Supporting Information

A Rapid Divergent Synthesis of Highly Substituted δ -Lactones

R. Karl Dieter* and Fenghai Guo

Howard L. Hunter Laboratory, Department of Chemistry, Clemson University, Clemson
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General. NMR spectra were recorded as CDCl₃ solutions on a 300 or 500 MHz instruments. The ¹H NMR chemical shifts are reported as δ values in parts per million (ppm) relative to tetramethylsilane (TMS) or CHCl₃ (δ = 7.24) as internal standard. The ¹³C NMR chemical shifts are reported as δ values in parts per million (ppm) downfield from TMS and are referenced with respect to the CDCl₃ signal (triplet, centerline δ = 77.0 ppm). Infrared (IR) spectra were recorded as neat samples (liquid films on NaCl plates). Gas chromatography-mass spectrometry measurements were performed on a GC coupled to a mass selective detector at 70 eV. Analytical thin layer chromatography (TLC) was

performed on silica gel plates, 200 μ mesh with F₂₅₄ indicator. Visualization was accomplished by UV light (254 nm), and/or a 10% ethanol solution of phosphomolybdic acid. Flash chromatography was performed with 200-400 μ silica gel. Elemental analyses were determined from doubly purified samples involving chromatography and/or kugelrohr distillation. The yields are of materials isolated by column chromatography. The preparation of (Z)-3-iodo-2-hepten-1-ol has been reported.¹

General procedure A: Preparation of (Z)-3-Iodo-2-alken-1-ols.² (Z)-3-iodo-2-alkenoate³ (20 mmol) was dissolved in dry Et₂O (30 mL) under an argon atmosphere, cooled to 0 °C (ice bath). Diisobutylaluminum hydride (DIBAL) (40 mL, 1.0 M in cyclohexane, 40 mmol) was added dropwise over 30 minutes, and then the solution was stirred for 30 minutes at 0 °C. The reaction mixture was quenched with 10 mL NH₃-NH₄Cl solution (pH $\approx$ 8), stirred for 1 hour, MgSO₄ (3.00 g) was added and the mixture was stirred for 1 hour. The reaction mixture was filtered through Celite, and the filtrate was concentrated in vacuo to give crude product. Purification by flash column chromatography (silica gel, 10% ethyl acetate: petroleum ether, v/v) gave pure title allylic alcohols.

General procedure B: Representative Procedure for Preparation of *in situ* Generated Phosphates from Alcohol 6a-c. ⁿBuLi (3.80 mL, 2.00 M, 7.60 mmol) was added to a solution of hexamethyldisilazane (1.29 g, 8.00mmol) in THF (6.0 mL) at 0 °C and stirred for 10 minutes. Then the solution was warmed up to 25 °C and stirred for 30 minutes. The solution was cooled to -78 °C, **6a** (705 mg, 6.18 mmol) in THF (6 mL) was added and solid material was observed floating on the solvent surface immediately. The reaction mixture was warmed to -20 °C and stirred for 1.5 hr. Then reaction was cooled to -78 °C, and diethyl chlorophosphate (1.12 g, 6.49 mmol) was added, keep stirring at -78 °C for 1.5 hr before it was used in *in situ* for cuprate reactions.

ⁿBuLi (3.80 mL, 2.00 M, 7.60 mmol) was added to a solution of hexamethyldisilazane (1.29 g, 8.00mmol) in THF (6.0 mL) at 0 °C and stirred for 10 minutes. Then the solution was warmed up to 25 °C and stirred for 30 minutes. The

solution was cooled to -78 °C, **6b/6c** (6.18 mmol) in THF (6 mL) was added and stirred for 1 hr at -78 °C. Diethyl chlorophosphate (1.12 g, 6.49 mmol) was added at -78 °C and stirred for 2 hrs at this temperature before it was used *in situ* for cuprate reactions.

General Procedure C: Reactions of Alkylcyanozinc cuprates (RCuCNZnLi) with the *in situ* Generated Phosphates to Form 7a-b. Alkyl lithium (1.00 mmol) was added to a flask under argon followed by addition of THF (2.0 mL). The flask was cooled to -78 °C, followed by addition of a THF solution of ZnBr₂ (236 mg, 1.05 mmol, 4 mL THF) and the solution was stirred for 15 min. Then a solution of CuCN (89 mg, 1.0 equiv) and LiCl (85 mg, 2.0 equiv) in THF (3.0 mL) was added at -78 °C, and the solution was stirred for an additional 45 minutes at -78 °C. The *in situ* generated phosphate prepared from alcohol **6a** (114 mg, 1.00 mmol) was added dropwise as a THF solution (3.0 mL) via syringe over 2.0 minutes to the cuprate solution at -78 °C. The reaction mixture was allowed to warm up to room temperature over night, quenched with saturated aqueous NH₄Cl (10 mL) solution, and extracted with Et₂O (3×15 mL). The combined organic phase was dried over anhydrous MgSO₄, concentrated in vacuo, and purified by flash column chromatography ((silica gel, 10-15% ethyl acetate: petroleum ether, v/v) to give **7a-b**.

General Procedure D: Reactions of Alkylcyanocuprates (RCuCNLi) with *in situ* Generated Phosphates to Form 8a-d, 9a-d. Alkyl lithium (1.00 mmol) was added to a flask under argon followed by addition of THF (2.0 mL). The flask was cooled to -78 °C, followed by addition of a solution of CuCN (89 mg, 1.0 equiv) and LiCl (85 mg, 2.0 equiv) in THF (3.0 mL) at -78 °C and the solution was stirred for an additional 45 minutes at -78 °C. The *in situ* generated phosphate prepared from alcohol **6b/6c** (1.00 mmol) was added dropwise as a THF solution (3.0 mL) via syringe over 2.0 minutes to the cuprate solution at -78 °C. The reaction mixture was allowed to warm up to room temperature over night, quenched with sat. aqueous NH₄Cl (10 mL) solution, and extracted with Et₂O (3×15 mL). The combined organic phase was dried over anhydrous MgSO₄, concentrated in vacuo, and purified by flash column chromatography (silica gel, 10-15% ethyl acetate: petroleum ether, v/v) to give **8a-d, 9a-d**.

General Procedure E: One-pot Tandem Phosphorylation, Cuprate Mediated Allylic Substitution, and Cuprate Mediated 1,4-Conjugate Addition with Allylic Alcohol (6a). R₁Li (1.00 mmol) was added to a flask under argon followed by addition of THF (2.0 mL). The solution was cooled to -78 °C, followed by addition of a THF (4.0 mL) solution of ZnBr₂ (236 mg, 1.05 mmol) and the solution was stirred for 15 min. Then a solution of CuCN (89 mg, 1.0 equiv) and LiCl (85 mg, 2.0 equiv) in THF (3.0 mL) was added at -78 °C and the solution was stirred for additional 45 minutes at -78 °C. The *in situ* generated phosphate prepared from alcohol **6a** (114 mg, 1.00 mmol) was added dropwise as a THF solution (3.0 mL) via syringe over 2.0 minutes to the cuprate solution at -78 °C. The reaction mixture was warmed up to 0 °C during 4 hrs stirring, then warmed up to r. t. during 2 hrs stirring, then reaction mixture was cooled to -78 °C, TMSCl (325 mg, 3.00 mmol) was added, followed by addition of R₂CuCNLi (mono cuprate, 1.0eq). The reaction mixture was stirred overnight, quenched with aqueous saturated NH₄Cl solution, and extracted with diethyl ether (3×30 mL). The organic phases were combined, dried over anhydrous MgSO₄, and concentrated in vacuo to give crude product. Purification by flash column chromatography gave **13a-b**, **14a-b**.

Preparation of (Z)-1-[Dimethyl(1,1-dimethylethyl)silyloxy]-3-iodo-hept-2-ene (4b).

(Z)-3-Iodo-2-hepten-1-ol (4.30 g, 17.00 mmol) was dissolved in dry dichloromethane, cooled to 0 °C. Triethyl amine (3.43 g, 34.00 mmol) and TBDMSCl (3.60 g, 24.00 mmol) were added, and the reaction mixture was stirred overnight. The reaction mixture was quenched with saturated aqueous NH₄Cl solution, extracted with dichloromethane (3×30 mL). The organic phase was dried over anhydrous MgSO₄, and concentrated in vacuo to give crude product. Purification by flash column chromatography (silica gel, 100% petroleum ether) gave **4b** (5.67 g, 91%): ¹H NMR δ 0.00 (s, 6H), 0.83 (s, 9H), 0.83 (t, *J* = 7.35 Hz, 3H), 1.22 (sex, *J* = 7.35 Hz, 2H), 1.42 (quint, 7.35 Hz, 2H), 2.38 (t, *J* = 6.85 Hz, 2H), 4.12 (dd, *J* = 4.15, 0.9 Hz, 2H), 5.66 (t, *J* = 4.15 Hz, 1H); ¹³C NMR δ - 4.9, 13.8, 18.3, 21.3, 25.9, 31.3, 44.7, 68.5, 107.4, 134.7.

Ethyl 2-hydroxy-4-[(1,1-dimethylethyl) dimethylsilyloxy]-3-pentenoate (5a):

Vinyl iodide **4a** (2.70 g, 9.00 mmol) was dissolved in dry diethyl ether (20 mL) in 100 mL RB flask under an argon atmosphere. The reaction was cooled to $-78\text{ }^{\circ}\text{C}$, $t\text{-BuLi}$ (11.25 mL, 1.60 M, 18.00 mmol) was added and the reaction was stirred for 15 minutes. Then ZnBr_2 (2.23 g, 9.90 mmol), dissolved in dry diethyl ether was added, and the mixture was stirred at $-78\text{ }^{\circ}\text{C}$ for 30 mins, followed by addition of ethyl glyoxylate (2.75 g, 13.50 mmol, 50% by wt, toluene solution). The reaction mixture was warmed up to r. t. during overnight stirring, quenched with saturated aqueous NH_4Cl solution, and extracted with diethyl ether ($3\times 30\text{ mL}$). The organic phases were combined, dried over anhydrous MgSO_4 and concentrated in vacuo to give crude product. Purification by silica gel column (10% EA/ Pet. Ether) gave **5a** (1.99 g, 81%): IR (neat) 3432 (br s), 2935 (m), 1733 (s), 1460 (m), 1372 (m), 1247 (m), 1083 (s), 826 (m) cm^{-1} ; ^1H NMR δ 0.11 (s, 6H), 0.93 (s, 9H), 1.30 (t, $J = 7.2\text{ Hz}$, 3H), 2.99 (br s, 1H), 4.25 (q, $J = 7.2\text{ Hz}$, 2H), 4.28–4.48 (m, 2H), 4.92 (d, $J = 8.1\text{ Hz}$, 1H), 5.40–5.53 (m, 1H), 5.73–5.87 (m, 1H); ^{13}C NMR δ - 5.1, 14.1, 18.3, 25.9, 60.0, 62.0, 67.9, 126.6, 134.8, 173.5.

Ethyl 3-butyl-2-hydroxy-4-[(1,1-dimethylethyl) dimethylsilyloxy]-3-pentenoate (5b).

Vinyl iodide **4b** (2.21 g, 6.00 mmol) was dissolved in dry diethyl ether (15 mL) under an argon atmosphere. The solution was cooled to $-78\text{ }^{\circ}\text{C}$, $t\text{-BuLi}$ (7.50 mL, 1.60 M, 12.00 mmol) was added via syringe and the reaction mixture was stirred for 15 minutes. Then ZnBr_2 [1.49 g, dissolved in dry diethyl ether (8.0 mL)] was added, and the mixture was stirred at $-78\text{ }^{\circ}\text{C}$ for 30 mins, followed by addition of ethyl glyoxylate (1.84 g, 9.00 mmol, 50% by wt, toluene solution). The reaction mixture was warmed up to r. t. while stirring overnight. Reaction was quenched with aq. saturated NH_4Cl solution, and extracted with diethyl ether ($3\times 20\text{ mL}$). The organic phases were combined, dried over anhydrous MgSO_4 , and concentrated in vacuo to give crude product. Purification by silica gel column (5% EA/ Pet. Ether) gave **5b** (1.77 g, 86%): ^1H NMR δ 0.10 (s, 6H), 0.88 (t, $J = 6.6\text{ Hz}$, 3H), 0.93 (s, 9H), 1.29 (t, $J = 6.9\text{ Hz}$, 3H), 1.25–1.46 (m, 4H), 2.05 (t, $J = 6.6\text{ Hz}$, 2H), 2.50–3.50 (br s, 1H), 4.15–4.30 (m, 2H), 4.30–4.45 (m, 2H), 4.93 (s, 1H), 5.58 (t, $J = 6.0\text{ Hz}$, 1H); ^{13}C NMR δ - 5.3, 13.9, 14.1, 18.3, 22.4, 25.9, 26.9, 30.2, 31.6, 59.8, 62.0, 69.5, 129.9, 137.0, 174.0.

3-Hydroxy-3,6-dihydro-2H-pyran-2-one (6a). Silyl ether **5a** (1.37 g, 5.00 mmol) was dissolved in MeOH (10.00 mL), fluosilicic acid (15.00 mmol, 6.80 mL, 25% wt water solution) was added. The reaction mixture was stirred overnight at r.t. MeOH was removed in vacuo, then the residue was extracted with dichloromethane (3×20 mL). The organic phases were combined, dried over anhydrous MgSO₄, and concentrated in vacuo to give crude product. Purification by flash column chromatography (silica gel, 50% ethyl acetate: petroleum ether, v/v) gave **6a** (451 mg, 89 %): IR (neat) 3415 (br s), 2912 (m), 2880 (m), 1722 (s), 1449 (m), 1400 (m), 1209 (s), 1116 (s), 1012 (m), 974 (m) cm⁻¹; ¹H NMR δ 3.24 (br s, 1H), 4.55-4.85 (m, 1H), 4.85-5.20 (m, 2H), 5.90-6.25 (m, 2H); ¹³C NMR δ 65.1, 68.5, 122.8, 128.4, 172.5.

4-Butyl-3-hydroxy-3,6-dihydro-2H-pyran-2-one (6b). Silyl ether **5b** (2.66 g, 7.70 mmol) was dissolved in MeOH (15.00 mL), fluosilicic acid (23.10 mmol, 10.50 mL, 25% wt water solution) was added. The reaction mixture was stirred overnight. MeOH was removed in vacuo, then the residue was extracted with dichloromethane (3×20 mL). The organic phases were combined, dried over anhydrous MgSO₄, and concentrated in vacuo to give crude product. Purification by flash column chromatography (silica gel, 20% ethyl acetate: petroleum ether, v/v) gave **6b** (1.20 g, 90 %): IR (neat) 3459 (sb), 2929 (m), 2863 (m), 1722 (s), 1460 (m), 1400 (m), 1219 (m), 1121 (m), 1012 (m), 875 (m) cm⁻¹; ¹H NMR δ 0.90 (t, *J* = 7.35 Hz, 3H), 1.34 (sex, *J* = 7.35 Hz, 2H), 1.38-1.55 (m, 2H), 2.17-2.35 (m, 2H), 3.17 (br s, 1H), 4.59 (s, 1H), 4.72-4.86 (m, 2H), 5.64 (t, *J* = 1.4 Hz, 1H); ¹³C NMR δ 13.9, 22.4, 29.4, 30.0, 67.0, 67.4, 115.4, 141.6, 173.7; mass spectrum *m/z* (relative intensity) EI 170 (0.18, M⁺), 141 (1.19), 126 (39), 111 (68), 97 (34), 83 (72), 69 (17), 55 (100).

4-Phenyl-3-hydroxy-3,6-dihydro-2H-pyran-2-one (6c). Silyl ether **5c** (0.90 g, 2.57 mmol) was dissolved in MeOH (10.00 mL), fluosilicic acid (7.71 mmol, 3.50 mL, 25% wt water solution) was added. The reaction mixture was stirred overnight. MeOH was removed in vacuo, then the residue was extracted with dichloromethane (3×20 mL). The organic phases were combined, dried over anhydrous MgSO₄, and concentrated in vacuo to give crude product. Purification by flash column chromatography (silica gel, 20% ethyl

acetate: petroleum ether, v/v) gave **6c** (425 mg, 87 %): ^1H NMR δ 3.44 (br s, 1H), 4.82–4.98 (m, 2H), 4.98–5.06 (m, 1H), 6.02 (t, J = 1.4 Hz, 1H), 7.14–7.44 (m, 5H); ^{13}C NMR δ 65.7, 67.6, 119.9, 127.1, 128.3, 128.4, 135.4, 138.8, 172.5.

5-Methyl-5,6-dihydropyran-2-one (7a).⁴ Employing General Procedure C, MeLi (0.67 mL, 1.50 M, 1.00 mmol), and the allylic phosphate of **6a** (114 mg, 1.00 mmol) gave, after purification by flash column chromatography (silica gel, 15–20% ethyl acetate: petroleum ether, v/v), **7a** (77 mg, 69 %): IR (neat) 3420 (br m), 2913 (s), 2847 (m), 1722 (s), 1465 (m), 1389 (m), 1263 (m), 1214 (m), 1105 (m), 1077 (m), 1039 (m), 821 (m) cm^{-1} ; ^1H NMR δ 1.16 (d, J = 7.2 Hz, 3H), 2.62–2.78 (m, 1H), 4.08 (dd, J = 8.4, 11.1 Hz, 1H), 4.42 (ddd, J = 1.2, 5.1, 11.1 Hz, 1H), 5.98 (dd, J = 2.1, 9.75 Hz, 1H), 6.81 (dd, J = 3.3, 9.6 Hz, 1H); ^{13}C NMR δ 15.5, 28.9, 72.1, 120.3, 151.5, 163.7; mass spectrum m/z (relative intensity) EI 112 (6.6, M^+), 97 (6), 82 (100), 54 (87).

5-Butyl-5,6-dihydropyran-2-one (7b). Employing General Procedure C, *n*-BuLi (0.50 mL, 2.0 M, 1.00 mmol), and the allylic phosphate of **6a** (114 mg, 1.00 mmol) gave, after purification by flash column chromatography ((silica gel, 10–15% ethyl acetate: petroleum ether, v/v), **7b** (108 mg, 70%): IR (neat) 3437 (br m), 2929 (s), 2853 (s), 1717 (s), 1465 (m), 1394 (m), 1274 (m), 1214 (m), 1159 (m), 1110 (m), 1077 (m), 952 (m), 821 (m) cm^{-1} ; ^1H NMR δ 0.14 (t, J = 6.9 Hz, 3H), 1.20–1.45 (m, 4H), 1.45–1.59 (m, 2H), 2.44–2.58 (m, 1H), 4.16 (dd, J = 7.5, 10.95 Hz, 1H), 4.43 (dd, J = 5.1, 11.1 Hz, 1H), 5.99 (dd, J = 1.8, 9.9 Hz, 1H), 6.87 (dd, J = 3.75, 9.75 Hz, 1H); ^{13}C NMR δ 13.8, 22.6, 28.9, 30.2, 33.8, 70.7, 120.4, 150.4, 163.9; GC mass spectrum m/z (relative intensity) EI 154 (0.17, M^+), 136 (2.27), 124 (16), 98 (73), 81 (100), 70 (19), 53 (48).

4-Butyl-5-methyl-5,6-dihydropyran-2-one (8a). Employing General Procedure D, MeLi (0.70 mL, 1.50 M, 1.05 mmol), and the allylic phosphate of **6b** (187 mg, 1.10 mmol) gave, after purification by flash column chromatography (silica gel, 10–15% ethyl acetate: petroleum ether, v/v), **8a** (120 mg, 71 %): IR (neat) 3514 (br w), 2957 (s), 2929 (s), 2869 (s), 1717 (s), 1460 (m), 1214 (m), 1050 (m), 995 (m), 870 (m) cm^{-1} ; ^1H NMR δ 0.94 (t, J = 7.3 Hz, 3H), 1.20 (d, J = 7.35 Hz, 3H), 1.38 (sex, J = 7.35 Hz, 2H), 1.41–1.57 (m, 2H), 2.25 (t, J = 7.8 Hz, 2H), 2.35–2.42 (m, 1H), 4.13 (dd, J = 4.15, 11.00 Hz, 1H),

4.36 (dd, $J = 4.15, 11.00$ Hz, 1H), 5.75 (s, 1H); ^{13}C NMR δ 13.8, 15.3, 22.3, 28.7, 32.2, 34.0, 71.7, 114.7, 164.6, 166.3; mass spectrum m/z (relative intensity) EI 168 (41, M^+), 140 (2.24), 127 (23), 111 (7.45), 96 (100), 81 (28), 67 (47), 53 (24). Anal. Calcd for $\text{C}_{10}\text{H}_{16}\text{O}_2$: C, 71.39; H, 9.58. Found: C, 71.01; H, 9.46.

4, 5-Dibutyl-5,6-dihydropyran-2-one (8b).⁵ Employing General Procedure D, 0.50 mL n -BuLi (2.00 M), and the allylic phosphate of **6b** (187 mg, 1.10 mmol) gave, after purification by flash column chromatography (silica gel, 10-15% ethyl acetate: petroleum ether, v/v), **8b** (152 mg, 72 %): ^1H NMR δ 0.82-0.94 (m, 6 H), 1.21-1.57 (m, 10 H), 2.07-2.13 (m, 1 H), 2.17-2.29 (m, 2 H), 4.25-4.32 (m, 2 H), 5.71 (s, 1 H); ^{13}C NMR δ 13.8, 13.9, 22.3, 22.6, 28.9, 29.2, 29.6, 34.4, 37.6, 68.9, 114.8, 164.7, 165.9; mass spectrum m/z (relative intensity) EI 210 (3.8, M^+), 153 (32), 137 (28), 112 (49), 95 (77), 82 (58), 67 (74), 55 (100).

4-Butyl-5-(1,1-dimethylethyl)-5,6-dihydropyran-2-one (8c). Employing General Procedure D, 0.60 mL t -BuLi (1.60 M), and the allylic phosphate of **6b** (187 mg, 1.10 mmol) gave, after purification by flash column chromatography (silica gel, 10-15% ethyl acetate: petroleum ether, v/v), **8c** (126 mg, 60 %): IR (neat) 3421 (br w), 2957 (s), 2869 (m), 1722 (s), 1465 (m), 1361 (m), 1225 (m), 1077 (m), 979 (m), 864 (m) cm^{-1} ; ^1H NMR δ 0.95 (t, $J = 7.5$ Hz, 3H), 1.07 (s, 9H), 1.29-1.43 (m, 2H), 1.45-1.63 (m, 2H), 1.96 (d, $J = 3.9$ Hz, 1H), 2.23-2.47 (m, 2H), 4.26 (dd, $J = 4.2, 11.7$ Hz, 1H), 4.60 (d, $J = 11.7$ Hz, 1H), 5.91 (s, 1H); ^{13}C NMR δ 13.8, 22.4, 29.1, 30.0, 34.8, 37.6, 47.0, 68.9, 117.4, 163.3, 164.8; mass spectrum m/z (relative intensity) EI 211 (0.04, $[\text{M} + 1]^+$), 165 (2.3), 154 (16), 139 (0.7), 125 (12), 112 (100), 84 (7.5), 57 (25).

4-Butyl-5-phenyl-5,6-dihydropyran-2-one (8d). Employing General Procedure D, PhLi (0.50 mL, 1.80 M), and the allylic phosphate of **6b** (187 mg, 1.10 mmol) gave, after purification by flash column chromatography (silica gel, 100% dichloromethane), **8d** (126 mg, 61 %): IR (neat) 3545 (br w), 2951 (s), 2924 (s), 2864 (s), 1717 (s), 1449 (m), 1241 (s), 1203 (s), 1066 (s), 859 (m), 750 (m) cm^{-1} ; ^1H NMR δ 0.88 (t, $J = 7.2$ Hz, 3H), 1.28 (sex, $J = 7.2$ Hz, 2H), 1.37-1.57 (m, 2H), 2.16 (t, $J = 7.5$ Hz, 2H), 3.55 (t, $J = 4.5$ Hz, 1H), 4.41 (dd, $J = 4.2, 11.40$ Hz, 1H), 4.62 (dd, $J = 4.5$ Hz, 1H), 6.03 (d, $J = 1.2$ Hz,

1H), 7.18–7.42 (m, 5H); ^{13}C NMR δ 13.7, 22.2, 28.6, 34.6, 44.0, 71.9, 116.6, 128.0, 128.1, 129.0, 137.3, 163.0, 164.5; mass spectrum m/z (relative intensity) EI 231 (1.36, $[\text{M} + 1]^+$), 230 (8.65, M^+), 200 (20), 188 (5.67), 158 (100), 141 (5.67), 129 (62), 115 (25), 91 (15), 77 (13), 51 (15).

5-Methyl-4-phenyl-5,6-dihydropyran-2-one (9a). Employing General Procedure D, MeLi (0.60 mL, 1.50 M, 0.90 mmol), and the allylic phosphate of **6c** (188 mg, 0.99 mmol) gave, after purification by flash column chromatography (silica gel, 15% ethyl acetate: petroleum ether, v/v), **9a** (125 mg, 75 %): IR (neat) 3497 (br m), 3049 (m), 2968 (s), 2929 (s), 1706 (s), 1613 (s), 1438 (m), 1351 (m), 1214 (m), 995 (m), 766 (m), 684 (m) cm^{-1} ; ^1H NMR δ 1.08 (d, $J = 7.35$ Hz, 3H), 2.97–3.04 (m, 1H), 4.14 (dd, $J = 11.0, 1.85$ Hz, 1H), 4.34 (dd, $J = 11.45, 3.65$ Hz, 1H), 6.08 (s, 1H), 7.39–7.54 (m, 5H); ^{13}C NMR δ 16.6, 30.7, 71.9, 114.1, 126.4, 129.1, 130.6, 135.0, 160.6, 164.9; mass spectrum m/z (relative intensity) EI 188 (100, M^+), 158 (90), 129 (67), 115 (51), 102 (21), 91 (11), 77 (17), 63 (15), 51 (21).

5-Butyl-4-phenyl-5,6-dihydropyran-2-one (9b). Employing General Procedure D, *n*-BuLi (0.50 mL, 1.60 M, 0.80 mmol), and the allylic phosphate of **6c** (167 mg, 0.88 mmol) gave, after purification by flash column chromatography (silica gel, 15% ethyl acetate: petroleum ether, v/v), **9b** (125 mg, 68 %): IR (neat) 3410 (w), 3060 (m), 2924 (s), 1706 (s), 1613 (s), 1443 (m), 1225 (m), 1077 (m), 865 (m) cm^{-1} ; ^1H NMR δ 0.84 (t, $J = 7.3$ Hz, 3H), 1.18–1.37 (m, 3H), 1.37–1.54 (m, 2H), 1.60–1.70 (m, 1H), 2.79–2.85 (m, 1H), 4.44–4.54 (m, 2H), 6.26 (s, 1H), 7.41–7.55 (m, 5H); ^{13}C NMR δ 18.8, 22.5, 29.7, 29.9, 36.0, 68.9, 114.3, 126.4, 129.1, 130.6, 135.4, 160.1, 165.1; mass spectrum m/z (relative intensity) EI 230 (8, M^+), 174 (70), 157 (30), 146 (85), 129 (43), 128 (46), 118 (100), 115 (36), 102 (10), 91 (15), 77 (13), 51 (10). Anal. Calcd for $\text{C}_{15}\text{H}_{18}\text{O}_2$: C, 78.23; H, 7.88. Found: C, 77.99; H, 7.99.

5-(1,1-Dimethylethyl)-4-phenyl-5,6-dihydropyran-2-one (9c). Employing General Procedure D, *t*-BuLi (0.60 mL, 1.60 M, 0.96 mmol), and the allylic phosphate of **6c** (201 mg, 1.06 mmol) gave, after purification by flash column chromatography (silica gel, 100% dichloromethane), **9c** (142 mg, 62 %). Title compound is clear solid with a melting

point 107.6–108.3 °C. IR (neat) 2953 (br s), 1706 (s), 1456 (m), 1359 (m), 1224 (m), 1090 (m), 767 (m), 704 (m) cm^{-1} ; ^1H NMR δ 0.88 (s, 9H), 2.72 (d, J = 3.9 Hz, 1H), 4.47 (dd, J = 4.2, 11.7 Hz, 1H), 4.75 (d, J = 12 Hz, 1H), 6.24 (s, 1H), 7.30–7.49 (m, 5H); ^{13}C NMR δ 29.2, 35.1, 45.4, 69.0, 118.1, 126.5, 128.9, 129.9, 139.5, 158.8, 165.3; mass spectrum m/z (relative intensity) EI 230 (1.2, M^+), 174 (67), 156 (7), 129 (100), 115 (18), 91 (7), 57 (34).

4, 5-Diphenyl-5,6-dihydropyran-2-one (9d). Employing General Procedure D, PhLi (0.67 mL, 1.80 M, 1.2 mmol), and the allylic phosphate of **6c** (190 mg, 1.00 mmol) gave, after purification by flash column chromatography (silica gel, 10-15% ethyl acetate: petroleum ether, v/v), **9d** (191.50 mg, 77 %). Light yellow solid with melting point 114.4–115.1 °C. IR (neat) 3454 (br s), 2077 (br m), 1706 (s), 1635 (s), 1443 (m), 1351 (m), 1203 (m), 1066 (m), 761 (m) cm^{-1} ; ^1H NMR δ 4.11 (t, J = 1.8 Hz, 1H), 4.50 (dd, J = 11.1, 1.8 Hz, 1H), 4.768 (dd, J = 11.1, 4.0 Hz, 1H), 6.61 (s, 1H), 7.10–7.60 (m, 10H); ^{13}C NMR δ 42.4, 72.6, 116.3, 126.6, 127.8, 127.9, 129.0, 129.2, 130.6, 135.4, 137.4, 156.1, 164.8; mass spectrum m/z (relative intensity) EI 251 (7, $[\text{M} + 1]^+$), 250 (38, M^+), 221 (16), 220 (100), 207 (15), 192 (65), 191 (54), 165 (17), 115 (9), 95 (7), 75 (7), 51 (5).

Methyl 4-(1,1-Dimethylethyl)-6,6-dimethyl-2-heptenoate (12). 4-(1,1-Dimethylethyl)-6,6-dimethyl-2-heptenoic acid (**11**) (40 mg, 0.19 mmol) was dissolved in MeOH (5.0 mL), followed by addition of TMSCl (217 mg, 2.0 mmol). The reaction mixture was stirred overnight. Solvent was removed to give crude product. Purification by flash column chromatography (silica gel, 10% diethyl ether: petroleum ether, v/v) gave **12** (40 mg, 94%): ^1H NMR 0.87 (s, 9H), 0.88 (s, 9H), 1.15-1.30 (m, 2H), 1.99 (t, J = 10.2 Hz, 1H), 3.72 (s, 3H), 5.78 (d, J = 15.6 Hz, 1H), 6.87 (dd, J = 15.6, 10.5 Hz, 1H); ^{13}C NMR 27.7, 30.2, 31.1, 33.9, 42.8, 49.2, 51.3, 121.7, 154.5, 167.0.

4, 5-Dibutyl-3,4,5,6-tetrahydropyran-2-one (13a). Employing General procedure E, *n*-BuLi (0.50 mL, 2.0 M, 1.00 mmol), the allylic phosphate of **6a** (114 mg, 1.00 mmol), and *n*-BuCuCNLi (mono cuprate, 1.0eq) gave, after purification by flash column chromatography (silica gel, 15% ethyl acetate: petroleum ether, v/v), **13a** (129 mg, 61 %): IR (neat) 3476 (br w), 2918 (s), 1749 (s), 1460 (m), 1372 (m), 1252 (m), 1154 (m), 1039

(m), 728 (m) cm^{-1} ; ^1H NMR δ 0.88 (t, J = 6.0 Hz, 6H), 1.18-1.37 (m, 10), 1.37-1.50 (m, 2H), 1.50-1.61 (m, 1H), 1.61-1.76 (m, 1H), 2.20 (dd, J = 16.2, 7.5 Hz, 1H), 2.59 (dd, J = 15.9, 6.6 Hz, 1H), 3.95 (dd, J = 11.4, 7.2 Hz, 1H), 4.26 (dd, J = 11.4, 4.5 Hz, 1H); ^{13}C NMR δ 13.9, 14.0, 22.6, 22.8, 28.5, 29.0, 31.4, 34.2, 35.2, 36.3, 38.5, 70.8, 173.0; mass spectrum m/z (relative intensity) EI 212 (1.8, M^+), 184 (1.6), 155 (91), 140 (61), 111 (25), 97 (26), 83 (26), 69 (57), 55 (100). Anal. Calcd for $\text{C}_{13}\text{H}_{24}\text{O}_2$: C, 73.53; H, 11.39. Found: C, 73.44; H, 11.42.

5-Butyl-4-methyl-3,4,5,6-tetrahydropyran-2-one (13b). Employing General procedure E, *n*-BuLi (0.50 mL, 2.0 M, 1.00 mmol), the allylic phosphate of **6a** (114 mg, 1.00 mmol), and MeCuCNLi (mono cuprate, 1.0eq) gave, after purification by flash column chromatography (silica gel, 15% ethyl acetate: petroleum ether, v/v), **13b** (100 mg, 59 %): IR (neat) 3448 (br s), 2951 (s), 2924 (s), 2864 (m), 1733 (s), 1640 (s), 1454 (m), 1372 (m), 1209 (m), 1088 (m), 1039 (m) cm^{-1} ; ^1H NMR δ 0.88 (t, J = 6.9 Hz, 3H), 0.97 (d, J = 6.9 Hz, 3H), 1.05–1.37 (m, 5 H), 1.39–1.64 (m, 2H), 1.64–1.84 (m, 1H), 2.16 (dd, J = 17.1, 8.7 Hz, 1H), 2.66 (dd, J = 16.9, 6.1 Hz, 1H), 3.86 (dd, J = 11.4, 9.3 Hz, 1H), 4.23 (dd, J = 11.1, 4.5 Hz, 1H); ^{13}C NMR δ 14.0, 15.7, 22.7, 28.1, 33.5, 34.0, 34.7, 37.8, 73.4, 172.3; mass spectrum m/z (relative intensity) EI 170 (0.35, M^+), 140 (1.58), 113 (100), 98 (80), 83 (97), 69 (53), 56 (78).

4-Butyl-5-methyl-3,4,5,6-tetrahydropyran-2-one (14a). Employing General procedure E, MeLi (0.57 mL, 1.75 M, 1.00 mmol), the allylic phosphate of **6a** (114 mg, 1.00 mmol), and *n*-BuCuCNLi (mono cuprate, 1.0eq) gave, after purification by flash column chromatography (silica gel, 15% ethyl acetate: petroleum ether, v/v), **14b** (110 mg, 65 %): IR (neat) 3465 (br m), 2957 (s), 2924 (s), 2858 (m), 1744 (s), 1465 (m), 1372 (m), 1203 (m), 1093 (m), 1039 (m) cm^{-1} ; ^1H NMR δ 0.88 (t, J = 6.7 Hz, 3H), 0.97 (d, J = 6.9 Hz, 3H), 1.06–1.40 (m, 6H), 1.40–1.62 (m, 1H), 1.62–1.80 (m, 1H), 2.16 (dd, J = 16.9, 8.5 Hz, 1H), 2.66 (dd, J = 16.9, 6.1 Hz, 1H), 3.86 (dd, J = 11.1, 9.3 Hz, 1H), 4.23 (dd, J = 11.1, 4.6 Hz, 1H); ^{13}C NMR δ 14.0, 15.7, 22.7, 28.1, 33.5, 34.0, 34.7, 37.8, 73.4, 172.3; mass spectrum m/z (relative intensity) EI 171 (0.6, $[\text{M} + 1]^+$), 140 (1.9), 113 (100), 98 (80), 83 (10), 69 (57), 56 (85).

4, 5-Dimethyl-3,4,5,6-tetrahydropyran-2-one (14b).⁶ Employing General procedure E, MeLi (0.57 mL, 1.75 M, 1.00 mmol), the allylic phosphate of **6a** (114 mg, 1.00 mmol), and MeCuCNLi (mono cuprate, 1.0eq) gave, after purification by flash column chromatography (silica gel, 15% ethyl acetate: petroleum ether, v/v), **14b** (70 mg, 55 %): ¹H NMR δ 0.99 (d, *J* = 6.0 Hz, 3H), 1.04 (d, *J* = 6.0 Hz, 3H), 1.56-1.76 (m, 2H), 2.15 (dd, *J* = 9.9, 17.4 Hz, 1H), 2.70 (dd, *J* = 5.7, 17.4 Hz, 1H), 3.88 (dd, *J* = 11.1, 13.5 Hz, 1H), 4.29 (dd, *J* = 4.2, 11.1 Hz, 1H); ¹³C NMR δ 14.5 (11.6), 19.5 (15.9), 33.2 (30.0), 35.0 (31.2), 37.6 (36.4), 74.2 (73.4), 171.5 (*cis*-isomer); mass spectrum *m/z* (relative intensity) EI 128 (11, M⁺), 98 (17), 69 (32), 56 (100).

Reference:

1. Marshall, J. M.; DuBay, W. J. *J. Org. Chem.* **1993**, 58, 3435-3443.
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5. Nangia, A.; Rao, P. B. *Tetrahedron Lett.* **1993**, 34, 2681-2684.
6. Fleming, I.; Reddy, N. L.; Takaki, K.; Ware, C. *J. Chem. Soc., Chem. Commun.*, **1987**, 1472-1474.

guo580-3.jdr

X: parts per Million : 1H

Filename
Author
Experiment
Sample_id
Solvent
Creation_time
Revision_time
Current_time

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- single_pulse.exp
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- 17-AUG-2005 17:33:59
- 17-AUG-2005 17:34:16

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X_freq
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X_resolution
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X_return
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Total_scans

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- 500.15991521 [MHz]
- 51 [ppm]
- 16384
- 0
- 0.45822189 [Hz]
- 7.50750751 [Hz]
- FALSE
- 1
- 8

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Recvr_gain
Relaxation_delay
Temp_get

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- 2 [us]
- 14
- 2 [s]
- 23.3 [deg]

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Data_title
Data_units
Dimensions
Site
Spectrometer

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- 1H
- [ppm]
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- DELTA_NMR

guo580-4.jdr

X : parts per Million : 13C

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Revision time   - 17-AUG-2005 17:40:14
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Data_units     - [ppm]
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Site           - Eclipse+ 500
Spectrometer   - DELTA_NMR

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JEOL

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SW 3.332 PPM
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TD 32

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FIDRES: 31.250000 Hz
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Current Data Parameters
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EXPNO 1
PROCNO 1

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F1 - Processing Parameters
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F3 - Processing Parameters
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6a

IR

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6.016
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6.002
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PROCNO 1

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6a

ER

Current Data Parameters
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guo646pure-3.jdf

6b

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JEOL

guo646pure-5.jdf

X : parts per Million : 13C

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 Site = Kelpset+ 500
 Spectrometer = DELTA_NMR
 Field_strength = 11.7473579 [T] (500 [MH
 X_acq_duration = 1.0420224 [s]
 X_domain = 13C
 X_freq = 125.76529768 [MHz]
 X_offset = 100 [ppm]
 X_points = 32768
 X_prescans = 4
 X_resolution = 0.95967227 [Hz]
 X_sweep = 31.44654088 [KHz]
 Irr_domain = 1H
 Irr_freq = 500.15991521 [MHz]
 Irr_offset = 5 [ppm]
 Clipped = FALSE
 Mod_return = 1
 Scans = 450
 Total_scans = 450
 X_90_width = 8.6 [us]

JEOL

guo731bottom-3.jdf

X : parts per Million : 1H

Filename = guo731bottom-3.jdf
 Author =
 Experiment =
 Sample_id =
 Solvent =
 Creation_time = 19-DEC-2005 14:21:43
 Revision_time = 19-DEC-2005 14:21:13
 Current_time = 19-DEC-2005 14:21:17

Content = Single Pulse Experiment
 Data_format = 1D COMPLEX
 Dia_size = 16384
 Dia_title =
 Dia_units = [ppm]
 Dimensions =
 Site =
 Spectrometer = DELTA_NMR

Field_strength = 11.7473579 [T] (500 [MHZ])
 X_acq_duration = 2.1823488 [s]
 X_domain = 1H
 X_freq = 500.15991521 [MHZ]
 X_offset = 5 [ppm]
 X_points = 16384
 X_prescans = 0
 X_resolution = 0.45822189 [Hz]
 X_sweep = 7.50750751 [KHz]
 X_clipped = FALSE
 Mod_return = 1
 Scans = 8
 Total_scans = 8

Initial_wait = 1 [s]
 Phase_preset = 2 [us]
 Recvt_gain = 15
 Relaxation_delay = 21 [s]
 Temp_get = 21.2 [degC]

JEOL

guo731bottom-3.jdf

X : parts per Million : 13C

```

Filename      = guo731bottom-3.jdf
Author        = NMR users
Experiment     = single_pulse_dec
Sample_id      = 2
Solvent        = CHLOROFORM-D
Creation_time   = 19-DEC-2005 14:30:01
Revision_time  = 19-DEC-2005 14:28:34
Current_time   = 19-DEC-2005 14:28:34

Content
Data_format    = Single Pulse with Bro
Dim_size       = 1D COMPLEX
Dim_title      = 13C
Dim_units      = [ppm]
Dimensions      = X
Site            = Eclipses+ 500
Spectrometer    = DEPTX_NMR

Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.0420224[s]
X_domain       = 13C
X_freq         = 125.76529768[MHz]
X_offset       = 100[ppm]
X_points       = 32768
X_prescans     = 4
X_rescans      = 0.95967227[Hz]
X_resolution    = 31.44654088[MHz]
X_sweep        = 1H
Xir_domain     = 1H
Xir_freq       = 500.15991521[MHz]
Xir_offset     = 5[ppm]
Xir_offset     = TRUE
Mod_return     = 1
Scans          = 214
Total_scans    = 214
X_90_width     = 8.6[us]
  
```

JEOL

7a

Current Data Parameters
NAME guo855
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060421
Time 16.09
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 4194.631 Hz
FIDRES 0.128010 Hz
AQ 3.9059956 sec
RG 512
DW 119.200 usec
DE 6.00 usec
TE 296.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCMRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 9.75 usec
PL1 0.00 dB
SFO1 300.131300 MHz

F1 - Acquisition parameters
ND0 1
TD 32
SFO1 300.1314 MHz
FIDRES 31.250000 Hz
SW 3.332 ppm
FNUC0E undefined

F3 - Acquisition parameters
ND0 1
SW 3.332 ppm
SFO1 300.1314 MHz
H2PPT 0.122070 Hz
TD 32

F2 - Processing parameters
SI 32
SF 300.32768 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 32
MC2 QF
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.00 Hz
GB 0

F3 - Processing parameters
SI 32
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.00 Hz
GB 0

Current Data Parameters
NAME guo855
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060421
Time 16.23

INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 265
DS 4
SWH 18115.944 Hz
FIDRES 0.276427 Hz
AQ 1.808436 sec
RG 1024
BW 27.600 usec
DE 296.8 K
D1 2.0000000 sec
d11 0.0500000 sec
DELTA 1.8999998 sec
ACQRES 0.0000000 sec
MCWRR 0.0150000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 10.00 usec
PL1 0.00 dB
SFO1 75.4752958 MHz

***** CHANNEL f2 *****
NAME waltz16
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
P12 17.00 dB
SFO2 300.1315007 MHz

F1 - Acquisition parameters
ND0 1
TD 32
SFO1 300.1314 MHz
PCPD2 80.00 usec
PL2 0.00 dB
P12 17.00 dB
SFO2 300.1315007 MHz
F1 - Acquisition parameters
ND0 1
TD 32
SFO1 300.1314 MHz
PCPD2 80.00 usec
PL2 0.00 dB
P12 17.00 dB
SFO2 300.1315007 MHz
F1 - Acquisition parameters
ND0 1
TD 32
SFO1 300.1314 MHz
PCPD2 80.00 usec
PL2 0.00 dB
P12 17.00 dB
SFO2 300.1315007 MHz

F2 - Processing parameters
SI 65536
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 32
SF 300.130000 MHz
WDW NO
SSB 2
LB 0.00 Hz
GB 0
PC 1.40

F2 - Processing parameters
SI 32
SF 300.130000 MHz
WDW NO
SSB 2
LB 0.00 Hz
GB 0

Current Data Parameters
NAME guo857
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060423
Time 22.16
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 16
SWH 4194.631 Hz
FIDRES 0.128010 Hz
AQ 3.9059956 sec
RG 512
RG 119.200 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCPRK 0.01500000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 9.75 usec
PL1 0.00 dB
SFO1 300.131300 MHz

F1 - Acquisition parameters
ND0 1
TD 32
SFO1 300.1314 MHz
FIDRES 31.250000 Hz
SW 3.332 ppm
FMODE undefined

F3 - Acquisition parameters
ND0 1
SM 3.332 ppm
SFO1 300.1314 MHz
H2DPR 0.12070 Hz
TD 32

F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 32
MC2 QF
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.00 Hz
GB 0

F3 - Processing parameters
SI 32
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.00 Hz
GB 0

7b

Current Data Parameters
NAME: 7b
EXPNO: 1
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20060423
Time: 22.12
INSTRUM: spect
PROBHD: 5 mm QNP 1H/13
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 302
DS: 0
SWH: 18115.941 Hz
FIDRES: 0.276427 Hz
AQ: 1.8088416 sec
RG: 912.3
DW: 27.600 usec
DE: 6.00 usec
TE: 300.2 K
DELTA: 2.0000000 sec
d11: 0.0300000 sec
DELTA: 1.8999998 sec
MCRRST: 0.0000000 sec
MCRRK: 0.0150000 sec

***** CHANNEL f1 *****
NUC1: 13C
P1: 10.00 usec
PL1: 0.00 dB
SFO1: 75.4752958 MHz

***** CHANNEL f2 *****
CPDPRG2: waltz16
NUC2: 1H
PCPD2: 80.00 usec
PL2: 0.00 dB
PL12: 17.00 dB
PL13: 17.00 dB
SFO2: 300.135007 MHz

F1 - Acquisition Parameters
NUC1: 1
TD: 32
SFO1: 300.1314 MHz
FIDRES: 31.25000 Hz
SM: 32.1312 ppm
F2H008: undefined

F3 - Acquisition Parameters
NUC1: 1
SW: 3.332 ppm
SFO1: 300.1314 MHz
RGPRP: 0.125070 Hz
TD: 32

F2 - Processing Parameters
SI: 65536
SF: 75.4677490 MHz
WDW: EM
SSB: 0
LB: 1.00 Hz
GB: 0
PC: 1.40

F1 - Processing Parameters
SI: 32
SF: 300.130000 MHz
WDW: no
SSB: 2
LB: 0.00 Hz
GB: 0

F3 - Processing Parameters
SI: 32
SF: 300.130000 MHz
WDW: no
SSB: 2
LB: 0.00 Hz
GB: 0

gno791-4.jdf

X : parts per Million : 1H

Filename = gno791-4.jdf
 Author = NMR users
 Experiment = single_pulse.exp
 Sample_id = 1
 Solvent = CHLOROFORM-D
 Creation_time = 2-MAR-2006 17:03:33
 Revision_time = 2-MAR-2006 17:18:32
 Current_time = 2-MAR-2006 17:18:46
 Content = Single Pulse Experiment
 Data_format = 1D COMPLEX
 Dim_size = 16384
 Dim_title = 1H
 Dim_units = [ppm]
 Dimensions = X
 Site = ECLIPSE 500
 Spectrometer = DELTA_NMR

Field_strength = 11.7473579 [T] (500 [MH]
 X_acq_duration = 2.1823486 [s]
 X_domain = 1H
 X_freq = 500.15991521 [MHz]
 X_offset = 5 [ppm]
 X_points = 16384
 X_prescans = 0
 X_resolution = 0.45822189 [Hz]
 X_sweep = 7.50750751 [kHz]
 Clipped = FALSE
 Mod_return = 1
 Scans = 8
 Total_scans = 8

Initial_wait = 1 [s]
 Phase_preset = 2 [us]
 Recvr_gain = 17
 Relaxation_delay = 2 [s]
 Temp_get = 22.3 [deg]

X_90_width = 12.1 [us]
 X_acq_time = 2.1823486 [s]
 X_angle = 45 [deg]
 X_pulse = 6.05 [us]

JEOL

guo791-3.jdf

X : parts per Million : 13C

```

Filename      = guo791-3.jdf
Author        = NMR users
Experiment     = single_pulse_dec
Sample_id     = 2
Solvent       = CHLOROFORM-D
Creation time  = 2-MAR-2006 17:25:57
Revision time  = 2-MAR-2006 17:24:04
Current_time   = 2-MAR-2006 17:24:15

Content
Data_format   = Single Pulse w/ich Bro
Dim_size      = 32768
Dim_cicle     = 13C
Dim_units     = [ppm]
Dimensions    = x
Site          = ECLIPSE+ 500
Spectrometer  = DELTA_NMR

Field strength = 11.7473579 [T] (500 [MH
X_acq duration = 1.0420224 [s]
X_domain       = 13C
X_freq        = 125.76529768 [MHz]
X_offset      = 100 [ppm]
X_points      = 32768
X_prescans    = 4
X_resolution   = 0.95967227 [Hz]
X_sweep       = 31.44654088 [kHz]
X_domain      = 1H
X_freq        = 500.15991521 [MHz]
X_offset      = 51 [ppm]
X_irr_freq    = FALSE
X_irr_offset   = FALSE
X_mod_return   = 1
X_scans       = 630
X_total_scans = 630
X_90_width    = 9.95 [us]
  
```

166.4631
164.7385

114.7730

77.3751
77.1233
76.8638
71.8658

34.1245
32.3313
28.8288

22.3962

15.3683
13.8880

JEOL

8b

X : parts per Million : 1H

Filename = guo789-5.jdf
 Author = NMR users
 Experiment = single_pulse.exp
 Sample_id = 1
 Solvent = CHLOROFORM-D
 Creation_time = 5-MAR-2006 23:10:51
 Revision_time = 5-MAR-2006 23:20:45
 Current_time = 5-MAR-2006 23:21:01
 Content = Single Pulse Experiment
 Data_format = 1D COMPLEX
 Dim_size = 16384
 Dim_title = 1H
 Dim_units = [ppm]
 Dimensions = X
 Site = Eclipse+ 500
 Spectrometer = DELTA_NMR

Field_strength = 11.7473579[T] (500[MH
 X_acq_duration = 2.182348[s]
 X_domain = 1H
 X_freq = 500.15991521[Mhz]
 X_offset = 5[ppm]
 X_points = 16384
 X_prescans = 0
 X_resolution = 0.4582189[Hz]
 X_sweep = 7.50750751[KHz]
 Clipped = FALSE
 Mod_return = 1
 Scans = 8
 Total_scans = 8
 X_90_width = 12.1[us]
 X_acq_time = 2.182348[s]
 X_angle = 45[deg]
 X_pulse = 6.05[us]

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88

```

Content
Data_format      = Single Pulse with Bro
Dim_size         = 1D COMPLEX
Dim_title        = 32768
Dim_units        = 13C
Dimensions       = [ppm]
Site             = X
Spectrometer     = Eclipse+ 500
                 = DELTA NMR

```

```
Field strength      = 11.74735791[T] (500[MH
X acc duration      = 1.0420224[s]
X domain            = 13C
X freq              = 125.76529768[MHz]
X offset            = 1001[ppm]
X points            = 32768
X prescans           = 4
X presol            = 0.9567227[Hz]
X resolution         = 31.44654088[kHz]
X sweep              = 1H
Irr domain           = 500.15991521[MHz]
Irr freq             = 5[ppm]
Irr offset           = FALSE
Clipped              = FALSE
Mod_return           = 1
Total_scans          = 463
X_90_width          = 9.95[us]
```

```
X_acq_time      = 1.0420224[s]
X_angle         = 30[deg]
X_pulse         = 3.31666667[us]
Initial_wait    = 1[s]
Phase_preset    = 2[us]
Recvr_gain      = 30
Relaxation_delay = 1[s]
Temp_get        = 22.9[dc]
```

VERA

8c

```

Current Data File: 8c0807
NAME: 8c0807
EXPNO: 1
PROCNO: 1

F2 - Acquisition Parameters
Date_ : 20060315
Time : 16.40
INSTRUM : spect
PROBHD : 5 mm QNP 1H/13
PULPROG : zgpg30
TD : 32768
SOLVENT : CDCl3
NS : 16
DS : 0
SWH : 4194.631 Hz
FIDRES : 0.128010 Hz
AQ : 3.905956 sec
RG : 645.1
DM : 119.200 usec
DE : 6.00 usec
TE : 295.1 K
D1 : 1.00000000 sec
MCREST : 0.00000000 sec
MCWPR : 0.01500000 sec

===== CHANNEL f1 =====
NUC1 : 13C
P1 : 9.75 usec
PL1 : 0.00 dB
SFO1 : 300.131300 MHz

F1 - Acquisition parameters
ND0 : 1
TD : 32
SFO1 : 300.1314 MHz
FIDRES : 31.250000 Hz
SW : 3.332 ppm
PULPROG : undefined

F3 - Acquisition parameters
ND0 : 1
SW : 3.332 ppm
SFO1 : 300.1314 MHz
H2PPT : 0.122070 Hz
TD : 32

F2 - Processing parameters
SI : 32768
SF : 300.130000 MHz
WDW : EM
SSB : 0
LB : 0.30 Hz
GB : 0
PC : 1.40

F1 - Processing parameters
SI : 32
MC2 : Q7
SF : 300.130000 MHz
WDW : no
SSB : 2
LB : 0.00 Hz
GB : 0

F3 - Processing parameters
SI : 32
SF : 300.130000 MHz
WDW : no
SSB : 2
LB : 0.00 Hz
GB : 0
  
```

8c

Current Data Parameters
NAME gms07
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060315
Time 16:47
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4005
DS 0
SWH 18115.941 Hz
FIDRES 0.276427 Hz
AQ 1.8088416 sec
RG 320
XG 17.500 usec
YB 2.000 usec
ZB 6.00 usec
TE 295.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
DECTA 0.00000000 sec
MCHRGST 0.01500000 sec
MCHRG 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
PL1 0.00 dB
SFO1 75.4732958 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 19.00 dB
PL13 17.00 dB
SFO2 300.1315007 MHz

F1 - Acquisition Parameters
ND0 1
TD 32
SFO1 300.1314 MHz
FIDRES 31.250000 Hz
SW 3.332 PPM
FMODER undefined

F3 - Acquisition Parameters
NU0 1
NU1 32
SFO1 300.1314 MHz
FIDRES 31.250000 Hz
SW 3.332 PPM
FMODER undefined

F2 - Processing Parameters
SI 65536
SF 75.46778 MHz
WDW 0
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

F1 - Processing Parameters
SI 32
SF 300.1300000 MHz
WDW 0
SSB 2
LB 0.00 Hz
GB 0

F3 - Processing Parameters
SI 32
SF 300.1300000 MHz
WDW 0
SSB 2
LB 0.00 Hz
GB 0

F2 - Acquisition Parameters
 Date_ 20060315
 Time_ 21.20
 INSTRUM spect
 PROBD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SMH 4194.631 Hz
 FIDRES 0.128010 Hz
 AQ 3.9059956 sec
 RG 456.1
 DW 119.200 usec
 DE 6.00 usec
 TE 295.3 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWPR 0.01500000 sec
 ===== CHANNEL f1 =====
 NUC1 1H
 P1 9.75 usec
 PL1 0.00 dB
 SFO1 300.131300 MHz
 F1 - Acquisition parameters
 ND0 1
 TD 32
 SFO1 300.1314 MHz
 FIDRES 31.250000 Hz
 SW 3.332 ppm
 FMODE undefined
 F3 - Acquisition parameters
 ND0 1
 SW 3.332 ppm
 SFO1 300.1314 MHz
 HZPPT 0.122070 Hz
 TD 32
 F2 - Processing parameters
 SI 32768
 SF 300.130000 MHz
 WDM EX
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00
 F1 - Processing parameters
 SI 32
 QF 0
 MC2 300.130000 MHz
 WDM no
 SSB 2
 LB 0.00 Hz
 GB 0
 F3 - Processing parameters
 SI 32
 SF 300.130000 MHz
 WDM no
 SSB 2
 LB 0.00 Hz
 GB 0

S-38

5.09

X : parts per Million : 1H

Filename = guo798pure-5.jdf
 Author = NMR users
 Experiment = single_pulse.exp
 Sample_id = 1
 Solvent = CHLOROFORM-D
 Creation_time = 8-MAR-2006 17:25:02
 Revision_time = 8-MAR-2006 17:25:40
 Current_time = 8-MAR-2006 17:30:57

Field_strength = 11.7473579(T) (500)MH
 X_acq_duration = 2.1823488(s)
 X_domain = 1H
 X_freq = 500.15991521(MHz)
 X_offset = 51(ppm)
 X_points = 16384
 X_prescans = 0
 X_resolution = 0.45822189(Hz)
 X_sweep = 7.50750751(KHz)
 X_clip = FALSE
 Mod_return = 1
 Scans = 8
 Total_scans = 8

Initial_wait = 1(s)
 Phase_preset = 2(us)
 Recvr_gain = 18
 Relaxation_delay = 21.4(dc)
 Temp_get

X_90_width = 12.1(us)
 X_acq_time = 2.1823488(s)
 X_angle = 45(deg)
 X_pulse = 6.05(us)

guo798pure-9.jdr

X : parts per Million : 13C

Filename	= guo798pure-9.jdr	Field strength	= 11.7473579 [T] (500 [MH
Author	= NMR users	X_acq_duration	= 1.0420224 [s]
Experiment	= single_pulse_dec	X_domain	= 13C
Sample_id	= 2	X_freq	= 125.76529768 [MHz]
Solvent	= CHLOROFORM-D	X_offset	= 100 [ppm]
Creation_time	= 8-MAR-2006 22:28:26	X_points	= 32768
Revision_time	= 8-MAR-2006 22:26:39	X_prescans	= 4
Current_time	= 8-MAR-2006 22:26:41	X_resolution	= 0.95967227 [Hz]
Content	= Single Pulse with Bro	X_sweep	= 31.44654088 [kHz]
Data_format	= 1D COMPLEX	irr_domain	= 1H
dim_size	= 32768	irr_freq	= 500.15991521 [MHz]
dim_title	= 13C	irr_offset	= 5 [ppm]
dim_units	= [ppm]	Clipped	= TRUE
Dimensions	= X	Mod_return	= 1
Site	= Eclipse+ 500	Scans	= 8886
Spectrometer	= DELTA_NMR	Total_scans	= 8886
		X_90_width	= 9.95 [us]

JEOL

X : parts per Million : 1H

Filename = guo799-7.jdf
 Author = NMR users
 Experiment = single_pulse.exp
 Sample_id = 1
 Solvent = CHLOROFORM-D
 Creation_time = 9-MAR-2006 17:31:31
 Revision_time = 9-MAR-2006 17:31:05
 Current_time = 9-MAR-2006 17:31:11
 Content = Single Pulse Experiment
 Data_format = ID COMPLEX
 Dim_size = 16384
 Dim_title = 1H
 Dim_units = [ppm]
 Dimensions = X
 Site = Eclipse+ 500
 Spectrometer = DELTA_NMR

Field_strength = 11.7473579 [T] (500 [MH]
 X_acq_duration = 2.1823488 [s]
 X_domain = 1H
 X_freq = 500.15991521 [MHz]
 X_offset = 51 [ppm]
 X_points = 16384
 X_prescans = 0
 X_resolution = 0.45822189 [Hz]
 X_sweep = 7.50750751 [KHz]
 X_return = FALSE
 Mod_return = 1
 Scans = 8
 Total_scans = 8
 X_90_width = 12.1 [us]
 X_acq_time = 2.1823488 [s]
 X_angle = 45 [deg]
 X_pulse = 6.05 [us]

Initial_wait = 1 [s]
 Phase_preset = 2 [us]
 Recvr_gain = 19
 Relaxation_delay = 2 [s]
 Temp_get = 21.8 [deg]

Filename = guo799-9.jdf
 Author = NMR users
 Experiment = single_pulse_dec
 Sample_id = 2
 Solvent = CHLOROFORM-D
 Creation_time = 9-MAR-2006 17:56:46
 Revision_time = 9-MAR-2006 17:54:45
 Current_time = 9-MAR-2006 17:54:51

Content = Single Pulse with Bro
 Data_format = ID COMPLEX
 Data_size = 32768
 Dim_title = 13C
 Dim_units = (ppm)
 Dimensions = X
 Site = Eclipse+ 500
 Spectrometer = DELTA_NMR

Field_strength = 11.7473579 [T] (500 [MH
 X_acq_duration = 1.0420224 [s]
 X_domain = 13C
 X_freq = 125.76529768 [MHz]
 X_offset = 100 [ppm]
 X_points = 32768
 X_prescans = 4
 X_resolution = 0.95967227 [Hz]
 X_sweep = 31.44654088 [kHz]
 X_domain = 1H
 Irr_freq = 500.15991521 [MHz]
 Irr_offset = 5 [ppm]
 Clipped = FALSE
 Mod_return = 1
 Scans = 713
 Total_scans = 713
 X_90_width = 9.95 [us]

JEOL

No title

Current Data Parameters
NAME guo81pure
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060411
Time 13.47
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 4194.631 Hz
FIDRES 0.128010 Hz
AQ 3.905956 sec
RG 456.1
DW 119.200 usec
DE 6.00 usec
TE 298.1 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRR 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 9.75 usec
PL1 0.00 dB
SFO1 300.1313300 MHz

F1 - Acquisition Parameters
NDO 1
TD 32
SFO1 300.1314 MHz
FIDRES 31.250000 Hz
SW 3.332 ppm
P1MODE undefined

F3 - Acquisition Parameters
NDO 1
SW 3.332 ppm
SFO1 300.1314 MHz
H2PFT 0.122070 Hz
TD 32

F2 - Processing Parameters
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

F1 - Processing Parameters
SI 32
MC2 QF
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.00 Hz
GB 0

F3 - Processing Parameters
SI 32
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.00 Hz
GB 0

9c

Current Data Parameters
NAME 9c
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050411
Time 12.48
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 367
DS 0
SWH 10115.841 Hz
FIDRES 0.276427 Hz
AQ 1.8088436 sec
RG 1149.4
DE 27.600 usec
TE 298.4 K
D1 2.00000000 sec
D11 0.03000000 sec
DELTA 1.89999998 sec
WALTZA 0.00000000 sec
WALTZB 0.01500000 sec
MORPH

===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
PL1 0.00 dB
SFO1 75.4752958 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 13C
PCPD2 80.00 usec
PL2 0.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 300.1315057 MHz

F1 - Acquisition Parameters
P1 10.00 usec
PL1 0.00 dB
SFO1 300.1314 MHz
FIDRES 31.250000 Hz
SM 3.332 ppm
PRMODES uncalibrated

F3 - Acquisition Parameters
P3 1.00 usec
PL3 0.00 dB
SFO3 300.1314 MHz
H2PPT 0.122070 Hz
TD 32

F2 - Processing Parameters
SI 32
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 1.40
PC 1.40

F1 - Processing Parameters
SI 32
SF 300.1300000 MHz
WDW OP
SSB 2
LB 0.00 Hz
GB 0

F3 - Processing Parameters
SI 32
SF 300.1300000 MHz
WDW NO
SSB 2
LB 0.00 Hz
GB 0

7.498
7.495
7.488
7.480
7.394
7.381
7.359
7.346
7.338
7.323
7.311
7.294
7.287
7.281
6.651
4.835
4.821
4.797
4.784
4.565
4.559
4.528
4.522
4.152
4.146
4.140

R

Current Data Parameters
NAME guo734
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060419
Time 21.02
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 4194.631 Hz
FIDRES 0.128010 Hz
AQ 3.9058956 sec
RG 812.7
WDW 119.200 usec
DE 6.00 usec
TE 298.4 K
D1 1.0000000 sec
MCREST 0.0000000 sec
MCMRK 0.0150000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 9.75 usec
PL1 0.00 dB
SFO1 300.1313300 MHz

F1 - Acquisition parameters
ND0 1
TD 32
SFO1 300.1314 MHz
FIDRES 31.250000 Hz
SW 3.332 ppm
FOMODE undefined

F3 - Acquisition parameters
ND0 1
SW 3.332 ppm
SFO1 300.1314 MHz
HZPPT 0.12070 Hz
TD 32

F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 32
MC2 QP
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.00 Hz
GB 0

F3 - Processing parameters
SI 32
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.00 Hz
GB 0

```

NAME          9d
EXPNO         1
PROCNO        1
Date_         20060419
Time          22.40
INSTRUM       spect
PROBHD        5 mm QNP 1H/13
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            1029
DS            0
SWH           18115.941 Hz
F2 - Acquisition Parameters
AQ            1.8088416 sec
RG            1149.4
DM            27.600 usec
DE            239.0 K
TE            21.1
D1            2.0000000 sec
d11           0.0100000 sec
DELTA         1.8999998 sec
MCHEBT        0.0000000 sec
MCWETK        0.0150000 sec

===== CHANNEL f1 =====
NUC1          13C
P1            10.00 usec
PL1           0.00 dB
SFO1          75.4752958 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          13C
PCPD2         80.00 usec
PL2           0.00 dB
PL12          17.00 dB
PL13          17.00 dB
SFO2          300.1315007 MHz

P2 - Processing parameters
SI            65536
SF            75.4677490 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

7.282
6.915
6.880
6.862
6.828

5.805
5.753

3.745
3.721
3.682
3.679
2.025
1.994
1.960
1.632
1.541
1.537
1.494
1.490
1.288
1.270
1.258
1.241
1.211
0.934
0.930
0.909
0.880
0.867
0.801

$J_1 = 10.5$
 $J_2 = 15.6$
 $J = 15.6$

```

SOLVENT      CDCl3
NS           32
DS           0
SMH          4194.631 Hz
FIDRES       0.128010 Hz
AQ           3.905956 sec
RG           181
DW           119.200 usec
DE           6.00 usec
TE           296.5 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRX        0.01500000 sec

===== CHANNEL f1 =====
NUC1         1H
P1           9.75 usec
PL1          0.00 dB
SFO1         300.131300 MHz

F1 - Acquisition parameters
ND0          1
TD           32
SF01         300.1314 MHz
FIDRES       31.250000 Hz
SFO1         3.332 ppm
SM           undefined
F1MODE       undefined

F3 - Acquisition parameters
ND0          1
SW           3.332 ppm
SF01         300.1314 MHz
HZPFT        0.12070 Hz
TD           32

F2 - Processing parameters
SI           32768
SF           300.1300000 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           1.40

F1 - Processing parameters
SI           32
MC2          OF
SF           300.1300000 MHz
WDW          no
SSB          2
LB           0.00 Hz
GB           0

F3 - Processing parameters
SI           32
WDW          no
SSB          2
LB           0.00 Hz
GB           0
  
```

167.018
154.484
121.687
77.431
77.008
76.585
51.346
49.249
42.843
33.877
31.119
30.162
27.716

CARS

```

===== CHANNEL F1 =====
PROC1 13C
P1 10.00 usec
P11 0.00 dB
SFO1 75.4752958 MHz

===== CHANNEL F2 =====
CPDPRG2 waltz16
PROC2 80.00 usec
P12 0.00 dB
P13 17.00 dB
P14 17.00 dB
SFO2 300.1315007 MHz

F1 - Acquisition parameters
NUC1 1
TD 32
SFO1 300.1314 MHz
FIDRES 31.250000 Hz
SW 3.332 ppm
F2MODES undefined

F2 - Acquisition parameters
NUC2 1
TD 32
SFO1 300.1314 MHz
FIDRES 31.250000 Hz
SW 3.332 ppm
F2MODES undefined

F3 - Processing parameters
SI 32
MC2 300.1300000 MHz
SF 300.1300000 MHz
WDW 2
SSB 0
LB 0.00 Hz
GB 0

F3 - Processing parameters
SI 32
MC2 300.1300000 MHz
SF 300.1300000 MHz
WDW 2
SSB 2
LB 0.00 Hz
GB 0

```


7.282

4.326
4.311
4.288
4.273
4.029
4.004
3.991
3.966
2.672
2.651
2.619
2.597
2.286
2.261
2.232
2.208
1.700
1.680
1.658
1.608
1.600
1.590
1.513
1.496
1.485
1.478
1.466
1.452
1.439
1.426
1.412
1.345
1.328
1.316

13a

Current Data Parameters
NAME guo875
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060505
Time 12.47

INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 4194.631 Hz
FIDRES 0.128010 Hz
AQ 3.9059956 sec
RG 322.5
DW 119.200 usec
DE 6.00 usec
TR 295.2 K
D1 1.0000000 sec
MCKEST 0.0000000 sec
MCKMRK 0.0150000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 9.75 usec
PL1 0.00 dB
SFO1 300.1313300 MHz

F1 - Acquisition parameters
ND0 1
TD 32
SFO1 300.1314 MHz
FIDRES 31.250000 Hz
SW 3.332 ppm
FMODE undefined

F3 - Acquisition parameters
ND0 1
SW 3.332 ppm
SFO1 300.1314 MHz
H2OPT 0.122070 Hz
TD 32

F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
GB 0
PC 1.00

F1 - Processing parameters
SI 32
MC2 QF
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.00 Hz
GB 0

F3 - Processing parameters
SI 32
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.00 Hz
GB 0

173.065

77.448
77.025
76.601
70.771

38.525
36.330
35.231
34.250
31.377
29.044
28.564
22.800
22.666
13.983
13.950

13a

Current Data Parameters
NAME gmo87spure
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060505
Time 15.29

INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 310
DS 0
SWH 18115.941 Hz
FIDRES 0.276427 Hz
AQ 1.8088436 sec
RG 1024
DM 277850 usec
TE 296.1 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCPRG 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
PL1 0.00 dB
SFO1 75.4752598 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 17.00 dB
SFO2 300.135067 MHz

F1 - Acquisition Parameters
ND0 1
TD 32
SFO1 300.1354 MHz
FIDRES 31.22000000 Hz
SF 31.312 ppm
PNUC2 undefined

F3 - Acquisition Parameters
ND0 1
SF 3.312 ppm
SFO1 300.1354 MHz
FIDRES 0.122076 Hz
TD 32

F2 - Processing parameters
SI 65536
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 32
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.00 Hz
GB 0

F3 - Processing parameters
SI 32
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.00 Hz
GB 0

7.282

4.296
4.281
4.259
4.244
3.934
3.903
3.896
3.865
2.737
2.717
2.681
2.660
2.245
2.216
2.188
2.159
1.804
1.782
1.756
1.751
1.733
1.725
1.718
1.710
1.703
1.687
1.626
1.606
1.582
1.559
1.553
1.496
1.371

13b

Current Data Parameters
NAME guo89top
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060501
Time 15.25

INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl₃
NS 16
DS 0
SWH 4194.631 Hz
FIDRES 0.128010 Hz
AQ 3.905956 sec
RG 362
DM 119.200 usec
DE 6.00 usec
TE 293.8 K
D1 1.00000000 sec
DCREST 0.00000000 sec
MCPRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 9.75 usec
PL1 0.00 dB
SFO1 300.131300 MHz

F1 - Acquisition parameters
ND0 1
TD 32
SF01 300.1314 MHz
FIDRES 31.250000 Hz
SW 3.332 ppm
FPMODE undefined

F3 - Acquisition parameters
ND0 1
SW 3.332 ppm
SF01 300.1314 MHz
HZPFT 0.122070 Hz
TD 32

F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 32
MC2 QF
SF 300.1300000 MHz
WDW HO
SSB 2
LB 0.00 Hz
GB 0

F3 - Processing parameters
SI 32
SF 300.1300000 MHz
WDW NO
SSB 2
LB 0.00 Hz
GB 0

13b

874pureProton

7.280

4.293
4.277
4.255
4.240
3.930
3.899
3.893
3.861
2.733
2.713
2.677
2.656
2.241
2.213
2.185
2.156
1.777
1.753
1.730
1.713
1.707
1.684
1.623
1.605
1.584
1.556
1.530
1.492
1.370
1.335
1.325
1.315
1.305

Current Data Parameters
NAME gno874
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060508
Time 12.53
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 4194.631 Hz
FIDRES 0.128010 Hz
AQ 3.905956 sec
RG 322.5
PW 119.280 usec
DE 6.00 usec
TE 295.3 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCMRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 9.75 usec
PL1 0.00 dB
SFO1 300.1313300 MHz

F1 - Acquisition Parameters
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

F3 - Acquisition Parameters
ND0 1
SW 3.332 Ppm
SFO1 300.1314 MHz
HZPFT 0.122070 Hz
TD 32

F2 - Processing Parameters
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

F1 - Processing Parameters
SI 32
SF 300.1300000 MHz
WDW NO
SSB 2
LB 0.00 Hz
GB 0

F3 - Processing Parameters
SI 32
SF 300.1300000 MHz
WDW NO
SSB 2
LB 0.00 Hz
GB 0

77.454
77.031
76.607
73.427

37.831
34.711
34.040
33.490
28.172
22.704
15.689
13.978

14a

S-54

14b

R

- 7.281
- 7.279
- 7.265
- 4.345
- 4.326
- 4.311
- 4.288
- 4.274
- 4.181
- 4.159
- 4.144
- 4.121
- 3.922
- 3.905
- 3.887
- 3.867
- 3.851
- 2.744
- 2.725
- 2.685
- 2.667
- 2.656
- 2.615
- 2.595
- 2.372
- 2.348
- 2.312
- 2.287
- 2.200
- 2.167
- 2.153
- 2.141
- 2.109
- 2.092
- 2.083
- 2.069
- 1.744
- 1.732
- 1.723
- 1.712
- 1.692
- 1.671
- 1.658
- 1.643

Current Data Parameters
NAME gno888
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060519
Time 15.49

INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 4194.631 Hz
FIDRES 0.128010 Hz
AQ 3.905956 sec
RG 724.1
DW 119.200 usec
DE 6.00 usec
TE 295.0 K
D1 1.00000000 sec
MCHRG 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 9.75 usec
PL1 0.00 dB
SFO1 300.131300 MHz
F1 - Acquisition parameters
ND0 1
TD 32
SFO1 300.1314 MHz
FIDRES 31.250000 Hz
SW 3.332 ppm
FMODE undefined

F3 - Acquisition parameters
ND0 1
SW 3.332 ppm
SFO1 300.1314 MHz
HZDPT 0.122070 Hz
TD 32

F2 - Processing parameters
SI 32768
SF 300.130000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 32
MC2 32
SF 300.130000 MHz
WDW NO
SSB 2
LB 0.00 Hz
GB 0

F3 - Processing parameters
SI 32
SF 300.130000 MHz
WDW NO
SSB 2
LB 0.00 Hz
GB 0

trans:cis = 92:8

14b

Current Data Parameters
NAME guo888
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060521
Time 14.31

INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
FIDRES 0.276427 Hz
AQ 1.8088416 sec
RG 172.8
RW 27.724 usec
DM 6.00 usec
DE 294.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCOREST 0.00000000 sec
MCRMK 0.01500000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 10.00 usec
PL1 0.00 dB
SFO1 75.4752958 MHz
***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PIL2 17.00 dB
SFO2 300.135007 MHz

F1 - Acquisition parameters
ND0 1
TD 32
SFO1 300.1314 MHz
FIDRES 31.430000 Hz
SW 3.312 ppm
PNUC1R undefined

F3 - Acquisition parameters
ND0 1
SW 3.312 ppm
SFO1 300.1314 MHz
FIDRES 31.430000 Hz
PCPD2 80.00 usec
PL2 0.00 dB
PIL2 17.00 dB
SFO2 300.135007 MHz

F2 - Processing parameters
SI 65536
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 65536
SF 300.130000 MHz
WDW no
SSB 2
LB 0.00 Hz
GB 0
PC 1.40

77.454
77.233
77.031
76.607
74.212
73.399

37.604
36.439
35.006
33.236
31.213
29.940
19.574
15.868
14.517
11.558