ω-Halonitriles: Domino Cyclizations to Oxa- and Carbocyclic Nitriles

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Experimental¹

General procedure for chloronitrile alkylations: Addition of the electrophile (1 equiv, neat) to a 0 °C, THF solution of *t*-BuOK (1 equiv) was followed immediately by the addition of the ω -chloronitrile. After 3 h, the reaction mixture was allowed to warm to the room temperature and saturated, aqueous NH₄Cl was then added. The crude reaction mixture was then separated, the aqueous fraction extracted with EtOAc, and the combined crude product passed through short plug of silica, concentrated, and purified by radial chromatography.

2-Phenyl-tetrahydro-pyran-3-carbonitrile (12a): Performing the general procedure with a THF solution (5 mL) of **10a** (144 mg, 1.23 mmol) and PhCHO (131 mg, 1.23 mmol) provided, after purification by radial chromatography (2:8 EtOAc/hexanes), 202.5 mg (88%, 1:1, *trans:cis*) of **12a** as oils. For **12a trans**: IR (film) 2239 cm⁻¹; ¹H NMR δ 1.72-2.05 (m, 3H), 2.36-2.42 (m, 1H), 2.63-2.72 (m, 1H), 3.56-3.66 (m, 1H), 4.13-4.18 (m, 1H), 4.34 (d, *J* = 9.5 Hz, 1H), 7.31-7.48 (m, 5H); ¹³C NMR δ 24.0, 28.0, 35.2, 68.2, 80.6, 119.3, 126.5, 128.3, 128.6, 138.7; HRMS (ESI) calcd for (M+Na⁺) C₁₂H₁₃NONa 210.0889, found 210.0889. For **12a cis**: IR (film) 2238 cm⁻¹; ¹H NMR δ 1.35-1.42 (m, 1H), 1.70-2.04 (m, 3H), , 2.75 (br s, 1H), 3.36-3.44 (m, 1H), 4.00-4.05 (m, 1H), 4.23 (d, *J* = 1.9 Hz, 1H), 7.02-7.41 (m, 5H); ¹³C NMR δ 21.6, 27.0, 35.4, 68.8, 78.5, 118.8, 125.4, 128.0, 128.2, 139.0; HRMS (ESI) calcd for (M+Na⁺) C₁₂H₁₃NONa 210.0889, found 210.0900.

2-Furan-2-yl-tetrahydro-pyran-3-carbonitrile (12b): Performing the general procedure with a THF solution (5 mL) of **10a** (144 mg, 1.23 mmol) and furan-2-carbaldehyde (119 mg, 1.23 mmol) provided, after purification by radial chromatography (2:8 EtOAc/hexanes), 164 mg

(75%, 1.5:1, *trans:cis*) of **12b** as oils. For **12b trans**: IR (film) 2236 cm^{-1} ; ^1H NMR δ 1.86-2.41 (m, 4H), 3.56-3.68 (m, 1H), 4.09 (d, $J = 12.3$ Hz, 1H), 4.48 (d, $J = 10.2$ Hz, 1H), 6.39 (br s, 1H), 6.46 (d, $J = 3.1$ Hz, 1H), 7.44 (br s, 1H); ^{13}C NMR δ 23.9, 27.7, 31.6, 68.2, 73.5, 109.1, 110.4, 119.0, 143.1, 150.7; MS m/e 178 ($M + H$); HRMS(ESI) calcd for ($M+\text{Na}^+$), $\text{C}_{10}\text{H}_{11}\text{NO}_2\text{Na}$ 200.0682 found: 200.0698. For **12b cis**: IR (film) 2241 cm^{-1} ; ^1H NMR δ 1.59-2.09 (m, 4H), 2.26 (d, $J = 13.3$ Hz, 2H), 3.15-2.16 (m, 1H), 3.56-3.65 (m, 1H), 4.19 (dd, $J = 11, 4$ Hz, 1H), 4.54 (d, $J = 2$ Hz, 1H), 6.39 (s, 1H), 6.52 (d, $J = 2.9$ Hz, 1H), 7.39 (br s, 1H); ^{13}C NMR δ 21.8, 26.5, 32.1, 68.6, 73.3, 107.3, 110.3, 118.9, 142.1, 151.3; MS m/e 178 ($M + H$); HRMS (ESI) calcd for ($M+\text{Na}^+$), $\text{C}_{10}\text{H}_{11}\text{NO}_2\text{Na}$ 200.0682 found: 200.0698.

2-tert-Butyl-tetrahydro-pyran-3-carbonitrile (12c): Performing the general procedure with a THF solution (5 mL) of *t*-BuOK (195 mg, 1.74 mmol), **10a** (136 mg, 1.16 mmol) and excess 2,2-dimethylpropionaldehyde² provided, after purification by radial chromatography (2:8 EtOAc/hexanes), 170 mg (88%, 2.5:1, *trans:cis*) of **12c** as oils. For **12c trans**: IR (film) 2235 cm^{-1} ; ^1H NMR δ 1.04 (s, 9H), 1.53-1.59 (m, 1H), 1.81 (dd, $J = 12, 5$ Hz, 1H), 1.89 (dd, $J = 12, 5$ Hz, 1H), 2.26-2.31 (m, 1H), 2.41-2.51 (m, 1H), 3.07 (d, $J = 9.4$ Hz, 1H), 3.32-3.41 (m, 1H), 3.97-4.00 (m, 1H); ^{13}C NMR δ 24.5, 26.6, 28.0, 29.7, 35.7, 61.8, 68.2, 85.1, 121.5; MS m/e 168 ($M + H$). HRMS (ESI) calcd for ($M+\text{Na}^+$): $\text{C}_{10}\text{H}_{17}\text{NONa}$ 190.1202 found 190.1206. For **12c cis**: IR (film) 2236 cm^{-1} ; ^1H NMR δ 1.03 (s, 9H), 1.54 (d, $J = 13.4$ Hz, 1H), 1.60-1.79 (m, 1H), 1.83-2.07 (m, 1H), 2.17 (d, $J = 13.1$ Hz, 1H), 2.84 (s, 1H), 2.90 (s, 1H), 3.45 (t, $J = 11.7$ Hz, 1H), 4.14 (dd, $J = 11, 4$ Hz, 1H); ^{13}C NMR δ 22.1, 26.2, 27.5, 28.7, 34.6, 69.3, 85.3, 120.6; MS m/e 168 ($M + H$).

2-Cyclopropyl-tetrahydro-pyran-3-carbonitrile (12d): Performing the general procedure with a THF solution (5 mL) of **10a** (145 mg, 1.23 mmol) and cyclopropanecarbaldehyde (87 mg, 1.23

mmol) provided, after purification by radial chromatography (1:9 EtOAc/hexanes), 157 mg (85%, 1:1, *trans:cis*) of **12d** as oils. For **12d trans**: IR (film) 2237 cm^{-1} ; ^1H NMR δ 0.36-0.77 (m, 4H), 0.99-1.06 (m, 1H), 1.58-1.85 (m, 3H), 2.26 (d, $J = 13.2$ Hz, 1H), 2.54-2.62 (m, 1H), 2.73-2.78 (m, 1H), 3.34-3.43 (m, 1H), 3.99 (d, $J = 11.9$ Hz, 1H); ^{13}C NMR δ 1.7, 3.2, 14.9, 24.2, 27.9, 33.6, 67.8, 81.0, 119.9; HRMS(ESI) calcd for ($\text{M}+\text{Na}^+$) $\text{C}_{19}\text{H}_{13}\text{NONa}$ 174.0889 found 174.0894. For **12d cis**: IR (film) 2236 cm^{-1} ; ^1H NMR δ 0.17-0.24 (m, 1H), 0.73 (m, 3H), 1.15-1.26 (m, 1H), 1.55 (d, $J = 13$ Hz, 1H), 1.71-2.10 (m, 2H), 2.17 (d, $J = 13$ Hz, 1H), 2.58-2.61 (m, 1H), 2.91 (br s, 1H), 3.40 (t, $J = 11.6$ Hz, 1H), 4.06-4.10 (m, 1H); ^{13}C NMR δ 1.4, 3.7, 14.3, 15.0, 21.9, 26.7, 32.8, 68.4, 81.0, 119.7; HRMS (ESI) calcd for ($\text{M}+\text{Na}^+$) $\text{C}_9\text{H}_{13}\text{NONa}$ 174.0889 found 174.08957.

2-Benzoyl-5-chloro-pentanenitrile (16a): Performing the general procedure with a THF solution (5 mL) of *t*-BuOK (290 mg, 2.58mmol), **10a** (144 mg, 1.23 mmol), and ethyl benzoate (185 mg, 1.23 mmol) provided, after purification by radial chromatography (1:9 EtOAc/hexanes), 158 mg (58%) of **16a** as an oil: IR (film) 2249, 1694, 1596 cm^{-1} ; ^1H NMR δ 1.96-2.31 (m, 4H), 3.62 (t, $J = 6$ Hz, 2H), 4.41-4.46 (m, 1H), 7.50-7.99 (m, 5H); ^{13}C NMR δ 26.9, 29.4, 39.0, 43.6, 116.9, 128.7, 129.1, 133.8, 134.6, 190.2; HRMS (ESI) calcd for ($\text{M}+\text{Na}^+$) $\text{C}_{12}\text{H}_{12}\text{ClNONa}$ 244.0500, found 244.0507.

5-Chloro-2-methyl-pentanenitrile (10b): To a 0 $^\circ\text{C}$ THF solution of *t*-BuOK (1.38 g, 12.3 mmol), neat **10a** (144 mg, 1.23 mmol) was added followed by (within 3 min) the addition of MeI (1.4, 9.8 mmol). After 10 min, the reaction mixture was allowed to warm to the room temperature, quenched with saturated NH_4Cl , extracted with EtOAc, passed through short plug of silica (3 x 1), and concentrated to yield 150 mg (93%) of **10b** as oil. IR (film): 2237 cm^{-1} ; ^1H

NMR δ 1.34 (d, J = 7.5 Hz, 3H), 1.72-1.80 (m, 2H), 1.84-2.02 (m, 2H), 2.60-2.72 (m, 2H), 3.58 (t, J = 5.9 Hz, 2H); ^{13}C NMR δ 18.0, 25.0, 29.7, 31.2, 43.9, 122.4.

3-Methyl-2-phenyl-tetrahydro-pyran-3-carbonitrile (12e): Performing the general procedure with a THF solution (5 mL) of (110 mg, 0.83 mmol) **10b** and PhCHO (88.7 mg, 0.83 mmol) provided, after purification by radial chromatography (2:8 EtOAc/hexanes), 120.2 mg (72%, 1:1 ratio of *trans* : *cis* isomers) of **12e** as a solid. Repetitive chromatography afforded a pure, crystalline solid allowing x-ray crystallographic identification of *trans* **12e**: IR (film) 2234 cm^{-1} ; ^1H NMR δ 1.02 (s, 3H), 1.51-1.60 (m, 2H), 2.04-2.18 (m, 2H), 3.46 (t, J = 11.7 Hz, 1H), 3.89 (s, 1H), 4.10-4.14 (m, 1H), 7.13-7.33 (m, 5H); ^{13}C NMR δ 23.2, 23.6, 36.5, 39.5, 46.8, 69.0, 85.8, 121.7, 126.6, 127.5, 128.1, 128.7, 137.0; MS m/e 202.

2-Phenyl-tetrahydro-furan-3-carbonitrile (11a):³ Performing the general procedure with a THF solution (5 mL) of **10c** (128mg, 1.23 mmol) and PhCHO (130 mg, 1.23 mmol) provided, after purification by radial chromatography (3:7 EtOAc/hexanes), 160 mg (75%, 2.5:1, *trans*:*cis*) of **11a** as oils. For **11a trans**: IR (film) 2240 cm^{-1} ; ^1H NMR δ 2.31-2.50 (m 2H), 3.30-3.41 (m, 1H), 3.93-4.07 (m, 1H), 4.27-4.38 (m, 1H), 4.91-5.01 (d, J = 7.6 Hz, 1H), 7.48-7.51 (m, 5H); ^{13}C NMR δ ; 31.3, 36.5, 67.3, 81.5, 118.9, 126.0, 128.4, 128.5, 137.1; HRMS(ESI) calcd for ($\text{M}+\text{Na}^+$) $\text{C}_{11}\text{H}_{11}\text{NONa}$ 196.0733 found 196.0747. For **11a cis**: IR (film) 2242 cm^{-1} ; ^1H NMR δ 2.32-2.57 (m, 2H), 2.87 (dd, J = 16, 8 Hz, 1H), 4.09 (dd, J = 16, 8 Hz, 1H), 4.20-4.28 (m, 1H), 4.95 (d, J = 7.7 Hz, 1H), 7.33-7.39 (m, 5H); ^{13}C NMR δ 30.9, 36.9, 67.8, 83.5, 119.7, 125.3, 128.4, 128.6, 138.6; HRMS (ESI) calcd for ($\text{M}+\text{Na}^+$), $\text{C}_{11}\text{H}_{11}\text{NONa}$ 196.0733, found 196.0725.

2,3,4,5-Tetrahydro-[2,2']bifuranyl-3-carbonitrile (11b): Performing the general procedure with a THF solution (5 mL) of **10c** (127 mg, 1.23 mmol) and furan-2-carbaldehyde (119 mg, 1.23 mmol) provided, after purification by radial chromatography (2:8 EtOAc/hexanes), 162 mg

(81%, 2:1, *trans:cis*) of **11b** as oils. For **11b trans**: IR (film) 2245 cm⁻¹; ¹H NMR δ 2.35 (ddd, *J* = 13, 13, 7 Hz, 1H), 2.46-2.58 (m, 1H), 3.28-3.35 (m, 1H), 4.00-4.17 (m, 2H), 5.03 (d, *J* = 7.0 Hz, 1H), 6.37-6.42 (m, 2H), 7.42 (s, 1H); ¹³C NMR δ 31.0, 33.1, 67.8, 109.1, 110.4, 119.5, 143.2, 150.3; HRMS (EI) calcd for C₉H₉NO₂ 163.0630 found 163.0620. For **11b cis**: IR (Film) 2244 cm⁻¹; ¹H NMR δ 2.42-2.58 (m, 2H), 3.38 (dd, *J* = 15, 7 Hz, 1H), 3.96 (dd, *J* = 16, 8 Hz, 1H), 4.24-4.32 (m, 1H), 5.12 (d, *J* = 6.8 Hz, 1H), 6.34-6.40 (m, 1H) 6.48 (d, *J* = 3.0 Hz, 1H), 7.45 (br s, 1H); ¹³C NMR δ 30.8, 33.8, 67.4, 75.2, 108.8, 110.4, 118.2, 143.0, 150.7; HRMS (EI) calcd for (M⁺) 163.0628, found 163.0617.

2-tert-Butyl-tetrahydro-furan-3-carbonitrile (11c): Performing the general procedure with a THF solution (5 mL) of **10c** (128 mg, 1.23 mmol) and excess 2,2-dimethylpropionaldehyde² provided, without requiring further purification, 175.2 mg (93%, 1:0) of **11c** as spectrally pure oil. For **11c trans**: IR (film): 2240cm⁻¹; ¹H NMR δ 1.01 (s, 9H), 2.20-2.26 (m, 2H), 2.76 (dd, *J* = 16, 8 Hz, 1H), 3.70 (d, *J* = 7.4 Hz, 1H), 3.82 (dd, *J* = 16, 8 Hz, 1H), 3.94-4.01 (m, 1H); ¹³C NMR δ 25.5, 28.6, 31.9, 34.1, 67.7, 90.5, 121.4; HRMS (EI) calcd for C₉H₁₅NO 153.1150 found: 153.1132.

2-Cyclopropyl-tetrahydro-furan-3-carbonitrile (11d): Performing the general procedure with a THF solution (5 mL) of **10c** (127 mg, 1.23 mmol) and cyclopropanecarbaldehyde (86 mg, 1.23 mmol) provided, after purification by radial chromatography (2:8 EtOAc/hexanes), 121 mg (72%, 1:1, *trans:cis*) of **11d** as oils. For **11d trans**: IR (film) 2243 cm⁻¹; ¹H NMR δ ; 0.44-0.68 (m, 4H), 0.91-1.02 (m, 1H), 2.14-2.45 (m, 2H), 2.79-2.88 (m, 1H), 3.37 (t, *J* = 8 Hz, 1H), 3.83-3.91 (dd, *J* = 15, 8 Hz, 1H), 4.02 (dd, *J* = 14, 8 Hz, 1H); ¹³C NMR δ 1.5, 2.7, 13.8, 30.8, 33.8, 67.2, 86.3, 120.2; HRMS (EI) calcd for C₈H₁₁NO 137.0835 found 137.0850; For **11d cis**: IR (film) 2241 cm⁻¹; ¹H NMR δ 0.22-0.28 (m, 1H), 0.45-0.50 (m, 1H), 0.62-0.76 (m, 2H), 1.12-

1.24 (m, 1H), 2.02-2.34 (m, 2H), 3.13-3.21 (m, 2H), 3.75 (dd, $J = 16, 7$ Hz, 1H), 4.09 (dd, $J = 15, 7$ Hz, 1H); ^{13}C NMR δ 1.8, 3.6, 12.1, 31.0, 33.6, 66.7, 84.0, 119.5; MS m/e 138 ($M + H$); HRMS(EI) calcd for $\text{C}_8\text{H}_{11}\text{NO}$ 137.0835 found 137.0850.

6-Chloro-2-(hydroxy-phenyl-methyl)-hexanenitrile (18a): Performing the general procedure with a THF solution (5 mL) of **10d** (131 mg, 1 mmol) and PhCHO (106 mg, 1 mmol) provided, after purification by radial chromatography (3:7 EtOAc/hexanes), 175 mg (74%, 1:1, dr) of an inseparable mixture of **18a** as an oils: IR (film) 2244 cm^{-1} ; ^1H NMR δ 1.49-1.76 (m, 12H), 2.48 (br s, 2H), 2.78-2.92 (m, 2H), 3.47-3.53 (m, 4H), 4.75 (d, $J = 5.9$ Hz, 1H), 4.80 (d, $J = 5.9$ Hz, 1H), 7.37 (s, 5H), 7.39 (s, 5H); ^{13}C NMR δ 24.5, 27.2, 28.3, 31.9, 32.0, 40.3, 40.9, 44.3, 73.6, 74.0, 119.8, 126.1, 126.4, 128.9; GCMS e/m 238 ($M + H$); HRMS calcd for $\text{C}_{13}\text{H}_{16}\text{ClNONa}$ 260.0813 found 260.0807.

7-chloro-2-(hydroxy-phenyl-methyl)-heptanenitrile (18b): Performing the general procedure with a THF solution (5 mL) of **10e** (180 mg, 1.23 mmol) and PhCHO (131 mg, 1.23 mmol) provided, after purification by radial chromatography (3:7 EtOAc/hexanes), 222 mg (72%, 1:1) of **18b** as a mixture of diastereomers. Repetitive chromatography provided a pure sample of (*R,R*)-**18b** whose structure was proven by x-ray crystallography: IR (film) 2053 cm^{-1} ; ^1H NMR δ 1.42-1.67 (m, 5H), 1.75 (pent, $J = 7$ Hz, 2H), 2.35-2.40 (m, 2H), 2.80-2.87 (m, 1H), 3.51 (t, $J = 6$ Hz, 2H), 4.78 (d, $J = 6, 3$ Hz, 1H), 7.38 (s, 5H); ^{13}C NMR δ 26.2, 26.4, 28.8, 32.1, 40.9, 44.7, 73.9, 120.0, 126.1, 128.7, 140.3; HRMS(EI) calcd for $\text{C}_{14}\text{H}_{18}\text{ClNONa}$ 274.0969 found 274.0971; For the diastereomeric mixture of **18b**: IR (film) 2242 cm^{-1} ; ^1H NMR δ 1.42-1.76 (m, 16H), 2.78-2.84 (m, 4H), 3.49-3.56 (m, 4H), 4.77-4.80 (m, 2H), 7.38 (s, 5H), 7.39 (s, 5H); HRMS calcd for $\text{C}_{14}\text{H}_{18}\text{ClNONa}$ 274.0969 found 274.0961.

2-Hydroxy-2, 3, 3-trimethyl-cyclohexanecarbonitrile (13), 2-Isopropyl-2-methyl-tetrahydro-pyran-3-carbonitrile (12f), and 2-(3-Hydroxy-propyl)-3,4-dimethyl-pent-2-enenitrile (22): Neat ketone **19** (322 mg, 3.72 mmol) was added to a 0 °C, THF solution of *t*-BuOK (700 mg, 6.23 mmol) followed, after 15 min, by neat **10f** (200 mg, 1.24 mmol). The reaction mixture was allowed to warm to room temperature over 1 h and then saturated aqueous NH₄Cl was added. The crude reaction mixture was extracted with EtOAc, and the combined organic extracts were passed through a short plug of silica gel (2 x 1 cm column), concentrated, and purified by radial chromatography (2:8 EtOAc/hexanes) to afford 66.3 mg (32%, 1:1 dr) of **13** as a solid, 16.6 mg (8%) of **12f** as an oil, and 42 mg (20%, 1:1, *E:Z*) of **22** as an oil. Repeating the procedure with the chloronitrile **10a** afforded 31.1 mg (15%) of **12f**, and 64.4 mg (31%, 1:1, *E:Z*) of **22**. Repeating the procedure with 5-iodopentanenitrile (**10g**) afforded 100.1 mg (63%, 1.5:1 equatorial : axial nitrile epimers) of **13** as a solid. Repetitive chromatography provided pure samples of each isomer of **13** with the stereochemistry being assigned by x-ray crystallography. For **13 trans**: IR (film) 3485, 2241 cm⁻¹; ¹H NMR δ 1.00 (s, 6H), 1.41 (s, 3H), 1.44-1.75 (m, 6H), 2.00-2.07 (m, 1H), 2.93 (dd, *J* = 12, 4 Hz, 1H); ¹³C NMR δ 20.2, 20.3, 21.5, 25.0, 27.9, 36.8, 38.0, 38.5, 74.2, 121.5; HRMS (ESI) calcd for (M+Na⁺) C₁₀H₁₇NONa 190.1202, found 190.1211. For **13 cis**: IR (film) 3467, 2242 cm⁻¹; ¹H NMR δ 0.94 (s, 3H), 1.01 (s, 3H), 1.38 (s, 3H), 1.13-1.61 (m, 5H), 1.75 (td, *J* = 13, 4.1 Hz, 1H), 1.87-2.18 (m, 1H), 2.66 (dd, *J* = 12, 5 Hz, 1H); ¹³C NMR δ 20.4, 23.3, 24.8, 26.1, 27.9, 35.0, 37.1, 37.7, 73.2, 121.3; HRMS (ESI) calcd for (M+Na⁺) C₁₀H₁₇NONa 190.1202, found 190.1196. For **12f**: IR (film) 2236 cm⁻¹; ¹H NMR δ 0.94-0.98 (m, 6H), 1.31 (s, 3H), 1.38-1.58 (m, 1H), 1.68-1.74 (m, 1H), 1.89-2.10 (m, 3H), 2.80 (dd, *J* = 9, 4 Hz, 1H), 3.62-3.65 (m, 2H); ¹³C NMR δ 16.2, 17.0, 17.3, 23.4, 23.6, 23.5, 33.1, 34.2, 60.4, 75.3, 120.9. For **22-E**: IR (film) 3446, 2209, 1618 cm⁻¹; ¹H NMR δ 1.02 (d, *J* = 6.7

Hz, 6H), 1.75-1.84 (m, 3H), 1.98 (s, 3H), 2.34-2.39 (m, 2H), 2.95 (sept, $J = 7$ Hz, 1H), 3.69 (t, $J = 6$ Hz, 2H); ^{13}C NMR δ 16.9, 20.2, 25.4, 30.0, 31.4, 60.4, 107.9, 119.5, 161.1 MS m/e (M+H) 168. For **22-Z**: IR (film) 3441, 2208, 1622 cm^{-1} ; ^1H NMR δ 1.05 (d, $J = 6$ Hz, 6H), 1.62-1.84 (m, 2H), 1.74 (s, 3H), 2.30 (t, $J = 7$ Hz, 2H), 3.15-3.24 (m, 1H), 3.68 (t, $J = 6$ Hz, 2H); ^{13}C NMR δ 12.6, 20.4, 26.1, 30.9, 35.7, 61.4, 107.1, 118.9, 161.5 MS m/e *Mass spec* (M+H) 168.

VGFF0302.301
DATE 30-3-2

12a *trans*

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SI 8192
TO 8192
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TE 297
FW 7600
O2 5684.205
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LB 0.0
GB 0.0
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CY 8.00
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HZ/CM 122.423
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PPM

VGFF0302-253
DATE 1-3-96

SF 75.469
SY 75.0
O1 6147.202
SI 16384
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SW 17857.143
HZ/PT 2.180
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RG 200
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FM 22400
O2 4004.452
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LB 6.000
GB 0.0
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CY 12.00
F1 200.016P
F2 -178P
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PPM/CM 8.008
SR -1381.58

138.654
128.558
128.328
126.507
119.307

35.223
27.989
23.955

VGFF0402.116
DATE 11-4-2

SF 300.133
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O1 4900.000
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SW 6024.096
HZ/PT 1.471
PM 5.0
RD 2.500
AQ .680
RG 4
NS 8
TE 297
FW 7600
O2 5684.205
DP 4L PD
LB 0.0
GB 0.0
CX 25.00
CY 10.00
F1 10.001P
F2 -196P
HZ/CM 122.423
PPM/CM .408
SR 3441.04

ppm

VGFF0302.246
DATE 25-3-2

SF	75.469
SY	75.0
O1	6147.202
SI	16384
TD	16384
SW	17857.143
HZ/PT	2.180
PW	4.0
RD	3.000
AG	.459
RG	200
NS	3213
TE	297
FW	22400
O2	4004.452
DP	20H 88
LB	6.000
GB	0.0
CX	25.00
CY	12.00
F1	200.016P
F2	-178P
HZ/CM	604.335
PPM/CM	8.008
SR	-1388.12

138.987

128.240
128.007
125.370

118.621

78.457
77.404
76.977
76.554
68.792

35.389

27.033

21.589

VGFF0402.253
DATE 25-4-2

SF 300.133
SY 210.0
O1 4900.000
SI 8192
TD 8192
SW 6024.096
HZ/PT 1.471
PW 5.0
RD 2.500
AG 1.680
RG 4
NS 8
TE 297
FW 7600
O2 5684.205
DP 4L PD
LB 0.0
GB 0.0
CX 25.00
CY 7.00
F1 10.001P
F2 -196P
HZ/CM 122.423
PPM/CM 408
SR 3366.03

VGFF0402.281
DATE 27-4-2

ppm

150.739

143.102

119.053

110.390

109.122

77.433

77.005

76.594

73.504

68.217

31.596

27.716

23.891

12b trans

SF 75.469
SY 75.0
O1 6147.202
S1 16384
T1 16384
SW 17857.143
HZ/PT 2.180
PM 4.0
RD 3.000
AQ .459
RG 200
NS 12786
TE 297
FM 22400
O2 4004.452
DP 20H BB
LB 0.0
GB 0.0
CX 25.00
CY 6.00
F1 200.017P
F2 -1.17BP
HZ/CW 604.335
PPM/CW 8.008
SR -1401.20

12b *trans:cis* mixture

VGFF0402.272
DATE 26-4-2

SF 300.133
SY 210.0
O1 4900.000
S1 8192
TD 8192
SM 6024.096
HZ/PT 1.471
PM 5.0
RD 2.500
AQ .680
RG 4
NS 8
TE 297
FW 7600
O2 5684.205
DP 4L P0
LB 0.0
GB 0.0
CX 25.00
CY 10.00
F1 10.001P
F2 -.196P
HZ/CW 122.423
PPM/CW .408
SR 3367.50

ppm

VGFE0502.091
DATE 8-5-2

SF 75.469
SY 75.0
O1 6147.202
S1 16384
TD 16384
SM 17857.143
HZ/P1 2.180

PM 4.0
RD 3.000
AQ .459
RG 200
NS 13837
TE 297

FM 22400
O2 4004.452
DP 20H BB

LB 0.0
GB 0.0
CX 25.00
CY 8.00
F1 200.016P
F2 -178P
HZ/CM 604.335
PPM/CM 8.008
SR -1394.66

151.375
150.711

142.983
142.087

118.997
118.883
110.420
110.304
109.011
107.287

77.433
77.002
76.588
73.390
73.310
68.598
68.096

32.143
31.479
27.579
26.454
23.797
21.775

12b *trans:cis* mixture

VGFF0602.084
DATE 7-6-2

SF 300.133
SY 210.0
O1 4900.000
SI 8192
TD 8192
SM 6024.096
HZ/PT 1.471

PM 5.0
RD 2.500
AQ .680
RG 2
NS 8
TE 297

FW 7600
O2 5684.205
DP 4L P0

LB 0.0
GB 0.0
CX 25.00
CY 12.00
F1 10.001P
F2 -.196P
HZ/CM 122.423
PPM/CM 3364.56
SR

12c *trans*

PPM

VGFF0604.0901
DATE 1-3-96

SF 75.469
SY 75.0
O1 6147.202
SI 16384
TD 16384
SW 17857.143
HZ/PT 2.180

PM 4.0
RD 3.000
AQ .459
RG 200
NS 14138
TE 297

FW 22400
O2 4004.452
DP 20H BB

LB 6.000
GB 0.0
CX 25.00
CY 10.00
F1 200.017P
F2 -178P
HZ/CM 604.335
PPM/CM 8.008
SR -1405.56

121.535

85.077

77.434
77.013
76.590

68.190

61.791

35.681

29.714
28.005
26.648
24.462

12c trans

PROTON .000
DATE 7-6-2

SF 300.133
SY 210.0
O1 4900.000
S1 8192
TD 8192
SW 6024.096
HZ/PT 1.471
PW 5.0
RD 2.500
AQ 1.680
RG 1
NS 8
TE 297
FW 7600
O2 5684.205
DP 4L P0
LB 0.0
GB 0.0
CX 25.00
CY 10.00
F1 10.001P
F2 -.196P
HZ/CM 122.423
PPM/CM .408
SR 3345.44

12c cis

ppm

VGFF0602.0810
DATE 7-6-2

SF 75.469
SY 75.0
O1 6147.202
S1 16384
TD 16384
SW 17857.143
HZ/PT 2.180

PM 4.0
RD 3.000
AQ .459
RG 200
NS 1062
TE 297

FW 22400
O2 4004.452
DP 20H BB

LB 0.0
GB 0.0
CX 25.00
CY 10.00
F1 200.016P
F2 -.178P
HZ/CM 604.335
PPM/CM 8.008
SR -1394.66

120.615

85.287

77.406

76.992

76.567

69.319

34.601

28.707

27.524

26.195

22.100

12c cis

VGFF0702.251
DATE 25-7-2

SF 300.133
SY 210.0
O1 4900.000
S1 8192
TD 8192
SW 6024.096
HZ/PT 1.471
PW 5.0
RG 2.500
AG 4.680
NS 8
TE 297
FW 7600
O2 5684.205
DP 4L P0
LB 0.0
GB 0.0
CX 25.00
CY 10.00
F1 10.001P
F2 -196P
HZ/CM 122.423
PPM/CM 408
SR 3360.15

PPM

VGFF0602.154
DATE 14-6-2

SF 75.469
SY 75.0
O1 6147.202
SI 16384
TD 16384
SM 17857.143
HZ/P1 2.180
PM 4.0
RD 3.000
AQ .459
RG 200
NS 548
TE 297
FM 22400
O2 4004.452
DP 20H BB
LB 0.0
GB 0.0
CY 25.00
F1 200.016P
F2 -2.979P
HZ/CM 612.793
PPM/CM 8.120
SR -1394.66

119.921

12d trans

81.020
77.429
76.999
76.567

67.839

33.587

27.868

24.171

14.949

3.216

1.692

VGFF0602.152
DATE 14-6-2

SF 300.133
SY 210.0
O1 4900.000
SI 6192
TD 8192
SM 6024.096
HZ/PT 1.471
PM 5.0
RD 2.500
AQ .680
RG 1
NS 8
TE 297
FW 7600
O2 5684.205
DP 4L P0
LB 0.0
GB 0.0
CX 25.00
CY 10.00
F1 10.001P
F2 .196P
HZ/CM 122.423
PPM/CM .408
SR 3349.85

12d cis

ppm

VGFF0602.159
DATE 14-6-2

SF 75.469
SY 75.0
O1 6147.202
SI 16384
TD 16384
SW 17857.143
HZ/P1 2.180

PW 4.0
RD 3.000
AQ 459
RG 200
NS 2527
TE 297

FW 22400
O2 4004.452
DP 20H BB

LB 0.0
GB 0.0
CX 25.00
CY 8.00
F1 200.016P
F2 -2.979P
HZ/CM 612.793
PPM/CM 8.120
SR -1396.84

119.668

81.019
77.438
77.007
76.586

68.397

32.784

26.660

21.895

15.043

14.344

3.669

1.416

12d cis

~~BRUKER~~

VGFF0402.1210
DATE 12-4-2

SF 300.133
SY 210.0
O1 4900.000
SI 8192
TD 8192
SW 6024.096
HZ/PT 1.471

PM 5.0
RD 2.500
AG .680
RG 2
NS 6
TE 297

FW 7600
O2 5584.205
DP 4L P0

LB 0.0
GB 0.0
CX 25.00
CY 10.00
F1 10.001P
F2 -.196P
HZ/CM 122.423
PPM/CM .408
SR 3364.56

16a

PPM
190.188

VGFF0402.131
DATE 13-4-2

SF 75.469
SY 75.0
O1 6147.202
SI 16384
TD 16384
SW 17857.143
HZ/PT 2.180
PM 4.0
RD 3.000
AQ 459
RG 200
NS 14979
TE 297
FW 22400
O2 4004.452
DP 20H 88
LB 0.0
GB 0.0
CX 25.00
CY 10.00
F1 210.010P
F2 -178P
HZ/CW 634.504
PM/CW 8.408
SR -1399.02

134.554
133.757
129.105
128.699

116.851

16a

77.406
76.985
76.557

43.604
39.019

29.430
26.888

VGFF0502.304
DATE 28-5-2

SF	300.133
SY	210.0
O1	4900.000
SI	8192
TD	8192
SW	6024.096
HZ/PT	1.471
PW	5.0
RD	2.500
RG	.680
NS	4
TE	8
TE	297
FM	7600
O2	5684.205
DP	4L P0
LB	0.0
GB	0.0
CX	25.00
CY	8.00
F1	10.001P
F2	-196P
HZ/CM	122.423
PPM/CM	.408
SR	3364.56

ppm

VGFF0802.085
DATE 7-8-2

SF 75.469
SY 75.0
O1 6147.202
SI 16384
TD 16384
SW 17857.143
HZ/PT 2.160

PW 4.0
RD 3.000
AG .459
RG 200
NS 729
TE 297

FW 22400
O2 4004.452
DP 20H BB

LB 5.000
GB 0.0
CX 25.00
CY 10.00
F1 200.017P
F2 -178P
HZ/CM 604.335
PPM/CM 8.008
SR -1401.20

122.381

77.439
77.019
76.595

10b

43.948

31.229

29.740

24.960

17.952

DE
DATE 3-6-2

SF 300.134
SY 210.0
O1 4900.000
SI 8192
TD 8192
SM 6024.096
HZ/PT 1.471
PW 5.0
RD 2.500
AQ .680
RG 2
NS 4
TE 297
FW 7600
O2 4451.428
DP 4L PD
LB 0.0
GB 0.0
CX 25.00
CY 10.00
F1 10.001P
F2 -.196P
HZ/CM 122.423
PPM/CM .408
SR 3405.74

12e trans

PPM

VGFF0602.011
DATE 3-6-2

SF 75.469
SY 75.0
O1 6147.202
SI 16384
TD 16384
SW 17857.143
HZ/PT 2.180
PW 4.0
RD 3.000
AQ .459
RG 200
NS 16976
TE 297
FW 22400
O2 4004.452
DP 20H BB
LB 0.0
GB 0.0
CX 29.00
CY 8.00
F1 200.017P
F2 -178P
HZ/CM 604.335
PPM/CM 8.008
SR -1399.02

137.023
128.673
128.098
127.462
126.629
121.680

85.780
77.407
76.995
76.567
69.028
46.817
39.539
36.507
23.629
23.194

VGFF0402.133
DATE 13-4-2

11a trans

SF 300.133
SY 210.0
O1 4900.000
SI 8192
TD 8192
SW 6024.096
HZ/PT 1.471
PW 5.0
RD 2.500
AQ .680
RG 4
NS 8
TE 297
FW 7600
O2 5684.205
DP 4L P0
LB 0.0
GB 0.0
CX 25.00
CY 10.00
F1 10.001P
F2 -.196P
HZ/CM 122.423
PPM/CM .408
SR 3370.44

PPM

VGFF0402.141
DATE 14-4-2

SF	75.469
SY	75.0
O1	6147.202
SI	16384
TD	16384
SW	17857.143
HZ/PT	2.180
PW	4.0
RD	3.000
RG	.459
NS	22404
TE	297
FW	22400
O2	4004.452
DP	20H BB
LB	0.0
GB	0.0
CX	25.00
CY	12.00
F1	200.017P
F2	-178P
HZ/CM	604.335
PPM/CM	8.008
SR	-1401.20

VGFE0402.1910
DATE 11-4-2

SF	300.133
SY	210.0
O1	4900.000
S1	8192
TD	8192
SW	6024.096
HZ/PT	1.471
PW	5.0
RD	2.500
AG	.680
RG	10
NS	8
TE	297
FW	7600
O2	5684.205
DP	4L P0
LB	2.000
GB	0.0
CX	25.00
CY	8.00
F1	10.001P
F2	-196P
HZ/CM	122.423
PPM/CM	.408
SR	3368.97

PPM

VGFF0402.083
DATE 6-4-2

SF 75.469
SY 75.0
O1 6147.202
SI 16384
TD 16384
SW 17857.143
HZ/PT 2.180

PM 4.0
RD 3.000
AQ .459
RG 200
NS 1312
TE 297

FW 22400
O2 4004.452
DP 20H BB

LB .500
GB 0.0
CX 25.00
CY 10.00
F1 200.016P
F2 .178P
HZ/CM 604.335
PPM/CM 8.008
SR -1392.48

138.590

128.617
128.440
125.346

119.688

83.499

77.429
77.000
76.572

67.786

36.852

30.901

~~B~~ ~~11b~~ ~~trans~~

11b trans

VGFE0402.305
DATE 29-4-2
SF 300.133
SY 210.0
O1 4900.000
S1 8192
TD 8192
SW 6024.096
HZ/PT 1.471
PM 5.0
RD 2.500
AQ .680
RG 4
NS 8
TE 297
FW 7600
O2 5684.205
DP 4L PD
LB 0.0
GB 0.0
CX 25.00
CY 7.00
F1 10.001P
F2 .196P
HZ/CM 122.423
PM/CM .408
SR 3366.03

PPM

VGFF0402.311
DATE 1-3-96SF 75.469
SY 75.0
O1 6147.202
SI 16384
TD 16384
SM 17857.143
HZ/PI 2.180PM 4.0
RD 3.000
AG .459
RG 200
NS 16754
TE 297FM 22400
O2 4004.452
DP 20H BBLB 0.0
GB 0.0
CX 25.00
CY 7.00
F1 200.017P
F2 -178P
HZ/CM 604.335
PPM/CM 8.008
SR -1399.02

150.277

143.207

119.547

110.423
109.09177.414
77.000
76.567

67.759

33.098
30.990 **11b trans**

11b cis

VGFF0402:3010
DATE 30-4-2

SF 300.133
SY 210.0
O1 4900.000
S1 8192
TD 8192
SN 6024.096
HZ/PT 1.471

PM 5.0
RD 2.500
AQ .680
RG 40
NS 8
TE 297

FW 7600
O2 5684.205
DP 4L P0

LB 0.0
GB 0.0
CX 25.00
CY 12.00
F1 10.001P
F2 -196P
HZ/CW 122.423
PMW/CW .408
SR 3367.50

VSFF0602.131
DATE 13-6-2

SF 300.133
SY 210.0
O1 4900.000
SI 8192
TD 8192
SM 6024.096
HZ/PT 1.471
PM 5.0
RD 2.500
AQ .680
RG 2
NS 8
TE 297
FW 7600
O2 5684.205
DP 4L P0
LB 0.0
GB 0.0
CX 25.00
CY 12.00
F1 10.001P
F2 -196P
HZ/CM 122.423
PPM/CM 408
SR 3364.56

11c trans

PPM

F.001
DATE 1-3-96

SF 75.469
SY 75.0
Q1 6147.202
SI 16384
TD 16384
SW 17857.143
HZ/PT 2.180

PM 4.0
RD 3.000
AQ .459
RG 200
NS 1171
TE 297

FW 22400
O2 4004.452
DP 20H BB

LB 0.0
GB 0.0
CX 25.00
CY 10.00
F1 200.017P
F2 -178P
HZ/CM 604.335
PPM/CM 8.008
SR -1403.38

121.423

90.519

77.428
77.001
76.570

67.702

11c trans

34.080
31.944
28.621
25.530

VGFF0702.303
DATE 29-7-2

SF 300.133
SY 210.0
O1 4900.000
S1 8192
TD 8192
SM 6024.096
HZ/PT 1.471
PM 5.0
RD 2.500
AQ 1.680
RG 16
NS 8
TE 297
FW 7600
O2 5684.205
DP 4L P0
LB 0.0
GB 0.0
CX 25.00
CY 10.00
F1 10.001P
F2 -196P
HZ/CW 122.423
PPM/CW .408
SR 3366.03

11d trans

PPM

VGFF0702.286
DATE 1-3-96

SF 75.469
SY 75.0
O1 6147.202
SI 16384
TD 16384
SW 17857.143
HZ/PT 2.180

PW 4.0
RD 3.000
AQ .459
RG 200
NS 714
TE 297

FW 22400
O2 4004.452
DP 20H BB

LB 4.000
GB 0.0
CX 25.00
CY 10.00
F1 200.016P
F2 -2.979P
HZ/CN 612.793
PPM/CN 8.120
SR -1396.B4

120.236

86.333

77.431
77.005
76.585

67.154

33.759
30.796

13.824

2.712
1.464

VGFF0702.282
DATE 27-7-2

SF 300.133
SY 210.0
D1 4900.000
S1 8192
TD 8192
SW 8024.096
HZ/PT 1.471
PM 5.0
RD 2.500
AQ .680
RG 4
NS 8
TE 297
FW 7600
D2 5684.205
DP 4L PD
LB 0.0
GB 0.0
CX 25.00
CY 5.00
F1 9.999P
F2 -.198P
HZ/CM 122.423
PPM/CM .408
SR 3368.15

PPM

VGFF0702.284
DATE 27-7-2

SF 75.469
SY 75.0
O1 6147.202
S1 16384
TD 16384
SW 17857.143
HZ/PT 2.180

PW 4.0
RO 3.000
AQ .459
RG 200
NS 556
TE 297

FW 22400
O2 4004.452
DP 20H BB

LB 4.000
GB 0.0
CX 25.00
CY 10.00
F1 200.016P
F2 -3.990P
HZ/CM 615.845
PPM/CM 8.160
SR -1394.66

119.527

84.021

77.408

76.983

76.561

66.662

33.562

30.984

12.102

3.583

1.825

VGFF0502.3410
DATE 30-5-2

SF 300.133
SY 210.0
O1 4900.000
S1 8192
TD 8192
SW 6024.096
HZ/PT 1.471
PW 5.0
RD 2.500
AQ .680
RG 2
NS B
TE 297
FM 7600
O2 5684.205
DP 4L P0
LB 0.0
GB 0.0
CX 25.00
CY 10.00
F1 10.001P
F2 -196P
HZ/CM 122.423
PMM/CM .408
SR 3367.50

18a diastereomeric mixture

PPM

VGFF0502.181
DATE 18-5-2

SF 75.469
SY 75.0
O1 6147.202
SI 16384
TD 16384
SW 17857.143
HZ/PT 2.180

PW 4.0
RD 3.000
AG .459
RG 200
NS 19715
TE 297

FW 22400
O2 4004.452
DP 20H 8B

LB 0.0
GB 0.0
CX 25.00
CY 13.00
F1 200.017P
F2 178P
HZ/CM 604.335
PPM/CM 8.008
SR -1403.38

128.859
126.367
126.133
119.810

18a diastereomeric mixture

77.433
77.002
76.585
73.970
73.648

44.280
40.923
40.318
31.951
31.884
28.323
27.175
24.522

VGFF0802.089
DATE 7-8-2

SF 300.133
SY 210.0
O1 4900.000
SI 8192
TD 8192
SW 6024.096
HZ/PT 1.471

PW 5.0
RD 2.500
AQ .680
RG 10
NS 8
TE 297

FW 7600
O2 5684.205
DP 4L P0

LB 0.0
GB 0.0
CX 25.00
CY 10.00
F1 10.001P
F2 -.196P
HZ/CM 122.423
PPM/CM .408
SR 3368.97

ppm

F.001
DATE 6-8-2

SF 75.469
SY 75.0
O1 6147.202
SI 16384
TD 16384
SM 17857.143
HZ/PT 2.180

PM 4.0
RD 3.000
AG .459
RG 200
NS 476
TE 297

FW 22400
O2 4004.452
DP 20H BB

LB 5.000
GB 0.0
CX 25.00
CY 10.00
F1 200.017P
F2 -178P
HZ/CW 604.335
PPM/CW 8.008
SR -1399.02

140.288

128.740

126.108

120.019

77.436

77.011

76.593

73.913

44.720

40.900

32.065

28.757

26.398

26.198

VGFF0402.119
DATE 11-4-2

SF 300.133
SY 210.0
O1 4900.000
S1 8192
TD 8192
SM 6024.096
HZ/PT 1.471
PW 5.0
RD 2.500
AQ .680
RG 2
NS 8
TE 297
FW 7600
O2 5684.205
DP 4L P0
LB 0.0
GB 0.0
CX 25.00
CY 12.00
F1 10.001P
F2 .196P
HZ/CM 122.423
PPM/CM .408
SR 3364.56

13 trans

ppm

VGFF0402.121
DATE 12-4-2

SF 75.469
SY 75.0
O1 6147.202
SI 16384
TD 16384
SW 17857.143
HZ/PT 2.180

PW 4.0
RD 3.000
AQ .459
RG 200
NS 16409
TE 297

FW 22400
O2 4004.452
DP 20H BB

LB 0.0
GB 0.0
CX 25.00
CY 12.00
F1 200.017P
F2 -178P
HZ/CM 604.335
PPM/CM 8.008
SR -1403.38

121.511

77.405
76.990
76.567
74.166

38.498
37.977
36.772

27.917
25.010
21.537
20.302
20.157

13 trans

VGFF0302.055
DATE 5-3-2

13 cis

SF 300.133
SY 210.0
Q1 4900.000
SI 8192
TD 8192
SM 6024.096
HZ/PT 1.471
PM 5.0
RD 2.500
AQ .680
RG 8
NS 8
TE 297
FW 7600
O2 5684.205
DP 4L P0
LB 0.0
GB 0.0
CX 25.00
CY 10.00
F1 10.001P
F2 -.196P
HZ/CM 122.423
PPM/CM .408
SR 3364.56

PPM

VGFF0202.081
DATE 8-2-2

SF 75.469
SY 75.0
O1 6147.202
SI 16384
TD 16384
SW 17857.143
HZ/PT 2.180
PM 4.0
RD 3.000
AQ .459
RG 200
NS 15969
TE 297
FW 22400
O2 4004.452
DP 20H 88
LB 4.000
GB 0.0
CX 25.00
CY 13.00
F1 200.017P
F2 -.178P
HZ/CM 604.335
PPM/CM 8.008
SR -1403.38

121.344

77.406
76.981
76.562
73.232

37.688
37.145
35.004

27.910
26.093
24.775
23.251
20.420

13 cis

VGFE0302.223
DATE 22-3-2

SF 300.133
SY 210.0
Q1 4900.000
SI 8192
TD 8192
SW 6024.096
HZ/PT 1.471
PM 5.0
RD 2.500
AQ 4.680
RG 4
NS 8
TE 297
FW 7600
O2 5684.205
DP 4L PD
LB 0.0
GB 0.0
CX 25.00
CY 10.00
F1 10.001P
F2 1.196P
HZ/CM 122.423
PRM/CM 408
SR 3366.03

PPM

VGFF0302.031
DATE 3-3-2

SF 75.469
SY 75.0
O1 6147.202
SI 16384
TD 16384
SW 17857.143
HZ/PT 2.180
PM 4.0
RD 3.000
AG 1.459
RG 200
NS 16990
TE 297
FM 22400
O2 4004.452
DP 20H BB
LB 6.000
GB 0.0
CX 25.00
CY 10.00
F1 200.017P
F2 -178P
HZ/CM 604.335
PPM/CM 8.008
SR -1405.56

120.885

77.426
77.004
76.581
75.289

60.360

34.150
33.122

23.596
23.447

17.260
17.012
16.233

VGFF0302.104
DATE 10-3-2

SF 300.133
SY 210.0
O1 4900.000
S1 8192
T0 8192
SW 8024.096
HZ/PT 1.471
PM 5.0
RD 2.500
AQ .680
RG 10
NS B
TE 297
FW 7600
O2 5684.205
DP 4L P0
LB 0.0
GB 0.0
CX 25.00
CY 7.00
F1 10.001P
F2 .196P
HZ/CM 122.423
PPM/CM .408
SR 3364.56

VGFF02021E02
DATE 20-2-2

PPM
176.544
175.801
174.596
165.216
163.709
161.542
161.052
148.800
140.054

119.524
118.909

107.947
107.064
103.349

77.442
77.023
76.597

61.400
60.369

35.691
31.448
30.885
30.030
26.051
25.395
23.583
20.394
20.188
16.925
14.132
12.603

SF 75.469
SY 75.0
O1 6147.202
SI 16384
TD 16384
SM 17857.143
HZ/PT 2.180

PM 4.0
RD 3.000
AQ 1.459
RG 200
NS 16214
TE 297

FW 22400
O2 4004.452
DP 20H 88

LB 5.000
GB 0.0
CX 25.00
CY 12.00
F1 200.017P
F2 207P
HZ/CM 604.422
PPM/CM 8.009
SR -1403.38

VGFF0202.232
DATE 23-2-2

SF 300.133
SY 210.0
O1 4900.000
SI 8192
TD 8192
SW 6024.096
HZ/PT 1.471

PM 5.0
RD 2.500
AQ .680
RG 20
NS 8
TE 297

FW 7600
O2 5684.205
DP 4L P0

LB 0.0
GB 0.0
CX 25.00
CY 10.00
F1 10.001P
F2 -196P
HZ/CM 122.423
PPM/CM 408
SR 3366.03

PPM
161.518

VGFF0202.251
DATE 1-3-96

SF 75.469
SY 75.0
O1 6147.202
SI 16384
TD 16384
SM 17857.143
HZ/PT 2.180

PM 4.0
RD 3.000
AG .459
RG 200
NS 14899
TE 297

FW 22400
O2 4004.452
DP 20H 88

LB 0.0
GB 0.0
CX 25.00
CY 10.00
F1 200.017P
F2 -178P
HZ/CM 604.335
PPM/CM 8.008
SR -1403.38

118.913

107.068

77.411
77.001
76.568

61.405

35.700

30.905

26.052

20.417

12.595

- ¹ For general experimental procedures see: Fleming, F.F.; Hussain, Z.; Weaver, D.; Norman, R.E. *J. Org. Chem.* **1997**, *62*, 1305. The high-resolution mass spectra were recorded at The Ohio State University Campus Chemical Instrumentation Center.
- ² 2,2-Dimethyl propionaldehyde in *t*-BuOH was passed through a short plug of silica immediately before using in the reaction.
- ³ Although **12a-trans** and **12a-cis** have been previously characterized by 200 MHz ¹H NMR full spectral data is provided since the 300 MHz NMR reported allows characterization of previously unassignable multiplets: Makosza, M.; Judka, M. *Chem. Eur. J.* **2002**, *8*, 4234.