DEVELOPMENT OF A METHOD FOR THE SYNTHESIS OF α-SUBSTITUTED α,β-UNSATURATED LACTONES BASED ON STILLE-TYPE Pd-CATALYZED CARBONYLATION OF (*Z*)-ω-IODOALKENOLS. AN EFFICIENT AND SELECTIVE SYNTHESIS OF (+)-HAMABIWALACTONE B†

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Abstract-Pd-Catalyzed carbonylative lactonization of (*Z*)-ω-iodoalkenols can be applied to synthesize α,β-unsaturated lactones containing an alkenyl or alkynyl substituent in the α position, providing a generally superior alternative to recently developed methods involving Pd-catalyzed α-substitution of α-stannyl esters, as exemplified by a highly expeditious synthesis of (+)-hamabiwalactone B.

As part of our long-standing project on the development of a highly selective α-substitution methodology[1] based on Pd- or Ni-catalyzed cross coupling of α-halo-α,β-unsaturated carbonyl compounds, we recently sought a general and satisfactory route to α-substituted lactones and lactams. To our disappointment, however, none of the recently developed methods for α-iodination of α,β-unsaturated enones[2] tested by us would convert 2(5*H*)-furanone to its 3-iodo derivative in detectable yields. Although some alternative protocols involving the use of α-stannyllactones have been reported recently,[3,4] they suffer from the facts that the requisite α-stannyllactones are not readily accessible and that their subsequent Pd-catalyzed cross coupling is generally low-yielding.

It then occurred to us that Stille's protocol[5] for Pd-catalyzed carbonylative lactonization based on earlier pioneering works by Heck[6] on intermolecular esterification and by Mori and Ban[7] on carbonylative

†This paper is dedicated to Professor Teruaki Mukaiyama in recognition of his pioneering and inspiring contributions to organic synthesis.

lactonization of *o*-bromo-ω-hydroxyalkylbenzenes would be a generally more attractive alternative. To the best of our knowledge, however, its scope has been limited to those cases where the α-substituents are some alkyl and one aryl, *i.e.*, Ph, groups.[5] Other sp^2- and sp-carbon groups, such as alkenyl and alkynyl groups that can chelate organopalladium intermediates and interfere with the desired Pd-catalyzed lactonization do not appear to have been previously employed.

To probe this matter, we prepared several representative (*Z*)-3-iodo-2-propenols (**1**) containing additional unsaturated substituents *via* Pd-catalyzed and other appropriate cross coupling reactions of propargyl alcohols followed by reduction with $LiAlH_4$—NaOMe and iodinolysis.[8] These (*Z*)-3-iodo-2-propenols were then subjected to carbonylation with CO (5-40 atm) in the presence of a catalytic amount (2-5 mol%) of $Cl_2Pd(PPh_3)_2$, and NEt_3 or K_2CO_3 (1-1.5 molar equiv.) in THF or DMF. As the experimental results summarized in Table 1 indicate, all reactions examined proceeded to give the corresponding butenolides **2** in good to excellent yields, even though these reactions were relatively slow at room temperature requiring 1-5 days. As the corresponding reactions of the Me or Ph substituted derivatives were notably faster, it is likely that an additional unsaturated group retards the desired reaction presumably through chelation. This phenomenon is more pronounced with an alkynyl group than with an alkenyl group. No additional efforts to further optimize the reaction conditions have so far been made. Despite some room for further improvement, the protocol presented herein appears to be considerably more efficient and satisfactory than the recently developed Pd-catalyzed α-substitution protocol.[1c,3] We have also briefly examined the applicability of the method reported herein to the synthesis of six-membered α-substituted α,β-unsaturated lactones through the use of stereoselective reduction-iodinolysis of homopropargyl alcohols with Red-Al and I_2.[9] To this end, 4-phenyl-3-butyn-1-ol was converted to (*Z*)-4-iodo-4-phenyl-3-butyn-1-ol (**3**) in 86% yield. Its lactonization proved to be more sluggish than the corresponding reaction of (*Z*)-3-iodo-3-phenyl-2-propen-1-ol. Nonetheless, the desired lactone (**4**) was obtained in 75% yield in 12 h at 40 atm of CO and 100 °C in DMF.

Scheme 1

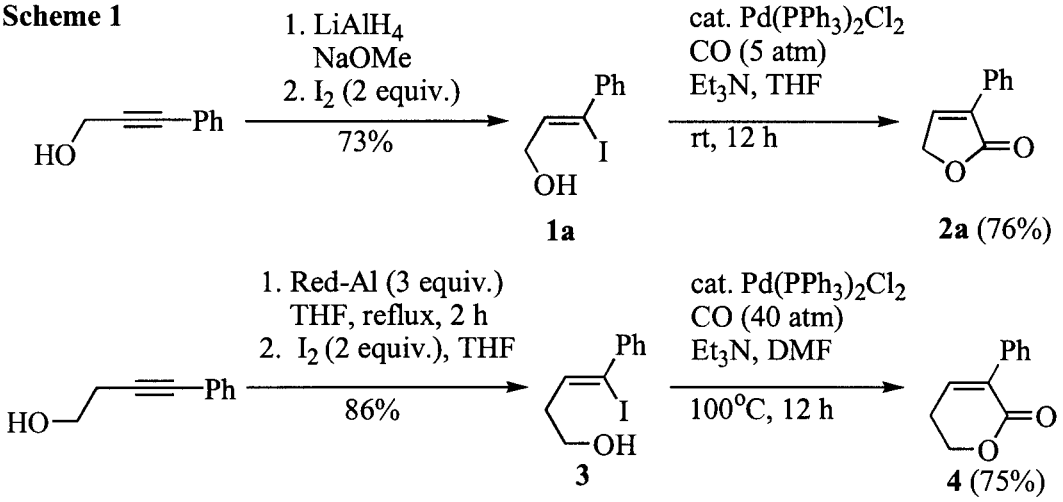
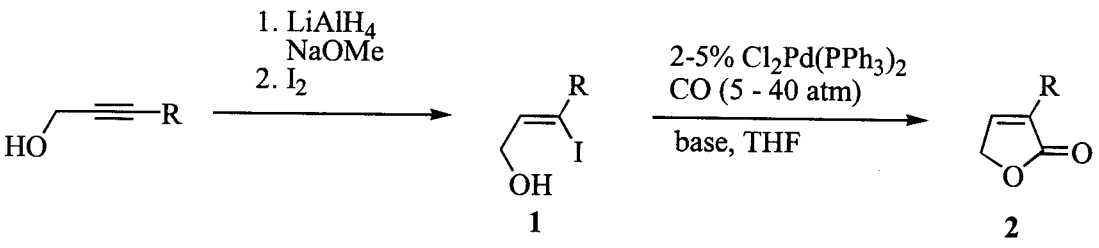


Table 1. Synthesis of α-Substituted Butenolides *via* Pd-Catalyzed Carbonylative Lactonization of (*Z*)-3-Iodo-2-propen-1-ols[a]

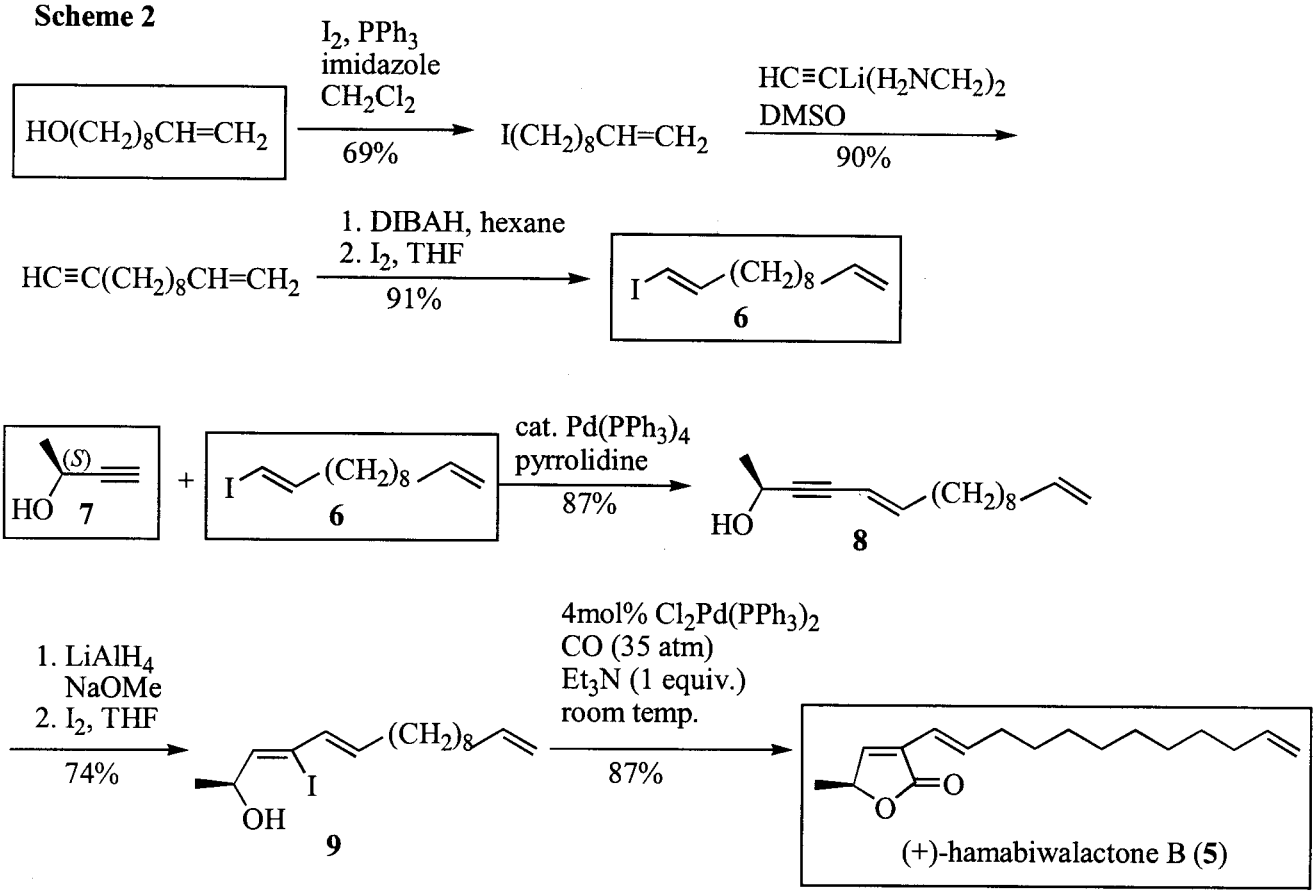


R of **1**	Method of Prep. of Starting alcohol[b]	Press. of CO (atm)	$Cl_2Pd(PPh_3)_2$ (mol %)	Base (equiv.)	Time (h)	Yield[c] (%) of **2**
$CH=CH_2$	A	5	2	K_2CO_3 (1)	24	76
(*E*)-CH=CHHex-*n*	B	5	2	NEt_3 (1)	48	73
(*E*)-$CH=CHCH_2OTBS$	B	25	4	NEt_3 (1)	72	77 (75)
$CH_2CH=CH_2$	C	5	2.5	K_2CO_3 (1)	72	67
$CH_2CH_2CH=CH_2$	D	40	5	NEt_3 (1.5)	14[d]	100
C≡CHex-*n*	E	10	3	NEt_3 (1)	120	83

[a] Unless otherwise indicated, all reactions were run in THF at room temperature.
[b] A: $BrCH=CH_2$, cat. $Cl_2Pd(PPh_3)_2$, cat. CuI, Et_2NH. B: ICH=CHR, cat. $Pd(PPh_3)_4$, pyrrolidine. C: $BrCH_2CH=CH_2$, CuI, DBU, THF, HMPA. D: Hydroxymethylation of 6-methyl-5-hepten-1-yne. E: IC≡CR, CuI, pyrrolidine. [c] By NMR. The number in parentheses is an isolated yield. [d] The lactonization reaction was preformed in DMF–MeOH at 100 °C.

To demonstrate the synthetic utility of the Pd-catalyzed carbonylative lactonization protocol herein discussed, (+)-hamabiwalactone B (**5**)[10] isolated from the roots of *Litsea japonica* (hamabiwa in Japanese) was chosen as a target. Its synthesis reported recently[3b] required (i) a five-step preparation of (+)-(*S*)-5-methyl-3-tributylstannylfuran-2(2*H*)-one and (ii) its Pd-catalyzed cross coupling with (*E*)-1-iodo-1,11-dodecadiene (**6**) the yield of the cross coupling step being 46%. As summarized in Scheme 2, our synthesis of **5** involves mere three steps from **6** and commercially available (*S*)-3-butyn-2-ol (**7**) proceeding in 56% overall isolated yield. Its spectral data are in excellent agreement with those reported in the literature,[3b,10] and the $[\alpha]_D^{25}$ value of +28.7° ($CHCl_3$, c 0.2) is very close to the value reported by Sweeney, *et al.*, supporting their correction of the originally reported value.[10]

Scheme 2



EXPERIMENTAL

General Procedures. Manipulations involving organometallics were carried out under a dry Ar atmosphere. Flash chromatographic separations were performed on 230-400 mesh silica gel 60. ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$, unless otherwise mentioned, on a 200 MHz spectrometer. NMR yields were determined by using dibromomethane or mesitylene as an internal reference. GLC analysis was carried out on a column packed with SE-30 Chromosorb W using a thermal conductivity detector. All commercially available reagents were used directly without further purification, unless otherwise indicated. THF was distilled from sodium benzophenone ketyl. Zinc bromide was flame dried under diminished pressure (< 1 mm Hg).

3-(3′-*tert*-Butyldimethylsilyloxy-1′-propenyl)-2(5*H*)-furanone (2d). Representative Procedure. To an autoclave purged with carbon monoxide was introduced a mixture of $Pd(PPh_3)_2Cl_2$ (28 mg, 0.04 mmol), Et_3N (101 mg, 1.0 mmol), (*Z*)-3-iodo-6-(*tert*-butyldimethylsilyloxy)-2,4-hexadien-1-ol (0.35 g, 1.0 mmol) in THF (2 mL). The mixture was stirred under 25 atm of CO for 72 h at 30 °C. Water (40 mL) and ether (40 mL) were then added, and the mixture was filtered. The ^{1}H NMR spectrum of the crude product indicated a 77% yield of **2d**. Flash chromatography (3/1 hexane-ethyl acetate) gave **2d** (75% isolated)as a light yellow oil: ^{1}H NMR ($CDCl_3$, Me_4Si) δ 0.12 (s, 6 H), 0.96 (s, 9 H), 4.35-4.4 (m, 2 H), 4.85 (bs, 2 H), 6.43 (bd, J = 14 Hz,

1 H), 6.98 (dt, J = 14 and 2 Hz, 1 H), 7.26 (bs, 1 H) ppm; ^{13}C NMR ($CDCl_3$, Me_4Si) δ -5.43 (2 C) 18.32, 25.83 (3 C), 62.88, 69.58, 118.72, 128.86, 136.10, 143.61, 172.29 ppm; IR (neat) 2954, 2936, 1755 cm^{-1}; MS (EI, 70 eV) m/z (relative intensity) 197 (72), 75 (100); MS (CI, 70 eV) m/z (relative intensity) 255 (M+H, 100), 123 (57); high-resolution MS calculated for $C_{13}H_{22}O_3Si$ (+H) 255.1416, found 255.1403.

3-Phenyl-2(5*H*)-furanone (2a).[5a] This compound was prepared as above using $Pd(PPh_3)_2Cl_2$ (28 mg, 0.04 mmol), anhydrous K_2CO_3 (0.27 g, 2 mmol), and (*Z*)-3-iodo-3-phenyl-2-propen-1-ol (0.52 g, 2 mmol) in THF (2 mL) and one drop of hydrazine hydrate. The mixture was stirred under 5 atm of CO for 24 h at 23-35 °C. The ^{1}H NMR spectrum of the crude product indicated a 76% yield of **2a**. The title compound was isolated and purified by flash column chromatography: ^{1}H NMR ($CDCl_3$, Me_4Si) δ 4.87 (d, J = 1.5 Hz, 2 H), 7.3-7.4 (m, 3 H), 7.58 (t, J = 1.5 Hz, 1 H), 7.75-7.8 (m, 2 H) ppm; ^{13}C NMR ($CDCl_3$, Me_4Si) δ 69.46, 126.78 (2 C), 128.50 (2 C), 129.14, 129.37, 131.24, 144.53, 172.19 ppm; IR (neat) 3100, 2924, 1742 cm^{-1}; MS (EI, 70 eV) m/z (relative intensity) 160 (M^+, 49), 132 (28), 115 (8), 103 (100), 77 (25), 51 (19); high-resolution MS calculated for $C_{10}H_8O_2$ 160.0524, found 160.0523.

3-Vinyl-2(5*H*)-furanone (2b). This compound was prepared according to the representative procedure. An autoclave was charged with a mixture of $Pd(PPh_3)_2Cl_2$ (28 mg, 0.04 mmol), anhydrous K_2CO_3 (0.27 g, 2 mmol), and (*Z*)-3-iodo-2,4-pentadien-1-ol (0.42 g, 2 mmol) in THF (2 mL), and one drop of hydrazine hydrate. The mixture was stirred under 5 atm of CO for 24 h at 23-35 °C. The ^{1}H NMR spectrum of the crude product indicated a 76% NMR yield of **2b**: ^{1}H NMR ($CDCl_3$, Me_4Si) δ 4.79 (bs, 2 H), 5.43 (dd, J = 11 and 1.5 Hz, 1 H), 6.22 (dd, J = 18 and 1.5 Hz, 1 H), 6.39 (dd, J = 18 and 11 Hz, 1 H), 7.22 (bs, 1 H) ppm. This compound was thermally unstable, and no further characterization of the compound was performed.

3-(*E*)-(1′-Octenyl)-2(5*H*)-furanone (2c). This compound was prepared according to the representative procedure. A mixture of $Pd(PPh_3)_2Cl_2$ (28 mg, 0.04 mmol), Et_3N (0.20 g, 2 mmol), and 3-iodo-(2*Z*,4*E*)-undecadien-1-ol (0.59 g, 2 mmol) in THF (2 mL) was placed in an autoclave. The mixture was stirred under 5 atm of CO for 48 h at 23-35 °C. The ^{1}H NMR spectrum of the crude product indicated a 73% yield of **2c**. The title compound was isolated and purified by flash column chromatography: ^{1}H NMR ($CDCl_3$, Me_4Si) δ 0.81 (t, J = 6.6 Hz, 3 H), 1.1-1.6 (m, 8 H), 2.05-2.15 (m, 2 H), 4.73 (bs, 2 H), 6.05 (d, J = 16.1 Hz, 1 H), 6.65-6.8 (m, 1 H), 7.07 (bs, 1 H) ppm; ^{13}C NMR ($CDCl_3$, Me_4Si) δ 14.09, 22.57, 28.70, 28.89, 31.66, 33.39, 69.66, 118.23, 129.61, 138.71, 141.98, 172.60 ppm; IR (neat) 3086, 2926, 1756 cm^{-1}; MS (EI, 70 eV) m/z (relative intensity) 194 (M^+, 45), 179 (3), 165 (12), 149 (8), 137 (16), 123 (89), 111 (28), 95 (43), 79 (100), 67 (66), 55 (43), 43 (42); high-resolution MS calculated for $C_{12}H_{18}O_2$ 194.1307, found 194.1306.

3-Allyl-2(5*H*)-furanone (2e). The compound was prepared in 67% NMR yield according to the representative procedure using $Pd(PPh_3)_2Cl_2$ (14 mg, 0.02 mmol), Et_3N (81 mg, 0.8 mmol), and (*Z*)-3-iodo-2,5-hexadien-1-ol (0.18 g, 0.8 mmol) in THF (2 mL) under 5 atm of CO for 72 h at 23-35 °C. Flash

chromatography (3/1 hexane-ethyl acetate) afforded **2e** as a light yellow oil: ^{1}H NMR ($CDCl_3$, Me_4Si) δ 2.95-3.0 (m, 2 H), 4.73 (bs, 2 H), 5.05-5.15 (m, 2 H), 5.7-5.9 (m, 1 H), 7.11 (bs, 1 H) ppm; ^{13}C NMR ($CDCl_3$, Me_4Si) δ 29.48, 70.21, 117.63, 132.50, 132.93, 145.17, 173.85 ppm; IR (neat) 3492, 3083, 1750 cm^{-1}; MS (EI, 70 eV) m/z (relative intensity) 124 (M^+, 7), 123 (40), 95 (15), 81 (19), 79 (100), 67 (61), 53 (29); high-resolution MS calculated for $C_7H_8O_2$ 124.0524, found 124.0518.

3-(1′-Octynyl)-2(5*H*)-furanone (2f). This compound was prepared in 83% NMR yield according the representative procedure using $Pd(PPh_3)_2Cl_2$ (56 mg, 0.08 mmol), Et_3N (0.40 g, 4.0 mmol), and 3-iodo-2-undecen-4-yn-1-ol (1.17 g, 4.0 mmol) in THF (4 mL). The mixture was stirred under 10 atm of CO for 120 h at 23-35 °C. Flash chromatography (3/1 hexane-ethyl acetate) afforded **2f** as a light red oil: ^{1}H NMR ($CDCl_3$, Me_4Si) δ 0.85-0.95 (t, J = 6.5 Hz, 3 H), 1.25-1.65 (m, 8 H), 2.40 (t, J = 6.5 Hz, 2 H), 4.89 (d, J = 2 Hz, 2 H), 7.45 (t, J = 2 Hz, 1 H) ppm; ^{13}C NMR ($CDCl_3$, Me_4Si) δ 14.00, 19.52, 22.46, 28.14, 28.53, 31.23, 69.76, 70.33, 98.79, 118.60, 150.11, 171.26 ppm; IR (neat) 2240, 1764 cm^{-1}; MS (EI, 70 eV) m/z (relative intensity) 177 (7), 163 (23), 147 (27), 135 (23), 131 (15), 119 (37), 105 (55), 94 (21), 91 (100), 79 (69), 65 (51), 55 (45), 43 (47), 41 (81); high-resolution MS calculated for $C_{12}H_{16}O_2$ (+H) 193.1229, found 193.1221.

3-Phenyl-5,6-dihydropyran-2-one (4). This compound was prepared as above using $Pd(PPh_3)_2Cl_2$ (56 mg, 0.08 mmol), Et_3N (0.40 g, 4.0 mmol), (*Z*)-4-iodo-4-phenyl-3-buten-1-ol (0.55 g, 2.0 mmol) in DMF (2 mL) in 75% NMR yield. The mixture was stirred under 40 atm of CO for 12 h at 100 °C. Water (40 mL) and ether (40 mL) were then added, and the mixture was filtered. Flash chromatography (3/1 hexane-ethyl acetate) afforded **4** as a light yellow oil: ^{1}H NMR ($CDCl_3$, Me_4Si) δ 3.7-3.8 (m, 2 H), 4.45 (t, J = 6 Hz, 2 H), 6.97 (t, J = 5 Hz, 1 H), 7.25-7.5 (m, 5 H) ppm; ^{13}C NMR ($CDCl_3$, Me_4Si) δ 24.68, 66.31, 128.10 (2 C), 128.15 (2 C), 128.40, 135.38, 137.14, 141.35, 163.83 ppm; IR (neat) 1707, 1045 cm^{-1}; MS (EI, 70 eV) m/z (relative intensity) 174 (13), 148 (31), 129 (4), 115 (100), 102 (5), 91 (25), 77 (4), 51 (11); high-resolution MS calculated for $C_{11}H_{10}O_2$ 174.0681, found 174.0685.

10-Iodo-1-decene. To a suspension of iodine (30.5 g, 0.12 mol), imidazole (8.2 g, 0.12 mol), and Ph_3P (31.9 g, 0.12 mol) in CH_2Cl_2 (300 mL) was added 9-decen-1-ol (15.6 g, 0.10 mol). The reaction mixture was stirred overnight. Pentane (200 mL) was then added to the reaction mixture. The white precipitate was filtered. Flash chromatography (hexane) afforded the title compound (18 g, 69%) as a colorless oil: ^{1}H NMR ($CDCl_3$, Me_4Si) δ 1.2-1.4 (m, 12 H), 1.75-1.9 (m, 2 H), 2.0-2.1 (m, 2 H), 4.9-5.05 (m, 2 H), 5.7-5.9 (m, 1 H) ppm; ^{13}C NMR ($CDCl_3$, Me_4Si) δ 22.61, 28.44, 28.81, 28.95, 29.21, 30.44, 33.49, 33.72, 114.13, 138.91 ppm.

1-Dodecen-11-yne. To a suspension of lithium acetylide-ethylenediamine complex (9.2 g, 100 mmol) in DMSO (50 mL) was added at 0 °C 10-iodo-1-decene (17.5 g, 66 mmol). The reaction mixture was stirred for 6 h. The completion of the reaction was indicated by GLC. Water was added to the reaction mixture which was then filtered and extracted with ether. The organic layer was concentrated. The title product (9.7

g, 90%) was obtained by Kugelrohr distillation (59 °C at 2 mmHg); ^{1}H NMR ($CDCl_3$, Me_4Si) δ 1.3-1.5 (m, 12 H), 1.91 (t, J = 3 Hz, 1 H), 2.0-2.15 (m, 2 H), 2.15-2.2 (m, 2 H), 4.9-5.05 (m, 2 H), 5.7-5.9 (m, 1 H) ppm; ^{13}C NMR ($CDCl_3$, Me_4Si) δ 18.31, 28.43, 28.68, 28.85, 29.02 (2 C), 29.30, 33.74, 68.02, 84.38, 114.07, 138.91 ppm.

(*E*)-1-Iodo-1,11-dodecadiene (6). 1-Dodecen-11-yne (6.3 g, 38 mmol) was treated with DIBAH (6.9 mL, 38 mmol) and dry hexane (50 mL). The reaction mixture was stirred overnight at 23 °C. A small sample was prepared and checked by GLC that indicated a 100% conversion to the corresponding alkenylalane. Hexane was removed *in vacuo*, and THF (20 mL) was added to the reaction mixture. The reaction mixture was then cooled to -78 °C, and I_2 (9.75 g, 38 mmol) in THF (40 mL) was added. The reaction mixture was sequentially treated with ether (120 mL), 1 N HCl (60 mL), saturated $Na_2S_2O_3$ (2 x 60 mL), and brine. The product (10.1 g, 91%) was obtained by concentration without further purification: ^{1}H NMR ($CDCl_3$, Me_4Si) δ 1.2-1.4 (m, 12 H), 1.8-1.9 (m, 2 H), 1.95-2.1 (m, 2 H), 4.9-5.05 (m, 2 H), 5.7-6.0 (m, 2 H), 6.4-6.55 (m, 1 H) ppm; ^{13}C NMR ($CDCl_3$, Me_4Si) δ 25.44, 28.19, 28.74, 28.92, 29.15, 29.21, 33.63, 35.89, 74.23, 114.00, 138.95, 146.44 ppm.

(*S*)-5,15-Hexadeca-1,11-dien-13-yn-2-ol (8). A mixture of 1-iodo-1*E*,11-dodecadiene (5.0 g, 17 mmol), (*S*)-3-butyn-2-ol (1.0 g, 14 mmol), $Pd(PPh_3)_4$ (0.81 g, 0.7 mmol) and pyrrolidine (20 mL) was stirred at 23 °C for 4 h. The reaction mixture was sequentially treated with 1N HCl (50 mL), ether (3 x 50 mL), saturated $NaHCO_3$ (50 mL), and the organic layer was dried over $MgSO_4$. After removal of the solvent, flash chromatography (3/1 hexane-ethyl acetate) afforded 2.85 g (87%) of **8** as a light yellow oil: ^{1}H NMR ($CDCl_3$, Me_4Si) δ 1.2-1.5 (m, 15 H), 2.0-2.15 (m, 4 H), 2.72 (bs, 1 H), 4.55-4.7 (m, 1 H), 4.9-5.05 (m, 2 H), 5.4-5.55 (m, 1 H), 5.7-5.9 (m, 1 H), 6.05-6.2 (m, 1 H) ppm; ^{13}C NMR ($CDCl_3$, Me_4Si) δ 24.22, 28.54, 28.76, 28.92, 28.96, 29.25, 32.90, 33.65, 58.45, 82.47, 89.46, 108.78, 114.02, 138.95, 145.03 ppm; IR (neat) 3339, 3076, 2926, 1640 cm^{-1}; MS (EI, 70 eV) m/z (relative intensity) 219 (2), 149 (2), 135 (10), 109 (16), 95 (66), 81 (49), 67 (54), 55 (100); high-resolution MS calculated for $C_{16}H_{26}O$ 234.1984, found 234.1981.

(*S*)-2-Hydroxy-4-iodo-3*Z*,5*E*,15-hexadecatriene (9). To a suspension of NaOMe (1.34 g, 29 mmol) in 13 mL of $LiAlH_4$ (1.0 M in THF, 13 mmol) at 0 °C was added (*S*)-2-hydroxy-5*E*,15-hexadecadien-3-yne (3.04 g, 13 mmol) in THF (5 mL), and the mixture was stirred at 0 °C for 6 h. To this was added 5 mL of EtOAc. The resulting mixture was cooled to -78 °C, and I_2 (6.3 g, 25 mmol) in THF (10 mL) was added. The reaction mixture was then warmed to 0 °C and worked up with 3N HCl. Flash chromatography (1/1 hexane-ethyl acetate) afforded 3.48 g (74%) of **9** as a light red oil: ^{1}H NMR ($CDCl_3$, Me_4Si) δ 1.1-1.5 (m, 15 H), 1.8-2.2 (m, 4 H), 2.60 (bs, 1 H), 4.6-4.7 (m, 1 H), 4.9-5.05 (m, 2 H), 5.7-6.1 (m, 4 H) ppm; ^{13}C NMR ($CDCl_3$, Me_4Si) δ 21.92, 28.57, 28.81, 29.01, 29.10, 29.30, 31.88, 32.96, 33.71, 72.25, 105.54, 114.08, 131.08, 139.03, 140.21,140.35 ppm; IR (neat) 3334, 3076, 2925, 1640 cm^{-1}.

(+)-Hamabiwalactone B (5). To an autoclave purged with carbon monoxide, a mixture of $Pd(PPh_3)_2Cl_2$ (56 mg, 0.08 mmol), Et_3N (0.20 g, 2.0 mmol), (*S*)-2-hydroxy-4-iodo-3*Z*,5*E*,15-hexadecatriene (0.72 g, 2.0 mmol) in THF (2 mL) was added. The mixture was stirred under 35 atm of CO for 96 h at 28 °C. Water (40 mL) and ether 40 mL) were then added, and the mixture was filtered. Analysis by ^1H NMR spectroscopy of the crude product using an internal standard indicated an 87% yield of **5**. Flash chromatography (3/1 hexane-ethyl acetate) afforded the title compound[10] as a light yellow oil: ^1H NMR ($CDCl_3$, Me_4Si) δ 1.2-1.5 (m, 15 H), 2.0-2.2 (m, 4 H), 4.9-5.1 (m, 3 H), 5.7-5.9 (m, 1 H), 6.05-6.15 (m, 1 H), 6.7-6.85 (m, 1 H), 7.07 (bs, 1 H) ppm; ^{13}C NMR ($CDCl_3$, Me_4Si) δ 18.93, 28.45, 28.54, 28.67, 28.88, 28.98, 29.17, 33.18, 33.57, 76.74, 113.91, 118.17, 129.01, 138.36, 138.85, 146.92, 171.82 ppm; IR (neat) 3077, 2979, 2927, 2855, 1758, 1084 cm^{-1}; MS (EI, 70 eV), m/z (relative intensity) 161 (1), 135 (4), 107 (5), 95 (21), 67 (20), 55 (39), 43 (100); high-resolution MS calculated for $C_{17}H_{26}O_2$ 262.1933, found 262.1928 $[\alpha]_D^{25}$ +28.7° ($CHCl_3$, c 0.2).

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