Barbier Reaction in the Regime of Metal-Oxide: Carbonyl Allylation over β -SnO/Cu₂O and Surface Diagnostics

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Spectral Characteristics of Homoallylic Alcohols

Entry 1: 1-(4-chlorophenyl)-2-methylbut-3-en-1-ol (*syn:anti*=50:50) [*lit. ref:* 1]:

^1H NMR (200 MHz; CDCl_3): δ (*anti* isomer) 0.88 (d, 3H, J = 5.0 Hz, Me), 2.19 (br s, 1H, -OH), 2.35-2.60 (m, 1H, CHMe), 4.33 (m, 1H, CHOH), 5.00-5.24 (m, 2H, $=\text{CH}_2$), 5.65-5.77 (m, 1H, $=\text{CH}$), 7.22-7.34 (m, 4H, Ar); δ (*syn* isomer) 0.99 (d, 3H, J = 5.0 Hz, Me), 1.92 (br s, 1H, -OH), 2.35-2.60 (m, 1H, CHMe), 4.59 (m, 1H, CHOH), 5.00-5.24 (m, 2H, $=\text{CH}_2$), 5.65-5.77 (m, 1H, $=\text{CH}$), 7.22-7.34 (m, 4H, Ar); ^{13}C NMR (CDCl_3): δ 14.02, 16.38 (*anti+syn*) [Me], 44.77, 46.24 (*anti+syn*) [CMe], 76.58, 77.13 (*anti+syn*) [COH], 115.82, 117.00 (*anti+syn*) [$=\text{CH}_2$], 127.21, 127.87, 128.16, 128.21, 128.35, 133.11, 133.39 (*anti+syn*) [Ar], 140.07, 140.15 (*anti+syn*) [$=\text{CH}$]; EIMS m/z (rel abundance): 196 (M^+ , <1), 178 [$(\text{M}-\text{H}_2\text{O})^+$, <1], 163 (<1), 143 (31), 141 (100), 113(14), 77 (41), 55 (8).

Entry 2: 2-Methyl-1-phenylbut-3-en-1-ol (*syn:anti*=19:81) [*lit. ref:* 2]:

^1H NMR (200 MHz; CDCl_3): δ (*anti* isomer) 0.87 (d, 3H, J = 6.8 Hz, Me), 2.17 (br s, 1H, -OH), 2.43-2.54 (m, 1H, CHMe), 4.38 (d, 1H, J =5.4 Hz, CHOH), 5.16-5.26 (m, 2H, $=\text{CH}_2$), 5.71-5.91 (m, 1H, $=\text{CH}$), 7.26-7.39 (m, 5H, Ar); δ (*syn* isomer) 0.99 (d, 3H, J = 6.8 Hz, Me), 1.62 (br s, 1H, -OH) 2.43-2.54 (m, 1H, CHMe), 4.63 (d, 1H, J =5.4 Hz, CHOH), 5.16-5.26 (m, 2H, $=\text{CH}_2$), 5.71-5.91 (m, 1H, $=\text{CH}$), 7.26-7.39 (m, 5H, Ar); EIMS m/z (rel abundance): 162 (M^+ , <1), 144 [$(\text{M}-\text{H}_2\text{O})^+$, (2)], 129 (3), 107 (100), 79 (21), 56 (3).

Entry 3: 3-Methyldec-1-en-4-ol (*syn:anti*=49:51) [*lit. ref.* 3]:

^1H NMR (200 MHz; CDCl_3): δ (*anti* isomer) 0.87 (t, 3H, J = 6.2 Hz, Me), 0.99 (d, 3H, J = 2.05 Hz, Me), 1.27-1.48 (m, 10H, 5 CH_2), 2.16-2.26 (m, 1H, CHMe), 3.36-3.45 (m, 1H, CHOH), 5.01-5.12 (m, 2H, $=\text{CH}_2$), 5.68-5.82 (m, 1H, $=\text{CH}$); δ (*syn* isomer) 0.87 (t, 3H, J = 6.2 Hz, Me), 1.03 (d, 3H, J = 2.05 Hz, Me), 1.27-1.48 (m, 10H, 5 CH_2), 2.16-2.26 (m, 1H, CHMe), 3.36-3.45 (m, 1H, CHOH), 5.01-5.12 (m, 2H, $=\text{CH}_2$), 5.68-5.82 (m, 1H, $=\text{CH}$); ^{13}C NMR (CDCl_3): δ 14.11, 14.15 (*anti+syn*) [2Me], 22.68 [CH_2], 25.76, 26.10 (*anti+syn*) [CH_2], 29.42, 29.45 (*anti+syn*) [CH_2], 31.91 [CH_2], 34.10, 34.32 (*anti+syn*) [CH_2], 43.52, 44.19 (*anti+syn*) [CMe], 74.67 [COH], 115.19, 116.22 (*anti+syn*) [$=\text{CH}_2$], 140.38, 141.15 (*anti+syn*) [$=\text{CH}$].

Entry 4: 3-Methyltridec-1-en-4-ol (*syn:anti*=50:50):

^1H NMR (200 MHz; CDCl_3): δ (*anti* isomer) 0.88 (m, 3H, Me), 1.01 (d, 3H, J = 2.1 Hz, Me), 1.25-1.35 (m, 16H, 8 CH_2), 2.13-2.27 (m, 1H, CHMe), 3.67 (m, 1H, CHOH), 5.01-5.12 (m, 2H, $=\text{CH}_2$), 5.65-5.86 (m, 1H, $=\text{CH}$); δ (*syn* isomer) 0.88 (m, 3H, Me), 1.04 (d, 3H, J = 2.1 Hz, Me), 1.25-1.35 (m, 16H, 8 CH_2), 2.13-2.27 (m, 1H, CHMe), 3.67 (m, 1H, CHOH), 5.01-5.12 (m, 2H, $=\text{CH}_2$), 5.65-5.86 (m, 1H, $=\text{CH}$); ^{13}C NMR (CDCl_3): δ 14.14, 16.31 (*anti+syn*) [2Me], 22.71, 25.81, 26.13, 28.98, 29.64, 31.95, 34.12, 34.31 [8 CH_2], 43.54, 44.14 (*anti+syn*) [CMe], 74.68 [COH], 115.08, 116.09 (*anti+syn*) [$=\text{CH}_2$], 140.39, 141.19 (*anti+syn*) [$=\text{CH}$]; EIMS m/z (rel abundance): 212 (M^+ , 14), 194 [$(\text{M}-\text{H}_2\text{O})^+$, 12], 157 (20), 97 (52), 83 (84), 69 (70), 56 (100).

Entry 5: 3-Methyltrideca-1,5-dien-4-ol (mixture of 2 diastereomers) [*lit. ref:* 4]:

^1H NMR (200 MHz; CDCl_3): δ (*anti* isomer) 0.87 (t, 3H, J = 6.1 Hz, Me), 0.99 (d, 3H, J = 6.1 Hz, Me), 1.16-1.36 (m, 10H, 5CH_2), 2.02-2.05 (m, 2H, CH_2), 2.16-2.27 (m, 1H, CHMe), 3.74-3.81 (m, 1H, CHOH), 5.03-5.17 (m, 2H, $=\text{CH}_2$), 5.38-5.48 (m, 1H, $=\text{CH}$), 5.56-5.79 (m, 2H, $\text{HC}=\text{CH}$); δ (*syn* isomer) 0.87 (t, 3H, J = 6.1 Hz, Me), 1.09 (d, 3H, J = 6.1 Hz, Me), 1.16-1.36 (m, 10H, 5CH_2), 2.02-2.05 (m, 2H, CH_2), 2.16-2.27 (m, 1H, CHMe), 3.94 (m, 1H, CHOH), 5.03-5.17 (m, 2H, $=\text{CH}_2$), 5.38-5.48 (m, 1H, $=\text{CH}$), 5.56-5.79 (m, 2H, $\text{HC}=\text{CH}$); ^{13}C NMR (CDCl_3): δ 14.09, 14.87 (*anti+syn*) [2Me], 16.09, 22.66, 29.14, 29.20, 31.84, 32.29, 43.68, 44.56 (*anti+syn*) [CMe], 75.96, 76.28 (*anti+syn*) [COH], 115.51, 116.22 (*anti+syn*) [$=\text{CH}_2$], 130.21, 130.56 (*anti+syn*) [$\text{C}=\text{C}$], 133.16, 133.76 (*anti+syn*) [$\text{C}=\text{C}$], 140.19, 140.62 (*anti+syn*) [$=\text{CH}$].

Entry 6: 2-Methyl-1-thiophenylbut-3-en-1-ol (*syn:anti*=34:66) [*lit. ref:* 5]:

^1H NMR (200 MHz; CDCl_3): δ (*anti* isomer) 0.97 (d, 3H, J = 6.8 Hz, Me), 2.40-2.62 (m, 1H, CHMe), 4.61-4.65 (m, 1H, CHOH), 5.03-5.26 (m, 2H, $=\text{CH}_2$), 5.77-5.91 (m, 1H, $=\text{CH}$), 6.91-6.97 (m, 2H, thiophene-H), 7.20-7.26 (m, 1H, thiophene-H); δ (*syn* isomer) 1.09 (d, 3H, J = 6.8 Hz, Me), 2.40-2.62 (m, 1H, CHMe), 4.77-4.80 (m, 1H, CHOH), 5.03-5.26 (m, 2H, $=\text{CH}_2$), 5.77-5.91 (m, 1H, $=\text{CH}$), 6.91-6.97 (m, 2H, thiophene-H), 7.20-7.26 (m, 1H, thiophene-H); ^{13}C NMR (CDCl_3): δ 14.78, 16.42 (*anti+syn*) [Me], 45.05, 45.53 (*anti+syn*) [CMe], 73.74, 73.82 (*anti+syn*) [COH], 115.90, 117.06 (*anti+syn*) [$=\text{CH}_2$], 124.19, 124.33 (*anti+syn*) [thiophene-C], 124.63 [thiophene-C], 126.32, 126.35 (*anti+syn*) [thiophene-C], 139.69, 140.05 (*anti+syn*) [$=\text{CH}$], 146.41, 146.63 (*anti+syn*) [thiophene-C].

Entry 7: 3,9-Dimethyl-undeca-1,10-diene-4,8-diol (mixture of 2 diastereomers):

^1H NMR (200 MHz; CDCl_3): δ (*anti* isomer) 0.99 (d, 6H, $J = 6.6$ Hz, 2Me), 1.35-1.57 (m, 6H, 3 CH_2), 1.92 (br s, 2H, 2-OH), 2.14-2.25 (m, 2H, 2 CHMe), 3.37-3.50 (m, 2H, 2 CHOH), 5.01-5.11 (m, 4H, 2= CH_2), 5.65-5.86 (m, 2H, 2=CH); ^1H NMR (CDCl_3): δ (*syn* isomer) 1.03 (d, 6H, $J = 6.6$ Hz, 2Me), 1.35-1.57 (m, 6H, 3 CH_2), 1.87 (br s, 2H, 2-OH), 2.14-2.25 (m, 2H, 2 CHMe), 3.37-3.50 (m, 2H, 2 CHOH), 5.01-5.11 (m, 4H, 2= CH_2), 5.65-5.86 (m, 2H, 2=CH); ^{13}C NMR (CDCl_3): δ 14.27, 14.37 (*anti+syn*) [Me], 16.17, 16.20 (*anti+syn*) [Me], 21.92, 22.05 (*anti+syn*) [CH_2], 33.71, 33.79 (*anti+syn*) [CH_2], 33.93, 34.05 (*anti+syn*) [CH_2], 43.53, 43.65 (*anti+syn*) [CHMe], 44.05, 44.17 (*anti+syn*) [CHMe], 74.50, 74.60 (*anti+syn*) [2 CHOH], 115.09, 116.09 (*anti+syn*) [2= CH_2], 140.29, 140.98 (*anti+syn*) [2=CH]; EIMS m/z (rel abundance): 211 [(M^+-H) , <1], 157 (18), 139 (44), 121 (60), 95 (72), 69 (68), 55 (100), 43 (62).

Entry 8: 2,2-Dimethyl-1-thiophenylbut-3-en-1-ol [*lit. ref:* 6]:

^1H NMR (200 MHz; CDCl_3): δ 1.04 (s, 3H, Me), 1.08 (s, 3H, Me), 2.16 (br s, 1H, -OH), 4.71 (s, 1H, CHOH), 5.08-5.20 (m, 2H, = CH_2), 5.90-6.04 (m, 1H, =CH), 6.93-6.98 (m, 2H, thiophene-H), 7.19-7.26 (m, 1H, thiophene-H); ^{13}C NMR (CDCl_3): δ 21.58 [Me], 24.24 [Me], 42.22 [CMe_2], 77.44 [COH], 114.26 [=CH $_2$], 124.34, 125.41, 125.99, 128.24 [4 thiophene-C], 144.49 [=CH]; EIMS m/z (rel abundance): 165 [$(\text{M}-\text{H}_2\text{O})^+$, (9)], 113 (100), 85 (24), 41 (21).

Entry 9: 1-Phenyl-3-propylpent-4-en-2-ol (3f): (mixture of 2 diastereomers):

^1H NMR (200 MHz; CDCl_3): δ 0.89 (m, 3H, Me), 1.29-1.48 (m, 4H, 2 CH_2), 1.63 (br s, 1H, -OH), 2.0-2.23 (m, 1H, CHC_3H_7), 2.46-2.90 (m, 2H, PhCH_2), 3.70-3.77 (m, 1H, CHOH), 5.05-5.23 (m, 2H, $=\text{CH}_2$), 5.60-5.79 (m, 1H, $=\text{CH}$), 7.24-7.36 (m, 5H, Ph); ^{13}C NMR (CDCl_3): δ 14.04, 14.11 (*anti+syn*) [Me], 20.34, 20.41 (*anti+syn*) [CH_2], 32.23, 33.25 (*anti+syn*) [CH_2], 40.94, 41.42 (*anti+syn*) [PhCH_2], 49.43, 50.06 (*anti+syn*) [CC_3H_7], 74.79, 75.25 (*anti+syn*) [COH], 117.05, 117.63 (*anti+syn*) [$=\text{CH}_2$], 126.30, 126.36 (*anti+syn*) [Ph], 128.33, 128.37 (*anti+syn*) [Ph], 129.37 [Ph], 138.51 [Ph], 139.01, 139.22 (*anti+syn*) [$=\text{CH}$]; EIMS m/z (rel abundance): 205 (M^+ , 6), 186 [$(\text{M}-\text{H}_2\text{O})^+$, 20], 143 (50), 121 (78), 92 (100), 91 (82), 84 (38), 77 (14), 55 (26).

Entry 10: 2-Hydroxy-1,2-diphenylpent-4-en-1-one [*lit. ref:* 6]:

^1H NMR (200 MHz; CDCl_3): δ 1.55 (s, 1H, -OH), 2.89-3.19 (m, 2H, CH_2), 4.97-5.14 (m, 2H, $=\text{CH}_2$), 5.63-5.84 (m, 1H, $=\text{CH}$), 7.26-7.75 (m, 10H, 2Ph); ^{13}C NMR (CDCl_3): δ 44.06 [CH_2], 81.39 [COH], 120.38 [$=\text{CH}_2$], 125.62, 128.06, 128.09, 128.88, 130.17, 132.37, 132.70, 134.61 [8Ph-C], 141.82 [$=\text{CH}$], 200.68 [CO]; EIMS m/z (rel abundance): 252 (M^+ , 4), 234 [$(\text{M}-\text{H}_2\text{O})^+$, 14], 211 (52), 147 (54), 105 (100), 77 (62), 51 (12).

Entry 11: 3-Hydroxy-3-methyl-5-hexenoic acid ethyl ester [lit. ref: 2]:

^1H NMR (200 MHz; CDCl_3): δ 1.23-1.37 (m, 6H, 2Me), 2.26 (m, 2H, CH_2) 2.42 (m, 2H, CH_2), 3.61 (br s, 1H, -OH), 4.18 (m, 2H, OCH_2), 5.02-5.12 (m, 2H, $=\text{CH}_2$), 5.73-5.90 (m, 1H, $=\text{CH}$); ^{13}C NMR (CDCl_3): δ 14.09 [Me], 26.75 [Me], 44.23 [CH_2], 46.43 [CH_2], 60.45 [OCH_2], 70.57 [COH], 118.40 [$=\text{CH}_2$], 133.61 [$=\text{CH}$], 172.75 [CO]; EIMS m/z (rel abundance): 172 (<1), 131 (76), 127 (12), 118 (20), 103 (20), 85 (100), 82 (48), 70 (16), 55 (22), 50 (42).

Entry 12: 4-Hydroxy-4-phenylhept-6-en-2-one:

^1H NMR (200 MHz; CDCl_3): δ 1.29 (s, 3H, Me), 2.36-2.39 (m, 2H, CH_2), 3.07 (d, 2H, $J = 7.9$ Hz, CH_2), 4.17 (br s, 1H, -OH), 4.99-5.12 (m, 2H, $=\text{CH}_2$), 5.77-5.94 (m, 1H, $=\text{CH}$), 7.35-7.96 (m, 5H, Ph); ^{13}C NMR (CDCl_3): δ 27.29 [Me], 46.51 [CH_2], 46.86 [CH_2], 71.82 [COH], 118.46 [$=\text{CH}_2$], 124.87, 128.12, 128.73, 133.64 [4 Ph-C], 134.11 [$=\text{CH}$], 201.87 [CO]; EIMS m/z (rel abundance): 205 (M^+ , 2), 163 (28), 105 (100), 77 (24), 43 (26).

Entry 13: 3-Allyl-3-hydroxy-3H-isobenzofuran-1-one:

^1H NMR (200 MHz; CDCl_3): δ 4.81-4.84 (m, 2H, CH_2), 5.26-5.44 (m, 2H, $=\text{CH}_2$), 5.92-6.09 (m, 1H, $=\text{CH}$), 6.56 (br s, 1H, -OH), 7.56-7.65 (m, 2H, Ph-H), 7.69-7.74 (m, 1H, Ph-H), 7.89-7.94 (m, 1H, Ph-H); ^{13}C NMR (CDCl_3): δ 66.43 [CH_2], 76.89 [COH], 118.93 [$=\text{CH}_2$], 128.72, 129.74, 130.14, 130.71, 131.52, 131.97 [6Ph-C], 133.22 [$=\text{CH}$], 167.49 [CO]; EIMS m/z (rel abundance): 190 (M^+ , 4), 188 (16), 149 (100), 132 (8), 122 (14), 104 (20), 93 (8), 76 (12), 65 (14), 57 (8), 50 (8).

Entry 14: 1-Allylcyclohexanol (3q) [*lit. ref:* 7]:

^1H NMR (200 MHz; CDCl_3): δ 1.36-1.63 (m, 10H, 5 CH_2), 2.17-2.22 (m, 2H, CH_2), 5.04-5.16 (m, 2H, $=\text{CH}_2$), 5.76-5.97 (m, 1H, $=\text{CH}$); ^{13}C NMR (CDCl_3): δ 22.06, 25.71, 37.32 [3 cyclohexyl-C], 46.74 [CH_2], 70.74 [COH], 118.55 [$=\text{CH}_2$], 133.58 [$=\text{CH}$]; EIMS m/z (rel abundance): 139 [$(\text{M}^+ - \text{H})$, 22), 121 (18), 97 (44), 83 (76), 56 (100), 43 (76).

Notes on XPS Analysis

Narrow scan XPS analyses were performed as described in main text. The binding energy values were compared to those in literature (Table T1 and T2).

Table T1. Selected B.E. (eV) of Sn (3d_{5/2}), Cu (2p_{3/2}), O (1s) and C (1s).

#	Sample	Sn (3d _{5/2})	Cu (2p _{3/2})	O (1s)	C (1s)	Ref
1	SnO	486.1		530.0		8
2	SnO ₂	486.4				8
3	[Me ₂ SnFe(CO) ₄] ₂	486.9				9
4	[Ph ₂ SnFe(CO) ₄] ₂	486.8				9
5	Bu ₄ Sn	486.2				8
6	Ph ₄ Sn	486.5				10
7	Ph ₃ SnCl	487.2				10
8	(PhCH ₂) ₃ SnCl	487.1				10
9	(PhCH ₂) ₂ SnCl ₂	487.8				10
10	Cu ₂ O		932.5	530.4		8, 11
11	CuO		933.7	529.6		11, 12
12	Cu(OH) ₂		934.7	531.2		11, 12
13	CuCl		932.4			8
14	CuCN		932.9			8
15	THF			535.5	287.2	13

Table T2. XPS data B.E. (eV) of β-SnO/Cu₂O [S_{UTR} and S_{TR}]

Entry	Element	S _{UTR}	Corr. Coeff.	S _{TR}	Corr. Coeff.
1	Sn (3d _{5/2})	486.2	0.98292	486.3, 487.3	0.99131
2	Cu (2p _{3/2})	932.3, 933.5 ^a , 934.6 ^b	0.99689	930.8, 932.5	0.096835
3	O (1s)	529.7 ^c , 530.0, 530.9, 531.3 ^c	0.99656	528.9, 530.0, 530.9, 532.0, 535.5, 537.6	0.99433
4	C (1s)	284.4, 287.7	0.97923	281.9, 284.4, 283.6, 287.5	0.99218

^a characterized as Cu(OH)₂, lit. 934.7 eV; ^b characterized as CuO, lit. 933.7 eV.; ^c signal 529.7 (CuO), and 531.3 [Cu(OH)₂] may arise due to partial oxidation of Cu₂O during sample work-up.

Sn (3d_{5/2}): The binding energy of electron emitted from 3d_{5/2} level in tin is very informative regarding the chemical environment of the atom, and increases on going from +II to +IV oxidation state (Table

T1, entry 1 and 2). For any given structural class of organotin(IV), the B.E. of Sn 3d_{5/2} is nearly invariant to organic group modification, provided other ligands remain the same, as shown for [Me₂SnFe(CO)₄]₂ versus [Ph₂SnFe(CO)₄]₂ (entry 3 and 4), and Bu₄Sn versus Ph₄Sn (entry 5 and 6). Moreover, successive replacement of an organic group by halogen in organotin(IV) causes progressive decrease in B.E. This is evident from the B.E. of Ph₄Sn, Ph₃SnCl, (PhCH₂)₃SnCl, and (PhCH₂)₂SnCl₂ (entry 6, 7, 8 and 9).

O (1s): The O(1s) signals at 530.0 and 530.9 in both **S_{TR}** and **S_{UTR}** are assigned to SnO and Cu₂O respectively (Table T2, entry 3). In sample **S_{TR}** the signal at 535.5 is characterized due to THF. The signal at 532.0 is assigned due to the proposed tin(IV) species, while the signal at 528.9 could be due to the copper(I) intermediate mentioned in main text. The signal at 537.6 remains uncharacterized.

C(1s): There are very few XPS data available for organotin compounds in C(1s) region, posing difficulty in comparative analyses. The signal at 287.5 in **S_{TR}** and 287.7 in **S_{UTR}** are assigned to coordinated THF. The new signal in **S_{TR}** at 281.9 is in the metal-carbide region. The other signal at 283.6 might be due to the proposed organotin(IV) species mentioned in main text.

XRD SPECTRA

XRD of β -SnO/ Cu_2O before (S_{UTR}) and after (S_{TR}) allyl halide treatment.

($\square$)= peaks in β -SnO; ($\circ$)= peaks in Cu_2O ; (Δ) = new peaks in S_{TR} .

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