Supporting Information for

Angew. Chem. **1999**.

Samarium-Mediated β-Elimination in Dihaloalcohols: Diastereoselective Synthesis of (*Z*)-Vinyl Halides

José M. Concellón,* Pablo. L. Bernad and Juan A. Pérez-Andrés

1,1-diiodonon-2-yl acetate (1a)

^{1}H NMR ($CDCl_3$, 300 MHz) δ 5.30 (1 H, d, *J* = 3.53), 4.46 (1 H, m), 2.16 (3 H, s), 1.52-1.12 (12 H, m), 0.89 (3 H, m); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 69.3 (C), 76.5 (CH), 33.4 (CH_2), 31.3 (CH_2), 28.8 (CH_2), 28.7 (CH_2), 24.7 (CH_2), 22.2 (CH_2), 20.7 (CH_3), 13.9 (CH_3), -21.4 (CH_3); MS 378.3 (24), 311.4 (33), 269.4 (20), 167.2 (21), 123.3 (65), 81.2 (68), 43.0 (100); IR (neat) 2051, 2928, 2856, 1745, 1464, 1371, 1222, 1078, 1020, 974; R_f = 0.5 (hexane/AcOEt 10/1).

1-cyclohexyl-2,2-diiodoethyl acetate (1b)

^{1}H NMR ($CDCl_3$, 300 MHz) δ 5.24 (1 H, d, *J* = 3.52), 4.38 (1 H, m), 2.05 (3 H, s), 1.72 (6 H, m), 1.15-0.85 (5 H, m); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 169.2 (C), 79.8 (CH), 42.2 (CH), 28.4 (CH_2), 27.2 (CH_2), 25.4 (CH_2), 25.1 (CH_2), 20.6 (CH_3), -23.6 (CH); MS 422.3 (M^+, <1), 362.3 (23), 295.4 (18), 253.3 (26), 235.5 (49), 125.3 (60), 107.2 (56), 55.1 (80), 43.1 (100); IR (neat) 2925, 2852, 1745, 1369, 1224, 1184, 1076, 1016, 974; R_f = 0.4 (hexane/AcOEt 20/1).

1,1-diiodo-3-phenylbut-2-yl acetate (1c)

^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.39-7.18 (5 H, m), 4.94 (1 H, dd, *J* = 10.03, 2.18), 4.81 (1 H, d, *J* = 2.18), 2.93 (1 H, m), 2.26 (3 H, s), 1.29 (3 H, d, *J* = 6.98); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 169.9 (C), 141.0 (C), 128.9 (CH),

127.3 (CH), 127.2 (CH), 80.5 (CH), 45.2 (CH), 20.9 (CH_3), 18.1 (CH_3), -23.3 (CH); MS 443.8 (M^+, 5), 383.8 (21), 316,9 (12), 257.0 (44), 130.1 (34), 105.0 (100), 77.0 (19), 43.0 (79); IR (neat) 3074, 3011, 2978, 2945, 1745, 1499, 1478, 1220, 1074, 975; R_f = 0.5 (hexane/AcOEt 10/1).

1,1-diiodo-4,8-dimethylnon-7-en-2-yl acetate (1d)

^{1}H NMR ($CDCl_3$, 300 MHz) δ 5.32-5.30 (1 H, m), 5.08 (1 H, m), 4.55-4.49 (1 H, m), 2.14 (3 H, s), 1.88-1.04 (13 H, m), 1.02-0.87 (3H, m); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 169.2 (C), 169.1 (C), 130.74 (C), 130.71 (C), 123.7 (CH), 74.5 (CH), 74.2 (CH), 40.0 (CH_2), 39.9 (CH_2), 36.6 (CH_2), 35.4 (CH_2), 28.3 (CH), 27.9 (CH), 25.2 (CH), 24.7 (CH_2), 24.4 (CH_2), 20.4 (CH_3), 19.4 (CH_3), 18.9 (CH_3), 17.2 (CH_3), -20.8 (CH), -21.2 (CH); MS 464.0 (M^+, 4), 422.0 (25), 404.1 (20), 340.0 (26), 337.1 (12), 43.0 (100); IR (neat) 2964, 1741, 1455, 1369, 1226, 1021; R_f = 0.5 (hexane/AcOEt 10/1).

(3*S*)-3-benzyloxy-1,1-diiodobut-2-yl acetate (1e)

^{1}H-NMR ($CDCl_3$, 200 MHz) δ 7.40-7.28 (10 H, m), 5.61, (1 H, d, *J* = 2.65), 5.26 (1 H, d, *J* = 8.51), 5.12 (1 H, dd, *J* =8.51, 3.52), 4.77 (1 H, dd, *J* = 7.92, 2.64), 4.66 y 4.44 (2 H, AB syst., *J* = 11.45), 4.64 and 4.47 (2 H, AB syst., *J* =10.86), 4.25 (1 H, qd, *J* = 6.16, 3.52), 3.62 (1 H, m), 2.27 (3 H, s), 2.20 (3 H, s), 1.24 (3 H, d, *J* =6.16), 1.21 (3 H, d, *J* =6.17); ^{13}C-NMR ($CDCl_3$, 50 MHz) δ 169.9 (C), 169.7 (C), 137.6 (C), 137.4 (C), 128.5 (CH), 128.4 (CH), 128.1 (CH), 128.0 (CH), 127.9 (CH), 79.1 (CH), 78.8 (CH), 73.0 (CH), 71.4 (CH), 71.3 (CH_2), 67.9 (CH_2), 21.0 (CH_3), 20.9 (CH_3), 16.2 (CH_3), 15.0 (CH_3), -25.8 (CH), -28.6 (CH); MS 287.3 (2), 181.2 (29), 135.3 (80), 107.2 (45), 91.1 (100), 43.2 (72); IR (neat) 2953, 2854, 1736, 1462; R_f = 0.3 (hexane/AcOEt 20/1).

3-chloro-1,1-diiodonon-2-yl acetate (1f)

^{13}C-NMR (75 MHz) δ 168.4 (C), 77.8 (CH), 76.6 (CH), 63.4 (CH), 61.2 (CH), 32.5 (CH_2), 31.2 (CH_2), 28.3 (CH_2), 28.2 (CH_2), 25.9 (CH_2), 25.6 (CH_2), 22.3 (CH_2), 22.1 (CH_2), 20.5 (CH_3), 20.3 (CH_3), 13.7 (CH_3), -26.1

(CH), -28.0 (CH); MS 412 (34), 345 (32), 121 (68), 43 (100); IR (neat) 2926, 2858, 1745, 1462, 1370, 1214, 1046, 932; ; R_f = 0.3 (hexane/AcOEt 20/1).

2,2-diiodo-1-phenylethyl acetate (1g)

^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.45-7.24 (5 H, m), 5.94 (1 H, d, *J* = 5.72), 5.31 (1 H, *J* = 5.73), 2.20 (3 H, s); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 168.6 (C), 136.3(C), 128.7 (CH), 128.1 (CH), 126.8 (CH), 78.8 (CH), 20.7 (CH_3), -22.1 (CH); MS 415.9 (M^+, 2), 289.0 (80), 162.1 (51), 91.0 (38), 77.0 (44), 43.0 (100); IR (neat) 3098, 3014, 2987, 2945, 1745, 1495, 1378, 1240, 1109, 985; R_f = 0.5 (hexane/AcOEt 10/1).

(*E*)-1,1-diiodopent-3-en-2-yl acetate (1h)

^{1}H NMR ($CDCl_3$, 300 MHz) δ 6.00-5.89 (1 H, m), 5.46 (1 H, ddq, *J* = 8.28, 6.98, 1.74), 5.14 (1 H, d, *J* = 3.93), 5.00 (1 H, dd, *J* = 6.97, 3.92), 2.05 (3 H, s), 1.76 (3 H, dd, *J* = 4.79, 1.75); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 168.8(C), 133.6 (CH), 126.3 (CH), 77.3 (CH), 20.8 (CH_3), 17.8 (CH), -21.3 (CH); MS 379.9 (M^+, <1), 319.9 (12), 267 (21), 41 (100); IR (neat) 2985, 2881, 1743, 1625, 1412, 1219, 945; R_f = 0.3 (hexane/AcOEt 20/1).

(*Z*)-1-iodonon-1-ene (2a) [1]

^{1}H NMR ($CDCl_3$, 300 MHz) δ 6.27-6.11 (2 H, m), 2.21-2.10 (2 H, m), 1.45-1.10 (10 H, m), 0.95-0.72 (3 H, m); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 141.4 (CH), 82.0 (CH), 34.6, 31.7, 29.1, 29.0, 27.9, 22.6 (6 × CH_2), 14.0 (CH_3); MS 252.0 (M^+, 60), 166.9 (23), 153.9 (14), 83.1 (58), 69.1 (100), 55.0 (61), 41.0 (52); IR (neat) 2924, 1458, 909; R_f = 0.8 (hexane).

(*Z*)-2-cyclohexyl-1-iodoethene (2b) [2]

---

[1] *E* isomer: G. C. M. Lee, B. Tobias, J. M. Holmes, D. A. Harcourt, M. E. Garst, *J. Am. Chem. Soc.* **1990**, *112*, 9330.

[2] a) H. C. Brown, C. Subrahmanyam, T. Hamaoka, N. Ravindran, D. H. Bowman, S. Misumi, M. K. Unni, V. Somayaji, N. G. Bhat, *J. Org. Chem.* **1989**, *54*, 6068. b) H. C. Brown, T. Hamaoka, N. Ravindran, C. Subrahmanyam, V. Somayaji, N. G. Bhat, *ibid*, 6075.

(*Z*)-1-iodo-3-phenylbut-1-ene (2c)

^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.40-7.21 (5 H, m), 6.33-6.24 (2 H, m), 3.90-3.82 (1 H, m), 1.43 (3 H, d, *J* = 6.98); min: 6.76-6.69 (1 H, m), 6.06 (1 H, dd, *J* = 14.38, 0.87), 3.56-3.47 (1 H, m); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 145.4 (CH), 143.7 (C), 128.4 (CH), 126.9 (CH), 126.4 (CH), 80.9 (CH), 44.5 (CH), 19.9 (CH_3); MS 257.9 (M^+, <1), 131.0 (100), 116.0 (20), 105.0 (10), 91.0 (17); IR (neat) 2980, 1602, 1492, 1450, 915; R_f = 0.6 (hexane); Anal. Calcd for $C_{10}H_{11}I$: C. 46.54, H. 4.30. Found: C 46.56, H 4.35.

(*Z*)-1-iodo-4,8-dimethylnona-1,7-diene (2d)

^{1}H NMR ($CDCl_3$, 300 MHz) δ 6.27-6.17 (2 H, m), 5.14-5-07 (1 H, m), 2.30-1.89 (4 H, m), 1.75-1.53 (7 H, m), 1.45-1.15 (2 H, m), 0.94 (3 H, d, *J* = 6.54); H min.: 6.57-6.43 (1 H, m), 6.00 (1 H, br d, *J* = 4.44); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 140.1 (CH), 131.3 (C), 124.5 (CH), 83.0 (CH), 41.6 (CH_2), 36.6 (CH_2), 32.0 (CH), 25.7 (CH_3), 25.5 (CH_2), 19.5 (CH_3), 17.6 (CH_3); MS 278.2 (M^+, <1), 194.1 (13), 167.0 (17), 109.2 (31), 95.2 (72), 81.2 (40), 69.2 (100), 41.1 (74); IR (neat) 2964, 2920, 1632, 1465, 1450, 1370, 738; R_f = 0.7 (hexane); Anal. Calcd for $C_{11}H_{19}I$: C. 47.49, H. 6.88; Found: C 47.47, H 6.92.

(3*S*, *Z*)-3-benzyloxy-1-iodobut-1-ene (2e)

^{1}H-NMR ($CDCl_3$, 200 MHz) δ 7.40 (5 H, m), 6.44 (1 H, d, *J* = 7.63), 6.27 (1 H, m), 4.52 (2 H, AB syst., *J* = 11.72), 4.32 (1 H, m), 1.32 (3 H, d, *J* = 6.27); ^{13}C-NMR ($CDCl_3$, 50 MHz) δ 142.9 (CH), 138.2 (C), 128.3 (CH), 127.8 (CH), 127.5 (CH), 82.8 (CH), 77.2 (CH), 70.5 (CH_2), 19.9 (CH_3); MS 288.2 (M^+, 29), 197.1 (22), 181.1 (21), 161.2 (70), 107.1 (30), 105.1 (71), 91.0 (100); IR (neat) 3063, 3030, 2974, 1606, 1454, 1271, 1064; R_f = 0.7 (hexane/AcOEt 10/1); $[\alpha]^{18}_{D}$ = + 64.3 (*c* 1.30, $CHCl_3$).

(*Z*)-3-chloro-1-iodonon-1-ene (2f)

^{1}H-NMR ($CDCl_3$, 300 MHz) δ 6.42 (1 H, d, *J* = 7.41), 6.27 (1 H, dd, *J* = 8.72, 7.41), 4.62 (1 H, m), 1.91-1.72 (2 H, m), 1.45-1.21 (8 H, m), 0.92-0.79 (3 H, m); ^{13}C-NMR ($CDCl_3$, 50 MHz) δ 141.3 (CH), 84.0 (CH), 63.0 (CH), 37.6 (CH_2), 31.5 (CH_2), 28.6 (CH_2), 26.0 (CH_2), 22.5 (CH_2), 14.0 (CH_3); C min. δ 145.5 (CH), 79.2 (CH), 63.5 (CH); MS 286 (M^+, 9), 201 (9), 180 (12), 123 (35), 81 (100), 67 (62), 43 (83); IR (neat) 2955, 2926, 1605, 1462, 1224, 1030; ; R_f = 0.8 (hexane).

(Z)-1-iodo-2-phenyletene (2g) [**Fehler! Unbekanntes Schalterargument.**]

(*Z*)-1-iodopenta-1,3-diene (2h) [3]

^{1}H NMR ($CDCl_3$, 300 MHz) δ 6.67 (1 H, dd, *J* = 9.59, 7.84) , 6.31-6.14 (1 H, m), 6.13-5.91 (2 H, m), 1.81 (3 H, d, *J* = 6.54); H min.: 6.97 (1 H, dd, *J* = 14.39, 10.46), 5.81-5.66 (1 H, m), 1.71 (3 H, d, *J* = 6.54); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 138.3 (CH), 134.9 (CH), 131.7 (CH), 79.0 (CH), 18.5 (CH_3); R_f = 0.6 (hexane).

1,1-dibromonon-2-yl acetate (3a)

^{1}H NMR ($CDCl_3$, 300 MHz) δ 5.68 (1 H, d, *J* = 3.48), 4.94 (1 H, m), 2.00 (3 H, s), 1.45-0.91 (12 H, m), 0.75 (3 H, m); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 169.4 (C), 75.8 (CH), 45.9 (CH), 31.4 (CH_2), 30.6 (CH_2), 28.8 (CH_2), 28.7 (CH_2), 24.8 (CH_2), 22.2 (CH_2), 20.3 (CH_3), 13.7 (CH_3); MS 171.1 (24), 123.1 (23), 111.1 (74), 43.0 (100); IR (neat) 2957, 2935, 2922, 1745, 1464, 1371, 1221, 1159, 1078, 1022, 717, 696; R_f = 0.7 (hexane/AcOEt 10/1).

1,1-dichloronon-2-yl acetate (3b)

^{1}H NMR ($CDCl_3$, 300 MHz) δ 5.78 (1 H, d, *J* = 3.52), 5.13 (1 H, m), 2.11 (3 H, s), 1.51-1-11 (12 H, m), 0.86 (3 H, m); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 169.8 (C), 75.8 (CH), 72.3 (CH), 31.5 (CH_2), 30.0 (CH_2), 28.9 (CH_2), 28.8 (CH_2), 24.8 (CH_2), 22.3 (CH_2), 20.4 (CH_3), 13.7 (CH_3); MS 111.2

[3] This compound decomposed on purification.

(12), 55.0 (10), 43.0 (100); IR (neat) 2957, 2926, 2858, 1751, 1465, 1438, 1373, 1226, 1082, 1026, 779; R_f = 0.6 (hexane/AcOEt 10/1).

1-chloro-1-iodonon-2-yl acetate (3c)

^{1}H NMR ($CDCl_3$, 300 MHz) δ 5.81 (2 H, m), 4.83 (1 H, m), 4.69 (1 H, m), 2.09 (3 H, s), 2.08 (3 H, s), 1.51-1.09 (24 H, m), 0.83 (6 H, m); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 169.8 (C), 77.0 (CH), 76.6 (CH), 31.6 (CH_2), 31.3 (CH), 31.2 (CH_2), 29.1 (CH_2), 29.0 (CH_2), 28.9 (CH_2), 25.0 (CH_2), 24.9 (CH_2), 22.5 (CH_2), 20.7 (CH_3), 13.9 (CH_3); MS 346.0 (M^+, <1), 219.1 (31), 123.1 (19), 81.1 (20), 43.0 (100); IR (neat) 2955, 2928, 2856, 1749, 1458, 1371, 1224, 1024; R_f = 0.5 (hexane/AcOEt 10/1).

2-bromo-2-chloro-1-cyclohexylethyl acetate (3d)

^{1}H NMR ($CDCl_3$, 300 MHz) δ 5.83 (1 H, d, *J* = 3.8), 5.77 (1 H, d, *J* = 4.1), 5.02 (1 H, dd, *J* = 7.3, 3.8), 4.88 (1 H, dd, *J* = 7.3, 4.1), 2.03 (3 H, s), 2.02 (3 H, s), 1.72-1.38 (12 H, m), 1.23-0.80 (10 H, m); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 169.4 (C), 79.0 (CH), 78.6 (CH), 59.7 (CH), 58.8 (CH), 39.6 (CH), 39.2 (CH), 28.5 (CH_2), 27.3 (CH_2), 27.0 (CH_2), 25.5 (CH_2), 25.2 (CH_2), 25.1 (CH_2), 20.2 (CH); MS 155.2 (14), 95.1 (100), 43.0; IR (neat) 2933, 2856, 1745, 1448, 1371, 1226, 1186, 1087, 1064, 1045, 1018, 758, 717; R_f = 0.5 (hexane/AcOEt 10/1).

1-chloro-1-iodo-3-phenylbut-2-yl acetate (3e)

^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.38-7.12 (10 H, m), 5.62 (1 H, dd, *J* = 10.0, 1.9), 5.49 (1 H, d, *J* = 2.2), 5.34 (1 H, d, *J* = 2.18), 4.75 (1 H, dd, *J* = 9.8, 2.2), 2.26 (1 H, m), 2.98 (1 H, m), 2.29 (3 H, s), 2.26 (3 H, s), 1.30 (6 H, dd, *J* = 7.08, 3.0); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 169.6 (C), 169.5 (C), 140.8 (C), 140.6 (C), 128.8 (CH), 128.7 (CH), 128.0 (CH), 127.1 (CH), 80.9 (CH), 79.2 (CH), 44.3 (CH), 41.8 (CH), 32.9 (CH), 28.2 (CH), 20.5 (CH_3), 20.4 (CH_3), 18.0 (CH), 17.6 (CH); MS 226.2 (9), 225.3 (24), 167.2 (22), 165.2 (62), 106.2 (31), 105.0 (100), 43.0 (74); IR (neat) 3029, 2975,

2934, 1750, 1494, 1453, 1371, 1218, 1077, 1050, 1019, 764, 701; R_f = 0.5 (hexane/AcOEt 10/1).

1-bromo-1-chloro-4,8-dimethylnon-7-en-2-yl acetate (3f)

^{1}H NMR ($CDCl_3$, 300 MHz) δ 5.81 (1 H, br d, *J* = 3.27), 5.26-5.0 (2 H, m), 2.10 (3 H, br s), 2.09-0.9 (16 H, m); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 169.6 (C), 169.5 (C), 169.4 (C), 131.0 (C), 130.9 (C), 124.0 (CH), 74.2 (CH), 74.04 (CH), 74.01 (CH), 73.8 (CH), 60.6 (CH), 60.5 (CH), 60.4 (CH), 60.3 (CH), 37.1 (CH_2), 36.8 (CH_2), 36.6 (CH_2), 36.4 (CH_2), 36.2 (CH_2), 35.6 (CH_2), 35.5 (CH_2), 28.5 (CH), 28.2 (CH), 25.4 (CH_3), 25.0 (CH_2), 24.7 (CH_2), 20.3 (CH_3), 19.8 (CH_3), 18.9 (CH_3), 18.8 (CH_3), 17.3 (CH_3); MS 327.3 (22), 325.3 (20), 285.2 (38), 283.2 (34), 267.2 (42), 265.2 (34), 249.2 (33), 247.2 (30), 203.2 (29), 201.2 (31), 183.2 (31), 43.0 (100); IR (neat) 2961, 2926, 1750, 1373, 1224; R_f = 0.5 (hexane/AcOEt 10/1).

(*Z*)-1-bromonon-1-ene (4a) [4]

(Z)-1-chlorononon-1-ene (4b) [**Fehler! Unbekanntes Schalterargument.**]

(*Z*)-1-chloro-2-cyclohexylethene (4d) [5]

(*Z*)-1-chloro-3-phenylbut-1-ene (4e)

^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.39-7.20 (5 H, m), 6.08 (1 H, d, *J* = 6.97), 5.93, (1 H, dd, *J* = 9.15, 6.97), 4.14 (1 H, m), 1.43 (3 H, d, *J* = 6.98); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 144.4 (C), 136.3, 128.4, 126.7, 126.2, 116.8 (5 × CH), 37.1 (CH), 20.5 (CH_3); MS 166 (M^+, 12), 131 (100), 115 (52), 91 (24), 77 (13), 51 (17); IR (neat) 3030, 2968, 2930, 2874, 1747, 1626, 1452, 1226; R_f = 0.5 (hexane); Anal. Calcd for $C_{10}H_{11}Cl$: C. 72.07, H. 6.65; Found: C 72.04, H 6.71.

(*Z*)-1-chloro-4,8-dimethylnona-1,7-diene (4f)

---

[4] J. Barluenga, J. L. Fernández-Simón, J. M. Concellón, M. Yus, *J. Chem. Soc., Perkin Trans. I* **1989**, 691.

[5] R. B. Miller, G. McGarvey, *J. Org. Chem.* **1978**, *43*, 4424.

^{1}H NMR ($CDCl_3$, 300 MHz) δ 6.06 (1 H, m), 5.78 (1 H, m), 5.11 (1 H, m), 2.32-1.82 (4 H, m), 1.70-1.49 (7 H, m), 1.45-1.09 (2 H, m), 0.92 (3 H, d, *J* = 6.54); H min.: 5.93-5.87 (1 H, m); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 131.2 (C), 130.4 (CH), 124.6 (CH), 118.5 (CH), 36.5 (CH_2), 33.9 (CH_2), 32.2 (CH), 25.6 (CH_3), 25.5 (CH_2), 19.4 (CH_3), 17.5 (CH_3); MS 186.1 (M^+, <1), 109.2 (25), 81.1 (20), 59.0 (100); IR (neat) 2960, 2926, 1629, 1460, 1450, 1377, 746; R_f = 0.5 (hexane); Anal. Calcd for $C_{11}H_{19}Cl$: C. 70.76, H. 10.26; Found: C 70.74, H 10.32.

2-trimethylsilyloxy-1,1-diiodononane

^{1}H NMR ($CDCl_3$, 300 MHz) δ 5.12 (1 H, d, *J* = 3.05), 3.11-3.01 (1 H, m), 1.80-1.63 (1 H, m), 1.54-1.15 (11 H, m); 0.96-0.79 (3 H, m), 0.17 (9 H. s); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 77.4 (CH), 35.9 (CH_2), 31.6 (CH_2), 29.2 (CH_2), 29.1 (CH_2), 25.2 (CH_2), 22.5 (CH_2), 14.0 (CH_3), 0.6 (CH_3), -10.5 (CH); MS 453.4 (3), 369.2 (12), 341.4 (24), 242.3 (35), 202.5 (44), 201.4 (100), 185.2 (49), 103.2 (40), 75.2 (73), 73.2 (92), 69.2 (58); IR (neat) 2955, 2924, 2854, 1251, 1095, 1084, 841; R_f = 0.6 (hexane).

1,1-diiodononan-2-ol

^{1}H NMR ($CDCl_3$, 300 MHz) δ 5.28 (1 H, d, *J* = 3.01), 3.15 (1 H, br s), 2.40 (1 H, br s), 1.74-1.18 (12 H, m), 0.87 (3 H. m); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 77.3 (CH), 36.0 (CH_2), 31.6 (CH_2), 29.3 (CH_2), 29.0 (CH_2), 25.3 (CH_2), 22.5 (CH_2), 14.0 (CH_3), -9.4 (CH); MS 395.9 (M^+, 15), 269.0 (48), 169.9 (40), 123.1 (56), 81.0 (100), 55.0 (60), 41.0 (74);); HRMS Calcd for $C_9H_{18}I_2O$ 395.9447, found 395.9449; IR (neat) 3402, 3410, 2924, 1076, 1464; R_f = 0.3 (hexane/AcOEt 10/1).