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Highly Diastereoselective Synthesis of Monocyclic and Bicyclic Secondary Diorganozinc Reagents with Defined Configuration**

Andreas Boudier, Eike Hupe, and Paul Knochel*

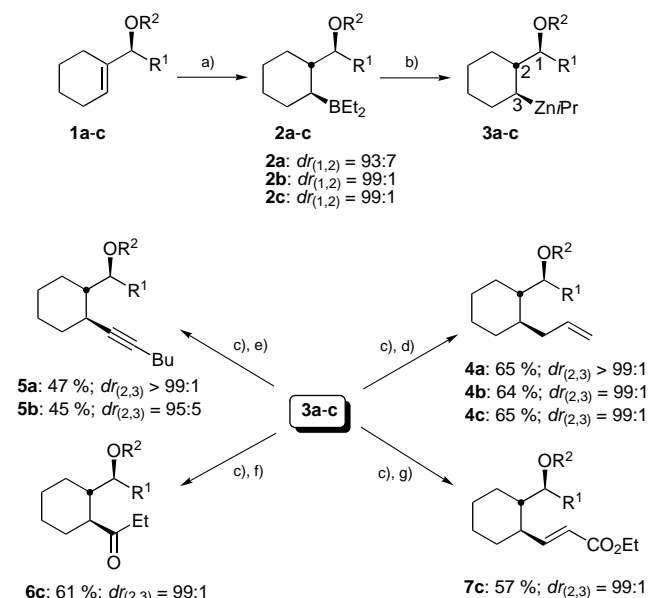
Dedicated to Professor Bernd Giese
on the occasion of his 60th birthday

The stereoselective formation of new C–C bonds is an important goal in organic synthesis. A requirement for stereoselective coupling is the availability of $\text{C}(\text{sp}^3)$ -hybridized organometallic compounds having a defined configuration.

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Organolithium compounds with high configurational stability are obtained only in strained cyclic systems or for organolithium reagents bearing a chelating heteroatom in the α -position.^[1, 2] Although remarkable progress has recently been made in this field,^[3, 4] a more general approach would be desirable. Recently, we have shown that secondary cyclic and acyclic chiral diorganozinc reagents can be prepared in the absence of any chelating heteroatom with high diastereoselectivity, allowing the stereochemical control of two adjacent stereocenters.^[5, 6, 7] Herein, we report the diastereoselective hydroboration of various monocyclic and bicyclic ring systems allowing, after subsequent boron–zinc exchange,^[8] the preparation of configurationally stable secondary dialkylzinc compounds with three adjacent chiral centers. Initially, we investigated the diastereoselective hydroboration of allylic ethers^[9] of type **1**, since the corresponding alcohols can be readily obtained in optically pure form.^[10] Thus, the hydroboration of **1a** ($\text{R}^1 = \text{Ph}$; $\text{R}^2 = \text{CH}_2\text{Ph}$) with Et_2BH in Me_2S ^[11] produces the corresponding organoborane **2a** with a diastereoselectivity of 93:7 (Scheme 1). An improved stereoselectivity of 99:1 is obtained by using an ethoxymethyl (EOM)



Scheme 1. Diastereoselective hydroboration of **1a–c**, boron–zinc exchange and reaction with electrophiles. For compounds of type **1–7**: **a**: $\text{R}^1 = \text{Ph}$; $\text{R}^2 = \text{Bn}$; **b**: $\text{R}^1 = \text{Ph}$; $\text{R}^2 = \text{CH}_2\text{OEt}$; **c**: $\text{R}^1 = t\text{Bu}$; $\text{R}^2 = \text{CH}_2\text{OEt}$. Reaction conditions: a) Et_2BH (3 equiv) in Me_2S , 50 °C, 16 h; b) $i\text{Pr}_2\text{Zn}$ (3 equiv), 25 °C, 5 h; c) $\text{CuCN} \cdot 2\text{LiCl}$ (1 equiv), –78 °C, 30 min; d) allyl bromide (3 equiv), –78 °C to 25 °C, 12 h; e) 1-bromo-1-hexyne (5 equiv), –55 °C, 2 d; f) $\text{EtC}(\text{O})\text{Cl}$ (3 equiv, –78 °C to 25 °C, 12 h; g) ethyl propiolate (3 equiv), –78 °C to 25 °C, 12 h.

protecting group (**2b**, $\text{R}^2 = \text{EOM}$). The presence of this acetal function also considerably facilitates the boron–zinc exchange, yielding secondary diorganozinc compounds of type **3**. After transmetalation with $\text{CuCN} \cdot 2\text{LiCl}$,^[12] a smooth C–C bond-forming reaction occurs with various electrophiles such as allyl bromide, 1-bromo-1-hexyne,^[13] propionyl chloride, or ethyl propiolate furnishing various polyfunctional products of type **4–7** (Table 1, entries 1–7).^[14] This one-pot sequence proceeds with good overall yields (45–73 %) and excellent

Table 1. Cu^I-mediated reactions of monocyclic secondary diorganozinc reagents with electrophiles.

Entry	Alkene ^[a]	EX ^[b]	Product	<i>dr</i> ^[c] (1,2)	<i>dr</i> ^[c] (2,3)	Yield ^[d] [%]
1		A		93:7	> 99:1	65
2		B		93:7	> 99:1	47
3		A		99:1	99:1	64
4		B		99:1	95:5	45
5		A		99:1	99:1	65
6		C		99:1	99:1	61
7		D		99:1	99:1	57
8		A		92:8	99:1	73
9		C		94:6	95:5	54
10		A		88:12	99:1	71
11		A		60:30	99:1	67
12		A		87:13	99:1	73
13		A		73:27	90:10	61
14		A		50:50	78:22	59
15		A		99:1	99:1	62

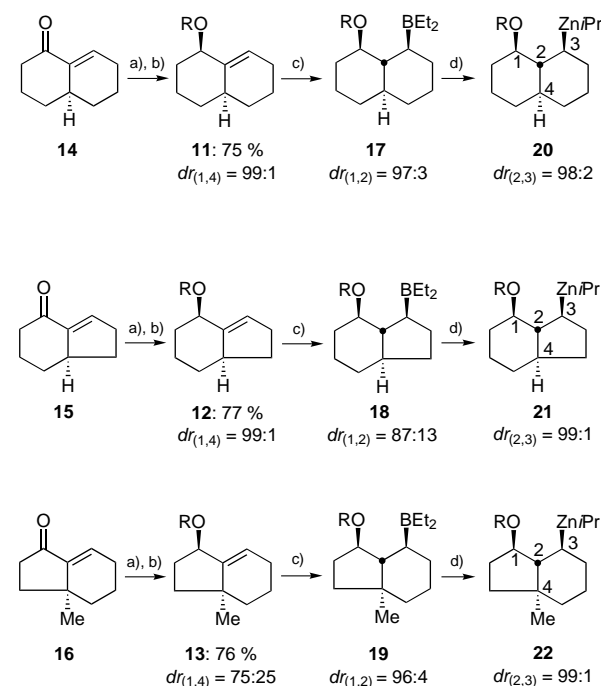
[a] Bn = benzyl, EOM = ethoxymethyl, TIPS = triisopropyl. [b] A = allyl bromide; B = 1-bromo-hex-1-yne; C = propionyl chloride; D = ethyl propiolate. [c] The diastereomeric ratio (*dr*) was determined by GC and NMR analysis of the crude product. [d] Yield of analytically pure product based on the starting alkene.

diastereoselectivities ($dr_{(2,3)} = 95:5$ to $99:1$ and $dr_{(1,2)} = 99:1$, with $R^2 = \text{EOM}$) allowing a relative control of three adjacent centers and the formation of a new C–C bond (Scheme 1 and Table 1).

The diastereoselectivity of the hydroboration is similarly dependent on the nature of the protecting group for the corresponding cyclopentene derivatives of type **8** (entries 8–15).^[15] Excellent diastereoselectivities were obtained for $R^2 = \text{Bn}$ or Me ($dr_{(1,2)}$ up to $99:1$), whereas protecting groups such as EOM or TIPS reduce dramatically the diastereoselectivity of the hydroboration (for $R^2 = \text{TIPS}$ and $R^1 = \text{Ph}$, $dr_{(1,2)} = 60:40$; for $R^2 = \text{EOM}$ and $R^1 = \text{Ph}$, $dr_{(1,2)} = 88:12$). Thus, by using the same reaction sequence as described in Scheme 1, the products **9a–g** were obtained with satisfactory overall yield. The trichloromethyl derivative **8g** ($R^2 = \text{Me}$ and $R^1 = \text{CCl}_3$) leads stereoselectively to the allylated product **10** (Table 1, entry 15).

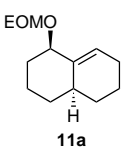
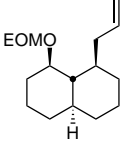
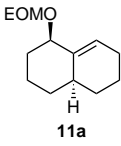
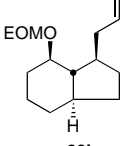
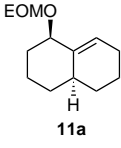
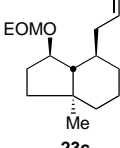
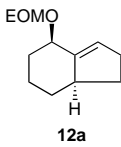
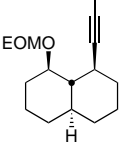
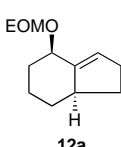
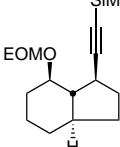
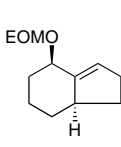
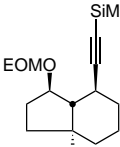
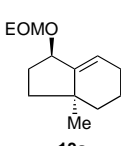
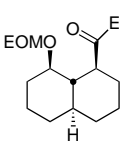
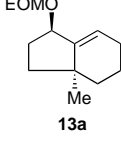
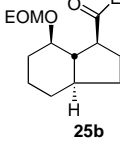
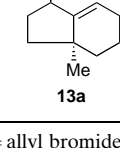
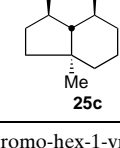
After these encouraging results, we examined the hydroboration of the bicyclic EOM-protected allylic alcohols **11**, **12**, and **13** (Scheme 2). These compounds were obtained by the reduction of the corresponding enones **14**, **15**,^[16] and **16**^[17] using $\text{NaBH}_4/\text{CeCl}_3$ (1 equiv, MeOH, 25 °C, 30 min)^[18] followed by a protection reaction with EtOCH_2Cl (Hünig's base, CH_2Cl_2 , 25 °C, 16 h).^[19]

Whereas for the enones **14** and **15** the reduction was highly diastereoselective furnishing the corresponding allyl alcohols as only one diastereomer, for the reduction of **16**, a 3:1 mixture of two separable diastereomers was



Scheme 2. Stereoselective synthesis of bicyclic secondary organozinc reagents and their reaction with electrophiles. $R = \text{CH}_2\text{OEt}$; reaction conditions: a) NaBH_4 (1 equiv), $\text{CeCl}_3 \cdot 7\text{H}_2\text{O}$ (0.4 M in MeOH, 1 equiv) 25 °C, 30 min; b) EtOCH_2Cl (1.5 equiv), Hünig's base (2 equiv), CH_2Cl_2 , 25 °C, 16 h; c) Et_2BH (3 equiv), $\text{CH}_2\text{Cl}_2:\text{Me}_2\text{S}$ (4:1), 25 °C, 48 h; d) $i\text{Pr}_2\text{Zn}$ (3 equiv), 25 °C, 5 h.

Table 2. Cu^I-mediated reactions of bicyclic secondary diorganozinc reagents with electrophiles.

Entry	Alkene	EX ^[a]	Product	<i>dr</i> ^[b] (1,2)	<i>dr</i> ^[b] (2,3)	Yield ^[c] [%]
1		A		97:3	> 98:2	65
2		B		97:3	> 99: < 1	42
3		C		97:3	94:6	59
4		A		87:13	99:1	65
5		B		87:13	> 99: < 1	43
6		C		87:13	96:4	62
7		A		96:4	98:2	59
8		B		96:4	> 99: < 1	41
9		C		96:4	94:6	61

[a] A = allyl bromide; B = 1-bromo-hex-1-yne; C = propionyl chloride. [b] The diastereomeric ratio (*dr*) was determined by GC and NMR analysis of the crude product. [c] Yield of analytically pure product based on the starting alkene.

obtained. The hydroboration of **11** under the standard reaction conditions (Et₂BH in Me₂S, 50 °C, 16 h) provided a 3:1 mixture of two diastereomeric alcohols (after oxidative work-up). However, the use of CH₂Cl₂ as cosolvent (Me₂S:CH₂Cl₂, 1:4) greatly improved the diastereoselectivity of the hydroboration (Et₂BH, 25 °C, 48 h), furnishing the organoborane **17** (*dr*_(1,2) = 97:3). Similarly, by using these improved hydroboration conditions, the bicyclic olefins **12** and **13** were converted to the corresponding organoboranes **18** (*dr*_(1,2) = 87:13) and **19** (*dr*_(1,2) = 94:6). The boron–zinc exchange of **17**–**19** proceeded smoothly with *i*Pr₂Zn (3 equiv, 25 °C, 5 h), affording the corresponding secondary organozinc reagents **20**–**22** (Scheme 2) which after transmetalation with CuCN·2LiCl react under almost complete retention of configuration (*dr*_(2,3) ≥ 96:4) with electrophiles such as allyl bromide, 1-bromoalkynes, or propionyl chloride. Bicyclic products of the type **23**–**25** have been obtained with excellent diastereoselectivities and satisfactory overall yields (Table 2).

Experimental Section

7c: A flame-dried 25-mL flask equipped with a magnetic stirring bar, an argon inlet, and a septum was charged with alkene **1c** (226 mg, 1 mmol). Et₂BH (0.4 mL, 7.3 M in Me₂S, 3 equiv) was added and the resulting mixture was stirred for 16 h at 50 °C. After the volatiles had been removed under vacuum (0.1 mm Hg, 25 °C, 2 h), *i*Pr₂Zn (0.6 mL, 5 M in Et₂O, 3 equiv) was added and the mixture was stirred 5 h at 25 °C. The boron–zinc conversion was about 85 % as monitored by GC analysis of oxidized aliquots (aqueous 3 M NaOH/aqueous 30 % H₂O₂). The volatiles were removed under vacuum (0.1 mm Hg, 25 °C, 0.5 h) and the gray-black residue was diluted with THF (4 mL) and cooled to –78 °C. A freshly prepared solution of CuCN·2LiCl (1.5 mL, 1 M in THF, 1.5 equiv) was added over 20 min. The mixture was stirred for 30 min at –78 °C. Then ethyl propiolate (294 mg, 3 mmol, 3 equiv) in anhydrous THF (2 mL) was slowly added (30 min). After stirring for 1 h at –78 °C, the mixture was allowed to warm to room temperature. Usual work-up and purification by flash chromatography (SiO₂, hexanes/Et₂O 19:1 → 9:1) afforded **7c** (186 mg; 57 %) as a colorless oil.

23a: A flame-dried 25-mL flask equipped with a magnetic stirring bar, an argon inlet, and a septum was charged with alkene **11** (210 mg, 1 mmol) in CH₂Cl₂ (2 mL). Et₂BH (0.4 mL, 7.3 M in Me₂S, 3 equiv) was added and the resulting mixture was stirred for 48 h at 25 °C. After the volatiles had been removed under vacuum (0.1 mm Hg, 25 °C, 2 h), *i*Pr₂Zn (0.6 mL, 5 M in Et₂O, 3 equiv) was added and the mixture was stirred 5 h at 25 °C. The boron–zinc conversion was about 80 % as monitored by GC analysis of oxidized aliquots (aqueous 3 M NaOH/aqueous 30 % H₂O₂). The volatiles were removed under vacuum (0.1 mm Hg, 25 °C, 0.5 h) and the gray-black residue was diluted with THF (2.5 mL) and cooled to –78 °C. At –78 °C, a freshly prepared solution of CuCN·2LiCl (0.7 mL, 1 M in THF, 0.7 equiv) was added over 1 h. The mixture was stirred for 20 min at –78 °C. Then allyl bromide (363 mg, 3 mmol, 3 equiv) in anhydrous THF (1.5 mL) was slowly added (40 min). After stirring for 1 h at –78 °C, the mixture was allowed to warm to room temperature. Usual work-up and purification by flash chromatography (SiO₂, hexanes/Et₂O = 49:1) afforded **23a** (164 mg; 65 %) as a colorless oil.

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Distance-Dependent Electron Transfer in Au/Spacer/Q-CdSe Assemblies

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Long-range electron transfer, that is, over distances of several molecular units, is an important phenomenon in biological systems; its role in protein and DNA function is currently a central issue in biological and chemical research.^[1] Long-range electron tunneling also plays a key role in

(opto)electronic devices. For instance, further miniaturization of the silicon-based metal/oxide/semiconductor transistor is seriously hindered due to electron tunneling through the oxide layer of molecular thickness.^[2] The dependence of electron transfer rate on the tunneling distance is an important topic in chemistry, biology, and physics. The electron transfer rate constant *k* between a filled (donor) and empty (acceptor) level is given by Equation (1), where *H*_{DA} describes the electronic coupling between the donor and acceptor, and *F*(Δ*G*,λ) is the Franck–Condon factor.^[3]

$$k = (2\pi/\hbar) H_{\text{DA}}^2 F(\Delta G, \lambda) \quad (1)$$

To enable elastic tunneling, a nuclear reorganization in the donor–acceptor system is often required: The expression *F*(Δ*G*,λ) accounts for the thermal activation that depends on the Gibbs free energy change Δ*G* and the reorganization energy parameter λ of the system. Due to the separation *r* between the electron donor and acceptor, the electronic coupling is much weaker than the maximum value *H*_{DA,max} and decays exponentially with *r* [Eq. (2)].

$$H_{\text{DA}}^2 = H_{\text{DA,max}}^2 e^{-\beta r} \quad (2)$$

Much research has been devoted to the experimental determination of the decay parameter β in crystalline and amorphous solids and in biologically relevant (protein, DNA) systems. Interestingly, the values for β, 0.1–1.5 Å^{−1}, are considerably smaller than the values for tunneling through a vacuum and depend markedly on the nature of the bridging molecule(s) between the donor and acceptor.^[1] An inherent problem in a reliable determination of β is the possible dependence of the Franck–Condon factor *F*(Δ*G*,λ) on the separation *r* because of Coulombic interactions in the reactant, product, and transition state.^[1, 3] The electron transfer rate is often investigated by time-resolved optical methods, for instance, fluorescence quenching of the photoexcited electron donor or acceptor. For a reliable interpretation of the data, the mechanism of excited-state decay, which involves light emission, electron transfer, and/or electron–phonon coupling, must be known. In addition, this mechanism must not change when the distance between donor and acceptor is increased by using larger bridging molecules.

Herein, we describe a class of assemblies, in which a photoexcitable entity (here a quantum dot, Q, in the form of a nanocrystalline particle) is covalently linked to a metal by spacer molecules of variable length. Relaxation of the excited state in Q may occur by two electronic transfers between the metal and Q; energy relaxation then occurs in the metal phase. Figure 1 shows an excited state in Q. Decay by two consecutive electron transfers may compete with relaxation of the excited state in Q. For this to occur, the Fermi level of the metal must be below the energy level of the excited electron in Q (the LUMO in Figure 1) but above the empty energy level (the unoccupied trap in Figure 1). Electron transfer between the energy levels in Q and the continuum of electron levels in the metal does not involve thermal activation; this means that the rate is solely determined by the electronic coupling between donor and acceptor [see Eq. (1) and (2)].

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