LDA-Catalyzed Cycloisomerization of 2-(2-Propynyloxy)ethyl Iodides Leading to 3-(Iodomethylene)tetrahydrofurans

Toshiro Harada,* Keiko Muramatsu, Takayuki Fujiwara, Hiroshi Kataoka, and Akira Oku
 Department of Chemistry and Materials Technology, Kyoto Institute of Technology,
 Matsugasaki, Sakyo-ku, Kyoto 606-8585, Japan

General. THF was dried and distilled over sodium benzophenone ketyl. LDA (1 M solution in THF) was prepared prior to use by treatment of diisopropylamine with butyllithium (1.6 M in hexane, 1.0 equiv) in THF. GC analyses were performed with a capillary column (OV-1, 30 m).

3-Iodomethylene-2-phenylethyltetrahydrofuran (**5a**).

To a solution of iodoalkyne **1a** (157 mg, 0.5 mmol) in THF (1 mL) under argon atmosphere at 0 °C was added LDA (1 M solution in THF, 0.10 mL, 0.1 mmol). The resulting solution was stirred at room temperature for 5 h. The mixture was poured into water and extracted twice with ether. The combined organic layers were dried over MgSO₄ and concentrated in *vacuo*. The residue was purified by flash chromatography (SiO₂, 2-3% ether in hexane) to afford 116 mg (74% yield) of **5a** (*E*:*Z* = 9.0:1) as an oil. (*E*)-**5a**: ¹H NMR (500 MHz, CDCl₃) δ 1.85-2.00 (2H, m), 2.56-2.66 (2H, m), 2.73 (1H, ddd, *J* = 6.9, 10.0, and 13.8 Hz), 2.82 (1H, ddd, *J* = 5.2, 10.2, and 13.8 Hz), 3.86 (1H, dt, *J* = 7.2 and 8.1 Hz), 4.11 (1H, ddd, *J* = 5.1, 7.1, and 8.7 Hz), 4.29 (1H, m), 6.00 (1H, q, *J* = 2.7 Hz), 7.20-7.36 (5H, m); NOESY experiment showed a cross peak between H_a (δ 6.00) and H_b (δ 1.9). ¹³C NMR (125.8 MHz) δ 31.5, 36.1, 38.3, 65.6, 68.4, 80.6, 125.9, 128.35, 128.38, 141.6, 155.5. (*Z*)-**5a**: ¹H NMR (500 MHz, CDCl₃) δ 1.91 (1H, m), 2.15 (1H, m), 2.65 (2H, m), 2.70-2.82 (2H, m), 3.87 (1H, q, *J* = 7.4 Hz), 4.12 (1H, m), 4.35 (1H, br d, *J* = 8.7 Hz), 6.06 (1H, q, *J* = 1.7 Hz), 7.17-7.30 (5H, m); NOESY experiment showed a cross peak between H_a (δ 6.06) and H_b (δ 2.65). ¹³C NMR (125.8 MHz) δ 31.5, 33.8, 36.1, 67.3, 67.5, 82.7, 125.8, 128.3, 128.5, 141.8, 153.3. (*E*)- and (*Z*)-**5a**: IR (liquid film) 3050, 1640, 1250, 740, 700 cm⁻¹; Anal. Calcd for C₁₃H₁₅IO: C, 49.70; H, 4.81. Found: C, 49.78; H, 4.82.

2-Cyclohexyl-3-iodomethylenetetrahydrofuran (5c**):** ¹H NMR (500 MHz, CDCl₃) δ 1.05-1.30 (5H, m), 1.52 (1H, m), 1.63-1.83 (4H, m), 2.51 (1H, m), 2.62 (1H, m), 3.84 (1H, dt, *J* = 7.2 and 8.4 Hz), 4.07 (1H, dt, *J* = 4.2 and 8.4 Hz), 4.14 (1H, m), 5.97 (1H, m), (minor *Z* isomer resonated at 6.11 (1H, m)); ¹³C NMR (125.8 MHz, CDCl₃) δ 26.2, 26.3, 26.4, 26.5, 29.7, 38.7, 42.2, 66.0, 68.3, 85.8, 154.4; IR (liquid film) 3050, 1640, 1250 cm⁻¹; MS (EI) *m/z* (relative intensity) 292 (*M*⁺, 2), 209 (100), 165 (53); HRMS calcd for C₁₁H₁₇IO 292.0324, found: 292.0309.; Anal. Calcd for C₁₁H₁₇IO: C, 45.22; H, 5.86. Found: C, 44.98; H, 5.76. The *E* stereochemistry of the major isomer was assumed by analogy.

4-Iodomethylene-1-oxa-2-phenylcyclopentane

(**5d**): (*Z*)-**5d**: ¹H NMR (500 MHz, CDCl₃) δ 2.58 (1H, br dd, *J* = 9.2 and 15.7 Hz), 2.99 (1H, br dd, *J* = 5.9 and 15.7 Hz), 3.95 (1H, dd, *J* = 2.4 and 15.9 Hz), 4.21 (1H, dd, *J* = 2.4 and 15.9 Hz), 5.08 (1H, dd, *J* = 5.9

and 9.2 Hz), 6.03 (1H, br s), 7.26-7.39 (5H, m); NOESY experiment showed a cross peaks between H_a (δ 6.03) and H_b (δ 2.58 and 2.99). ¹³C NMR (125.8 MHz, CDCl₃) δ 43.2, 66.9, 75.6, 82.4, 125.8, 127.9, 128.5, 140.8, 151.2. (*E*)-**5d**: ¹H NMR (500 MHz, CDCl₃) δ 2.53 (1H, br dd, *J* = 8.8 and 16.9 Hz), 3.01 (1H, br dd, *J* = 6.4 and 16.9 Hz), 4.40 (1H, br d, *J* = 13.0 Hz), 4.59 (1H, d, *J* = 13.0 Hz), 5.05 (1H, dd, *J* = 6.4 and 8.8 Hz), 6.08 (1H, br s), 7.26-7.39 (5H, m); NOESY experiment showed a cross peaks between H_a (δ 6.08) and H_b (δ 4.59 and 5.05). ¹³C NMR (125.8 MHz, CDCl₃) δ 45.6, 67.4, 71.6, 80.3, 125.9, 127.9, 128.5, 140.9, 152.2. (*Z*)- and (*E*)-**5d**: IR (liquid film) 3050, 1645, 1260 cm⁻¹; Anal. Calcd for C₁₁H₁₀IO: C, 46.18; H, 3.88. Found: C, 46.46; H, 3.95.

4-Iodomethylene-2-(prop-2-ynyloxy)tetrahydrofuran (5e): a 1.3:1 mixture of *Z* and *E* isomers; ¹H NMR (500 MHz, CDCl₃) δ 2.46 (1H, br s), 2.61-2.78 (2H, m), 4.21-4.47 (4H, m), 5.49 (1H for the major isomer, d, *J* = 4.5 Hz), 5.64 (1H for the minor isomer, d, *J* = 4.5 Hz), 6.05 (1H or the minor isomer, br s), 6.11

(1H for the major isomer, br s); ¹³C NMR (125.8 MHz, CDCl₃) δ 40.9 (minor isomer), 44.0 (major isomer), 53.8 (minor isomer), 54.0 (major isomer), 67.4 (minor isomer), 68.2 (major isomer), 69.5 (major isomer), 74.0 (minor isomer), 74.3 (major isomer), 74.3 (minor isomer), 79.2 (minor isomer), 79.3 (major isomer), 101.4 (major isomer), 103.0 (minor isomer), 148.4 (minor isomer), 149.5 (major isomer). Determination of the stereochemistry was based on upfield shifts of the allylic carbons syn to the iodine atom.^a IR (liquid film) 3275, 3060, 2120, 1640, 1245, 1085 cm⁻¹; MS, *m/z* (relative intensity) 264 (M⁺, 21), 225 (10), 209 (37), 178 (100); HRMS calcd for C₈H₉ IO₂ 263.9647, found; 263.9657. Anal. Calcd for C₈H₉ IO₂: C, 36.39; H, 3.44. Found: C, 36.29; H, 3.37.

(E)-5-Ethoxy-3-iodomethylene-2-phenethyl-tetrahydrofuran (5f). (*E*)-**5f**: *R*_f = 0.53 in 10% ethyl acetate in hexane; ¹H NMR (500 MHz, C₆D₆) δ 1.16 (3H, t, *J* = 7.0 Hz), 1.73 (2H, m), 2.49 (1H, quiet d, *J* = 2.8 and 17.3 Hz), 2.67-2.73 (2H, m), 2.81 (1H, ddd, *J* = 2.0, 10.0, and 13.5 Hz), 3.34 (1H, qd, *J* = 7.0 and 9.6 Hz), 3.79 (1H, qd, *J* = 7.0 and 9.6 Hz), 4.43 (1H, br d, *J* = ca. 8 Hz), 5.05 (1H, d, *J* = 5.3 Hz), 5.62 (1H, q, *J* = 2.7 Hz),

7.17 (2H, m), 7.25 (3H, m); NOESY experiment showed a cross peaks between H_a (δ 5.62) and H_b (δ 1.73). ¹³C NMR (125.8 MHz, C₆D₆) δ 15.1, 31.5, 36.4, 45.1, 62.4, 68.9, 78.1, 101.0, 125.7, 128.4, 128.6, 141.8, 154.0. (*E*)-**5f***: *R*_f = 0.48 in 10% ethyl acetate in hexane; ¹H NMR (500 MHz, C₆D₆) δ 1.29 (3H, t, *J* = 7.0 Hz), 1.70 (1H, m), 2.04 (1H, m), 2.49 (1H, br d, *J* = ca. 17 Hz), 2.72-2.77 (2H, m), 2.87 (1H, ddd, *J* = 4.8, 9.7, and 14.1 Hz), 3.31 (1H, qd, *J* = 7.0 and 9.6 Hz), 3.81 (1H, qd, *J* = 7.0 and 9.6 Hz), 4.30 (1H, br d, *J* = ca. 10 Hz), 5.02 (1H, d, *J* = 5.6 Hz), 5.57 (1H, q, *J* = 2.0 Hz), 7.18 (2H, m), 7.25 (3H, m); NOESY experiment showed a cross peaks between H_a (δ 5.57) and H_b (δ 1.70). ¹³C NMR (125.8 MHz, C₆D₆) δ 15.01, 32.4, 39.4, 44.8, 62.7, 69.1, 80.7, 102.2, 125.7, 128.4, 128.6, 141.8, 154.2. (*Z*)-**5f**: *R*_f = 0.40 in 10% ethyl acetate in hexane; ¹H NMR (500 MHz, C₆D₆) δ 1.16 (3H, t, *J* = 7.0 Hz), 2.18 (1H, m), 2.38 (1H, dddd, *J* = 0.5, 2.0, 5.7, and 17.0 Hz), 2.43-2.52 (2H, m), 2.92 (1H, ddd, *J* = 7.3, 9.7, and 13.7 Hz), 3.07 (1H, m), 3.35 (1H, qd, *J* = 7.0 and 9.6 Hz), 3.90 (1H, qd, *J* = 7.0 and 9.6 Hz),

^a Gaudemer, A.; Gofier, M.; Mandelbaum, A.; Parthasarathy, R. *Determination of Configuration by Spectrometric Methods*; Kagan, H. B. Morrison, J. D., Ed.; *Stereochemistry Fundamentals and Methods*, vol. 1; Georg Thieme Publishers: Stuttgart, 1977; pp 55.

4.63 (1H, br d, $J = 10.6$ Hz), 5.08 (1H, dd, $J = 2.4$ and 5.7 Hz), 5.50 (1H, q, $J = 2.1$ Hz), 7.17 (1H, m), 7.26 (2H, m), 7.34 (2H, m); NOESY experiment showed a cross peaks between H_a (δ 5.50) and H_b (δ 2.38 and 2.43-2.52). ^{13}C NMR (125.8 MHz, C_6D_6) δ 15.0, 32.5, 35.9, 41.1, 63.0, 67.2, 83.7, 104.4, 125.9, 128.4, 128.7, 141.9, 152.2. (*E*)-**5f**, **5f'** and (*Z*)-**5f**: IR (liquid film) 3060, 1640, 1600, 1185, 970, 740, 700 cm^{-1} ; MS, m/z (relative intensity) 357 ($\text{M}^+ - 1$, 1), 329 (28), 231 (100); HRMS calcd for $\text{C}_{15}\text{H}_{18}\text{IO}_2$ 357.0352, found; 357.0358.

5-Ethoxy-3-iodomethylene-2-(oct-1-enyl)tetrahydrofuran (5g).

(*E*)-**5g** and -**5g'**: $R_f = 0.43$ and 0.50 in 10% ether in hexane; ^1H NMR (500 MHz, C_6D_6) δ 0.97 (3H, t, $J = 7.2$ Hz), 1.15 (3H, t, $J = 7.0$ Hz), 1.20-1.38 (8H, m), 1.96 (2H, m), 2.54 (1H, m), 2.81 (1H, br d, $J = 17.0$ Hz), 3.35 (1H, qd, $J = 7.0$ and 9.5 Hz), 3.87 (1H, qd, $J = 7.0$ and 9.5 Hz), 4.80 (1H, br d, $J = 8.4$ Hz), 5.05 (1H, d, $J = 5.2$ Hz), 5.51 (1H, td, $J = 6.7$ and 15.3 Hz), 5.67 (1H, dd, $J = 5.2$ and 15.2 Hz), 5.94 (1H, m) for (*E*)-**5g** and δ 0.96 (3H, t, $J = 7.2$ Hz), 1.16 (3H, t, $J = 7.1$ Hz), 1.20-1.42 (8H, m), 1.96 (2H, m), 2.40 (1H, tdd, $J = 2.8, 5.4$, and 17.5 Hz), 2.76 (1H, br d, $J = 17.5$ Hz), 3.34 (1H, qd, $J = 7.1$ and 9.6 Hz), 3.81 (1H, qd, $J = 7.1$ and 9.6 Hz), 4.95 (1H, br d, $J = 7.8$ Hz), 5.09 (1H, d, $J = 5.3$ Hz), 5.44 (1H, dd, $J = 7.8$ and 15.3 Hz), 5.79 (1H, td, $J = 6.7$ and 15.3 Hz), 5.96 (1H, m) for (*E*)-**5g'**; ^{13}C NMR (125.8 MHz, C_6D_6) δ 14.3, 15.3, 22.9, 29.2, 29.3, 31.9, 32.3, 44.6, 62.7, 71.0, 83.4, 102.2, 128.5, 134.3, 153.9 for (*E*)-**5g** and δ 14.3, 15.3, 22.9, 29.1, 29.2, 31.9, 32.4, 44.6, 62.8, 70.8, 81.1, 101.4, 128.5, 135.6, 154.3 for (*E*)-**5g'**. Isomerization was observed between (*E*)-**5g** and (*E*)-**5g'** in the attempted separation of these diastereomers by silica gel column chromatography. (*Z*)-**5g**: $R_f = 0.31$ in 10% ether in hexane; ^1H NMR (500 MHz, C_6D_6) δ 0.97 (3H, t, $J = 7.2$ Hz), 1.19 (3H, t, $J = 7.0$ Hz), 1.24-1.38 (6H, m), 1.46 (2H, m), 2.12 (2H, m), 2.40 (1H, ddd, $J = 2.0, 5.4$, and 16.8 Hz), 2.54 (1H, br d, $J = 16.8$ Hz), 3.35 (1H, qd, $J = 7.0$ and 9.5 Hz), 3.93 (1H, qd, $J = 7.0$ and 9.5 Hz), 5.08 (1H, d, $J = 8.2$ Hz), 5.11 (1H, dd, $J = 2.1$ and 5.4 Hz), 5.69 (1H, br s), 5.79 (1H, dd, $J = 8.3$ and 15.4 Hz), 5.99 (1H, td, $J = 6.8$ and 15.4 Hz); NOESY experiment showed a cross peaks between H_a (δ 5.99) and H_b (δ 2.40 and 2.54). ^{13}C NMR (125.8 MHz, C_6D_6) δ 14.3, 15.2, 22.9, 29.2, 29.3, 32.0, 32.6, 41.6, 62.8, 69.2, 85.6, 104.2, 128.8, 136.3, 151.0. (*Z*)-**5g'**: $R_f = 0.36$ in 10% ether in hexane; ^1H NMR (500 MHz, C_6D_6) δ 0.96 (3H, t, $J = 8.7$ Hz), 1.12 (3H, t, $J = 7.0$ Hz), 1.24-1.38 (6H, m), 1.44 (2H, m), 2.12 (2H, m), 2.40 (2H, m), 3.34 (1H, qd, $J = 7.0$ and 9.5 Hz), 3.81 (1H, qd, $J = 7.0$ and 9.5 Hz), 5.15 (2H, m), 5.63 (1H, dd, $J = 7.1$ and 15.3 Hz), 5.79 (1H, br s), 6.05 (1H, td, $J = 6.8$ and 6.9 Hz); NOESY experiment showed a cross peaks between H_a (δ 5.79) and H_b (δ 2.40). ^{13}C NMR (125.8 MHz, C_6D_6) δ 14.3, 15.2, 23.0, 29.2, 29.4, 32.0, 32.7, 42.4, 62.7, 69.6, 83.3, 102.3, 126.7, 135.3, 150.6. (*E*)-**5g**, **5g'** and (*Z*)-**5g**: IR (liquid film) 3060, 1640, 1195, 970 cm^{-1} ; MS, m/z (relative intensity) 364 (M^+ , 2), 319 (7), 243 (95), 205 (100); HRMS calcd for $\text{C}_{15}\text{H}_{25}\text{IO}_2$ 364.0899, found; 364.0895.

(*Z*)-**5g** and (*Z*)-**5g'**

(E)-5-Ethoxy-3-iodomethylene-2,2-dimethyltetrahydrofuran ((E)-5h).

^1H NMR (500 MHz, C_6D_6) δ 1.13 (3H, t, $J = 7.0$ Hz), 1.20 (3H, s), 1.38 (3H, s), 2.63 (1H, ddd, $J = 3.1, 5.6$, and 17.5 Hz), 2.81 (1H, ddd, $J = 1.0, 2.1$, and 17.5 Hz), 3.29 (1H, qd, $J = 7.5$ and 9.5 Hz), 3.78 (1H, qd, $J = 7.5$ and 9.5 Hz), 5.00 (1H, d, $J = 5.5$ Hz), 5.71 (1H, t, $J = 2.7$ Hz); NOESY experiment showed a cross peaks between H_a (δ 5.71) and CH_3 (δ 1.20 and 1.38). ^{13}C NMR (125.8 MHz, C_6D_6) δ 14.9, 28.9, 29.8, 45.0, 62.2, 68.8, 83.4, 100.6, 159.0; IR (liquid film) 3060, 1640, 1280, 1195 cm^{-1} ; MS, m/z (relative intensity) 282 (M^+ , 2), 267 (100), 208 (38); HRMS calcd for $\text{C}_9\text{H}_{15}\text{O}_2\text{I}$ 282.0117, found; 358.0116.

(*E*)-**5h**

(E)-2-Ethoxy-4-iodomethylene-1-oxaspiro[4.5]decane ((E)-5i). ^1H NMR (300 MHz, C_6D_6) δ 1.08 (3H, t, $J = 7.1$ Hz), 1.15 (1H, m), 1.44-1.86 (8H, m), 2.07 (1H, br d, $J = ca. 13$ Hz),

2.62 (1H, ddd, $J = 3.2, 5.6$ and 17.4 Hz), 2.86 (1H, ddd, $J = 1.0, 2.4$, and 17.4 Hz), 3.32 (1H, qd, $J = 7.1$ and 9.4 Hz), 3.82 (1H, qd, $J = 7.1$ and 9.4 Hz), 5.03 (1H, d, $J = 5.5$ Hz), 5.76 (1H, dd, $J = 2.2$ and 2.8 Hz); ^{13}C NMR (125.8 MHz, CDCl_3) δ 15.1, 22.4, 22.6, 25.3, 38.4, 38.6, 44.9, 62.4, 69.1, 85.4, 100.5, 158.7; IR (liquid film) 3050, 1635, 1195, 980 cm^{-1} ; MS, m/z (relative intensity) 322 (M^+ , 14), 279 (15), 195 (100); HRMS calcd for $\text{C}_{12}\text{H}_{19}\text{IO}_2$ 322.0430, found: 322.0413. Anal. Calcd for $\text{C}_{12}\text{H}_{19}\text{IO}_2$: C, 44.74; H, 5.94. Found: C, 44.63; H, 5.68. The *E* stereochemistry was assumed by analogy.

(Z)-3-Iodomethylenehexahydrofuro[2,3-b]pyran ((Z)-5j):^b ^1H NMR (500 MHz, C_6D_6) δ 0.78 (1H, m), 1.25 (2H, m), 1.40 (1H, m), 2.16 (1H, br s), 3.05 (1H, m), 3.66 (1H, br d, $J = \text{ca. } 11.5$ Hz), 4.41 (1H, br d, $J = \text{ca. } 14$ Hz), 4.67 (1H, td, $J = 2.4$ and 14.3 Hz), 5.14 (1H, d, $J = 3.6$ Hz), 5.53 (1H, q, $J = 2.6$ Hz); NOESY experiment showed a cross peaks between H_a (δ 5.53) and H_b (δ 2.16) and between H_b (δ 2.16) and H_c (δ 5.14). ^{13}C NMR (125.8 MHz, C_6D_6) δ 20.4, 22.4, 45.0, 63.6, 66.8, 75.1, 102.2, 151.4.

1-Deuterio-3-(2-iodoethoxy)-5-phenyl-1-pentyne (1a-d).

To a solution of tosylate **i**^c (1.43 g, 4.0 mmol) in THF (8 mL) at -78°C was added butyllithium (1.6 M in hexane, 2.6 mL, 4.2 mmol). The mixture was stirred for 2 h at -78°C and quenched by the addition of D_2O (1 mL) in THF (4 mL). The mixture was extracted twice with ether. The organic layers were dried over MgSO_4 and concentrated in *vacuo*. The residue was purified by flash chromatography (SiO_2 , 30% ethyl acetate in hexane) to give 1.33 g (93% yield) of the 1-deuterio tosylate: ^1H NMR (300 MHz, CDCl_3) δ 1.90-2.05 (2H, m), 2.41 (3H, s), 2.45 (0.02H, s), 2.71 (2H, t, $J = 7.4$ Hz), 3.55-3.61 (1H, m), 3.87-3.99 (2H, m), 4.18-4.22 (2H, m), 7.15-7.36 (7H, m), 7.81 (2H, d, $J = 8.3$ Hz). To a solution of dry sodium iodide (5.6 mmol) in acetone (6 mL) was added a solution of the 1-deuterio tosylate (1.33g, 3.7 mmol) in acetone (4 mL) at room temperature. The mixture was stirred for 12 h at reflux. The reaction mixture was poured into water and extracted twice with ether. The organic layers were dried over MgSO_4 and concentrated in *vacuo*. The residue was purified by flash chromatography (SiO_2 , 3-5% ethyl acetate in hexane) to give **1a-d** (97% yield, 98%-d): ^1H NMR (300 MHz, CDCl_3) δ 1.98-2.16 (2H, m), 2.48 (0.02H, d, $J = 3.49$ Hz), 2.82 (2H, t, $J = 7.5$ Hz), 3.26-3.30 (2H, m), 3.68 (1H, td, $J = 6.9$ and 10.4 Hz), 3.95-4.08 (2H, m), 7.20-7.29 (5H, m).

Deuterium Scrambling Experiment. To a solution of **1a-d** (185 mg, 0.61 mmol) and **1c** (177 mg, 0.62 mmol) in THF (2.4 mL) at 0°C was added LDA (1 M solution in THF, 0.24 mL, 0.24 mmol). The resulting solution was stirred at room temperature for 5 h. The mixture was poured into water and extracted twice with ether. The combined organic layers were dried over MgSO_4 and concentrated in *vacuo*. The residue was purified by flash

^b Yanada, R.; Koh, Y.; Nishimori, N.; Matsumura, A.; Obika, S.; Mitsuya, H.; Fujii, N.; Takemoto, Y. *J. Org. Chem.* **2004**, 69, 2417.

^c (a) Harada, T.; Fujiwara, T.; Iwazaki, K.; Oku, A. *Org. Lett.* **2000**, 2, 1855. (b) Harada, T.; Iwazaki, K.; Otani, T.; Oku, A. *J. Org. Chem.* **1998**, 63, 9007.

chromatography (SiO₂, 2-3% ether in hexane) to give 203 mg of a 1.1:1 mixture of (*E*)-**5c-d** (51%-*d*, 58% yield) and (*E*)-**5a-d** (48%-*d*, 52% yield) and 26.4 mg of (*E*)-**5a-d** (13% yield).

Current Data Parameters
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 PROCNO 1

F2 - Acquisition Parameters

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 TE 299.4 K
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 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

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F2 - Processing parameters

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 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

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 F1 4001.04 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 0.40000 ppm/cm
 HZCM 200.05200 Hz/cm

Current Data Parameters
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 PROCNO 1

F2 - Acquisition Parameters

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 TD 65536
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 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 406.4
 DW 48.400 usec
 DE 6.00 usec
 TE 299.2 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

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

NUC1 1H
 P1 10.30 usec
 PL1 -6.00 dB
 SF01 500.1330885 MHz

F2 - Processing parameters

SI 32768
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 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

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 CY 12.50 cm
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 F2P 0.000 ppm
 F2 0.00 Hz
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 HZCM 200.05200 Hz/cm

Current Data Parameters
 NAME 19D-114-PD
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20041021
 Time 21.44
 INSTRUM drx500
 PROBHD 5 mm Multinucl
 PULPROG zg30
 TD 65536
 SOLVENT C6D6
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 456.1
 DW 48.400 usec
 DE 6.00 usec
 TE 296.9 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

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

NUC1 1H
 P1 10.30 usec
 PL1 -6.00 dB
 SF01 500.1330885 MHz

F2 - Processing parameters

SI 32768
 SF 500.1300129 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

CX 20.00 cm
 CY 0.00 cm
 F1P 8.000 ppm
 F1 4001.04 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 0.40000 ppm/cm
 HZCM 200.05200 Hz/cm

19D-100-PA

Current Data Parameters
 NAME 19D-100-PA
 EXPNO 1
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 500000
 Time 14.41
 INSTRUM dpx300
 PROBHD 5 mm QNP 1H/13C/31P/19F
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 6172.839 Hz
 FIDRES 0.166360 Hz
 AQ 2.6542680 sec
 RG 256
 DM 81.000 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 10.80 usec
 DE 6.50 usec
 SFO1 300.1318634 MHz
 NUC1 1H
 PL1 -5.00 dB
 F2 - Processing parameters
 SI 16384
 SF 300.1300000 MHz
 MDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00
 1D NMR plot parameters
 CX 20.00 cm
 F1P 10.734 ppm
 F1 3221.63 Hz
 F2P -0.266 ppm
 F2 -79.80 Hz
 PPMCM 0.55000 ppm/cm
 HZCM 165.07150 Hz/cm

19D-101-PI

Current Data Parameters

NAME 19D-101-PI
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20040921
Time 16.33
INSTRUM drx500
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT C6D6
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 456.1
DM 48.400 usec
DE 6.00 usec
TE 299.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

NUC1 1H
P1 10.30 usec
PL1 -6.00 dB
SF01 500.1330885 MHz

F2 - Processing parameters

SI 32768
SF 500.1300129 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
CY 0.00 cm
F1P 8.000 ppm
F1 4001.04 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.40000 ppm/cm
HZCM 200.05200 Hz/cm

S-10

Current Data Parameters
 NAME 19D-101-PK
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20040921
 Time 15.49
 INSTRUM drx500
 PROBHD 5 mm Multinucl
 PULPROG zg30
 TD 65536
 SOLVENT C6D6
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 143.7
 DW 48.400 usec
 DE 6.00 usec
 TE 299.3 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWPK 0.01500000 sec

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

NUC1 1H
 P1 10.30 usec
 PL1 -6.00 dB
 SF01 500.1330885 MHz

F2 - Processing parameters

SI 32768
 SF 500.1300129 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

CX 20.00 cm
 CY 0.00 cm
 F1P 8.000 ppm
 F1 4001.04 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 0.40000 ppm/cm
 HZCM 200.05200 Hz/cm

Current Data Parameters
 NAME 19D-118-PA
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20040729
 Time 21.37
 INSTRUM drx500
 PROBHD 5 mm Multinucl
 PULPROG zg30
 TD 65536
 SOLVENT C6D6
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 181
 DW 48.400 usec
 DE 6.00 usec
 TE 299.6 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRK 0.01500000 sec

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

NUC1 1H
 P1 10.30 usec
 PL1 -6.00 dB
 SF01 500.1330885 MHz

F2 - Processing parameters

SI 32768
 SF 500.1300129 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

CX 20.00 cm
 CY 12.50 cm
 F1P 8.000 ppm
 F1 4001.04 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 0.40000 ppm/cm
 HZCM 200.05200 Hz/cm

Current Data Parameters
 NAME 1A-178-PA
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 500000
 Time 17.30
 INSTRUM DRX500
 PROBHD 5 mm Multinu
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.315264 Hz
 AQ 1.5860212 sec
 RG 128
 DM 48,400 us
 DE 6.00 us
 TE 300.0 K
 1.00000000 sec
 P1 9.30 us
 P2 6.00 us
 DE 6.00 us
 SFO1 500.1330885 MHz
 NUC1 1H
 PL1 -6.00 dB

F2 - Processing parameters
 SI 16384
 SF 500.1300000 MHz
 MDW EN
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 25.00 cm
 F1P 8.000 pp
 F1 4001.04 Hz
 F2P 0.000 pp
 F2 0.00 Hz
 PPM0H 0.32000 pp
 HZCM 150.04160 Hz

1.1898
1.2042
1.2182
1.4047
1.4119
1.4307
1.5963
1.6082
1.6154
1.6218
1.6280
1.6353
1.6419
1.6470
1.6539
1.6621
1.6692
1.6873
1.9352
2.6816
2.6839
2.7150
2.7168
2.7750
2.7813
2.7857
2.7920
3.4685
3.4826
3.4875
3.5015
3.7956
3.8101
3.8149
3.8292

5.2052
5.2160
5.9698
5.9748
5.9800

7.2866