Synthesis of α-Methylene-γ-butyrolactones via Addition of Tin Hydride to Enynes Induced by Triethylborane

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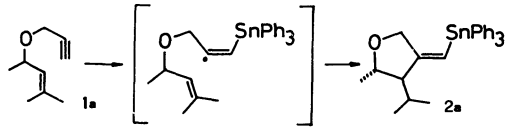
Synopsis. Et_3B induced addition of Ph_3SnH to suitably constituted enynes provides (triphenylstannylmethylene) oxolanes stereoselectively in one pot. Destannylation followed by oxidation affords α-methylene-γ-butyrolactones in fair yields.

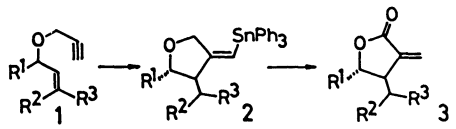
We have recently shown[1] that triphenyltin hydride adds easily to acetylenes regioselectively in the presence of Et_3B under mild conditions. The reaction has been found to be effective for vinyl-radical cyclization[2] of suitably constituted acetylenic olefins.[1,3] Here we wish to report the applicaton of this new method to the stereoselective synthesis of α-methylene-γ-butyrolactones, which represent a major class of known natural products and possess wide-ranging biological activities.[4]

A hexane solution of Et_3B was added to a solution of Ph_3SnH and enyne, 1,3-dimethyl-2-butenyl propargyl ether (**1a**) in toluene at 25 °C under an argon atmosphere. The mixture was stirred for 3 h at 25 °C and workup followed by preparative tlc purification gave cyclized product **2a** in 84% yield.



The results are summarized in Table 1. The yields of cyclized products depend on the concentration of the substrate and Ph_3SnH. High dilution favors the formation of the desired cyclized product **2**. On the other hand, without solvent or high concentration provided uncyclized product along with **2**.[5] It is worth noting that the cyclized products, **2a—d**, consist of only (Z)-trans-isomer independently of the stereochemistry of the double bond in the starting enynes (**1c** and **1d**). In contrast, treatment of **1e** with Ph_3SnH gave cis-fused oxolane **2e** exclusively which is thermodynamically more stable than trans-isomer.

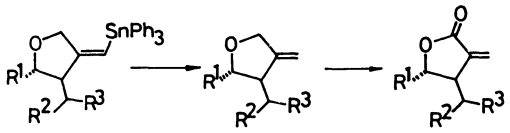
Table 1. Synthesis of α-Methylene-γ-butyrolactone



1	R^1	R^2	R^3	Yield/%	
				2	3[a]
a	Me	Me	Me	84	57
b	Ph	Me	Me	70	39
c	*n*-Bu	H	*n*-Pr	83	41
d	Me	*n*-Bu	H	75	59
e	$-(CH_2)_4-$		H	71[b]	31

a) Overall yield from **2**. b) Cis product was obtained. See experimental part.

Destannylation[6] (*n*-BuLi/THF and H_2O) followed by oxidation[7] with $CrO_3 \cdot 2py$ gave the desired α-methylene-γ-butyrolactones.[8]



Experimental

The IR spectra were determined on a JASCO IR-810 spectrometer, the mass spectra on a Hitachi M-80 machine, the ^1H NMR spectra were recorded on a Varian EM-390H and XL-200 spectrometers, and the ^{119}Sn NMR spectra on a JEOL JNM-FX 90Q spectrometer. The chemical shifts of the proton NMR are given in δ with TMS as an internal standard, and those of the ^{119}Sn NMR are given in δ with Me_4Sn as an internal standard. The analyses were performed at the Elemental Analyses Center of Kyoto University. Tetrahydrofuran was dried in benzophenone ketyl and distilled. All the experiments were carried out under an argon atmosphere.

Preparation of Alkenyl Propargyl Ether. Generation of (*E*)-1-butyl-2-hexenyl propargyl ether (**1c**) is representative. A hexane solution of butyllithium (1.5 M, 1 M=1 mol dm^{-3}, 13.3 ml, 20.0 mmol) was added to a solution of (*E*)-2-hexenal (1.96 g, 20.0 mmol) in THF (30 ml) at 0 °C. After stirring for 15 min, HMPA (5 ml) and propargyl bromide (1.87 ml, 21.0 mmol) was added. The resulting mixture was stirred at 0 °C for 15 min, then at 25 °C for 3 h. Workup (AcOEt, 1 M HCl) followed by purification by silica-gel column chromatography and distillation gave **1c** (2.6 g, 67% yield) as a colorless oil: Bp 120 °C (20 Torr, 1 Torr=133.322 Pa); IR (neat) 3308, 2956, 2928, 2860, 1655, 1638, 1459, 1076 cm^{-1}; ^1H NMR ($CDCl_3$) δ=0.84—0.97 (m, 6H), 1.27—1.70 (m, 8H), 2.00—2.12 (m, 2H), 2.37 (dd, *J*=2.0, 2.0 Hz, 1H), 3.75—3.87 (m, 1H), 4.01 (dd, *J*=16.0, 2.0 Hz, 1H), 4.18 (dd, *J*=16.0, 2.0 Hz, 1H), 5.25 (dd, *J*=15.5, 9.0 Hz, 1H), 5.65 (dt, *J*=15.5, 6.5 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ=13.6, 14.0, 22.3, 22.6, 27.6, 34.2, 35.2, 54.6, 73.4, 79.8, 80.5, 129.9, 135.1. Found: C, 80.34; H, 11.64%. Calcd for $C_{13}H_{22}O$: C, 80.35; H, 11.41%.

1,3-Dimethyl-2-butenyl Propargyl Ether (1a): Bp 120 °C (760 Torr); IR (neat) 3300, 2972, 2928, 1671, 1445, 1376, 1265, 1208, 1150, 1104, 1077, 1037, 844 cm^{-1}; ^1H NMR ($CDCl_3$) δ=1.20 (d, *J*=7.0 Hz, 3H), 1.69 (s, 3H), 1.72 (s, 3H), 2.38 (bs, 1H), 4.02 (d, *J*=15.0 Hz, 1H), 4.13 (d, *J*=15.0 Hz, 1H), 4.40 (dq, *J*=10.0, 7.0 Hz, 1H), 5.02 (d, *J*=10.0 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ=18.0, 21.2, 25.27, 54.4, 70.5, 73.3, 80.6, 126.2, 136.3.

1-Phenyl-3-methyl-2-butenyl Propargyl Ether (1b): Bp

160 °C (20 Torr); IR (neat) 3290, 2968, 2916, 2852, 1451, 1376, 1066, 1027, 755, 698 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ=1.76 (s, 3H), 1.83 (s, 3H), 2.42 (dd, J=2.4, 2.4 Hz, 1H), 4.07 (dd, J=17.0, 2.4 Hz, 1H), 4.20 (dd, J=17.0, 2.4 Hz, 1H), 5.33 (s, 2H PhCH–OCH_2+Me_2C=CH), 7.35 (m, 5H), ^{13}C NMR ($CDCl_3$) δ 18.4, 26.0, 54.8, 74.0, 76.5, 80.4, 125.2, 126.8, 127.6, 128.5, 137.1, 142.1. Found: C, 83.88; H, 8.07%. Calcd for $C_{14}H_{16}O$: C, 83.96; H, 8.05%.

(*Z*)-1-Methyl-2-heptenyl Propargyl Ether (1d): Bp 130 °C (20 Torr); IR (neat) 3306, 2956, 2926, 2858, 1460, 1440, 1371, 1132, 1087, 1071, 1042, 913, 734 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ= 0.91 (t, J=10.0 Hz, 3H), 1.24 (d, J=6.3 Hz, 3H), 1.25—1.50 (m, 4H), 2.05—2.25 (m, 2H), 2.39 (dd, J=2.4, 2.4 Hz, 1H), 4.04 (dd, J=15.7, 2.4 Hz, 1H), 4.14 (dd, J=15.7, 2.4 Hz, 1H), 4.49 (dq, J=9.2, 6.3 Hz, 1H), 5.22 (dd, J=9.2, 11.0 Hz, 1H), 5.60 (dt, J=11.0, 7.5 Hz, 1H): ^{13}C NMR ($CDCl_3$) δ=13.8, 21.4, 22.3, 27.3, 31.9, 54.7, 69.5, 73.5, 80.1, 130.6, 133.9. Found: C, 79.25; H, 11.11%. Calcd for $C_{11}H_{18}O$: C, 79.46; H, 10.91%.

2-Cyclohexenyl Propargyl Ether (1e): Bp 140 °C (760 Torr); IR (neat) 3296, 3024, 2930, 2854, 1438, 1263, 1081, 1020, 725 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ=1.43—2.20 (m, 8H), 2.42 (dd, J=2.4, 2.4 Hz, 1H), 4.05—4.17 (m, 1H), 4.20 (d, J=2.4 Hz, 1H), 4.22 (d, J=2.4 Hz, 1H), 5.81 (d, J=10.0 Hz, 1H), 5.92 (dt, J=10.0, 3.8 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ=19.0, 25.1, 28.0, 55.2, 71.7, 73.8, 81.4, 127.0, 131.4. Found: m/z 136.0742, 135.0768. Calcd for $C_9H_{12}O$: M, 136.0887, M−1, 135.0808.

General Procedure for the Enyne Cyclization Mediated by Et_3B. A hexane solution of Et_3B (1.0 M, 0.2 ml, 0.2 mmol) was added to a solution of Ph_3SnH (0.42 g, 1.2 mmol) and enyne **1a** (0.14 g, 1.0 mmol) in toluene (100 ml) at 25 °C. After stirring for 3 h at 25 °C, the reaction mixture was poured into water and extracted with ethyl acetate. Purification by preparative tlc gave (*Z*)-*trans*-3-isopropyl-2-methyl-4-(triphenylstannylmethylene)oxolane (**2a**) as a colorless oil (0.41 g, 84% yield): Bp 170 °C (bath temp, 0.1 Torr); ^{13}C NMR ($CDCl_3$) δ=18.9, 20.7, 21.6, 30.4, 59.9, 72.3, 77.5, 112.8, 128.6, 136.2, 138.3, 165.0. Found: C, 66.43; H, 6.29%. Calcd for $C_{27}H_{30}OSn$: C, 66.29; H, 6.18%. IR, ^{1}H NMR, and ^{119}Sn NMR data are shown in Ref. 1.

(*Z*)-*trans*-3-Isopropyl-2-phenyl-4-(triphenylstannylmethylene)oxolane (2b): Bp 220 °C (bath temp, 0.2 Torr); IR (neat) 3060, 2956, 2924, 1429, 1075, 1059, 1023, 908, 727, 697 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ=1.01 (d, J=7.0 Hz, 3H), 1.11 (d, J=7.0 Hz, 3H), 2.73 (m, 1H), 4.22 (d, J=14.0 Hz, 1H), 4.35 (d, J=14.0 Hz, 1H), 4.97 (d, J=5.0 Hz, 1H), 6.12 (s, 1H), 7.28—7.70 (m, 20H); ^{13}CNMR ($CDCl_3$) δ=19.5, 20.5, 30.8, 61.7, 73.2, 83.5, 114.1, 125.9, 127.3, 128.3, 128.4, 128.6, 136.8, 137.2, 138.1 164.3; ^{119}Sn NMR ($CDCl_3$) δ=−145.99. Found: C, 69.87; H, 5.81%. Calcd for $C_{32}H_{32}OSn$: C, 69.72; H, 5.85%.

(*Z*)-*trans*-2,3-Dibutyl-4-(triphenylstannylmethylene)-oxolane (2c): Bp 200 °C (bath temp, 20 Torr); IR (neat) 3060, 3010, 2952, 2926, 2854, 1466, 1458, 1429, 1075, 726, 697 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ=0.85—0.99 (m, 6H), 1.25—1.68 (m, 12H), 2.33—2.43 (m, 1H), 3.62—3.75 (m, 1H), 4.06 (dd, J=16.0, 2.2 Hz, 1H), 4.18 (dd, J=16.0, 2.2 Hz, 1H), 6.06 (dd, J=2.2, 2.2 Hz, 1H), 7.34—7.79 (m, 15H); ^{13}C NMR ($CDCl_3$) δ=14.0, 22.7, 23.0, 28.2, 29.3, 32.1, 35.1, 51.9, 72.0, 84.5, 111.2, 128.6, 129.0, 136.8, 138.3, 166.5; ^{119}Sn NMR ($CDCl_3$) δ=−143.98. Found: C, 68.36; H, 6.89%. Calcd for $C_{31}H_{38}OSn$: C, 68.28; H, 7.02%.

(*Z*)-*trans*-2-Methyl-3-pentyl-4-(triphenylstannylmethylene)-oxolane (2d): Bp 190 °C (bath temp, 0.1 Torr); IR (neat) 3060, 3046, 3012, 2954, 2926, 2854, 1740, 1619, 1480, 1459, 1429, 1380, 1240, 1111, 1075, 1048, 1023, 997 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ=0.91 (t, J=6.8Hz, 3H), 1.27 (d, J=6.0Hz, 3H), 1.15—1.65 (m, 8H), 2.15—2.30 (m, 1H), 3.68—3.78 (m, 1H), 3.92 (d, J=14.0 Hz, 1H), 4.10 (d, J=14.0 Hz, 1H), 6.03 (s, 1H), 7.23—7.68 (m, 15H); ^{13}C NMR ($CDCl_3$) δ=14.1, 22.6, 24.2, 26.7, 31.6, 32.2, 53.4, 72.1, 80.7, 111.0, 128.6, 129.0, 136.8, 138.3, 166.5; ^{119}Sn NMR ($CDCl_3$) δ=−144.96. Found: C, 67.36; H, 6.52%. Calcd for $C_{29}H_{34}OSn$: C, 67.34; H, 6.63%.

(*Z*)-*cis*-Hexahydro-3-(triphenylstannylmethylene)benzofuran (2e): Mp 85 °C (hexane); IR (neat, before crystallization) 3060, 2928, 2825, 1623, 1480, 1428, 1074, 1052, 727, 697 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ=1.20—1.83 (m, 8H), 2.63—2.77 (m, 1H), 4.00—4.33 (m, 3H), 6.06 (s, 1H), 7.35—7.80 (m, 15H); ^{13}C NMR ($CDCl_3$) δ=21.4, 23.0, 27.2, 27.8, 46.8, 71.3, 78.0, 110.3, 128.6, 129.0, 136.8, 138.2, 166.4; ^{119}Sn NMR ($CDCl_3$) δ=−143.01. Found: C, 66.36; H, 5.77%. Calcd for $C_{27}H_{28}OSn$: C, 66.56; H, 5.79%.

Typical Procedure for Destannylation. Butyllithium (1.5 M hexane solution, 2.7 ml, 4.0 mmol) was added to a soluton of (triphenylstannylmethylene)oxolane (**2a**) (0.49 g, 1.0 mmol) in THF (5 ml) at −78 °C. After stirring for 15 min, MeOH (1.0 ml) was added and the resulting mixture was allowed to warm to 25 °C. The mixture was poured into 1 M HCl solution and extracted with ethyl acetate. The organic extracts were washed with brine, dried (Na_2SO_4), and concentrated. The crude product was purified by preparative TLC (silica gel, hexane–ethyl acetate=20:1) to give (*Z*)-*trans*-3-isopropyl-2-methyl-4-methyleneoxolane (IR (neat) 3064, 2956, 2924, 2872, 1659, 1459, 1371, 1090, 1048 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ=0.94 (d, J=6.8 Hz, 3H), 0.96 (d, J=6.9 Hz, 3H), 1.24 (d, J=6.2 Hz, 3H), 1.68—1.97 (m, 1H), 2.08—2.20 (m, 1H), 3.98—4.17 (m, 1H), 4.24 (d, J=12.5 Hz, 1H), 4.36 (d, J=12.5 Hz, 1H), 4.96 (bs, 1H), 5.01 (bs, 1H)) which was contaminated by unidentified compounds and converted into α-methylene-γ-butyrolactone without further purification.

Transformation of Methyleneoxolane to α-Methylene-γ-butyrolactone. According to the procedure described in the literature,[7] methyleneoxolane derived from **2a** was oxidized with Collins reagent to give *trans*-4,5-dihydro-4-isopropyl-5-methyl-3-methylene-2(3*H*)-furanone (**3a**, 88 mg, 57% yield): Bp 90 °C (bath temp, 20 Torr); IR (neat) 2962, 2928, 2874, 1762, 1654, 1459, 1275, 1126, 1099, 1033 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ=0.94 (d J=6.7 Hz, 3H), 0.95 (d, J=6.9 Hz, 3H), 1.37 (d, J=6.4 Hz, 3H), 1.84—2.05 (m, 1H), 2.52—2.61 (m, 1H), 4.47 (dq, J=3.3, 6.4 Hz, 1H), 5.62 (d, J=2.1 Hz, 1H), 6.35 (d, J=2.1 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ=18.2, 19.0, 22.7, 31.4, 52.1, 76.8, 123.5, 137.6, 170.7. Found: m/z 154.0831, 139.0744. Calcd for $C_9H_{14}O_2$: M, 154.0992, M−CH_3, 139.0758.

***trans*-4,5-Dihydro-4-isopropyl-3-methylene-5-phenyl-2(3*H*)-furanone (3b)**: Bp 120 °C (bath temp, 20 Torr); IR (neat) 2960, 2872, 1765, 1656 1458, 1401, 1390, 1270, 1126, 1021, 1001, 964 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ=1.01 (d, J=5.7 Hz, 3H), 1.05 (d, J=5.6 Hz, 3H), 1.95—2.13 (m, 1H), 2.85—2.93 (m, 1H), 5.31 (d, J=2.9 Hz, 1H), 5.65 (d, J=2.2 Hz, 1H), 6.41 (d, J=2.2 Hz, 1H), 7.49—7.70 (m, 5H); ^{13}C NMR ($CDCl_3$) δ=18.5, 18.9, 32.2, 53.6, 81.1, 123.9, 125.3, 128.3, 128.8, 136.8, 140.6, 170.6. Found: C, 77.47; H, 7.65%. Calcd for $C_{14}H_{16}O_2$: C, 77.75; H, 7.46%.

***trans*-4,5-Dibutyl-4,5-dihydro-3-methylene-2(3*H*)-furanone (3c)**:[9] Bp 155 °C (bath temp, 20 Torr); IR (neat) 2954, 2928, 2860, 1763, 1467, 1459, 1268, 1151, 1110 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ=0.90—1.01 (m, 6H), 1.33—1.76 (m, 12H), 2.60—2.75 (m, 1H), 4.22 (dt, J=4.1, 6.2 Hz, 1H), 5.59 (d, J=2.4 Hz, 1H), 6.26 (d, J=2.4 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ=13.9, 22.4, 22.6, 27.2, 28.5, 33.9, 35.9, 44.5, 83.5, 122.0, 139.5, 170.3.

***trans*-4,5-Dihydro-5-methyl-4-pentyl-3-methylene-2(3*H*)-furanone (3d)**: Bp 150 °C (bath temp, 20 Torr); IR (neat) 2956, 2928, 2856, 1764, 1664, 1459, 1402, 1382, 1271, 1162, 1126, 1102, 1053, 948, 815 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ=0.90 (t, J=5.0 Hz, 3H), 1.33—1.74 (m, 9H), 1.41 (d, J=6.3 Hz, 3H), 2.56—2.70 (m, 1H), 4.37 (dq, J=4.8, 6.3 Hz, 1H), 5.59 (d, J=2.6 Hz, 1H), 6.26 (d, J=2.6 Hz, 1H); ^{13}C NMR ($CDCl_3$)

δ=13.9, 21.7, 22.4, 26.1, 31.7, 33.5, 46.3, 79.8, 121.9, 139.6, 170.3. Found: C, 72.55; H, 10.24%. Calcd for $C_{11}H_{18}O_2$: C, 72.49; H, 9.95%.

***cis*-Hexahydro-3-methylene-2(3*H*)-benzofuranone (3e)**: The compound was obtained from **1e** in similar fashion described for the conversion of **1a** into **3a**. The physical deta of **3e** were identical with those in the literature.[10]

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5) High dilution favors the intramolecular radical cyclization. Y. Ueno, K. Chino, and M. Okawara, *Tetrahedron Lett.*, **23**, 2575 (1982) For instance, the enyne, Me_2C=$CHCH_2CH_2C(OH)MeC{\equiv}CH$ gave cyclized product, 3-isopropyl-1-methyl-2-(triphenylstannylmethylene)cyclopentanol exclusively at 0.012 M concentration of Ph_3SnH. Meanwhile, the reaction at 80 °C without solvent gave a complex mixture consisting of (*E*)- and (*Z*)-vinylstannanes (Me_2C=$CHCH_2CH_2C(OH)MeCH$=$CHSnPh_3$, 46%), regioisomer (Me_2C=$CHCH_2CH_2C(OH)MeC(SnPh_3)$=$CH_2$, 9%), and the desired cyclized product (38%).

6) Triphenylstannylalkenes were easily transformed into alkenyllithium upon treatment with *n*-BuLi as tributylstannylalkenes. E. J. Corey, P. Ulrich, and J. M. Fitzpatrick, *J. Am. Chem. Soc.*, **98**, 222 (1976).

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8) An addition of galvinoxyl (0.1 mmol) to a reaction mixture of 1-dodecyne (1.0 mmol), Ph_3SnH (1.1 mmol), and Et_3B (0.1 mmol) resulted in a recovery of the acetylene (85%). The organoboranes are known to be excellent sources of free radicals in the presence of oxygen (H. C. Brown and M. M. Midland, *Angew. Chem., Int. Ed. Engl.*, **11**, 692 (1972); A. Suzuki, S. Nozawa, M. Itoh, H. C. Brown, G. W. Kabalka, and G. W. Holland, *J. Am. Chem. Soc.*, **92**, 3503 (1970)). Thus, we are tempted to assume a radical chain mechanism for the reaction. A trace of oxygen could be in a reaction mixture and initiate the free-radical reaction, although the reactions have been achieved under an argon atmosphere.

9) *cis*-Dibutyl-4,5-dihydro-3-methylene-2(3*H*)-furanone[10d] was obtained as minor product (trans:cis=96:4). ^1H NMR ($CDCl_3$) δ=4.56 (m, 1H), 5.52 d, *J*=2.4 Hz, 1H), 6.21 (d, *J*=2.4 Hz, 1H).

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