surements and electronic structure calculations are consistent with the formation of delocalized, 1-D electronic states derived from what is formally an unpaired electron in the nonbonding orbital on a Te_3^{3-} unit through intervening Au centers. Magnetic susceptibility, conductivity, and optical measurments show that this 1-D Zintl anion exists in a Peierls distorted state exhibiting semiconducting, diamagnetic behavior. This concept of an organic-insulated conductive wire, combined with the rich structural diversity of the Zintl materials, suggest that many more materials of this type could be prepared.

Experimental Section

All manipulations were performed under helium, and en was distilled from a red solution of K_4Sn_9. An alloy of composition $K_6Ag_2Sn_2Te_9$ was prepared by fusing (about 800 °C) K_2Te (2.000 g, 9.72 mmol), Au (0.638 g, 3.24 mmol), Ag (0.698 g, 6.48 mmol), Sn (0.770 g, 6.48 mmol), and Te (2.894 g. 22.7 mmol) in a quartz tube under argon (1 atm) for 1 min with swirling. This homogeneous melt was crushed to a fine powder, extracted with en (l g alloy per 10 mL en) for 12–24 hr, and filtered to remove undissolved solid. The dark red-brown extract was added to a vial containing a saturated solution of tetraethylammonium iodide (TEA I, 0.600 g, 2.33 mmol) in en (9–10 mL). The first crystals of $(Et_4N)_4[Au(Ag_{1-x}Au_x)_2Sn_2Te_9]$, which form as tiny, radially protruding intergrowths of black blades, appear after approximately 2–3 months and continue to form for several months thereafter. Yields are typically 1–5% by weight based on alloy; it is noteworthy that there are no other solid products. Elemental analysis calcd for $(Et_4N)_4Au(Ag_{1-x}Au_x)_2Sn_2Te_9$: C 16.17, H 3.39, N 2.36, Au 13.59, Ag 6.17, Sn 9.99, Te 48.32; found: C 16.35, H 3.57, N 2.59, Au 9.25, Ag 5.26, Sn 10.42, Te 50.49.

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Keywords: semiconductors · solid-state chemistry · structure elucidation · tellurium · Zintl anions

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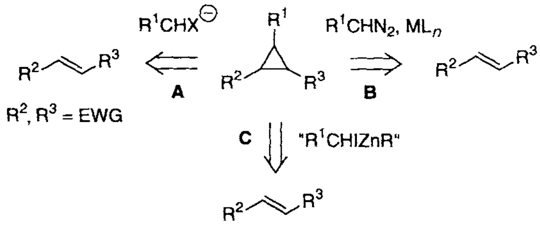
Diastereo- and Enantioselective Synthesis of 1,2,3-Substituted Cyclopropanes with Zinc Carbenoids[**]

André B. Charette[*] and Jacinthe Lemay

A significant number of natural products containing 1,2,3-substituted cyclopropanes have been isolated in the last few decades.[1, 2] Traditionally, these cyclopropane units have been prepared by a nucleophilic addition–elimination sequence on α,β-unsaturated carbonyl derivatives (Scheme 1, **A**)[3] or by a cyclopropanation reaction involving the transition metal catalyzed decomposition of diazoesters (**B**).[4, 5] It is clear that the diastereo- and enantiocontrol is a serious limitation in some of these methods. The use of the Simmons–Smith cyclopropanation reaction[6] to generate 1,2,3-substituted cyclopropanes is very rare, and so far no method has provided good diastereo- and enantiomeric levels (**C**).[7, 8] In this communication, we report a new method to generate 1,2,3-substituted cyclopropanes with excellent diastereo- and enantiocontrol by using substituted zinc carbenoids, and we show for the first time that functionalized zinc carbenoids[9] can be prepared and are effective cyclopropanating reagents.



Scheme 1. Different strategies for the cyclopropanation of olefins (EWG = electron-withdrawing group).

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[*] Prof. A. B. Charette, J. Lemay
Département de Chimie
Université de Montréal
P. O. Box 6128, Station Downtown, Montréal H3C 3J7 (Canada)
Fax: Int. code +(514)343-5900
e-mail: charetta@ere.umontreal.ca

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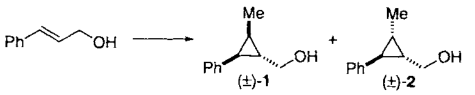
The use of substituted diiodoalkanes as precursors to substituted zinc carbenoids was first reported by Kawabata and Furukawa.[10, 11] Very modest product ratios (1.6–5.4:1) and yields (23–85%) were obtained when allylic alcohols were treated with a mixture of 1,1-diiodoethane (2.0 equiv) and Et_2Zn (1.5 equiv). Since we have already shown that the stoichiometry of the diiodoalkane and Et_2Zn is very important for optimizing diastereomeric ratios and yields,[12] we have initially surveyed different reaction conditions to optimize the diastereoselectivities in the achiral version of this reaction (Table 1). Cyclopropanes **1** and **2** were obtained in greater than 10% yield when cinnamyl alcohol was treated with 1,1-diiodoethane and Et_2Zn under Kawabata's conditions (entry 1). However, the yield, diastereoselectivity, and rate of the reaction increased dramatically if CH_2Cl_2 was used as the solvent (entry 2) or if a twofold excess of the preformed reagent[13] in CH_2Cl_2 was used (entry 3). Finally, significantly good diastereomeric ratios were also observed with five equivalents each of Et_2Zn and CH_3CHI_2 but the yield dropped to 58% (entry 4). These data clearly illustrate the importance of using a noncomplexing solvent in this reaction.

Table 1. Diastereoselective cyclopropanation of cinnamyl alcohol [a].

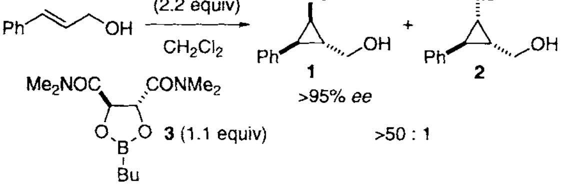


(±)-**1** (±)-**2**

Entry	Et_2Zn [equiv]	CH_3CHI_2 [equiv]	Yield [%] [b]	Ratio (**1**:**2**) [c]
1	1.5	2.0 [d]	<10	–
2	1.5	2.0	85	93:7
3	2.2	4.4 [e]	82	89:11
4	5.0	5.0	58	93:7

[a] Unless otherwise stated, all the reactions were carried out at 0 °C in CH_2Cl_2. [b] Yield of isolated product from chromatographically homogeneous material. [c] Determined by ^1H NMR spectroscopy (400 MHz) and by GC of the crude reaction mixture. [d] Solvent: *t*BuOMe. [e] The reagent was preformed prior to the addition of cinnamyl alcohol.

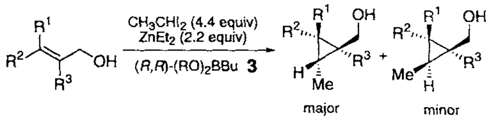
The addition of the chiral dioxaborolane ligand **3**[14] to the reaction had a spectacular effect on stereocontrol in the enantioselective version of this cyclopropanation reaction (Scheme 2). The treatment of a mixture of cinnamyl alcohol and **3** with 2.2 equivalents of the preformed reagent in CH_2Cl_2 at 0 °C resulted not only in a significant increase in the diastereomeric ratio (from 93:7 to 98:2), but also gave an outstanding enantiomeric excess of the major diastereomer.[15]

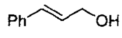
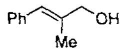
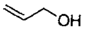
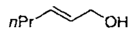


Scheme 2. Enantioselective cyclopropanation with the chiral dioxaborolane **3**.

Several substituted allylic alcohols were submitted to these reaction conditions, and the results are illustrated in Table 2. A particularly striking feature is that excellent diastereo- and enantioselectivities were obtained regardless of the olefin configuration. In all the cases, the relative stereochemistry of the new chiral center was *anti* to the CH_2OH group. The substantial increase in the diastereoselectivity of the reaction mediated by dioxaborolane **3** relative to the achiral version is presumed to be the result of an increase of the steric size of the CH_2OZnR group when bonded to the chiral ligand **3**.

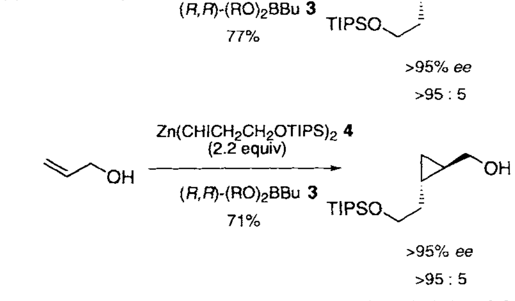
Table 2. Enantioselective cyclopropanation of allylic alcohols [a].



Entry	Allyl alcohol	*ds* [b]	*ee* [%] [c]	Yield [%] [d]
1		>50:1	98	96
2		14:1	90	83
3		>50:1	94	80
4		20:1	90	84 [e]
5		15:1	94	87 [e]
6		10:1	93	80 [e]

[a] All the reactions were carried out at 0 °C in CH_2Cl_2. [b] The diastereoselectivities were determined by ^1H NMR (400 MHz) and capillary GC analysis of the crude reaction mixture. [c] Determined by NMR analysis of the derived Mosher's esters (entry 2–4), by capillary GC analysis on chiral stationary phase (entry 1), or by conversion into the chiral α-methylbenzylamide. [d] Yield of isolated product from chromatographically homogeneous material. [e] The yield refers to the corresponding benzoate that was isolated by treating the crude mixture with PhCOCl.

On the basis of these results, we hypothesized that we could use functionalized iodoalkylzinc reagents in these enantioselective cyclopropanation reactions. Knochel has extensively shown that organozinc reagents are compatible with a variety of functional groups[16] but the related functionalized iodoalkylzinc reagents have never been prepared and used as cyclopropanating reagents. This variant would greatly enhance the scope of the reaction, since cyclopropanation with substituted iodoalkylzinc reagents has been limited to $CH_3CHIZnX$ and $PhCHIZnX$.[10, 11, 17] Furthermore, a variety of substituted diiodoalkanes are directly accessible by the alkylation of diiodomethane.[18] The following examples clearly show the potential of this methodology as a convergent synthesis of structurally complex, polysubstituted cyclopropanes. A single isomer was obtained when cinnamyl alcohol or allyl alcohol was



Scheme 3. Reaction of cinnamyl or allyl alcohol with **4** and the chiral ligand **3** (TIPS = triisopropylsilyl).

treated with 2.2 equivalents of **4** (prepared from 1-triisopropylsilyloxy-3,3-diiodopropane[19] and Et_2Zn) and 1 equivalent of **3** (Scheme 3). Furthermore, the diastereo- and enantioselectivity for these processes were excellent.[20]

In summary, we have developed an expedient and efficient synthesis of enantiomerically enriched 1,2,3-substituted cyclopropanes from allylic alcohols. In addition, we have presented the first cyclopropanation reaction of a functionalized iodoalkylzinc reagent. Applications of this methodology to natural product synthesis is currently underway.

Experimental Section

To a solution of Et_2Zn (113 µL, 1.1 mmol) in CH_2Cl_2 (5 mL) at 0 °C was added CH_3CHI_2 (222 µL, 2.2 mmol) over 5 min. After 10 min of stirring at 0 °C, a solution of the allylic alcohol (0.5 mmol) and (*R,R*)-dioxaborolane (**3**) (149 mg, 0.55 mmol) in CH_2Cl_2 (2 mL) was added rapidly by cannula. The ice bath was removed, and the reaction was stirred until TLC analysis showed complete consumption of the starting material (1 h). The reaction mixture was then poured into saturated aqueous NH_4Cl (20 mL), and 10% aqueous HCl (10 mL) was added. The mixture was extracted three times with ether (10 mL), and the combined organic layers were stirred vigorously with 5 M aqueous KOH (15 mL) for 12 h, washed with saturated aqueous Na_2SO_3 (15 mL), with saturated aqueous NH_4Cl (15 mL), and with saturated aqueous NaCl (15 mL), dried over $MgSO_4$, and concentrated under reduced pressure. The residue was purified by flash chromatography to afford the desired cyclopropane.

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Keywords: asymmetric synthesis · carbenoids · cyclopropanations · iodine · zinc

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[19] Prepared in three steps from diiodomethane by the following sequence: a) LiHMDS, allyl bromide (63%); b) O_3, $NaBH_4$ (79%); c) TIPSOTf, Et_3N (94%).

[20] Quite surprisingly, the desired cyclopropane was obtained only in the presence of the chiral ligand. It is conceivable that the chiral ligand plays the important role of stabilizing the reagent. 1-Triisopropylsilyloxy-2-propene that is presumably generated by the decomposition of the carbene was isolated as the major by-product.

Generation of Alkyl(dicarbonyl)(chloro)ruthenium Dimers in the Ruthenium-Catalyzed Addition of Alkyl Formates to Ethylene**

Sylvie Fabre, Philippe Kalck,* and Guy Lavigne*

The complex $[PPN][Ru(CO)_3(Cl)_3]$ (**1**, PPN = $(PPh_3)_2N^+$) is a convenient catalyst precursor for the hydroesterification of ethylene with methyl formate to furnish methyl propionate[1] (100% conversion, 99% selectivity, initial turnover frequency $TOF_0 = 700\ h^{-1}$).[1–3] To clarify the nature of the active species, we terminated the reaction by freezing once the maximum activity was reached. This gave a solid whose ν(CO) absorption bands [2017(s), 1945(s) cm^{-1}] appeared at slightly higher wavenumbers relative to the active catalyst solution [2003(s, br), 1918(s, br) cm^{-1}]. The solution NMR spectrum of this material indicated that it consists of a mixture of three closely related species **2–4**, which exhibit signals characteristic of Ru-bound ethyl and methyl groups.

$[PPN][Ru(CO)_3(Cl)_3]$ **1**

$[PPN][Ru_2(\mu\text{-}Cl)_3(\eta^1\text{-}CH_2CH_3)_2(CO)_4]$ **2**

$[PPN][Ru_2(\mu\text{-}Cl)_3(\eta^1\text{-}CH_2CH_3)(\eta^1\text{-}CH_3)(CO)_4]$ **3**

$[PPN][Ru_2(\mu\text{-}Cl)_3(\eta^1\text{-}CH_3)_2(CO)_4]$ **4**

Single crystals obtained from this mixture were shown by X-ray diffraction analysis to consist of a statistical packing of isomorphous units of these three complexes, which differ only in the nature of the alkyl groups.[4] The first of these compounds to be obtained in pure form was **2**, the crystal structure of which is shown in Figure 1.[5] This is the first ternary alkyl(carbonyl)-

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[*] Prof. P. Kalck, S. Fabre
Ecole Nationale Supérieure de Chimie de Toulouse
Institut National Polytechnique
118 route de Narbonne, F-31077 Toulouse Cedex (France)
Fax: Int. code + 561175600

Dr. G. Lavigne
Directeur de Recherches CNRS
Laboratoire de Chimie de Coordination associé à l'Université Paul Sabatier et à l'Institut National Polytechnique
205 route de Narbonne, F-31077 Toulouse Cedex (France)
Fax: Int. code + 561553003
e-mail: lavigne@lcctoul.lcc-toulouse.fr

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