initiated by an enoyl-CoA isomerase catalyzed 1,3-hydrogen shift yielding **7b** and, after an additional β-oxidation cycle, 5-hydroxy-(*E*)-3-decenoyl-CoA **10**. Assuming that the 5-hydroxy group prevents a further degradation of **10** via the usual pathway, **11** can be generated by reduction of the Δ^3 double bond. This is similar to the recently described enzymatic hydrogenation of (*Z*)-5-dodecenoyl-CoA as an essential step in the metabolism of oleic acid in rat liver cells.[11] The latter pathway is supported by the fact that a 1,3-proton shift, catalyzed by enoyl-CoA isomerase, proceeds intramolecularly and by the stipulation that the CoA-group and the C2–C3 bond are rigidly fixed to the enzyme.[12]

Although further studies are required in order to fully elucidate the mechanism of the degradation of **1**, it is evident that the reactions responsible for the stereochemical outcome are independent from the presence of double bonds and are different from the proposed mechanisms previously described.[4,9,13] Additionally, the possible role of **1** in the biosynthesis of (*R*)-**12**, produced by *Sp. odorus*, was demonstrated.

Supplementary Material Available: Procedures and selected NMR, UV, and mass spectra of **1**–**5** (20 pages). This material is contained in many libraries on microfiche, immediately follows this article in the microfilm version of the journal, and can be ordered from the ACS; see any current masthead page for ordering information.

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Preparation of Functionalized Dialkylzinc Reagents via an Iodine–Zinc Exchange Reaction. Highly Enantioselective Synthesis of Functionalized Secondary Alcohols

Michael J. Rozema, AchyuthaRao Sidduri, and Paul Knochel*

The Willard H. Dow Laboratories, Department of Chemistry, The University of Michigan, Ann Arbor, Michigan 48109-1055

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Summary: Functionalized dialkylzincs are obtained by the reaction of polyfunctional alkyl iodides with Et_2Zn in excellent yield. Their treatment with aldehydes, in the presence of the titanium catalyst **6**, affords functionalized secondary alcohols with high enantioselectivity.

Dialkylzincs (R_2Zn) are important intermediates in organic synthesis since their addition to aldehydes in the presence of various chiral catalysts allows the preparation of optically active secondary alcohols with a high level of enantioselectivity.[1-3] No general preparation[4] of R_2Zn is available, and this has limited their applications in organic synthesis. For the lower terms ($R \leq Bu$) a direct insertion of zinc to an alkyl iodide followed by the distillation of R_2Zn is possible.[5] The transmetalation of lithium,[6] magnesium,[3d,7] aluminum,[8] boron,[9] and mercury[10] organometallics with zinc salts has also been frequently used for the preparation of R_2Zn. We report herein a new preparation of highly functionalized primary dialkylzincs **1** by using an iodine–zinc exchange[11] reaction (eq 1).

$$\underset{\mathbf{2}}{\text{FG-RI}} + \underset{(3\text{–}5\ \text{equiv})}{\text{Et}_2\text{Zn}} \xrightarrow[45\text{–}55\ {}^\circ\text{C},\ 1\text{–}12\ \text{h}]{\text{neat}} \underset{\mathbf{3}}{\text{FG-RZnEt}} + \text{EtI} \xrightarrow[(-\text{Et}_2\text{Zn},\ -\text{EtI})]{\substack{0.1\ \text{mmHg} \\ 40\text{–}50\ {}^\circ\text{C},\ 2\ \text{h}}} \underset{\mathbf{1}}{(\text{FG-R})_2\text{Zn}} \qquad (1)$$

Thus, the treatment of an alkyl iodide, FG-RI, **2** with an excess of diethylzinc[12] (3–5 equiv) at 45–55 °C without

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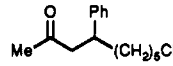
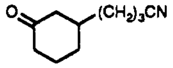
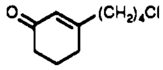
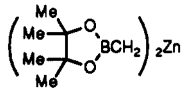
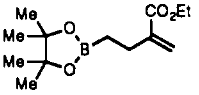
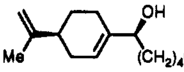
Table I. Preparation of the Functionalized Dialkylzincs 1a–g and Their Reaction with Electrophiles in the Presence of CuCN•2LiCl

$(FG\text{-}R)_2Zn$		reaction condns[a] (°C, h)	electrophile	products		yield[b,c] (%)
$(AcO(CH_2)_5)_2Zn$	**1a**	(50, 4)	nitrostyrene	$O_2NCH_2CH(Ph)(CH_2)_5OAc$	**5a**	82
	1a	(50, 4)	ethyl α-(bromomethyl)acrylate	$CH_2{=}C(CO_2Et)(CH_2)_6OAc$	**5b**	88
	1a	(50, 4)	PhCOCl	$PhCO(CH_2)_5OAc$	**5c**	87
$(t\text{-}BuCO_2CH_2)_2Zn$	**1b**	(40, 2)	$IC{\equiv}CBu$	$BuC{\equiv}CCH_2OCO\text{-}t\text{-}Bu$	**5d**	74
$(NC(CH_2)_5)_2Zn$	**1c**	(55, 40)	benzylideneacetone		**5e**[d]	72
$(NC(CH_2)_3)_2Zn$	**1d**	(50, 12)	$HC{\equiv}CCO_2Et$	$(E)\text{-}NC(CH_2)_3CH{=}CHCO_2Et$	**5f**	85
	1d	(50, 12)	cyclohexenone		**5g**[d]	83
$(Cl(CH_2)_4)_2Zn$	**1e**	(50, 20)	3-iodocyclohexenone		**5h**	86
	**1f**	(0, 1)	ethyl α-(bromomethyl)acrylate		**5i**	90

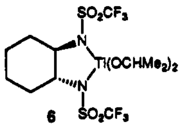
[a] Reaction conditions used to prepare the dialkylzincs 1 by the reaction of FG-RI and Et_2Zn (3–5 equiv). [b] All yields refer to isolated yields of analytically pure products. Satisfactory spectral data were obtained for all compounds (supplementary material). [c] The zinc reagent was converted to the copper reagent FG-RCu(CN)Zn(FG-R) [(CuCN·2LiCl (1 equiv, −10 to 0 °C, 5 min)] before the addition of the electrophile. [d] The reaction has been performed in the presence of Me_3SiCl (ca. 2 equiv).

Table II. Reaction of the Functionalized Dialkylzincs 1a–j with Aldehydes in the Presence of the Chiral Catalyst 6 Affording the Chiral Polyfunctional Secondary Alcohols 7a–j

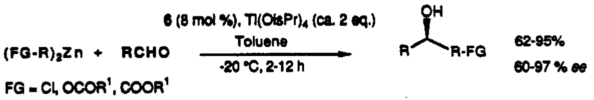
entry	$(FG\text{-}R)_2Zn$		reaction condns[a] (°C, h)	electrophile	products		% ee	yield[b] (%)
1	$(AcO(CH_2)_5)_2Zn$	**1a**	(50, 4)	PhCHO	(*S*)-$PhCH(OH)(CH_2)_5OAc$	**7a**	93	79
2		**1a**	(50, 4)	Pent-CHO	(*R*)-$PentCH(OH)(CH_2)_5OAc$	**7b**	97	62
3		**1a**	(50, 4)	c-HexCHO	(*S*)-c-$HexCH(OH)(CH_2)_5OAc$	**7c**	97	83
4	$(Cl(CH_2)_4)_2Zn$	**1e**	(50, 20)	PhCHO	(*S*)-$PhCH(OH)(CH_2)_4Cl$	**7d**	93	95
5		**1e**	(50, 20)	(*S*)-perillaldehyde		**7e**	97	95
6	$(Oct)_2Zn$	**1g**	(55, 12)	*p*-MeOPhCHO	(*S*)-*p*-MeOPhCH(OH)Oct	**7f**	94	95
7	$(AcO(CH_2)_4)_2Zn$	**1h**	(50, 4)	PhCHO	(*S*)-$PhCH(OH)(CH_2)_4OAc$	**7g**	92	72
8	$(AcO(CH_2)_3)_2Zn$	**1i**	(50, 4)	PhCHO	(*S*)-$PhCH(OH)(CH_2)_3OAc$	**7h**	86	75
9	$(t\text{-}BuCO_2(CH_2)_3)_2Zn$	**1j**	(50, 12)	PhCHO	(*S*)-$PhCH(OH)(CH_2)_3OCOt\text{-}Bu$	**7i**	92	90
10	$(EtO_2C(CH_2)_3)_2Zn$	**1k**	(55, 48)	PhCHO	(*S*)-$PhCH(OH)(CH_2)_3CO_2Et$	**7j**	60	75

[a] Reaction conditions used to prepare the dialkylzincs 1 by the reaction of FG-RI and Et_2Zn (3–5 equiv). [b] All yields refer to isolated yields of analytically pure products. Satisfactory spectral data were obtained for all compounds (supplementary material).

solvent leads to a rapid exchange (1–12 h) with the formation of an intermediate mixed dialkylzinc **3**.[13] The excess of Et_2Zn and EtI is pumped off (ca. 0.1 mmHg, 40–50 °C, 2 h) affording the dialkylzinc **1** in excellent yields. Remarkably, the reaction tolerates the presence of functional groups such as an ester, nitrile, chloride, or boronic ester (eq 1 and Table I). After the addition of CuCN·2LiCl,[14] new organocopper reagents of the type FG-RCu(CN)Zn(FG-R) **4** are formed. Compared to the zinc–copper reagents FG-RCu(CN)ZnI, the copper compounds **4** (both alkyl groups (FG-R) are transferred) display a significantly higher reactivity toward various electrophiles (nitro olefins, allylic and alkynyl halides, acid chlorides, enones, and ethyl propiolate) and under mild conditions afford a wide range of polyfunctionalized molecules **5** (Table I) in high yields (72–90%). The reaction of $(FG\text{-}R)_2Zn$ with various aldehydes (1 equiv) in the presence of a catalytic amount of the readily prepared titanium complex **6** (8 mol %)[15] and of $Ti(OisPr)_4$ (ca. 2 equiv) allows for the first time the preparation of polyfunctional secondary alcohols of type **7** (containing an ester or a halide function) in good yields (62–95%) and with excellent enantioselectivity (60–97% ee, eq 2 and Table II).[15,16] The position of the functional group in the carbon



6



(2)

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(12) Diethylzinc was purchased from Aldrich Chemical Co.

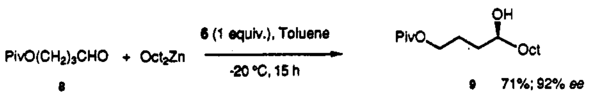
(13) The reaction mixture obtained by the reaction of iodomethyl pivalate with Et_2Zn was analyzed by ^1H NMR. After completion of the reaction, a broad signal at 3.15 ppm (attributed to $PivOCH_2ZnEt$) was observed as well as signals corresponding to EtI. After evaporation of the excess of Et_2Zn, only the signals corresponding to $(PivOCH_2)_2Zn$ were observed (supplementary material).

(14) (a) Knochel, P.; Yeh, M. C. P.; Berk, S. C.; Talbert, J. *J. Org. Chem.* **1988**, *53*, 2390. (b) AchyuthaRao, S.; Knochel, P. *J. Am. Chem. Soc.* **1991**, *113*, 5735 and references cited therein.

(15) The titanium complex **6** is derived from (−)-*trans*-cyclohexane-1(*R*),2(*R*)-diamine and has been used by Yoshioka and Ohno to catalyze the asymmetric addition of Et_2Zn to aldehydes (ref 2a,b).

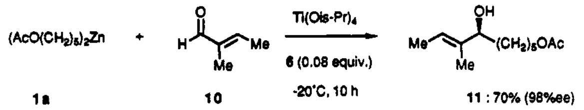
chain strongly influences the level of the stereoselectivity in these addition reactions. Thus, excellent enantioselectivites are observed if the acetoxy group is separated from the metal by four or five carbon atoms (92–93% ee, entries 1 and 7 of Table II). However, an erosion of the stereoselectivity is observed if only three carbons separate the metal from the acetoxy functionality (86% ee, entry 8). This lower enantioselectivity can be improved by replacing the acetoxy group by a pivaloyloxy group (92% ee, entry 9). The lowest selectivity has been observed with bis(3-carbethoxypropyl)zinc (**1j**) which adds to benzaldehyde affording ethyl 5-hydroxy-5-phenylpentanoate (**7j**) in only 60% ee (75% yield, entry 10).

Also, the enantioselective addition of Oct_2Zn to the 4-(pivaloyloxy)butyraldehyde **8**[18,19] is possible if stoichiometric amounts of **6** are used and allows the preparation of the monoprotected 1,4-diol **9** in 71% yield and 92% ee (eq 3).



(3)

This method can also be extended to the preparation of optically pure (98% ee) functionalized allylic alcohols.[10,8] Thus, the addition of **1a** (2 equiv) to (*E*)-2-methylbutenal (**10**) in the presence of a catalytic amount of **6** (0.08 equiv) and $Ti(OisPr)_4$ (ca. 2 equiv, −20 °C, 10 h) provides the allylic alcohol **11** in 70% yield (98% ee), eq 4.



(4)

In conclusion, we have reported a new iodine–zinc exchange reaction which allows the first preparation of functionalized dialkylzincs and their successful asymmetric addition to aldehydes in the presence of the chiral catalyst **6**. Extensions of this methodology are currently underway in our laboratories.

Acknowledgment. We thank the donors of the Petroleum Research Fund, administered by the American Chemical Society, and the University of Michigan (office of the Vice President for Research) for support of this research. We also thank S. Chi for some preliminary experiments.

Supplementary Material Available: Spectral data of the compounds described in Tables I and II (9 pages). Ordering information is given on any current masthead page.

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(16) **Typical Procedure. (a) Typical Procedure for the Reaction of the Copper Reagents FG-RCu(CN)Zn(FG-R) 4 with an Electrophile.** Preparation of 3-(3-cyanopropyl)cyclohexanone **5g**: A Schlenk tube equipped with a septum cap and an argon outlet was charged with 4-iodobutyronitrile (1.20 g, 6 mmol) and diethylzinc (3.0 mL, 30 mmol). The reaction mixture was warmed to 50–55 °C and was stirred for 12 h at this temperature. GLC analysis of a hydrolyzed reaction aliquot indicates the completion of the reaction. The ethyl iodide formed, and excess diethylzinc was removed in vacuum (50 °C, 2 h; ca. 0.1 mmHg). The resulting oil of bis(3-cyanopropyl)zinc **1d** was dissolved in dry THF (3 mL) and added to a THF solution (6 mL) of CuCN (270 mg, 3 mmol) and LiCl (255 mg, 6 mmol, dried 2 h at 150 °C under 0.1 mmHg) at −20 °C. The resulting light green solution was cooled to −78 °C, and Me_3SiCl (0.8 g, 7 mmol) and 2-cyclohexenone (0.335 g, 3.5 mmol) were added successively. The reaction mixture was slowly allowed to warm to −10 °C overnight and was worked up in the usual way to afford after purification by flash chromatography (30% AcOEt in hexane) the ketone **5g** (483 mg, 83% yield). **(b) Typical Procedure for the Catalytic Asymmetric Addition of Dialkylzinc $(FG-R)_2Zn$ 1 to an Aldehyde.** Preparation of (*S*)-(−)-6-hydroxy-6-phenylhexyl acetate **7a**: A Schlenk tube equipped with a septum cap and an argon outlet was charged with 5-iodopentyl acetate (2.05 g, 8.0 mmol) and diethylzinc (4.0 mL, 40 mmol). The reaction mixture was warmed to 50 °C and was stirred for 4 h at this temperature. GLC analysis of a hydrolyzed reaction aliquot indicates the completion of the reaction. The ethyl iodide formed, and excess diethylzinc was removed in vacuum (50 °C, 2 h; ca. 0.1 mmHg). The resulting clear viscous oil of bis(5-acetoxypentyl)zinc was dissolved in toluene (4 mL) and was added at −60 °C to the titanium catalyst **6**, followed by benzaldehyde (210 mg, 2.0 mmol). The reaction mixture was warmed to −20 °C and was stirred for 2 h. After the usual workup (using 10% aqueous HCl/ether) and flash chromatography (10% EtOAc in hexane), the alcohol **7a** (370 mg, 79% yield, 93% enantiomeric excess) was obtained as a clear colorless oil. Its optical purity was determined by the ^1H NMR analysis of its derivative with (*S*)-(−)-*O*-acetylmandelic acid.[17] **Preparation of the Catalyst.** The catalyst **6** was prepared in the following way: a three-necked flask equipped with a septum, a thermometer, and an argon outlet was charged with *trans*-1(*R*),2(*R*)-bis(trifluorosulfinamido)cyclohexane (63 mg, 0.16 mmol, 8 mol %; ref 2a,b) and titanium tetraisopropoxide (1.2 mL, 4.0 mmol) in toluene (1 mL), and the solution was warmed to 40 °C for 0.5 h.

(17) Parker, D. *J. Chem. Soc., Perkin Trans. 2*, **1983**, 83.

(18) 4-(Pivaloyloxy)butyraldehyde has been prepared from 1,4-butanediol in two reaction steps: (i) PivCl (0.4 equiv), DMAP cat., pyridine, CH_2Cl_2, 0–25 °C, 1 h; 50% yield; (ii) ClCOCOCl (1.1 equiv), DMSO (2.4 equiv), −60 °C, then Et_3N (excess), −60 to +10 °C, 95% yield.

(19) The presence of an oxygen substituent close to the carbonyl group or to the carbon–metal bond strongly changes the rate of the addition reaction. See also the excellent review article: Klumpp, G. W. *Recl. Trav. Chim. Pays-Bas* **1986**, *105*, 1.

(20) Most of the asymmetric additions have been performed on a scale using 2 mmol of aldehyde. The scale-up of the reaction (10 mmol of RCHO) is possible; comparable yields and the *same enantioselectivity* is observed. However, we noticed during the larger scale preparation of some dialkylzincs, such as **1j**, a rate decrease and a lower final conversion. This problem can be solved by the addition of a catalytic amount (0.3%) of CuI to the reaction mixture. The mechanism of the iodine–zinc exchange reaction is currently being investigated.

Asymmetric Synthesis of Calyculin A. 1. The C_1–C_{25} Spiroketal Fragment

David A. Evans* and James R. Gage

Department of Chemistry, Harvard University, Cambridge, Massachusetts 02138

Received January 3, 1992

Summary: An asymmetric synthesis of the C_1–C_{25} portion of calyculin A has been developed.

In 1986, Fusetani and co-workers reported the isolation of the unique marine natural product, calyculin A, from the sponge *Discodermia calyx*, along with its X-ray structure (relative configuration).[1] In subsequent papers, this group has reported the related structures, calyculins B–H, differing only in olefin geometry in the C_1–C_9 region

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(1) (a) Structure: Kato, Y.; Fusetani, N.; Matsunaga, S.; Hashimoto, K.; Fujita, S.; Furuya, T. *J. Am. Chem. Soc.* **1986**, *108*, 2780–2781. (b) Activity: Ishihara, K.; Martin, B. L.; Brautigan, D. L.; Karaki, H.; Ozaki, H.; Kato, Y.; Fusetani, N.; Watabe, S.; Hashimoto, H.; Uemura, K.; Hartshorne, K. *J. Biochem. Biophys. Res. Commun.* **1989**, *159*, 871–877.

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