Indian Journal of Chemistry
Vol. 37B, August 1998, pp.731 - 736

Zinc mediated allylation of aldehydes and ketones using allyl bromides and commercial zinc dust. The issue of *regio*- and stereoselectivity

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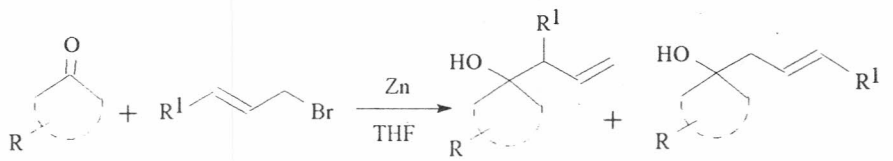
Received 29 May 1998; accepted 30 June 1998

An efficient procedure for the preparation of homoallylic alcohols has been achieved by a simple reaction of an aldehyde or a ketone with allyl bromide and commercial zinc dust in tetrahydrofuran. Excellent regioselectivity has been observed in the reaction of substituted allyl bromides.

Allylation of carbonyl compounds with allyl organometallic compounds or by allylic halides, assisted by metals, to produce homoallylic alcohols is one of the most useful processes in organic synthesis[1] because of the great potentiality of the homoallylic alcohols to be converted to important building blocks in natural product synthesis.[2] Acyclic stereocontrol during the carbon-carbon bond formation has also increased the importance of this process.[3] Different methods have been developed based on the use of a variety of metals such as magnesium,[4] zinc,[5] tin,[6] indium[7] and barium.[8] But, zinc offers certain specific advantages being moderately reactive to allow the preparation of polyfunctional allylzinc derivatives[9] and avoiding formation of side products derived from reductive coupling of the carbonyl compound such as pinacol condensation, often observed using other reagents.[10] However, in general, the preparation of allylzinc derivatives is reported to require activated zinc to initiate the reaction,[11] although some allylations have been performed using non-activated zinc in harsher conditions or in aqueous media in the presence of ammonium chloride and other additives.[12] Several methods are available to activate zinc such as washing with HCl solution,[13] ultrasound irradiation in the presence of lithium,[14] use of Zn/Cu couple,[15] reduction of zinc chloride with alkali metals,[16] electroreduction[11] or sonoelectrochemical reduction[17] of zinc chloride, etc. Considering the increasing importance of allylzinc compounds in organic syntheses, we have recently reported in a communication[18] a straightforward method for the formation of allylzinc bromide through a simple reaction of allyl bromide and commercial zinc dust in THF, which adds on to aldehydes or ketones to produce the corresponding homoallylic alcohols in excellent yields. We now wish to disclose the regio- and stereochemical outcome of this reaction with substituted allyl bromides and carbonyl compounds as well as the details of earlier results[18] **(Scheme I)**.

Results and Discussion

The experimental procedure is very simple. The allyl bromide in THF was added dropwise to a stirred suspension of commercial zinc dust in THF



R , R^1 = H , alkyl or aryl

Scheme I

and mixture was stirred for half an hour after which the carbonyl compound was added. The reaction mixture was stirred for a certain period of time as required to complete the reaction (TLC), decomposed with water and worked-up as usual to furnish the homoallylic alcohol.

A wide range of structurally varied aldehydes and ketones underwent allylation by this procedure to produce the corresponding homoallylic alcohols in excellent yields.The results are presented in **Table I**. The reactions are reasonably fast and clean. The reaction condition is mild enough not to affect the conjugated as well as the isolated double bonds (entries 3-5,18,28), methoxyl (entries 7-9), chloro (entry 10) and carboxylic ester functionality (entry 28). In the reactions of substituted allyl bromides such as crotyl bromide and cinnamyl bromide with several aldehydes and ketones, excellent regioselectivity was observed providing γ-addition products as the only isolable compounds (entries 6-10, 20-23). However, the addition of allyl bromide to substituted cyclohexanones is not always stereoselective. No selectivity was observed in the addition to 2-methyl-, 3-methyl- and 4-*t*-butyl cyclohexanones (entries 24-26) whereas 4-methyl cyclohexanone and 4-carboethoxy-3-methylcyclohex-2-en-1-one (Hagemann's ester) produce *trans* isomers almost exclusively (entries 27, 28). The reason for this anomolous behaviour is not well-understood.

Allyl chloride is found to be much less reactive than allyl bromide as it has been observed that the reactions of allyl chloride with cyclohexanone and 1-octanal under identical conditions are only 10-15% complete. Similar observation has been reported earlier by Rollin *et al*[11]. Zinc dust from different sources are found to give the same results.

Conclusions

The present procedure for allylation of carbonyl compounds using commercial zinc dust provides much improvement with regard to operational simplicity, generality, yield and regioselectivity over the existing methods and thus, will make a useful and important addition to the present methodologies.

Experimental Section

General. ^{1}H NMR spectra were recorded at 60, 200 or 300 MHz in CCl_4 or $CDCl_3$ solutions with Me_4Si as internal standard. ^{13}C NMR were recorded at 50 and 75 MHz. IR spectra were taken as thin film (neat) on a Perkin-Elmer 298 spectrometer. GC was run on a Shimadzu GC-9A instrument using SE-30 column. Analyses were carried out by Mr S Sarkar of this laboratory. Thin layer chromatography(TLC) was performed on precoated silica gel plates (Sigma). Silica gel (60-120 mesh) was used for column chromatography. Commercial zinc dust was used without any treatment. Tetrahydrofuran was distilled from potassium-benzophenone prior to use. Carbonyl compounds and allyl bromides were commercial products and were distilled before use.

General Procedure for Allylation. The allyl bromide (1 mmole) in THF (1 mL) was added dropwise to a stirred suspension of commercial zinc dust (1 mmole) in THF (2 mL) at room temperature and the mixture was stirred for half an hour after which the carbonyl compound (1 mmole) in THF (1 mL) was added. Stirring was continued for a certain period of time as required to complete the reaction (TLC). The reaction mixture was decomposed by careful addition of a few drops of water and extracted with ether (3x20 mL). The ethereal extract was washed with brine, dried over Na_2SO_4 and evaporated to leave the crude product which was purified through a short column of silica gel.

Although the procedure has been written in 1 mmole scale, gram-scale reactions were also carried out and found to afford the corresponding products in analogously good yields.

The products were easily characterised by their spectral data (IR, ^{1}H and ^{13}C NMR) and elemental analyses. Many of these compounds have been reported in the literature earlier (cf. **Table I**) and the spectral and analytical data of the new compounds are presented below designated by their entries in the **Table I**.

1 : IR: 1640, 3200-3600 cm^{-1}; ^{1}H NMR (60 MHz, CCl_4): δ 0.83-1.8 (15H, m), 2.13 (2H,d, *J*=5.9 Hz), 3.5 (2H, broad), 4.8-6.1 (3H, m). Anal. Calcd for $C_{11}H_{22}O$: C, 77.58; H, 13.02. Found : C, 77.62; H, 13.22%.

5 : IR: 1640, 3200-3600 cm^{-1}; ^{1}H NMR (60 MHz, CCl_4): δ 1.0-2.8 (16H, m), 4.0-4.5 (1H, m), 4.8-6.1 (5H, m). Anal. Calcd for $C_{13}H_{22}O$: C, 80.35; H, 11.41. Found : C, 80.40; H, 11.51%.

Table I — Allylation of carbonyl compounds

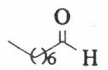
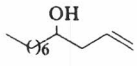
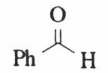
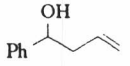
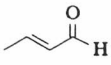
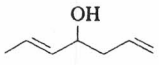
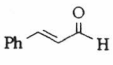
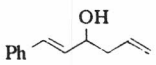
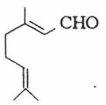
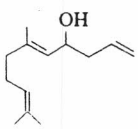
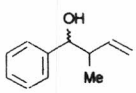
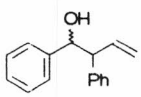
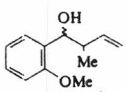
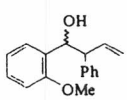
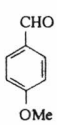
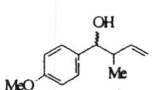
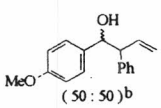
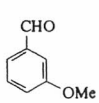
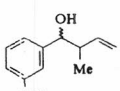
Entry (Ref.)	Carbonyl compd	RC*	RP† (hr)	Product (ratio)	Yield (%)[a]
1		A	1.5		88
2 (1i)		A	3		90
3 (19)		A	1.5		91
4 (1i)		A	6		94
5		A	2		85
6 (7)		B	1.5	 (57 : 43)[b]	90
		C	2	 (60 : 40)[b]	85
7		B	1	 (63 : 37)[b]	88
		C	2	 (48 : 52)[b]	90
8		B	2	 (60 : 40)[b]	91
		C	2	 (50 : 50)[b]	80
9		B	1.5	 (50 : 50)[b]	90

Table I —*Contd*

Table I — Allylation of carbonyl compounds — *Contd*

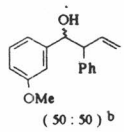
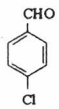
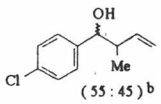
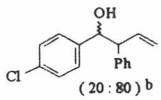
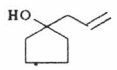
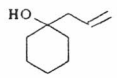
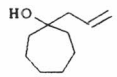
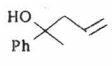
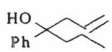
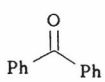
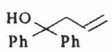
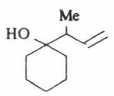
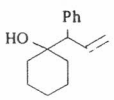
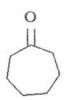
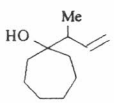
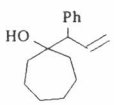
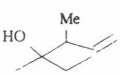
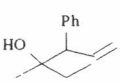
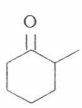
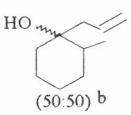
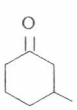
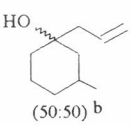
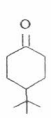
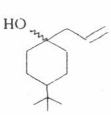
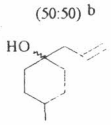
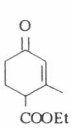
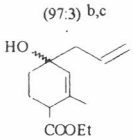
Entry (Ref.)	Carbonyl compd	RC*	RP† (hr)	Product (ratio)	Yield (%)
		C	2.5	 (50 : 50)[b]	85
10 (4)		B	1	 (55 : 45)[b]	95
(19)		C	2	 (20 : 80)[b]	92
11 (4)		A	1.5		80
12 (19)		A	3		92
13		A	1		84
14		A	0.5		94
15		A	1		85
16		A	2		89
17		A	2.5		91
18		A	2		92
19		A	2		94
20 (22)		B	1		92
		C	1.5		94

Table I —*Contd*

Table I — Allylation of carbonyl compounds —*Contd*

Entry (Ref.)	Carbonyl compd	RC*	RP† (hr)	Product (ratio)	Yield (%)[a]
21 (22)		B	2		76
(20)		C	2		90
22		B	2		91
		C	2		90
23		B	1.5		86
		C	2		95
24 (20)		A	1.5	 (50:50) [b]	92
25 (20)		A	1.5	 (50:50) [b]	90
26 (20)		A	2	 (50:50) [b]	91
27 (20)		A	1.5	 (97:3) [b,c]	90
28	 COOEt	A	3	 COOEt (98:2) [b,c]	90

A: Reaction with allyl bromide, B: Reaction with crotyl bromide C: Reaction with cinnamyl bromide.

[a] All yields refer to pure isolated products.

[b] The ratio of stereoisomers (*anti/syn* or *trans/cis*) has been determined by GC and NMR.

[c] The major isomer has been assigned *trans*-geometry based on ^{13}C NMR data.

* RC : Reaction condition.

† RP : Reaction period.

7B: IR: 1600, 1640, 3200-3600 cm^{-1}; ^1H NMR (60 MHz, CCl_4): δ 0.93 and 0.96 (total 3H, 2d, J = 7,6 Hz), 2.09 (1H, broad), 2.53 (1H, quintet, J = 6 Hz), 3.79 (3H, s), 4.56-4.93 (2H, m), 4.96-5.23 (1H, m), 5.49-6.23 (1H, m), 6.66-7.39 (4H, m). Anal. Calcd for $C_{12}H_{16}O_2$: C, 74.97; H, 8.39. Found : C, 74.92; H, 8.07%.

7C : IR: 1600, 1640, 3200-3600 cm^{-1}; ^1H NMR (60 MHz, CCl_4): δ 2.33 (1H, broad), 3.59 (3H, s), 3.69 (1H, t, J = 6 Hz), 4.66-5.33 (3H, m), 5.73-6.43 (1H, m), 6.49-7.4 (9H, m). Anal. Calcd for $C_{17}H_{18}O_2$: C, 80.28; H, 7.13. Found : C, 80.30; H, 7.11%.

8B : IR: 1610, 1640, 3200-3600 cm^{-1}; ^1H NMR (60 MHz, CCl_4): δ 0.83 and 0.96 (total 3H, 2d, J = 7,6.5 Hz), 2.03 (1H, broad), 2.03-2.63 (1H, m), 3.79 (3H, s), 4.39 and 4.56 (total 1H, 2d, J = 7,6 Hz), 4.6-5.23 (2H, m), 5.33-5.99 (1H, m), 6.76 (2H, d, J = 10 Hz), 7.16 (2H, d, J = 10 Hz). Anal. Calcd for $C_{12}H_{16}O_2$: C, 74.97; H, 8.39. Found : C, 74.95; H, 8.22%.

8C : IR: 1600, 1640, 3200-3600 cm^{-1}; ^1H NMR (60 MHz, CCl_4): δ 1.76 (1H, broad), 3.26-3.73 (1H, m), 3.79 (3H, s), 4.66-5.33 (3H, m), 5.66-6.49 (1H, m), 6.89 (2H, d, J= 10 Hz), 6.99 (5H, s), 7.56 (2H, d, J = 10 Hz). Anal. Calcd for $C_{17}H_{18}O_2$: C, 80.28; H, 7.13. Found : C, 80.18; H, 7.23%.

9B : IR: 1605, 1640, 3200-3600 cm^{-1}; ^1H NMR (60 MHz, CCl_4): δ 0.83 and 0.96 (total 3H, 2d, J = 6 Hz), 2.33 (2H, broad), 3.73 (3H, s), 4.19 and 4.39 (total 1H, 2d, J = 7,6 Hz), 4.69-5.19 (2H, m), 5.73-6.09 (1H, m), 6.49-7.43 (4H, m).Anal. Calcd for $C_{12}H_{16}O_2$: C, 74.97; H, 8.39. Found : C, 75.01; H, 8.29%.

9C : IR: 1600, 1640, 3200-3600 cm^{-1}; ^1H NMR (60 MHz, CCl_4): δ 2.43 (1H, broad), 3.46 (1H, t, J = 6 Hz), 3.96 (3H, s), 4.33-5.16 (3H, m), 5.16-5.99 (1H, m), 6.66-7.33 (9H, m). Anal. Calcd for $C_{17}H_{18}O_2$: C, 80.28; H, 7.13. Found : C, 80.50; H, 7.01%..

10B : IR: 1610, 1640, 3200-3600 cm^{-1}; ^1H NMR (60 MHz, CCl_4): δ 0.86 and 0.96 (total 3H, 2d, J = 5,4 Hz), 2.23 (1H, broad), 2.23-2.66 (1H, m), 4.26 and 4.46 (total 1H, 2d, J = 6,5 Hz), 4.76-5.33 (2H, m), 5.46-6.13 (1H, m), 7.26 (4H, s). Anal. Calcd for $C_{11}H_{13}ClO$: C, 67.21; H, 6.61. Found : C, 67.40; H, 6.50%.

10C : IR: 1600, 1640, 3200-3600 cm^{-1}; ^1H NMR (60 MHz, CCl_4): δ 2.13 (1H, broad), 3.03 (1H, t, J = 7 Hz), 4.69 and 4.76 (total 1H, 2d, J = 7,6 Hz), 4.89-5.33 (2H, m), 5.83-6.49 (1H, m), 6.83-7.06 (9H, m).Anal. Calcd for $C_{16}H_{15}ClO$: C, 74.30; H, 5.80. Found : C, 74.11; H, 5.83%.

13 : IR: 1640, 3200-3600 (broad) cm^{-1}; ^1H NMR (60 MHz, CCl_4): δ 1.3-1.9 (12H, m), 2.15 (2H, d, J = 5.9 Hz), 2.4 (1H, broad), 4.8-6.1 (3H, m).Anal. Calcd for $C_{10}H_{18}O$: C, 77.86; H, 11.76. Found : C, 77.90; H, 11.96%.

16 : IR: 1640, 2960, 3200-3600 cm^{-1}; ^1H NMR (60 MHz, CCl_4): δ 0.8-2.0 (6H, m), 2.5 (2H, t, J = 5.9 Hz), 2.8 (2H, t, J = 5.9 Hz), 4.8-6.1 (3H, m), 7.1-7.5 (5H, m).Anal. Calcd for $C_{13}H_{18}O$: C, 82.06; H, 9.54. Found : C, 82.16; H, 9.66%.

18 : IR: 1640, 3200-3600 cm^{-1}; ^1H NMR (60 MHz, CCl_4): δ 1.4-1.8 (10H, m), 2.15 (2H, d, J = 5.9 Hz), 4.8-6.1 (4H, m). Anal. Calcd for $C_{10}H_{16}O$: C, 78.89; H, 10.59. Found : C, 78.60; H, 10.71%.

19 : IR: 1640, 2920, 2960, 3200-3600 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 1.58 (3H, s), 2.39 (3H, s), 2.50-2.76 (3H, m), 5.11-5.22 (2H, m), 5.60-5.81 (1H, m), 7.19 (2H, d, J = 8.2 Hz), 7.39 (2H, d, J = 8.2 Hz); ^{13}C NMR (50 MHz, $CDCl_3$): δ 20.75, 29.61, 48.35, 73.40, 118.82, 124.60, 128.65, 133.79, 135.85, 144.66.Anal. Calcd for $C_{12}H_{16}O$: C, 81.77; H, 9.15. Found : C, 81.97; H, 9.21%.

20C : IR: 1640, 2960, 3200-3600 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 1.28-2.78 (8H, m), 3.32 (2H, d, J = 9 Hz), 5.11 (2H, m), 6.25-6.40 (1H, m), 7.15-7.34 (5H, m); ^{13}C NMR (75 MHz, $CDCl_3$): δ 23.31, 23.41, 38.03, 38.49, 59.56, 83.88, 116.79, 126.73, 128.05, 128.78, 138.13, 141.29.Anal. Calcd for $C_{14}H_{18}O$: C, 83.12; H, 8.97. Found : C, 83.31; H, 8.87%.

22B : IR: 1640, 3200-3600 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 0.96 (3H, d, J = 7 Hz), 1.30-1.69 (13H, m), 2.13 (1H, m), 4.97 (2H, d, J = 12 Hz), 5.70-5.82 (1H, m).Anal. Calcd for $C_{11}H_{20}O$: C, 78.51; H, 11.98. Found : C, 78.67; H, 11.72%.

22C : IR: 1640, 2960, 3200-3600 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 1.29-1.79 (12H, m), 3.26 (2H, d, J = 9 Hz), 5.06-5.18 (2H, m), 6.27-6.36 (1H, m), 7.11-7.33 (5H, m).Anal. Calcd for $C_{16}H_{22}O$: C, 83.43; H, 9.63. Found : C, 83.58; H, 9.53%.

23B : IR: 1640, 3200-3600 cm^{-1}; ^1H NMR (60 MHz, CCl_4): δ 0.8-2.4 (12H, m with a singlet at 1.0), 3.3 (1H, broad), 4.8-6.1 (3H, m). Anal. Calcd for $C_8H_{16}O$: C, 74.94; H, 12.58. Found : C, 74.80; H, 12.51%.

23C : IR: 1640, 2960, 3200-3600 cm^{-1}; ^1H NMR (60 MHz, CCl_4): δ 1.0 (3H, t, J = 6 Hz), 1.0 (3H, s), 1.07-2.56 (3H, m), 3.20 (1H, dd, J_1, J_2 = 6,2 Hz), 4.90 (1H, dd, J_1,J_2 = 6,2 Hz), 5.2 (1H, s), 5.93-6.6 (1H, m), 7.2 (5H, s). Anal. Calcd for $C_{13}H_{18}O$: C, 82.06; H, 9.54. Found : C, 82.31; H, 9.55%.

27 : IR: 1640, 3200-3600 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 0.83 (3H, s), 1.23-1.54 (9H, m), 1.70 (1H, broad), 2.09 (2H, d, J = 7 Hz), 4.96-5.04 (2H, m), 5.76-5.87 (1H, m); ^{13}C NMR (75 MHz, $CDCl_3$): δ 22.20, 30.10, 32.12, 36.70, 48.47, 70.10, 118.16, 133.86.

28 : IR: 1640, 1680, 1740, 2960, 3200-3600 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 1.19 (3H, t, J = 7 Hz), 1.57-2.03 (8H, m), 2.27 (2H, d, J = 6 Hz), 2.90 (1H, t, J = 4.7 Hz), 4.11 (2H, q, J = 7 Hz), 5.02-5.12 (2H, m), 5.47 (1H, broad s), 5.72-5.93 (1H, m); ^{13}C NMR (50 MHz, $CDCl_3$): δ 14.08, 22.63, 22.87, 32.05, 45.25, 46.67, 60.50, 69.18, 118.62, 133.36, 133.89, 173.49. Anal. Calcd for $C_{13}H_{20}O_3$: C, 69.61; H, 8.99. Found : C, 69.49, H, 9.12%.

Acknowledgement

We are pleased to acknowledge the financial support from CSIR, New Delhi [Grant No. 01(1364)/95)]. Two of the authors (AM and ARD) also thank CSIR for their fellowships.

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