), 3.87 (td, *J* = 11.7, 2.8 Hz, 1H), 3.70–3.80 (m, 2H), 3.23–3.33 (m, 2H), 2.72 (dd, *J* = 14.5, 4.6 Hz, 1H), 2.54–2.63 (m, 1H), 2.22 (q, *J* = 6.8 Hz, 2H), 1.95–2.06 (m, 1H), 1.82–1.93 (m, 2H), 1.50–1.65 (m, 3H), 1.10–1.40 (m, 5H); ¹³C NMR (125 MHz, CDCl₃) 173.4, 135.5, 116.8, 98.6, 58.9, 42.8, 40.5, 38.3, 36.5, 33.8, 28.7, 28.0, 25.6, 24.7, 22.2 ppm; IR (film) 3292 (br), 3078, 2933, 2861, 1640 cm⁻¹; analysis calculated for C₁₅H₂₅NO₃: C, 67.38; H, 9.42; N, 5.24. Found: C, 67.09; H, 9.47; N, 5.16. MS (ES) calculated for C₁₅H₂₅NO₃ (M + H⁺): 268.19, found: 268.20.

[2-(1,5-Dioxaspiro[5.5]undec-7-yl)-ethyl]but-3-enylamine (10). Lithium aluminum hydride (19.6 mL of a 1 M solution in diethyl ether, 19.6 mmol) was added dropwise to a stirred 0 °C solution of amide **9** (1.56 g, 5.8 mmol) in diethyl ether (19.3 mL) and the reaction allowed to warm to 25 °C. After 16 h at 25 °C, the reaction was cooled to 0 °C and water (0.745 mL), 2.5 M sodium hydroxide (0.745 mL) and water (0.745 mL) were added dropwise in a sequential manner with vigorous stirring. The resulting slurry was stirred for 2 h, filtered through a pad of celite and concentrated *in vacuo* to provide 1.45 g (5.72 mmol, 99 %) of a colorless oil: ¹H NMR (500 MHz, CDCl₃) δ 5.75 (ddt, *J* = 17.1, 10.2, 6.8 Hz, 1H), 5.04 (dd, *J* = 17.1, 0.8 Hz, 1H), 4.99 (dd, *J* = 10.2, 0.9 Hz, 1H), 3.98 (td, *J* = 11.2, 2.9 Hz, 1H), 3.86 (td, *J* = 11.3, 2.9 Hz, 1H), 3.71–3.77 (m, 2H), 2.60–2.69 (m, 3H), 2.55 (ddd, *J* = 10.8, 10.5, 6.6 Hz, 1H), 2.44 (br d, *J* = 2.3 Hz, 1H), 2.21 (q, *J* = 6.8 Hz, 2H), 1.92–1.99 (m, 1H), 1.82–1.92 (m, 1H), 1.48–1.62 (m, 4H), 1.17–1.40 (m, 7H); ¹³C NMR (125 MHz, CDCl₃) 136.6, 116.2, 99.0, 58.93, 58.86, 48.9, 48.8, 34.32, 34.26, 28.5, 28.1, 28.0, 25.6, 24.3, 22.3 ppm; IR (film) 2933, 2860, 2811 cm⁻¹; analysis calculated for C₁₅H₂₇NO₂: C, 71.10; H, 10.74; N, 5.53. Found: C, 70.88; H, 10.79; N, 5.27. HRMS (ES) calculated for C₁₅H₂₇NO₂ (M + Na⁺): 276.1939, found: 276.1932.

Typical procedure for methylene transfer driven cationic 2-aza-Cope rearrangements. Preparation of 7a-Allyloctahydroindole-1-carboxylic acid benzyl ester (11). A solution of amine ketal **10** (0.297 g, 1.17 mmol), morpholine (10.2 μ L, 0.117 mmol) and TFA (90.4 μ L, 1.17 mmol) was heated to 120 °C with vigorous stirring for *ca.* ~15 s until the formation of a homogenous mixture was observed. To this mixture was added dimedone (0.411 g, 2.93 mmol) and the reaction was heated to 120 °C for 2 h 20 min to give complete conversion to the 2-aCr product as determined by ¹H NMR of the crude reaction mixture.

After cooling to 25 °C, the reaction mixture was dissolved in chloroform (7.8 mL) and transferred to a larger reaction vessel. To this flask was added saturated aqueous Na₂CO₃ (3.9 mL), water (3.9 mL) and benzyl chloroformate (0.418 mL, 2.93 mmol). The reaction was vigorously stirred at 25 °C for 13 h, partitioned from brine (10 mL) and the aqueous layers were extracted with methylene chloride (3 X 30 mL). The combined organic extracts were dried (MgSO₄), filtered, concentrated *in vacuo* and the residue was purified on silica gel (3:40 Et₂O:pentane) to provide 0.278 g (0.927 mmol, 79.2%) of a colorless oil: ¹H NMR (500 MHz, DMSO, 398 K) δ 7.26–7.39, (m, 5H), 5.67–5.78 (m, 1H), 4.99–5.13 (m, 4H), 3.59 (ddd, *J* = 10.9, 8.3, 4.4 Hz, 1H), 3.30 (dt, *J* = 10.9, 7.9 Hz, 1H), 2.73 (dd, *J* = 14.0, 6.4 Hz, 1H), 2.46 (dd, *J* = 14.0, 7.9 Hz, 1H), 2.07–2.15 (m, 1H), 1.87–1.94 (m, 2H), 1.68–1.82 (m, 2H), 1.58–1.67 (m, 1H), 1.28–1.53 (m, 5H); ¹³C NMR (125 MHz, DMSO, 398 K) 152.9, 136.8, 133.8, 127.4, 126.7, 126.6, 116.4, 64.8, 63.0, 45.4, 39.4, 38.7, 31.3, 25.4, 24.7, 20.9, 20.5 ppm; IR (film) 3073, 3034, 3007, 2930, 2860, 1695 cm⁻¹; analysis calculated for C₁₉H₂₅NO₂: C, 76.22; H, 8.42; N, 4.68; found: C, 75.96; H, 8.51; N, 4.72. HRMS (ES) calculated for C₁₉H₂₅NO₂N (M + Na⁺): 322.1783, found: 322.1794.

This reaction was also examined on a 0.30 g scale in refluxing PhMe for six hours to provide **11** in 50% yield.

Syntheses of **13**, **15**, **17**, **19**, **21**, **22**, **24**, **25**, **27**, **28**, **30** and **31** were carried out in a similar manner; characterization data for these products follows.

6a-Allylhexahydro-cyclopenta[*b*]pyrrole-1-carboxylic acid benzyl ester (13). Isolated as a colorless oil in 86% yield on a 0.26 g scale. Changes from the standard procedure include heating **12** to 120 °C for 40 min: ^1H NMR (500 MHz, DMSO, 388 K) δ 7.26–7.38 (m, 5H), 5.74 (ddt, J = 16.4, 11.2, 7.4 Hz, 1H), 5.07–5.14 (m, 2H), 4.99–5.04 (m, 2H), 3.42–3.53 (m, 2H), 2.80 (dd, J = 13.5, 7.1 Hz, 1H), 2.43–2.49 (m, 1H), 2.36 (dd, J = 13.8, 7.5 Hz, 1H), 2.11–2.21 (m, 1H), 1.92 (dq, J = 12.6, 8.0 Hz, 1H), 1.82 (dq, J = 12.6, 7.4 Hz, 1H), 1.70 (dt, J = 13.4, 6.6 Hz, 1H), 1.49–1.63 (m, 3H), 1.37–1.44 (m, 1H); ^{13}C NMR (125 MHz, DMSO, 388 K) 152.6, 136.8, 134.2, 127.5, 126.8, 126.6, 116.5, 73.1, 64.9, 47.3, 47.0, 40.6, 37.3, 31.2, 27.8, 24.1 ppm; IR (film) 3073, 3034, 2949, 2868, 1695 cm^{-1} ; analysis calculated for $C_{18}H_{23}NO_2$: C, 75.76; H, 8.12; N, 4.91; found: C, 75.74; H, 8.23; N, 4.97. HRMS (ES) calculated for $C_{18}H_{23}NO_2$ ($M + Na^+$): 308.1627, found: 308.1624.

This reaction was also carried out on a 0.20 g scale at 80 °C for three hours to provide **13** in 96% yield.

7a-Allyloctahydro-[1]pyrindine-1-carboxylic acid benzyl ester (15). Isolated as a colorless oil in 85% yield on a 0.26 g scale. Changes from the standard procedure include heating **14** to 120 °C for 1 h 40 min: ^1H NMR (500 MHz, DMSO, 298 K) δ 7.28–7.38 (m, 5H), 5.80 (ddt, J = 16.8, 10.2, 7.4 Hz, 1H), 4.96–5.09 (m, 4H), 3.82 (ddd, J = 13.4, 6.3, 3.1 Hz, 1H), 2.95 (ddd, J = 11.4, 11.3, 4.7 Hz, 1H), 2.62 (dd, J = 13.7, 7.4 Hz, 1H), 2.25 (dd, J = 13.7, 7.5 Hz, 1H), 2.16 (dt, J = 13.2, 8.5 Hz, 1H), 1.94–2.05 (m, 2H), 1.68–1.78 (m, 1H), 1.57–1.67 (m, 2H), 1.32–1.56 (m, 4H); ^{13}C NMR (125 MHz, DMSO, 298 K) 155.3, 137.1, 134.7, 128.3, 127.6, 127.5, 117.5, 66.6, 65.7, 41.6, 40.9, 40.1, 37.6, 28.7, 24.0, 21.1, 20.7 ppm; IR (film) 3073, 3034, 2945, 2868, 1695 cm^{-1} ; analysis calculated for $C_{19}H_{25}NO_2$: C, 76.22; H, 8.42; N, 4.68; found: C, 76.12; H, 8.44; N, 4.68. HRMS (ES) calculated for $C_{19}H_{25}NO_2$ ($M + Na^+$): 322.1783, found: 322.1776.

7a-Allyl-3a-benzyloxymethyloctahydroindole-1-carboxylic acid benzyl ester (17).

Isolated as a colorless oil in 80% yield on a 0.31 g scale. Changes from the standard procedure include heating **16** to 130 °C and the addition of 4 equiv of dimedone portionwise in 1.5 h intervals over 4.5 h (addition of 1 equiv initially, then addition of 1 equiv every 1.5 h) at which point the reaction was maintained a further 3 h prior to cbz protection using 4.0 equiv of benzyl chloroformate: ¹H NMR (500 MHz, DMSO, 408 K) δ 7.26–7.41 (m, 10 H), 5.86 (ddt, J = 17.1, 10.0, 7.2 Hz, 1H), 5.09 (q, J = 12.7 Hz, 2H), 4.90–4.98 (m, 2H), 4.50–4.57 (m, 2H), 3.65 (d, J = 9.6 Hz, 1H), 3.44–3.59 (m, 3H), 2.68–3.77 (m, 1H), 2.43 (dd, J = 14.4, 6.9 Hz, 1H), 2.35 (dd, J = 14.3, 7.7 Hz, 1H), 1.90 (dt, J = 12.5, 8.5 Hz, 1H), 1.79 (dt, J = 13.5, 3.7 Hz, 1H), 1.40–1.55 (m, 6H), 1.33 (ddd, J = 13.6, 11.5, 5.1 Hz, 1H), 1.18–1.28 (m, 1H); ¹³C NMR (125 MHz, DMSO, 408 K) 153.0, 137.9, 136.8, 134.6, 127.3, 127.2, 126.6, 126.51, 126.46, 126.4, 115.6, 72.6, 72.3, 66.4, 64.7, 47.1, 44.0, 40.0, 31.0, 30.4, 30.0, 20.9, 20.7 ppm; IR (film) 2934, 2895, 2860, 1702 cm⁻¹; analysis calculated for C₂₇H₃₃NO₃: C, 77.29; H, 7.93; N, 3.34; found: C, 77.32; H, 7.90; N, 3.45. HRMS (ES) calculated for C₂₇H₃₃NO₃ (M + Na⁺): 442.2358, found: 442.2355.

7a-(1-Methylallyl)octahydroindole-1-carboxylic acid benzyl ester (19). Isolated as an impure (ca. ~90% purity) colorless oil in <15% yield on a 0.10 g scale. Changes from the standard procedure include heating **18** to 130 °C and the addition of 4 equiv of dimedone portionwise at 1.5 h intervals over 4.5 h (addition of 1 equiv initially, then addition of 1 equiv every 1.5 h) at which point the reaction was maintained a further 46 h prior to Cbz protection using 2.5 equiv of benzyl chloroformate. Subsequent purification provided **19** in 80-90% purity (partial characterization data follows): ¹H NMR (500 MHz, DMSO, 388 K) δ 7.27–7.38 (m, 5H), 5.87 (ddd, J = 17.2, 10.2, 8.5 Hz, 1H), 5.02–5.10 (m, 2H), 4.99 (d, J = 17.0 Hz, 1H), 4.94 (d, J = 10.2 Hz, 1H), 3.63 (ddd, J = 11.0, 8.5, 6.0 Hz, 1H), 3.32 (ddd, J = 11.0, 8.5, 6.1 Hz, 1H), 2.87

(br s, 1H), 2.78 (br quint, $J = 7.2$ Hz, 1H), 2.15–2.28 (m, 2H), 1.84–1.92 (m, 1H), 1.74–1.80 (m, 1H), 1.38–1.62 (m, 4H), 1.23–1.36 (m, 4H), 0.95 (d, $J = 7.0$ Hz, 1H); ^{13}C NMR (125 MHz, DMSO, 388 K) 153.1, 140.5, 127.5, 127.2, 126.8, 126.7, 113.8, 68.5, 66.6, 64.8, 45.7, 44.2, 38.3, 28.0, 26.6, 20.5, 20.0, 14.8 ppm; HRMS (ES) calculated for $\text{C}_{20}\text{H}_{27}\text{NO}_2$ ($\text{M} + \text{Na}^+$): 336.1939, found: 336.1943.

7a-Allyl-7-methyloctahydroindole-1-carboxylic acid benzyl esters (**21** and **22**).

Isolated as a 11:1 mixture of **21** and **22** in 82% yield on a 0.29 g scale. Changes from the standard procedure include heating **20** to 130 °C and the addition of 4 equiv of dimedone portion wise at 1.5 h intervals over 4.5 h (addition of 1 equiv initially, then addition of 1 equiv every 1.5 h) at which point the reaction was maintained a further 6 h prior to Cbz protection using 4.0 equiv of benzyl chloroformate.

21: ^1H NMR (500 MHz, DMSO, 408 K) δ 7.26–7.37 (m, 5H) 5.64 (dddd, $J = 18.7, 10.2, 8.5, 5.4$ Hz, 1H), 5.00–5.10 (m, 4H), 3.52 (ddd, $J = 11.3, 9.3, 2.5$ Hz, 1H), 3.34–3.42 (m, 1H), 3.28 (dd, $J = 13.9, 4.2$ Hz, 1H), 3.35–3.43 (m, 1H), 3.25–3.31 (m, 1H), 2.19–2.27 (m, 2H), 1.86–1.97 (m, 2H), 1.66–1.73 (m, 1H), 1.53–1.63 (m, 2H), 1.39–1.52 (m, 3H), 1.14–1.25 (m, 1H), 1.00 (d, $J = 6.9$ Hz, 1H); ^{13}C NMR (125 MHz, DMSO, 408 K) 153.3, 136.8, 134.1, 127.3, 126.6, 126.5, 116.3, 66.2, 64.7, 45.2, 39.5, 35.9, 30.7, 30.5, 23.8, 23.4, 19.0, 15.9 ppm; IR (film) 3069, 3034, 2957, 2930, 2887, 2856, 1702 cm^{-1} ; analysis calculated for $\text{C}_{20}\text{H}_{27}\text{NO}_2$: C, 76.64; H, 8.68; N, 4.47; found: C, 76.77; H, 8.78; N, 4.45. HRMS (ES) calculated for $\text{C}_{20}\text{H}_{27}\text{NO}_2$ ($\text{M} + \text{Na}^+$): 336.1939, found: 336.1948.

22: ^1H NMR (500 MHz, d_5 -nitrobenzene, 388 K) δ 7.44–7.49 (m, 2H), 7.33–7.39 (m, 2H), 7.26–7.33 (m, 1H), 5.73–5.84 (m, 1H), 5.19–5.34 (m, 2H), 5.03–5.11 (m, 2H), 3.69 (t, $J = 9.8$ Hz, 1H), 3.39 (br q, $J = 10.2$ Hz, 1H), 3.12 (br d, $J = 14.9$ Hz, 1H), 2.54 (dd, $J = 14.0, 8.6$ Hz, 1H), 2.35–2.42 (m, 1H), 2.23–2.31 (m, 1H), 1.77–1.90 (m, 2H), 1.54–1.67 (m, 4H), 1.25–1.52 (m, 2H), 0.96 (d, $J = 7.1$, 3H); ^{13}C NMR (125 MHz, d_5 -nitrobenzene, 388 K) 154.1, 138.0, 135.2, 128.2, 127.53, 127.46, 116.9, 66.0, 65.8, 47.6, 40.3, 40.0, 36.3, 29.0, 27.8, 24.7, 17.1, 15.1 ppm;

IR (film) 3069, 3034, 2930, 1702 cm^{-1} ; HRMS (ES) calculated for $\text{C}_{20}\text{H}_{27}\text{NO}_2$ ($\text{M} + \text{Na}^+$): 336.1939, found: 336.1944.

8a-Allyloctahydroquinoline-1-carboxylic acid benzyl esters (24 and 25). Isolated as a 1.2:1 mixture of **24** and **25** in 81% yield on a 0.30 g scale. Changes from the standard procedure include heating **23** to 120 $^{\circ}\text{C}$ for 100 min.

24: ^1H NMR (500 MHz, DMSO 348 K) δ 7.28–7.39 (m, 5H), 5.77 (ddt, $J = 17.2, 10.0, 7.1$ Hz, 1H), 5.00–5.09 (m, 4H), 3.79 (dt, $J = 13.3, 4.6$ Hz, 1H), 3.23 (ddd, $J = 14.9, 10.8, 4.4$ Hz, 1H), 2.68 (d, $J = 7.3$ Hz, 2H), 2.34 (ddd, $J = 13.0, 8.7, 3.8$ Hz, 1H), 1.63–1.85 (m, 4H), 1.46–1.61 (m, 5H), 1.27–1.46 (m, 3H); ^{13}C NMR (125 MHz, DMSO, 348 K) 154.9, 136.9, 133.8, 127.8, 127.1, 126.9, 116.9, 65.3, 60.1, 40.7, 38.3, 35.7, 32.2, 27.3, 23.1, 21.9, 21.8, 20.6 ppm; IR (film) 3069, 3034, 3007, 2934, 2864, 1698 cm^{-1} ; analysis calculated for $\text{C}_{14}\text{H}_{27}\text{NO}_2$: C, 76.64; H, 8.68; N, 4.47; found: C, 76.47; H, 8.66; N, 4.42. HRMS (ES) calculated for $\text{C}_{20}\text{H}_{27}\text{NO}_2$ ($\text{M} + \text{Na}^+$): 336.1939, found: 336.1949.

25: ^1H NMR (500 MHz, DMSO) δ 7.27–7.38 (m, 5H), 5.70 (ddt, $J = 17.1, 10.0, 7.1$ Hz, 1H), 5.10 (br d, $J = 17.1$ Hz, 1H), 4.93–5.03 (m, 3H), 3.94–4.01 (m, 1H), 3.08 (ddd, $J = 14.3, 11.2, 3.2$ Hz, 1H), 2.88 (br d, $J = 13.2$ Hz, 1H), 2.64 (dd, $J = 14.7, 7.4$ Hz, 1H), 2.41 (dd, $J = 14.8, 6.9$ Hz, 1H), 1.52–1.64 (m, 5H), 1.31–1.50 (m, 5H), 1.18–1.29 (m, 2H); ^{13}C NMR (125 MHz, DMSO) 155.1, 137.3, 134.0, 128.3, 127.6, 127.4, 117.2, 65.5, 62.5, 45.2, 41.2, 35.0, 30.7, 29.7, 26.6, 25.3, 24.8, 22.7 ppm; IR (film) 3069, 3034, 3007, 2930, 2864, 1702 cm^{-1} ; analysis calculated for $\text{C}_{14}\text{H}_{27}\text{NO}_2$: C, 76.64; H, 8.68; N, 4.47; found: C, 76.63; H, 8.73; N, 4.47. HRMS (ES) calculated for $\text{C}_{20}\text{H}_{27}\text{NO}_2$ ($\text{M} + \text{Na}^+$): 336.1939, found: 336.1946.

8a-Allyloctahydrocyclohepta[*b*]pyrrole-1-carboxylic acid benzyl esters (27** and **28**).**

Isolated as a 9:1 mixture of **27** and **28** in 64% yield on a 0.19 g scale. Changes from the standard procedure include heating **26** to 130 °C and the addition of 4 equiv of dimedone portionwise in 1.5 h intervals over 4.5 h (addition of 1 equiv initially, then addition of 1 equiv every 1.5 h) at which point the reaction was maintained a further 6 h prior to cbz protection using 4.0 equiv of benzyl chloroformate. Although these compounds were separated by HPLC (EtOAc:hexanes), **28** was not recovered. Accordingly, characterization data for **27** and partial characterization data for the 9:1 mixture of **27** and **28** are included:

Data for the 9:1 mixture of **27** and **28**: ^1H NMR (500 MHz, DMSO, 388 K) δ 7.29–7.40 (m, 5H), 5.80–5.89 (m, 0.1H), 5.71–5.80 (m, 0.9H), 5.01–5.14 (m, 3.8H), 4.94–5.00 (m, 0.2H), 3.50–3.59 (m, 1H), 3.33–3.42 (m, 1H), 2.69–2.79 (m, 1H), 2.25–2.45 (m, 2H), 2.08–2.17 (m, 0.9H), 1.85–2.01 (m, 1.8H), 1.73–1.83 (m, 0.4H), 1.60–1.71 (m, 4.1H), 1.42–1.58 (m, 4.8H), 1.26–1.39 (m, 0.3H); analysis calculated for $\text{C}_{14}\text{H}_{27}\text{NO}_2$: C, 76.64; H, 8.68; N, 4.47; found: C, 76.77; H, 8.68; N, 4.55.

Data for **27** alone: ^1H NMR (500 MHz, DMSO, 388 K) δ 7.27–7.37 (m, 5H), 5.70–5.80 (m, 1H), 4.99–5.11 (m, 4H), 3.54 (ddd, $J = 10.7, 8.0, 6.2$ Hz, 1H), 3.36 (dt, $J = 10.7, 7.3$ Hz, 1H), 2.72 (dd, $J = 13.7, 6.8$ Hz, 1H), 2.30–2.40 (m, 2H), 2.08–2.15 (m, 1H), 1.94–2.01 (m, 1H), 1.85–1.93 (m, 1H), 1.61–1.71 (m, 4H), 1.41–1.57 (m, 5H); ^{13}C NMR (125 MHz, DMSO, 388 K) 152.6, 136.8, 133.9, 127.4, 126.6, 126.5, 116.6, 68.2, 64.7, 46.1, 45.3, 42.1, 34.2, 29.5, 29.2, 27.8, 24.2, 22.0 ppm; IR (film) 3073, 3034, 2926, 2860 cm^{-1} ; HRMS (ES) calculated for $\text{C}_{14}\text{H}_{27}\text{NO}_2$ ($\text{M} + \text{H}^+$): 314.2120 found: 314.2117.

9a-Allyldecahydrocyclohepta[*b*]pyridine-1-carboxylic acid benzyl esters (30** and **31**).**

Isolated as a 3.5:1 mixture of **30** and **31** in 71% yield on a 0.27 g scale. Changes from the standard procedure include heating **29** to 130 °C and the addition of 4 equiv of dimedone portionwise in 1.5 h intervals over 4.5 h (addition of 1 equiv initially, then addition of 1 equiv every 1.5 h) at which point the reaction was maintained a further 6 h prior to cbz protection using 4.0 equiv of benzyl chloroformate. Separation of **30** and **31** by preparative HPLC (EtOAc:hexanes) provided both **30** and **31** analytically pure:

30: ^1H NMR (500 MHz, C_6D_6) δ 7.25–7.29 (m, 2H), 7.10–7.14 (m, 2H), 7.03–7.06 (m, 1H), 5.92 (dddd, $J = 17.0, 10.1, 7.9, 6.8$ Hz, 1H), 5.03–5.18 (m, 4H), 3.76 (ddd, $J = 13.1, 6.3, 4.7$ Hz, 1H), 3.17–3.24 (m, 1H), 2.91 (dd, $J = 13.9, 6.7$ Hz, 1H), 2.53–2.64 (m, 2H), 2.19 (ddd, $J = 14.7, 11.5, 1.8$ Hz, 1H), 1.48–1.65 (m, 5H), 1.16–1.47 (m, 8H); ^{13}C NMR (125 MHz, C_6D_6) 156.8, 138.4, 135.9, 129.0, 128.7, 128.3, 117.6, 67.1, 64.4, 42.8, 41.4, 40.9, 37.0, 31.2, 29.5, 27.30, 27.24, 22.8, 21.7 ppm; IR (film) 3073, 3034, 2930, 2864, 1702 cm^{-1} ; HRMS (ES) calculated for $C_{15}H_{29}NO_2$ ($M + H^+$): 328.2277, found: 328.2274.

31: ^1H NMR (500 MHz, C_6D_6) δ 7.24–7.28 (m, 2H), 7.09–7.13 (m, 2H), 7.03–7.07 (m, 1H), 5.94 (ddt, $J = 17.0, 10.0, 6.9$ Hz, 1H), 5.03–5.17 (m, 4H), 3.67 (dt, $J = 14.2, 5.5$ Hz, 1H), 3.30 (ddd, $J = 13.2, 8.8, 4.5$ Hz, 1H), 2.74 (ddd, $J = 13.3, 10.5, 1.5$ Hz, 1H), 2.61 (dd, $J = 14.8, 7.5$ Hz, 1H), 2.49 (dd, $J = 14.8, 6.6$ Hz, 1H), 1.95 (dd, $J = 14.8, 7.7$ Hz, 1H), 1.72–1.86 (m, 2H), 1.57–1.67 (m, 2H), 1.48–1.56 (m, 1H), 1.28–1.47 (m, 4H), 1.13–1.27 (m, 3H), 1.04–1.11 (m, 1H); ^{13}C NMR (125 MHz, C_6D_6) 155.7, 138.4, 135.7, 129.0, 128.7, 128.3, 117.2, 67.0, 65.2, 44.7, 41.2, 39.5, 38.0, 32.1, 29.9, 29.2, 28.5, 25.0 ppm; IR (film) 3069, 3011, 2926, 2864, 1702 cm^{-1} ; HRMS (ES) calculated for $C_{15}H_{29}NO_2$ ($M + Na^+$): 328.2277, found: 350.2090.

cis-7a-Allyloctahydroindole (36) A solution of protected amine **11** (0.020 g, 0.067 mmol) in *t*-BuOH (3.3 mL) and THF (13.3 mL) was cooled to $-78\text{ }^{\circ}\text{C}$ and ammonia (*ca.* 60 mL) was doubly distilled into the reaction mixture. Solid sodium metal (*ca.* 0.04 g, 1.6 mmol) was added portionwise until a dark blue color persisted, and the reaction was maintained at $-78\text{ }^{\circ}\text{C}$ for 10 min. At this point the reaction was quenched by the addition of *i*-PrOH (10 mL) and solid ammonium chloride (0.010 g) and warmed to $25\text{ }^{\circ}\text{C}$ while the ammonia was allowed to boil off. The resulting solution was filtered through Celite, concentrated *in vacuo*, and purified on silica gel (CH₂Cl₂, saturated with NH₄OH) to provide 0.0073 g (0.44 mmol, 66%) of a colorless oil: ¹H NMR (500 MHz, CDCl₃) δ 5.83 (dddd, $J = 14.9, 10.2, 7.9, 7.0\text{ Hz}$, 1H), 5.02–5.51 (m, 2H), 2.91–3.05 (m, 2H), 2.26 (dd, $J = 13.9, 7.9\text{ Hz}$, 1H), 2.12 (dd, $J = 13.9, 7.0\text{ Hz}$, 1H), 1.65–1.90 (m, 4H), 1.51–1.58 (m, 1H), 1.39–1.49 (m, 5H), 1.28–1.38 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) 134.8, 117.6, 61.7, 42.6, 42.3, 42.1, 31.2, 29.6, 26.5, 22.3, 21.9 ppm; IR (film) 3300 (br), 3076, 2930, 2860, 1729, 1664, 1640 cm⁻¹; HRMS (ES) calculated for C₁₁H₁₉N (M + H⁺): 166.1596, found: 166.1604.

cis-7a-Allyl-3a,7,7-trideuterooctahydroindole (d₃-36). A solution of amine ketal **32** (0.039 g, 0.16 mmol), dimethoxybenzene (0.10 mL of a 0.21 M solution in deuterated MeOH, 0.021 mmol), deuterated MeOH (0.31 mL) and trifluoroacetic acid (0.012 mL, 0.15 mmol) were heated in a sealed reaction vessel to $60\text{ }^{\circ}\text{C}$ for 16 h. To this was added dimedone (0.40 mL of a 1 M solution in deuterated MeOH, 0.40 mmol) and deuterated water (0.0097 mL, 0.49 mmol). This was sealed and heated to $110\text{ }^{\circ}\text{C}$ for 25.5 h. The reaction was concentrated *in vacuo* and purified on silica gel (1:1:98-1:4:95 NH₄OH:MeOH:methylene chloride) to give 0.018 g (65%) of a pale yellow oil: ¹H NMR (500 MHz, CDCl₃) δ 5.82 (ddt, $J = 17.1, 10.2, 7.8\text{ Hz}$, 1H), 5.01–5.09 (m, 2H), 2.91–3.03 (m, 2H), 2.25 (dd, $J = 13.9, 7.9\text{ Hz}$, 1H), 2.13 (dd, $J = 13.9, 7.0\text{ Hz}$, 1H), 1.91 (br s, 1H), 1.81–1.87 (m, 1H), 1.62–1.69 (m, 1H), 1.49–1.58 (m, 1H), 2.36–1.48 (m, 3H), 1.27–1.35 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) 134.78, 117.6, 61.5, 42.6, 42.2, 41.6 (t, $J_{CD} = 18.8\text{ Hz}$),

30.44 (m), 29.5, 26.4, 22.2, 21.7 ppm; IR (film) 3296 (br), 2926, 2856, 1640, 1459, 1413 cm^{-1} ; MS (ES) calculated for $\text{C}_{11}\text{H}_{17}\text{D}_3\text{N}$ ($\text{M} + \text{H}^+$): 169.18, found: 169.10.

7a-Allyl-7-methyloctahydroindole (s8). A solution of protected amine **21** (0.042 g, 0.13 mmol) in *t*-BuOH (3.4 mL) and THF (13 mL) was cooled to $-78\text{ }^{\circ}\text{C}$ and ammonia (*ca.* 60 mL) was doubly distilled into the reaction mixture. Solid sodium metal (*ca.* 0.04 g, 1.6 mmol) was added portionwise until a dark blue color persisted, and the reaction was maintained at $-78\text{ }^{\circ}\text{C}$ for 10 min. At this point, the reaction was quenched by the addition of MeOH (10 mL) and warmed to $25\text{ }^{\circ}\text{C}$ while the ammonia was allowed to boil off. The resulting solution was concentrated *in vacuo*, partitioned between 1N NaOH (20 mL) and CH_2Cl_2 (25 mL) and the organic layer was discarded. The organic layer was diluted with brine (15 mL) and extracted with Et_2O . The ethereal extracts were dried (Na_2SO_4), filtered and concentrated *in vacuo* to provide 0.0013 g (0.0073 mmol, 5.4%) of a colorless oil: ^1H NMR (500 MHz, C_6D_6) δ 5.96 (ddt, $J = 17.3, 9.8, 7.5\text{ Hz}$, 1H), 5.02–5.09 (m, 2H), 2.87 (td, $J = 10.5, 3.8\text{ Hz}$, 1H), 2.78 (ddd, $J = 17.8, 16.0, 7.3\text{ Hz}$, 1H), 2.09–2.20 (m, 2H), 1.80–1.87 (m, 1H), 1.61–1.69 (m, 1H), 1.50–1.56 (m, 2H), 1.27–1.49 (m, 6H), 0.95–1.05 (m, 1H), 0.89 (d, $J = 6.9\text{ Hz}$, 3H); ^{13}C NMR (125 MHz, C_6D_6) 135.8, 117.9, 64.4, 43.4, 41.2, 34.9, 34.8, 31.5, 29.3, 25.6, 21.5, 16.2 ppm; IR (film) 3327 (br), 2926, 2856 cm^{-1} ; HRMS (ES) calculated for $\text{C}_{12}\text{H}_{21}\text{N}$ ($\text{M} + \text{H}^+$): 180.1752, found: 180.1752.

0.91206
0.90606
0.90193
0.89806
0.89676
0.98481
1.25947
1.26885
1.28547
1.29843
1.30280
1.31644
1.31906
1.32773
1.33666
1.36305
1.37781
1.38736
1.39425
1.40555
1.41350
1.42305
1.42328
1.44558
1.45723
1.46557
1.47425
1.48331
1.49151
1.50194
1.51016
2.04100
2.04438
2.06005
2.06344
2.07358
2.07706
2.26843
2.28104
2.42617
2.64787
2.65236
2.65934
2.66381
2.68506
3.43875
3.44935
3.45828
3.47083
3.47685
3.48562
3.49605
3.50615
3.51413
4.46175

7.15984

ppm
9.63693

s2

Current Data Parameters
 USER Jester
 NAME 2A-V-95-1
 EXPNO 7
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20040428
 Time_ 12.21
 INSTRUM gm500
 PROBHD 5 mm broadband
 PULPROG zg30
 TD 81728
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.098043 Hz
 AQ 5.0988774 sec
 RG 181
 DW 62.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.10000000 sec
 ===== CHANNEL f1 =====
 NUC1 1H
 P1 11.50 usec
 PL1 -3.00 dB
 SFO1 500.1535010 MHz
 F2 - Processing parameters
 SI 65536
 SF 500.1459963 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 4.00
 LD NMR plot parameters
 HY 27.80 cm
 FID 10.00 cm
 F1P 5001.50 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 FREQM 0.43860 ppm/cm
 HZCM 219.36404 Hz/cm

s19

Current Data Parameters
USER jester
NAME ZA-V-95-1
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040428
Time 12.31
INSTRUM gn500
PROBHD 5 mm broadband
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 552
DS 4
SHE 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.081390 sec
RG 4096
DW 16.500 usec
DE 4.50 usec
TE 300.2 K
D1 0.2500000 sec
D11 0.0300000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 6.80 usec
PL1 0.00 dB
SFO1 125.7766019 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P2 80.00 usec
PL2 -3.00 dB
SFO2 500.153510 MHz

F2 - Processing parameters
SI 65536
SF 125.7627111 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
FIP 230.925 ppm
F1 29041.73 Hz
F2P -10.025 ppm
F2 -1261.30 Hz
PPM0 10.56816 ppm/cm
HZ0N 1329.08932 Hz/cm

20

Current Data Parameters
 USER Jester
 NAME ZA-V-97-1-1
 EXPNO 4
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20040614
 Time 19:37
 INSTRUM gnm300
 PROBRD 5 mm broadband
 PULPROG zgpg30
 TD 65536
 SFO1 125.7629896 MHz
 SOLVENT CDCl3
 NS 515
 DS 4
 SWH 30303.031 Hz
 FIDRES 0.462388 Hz
 AQ 1.0813940 sec
 RG 4096
 DW 16.500 usec
 DE 4.50 usec
 TE 300.0 K
 D1 0.25000000 sec
 D11 0.03000000 sec
 ===== CHANNEL f1 =====
 NUC1 13C
 P1 6.50 usec
 PL1 0.00 dB
 SFO1 125.7766019 MHz
 ===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 P2 80.00 usec
 PL2 -3.00 dB
 PL12 15.40 dB
 SFO2 500.1535010 MHz
 F2 - Processing parameters
 SI 65536
 SF 125.7629896 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 2.00
 1D NMR plot parameters
 CX 22.80 cm
 F1P 229.520 ppm
 F1 28865.08 Hz
 F2P -10.507 ppm
 F2 -1321.34 Hz
 PPM0M 10.52747 ppm/cm
 HZCM 1323.96594 Hz/cm

7.25997

5.62816
5.61577
5.6106
5.60802
5.59863
5.59496
5.59107
5.58165
5.57776
5.57408
5.56466
5.56166
5.55697
5.54462
5.50190
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4.92223
4.92040
4.16336
4.15603
4.14915
4.14182
4.12748
4.11287
4.09856
4.09115
4.08435
4.07700
3.95770
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3.93427
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3.91092
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3.88831
3.86572
3.78175
3.77827
3.77486
3.77117
3.76778
3.75863
3.75410
3.74996
3.74380
3.73956
3.72716
3.71628
2.85722
2.84551
2.82947
2.81783
2.26628
2.24923
2.23843
2.22138
1.91557
1.91005
1.90235
1.89887
1.88807
1.88204
1.87706
1.87110
1.86215
1.64235
1.58463
1.58018
1.57537
1.47886
1.47124
1.46236
1.45441
1.44886
1.44171
1.43720
1.37271
1.35185
1.33115
1.31127
1.30780
1.26803
1.24175
1.23874
1.22666
1.21243
1.19823
0.81984
0.80565

s4

Current Data Parameters
USER Jester
NAME 2A-V-75-2
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040708
Time_ 18.09
INSTRUM gm500
PROBHD 5 mm broadband
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 1
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0988774 sec
RG 20.2
DW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
===== CHANNEL f1 =====
NUC1 1H
P1 11.50 usec
PL1 -3.00 dB
SFO1 500.1535010 MHz
F2 - Processing parameters
SI 65536
SF 500.1500272 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
LD NMR plot parameters
SI 32768
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
FID 81728
F1F 4001.50 Hz
F2F 0.000 ppm
F2 0.00 Hz
PWCN 0.35088 ppm/cm
HZCN 175.49124 Hz/cm

s23

ppm
7
6
5
4
3
2
1

s4

s24

Current Data Parameters
NAME Z4-V15
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040708
Time_ 18.11
INSTRUM gn500
PROBHD 5 mm broadband
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 68
DS 4
SWH 30303.031 Hz
FIRES 0.462388 Hz
AQ 1.0613940 sec
RG 327.5
AQ 16.50 usec
RG 4.50 usec
TE 300.0 K
D1 0.25000000 sec
D11 0.03000000 sec
===== CHANNEL f1 =====
NUC1 13C
P1 6.80 usec
PL1 0.00 dB
SFO1 125.7766019 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
P2 3.00 dB
PL2 15.40 dB
SFO2 500.1559010 MHz
F2 - Processing parameters
SI 65536
SF 125.7628381 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
ID NMR plot parameters
CX 22.80 cm
FIP 229.520 ppm
F1 28865.05 Hz
F2 -10.507 ppm
F2 13211.94 Hz
F3 10.3247 ppm/cm
H2O 1323.9436 Hz/cm

s26

Current Data Parameters
 USER Jester
 NAME 2A-V-83-2
 EXPNO 2
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20040712
 Time 21:43
 INSTRUM gn300
 PROBHD 5 mm broadband
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 259
 DS 4
 SWH 30303.081 Hz
 SF 125.762788 MHz
 FIDRES 0.46238 Hz
 AQ 1.083580 sec
 RG 1024
 DW 16.500 usec
 DE 4.50 usec
 TE 300.0 K
 D1 0.25000000 sec
 D11 0.0300000 sec
 ===== CHANNEL f1 =====
 NUC1 13C
 P1 6.80 usec
 PL1 0.00 dB
 SF01 125.7766019 MHz
 ===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 P2 80.00 usec
 PL2 3.00 dB
 PL12 15.40 dB
 SF02 500.1355010 MHz
 F2 - Processing parameters
 SI 65536
 SF 125.762788 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 2.00
 ID NMR plot parameters
 CX 22.80 cm
 F1P 230.89 ppm
 F2P 2904.63 Hz
 F1 101.62 ppm
 F2 -1278.40 ppm
 FWHM 10.56616 ppm/cm
 HZCN 1329.08000 Hz/cm

ppm

150

100

50

0

ppm

Current Data Parameters
 USER Jester
 NAME 2A-V-90-1
 EXPNO 6
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040711
 Time_ 20.39
 INSTRUM omega500
 PROBHD 5 mm broadband
 PULPROG zg30
 TD 81728
 SOLVENT CDCl₃
 NS 8
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.098043 Hz
 AQ 5.098774 sec
 RG 256
 DW 62.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.1000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 13.00 usec
 PL1 -1.00 dB
 SF01 500.2235015 MHz

F2 - Processing parameters
 SI 65536
 SF 500.219999 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 4.00

LD NMR plot parameters
 FWHM 22.80 cm
 FID 81.00 cm
 F1P 4001.56 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPM0M 0.35088 ppm/cm
 HZCM 175.51579 Hz/cm

s6

```

Current Data Parameters
=====
NAME      2A-V-90-1
EXPNO     7
PROCNO    1

F2 - Acquisition Parameters
=====
Date_     20040711
Time      20.25
INSTRUM   omega500
PROBHD    5 mm broadband
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         1016
DS         4
SWH        30303.031 Hz
FIDRES     0.462388 Hz
AQ         1.0813940 sec
RG         5150.6
DM         16.500 usec
DE         4.50 usec
TE         300.0 K
D1         0.25000000 sec
D11        0.03000000 sec

===== CHANNEL f1 =====
NUC1       13C
P1         22.50 usec
PL1        -6.00 dB
SFO1       125.7942048 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
P2         80.00 usec
PL2        -1.00 dB
PL12       14.40 dB
SFO2       500.2230013 MHz

F2 - Processing parameters
=====
SI         65536
SF         125.780322 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         2.00

1D NMR plot parameters
=====
RG         327.680 cm
FIDRES     0.462388 Hz
P1         22.50 usec
PL1        -6.00 dB
PL12       14.40 dB
PL2        -1.00 dB
P2         80.00 usec
SFO1       125.780322 MHz
SFO2       500.2230013 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         2.00
H2COW      1332.08044 Hz/cm
  
```

9.80178
9.79613
9.78838

7.35262
7.35045
7.34750
7.33811
7.33588
7.33412
7.32663
7.32222
7.31674
7.31333
7.30745
7.30013
7.28632
7.28302
7.27845
7.26922
7.26002
7.25567

5.29605
4.55882
4.53453
4.48745
4.46315
3.94125
3.93627
3.91605
3.91112
3.89258
3.88753
3.77236
3.76910
3.76461
3.76152
3.74907
3.74593
3.73825
3.72626
3.72325
3.71975
3.71548
3.71233
3.69168
2.57432
2.56645
2.54485
2.53682
2.52981
1.95414
1.94363
1.93245
1.92813
1.91732
1.90652
1.88055
1.87585
1.84945
1.84210
1.66870
1.65962
1.64155
1.59901
1.51280
1.50565
1.48975
1.48113
1.47158
1.46151
1.45241
1.43556
1.40880
1.27395
1.27127
1.26864
1.24996
1.24746
1.24475
1.24214
1.22206
1.20802
0.88248

s7

s29

Current Data Parameters
USER jester
NAME 2A-V-96-2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040428
Time_ 12.04
INSTRUM gn500
PROBHD 5 mm broadband
PULPROG zg30
TD 81728
SOLVENT CDCl3T
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 181
DM 62.400 usec
DE 6.00 usec
TE 300.0 K
RB 0.1000000 sec
===== CHANNEL f1 =====
NUC1 1H
P1 11.50 usec
PL1 -3.00 dB
SFO1 500.1535010 MHz
F2 - Processing parameters
SI 65536
SF 500.1500274 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
AQ 12.00 cm
FID 10.00 cm
P1P 5001.50 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.43860 ppm/cm
HZCM 219.36406 Hz/cm

ppm

Current Data Parameters
USER: jester
NAME: ZA-V-96-2
EXPNO: 2
PROCNO: 1
F2 - Acquisition Parameters
Date_ 20040228
Time 12.08
INSTRUM: zgpg30
PROBHD: 5 mm broadband
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 72
DS: 4
SWH: 30303.031 Hz
FIDRES: 0.462288 Hz
AQ: 1.081394 sec
RG: 4096
DW: 16.500 usec
DE: 4.50 usec
TE: 300.0 K
D1: 0.25000000 sec
D11: 0.03000000 sec
===== CHANNEL f1 =====
NUC1: 13C
P1: 6.80 usec
PL1: 0.00 dB
SFO1: 125.7766019 MHz
===== CHANNEL f2 =====
CPDPRG2: waltz16
NUC2: 1H
P2: 80.00 usec
PL2: -3.00 dB
PL12: 15.40 dB
SFO2: 500.1355010 MHz
F2 - Processing parameters
SI: 65536
SF: 125.7628260 MHz
WDW: EM
SSB: 0
LB: 1.00 Hz
GB: 0
PC: 2.00
1D NMR plot parameters
CX: 22.89 cm
FIP: 230.015 ppm
F1: 28927.38 Hz
F2P: -10.938 ppm
F2: -1375.66 Hz
PPM0: 10.52747 ppm/cm
HZCM: 1323.96423 Hz/cm

s30

ppm

Current Data Parameters
 USER Jester
 NAME ZA-V-97-2-1
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040614
 Time 18.20
 INSTRON 95500
 PROBHD 5 mm broadband
 PULPROG zgpg30
 TD 65536
 SFO1 500.150277 MHz
 SOLVENT CDCl3
 NS 1
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.09843 Hz
 AQ 5.0998774 sec
 RG 40.3
 DW 62.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.10000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 11.50 usec
 PL1 -3.00 dB
 SFO1 500.150277 MHz

F2 - Processing parameters
 SI 65536
 SF 500.150277 MHz
 WDW EM
 SSF 0
 LB 0.30 Hz
 GB 0
 PC 4.00

1D NMR plot parameters
 CX 22.80 cm
 FIP 8.000 ppm
 FI 4001.20 Hz
 FZP 0.000 ppm
 FZ 0.00 Hz
 FFCN 0.35088 ppm/cm
 FZCN 175.45124 Hz/cm

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```

Current Data Parameters
=====
NAME      Jester
EXPNO     3
PROCNO    1

F2 - Acquisition Parameters
=====
Date_     20040614
Time      18.23
INSTRUM   spect
PROBHD    5 mm broadband
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         1024
DS         4
SWH        30303.081 Hz
FIDRES     0.462388 Hz
AQ         1.0813940 sec
RG         2896.3
DW         16.500 usec
DE         4.50 usec
TE         300.0 K
D1         0.25000000 sec
D11        0.0300000 sec

===== CHANNEL f1 =====
NUC1       13C
P1         6.80 usec
PL1        0.00 dB
SFO1       125.776013 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
PCPD2       80.00 usec
PL2         -3.00 dB
PL12        15.40 dB
SFO2        500.1535010 MHz

F2 - Processing parameters
=====
SI          65536
SF          125.762811 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          2.00

1D NMR plot parameters
=====
CY          228.975 ppm
F1          28922.10 Hz
F2          -10.979 ppm
P1          -1380.74 Hz
PPM0N       10.56815 ppm/cm
HZ0N        1329.08044 Hz/cm
    
```

Current Data Parameters
 Name: 2A-IV-146-67-2
 Date: 20031203
 Time: 15.49
 Instrument: 5 mm broadband
 PULPROG: zgpg30
 TD: 65536
 SOLVENT: CDCl3
 NS: 139
 DS: 4
 SWH: 30303.031 Hz
 FIDRES: 0.462388 Hz
 AQ: 1.0913940 sec
 RG: 256
 DW: 16.50 usec
 DE: 4.50 usec
 TE: 300.0 K
 D1: 0.25000000 sec
 D11: 0.03000000 sec

===== CHANNEL f1 =====
 NUC1: ¹³C
 P1: 15.00 usec
 PL1: 0.00 dB
 SF01: 125.6961791 MHz

===== CHANNEL f2 =====
 CPDPRG2: waltz16
 NUC2: ¹H
 PCPD2: 80.00 usec
 PL2: -3.00 dB
 PL12: 15.00 dB
 SF02: 499.833498 MHz

F2 - Processing parameters
 SI: 125.6823821 MHz
 SF: 65536
 WDW: EM
 SSB: 0
 LB: 1.00 Hz
 GB: 0
 PC: 2.00

1D NMR plot parameters
 CX: 22.80 cm
 FIP: 230.330 ppm
 FI: 29948.47 Hz
 F2P: -10.778 ppm
 F2: 13541.56 Hz
 FWHM: 10.5191 ppm/cm
 HZCX: 1325.0808 Hz/cm

Current Data Parameters
 Date_ 20031211
 Time_ 13.58
 INSTRUM gn500
 PROCESSED 5 mm broadband
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 184
 DS 4
 SWH 30303.031 Hz
 FIDRES 0.462388 Hz
 AQ 1.0813940 sec
 RG 572.6
 DW 16.60 usec
 DE 4.50 usec
 TE 300.0 K
 D1 0.25000000 sec
 D11 0.03000000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 15.00 usec
 PL1 0.00 dB
 SF01 125.6961791 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -3.00 dB
 SF02 499.833498 MHz

F2 - Processing parameters
 SI 65536
 SF 125.6823659 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 2.00

1D NMR plot parameters
 CX 22.80 cm
 FIP 230.459 ppm
 FI 29964.65 Hz
 F2P -10.649 ppm
 F2 13381.38 Hz
 FWHM 10.5191 ppm/cm
 HZCX 1325.0832 Hz/cm

Current Data Parameters
 USER jester
 NAME ZA-IV-154-1
 EXPNO 5
 PROCNO 1
 P2 - Acquisition Parameters
 Date_ 20040326
 Time 9.28
 INSTRUM omega500
 PROBD 5 mm broadband
 PULPROG zg30
 TD 81728
 SOLVENT CDCl3
 NS 1
 DS 2
 SWE 8012.820 Hz
 FIDRES 0.098043 Hz
 AQ 5.0998774 sec
 RG 32
 DW 62.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.10000000 sec
 ===== CHANNEL f1 =====
 NUC1 1H
 P1 13.00 usec
 PL1 -1.00 dB
 SFO1 500.225015 MHz
 P2 - Processing parameters
 SI 65536
 SF 500.2200316 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 4.00
 1D NMR Plot parameters
 C 24.80 cm
 FID 16384
 P1 4001.76 Hz
 P2 0.000 Hz
 F2 0.00 Hz
 FPMCM 0.35088 ppm/cm
 HZCM 175.51581 Hz/cm

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Current Data Parameters
 User: jg
 Name: ZA-IV-154-1
 ExpNO: 3
 PROCNO: 1

F2 - Acquisition Parameters
 Date_: 20031213
 Time: 15.11
 INSTRUM: gn500
 PROBHD: 5 mm broadband
 PULPROG: zgpg30
 TD: 65536
 SOLVENT: CDCl3
 NS: 296
 DS: 4
 SWH: 30303.031 Hz
 FIDRES: 0.46288 Hz
 AQ: 1.081390 sec
 RG: 832
 DW: 1.500 usec
 DE: 4.50 usec
 TE: 300.0 K
 D1: 0.2500000 sec
 D11: 0.0300000 sec

===== CHANNEL f1 =====
 NUC1: 13C
 P1: 15.00 usec
 PL1: 0.00 dB
 SFO1: 125.6961791 MHz

===== CHANNEL f2 =====
 CPDPRG2: waltz16
 NUC2: 1H
 P2: 80.00 usec
 PL2: -3.00 dB
 PL12: 19.00 dB
 P22: 13.00 dB
 SFO2: 499.834986 MHz

F2 - Processing parameters
 SI: 65536
 SF: 125.6823608 MHz
 WDW: EN
 SSB: 0
 LB: 1.00 Hz
 GB: 0
 PC: 2.00

1D NMR plot parameters
 CX: 22.80 cm
 FIP: 230.500 ppm
 F1: 28969.75 Hz
 F2: -10.608 ppm
 FZ: -1333.28 Hz
 EQN: 10.57451 ppm/cm
 HZCN: 1329.08052 Hz/cm

Current Data Parameters
 USER Jester
 NAME ZA-V-106-3
 EXPNO 5
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040622
 Time 13:16
 INSTRUM console
 PROBRD 5 mm broadband
 PULPROG zg30
 TD 81728
 SOLVENT CDCl3
 NS 8
 DS 2
 SMR 801.2820 Hz
 FIDRES 0.098043 Hz
 AQ 5.0598774 sec
 RG 64
 DM 62.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.10000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 13.00 usec
 PL1 -1.00 dB
 SFO1 500.2235015 MHz

F2 - Processing parameters
 SI 65536
 SF 500.2200132 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 4.00

1D NMR Plot Parameters
 CX 22.80 cm
 FIP 8.000 ppm
 F1 4001.76 Hz
 F2 0.000 ppm
 F3 0.000 ppm
 PRGM 0.35088 ppm/cm
 HZCN 175.51579 Hz/cm

Current Data Parameters
 USER jester
 NAME ZA-V-106-3
 EXPNO 6
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20040622
 Time_ 23.58
 INSTRUM omegad500
 PROBHD 5 mm broadband
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 839
 DS 4
 SWH 30303.031 Hz
 FIDRES 0.4622388 Hz
 AQ 1.0813940 sec
 RG 5160.6
 DW 16.500 usec
 DE 4.50 usec
 TE 300.0 K
 D1 0.25000000 sec
 D11 0.03000000 sec
 ===== CHANNEL f1 =====
 NUC1 13C
 P1 22.50 usec
 PL1 -6.00 dB
 SFO1 125.7942048 MHz
 ===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 P2 80.00 usec
 PL2 -1.00 dB
 PL12 14.40 dB
 SFO2 500.2230013 MHz
 F2 - Processing parameters
 SI 32768
 SF 125.7605745 MHz
 EQ 1
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 2.00
 1D NMR plot parameters
 CX 22.80 cm
 F1P 228.824 ppm
 F1 28781.62 Hz
 F2P -12.096 ppm
 F2 -1521.40 Hz
 PPMX 10.5666 ppm/cm
 HZCN 1329.08020 Hz/cm

```

Current Data Parameters
=====
USRP          USRP-Jes2ar
NAME          ZA-V-104-3
EXPNO         5
PROCNO        1

F2 - Acquisition Parameters
=====
Date_         20040622
Time_         21.46
INSTRUM       omega500
PROBHD        5 mm broadband
PULPROG       zg30
TD            81728
SOLVENT       CDCl3
NS            1
DS            2
SWH           8012.850 Hz
FIDRES        0.090043 Hz
AQ            5.039874 sec
RG            62.400 usec
DE            6.00 usec
TE            300.0 K
D1            0.1000000 sec

===== CHANNEL f1 =====
NUC1          1H
P1            13.00 usec
PL1           -1.00 dB
SFO1          500.235015 MHz

F2 - Processing parameters
=====
SI            65536
SF            500.220128 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            4.00

ID NMR plot parameters
=====
CX            22.80 cm
CY            8.000 ppm
F1P           4001.76 Hz
F2P           0.000 ppm
F2            0.00 Hz
PPHMM         0.35088 ppm/cm
HPCPM         175.51579 Hz/cm

```

s41

Current Data Parameters
 USSR Jester
 NAME 2A-V-104-3
 EXPNO 6
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20040622
 Time_ 21.48
 INSTRUM omeg500
 PROBED 5 mm broadband
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl₃
 NS 366
 DS 4
 SWE 30303.031 Hz
 FIDRES 0.462388 Hz
 AQ 1.0813940 sec
 RG 5160.6
 DW 16.500 usec
 DB 4.50 usec
 TB 180.0 K
 TS 0.2500000 sec
 D1 0.1000000 sec
 D11 0.0300000 sec
 ===== CHANNEL f1 =====
 NUC1 ¹³C
 P1 22.50 usec
 PL1 -6.00 dB
 SFO1 125.7942048 MHz
 ===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 ¹H
 P2 80.00 usec
 PL2 -1.00 dB
 PL12 14.40 dB
 SFO2 500.2230013 MHz
 F2 - Processing parameters
 SI 32768
 SF 125.7805596 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 2.00
 1D NMR plot parameters
 CX 22.80 cm
 FIP 228.864 ppm
 F1 28786.71 Hz
 F2 -12.055 ppm
 F2 -1516.32 Hz
 PPMON 10.56666 ppm/cm
 HZCN 1329.08032 Hz/cm

Current Data Parameters
USER Jester
NAME ZA-V-105-3
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040622
Time 12:00
INSTRUM spect
PROBHD 5 mm broadband
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.096043 Hz
AQ 5.0988774 sec
RG 32
DM 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.1000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 13.00 usec
PL1 -1.00 dB
SF01 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2200000 MHz
WDW EN
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

ID NMR plot parameters
CX 22.80 cm
F1P 8.000 ppm
F1 4001.76 Hz
F2P 0.000 ppm
F2 0.000 Hz
FREQW 0.35088 Hz/cm
HZON 175.51579 Hz/cm

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Current Data Parameters
 USER Jester
 NAME ZA-V-105-3
 EXPNO 3
 PROCNO 1
 F2 - Acquisition Parameters
 Date 20040622
 Time 22:52
 INSTRUM omegab500
 PROBHD 5 mm broadband
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 154
 DS 4
 SWH 30303.001 Hz
 FIDRES 0.46238 Hz
 AQ 1.0813840 sec
 RG 5160.6
 DW 16.500 usec
 DE 4.50 usec
 TE 300.0 K
 D1 0.25000000 sec
 D11 0.0300000 sec
 ===== CHANNEL f1 =====
 NUC1 13C
 P1 22.50 usec
 PL1 -6.00 dB
 SFO1 125.7942048 MHz
 ===== CHANNEL f2 =====
 PULPROG waltz16
 NUC2 1H
 PCPDZ 80.10 usec
 PL2 -1.00 dB
 PL12 14.40 dB
 SFO2 500.2230013 MHz
 F2 - Processing parameters
 SI 65536
 SF 125.7804664 MHz
 WDW EN
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 2.00
 1D NMR plot parameters
 CX 22.60 cm
 F1P 229.546 ppm
 F1 28.694 Hz
 F2P -143.584 ppm
 F2 -143.09 Hz
 FWHM 10.56666 ppm/cm
 HZWN 1329.08332 Hz/cm

Current Data Parameters
 USER Jester
 NAME ZA-V-115-H19
 EXPNO 8
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20070726
 Time 22:27
 INSTRUM spect
 PROBRW 5 mm broadband
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 7500
 DS 4
 SWH 30303.031 Hz
 FIDRES 0.462388 Hz
 AQ 1.0813940 sec
 RG 8192
 DM 16.500 usec
 DB 4.50 usec
 TE 300.0 K
 D1 0.25000000 sec
 D11 0.03000000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 6.80 usec
 PL1 0.00 dB
 SF01 125.7766019 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 P2 80.00 usec
 PL2 -3.00 dB
 PL12 15.40 dB
 SF02 500.1535010 MHz

F2 - Processing parameters
 SI 65536
 SF 125.7675903 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 2.00

1D NMR plot parameters
 CX 22.80 cm
 F1P 230.300 ppm
 F1 28963.15 Hz
 F2P -10.654 ppm
 F2 -1339.87 Hz
 PPMCM 10.56815 ppm/cm
 HZCM 1329.08020 Hz/cm

Current Data Parameters
 USER Jester
 NAME ZA-V-116-3
 EXPNO 5
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040625
 Time 1:55
 INSTRUM gn500
 PULPROG 5 mm broadband
 FULPROG zgpg30
 GAMMA 13
 SOLVENT CDCl3
 NS 8
 DS 2
 SFO1 500.1535010 MHz
 FIDRES 0.098043 Hz
 AQ 5.0598774 sec
 RG 90.5
 DW 62.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.10000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 11.50 usec
 PL1 -3.00 dB
 SFO1 500.1535010 MHz

F2 - Processing parameters
 SI 65536
 SF 500.1500000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 4.00

1D NMR Plot parameters
 CX 22.80 cm
 FIP 8.000 ppm
 F1 4001.20 Hz
 F2 0.000 ppm
 P2 0.000 Hz
 PPMQN 0.35088 ppm/cm
 PZCN 175.45123 Hz/cm

```

Current Data Parameters
=====
NAME      ZB-V-1163
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
=====
Date_     20040625
Time      1.58
INSTRUM   spect
PROBHD    5 mm broadband
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         224
DS         4
SWH        30303.001 Hz
FIDRES     0.462388 Hz
AQ          1.0813840 sec
RG          327.8
DE          16500 usec
TE          300.0 K
D1          0.25000000 sec
D11         0.03000000 sec
===== CHANNEL f1 =====
NUC1       13C
P1          6.80 usec
PL1         0.00 dB
SFO1       125.776019 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
P2          80.00 usec
PL2         3.00 dB
SFO2       500.1555010 MHz

F2 - Processing parameters
=====
SI          65536
SF          125.7629823 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          2.00

1D NMR plot parameters
=====
CX          22.80 cm
F1P         228.772 PPM
F1          28771.09 Hz
F2P         -12.181 PPM
F2          -1531.95 Hz
PRGN        10
H2CH        1323.06044 PPM/cm
    
```

Current Data Parameters
 USER Jester
 NAME ZA-V-158-3
 EXPNO 4
 PROCNO 1
 P2 - Acquisition Parameters
 Date_ 20040727
 Time_ 0.14
 INSTRUM gp500
 PROBHD 5 mm broadband
 PULPROG zg30
 TD 81728
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.098043 Hz
 AQ 5.098774 sec
 RG 362
 DW 62.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.1000000 sec
 ===== CHANNEL f1 =====
 NUC1 1H
 P1 11.50 usec
 PL1 -3.00 dB
 SFO1 500.1535010 MHz
 P2 - Processing parameters
 SI 65536
 SF 500.1500000 MHz
 WDM 0
 EN 0
 SS 0
 LB 0.30 Hz
 GB 0
 PC 4.00
 LD NMR plot parameters
 XY 27.80 cm
 YP 8.00 cm
 FIP 4001.20 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPM0 0.35088 ppm/cm
 HZCM 175.49123 Hz/cm

s53

Current Data Parameters
 USER Jester
 NAME ZA-V-107-3
 EXPNO 4
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20040624
 Time 21.01
 INSTRUM gn500
 PROBHD 5 mm broadband
 PULPROG zg30
 TD 81728
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.73842 Hz
 AQ 5.093971 sec
 RG 50.5
 DQ 62.400 usec
 DB 6.00 usec
 TE 300.0 K
 D1 0.1000000 sec
 ===== CHANNEL f1 =====
 NUC1 1H
 P1 11.50 usec
 PL1 -3.00 dB
 SFO1 500.1535010 MHz
 F2 - Processing parameters
 SI 65536
 SF 500.1500095 MHz
 WDW EN
 SS 0
 LB 0.30 Hz
 GB 0
 PC 4.00
 1D NMR plot parameters
 CX 22.80 cm
 FIP 8.000 ppm
 F1 4001.20 Hz
 F2 0.000 ppm
 F2 0.00 Hz
 PPM0 0.35088 ppm/cm
 HZCM 175.49124 Hz/cm

Current Data Parameters
 USER jester
 NAME ZA-V-107-3
 EXPNO 5
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20040624
 Time 21.03
 INSTRUM gds500
 PROBHD 5 mm broadband
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl₃
 NS 345
 DS 4
 SWH 30303.031 Hz
 FIDRES 0.462388 Hz
 AQ 1.0813940 sec
 RG 4096
 DW 16.500 usec
 DE 4.50 usec
 TE 300.2 K
 D1 0.25000000 sec
 D11 0.03000000 sec
 ===== CHANNEL f1 =====
 NUC1 ¹³C
 P1 6.80 usec
 PL1 0.00 dB
 SFO1 125.7766019 MHz
 ===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 ¹H
 P2 80.00 usec
 PL2 -3.00 dB
 PL12 15.40 dB
 SFO2 500.1355010 MHz
 F2 - Processing parameters
 SI 32768
 SF 125.762963 MHz
 DSF 125.762963 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 2.00
 1D NMR plot parameters
 CX 22.80 cm
 FIP 229.134 ppm
 F1 28816.53 Hz
 F2P -11.820 ppm
 F2 -1486.50 Hz
 PPM0V 10.56614 ppm/cm
 BZCN 1329.08020 Hz/cm

25

Current Data Parameters
 USER jester
 NAME ZA-V-107-5
 EXPNO 2
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20040624
 Time 22.13
 INSTRUM gn500
 PROBRD 5 mm broadband
 PULPROG zg30
 TD 81728
 SOLVENT CDCl3T
 NS 8
 DS 2
 SFE 8012.820 Hz
 FIDRES 0.098043 Hz
 AQ 5.0398774 sec
 RG 181
 DW 62.400 usec
 DE 6.000 usec
 TE 300.0 K
 D1 0.10000000 sec
 ===== CHANNEL f1 =====
 NUC1 1H
 P1 11.50 usec
 PL1 -3.00 dB
 SFO1 500.1535010 MHz
 F2 - Processing parameters
 SI 65536
 SF 500.1500097 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 4.00
 FID NMR plot parameters
 CX 22.80 cm
 FIP 8.000 ppm
 F1 4001.20 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 0.35088 ppm/cm
 HZCM 175.49124 Hz/cm

25

Current Data Parameters
 USER Jester
 NAME ZA-V-107-5
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040624
 Time_ 22.15
 INSTRUM 5 mm broadband
 PULPROG zgpg30
 TD 65536
 SFO1 125.76013 MHz
 SOLVENT CDCl3
 NS 1300
 DS 4
 SWH 30303.031 Hz
 FIDRES 0.462388 Hz
 AQ 1.0813940 sec
 RG 4096
 DW 16.500 usec
 DE 4.50 usec
 TE 300.0 K
 D1 0.25000000 sec
 D11 0.03000000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 6.80 usec
 PL1 0.00 dB
 SFO1 125.766013 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -3.00 dB
 PL12 15.40 dB
 SFO2 500.1535010 MHz

F2 - Processing parameters
 SI 65536
 SF 125.7628814 MHz
 NQW 0
 EN 0
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 2.00

1D NMR plot parameters
 CY 22.80 cm
 F1P 229.575 ppm
 F1 28872.01 Hz
 F2P -11.379 ppm
 F2 -1431.02 Hz
 PPMCN 10.56814 ppm/cm
 HZCN 1329.08032 Hz/cm

Current Data Parameters

USER Jester
 NAME ZA-V-117-4
 EXPNO 4
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040625
 Time 2.59
 INSTRON 95500
 PROBO 5 mm broadband
 PULPROG zgpg30
 TD 65536
 SFO1 500.150000 MHz
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.09843 Hz
 AQ 5.0998774 sec
 RG 90.5
 DW 62.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.10000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 11.50 usec
 PL1 -3.00 dB
 SFO1 500.150000 MHz

F2 - Processing parameters
 SI 65536
 SF 500.150000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 4.00

1D NMR plot parameters
 CX 22.80 cm
 FIP 8.000 ppm
 FI 4001.20 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 FPCNO 0.35088 ppm/cm
 FZCN 175.45123 Hz/cm

Current Data Parameters
 USER Jester
 NAME ZA-V-143-5
 EXPNO 4
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040722
 Time 18.28
 INSTRUM gms500
 PROBED 5 mm broadband
 PULPROG zg30
 TD 81728
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.098043 Hz
 AQ 5.0989774 sec
 RG 181
 DW 62.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.1000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 11.50 usec
 PL1 -3.00 dB
 SF01 500.1535010 MHz

F2 - Processing parameters
 SI 65536
 SF 500.1500087 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 4.00

1D NMR plot parameters
 CH 22.00 cm
 CP 8.00 cm
 F1 4001.20 Hz
 F2 0.000 Hz
 F3 0.000 Hz
 PPM0 0.35088 ppm/cm
 HZ0M 175.49123 Hz/cm

Current Data Parameters
 User: Jester
 Sample: ZA-V-144-S
 ExpNO: 2
 PROCNO: 1

F2 - Acquisition Parameters
 Date_: 20040722
 Time: 16.19
 INSTRUM: gn500
 PROCNO: 5 mm broadband
 PULPROG: zgpg30
 TD: 2930
 SFO1: 500.135010 MHz
 SOLVENT: CDCl3
 NS: 8
 DS: 2
 SWH: 8012.820 Hz
 FIDRES: 0.098043 Hz
 AQ: 5.099774 sec
 RG: 261
 DW: 62.400 usec
 DE: 2.00 usec
 TE: 300.0 K
 D1: 0.1000000 sec

===== CHANNEL f1 =====
 NUCL: 1H
 P1: 11.50 usec
 PL1: -3.00 dB
 SFO1: 500.135010 MHz

F2 - Processing parameters
 SI: 65536
 SF: 500.1499869 MHz
 WDW: EM
 SSB: 0
 LB: 0.30 Hz
 GB: 0
 PC: 4.00

1D NMR plot parameters
 CX: 32.80 cm
 FIP: 8.000 ppm
 F1: 4001.20 Hz
 F2: 0.000 ppm
 F2: 0.00 Hz
 PPMCM: 0.35088 ppm/cm
 HZCM: 175.49123 Hz/cm

Current Data Parameters
Date: 20040702
Time: 11:00
Name: 30
Pulse: 1
Acq: 1
F2 - Acquisition Parameters
Date: 20040702
Time: 11:00
Name: 30
Pulse: 1
Acq: 1
F2 - Processing parameters
SI: 32768
SF: 400.146
WDW: EM
SSB: 0
GB: 0
PC: 1.00
SC: 1.00
F2 - 1D and 2D Parameters
SI: 32768
SF: 400.146
WDW: EM
SSB: 0
GB: 0
PC: 1.00
SC: 1.00
F2 - 1D and 2D Parameters
SI: 32768
SF: 400.146
WDW: EM
SSB: 0
GB: 0
PC: 1.00
SC: 1.00

Current Data Parameters
 USER Jester
 NAME 2A-V-144-6
 EXPNO 3
 PROCNO 1
 P2 - Acquisition Parameters
 Date_ 20040702
 Time_ 16.54
 INSTRUM gm500
 PROBD 5 mm broadband
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl₃
 NS 892
 DS 4
 SWH 30303.031 Hz
 FIDRES 0.462388 Hz
 AQ 1.0813940 sec
 RG 3251
 DW 16.500 usec
 DE 4.50 usec
 TE 300.0 K
 D1 0.25000000 sec
 D11 0.03000000 sec
 ===== CHANNEL f1 =====
 NUC1 13C
 P1 6.80 usec
 PL1 0.00 dB
 SFO1 125.7766019 MHz
 ===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCD2 80.00 usec
 PL2 -3.00 dB
 PL12 15.40 dB
 SFO2 500.1535010 MHz
 P2 - Processing parameters
 SI 32768
 SF 125.7627250 MHz
 WDW EM
 SSF 0
 LB 1.00 Hz
 GB 0
 PC 2.00
 1D NMR plot parameters
 CX 22.80 cm
 FIP 230.781 ppm
 F1 29023.70 Hz
 F2P -10.173 ppm
 F2 -1279.33 Hz
 PRON 10.56816 ppm/cm
 HZCM 1329.08032 Hz/cm

537.87
554.06
649.20
685.12
688.42
692.31
694.72
699.91
704.36
707.62
726.91
733.21
739.82
742.66
746.80
750.52
757.31
760.77
763.63
769.17
794.32
798.86
803.22
867.12
869.44
875.19
878.71
884.27
889.80
897.28
902.67
904.71
942.15
950.03
951.59
955.20
959.46
964.51
1078.56
1085.54
1092.43
1099.41
1149.50
1157.41
1163.36
1171.29
1499.50
1503.55
1505.21
1508.83
1514.67
1522.87
1527.97
1531.94
1533.85
1537.51
1700.50
1752.57

2543.17
2544.26
2545.41
2546.75
2558.60
2559.96
2560.92
2562.24
2563.62
2568.77
2569.71
2571.03
2673.97
2925.04
2932.09
2932.92
2935.26
2939.97
2942.14
2950.00
2952.14
2956.87
2959.19
2960.04
2967.07

Current Data Parameters
 USER Jester
 NAME ZA-III-197-2-2
 EXPNO 1
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20020619
 Time 22.09
 INSTRUM gm500
 PROBDW 5 mm broadband
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 8072.82 Hz
 FIDRES 0.11226 Hz
 AQ 4.08496 sec
 RG 181
 DW 62.400 usec
 DB 6.00 usec
 TE 300.0 K
 D1 0.1000000 sec
 ===== CHANNEL f1 =====
 NUC1 1H
 P1 12.00 usec
 PL1 -3.00 dB
 SFO1 499.9934995 MHz
 F2 - Processing parameters
 SI 65536
 SF 499.9934995 MHz
 WDW EN
 SS 0
 LB 0.30 Hz
 GB 0
 PC 4.00
 1D NMR plot parameters
 CX 22.80 cm
 FIP 8.000 ppm
 F1 3999.44 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PWDW 0.35088 ppm/cm
 HZCM 175.41403 Hz/cm

Current Data Parameters
 USER Jester
 NAME ZA-III-197-2-2
 EXNO 3
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20020619
 Time 22.23
 INSTRUM gms500
 PROBED 5 mm broadband
 PULPROG zgpg30
 ID 65536
 SOLVENT CDCl₃
 NS 132
 DS 2
 SWH 30303.031 Hz
 FIDRES 0.462388 Hz
 AQ 1.0413940 sec
 RG 5160.6
 DW 16.500 usec
 DE 4.50 usec
 TE 300.0 K
 D1 0.25000000 sec
 D11 0.03000000 sec
 ===== CHANNEL f1 =====
 NUC1 ¹³C
 P1 6.82 usec
 PL1 0.00 dB
 SFO1 125.7713038 MHz
 ===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 ¹H
 P2 80.00 usec
 PL2 -3.00 dB
 PL12 15.20 dB
 SFO2 499.9334995 MHz
 F2 - Processing parameters
 SI 65536
 SF 125.7075086 MHz
 MDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 2.00
 1D NMR plot parameters
 CT 22.80 cm
 F1P 230.270 ppm
 F1 28946.71 Hz
 F2P -10.790 ppm
 F2 -1356.32 Hz
 PPMCH 10.57280 ppm/cm
 HZCM 1329.08032 Hz/cm

8

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Current Data Parameters
=====
USER      jsm
NAME      ZA-V-166-2
EXPNO     4
PROCNO    1

F2 - Acquisition Parameters
=====
Date_     20040727
Time      23.12
INSTRUM   spect
PROBHD    5 mm broadband
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         941
DS         4
SHE       30303.031 Hz
SF         0.462368 Hz
AQ         1.0613940 sec
RG         680
PC         16.800 usec
DE         4.50 usec
TE         300.0 K
D1         0.25000000 sec
D11        0.03000000 sec

===== CHANNEL f1 =====
NUC1       13C
P1         6.80 usec
PL1        0.00 dB
SFO1       125.7766013 MHz

===== CHANNEL f2 =====
CPRPG2     waitz16
NUC2       1H
P2         80.00 usec
PL2        19.00 dB
SFO2       500.1535010 MHz

F2 - Processing parameters
=====
SI         65536
SF         125.762797 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         2.00

1D NMR plot parameters
=====
CX         22.80 cm
FIP        230.781 ppm
F1         29023.70 Hz
F2         -10.173 ppm
F3         -12.943 Hz
PRGMM      1D1665
HZCM       1329.08032 Hz/cm
  
```