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High γ-selectivity in the coupling of penta-2,4-dienyl- and pent-2-en-4-ynylindium reagents with aldehydes

Tsunehisa Hirashita, Shin'ichirou Inoue, Hatsuo Yamamura, Masao Kawai, Shuki Araki *

Department of Applied Chemistry, Nagoya Institute of Technology, Gokiso-cho, Showa-ku, Nagoya 466, Japan

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Abstract

A variety of penta-2,4-dienyl- and pent-2-en-4-ynylindium reagents have been prepared in situ from the reaction of the corresponding allylic bromides with indium metal, and their reactions with carbonyl compounds have been examined. The reaction with aldehydes gives the corresponding homoallyl alcohols in high yields. The coupling occurs regioselectively at the γ-position of these indium reagents. No α- and ε-coupling products are formed. © 1997 Elsevier Science S.A.

Keywords: Indium; Regioselectivity; Penta-2,4-dienyl system; Pent-2-en-4-ynyl system; Allylic alcohol

1. Introduction

Regioselective coupling of penta-2,4-dienylmetal reagents with electrophiles is a useful method in organic synthesis. When the coupling occurs at the terminal α- and/or ε-carbon(s) in a penta-2,4-dienylmetal, a conjugated penta-1,3-diene is formed, whereas at the internal γ-carbon a non-conjugated penta-1,4-diene is obtained. A variety of penta-2,4-dienylmetal species have hitherto been studied, of which stannane and silane reagents have attracted much attention. Pentadienylstannanes and -silanes are known to react with carbonyl compounds in the presence of Lewis acids giving homoallyl alcohols [1]. Lewis acids play an important role to determine the regioselectivity. For example, in the presence of strong Lewis acids such as boron trifluoride etherate and aluminium chloride, pentadienylstannanes react at their terminal ε-carbon. In contrast, the pentadienylation occurs regioselectively at the γ-position when zinc chloride with a lower Lewis acidity was used [2,3]. Although the metallic tin- and zinc-mediated reactions of pentadienyl bromides with carbonyl compounds have also been described to proceed selectively at the γ-position [4,5], no detailed study has been reported.

In order to control the regioselectivity of the reaction of pentadienylmetal with carbonyl compounds, we have focused on indium metal and undertaken the preparation and reactions of pentadienylindium reagents. Indium-mediated reactions have recently emerged as a useful tool in organic synthesis [6,7]. Allylindium reagents show high γ-selective allylation of a variety of carbonyl compounds, and some applications of allylindium reagents to natural products synthesis in aqueous media have been reported [8–10]. It has also been demonstrated that indium trichloride promotes the reaction of allylstannane with aldehydes to afford γ-adducts [11]. Recently, the reaction of pentadienylstannane with aldehyde promoted by indium trichloride was reported [12]. In these cases, indium trichloride is considered not to act as a Lewis acid for the activation of carbonyl compounds, but to form an allylic indium species via transmetalation from allylic stannane reagents [11,12].

In this paper, we describe the preparation of penta-2,4-dienyl- and pent-2-en-4-ynylindium reagents, which are higher homologues of allylindium with an extended conjugated double bond or triple bond, and their regioselective coupling reactions with carbonyl compounds.

2. Results and discussion

Penta-2,4-dienylindium reagents were readily prepared from indium powder and the corresponding allylic bromides in *N,N*-dimethylformamide (DMF) at ambient temperature. Penta-2,4-dienylindium prepared in situ

* Corresponding author. Fax: 81-52-735-5247.

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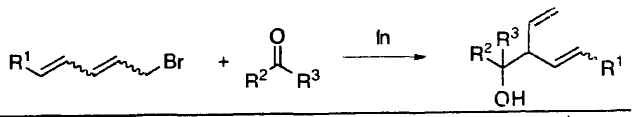
Table 1
Reaction of pentadienylindium reagents with carbonyl compounds



Entry	R^1	R^2	R^3	Conditions[a]	Yield (%)[b]
1	H	Ph	H	A	97
2	Me	H	H	A	56 (32:68)[c]
3	Me	Ph	H	A	76[d]
4	Me	Ph	H	B	23[d]
5	Me	n-C_7H_{15}	H	A	89[d]
6	Me	c-C_6H_{11}	H	A	72 (68:20:12)[e]
7	Me	(E)-PhCH=CH	H	A	100[d]
8	Ph	Ph	H	A	85 (54:46)[f]
9	Me	t-Bu	H	C	0
10	Me	Ph	Me	A	0

[a]A:in DMF, 0°C, 3 h; B: in water, room temperature, overnight; C: in DMF, room temperature, overnight.
[b]Isolated yield.
[c]E:Z ratio.
[d]Mixture of isomers. Diastereomeric ratio was not determined.
[e]Diastereomeric ratio determined by ^{13}C NMR analysis.
[f]Diastereomeric ratio determined by ^1H NMR analysis.

from penta-2,4-dienyl bromide (E:Z = 90:10) reacted with benzaldehyde regioselectivity at the γ-position to give the coupling product quantitatively (Table 1, entry 1). The indium-mediated reaction of hexa-2,4-dienyl bromide with aldehydes also gave the corresponding homoallylic alcohols in good yields (entries 2–7). Again, The γ-regioselectivity was perfectly achieved. The coupling product with formaldehyde was a mixture of geometrical isomers (E:Z = 32:68) (entry 2). 5-Phenylpenta-2,4-dienylindium also provided the γ-selectively coupled homoallyl alcohol (entry 8).

One of the characteristics of allylindium reagents is the variety of usable solvents. Allylation with allylindium can be carried out effectively in water [8–10,13], but the indium-mediated reaction of hexa-2,4-dienyl bromide with benzaldehyde in water was sluggish to give the homoallyl alcohol in a poor yield and unreacted benzaldehyde was recovered (entry 4). The regio- and diastereoselectivity were almost coincident to those in DMF. From the reaction of a ketone or bulky aldehyde like pivaraldehyde, no cross-coupling product was obtained (entries 9 and 10). Addition of a Lewis acid is an effective method for the activation of carbonyl compounds. However, the addition of boron trifluoride etherate to the reaction of hexa-2,4-dienylindium and octanal resulted in only decrease of the yield, and the regio- and diastereoselectivities were not changed.

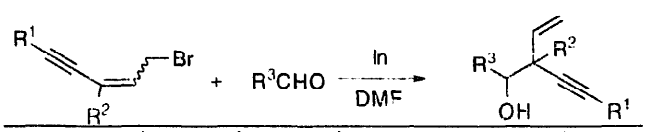
In principle, the γ-coupling reaction of hexadienylindium with aldehydes will give four diastereoisomers: pairs of syn/anti diastereomers and E/Z isomers. The homoallylic alcohol obtained in entry 6 was revealed by ^{13}C NMR analysis to be a mixture of three isomers in the ratio of 68:20:12. In the other cases, the homoallylic alcohols obtained are also considered to be mixtures of three or four possible isomers; however, the ratios were not determined.

It has been reported that both (E)- and (Z)-cinnamyl bromides give almost coincident diastereoselectivities with high anti-selectivity (> 90%) in the reaction with aldehyde [14]. This fact suggests that the E,Z-isomerization occurs during the oxidative addition of indium to cinnamyl bromide, or that the E,Z-isomerization of cinnamylindium is faster than the coupling reaction. The reaction of (E,E)-5-phenylpenta-2,4-dienyl bromide gave a diastereoselectivity of 54:46, whereas the E-geometry of the C^4 double bond was completely retained during the reaction (entry 8). If the transition state is assumed to be a six-membered cyclic type, the (E,E)-dienyl bromide, and even the (Z,E)-isomer, are expected to afford the anti-adduct as in the cinnamyl case. The observed low diastereoselectivity may be owing to the small energy difference between the two transition states leading to the syn- and anti-products.

The indium-mediated reaction of pent-2-en-4-ynyl bromides with aldehydes also proceeded smoothly (Table 2). The coupling occurred only at the γ-position of the enynylindium, as in the cases of penta-2,4-dienyl bromides. The enynylindium having no substituent at the γ-position reacted smoothly with benzaldehyde to give the corresponding homoallylic alcohol in good yield (entry 1). The methyl group at the γ-position of the enynylindium reagent diminished the reactivity, giving lower yields of the products (entries 2–4). Sterically bulky cyclohexylcarboxyaldehyde did not react at all with this enynylindium (entry 5).

In summary, the indium-mediated reactions of penta-2,4-dienyl bromides and pent-2-en-4-ynyl bromides with aldehydes have been found to give the corresponding homoallylic alcohols with complete γ-selectivity. Because allylic indium reagents have strong Lewis acidity, the coupling reaction with carbonyl compounds is considered to proceed via a coordination to the carbonyl

Table 2
Reaction of enynylindium reagents with aldehydes[a]



Entry	R^1	R^2	R^3	Yield (%)[b]
1	Ph	H	Ph	89 (67:33)
2	H	Me	Ph	30 (61:39)
3	H	Me	n-C_7H_{15}	36 (70:30)
4	H	Me	H	25
5	H	Me	c-C_6H_{11}	0

[a]All reactions were carried out in DMF at 0°C for 3 h.
[b]Isolated yield. Numbers in parentheses show diastereomeric ratio by ^1H NMR analysis.

oxygen, forming a six-membered transition state. This is the source of the high γ-regioselectivity of these reactions. The poor diastereoselectivity may be attributable to the distorted six-membered ring, because the indium atom is too bulky to form a rigid six-membered ring in the transition state. It has also been demonstrated that the geometry of the starting dienyl bromides is not important to the regioselectivity of the reaction of pentadienylindium reagents. This is in sharp contrast to the cases of dienylstannane reagents, in which the regioselectivity largely depends on the geometry of the reagents [15]. The present indium-prompted Barbier-type reactions are synthetically superior to the existing stannane- and silane-based ones, because of their high regioselectivity and experimental simplicity; there is no need to handle hazardous chemicals such as organometallics and Lewis acids.

3. Experimental section

3.1. General

IR spectra were recorded on a JASCO IRA-102 spectrophotometer. ^{1}H NMR spectra were obtained for solutions in $CDCl_3$ on a Varian Gemini 200 spectrometer (200 MHz) with Me_4Si as internal standard; J values are given in Hz. ^{13}C NMR spectra were measured for solutions in $CDCl_3$ with a Varian Gemini 200 spectrometer (50 MHz). Elemental analyses were done at the Elemental Analysis Centre of Kyoto University. All reactions were carried out under argon. Indium powder (99.99%) was obtained from Aldrich Chemical.

All dienyl bromides and enynyl bromides were prepared from the reaction of the corresponding alcohols with phosphorous tribromide in ether, and used without further purification. Penta-2,4-dien-1-ol [16], hexa-2,4-dien-1-ol [17], 5-phenylpenta-2,4-dien-1-ol [18], and 5-phenylpent-2-en-4-yn-1-ol [19] were prepared according to the literature methods.

3.2. Reactions of dienyl and enynylindium reagents with aldehydes. Typical procedure

A mixture of penta-2,4-dienyl bromide (*E/Z* mixture) (0.12 g, 1.0 mmol), indium powder (57 mg, 0.50 mmol) and benzaldehyde (51 μl, 0.50 mmol) was stirred in DMF (2.0 cm^3) at 0°C for 3 h. The reaction mixture was quenched with dilute hydrochloric acid. The product was extracted with diethyl ether. The extracts were washed with brine and dried (Na_2SO_4). After the solvent was removed under reduced pressure, the residue was column chromatographed (silica gel; dichloromethane) to give 2-ethenyl-1-phenylbut-3-en-1-ol (72 mg, 76%). Other reactions were similarly carried out and the results are summarised in Tables 1 and 2.

3.3. 2-Ethenyl-1-phenylbut-3-en-1-ol [20,21]

^{1}H NMR: 7.17–7.44 (m, 5H, Ph), 5.58–5.97 (m, 2H, C*H*=CH$_2$), 4.88–5.32 (m, 4H, CH=C*H*$_2$), 4.60 (dd, J = 7.1, 3.2, 1H, C*H*OH), 3.05–3.17 (m, 1H, CH), 2.17 (d, J = 3.2, 1H, OH). IR (neat, cm^{-1}): 3430, 3080, 3030, 2980, 2876, 1630, 1600, 1490, 1450, 1410, 1380, 1290, 1190, 1150, 1080, 1030, 996, 910, 830, 760, 716, 696.

3.4. 2-Ethenylpent-3-en-1-ol

^{1}H NMR: 5.05–5.85 (m, 5H, olefinic), 3.49–3.55 (m, 2H, C*H*$_2$OH), 3.32 (br. quint, J = 7.5, CH, *Z* isomer) and 2.92 (br. quint, J = 7.3, CH, *E* isomer) (total 1H), 1.65–1.73 (m, 3H, Me), 1.48 (br. t, J = 6.7, 1H, OH). IR (neat, cm^{-1}): 3360, 2920, 1638, 1444, 1418, 1378, 1050, 992, 962, 918, 720.

3.5. 2-Ethenyl-1-phenylpent-3-en-1-ol [20–22]

^{1}H NMR: (major isomer) 7.41 − 7.12 (m, 5H, Ph), 4.85–5.92 (m, 5H, olefinic), 4.45–4.55 (m, 1H, C*H*OH), 3.03 (q, J = 6.8, 1H, CH), 2.29 (d, J = 2.8, 1H, OH), 1.60 (dd, J = 5.7, 1.1, 3H, Me). Peaks of minor isomers were also observed: 3.38 (m, CH), 2.32 (br. d, J = 2.8, OH), 1.73 (dd, J = 6.3, 0.82, Me), 1.36 (dd, J = 6.6, 1.6, Me). IR (neat, cm^{-1}): 3450, 3100, 3050, 3000, 2950, 2900, 1640, 1600, 1500, 1436, 1380, 1190, 1080, 1040, 1000, 970, 920, 840, 760, 702.

3.6. 4-Ethenyldodec-2-en-5-ol

^{1}H NMR: 5.02–5.90 (m, 5H, olefinic), 3.48 (br. s, 1H, C*H*OH), 2.65–3.22 (m, 1H, C*H*CH=), 1.60–1.78 (m, 3H, =CHC*H*$_3$), 1.50 (m, 1H, OH), 1.27 (s, 12H, CH$_2$), 0.88 (t, J = 6.0, 3H, CH$_2$C*H*$_3$). IR (neat, cm^{-1}): 3350, 3050, 3000, 2940, 2910, 2840, 1700, 1630, 1450, 1370, 1116, 1060, 986, 960, 906. Anal. Found: C, 79.86; H, 12.34. $C_{14}H_{26}O$ Calc.: C, 79.94; H, 12.46%.

3.7. 1-Cyclohexyl-2-ethenylpent-3-en-1-ol

^{1}H NMR: 5.00–5.94 (m, 5H, olefinic), 3.18–3.28 (m, C*H*OH and C*H*CH=) and 2.90 (q, J = 7.2, C*H*CH=) (total 2H), 0.95–1.85 (m, 15H, Me, *c*-C_6H_{11}, and OH). ^{13}C NMR (olefinic carbon): diastereomer 1:137.7, 130.9, 126.2, 116.1; diastereomer 2:139.0, 127.8, 127.6, 115.0; diastereomer 3:137.8, 129.4, 124.5, 115.7. IR (neat, cm^{-1}): 3400, 3070, 3020, 2930, 2850, 1630, 1444, 1410, 1390, 1370, 1240, 1304, 1260, 1200, 1180, 1140, 1080, 1060, 1036, 960, 940, 904, 894, 864, 838, 760, 720. Anal. Found: C, 80.56; H, 11.65. $C_{13}H_{22}O$ Calc.: C, 80.36; H, 11.41%.

3.8. 4-Ethenyl-1-phenylhepta-1,5-dien-3-ol

^{1}H NMR: (major isomer) 7.16–7.50 (m, 5H, Ph), 6.61 (dd, J = 16, 1.3, 1H, PhC*H*=CH), 6.22 (dd, J = 16, 6.2, 1H, PhCH=C*H*), 5.06–5.97 (m, 5H, olefinic), 4.17–4.26 (m, 1H, C*H*OH), 2.96 (q, J = 5.0, 1H, C*H*OH), 1.88 (d, J = 5.0, 1H, OH), 1.71 (dd, J = 6.0, 1.0, 3H, Me). Peaks of minor isomers were also observed: 3.25–3.40 (m, CH), 1.92 (d, J = 3.6, OH), 1.65 (dd, J = 6.8, 1.7, Me), 1.74 (dd, J = 7.4, 1.3, Me). IR (neat, cm^{-1}) 3400, 3075, 3020, 2970, 2900, 2875, 1660, 1630, 1594, 1574, 1490, 1444, 1370, 1200, 1150, 1060, 1020, 990, 960, 910, 830, 744, 690. Anal. Found: C, 83.78; H, 8.59. $C_{15}H_{18}O$ Calc.: C, 84.07; H, 8.47%.

3.9. 2-Ethenyl-1,4-diphenylbut-3-en-1-ol [23]

^{1}H NMR: 7.1–7.4 (m, 10H, Ph), 6.49 (d, J = 16, =C*H*Ph, minor isomer) and 6.33 (dd, J = 16, 1.0, =C*H*Ph, major isomer) (total 1H), 6.29 (dd, J = 16, 8.2, C*H*=CHPh, minor isomer) and 6.07 (dd, J = 16, 7.4, C*H*=CHPh, major isomer) (total 1H), 5.92 (ddd, J = 17, 10, 8.5, C*H*=CH$_2$, major isomer) and 5.77 (ddd, J = 17, 11, 7.1, C*H*=CH$_2$, minor isomer) (total 1H), 5.17–5.28 (m, CH=CH_2) and 5.00–5.10 (m, CH=CH_2) (total 2H), 4.46 and 4.67 (each d, J = 6.9, 1H, C*H*OH), 3.20–3.31 (m, 1H, C*H*CH(Ph)OH), 2.28 (s, 1H, OH). IR (neat, cm^{-1}): 3400, 3005, 1628, 1592, 1488, 1442, 1380, 1296, 1080, 1038, 910, 740, 682.

3.10. 2-Ethenyl-1,4-diphenylbut-3-yn-1-ol

^{1}H NMR: 7.25–7.50 (m, 10H, Ph), 5.91 (ddd, J = 17, 10, 6.8, C*H*=CH$_2$, minor isomer) and 5.72 (ddd, J = 17, 10, 5.5, C*H*=CH$_2$, major isomer) (total 1H), 5.48 (dt, J = 17, 1.6, *E*-CH=CH_2, major isomer) and 5.46 (dt, J = 17, 1.6, *E*-CH=CH_2, minor isomer) (total 1H), 5.24 (dt, J = 10, 1.6, *Z*-CH=CH_2, major isomer) and 5.30 (dt, J = 10, 1.6, *Z*-CH=CH_2, minor isomer) (total 1H), 4.74 (dd, J = 6.2, 3.9, C*H*OH, major isomer) and 4.86 (dd, J = 6.4, 3.6, C*H*OH, minor isomer) (total 1H), 3.63–3.72 (m, 1H, CH(OH)C*H*), 2.59 (d, J = 3.9, OH, major isomer) and 2.38 (d, J = 3.6, OH, minor isomer) (total 1H). IR (neat, cm^{-1}): 3450, 3045, 1640, 1598, 1490, 1452, 1400, 1042, 920, 756, 688. Anal. Found: C, 83.78; H, 8.59. $C_{15}H_{18}O$ Calc.: C, 84.07; H, 8.47%.

3.11. 2-Ethynyl-2-methyl-1-phenylbut-3-en-1-ol

^{1}H NMR: 7.22–7.50 (m, 5H, Ph), 5.77 (dd, J = 17, 10, C*H*=CH$_2$, major isomer) and 5.72 (dd, J = 17, 10, C*H*=CH$_2$, minor isomer) (total 1H), 5.56 (dd, J = 17, 1.7, *E*-CH=CH_2, major isomer) and 5.37 (dd, J = 17, 1.4, *E*-CH=CH_2, minor isomer) (total 1H), 5.26 (dd, J = 10, 1.7, *Z*-CH=CH_2, major isomer) and 5.17 (dd, J = 10, 1.4, *Z*-CH=CH_2, minor isomer) (total 1H), 4.53 (d, J = 3.3, C*H*OH, major isomer) and 4.63 (d, J = 4.1, C*H*OH, minor isomer) (total 1H), 2.43 and 2.46 (each s, total 1H, OH), 2.44 (s, 1H, CH), 1.32 (s, Me, minor isomer) and 1.20 (s, Me, major isomer) (total 3H). IR (neat, cm^{-1}): 3450, 3300, 3080, 2860, 1634, 1490, 1450, 1400, 1364, 1240, 1190, 1130, 1080, 1040, 1020, 990, 920, 820, 780, 720, 700. Anal. Found: C, 83.90; H, 7.67 $C_{13}H_{14}O$ Calc.: C, 83.83; H,7.58%.

3.12. 3-Ethynyl-3-methylundec-1-en-4-ol

^{1}H NMR: 5.78 (dd, J = 17, 11, C*H*=CH$_2$, minor isomer) and 5.73 (dd, J = 17, 10, C*H*=CH$_2$, major isomer) (total 1H), 5.48 (dd, J = 17, 1.5, *E*-CH=CH_2, minor isomer) and 5.44 (dd, J = 17, 1.5, *E*-CH=CH_2, major isomer) (total 1H), 5.22 (dd, J = 11, 1.5, *Z*-CH=CH_2, minor isomer) and 5.17 (dd, J = 10, 1.5, *Z*-CH=CH_2, major isomer) (total 1H), 3.29–3.45 (m, 1H, C*H*OH), 2.35 (s, 1H, CH), 1.78 (d, J = 4.9, OH, minor isomer) and 1.61 (d, J = 3.1, 1H, OH, major isomer) (total 1H), 1.36 (s, Me, minor isomer) and 1.30 (s, Me, major isomer) (total 3H), 1.27 (m, 12H, CH$_2$), 0.88 (t, J = 6.5, 3H, Me). IR (neat, cm^{-1}): 3450, 3300, 2925, 2105, 1638, 1558, 1404, 1378, 1260, 1064, 996, 964, 920. Anal. Found: C, 80.47; H, 11.59. $C_{14}H_{24}O$ Calc.: C, 80.71; H; 11.61%.

3.13. 2-Ethynyl-2-methylbut-3-en-1-ol

^{1}H NMR: 5.73 (dd, J = 17, 10, 1H, C*H*=CH$_2$), 5.48 (dd, J = 17, 1.5, 1H, *E*-CH=CH$_2$), 5.23 (dd, J = 10, 1.5, 1H, *Z*-CH=CH$_2$), 3.50 (s, 2H, CH_2OH), 2.36 (s, 1H, CH), 1.77 (br. s, 1H, OH), 1.32 (s, 3H, Me). IR (neat, cm^{-1}): 3400, 3300, 2890, 2110, 1640, 1450, 1406, 1306, 1268, 1048, 998, 922. Anal. Found: C, 75.67; H, 9.24. $C_7H_{10}O$ Calc.: C, 76.33; H, 9.15%.

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