There is a fundamental difference between detecting a CH6 and a CH5 proton in the $\omega 3$ dimension. If one detects CH6, and if the frequency of CH6 overlaps with the resonance of another H6 proton, nothing is gained by using the 3D experiment relative to 2D NMR because the magnetization transfer pathway (sugar protons $\xrightarrow{J_1}$ CH6 $\xrightarrow{J_2}$ CH6) is the same as the original 2D NOESY transfer. Overlap of the H6/sugar proton NOESY cross peaks remains. In addition, the true 3D cross peaks which could possibly resolve the overlap (sugar protons $\xrightarrow{J_1}$ CH5 $\xrightarrow{J_2}$ CH6) have little or no intensity because the direct NOESY magnetization transfer between sugar protons and CH5 is very inefficient.

However, if the CH5 frequency in the $\omega 3$ dimension is detected, the CH6 of the same base via the TOCSY mixing process is detected (sugar protons $\xrightarrow{J_1}$ CH6 $\xrightarrow{J_2}$ CH5). In that case if CH6 overlaps with another H6 resonance then the CH6 will be *selectively detected*. Parts A and B of Figure 3 compare two $\omega 3$ planes ($\omega 2$ = 7.32 ppm region shown only) detected at the frequency of C9 H5 or C9 H6. In the regular 2D NOESY spectrum, T8 H6 resonates at the same frequency as C9 H6 and therefore the cross peaks C9 H6/T8 H1′, C9 H6/T8 H2′, and C9 H6/T8 H2″ are undetectable because they are hidden by the cross peaks T8 H6/T8 H1′, T8 H6/T8 H2′, and T8 H6/T8 H2″. By detecting the $\omega 3$ plane at the C9 H5 frequency, one clearly sees that the signals originating from T8 H6 have disappeared. One can now detect and unambiguously assign C9 H6/T8 H1′, C9 H6/T8 H2′, and C9 H6/T8 H2″.

This single 3D spectrum provides the information for assigning all of the nonexchangeable protons including even strongly overlapping peaks in crowded spectral regions such as the H5′ and H5″ protons in a much more reliable manner than 2D NMR and greatly simplifies the process for assignment of all the protons in the system. This experiment is particularly useful for C and T bases, where the scalar coupling between CH5 and CH6, and between TH6 and TCH3, allows one to take full advantage of the increased resolution provided by the TOCSY mixing process.[25]

Acknowledgment. This work was supported by the NIH (AI27744), the Purdue University Biochemical Magnetic Resonance Laboratory, which is supported by the National AIDS Research Center at Purdue (AI727713), and the NSF National Biological Facilities Center on Biomolecular NMR, Structure and Design at Purdue (Grants BBS 8614177 and 8714258 from the Division of Biological Instrumentation). We greatly appreciate the contributions of Donna Beiswanger as well as Claude Jones and Dean Carlson for NMR discussion and those of Vikram Roongta for the synthesis of the dodecamer.

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Novel Zirconocene-Promoted Carbon–Carbon Bond Formation via a 1,2-Migration Reaction of Alkynylzirconium Derivatives

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We report on a novel class of carbon–carbon bond forming reaction of alkynylzirconocene derivatives which, we believe, proceeds via 1,2-migration as in eqs 1 and 2 (Scheme I). One distinguishing feature of the reaction is that, unlike some other known 1,2-migration reactions such as those involving α,β-unsaturated organoborates,[2] *it proceeds without the assistance of an external electrophile*. In search for new migratory insertion reactions of organotransition metals,[3-5] we generated $Li[Cp_2Zr(C{\equiv}CPh)_3]$ (**1a**) by treating Cp_2ZrCl_2, where $Cp = \eta^5\text{-}C_5H_5$, with 3 equiv of LiC≡CPh in THF at −78 to 25 °C over a few hours. Treatment of **1a** with 3 N HCl indeed produced (*Z*)-1,4-diphenyl-1-buten-3-yne (**2a**) as a >96% isomerically pure compound in 61% GLC yield based on Zr (eq 1). Similarly, the reaction of Cp_2ZrCl_2 with 3 equiv of LiC≡CC(Me)=CH_2, followed by iodinolysis with 2 equiv of I_2, produced **3c** in almost quantitative GLC yield along with 4-iodo-2-methyl-1-buten-3-yne (100% based on Zr) (eq 1).

Zirconocene derivatives containing both alkynyl and aryl groups also undergo a similar reaction. Thus, treatment of preformed $Cp_2Zr(C{\equiv}CPh)_2$[6a] with 2 equiv of PhLi (−78 to 25 °C) provided, upon quenching with 3 N HCl, (*Z*)-stilbene (**5a**) in 85% GLC yield based on Zr along with only a 6% yield of **2a**, and the reaction of Cp_2ZrPh_2[6b] with 1 equiv of LiC≡CPh provided, after treatment with 3 N HCl, >98% isomerically pure **5a** in 81% GLC yield (eq 2). The amount of **2a** was trace, if any. The use of DCl in place of HCl gave (*Z*)-dideuteriostilbene with >95% deuterium in-

(1) (a) On leave from Okayama University, Okayama, Japan. A part of this work was performed at Okayama University subsequent to the leave of absence. (b) David Ross Fellow, Purdue University (1988-1990).
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Scheme I

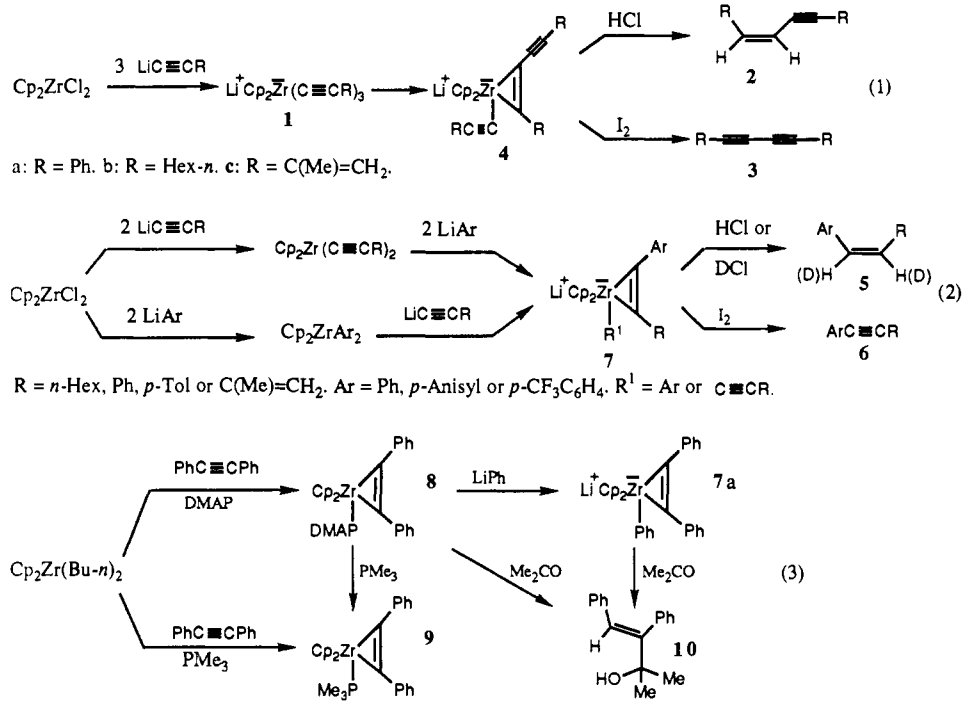

Table I. Carbon–Carbon Bond Formation of Alkynylzirconium Derivatives[a]

R^1 of $Cp_2ZrR^1_2$	LiR^2	quenching		product yield	
		reagent	product[b]	GLC	isolated
C≡CPh	LiC≡CPh	HCl	(*Z*)-PhCH=CHC≡CPh	61	
C≡CHex-*n*	LiC≡CHex-*n*	HCl	(*Z*)-*n*-HexCH=CHC≡CHex-*n*	60	55
C≡CC(Me)=CH_2	LiC≡CC(Me)=CH_2	I_2[c]	CH_2=(Me)CC≡CC≡CC(Me)=CH_2	100	
C≡CPh	LiPh[d]	HCl	(*Z*)-PhCH=CHPh	85	54
C≡CTol-*p*	LiPh[d]	HCl	(*Z*)-PhCH=CHTol-*p*		77
C≡CPh	LiC_6H_4OMe-*p*[d]	HCl	(*Z*)-PhCH=CHC_6H_4OMe-*p*		75
C≡CPh	$LiC_6H_4CF_3$-*p*[d]	HCl	(*Z*)-PhCH=CH$C_6H_4CF_3$-*p*		43
Ph	LiC≡CPh	HCl	(*Z*)-PhCH=CHPh	81	60
Ph	LiC≡CHex-*n*	HCl	(*Z*)-PhCH=CHHex-*n*	55	45
Ph	LiC≡CC(Me)=CH_2	I_2[c]	PhC≡CC(Me)=CH_2	80	60

[a] The reactions were run in THF at −78 to 25 °C for a few to several hours. [b] The alkene products were >96% *Z*. [c] Two to three molar equivalents of I_2 used. [d] Two equivalents of LiR^2 used.

corporation. The experimental results are summarized in Table I.

Although clarification of the mechanistic details is still continuing, the following are worth noting. First, $Cp_2Zr(C{\equiv}CR)_2$[6a] are generally stable for days at 25 °C, and their protonolysis provides quantitatively the starting alkynes. Therefore, the third equivalent of LiC≡CR or LiAr is essential to obtaining the products of carbon—carbon bond formation. Secondly, the following results indicate that the reaction proceeds via zirconate species and that it is not assisted by an external electrophile. Thus, for example, treatment of Cp_2ZrCl_2 with 3 equiv of LiC≡CHex-*n* at −78 to 25 °C (over 1 h) gave a Cp_2Zr derivative which exhibited a ^1H NMR singlet for the Cp group at δ 5.93. A very minor signal (≤4%) at δ 5.62 was also discernible. Quenching the mixture with 3 N HCl at this time produced only a ≤4% yield of **2b**, along with 1-octyne recovered to the extent of 90%. These results are consistent with the intermediary formation of Li[Cp_2Zr(C≡CHex-*n*)$_3$] (**1b**). The signal at δ 5.62 gradually grew, and its integration after 48 h at 25 °C corresponded to a 70% NMR yield of a new $ZrCp_2$ derivative. Analysis by GLC of the protonolysis product indicated that **2b** was produced in 60% yield by GLC based on Zr as the only octyne dimerization product. Clearly, formation of **2b** requires some transformation of the initially formed intermediate prior to protonolysis, establishing that it is not induced by HCl. Thirdly, the formation of **4** or **7** as the products before quenching with HCl or I_2 has been further supported by the following data. As reported previously, treatment of $Cp_2Zr(Bu\text{-}n)_2$ with 1 equiv of PhC≡CPh in the presence of 4-(dimethylamino)pyridine (DMAP) or PMe_3 in THF gave a 90% NMR yield of **8**[7] (δ 5.85) or a 98% NMR yield of **9**[8] [δ 5.77 (d, J = 2 Hz)], respectively. Treatment of **8** with PMe_3 cleanly provided **9** in 80% NMR yield. Significantly, treatment of **8** with PhLi (2 equiv) produced a 70% NMR yield of a $ZrCp_2$ derivative (δ 5.62) indistinguishable from that obtained by treatment of Cp_2ZrPh_2 (δ 6.41) with LiC≡CPh.[9] The structure **7a** may now be assigned to this $ZrCp_2$ derivative. Although both samples of **7a** proved to be inert to alkynes,[7,8,10] such as 4-octyne, their reaction with acetone[7,8,10] (4–6 equiv) at 25 °C for 6 h followed by treatment with 3 N HCl provided **10** in 70–75% isolated yield,[11] further supporting the assignment made above (eq 3). Unfortunately, both samples of **7a** obtained as described above contained a ca.

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(9) In sharp contrast, neither treatment of **9** with PhLi nor that of **7a** with PMe_3 at room temperature led to interconversion between **7a** and **9**. Other unsuccessful attempts to prepare **7a** include treatment of $Cp_2Zr(Cl)Me$ with PhCH=C(Li)Ph, which was complicated by facile *Z*-to-*E* isomerization of the alkenyllithium, and treatment of PhC≡CPh with $Cp_2Zr(Cl)H$, which did not give the desired hydrozirconation product in more than 5–10% yield.

15% NMR yield of another $ZrCp_2$ derivative, which was indistinguishable from a species obtained by treating 1,1-bis(η^5-cyclopentadienyl)-2,3,4,5-tetraphenyl-1-zirconacyclopentadiene with PhLi. This contamination hampered attempts to obtain **7a** as a pure compound. Furthermore, its ^1H and ^{13}C NMR spectra were relatively uninformative.[12,13] Fourthly, treatment of $Cp_2Zr(C{\equiv}CTol\text{-}p)_2$ with 2 equiv of PhLi in the presence of PhC≡CPh produced, after protonolysis with 3 N HCl, a 62% GLC yield of (*Z*)-PhCH=CHTol-*p* without producing (*Z*)-stilbene (<0,2% if any). Tolan was recovered to the extent of 86%. The results clearly rule out dissociative mechanisms, such as that proceeding via reductive elimination to give free PhC≡CTol-*p* followed by complexation, and point to a nondissociative mechanism, such as 1,2-migration.

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Supplementary Material Available: IR and ^1H and ^{13}C NMR data for product enynes and alkenes (3 pages). Ordering information is given on any current masthead page.

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(11) Under comparable conditions, **8** reacted readily with alkynes, acetone, and benzaldehyde to give the expected five-membered zirconacycles, which were protonolyzed to give the corresponding dienes and allylic alcohols.[7,8,10]

(12) Attempts to obtain crystalline samples of **7a** for X-ray analysis have so far failed.

(13) The most informative NMR data were obtained with **4c**. ^1H NMR (C_6D_6, Me_4Si): δ 1.73 (s, 3 H), 1.81 (s, 3 H), 2.33 (s, 3 H), 4.8–4.85 (m, 2 H), 4.9–4.95 (m, 1 H), 5.0–5.05 (m, 1 H), 5.25–5.3 (m, 2 H), 5.57 (s, 10 H). In addition, the ^1H signals for approximately two molecules of THF per complex were seen at δ 1.15–1.3 and 3.4–3.55. ^{13}C NMR (THF-d_8, Me_4Si): δ 23.17, 24.49, 24.83, 97.84, 104.97 (Cp), 107.75, 114.39 (CH_2), 114.91 (CH_2), 114.99 (CH_2), 126.73, 130.28, 130.77, 131.70, 134.82, 148.34, 207.32.

Regioselective and Enantioselective Substitution of Allylic Sulfoximines with Organocopper Reagents. A Versatile Approach to Optically Active Isocarbacyclins

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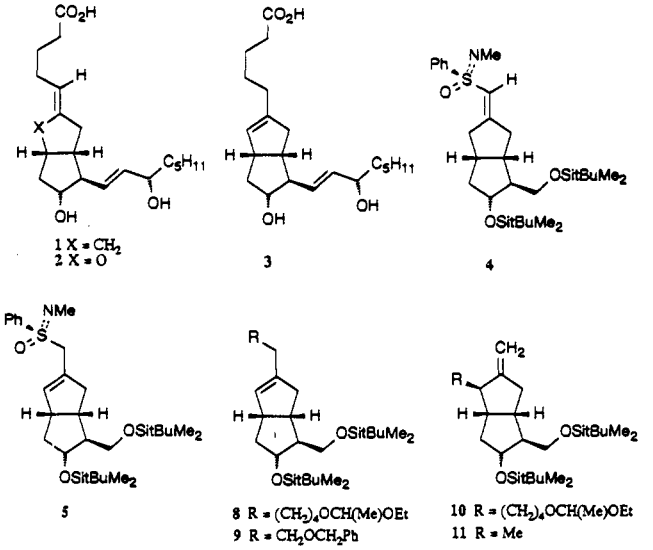
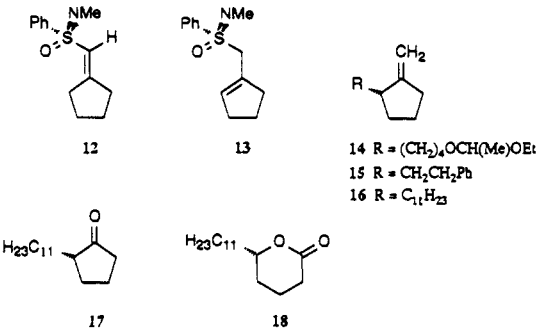
Carbacyclin (**1**)[1a] and isocarbacyclin (**3**),[1b] stable analogues of the unstable hemostase regulator prostacyclin (**2**),[1c] are the prototypes of a new generation of antithrombotic drugs. Modification mainly of their side chains has led to the attainment of highly potent derivatives[2] which show great promise for the treatment of obliterative peripheral artery disease.[3] Recently we described a stereoselective synthesis of carbacyclins via Ni-catalyzed cross-coupling reactions of the enantiomerically pure alkenyl sulfoximine **4**[4] with organozinc compounds.[5] We reasoned that **4** would likewise allow for a versatile synthesis of isocarbacyclins[6] provided it can be isomerized to the allylic sulfoximine **5** and this in turn made to allylate organocopper reagents such as, e.g., $LiCu[(CH_2)_4OCH(Me)OEt]_2$ (**6a**)[7a] or $Cu(CH_2)_4OCH(Me)OEt$ (**6b**),[7a] and $ClMgCu(CH_2OCH_2Ph)_2$ (**7a**) or $CuCH_2OCH_2Ph$ (**7b**),[7b] to give the precursors **8**[6b] and **9**,[6b,2e] respectively (Scheme I),[8] which were synthesized previously (with other protecting groups) by less direct routes.

Scheme I



Scheme II



Allylic sulfones gained synthetic importance since they allow the allylation of carbon electrophiles as well as nucleophiles via alkylation of their mono-[9] and dicarbanion[10,11f] salts and transition metal mediated substitution with organometallics or carbanion

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