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Stereospecific Reduction and Cross-Coupling of γ-Monosubstituted Allylic Chlorides Using Coordinatively Unsaturated Palladium Catalysts[1]

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A common strategy in the elaboration and transformation of allylic compounds involves transition metal activation of the allylic moiety in an initial step followed by capture of the resultant organometallic intermediates. In this regard, Pd(0)- and Pd(II)-based catalysts have emerged as highly versatile reagents, giving rise to reaction products in excellent yields and of high isomeric purity.

Stereospecific replacement of allylic functional groups by hydrogen remains a challenging problem in organic chemistry. The regio- and stereochemical outcome in these reactions is dependent on steric and electronic factors in the allylic substrate,[2-4] the allylic group being displaced,[5] the nature of the reducing agent,[5-7] and the ligands of the transition-metal catalyst.[8a,c] Excellent selectivity in favor of 1-olefin formation has been achieved in palladium-catalyzed reductions of allylic formates, esters, carbonates, phenyl ethers, chlorides, and vinyl epoxides using formates as the hydride source.[8a-c] Palladium-catalyzed reductions of (*E*)-γ-monosubstituted allylic compounds have been achieved to yield the corresponding 2(*E*)-olefins in high yields (>95%) and with a high degree of stereoselectivity.[2,3,5] Likewise, (*Z*)-γ,γ-disubstituted allylic compounds exhibit excellent regio- and stereoselectivity under similar reaction conditions.[5] However, to our knowledge, palladium-catalyzed reduction of (*Z*)-γ-*mono*substituted allylic compounds such as **3a** to 2(*Z*)-olefins has not been achieved with 100% stereospecificity.

Palladium-catalyzed cross-coupling reactions of allylic substrates with vinylorganometals has been the subject of intense investigation over the past decade.[9] While cross-coupling of γ,γ-*di*substituted allylic compounds generally proceeds with a high degree of stereo- and regioselectivity, similar reactions with γ-*mono*substituted analogues give rise to mixtures of regio- and stereoisomers.[9g-j] Superior selectivity in palladium-catalyzed reactions involving γ,γ-disubstituted allylic compounds is likely due to steric effects in the resultant allylpalladium complex. Presumably, greater steric repulsion at C_3 disfavors formation of a σ-bonded palladium–C_3 intermediate, essential for *E* to *Z* interconversion (i.e. between **7a** and **7b**, Scheme I).[10] In sterically less encumbered allylpalladium complexes therefore, isomerization is expected to be more facile. A rate of isomerization on the order of the ensuing C–H (reduction) or C–C (cross-coupling) bond forming reactions would account for the formation of regio- and stereoisomers.

Based on this line of reasoning, the possibility of capturing allylpalladium complexes prior to σ to π interconversion was investigated. The strategy we have adopted

Scheme I. Palladium-Catalyzed Reduction of γ-Monosubstituted Allylic Compounds

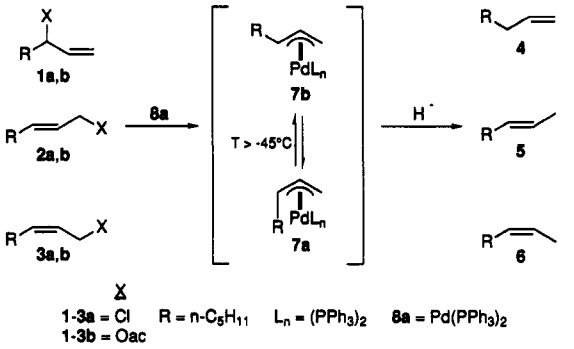

Table I. Diisobutylaluminum Hydride Capture of 3a Derived π-Allylpalladium Complex at Various Temperatures[a]

	product ratios (%)[b]			
temp, °C	4	5	6	yield (%)[b]
−78	–	–	100	100
−45	24	6	70	87
−23	42	21	37	73
0	56	18	26	76
rt	52	14	34	69

[a] Stoichiometric amount of Pd(0) used, 5 equiv of DIBAH added. [b] Product ratios and yields determined by GC.

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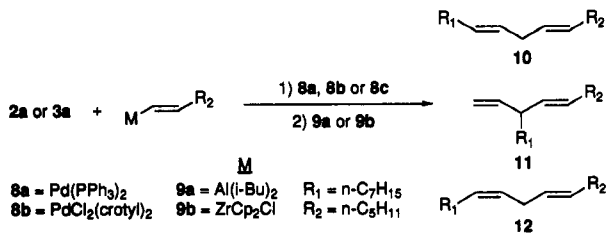
Scheme II. Palladium-Catalyzed Cross-Coupling of γ-Monosubstituted Allylic Compounds with (*E*)-Vinylorganometals



involves low-temperature oxidative addition of reactive allylic compounds to *preformed* coordinatively unsaturated palladium species, **8a**[11] and **8b**,[12] followed by addition of hydride or vinylorganometallic reagents. We wish to report that at temperatures below −45 °C DIBAH reduction of **2a** and **3a** in the presence of **8a** proceeds stereospecifically to yield **5** and **6**, respectively. We also report the stereospecific synthesis of a (*Z*,*E*)-1,4-diene in which the double bond geometry of the starting (*Z*)-γ-monsubstituted allylic chloride **3a** is maintained throughout the coupling sequence.

Results and Discussion

Reduction of γ-Monosubstituted Allylic Chlorides. Allylic chlorides and acetates have been reported to react rapidly with Pd(0) catalysts[9b] and were therefore selected for study. Oxidative addition of **2a** and **3a** to catalyst **8a**, prepared in situ by reduction of $PdCl_2(PPh_3)_2$ with 2 equiv of diisobutylaluminum hydride (DIBAH),[11] was rapid at −78 °C. Delivery of 1 equiv of DIBAH to solutions comprised of allylic substrates and **8a** at −78 °C generated exclusively **5** from **2a** and **6** from **3a** (Scheme I, Table I).[13] The involvement of **8a** in the reduction of **2a** and **3a** was established by exposing these allylic chlorides to DIBAH at −78 °C, in the absence of catalyst. Under these reaction conditions formation of products did not occur even after prolonged reaction times at room temperature.

Having established that the reduction of **2a** or **3a** with DIBAH/**8a** was stereospecific at −78 °C we determined at what temperature isomerization processes became competitive with hydride reduction. With this in mind reductions were carried out at temperatures for −78 °C to room temperature at ca. 20 °C increments.[14] It was reasoned that loss of stereochemical integrity of **7a** and **7b**, generated from **2a** and **3a**, respectively, would be reflected in the formation of mixtures of **5** and **6** upon treatment with hydride. Indeed, addition of DIBAH to these solutions at temperatures above −45 °C gave mixtures of **5** and **6**, suggesting that onset of interconversion between **7a** and **7b** occurred near this temperature (Table I). Competing processes such as β-elimination probably account for the lower yields in reductions carried out at elevated temperatures.[7] Stereospecific reduction of allylic acetates **2b** and **3b** was not possible since oxidative addition of these compounds to **8a** required elevated temperatures (ca. 0 °C).

Table II. $PdCl_2(crotyl)_2$ (8b) Catalyzed Cross-Coupling of 3a and 9b[a]

	product ratios (%)[b]			
8b[c]	**10**	**11**	**12**	yield (%)[b]
0.1	–	25	75	9
0.2	–	33	67	18
1	–	33	67	55
1[d]	–	22	78	87

[a] −78 °C to room temperature overnight. [b] Product ratios and yields determined by GC. [c] Equivalents (based on **3a**). [d] **8b** equilibrated with 5 equiv of maleic anhydride prior to addition of **3a** and **9b**.

Cross-Coupling of γ-Monosubstituted Allylic Chlorides. The mildness of these reaction conditions led us to explore the possibility of extending this methodology to cross-coupling reactions with vinylorganometals (Scheme II). The envisioned process would yield 1,4-dienes of high stereochemical purity if the starting geometry of both reacting partners was maintained throughout the coupling sequence. Vinylorganometals based on Al[15a] and Zr[15b] were selected for initial cross-coupling experiments because they are known to undergo palladium-catalyzed cross-coupling and are readily prepared by hydrometalation of terminal alkynes. Experiments carried out employing **8a** as the catalyst resulted in the loss of *Z* double bond geometry in cross-coupling reactions of **3a** and **9a**[15a] or **9b**.[15b,16] These results prompted the investigation of other, more reactive, palladium catalysts. Cross-coupling of **3a** with alkenylzirconium reagent **9b** in the presence of di-μ-chloro-di(π-crotyl)dipalladium **8b**[12] (yielding a coordinatively unsaturated palladium species in situ)[17] afforded **12** as the major product without the formation of the stereoisomer **10** (Table II).[18]

Based on Schwartz's observations that equilibration of π-allylpalladium complexes with maleic anhydride favored "head to head" coupling in palladium-catalyzed cross-coupling reactions,[9g,h] application of this approach was investigated in the hope of improving the regioselectivity in these reactions. Conducting cross-coupling reactions in the presence of maleic anhydride did not significantly affect the regioselectivity but resulted instead in a dramatic increase in product yield. Thus, reaction of **3a** with **9b** using **8b**/maleic anhydride (5 equiv based on palladium) yielded 87% of **11** and **12** (22:78). This fact along with earlier observations that yields varied directly with the

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(13) Reduction of the π-allylpalladium complex employing $LiBEt_3D$, LAH, or $NaBH_4$ was not stereospecific and gave rise to mixtures of **4**, **5**, and **6** (supplementary material).
(14) To ensure isothermal mixing, a specially designed addition tube containing DIBAH was immersed in the reaction mixture and the temperature allowed to equilibrate for 5 min. The reducing agent was then delivered to the solution by pressurizing the addition tube with N_2 (supplementary material).
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(16) Modest regioselectivity was achieved in palladium (**8a**) catalyzed reactions employing **9a** or **9b** (85:15 mixture of **10** and **11**). None of the expected (*Z*,*E*)-1,4 diene, **12**, was formed when the (*Z*)-allylic chloride, **3a**, was subjected to identical reaction conditions. Loss of *Z* double bond geometry in the allylic fragment suggested that the coupling reaction occurred above −45 °C, allowing interconversion of **7b** to **7a** prior to coupling (supplementary material).
(17) In this process, the crotyl ligands of **8b** are consumed in an initial step by an organometallic reagent (**9b**) giving rise to the desired coordinatively unsaturated palladium species. In addition, two products were formed which had shorter GC retention times than **10**–**12**. Both products gave M^+ ion peaks of 180 amu and exhibited similar mass spectral fragmentation patterns: *m*/*e* 41, 55, 68, 81, 95, 109, 123, 151, 165, 180 (M^+). This data is consistent with the formation of isomers of tridecadiene, expected in the coupling of a crotyl fragment and **9b**.
(18) As one referee astutely pointed out, formation of **12** without **10** is counterintuitive since mechanisms of stereo- and regioisomerization go hand-in-hand.[9] A plausible explanation of this apparent disparity is that formation of the Pd–C_3 intermediate is immediately followed by reductive elimination. In order to account for the present observations this process must be much more rapid than the subsequent sequence of events involved in double bond isomerization.

amount of catalyst used indicate that this palladium species is unstable and decomposes readily in the absence coordinating ligands.

In summary, stereospecific reduction of (*E*)- and (*Z*)-γ-monosubstituted allylic chlorides has been accomplished via low-temperature hydride capture of the corresponding π-allylpalladium complexes. In addition, cross-coupling of a (*Z*)-γ-monosubstituted allylic chloride with an organometallic reagent has been achieved without loss of double bond geometry. Key to these processes is the use of reactive, coordinatively unsaturated palladium species which promote C–H and C–C bond forming reactions prior to isomerization of the allylic double bond. Although the latter process furnishes predominantly "head to head" coupled products, formation of regioisomers remains a problem. Further refinement of this coupling methodology is currently underway.

Experimental Section

General. THF was freshly distilled under N_2 from K containing benzophenone as an indicator. Manipulations involving air- and moisture-sensitive reagents were carried out using standard Schlenk techniques. Purification of dienes was achieved by preparative thin-layer chromatography using SiO_2 impregnated with $AgNO_3$ (20%) as the stationary phase and hexanes (100%) as the eluant. Gas chromatographic analyses were carried out on a Hewlett-Packard Model 5890A chromatograph equipped with a flame-ionization detector and a DB-1 capillary column (15 m × 0.25 mm i.d., film thickness: 0.25 μm). Mass spectral analyses were performed on a Hewlett-Packard Model 5985B GC/MS/DS using electron impact (70 eV). Gas-phase IR spectra were obtained using a Bruker GC/FT/IR Model IFS85. ^1H and ^{13}C NMR spectra were obtained on a Bruker Model WM400 NMR spectrometer.

Preparation of Octene Isomers. Authentic samples of 1-octene,[15a] 2(*E*)-octene,[19] and 2(*Z*)-octene[15a] were prepared by standard literature procedures.

Preparation of Allylic Substrates.[20] 1-Chloro-2(*E*)-octene and 1-chloro-2(*Z*)-octene were prepared[21] from the corresponding alcohols[22,23] which were obtained by stereospecific reduction of 2-octyn-1-ol.[24] 1-Acetoxy-2(*E*)-octene and 1-acetoxy-2(*Z*)-octene were obtained by acetylation of the corresponding alcohols with acetic anhydride/pyridine.

Hydride Reduction of Allylic Chlorides in the Presence of Pd(0). To a stirred solution of $Pd(PPh_3)_2$, **8a** (0.1 g, 0.14 mmol) in THF (20 mL), cooled to the desired temperature in a specially designed reaction flask[14] was added, via cannula, *E* or *Z* isomers of 1-chloro-2-octene (0.02 g, in 2 mL of THF, 0.14 mmol). After 10 min 5 equiv of DIBAH were added to the addition tube and allowed to equilibrate with the temperature of the surrounding reaction mixture for 15 min. These reagents were then delivered to the main reaction chamber by carefully pressurizing the addition tube with N_2. Reactions were monitored by gas chromatographic analysis of aliquots removed at 10-min intervals. Octene peaks (GC trace) were identified by GC/MS and by coinjection of authentic samples. Yields were calculated using *n*-decane as an internal standard.

Coupling Reactions Using Di-μ-chloro-di(π-crotyl)dipalladium (8b).[12] A solution of **8b** (0.56 g, 0.14 mmol) in THF (20 mL) was stirred at room temperature under an atmosphere of N_2 until it had completely dissolved (ca. 5 min). The solution was then cooled to −78 °C, and **3a** (0.14 mmol in 1 mL of THF) was added via cannula, followed by vinylzirconene **9b** (1.5 equiv). Solutions were warmed overnight to room temperature. Equilibration of π-allylpalladium complexes with maleic anhydride was achieved by adding this ligand (dissolved in THF) by syringe, to cooled solutions (−78 °C) of **8b**. After stirring at −78 °C for 30 min **9b** was added, and the reaction mixture warmed to room temperature overnight.

6(*E*),8(*E*)-Heptadecadiene (10): FT/IR (vapor) 3171 (w), 2934 (s), 2864 (m), 1462 (w), 1352 (w), 984 (w), cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 6.05–5.95 (2 H, m), 5.63–5.50 (2 H, m), 2.05 (4 H, q, *J* = 7 Hz), 1.42–1.18 (18 H, 2 br s), 0.88 (6 H, t, *J* = 7 Hz); ^{13}C NMR, alkene-C, δ 132.46, 30.48; methyl and methylene-C, δ 32.53, 31.77, 31.69, 29.63, 29.40, 29.36, 29.11, 28.82, 22.57, 22.52, 13.94; MS *m/e* (%, rel int) 236 (33), 165 (3), 152 (4), 151 (5), 138 (14), 137 (8), 124 (25), 123 (17), 110 (46), 109 (42), 96 (54), 95 (69), 83 (19), 82 (68), 81 (85), 79 (44), 69 (19), 68 (25), 67 (100), 55 (17), 54 (12), 43 (13), 41 (18). Anal. Calcd for $C_{17}H_{32}$: C, 86.44; H, 13.56. Found: C, 86.55; H, 13.41.

3-Pentyl-1,4(*E*)-dodecadiene (11): FT/IR (vapor) 3084 (w), 2966 (m), 2934 (s), 2866 (m), 1637 (w), 1556 (w), 1464 (w), 1352 (w), 968 (w), 914 (w) cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.64 (1 H, ddd, *J* = 17, 10, 7.5 Hz), 5.33 (1 H, dt, *J* = 15.5, 6 Hz), 5.20 (1 H, ddt, *J* = 15.5, 7.5, 1.5 Hz), 4.92 (1 H, ddt, *J* = 17, 2, 1 Hz), 4.87 (1 H, ddt, *J* = 10, 2, 1.5 Hz), 2.54 (1 H, br quintet, *J* = 7 Hz), 1.96 (2 H, q, *J* = 7 Hz), 1.32–1.12 (18 H, m), 0.81 (6 H, t, *J* = 7 Hz); ^{13}C NMR, alkene-C, δ 142.64, 132.98, 130.48, 113.20; methyl and methlene-C, δ 46.68, 34.86, 32.54, 29.09, 29.04, 27.05, 26.73, 22.56, 22.52, 22.47, 13.92; MS *m/e* (%, rel int) 236 (3), 207 (8), 165 (16), 138 (7), 137 (29), 124 (13), 123 (27), 110 (27), 109 (61), 96 (24), 95 (88), 83 (18), 82 (27), 81 (89), 79 (39), 69 (23), 68 (37), 67 (100), 55 (21), 43 (18), 41 (23). Anal. Calcd for $C_{17}H_{32}$: C, 86.44; H, 13.56. Found: C, 86.41; H, 13.56.

6(*Z*),9(*E*)-Heptadecadiene (12): FT/IR (vapor) 3018 (w), 2966 (m), 2934 (s), 2866 (m), 1460 (w), 1385 (w), 1350 (w), 966 (w), 714 (w) cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.47–5.31 (4 H, m), 2.72 (2 H, t, *J* = 6 Hz), 2.01 (4 H, q, *J* = 7 Hz), 1.39–1.16 (16 H, bm), 0.87 (6 H, t, *J* = 7 Hz); ^{13}C NMR, alkene-C, δ 130.94, 130.56, 128.42, 127.86; methyl and methylene-C, δ 32.49, 31.80, 31.50, 30.76, 30.39, 29.64, 29.51, 29.28, 29.10, 27.04, 22.47, 22.26, 13.92; MS *m/e* (%, rel int) 236 (15), 152 (4), 151 (3), 138 (10), 137 (7), 124 (21), 123 (14), 110 (40), 109 (34), 96 (57), 95 (67), 83 (21), 81 (95), 79 (39), 69 (28), 18 (37), 67 (100), 55 (34), 54 (26), 43 (17), 41 (34). Anal. Calcd for $C_{17}H_{32}$: C, 86.44; H, 13.56. Found: C, 86.32; H, 13.41.

Supplementary Material Available: Tables of hydride reduction, hydride capture, and product ratios, apparatus figure, and in situ generation of coordinatively unsaturated palladium from **8b** (figure) (6 pages). Ordering information is given on any current masthead page.

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Reductive Decyanization of α-Amino Nitriles by Borane

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α-Amino nitriles 1 are useful intermediates for the preparation of α-amino acids (via the Strecker synthesis),[1] aldehydes, ketones,[2] enamines,[3] β-diamines,[4] and other functionalized organic compounds.[5] The synthesis of

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