Table II. ^{13}C NMR Data for 8[a]

δ	J_{C-H} (mult)	$J_{long-range}$ (mult)	assignment[b]
23.3	129.5 (q)	4.5 (q)	n
27.2	129.5 (q)	5.2 (q)	n
48.0		<2 (m)	o
53.8		<2 (m)	o
88.8	164.5 (d)		p
110.5	162.3 (d)	8.2 (d)	q
121.2	158.5 (d)	7.4 (d)	r
122.3	158.5 (d)	?	r
122.5	158.5 (d)	?	r
122.7	158.5 (d)	?	r
127.5	160.8 (d)	?	r
127.7	160.8 (d)	?	r
128.0	160.8 (d)	?	r
138.4		? (m)	s
142.2		6 & 6 & ? (m) d of d of d	t
148.6		? (m)	s
152.8		? (m)	s
174.6		1.5 & ~2 (m) d of hep	u
182.6		~2 (m) heptet	v
184.5		1.5 (d)	w

[a] $CDCl_3$ solution. [b] See structure 8.

selective methyl decoupling experiment, which is consistent with a cis relationship between C_v and the vinylic proton.[5] The sizable downfield shift of the N–H due to hydrogen bonding and the NOE between the N–H and carbonyl carbon are also supportive of the cis conformation. Spectra of model compounds[10] suggest that C_q absorbs at δ 110 and the proton at δ 7.8 is ortho to the imino nitrogen.

A possible mechanism for the formation of 8 is shown in Scheme I. Oxidation of the 2-methyl in 1 to an aldehyde is similar to the photooxidation of methylenes observed by Kanaoka et al.; however, they did not observe the oxidation of 2-methyl-3*H*-indoles under their conditions.[8a,b] Aldehydes have been formed from 2-methylindoles in this way.[8c,d] The aldehyde undergoes an aldol-like condensation with another molecule of 1, and the dimer is oxidized to the fully conjugated system, which rearranges to the ketone 8.

Experimental Section

^1H NMR and ^{13}C NMR spectra were taken on Bruker 200-MHz Supercon (FT) and Bruker UM 360-MHz spectrometers in $CDCl_3$ solution, using Me_4Si as an internal standard. Mass spectra were determined on an AEI MS 902 instrument.

12,12,12a,14,14-Pentamethyl-6*H*,12*H*,12a*H*,14*H*-pyrimido[1,6-*a*:3,4-*a*']diindole (6). A mixture of methylene iodide (25 g) and 1 (30 g) was stirred with pulverized KOH (16 g) for 18 h at 80 °C under nitrogen. The KOH was filtered off and the reaction mixture chromatographed on silica gel (CH_2Cl_2 solvent). Compound 6 was obtained in the early fractions, while 1 (5 g impure) was found in the last fraction that was eluted with 1–2% methanol. Compound 6 was recrystallized from ethanol: 3.42 g (11%); mp 124–127 °C; mass spectrum, *m/e* (relative intensity) 330 (29), 315 (100), 300 (6), 285 (15); UV (95% EtOH) 280 nm (ε 23 000), 244 (12 000), 206 (36 000); IR (CCl_4 solvent) 1605 cm^{-1}; ^1H NMR ($CDCl_3$) δ 7.24 (t of d, *J* = 8, 1.3 Hz, 1 H), 7.11 (d of t, *J* = 8, 1.3 Hz, 2), 7.08 (t of d, *J* = 8, 1.3 Hz, 1), 6.86 (t of d, *J* = 8, 0.9 Hz, 1), 6.84 (t of d, *J* = 8, 0.9 Hz, 1), 6.73 (d of t, *J* = 8, 0.9 Hz, 1), 6.71 (d of t, *J* = 8, 0.9 Hz, 1), 5.36 (d, *J* = 12 Hz, 1), 4.71 (d, *J* = 12 Hz, 1), 4.71 (s, 1), 1.51 (s, 3), 1.49 (s, 3), 1.38 (s, 3), 1.30 (s, 3), 1.07 (s, 3); see Table I for ^{13}C NMR data; exact mass calcd for $C_{23}H_{26}N_2$ 330.2096, found 330.2084 (M^+).

(3,3-Dimethyl-2-indolinylidene)methyl 3,3-Dimethyl-3*H*-indol-2-yl Ketone (8). Oxygen was bubbled into a suspension of K_2CO_3 (3.0 g) in 1 (25.0 g) and benzoyl peroxide (1.0 g) at 140 °C for 4 h. After filtration, the reaction mixture was chromatographed on silica gel (CH_2Cl_2 solvent). The early fractions were recrystallized from ethanol to give 6.96 g (27%): mp 203–204 °C; mass spectrum, *m/e* (relative intensity) 330 (18), 315 (8), 186 (16), 171 (6), 159 (5), 145 (91), 144 (100), 130 (41), 115 (24), 77 (17); UV (95% EtOH) 415 nm (ε 25 000), 275 (11 600), 247 (11 400), 243 (10 800), 206 (31 000); IR (thin film) 3240 (NH), 1600 (C=O) cm^{-1}; ^1H NMR ($CDCl_3$) δ 11.5 (br, 1), 7.79 (m, 1), 7.44–6.98 (m, 7), 6.69 (s, 1), 1.61 (s, 6), 1.50 (s, 6); see Table II for ^{13}C NMR data; exact mass calcd for $C_{22}H_{22}N_2O$ 330.1732, found 330.1727 (M^+).

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Registry No. 1, 1640-39-7; 6, 87697-25-4; 8, 87697-26-5; methylene iodide, 75-11-6.

(10) Sadtler Research Laboratories, Inc. Spectra, ^1H NMR 9761M and 16082M; ^{13}C NMR 3167 and 3615.

Facile One-Pot Synthesis of Bromo Homoallyl Alcohols and 1,3-Keto Acetates via Allyltin Intermediates

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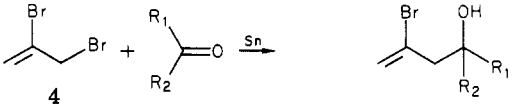
Allylation of carbonyl compounds by means of allyltin intermediates has been the subject of current interest, and we have previously disclosed that the reaction is extensively accelerated by the presence of water.[1]

Bromo homoallyl alcohols 1, useful intermediates for α-methylene γ-lactones, can be prepared from [1-(trimethylsilyl)vinyl]magnesium bromide and epoxy compounds as reported first by Matsuda[2] and later by Stille et al.[3] More recently, Trost et al. have reported the preparation from 2-bromo-3-(trimethylsilyl)propene.[4] These papers prompted us to describe here our own synthetic method of 1 by the allyltin method. Furthermore, acetonylation of the carbonyl compounds leading to 1,3-keto acetates 3 will also be described.

When 2,3-dibromoprop-1-ene (4) was subjected to the reaction with carbonyl compounds in the presence of metallic tin in a 1:1 ether–water mixture (eq 1), 1 was



4 (1)

1a, R_1 = *n*-C_4H_9; R_2 = H
b, R_1 = $PhS(CH_2)_2$; R_2 = H
c, R_1 = Ph; R_2 = H
d, R_1 = $EtO_2C(CH_2)_8$; R_2 = H
e, R_1 = $NC(CH_2)_2$; R_2 = H
f, R_1,R_2 = $(CH_2)_5$

obtained in good yields as shown in Table I. The notable features of the present method consist of the following aspects. (1) The reaction is a one-pot synthesis, and yields are high. (2) Operations are simple and do not need an inert atmosphere. (3) 2,3-Dibromoprop-1-ene is readily or commercially available. (4) Masked aldehydes such as acetals or ketals are employable without deblocking pro-

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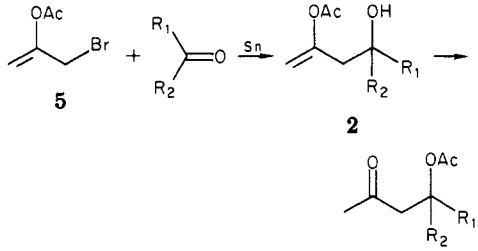
0022-3263/84/1949-0172$01.50/0

Table I. Synthesis of Bromo Homoalkyl Alcohols 1

entry	carbonyl compd	reaction time, h	product	yield, %
1	$CH_3(CH_2)_3CHO$	6	1a	81
2	$PhS(CH_2)_2CHO$	3	1b	70
3	PhCHO	5	1c	97
4		5	1c	80
5	$EtO_2C(CH_2)_8CHO$	4	1d	83
6	$NC(CH_2)_2CH(OCH_3)_2$	6	1e	88
7		4	1f	78

cess. The present method, therefore, seems to offer a promising procedure for 1.

Next, reactions of 2-acetoxy-3-bromoprop-1-ene (5) with a variety of carbonyl compounds in the presence of tin were investigated (eq 2), the results of which are shown in Table

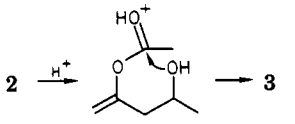


5 2

(2)

3a, R_1 = n-C_4H_9; R_2 = H
b, R_1 = $PhS(CH_2)_2$; R_2 = H
c, R_1 = Ph; R_2 = H
d, R_1,R_2 = $(CH_2)_5$
e, R_1 = $CH_3CH{=}CH$; R_2 = H
f, R_1 = $PhCH{=}CH$; R_2 = H

II. When the reactions were conducted under the same conditions as for reaction 1 (method A), there were unexpectedly obtained 1,3-keto acetates 3 in addition to normal products 2 (entries 1, 3, 5, and 9). Furthermore, it was found that employment of carbonyl compounds in excess (method B) resulted in the exclusive formation of 3 except for 3-(phenylthio)propionaldehyde and benzaldehyde (entries 4 and 6). Facile conversion of 2 to 3 may be interpreted in terms of the acid-prompted mechanism in eq 3.



(3)

Experimental Section

^{1}H NMR spectra were measured with a Hitachi R-24B spectrometer operating at 60 MHz at 35 °C. Commercially available aldehydes, ketones, and 2,3-dibromoprop-1-ene (4) were purified by distillation. 3-(Phenylthio)propionaldehyde was prepared by 1,4-addition of sodium thiophenolate to acrolein. ω-(Ethoxycarbonyl)nonanal was obtained by ozonolysis of ethyl undecylenate. 2-Acetoxy-3-bromoprop-1-ene (5) was prepared by acetoxymercuration of propargyl bromide.[5]

Reaction of 4 with Carbonyl Compounds. To a suspension of tin powder (178 mg, 1.5 mmol) and 4 (599 mg, 3 mmol) in an ether–water (each 2 mL) mixture were added a few drops of an aqueous HBr solution and pentanal (86 mg, 1 mmol). The mixture was stirred for 6 h at room temperature and extracted with ether and water followed by drying ($MgSO_4$) and evaporation. The resulting oil was purified by column chromatography on silica gel (hexane–ether, 50:1) to give **1a**:[4] yield 168 mg (81%); ^{1}H NMR (CCl_4) δ 0.62–1.68 (m, 9 H, C_4H_9), 1.90 (s, 1 H, OH), 2.52 (d, 2 H, J = 6.4 Hz, $CH_2C{=}C$), 3.93 (m, 1 H, CHOH), 5.55 (d, 1 H, J = 1.6 Hz, =CH), and 5.72 (br s, 1 H, =CH).

Other bromo homoallyl alcohols were synthesized analogously.

1b: purified by column chromatography on silica gel (hexane–ether 30:1): ^{1}H NMR (CCl_4) δ 1.77 (dt, 2 H, J = 6.0, 8.0 Hz, $SCCH_2$), 2.14 (s, 1 H, OH), 2.52 (d, 2 H, J = 6.0 Hz, $CH_2C{=}C$), 3.07 (t, 2 H, J = 8.0 Hz, SCH_2), 4.11 (quintet, 1 H, J = 6.0 Hz, CHOH), 5.56 (d, 1 H, J = 1.2 Hz, =CH), 5.72 (br s, 1 H, =CH), 7.32 (m, 5 H, C_6H_5). Anal. Calcd for $C_{12}H_{15}BrOS$: C, 50.18; H, 5.26. Found: C, 49.89; H, 5.52.

1c: purified by column chromatography on silica gel (hexane–ether 50:1); ^{1}H NMR (CCl_4) δ 2.34 (s, 1 H, OH), 2.72 (d, 2 H, J = 6.7 Hz, $CH_2C{=}C$), 4.94 (t, 1 H, J = 6.7 Hz, CHOH), 5.45 (d, 1 H, J = 1.6 Hz, =CH), 5.59 (br s, 1 H, =CH), 7.31 (s, 5 H, C_6H_5). Anal. Calcd for $C_{10}H_{11}BrO$: C, 52.89; H, 4.88. Found: C, 52.54; H, 4.98.

1d: purified by preparative TLC on silica gel (hexane–ether, 5:2); ^{1}H NMR (CCl_4) δ 1.35 (br s, 16 H, $C(CH_2)_8C$), 1.78 (s, 1 H, OH), 2.20 (d, 2 H, J = 6.0 Hz, CH_2CO), 2.50 (d, 2 H, J = 7.0 Hz, $CH_2C{=}C$), 3.67 (s, 3 H, OCH_3), 3.88 (m, 1 H, CHOH), 5.55 (d, 1 H, J = 1.4 Hz, =CH), 5.72 (br s, 1 H, =CH). Anal. Calcd for $C_{15}H_{27}BrO_3$: C, 53.74; H, 8.12. Found: C, 53.34; H, 7.98.

1e: purified by column chromatography on silica gel (hexane–ether 20:1); ^{1}H NMR (CCl_4) δ 1.29–2.15 (m, 2 H, CH_2CCN), 2.27–2.69 (m, 5 H, $CH_2C{=}C$, CH_2CN, and OH), 3.99 (m, 1 H, CHOH), 5.50 (d, 1 H, J = 2.0 Hz, =CH), 5.64 (br s, 1 H, =CH). Anal. Calcd for $C_7H_{10}BrNO$: C, 41.20; H, 4.94; N, 6.86. Found: C, 41.45; H, 5.35; N, 7.02.

1f[2]: purified by column chromatography on silica gel (hexane–ether 50:1); ^{1}H NMR (CCl_4) δ 1.55 (br s, 10 H, CH_2), 1.82 (s, 1 H, OH), 2.65 (s, 2 H, $CH_2C{=}C$), 5.65 (d, 1 H, J = 1.0 Hz, =CH), and 5.72 (br s, 1 H, =CH).

Reaction of 5 with Carbonyl Compounds (Method B). To a suspension of tin powder (356 mg, 3 mmol) and 5 (358 mg, 2 mmol) in a ether–water (each 2 mL) mixture was added a few drops of an aqueous HBr solution and pentanal (258 mg, 3 mmol). The mixture was stirred for 4 h at room temperature and extracted with ether and water. The organic layer was washed with brine and water, dried ($MgSO_4$), and evaporated. The resulting oil was purified by preparative TLC on silica gel (hexane–ether 3:1) to give **3a**: yield 272 mg (73%); ^{1}H NMR (CCl_4) δ 0.56–1.64 (m, 9 H, C_4H_9), 1.99 (s, 3 H, CH_3CO), 2.11 (s, 3 H, OCH_3), 2.57 (d, 2

Table II. Synthesis of 1,3-Keto Acetates 3

entry	carbonyl compd	reaction method[a]	reaction time, h	yield of 2 and 3,[b] % (2/3)[c]
1	$CH_3(CH_2)_3CHO$	A	10	70 (23/77)
2		B	4	73 (0/100)
3	$PhS(CH_2)_2CHO$	A	3	82 (78/22)
4		B	4	76 (32/68)
5	PhCHO	A	3	99 (30/70)
6		B	3	65 (27/73)
7		B	4	42 (0/100)
8	$CH_3CH{=}CHCHO$	B	4	50 (0/100)
9	$PhCH{=}CHCHO$	A	4	77 (54/46)
10		B	6	71 (0/100)

[a] Method A: ratio of reactants was 1:3:1.5 carbonyl/5/Sn. Method B: ratio of reactants was 3:2:3 carbonyl/5/Sn. [b] Based on carbonyl compounds (method A) and on 5 (method B). [c] The ratio was determined on the basis of ^{1}H NMR spectra.

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H, J = 6.0 Hz, CH_2CO), 5.11 (m, 1 H, CHOAc). Anal. Calcd for $C_{10}H_{18}O_3$: C, 64.45; H, 9.76. Found: C, 64.36; H, 9.87.

Other 1,3-keto acetates were synthesized analogously.

3d: purified by column chromatography on silica gel (hexane–ether 50:1); ^1H NMR (CCl_4) δ 1.51 (br s, 10 H, CH_2), 2.06 (s, 3 H, CH_3CO), 2.13 (s, 3 H, OCH_3), 3.10 (s, 2 H, CH_2CO). Anal. Calcd for $C_{11}H_{18}O_3$: C, 66.62; H, 9.17. Found: C, 66.24; H, 9.32.

3e: purified by distillation [bp 120 °C (20 mm; Kugelrohr bath temperature)]; ^1H NMR (CCl_4) δ 1.71 (d, 3 H, J = 5.8 Hz, CH_3C=C), 2.00 (s, 3 H, CH_3CO), 2.14 (s, 3 H, OCH_3), 2.68 (dd, 2 H, J = 6.0, 2.0 Hz, CH_2CO), 5.20–6.25 (m, 3 H, CHOAc and CH=CH). Anal. Calcd for $C_9H_{14}O_3$: C, 63.50; H, 8.31. Found: C, 63.06; H, 8.44.

3f: purified by column chromatography on silica gel (hexane–ether 30:1); ^1H NMR (CCl_4) δ 2.04 (s, 3 H, CH_3CO), 2.17 (s, 3 H, OCH_3), 2.80 (dd, 2 H, J = 8.0, 2.0 Hz, CH_2CO), 5.76–6.34 (m, 2 H, CHOAc and CH=CPh), 6.72 (d, 1 H, J = 15 Hz, C=CHPh), 7.35 (br s, 5H, C_6H_5). Anal. Calcd for $C_{14}H_{16}O_3$: C, 70.87; H, 7.34. Found: C, 71.21; H, 7.05.

Registry No. **1a**, 83650-02-6; **1b**, 87763-89-1; **1c**, 87763-90-4; **1d**, 87763-91-5; **1e**, 87763-92-6; **1f**, 67964-39-0; **2a**, 87763-93-7; **2b**, 87763-94-8; **2c**, 87763-95-9; **2f**, 87763-96-0; **3a**, 87763-97-1; **3b**, 87763-98-2; **3c**, 56894-87-2; **3d**, 87763-99-3; **3e**, 87764-00-9; **3f**, 87764-01-0; **4**, 513-31-5; **5**, 87764-02-1; $CH_3(CH_2)_3CHO$, 110-62-3; $PhS(CH_2)_2CHO$, 27098-65-3; PhCHO, 100-52-7; $EtO_2C(CH_2)_8CHO$, 692-87-5; $NC(CH_2)_2CH(OCH_3)_2$, 14618-78-1; CH_3CH=CHCHO, 4170-30-3; PhCH=CHCHO, 104-55-2; benzaldehyde ethylene ketone, 936-51-6; cyclohexanone, 108-94-1; tin, 7440-31-5.

Synthesis of Carbon-13 Labeled Aldehydes, Carboxylic Acids, and Alcohols via Organoborane Chemistry

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Isotopically labeled materials have played a unique role in chemical and biological research.[1,2] The regiospecific incorporation of carbon-13 has become increasingly important in recent years with the advent of the multinuclear NMR instruments and promises to assume an even greater role in medical research as the new whole-body multinuclear NMR scanners become more widespread.[3,4] The enriched materials that are required for biological and medical studies often present formidable synthetic challenges due to the variety of functional groups that impart the physiological activity to the molecules of interest. The traditional incorporation routes for carbon-13 (Grignard reactions, hydrolysis of labeled nitriles, oxidation of labeled methyl groups, etc.,) are often unsuitable for synthesizing these functionally substituted molecules.

Organoboranes have been utilized to prepare a wide variety of functionally substituted reagents.[5,6] In recent years, we have used the organoboranes to incorporate a number of isotopes.[7,8] It occurred to us that the carbonylation reaction developed by Brown and his co-workers would be an ideal method for regiospecifically incorporating carbon-13.[9-11] We now report that the carbonylation of organoboranes using carbon-13 enriched carbon monoxide produces the labeled aldehydes in excellent yield. Furthermore, the ^{13}C-labeled aldehydes are readily converted into the corresponding ^{13}C-labeled carboxylic acids and alcohols.

The synthesis of the labeled carboxylic acids could be carried out in a variety of ways, including a direct oxidation of the initial organoborane adduct. However, we find that silver oxide oxidation of the aldehydes leads to higher yields of the acids.

The synthesis of the carbon-13-labeled alcohols could also be achieved in a number of ways, including the direct hydrolysis of the initially formed organoborane adduct[9] (replacing the oxidation–reduction sequence). We find that higher yields are obtained by avoiding the harsh, direct hydrolysis reaction. We prefer to reduce the aldehyde with a borane reagent. Since the initial step in the sequence involved a hydroboration, few difficulties are encountered with a borane reagent in the final step.

Our results are summarized in Table I.

Experimental Section

Melting points and boiling points are uncorrected. Routine NMR spectra were recorded on a Varian Associates T-60 spectrometer. All chemical shifts are reported in parts per million downfield from Me_4Si. ^{13}C NMR spectra were run on a JEOL FX-90Q spectrometer and referenced to external CCl_4, $CDCl_3$, or acetone-d_6. The mass spectra were obtained with a HP-5982-A GC-mass spectrometer. Elemental Analyses were performed by Galbraith Laboratories, Knoxville, TN.

Commercially available samples (Aldrich) of cyclohexene, 10-undecen-1-ol, 1-nonene, and safrole were distilled prior to use. 3-(*p*-Tolylthio)-2-methylpropene[12] and 9-BBN[13] were prepared according to published procedures.

Hydroboration. General Procedure. 9-BBN (10 mmol, 33.3 mL of a 0.3 M solution in THF) was placed in a dry, 50-mL, nitrogen-flushed flask fitted with a septum inlet and cooled to 0 °C. The alkene (10 mmol) was added via syringe and the solution stirred overnight at room temperature to yield the corresponding organoborane. [For alkenes containing acidic hydrogens, a twofold excess of 9-BBN was employed during the hydroboration.]

Carbonylation. General Procedure. The organoborane (10 mmol) was cooled to 0 °C and potassium triisopropoxyborohydride (KIPBH) (10 mmol, 10 mL of a 1.0 M solution) was then added. [**Caution**: excess KIPBH will inhibit the uptake of CO.] An atmosphere of carbon-13-enriched carbon monoxide was then maintained over the solution by using a syringe needle connected to a rubber bladder containing the ^{13}C-enriched CO. (An initial flush of the N_2 atmosphere with ^{13}CO is helpful.) The mixture was stirred vigorously for 30 min, and then NaOAc (12 mmol, 12 mL of a 1.0 M solution) and H_2O_2 (12 mmol, 1.3 mL of a 30% solution) were added at 0 °C to oxidize the intermediate organoborane complex to form the ^{13}C-labeled aldehyde. Excess diethanolamine was then added to precipitate the borinic acid byproduct.[14] The mixture was saturated with NaCl and the product extracted into ether. The ether was removed, and the aldehyde was purified via column chromatography.

Oxidation. General Procedure. The crude aldehyde was added to a suspension of freshly prepared Ag_2O [$AgNO_3$ (30 mmol, 30 mL of a 1.0 M solution) is mixed with NaOH (30 mL of a 2.0

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