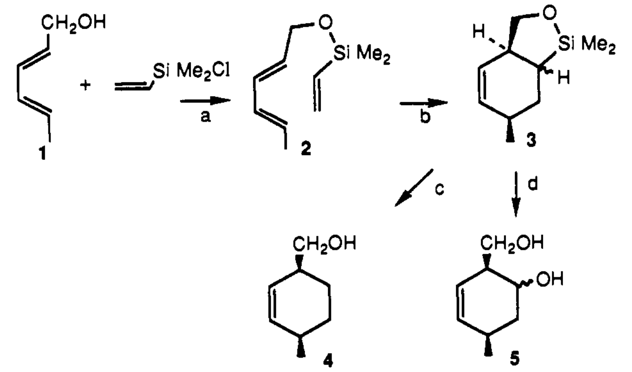
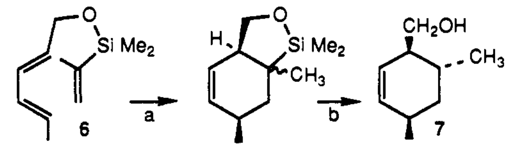
**Scheme I**$^a$



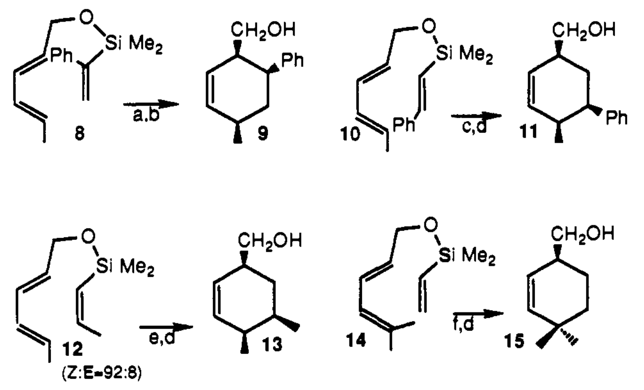
$^a$Conditions: (a) Et$_3$N-THF; (b) 160 °C,[2] 3.5 h; 70%; (c) 4 equiv of TBAF-DMF, 75 °C, 4 h; 75%; (d) 1 equiv of TBAF-DMF, 10 equiv of 30% H$_2$O$_2$, 55 °C, 2 h; 85% (cis-1,2:trans-1,2 = 70:30).

**Scheme II**$^a$



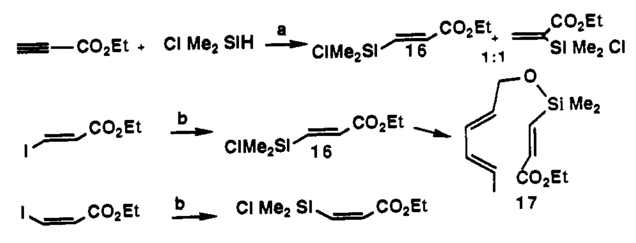
$^a$Conditions: (a) 160 °C, 5 h; 80%, 2 isomers (60:40); (b) as in **3** → **4**; 65% (1 isomer; see footnote 7).

**Scheme III**$^a$



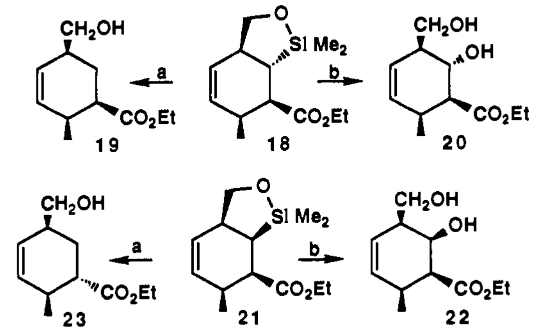
$^a$Conditions: (a) 160 °C, 4 h; 75%; (b) TBAF-DMF; 85%; (c) 180 °C, 10 h; 80%; (d) TBAF-DMF, 85% (cis-4,5:trans-4,5 = 85:15); (e) 200 °C, 10 h; 45% (cis-4,5:trans-4,5 = 81:19); (f) 200 °C, 30 h; 50–60%.

**Scheme IV**$^a$



$^a$Conditions: −100 °C; then 1 equiv of acetyl chloride.

**Scheme V**$^a$



$^a$Conditions: (a) 2 equiv of TBAF; DMF, 60 °C, 4 h; 75%; (b) as in **3** → **5**; 80%.

context. This proved very successful. We were able to prepare the (*E*)-(chlorosilyl)acrylate **16** in two different ways, as shown in Scheme IV: as a component of a 1:1 mixture of regioisomers[11] or, regiospecifically, from (*E*)-ethyl 3-iodoacrylate.[12,13] Addition of **16** to **1** then gave the silicon-tethered acrylate **17** (50% from the iodoacrylate).

Cyclization of **17** readily (4–6 h, 80 °C) gave **18** (see Scheme V), the product of endo addition, in 90% yield. A similar sequence starting with (*Z*)-ethyl 3-iodoacrylate[12] gave the corresponding product of endo addition, as judged by its conversion to **22**. In this particular case, however, the initial, unstable, all-cis adduct **21** was epimerized by fluoride ion in the course of the desilylation process, with the formation of **23**.

The important feature of *controlled regiochemistry* which the temporary silicon connection can afford in the context of 4 + 2 cycloadditions is particularly well illustrated by the syntheses of **20** and **22**, in which, in addition, four new asymmetric centers are generated stereospecifically. Regiocontrol is also notable in the syntheses of **9** and **11**, inter alia.

**Acknowledgment.** We thank the National Science Foundation and the National Institutes of Health for their support of this work.

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[11] Both isomers can be successfully used in silicon-tethered 4 + 2 cycloadditions, e.g., to **1**.

[12] Biougne, J.; Theron, F. *C. R. Seances Acad. Sci. Ser. C* **1971**, *272*, 858.

[13] Use of chlorosilyl rather than (dimethylamino)silyl derivatives is necessary when electrophilic olefins are involved to avoid conjugate addition of the dimethylamine released during silyl ether formation.

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## New Preparation of α-Methylene-γ-butyrolactones Mediated by (Iodomethyl)zinc Iodide

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A number of α-methylene-γ-butyrolactones display significant biological activity, and many syntheses of this class of molecules have been described.[2] We report a new stereoselective one-pot

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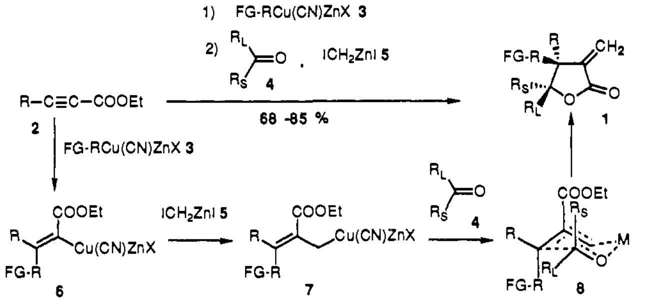
**Table I.** $\alpha$-Methylene-$\gamma$-butyrolactones **1** Prepared by the Reaction of an Acetylenic Ester **2**, (FG-R)Cu(CN)ZnX **3**, a Carbonyl Compound **4**, and (Iodomethyl)zinc Iodide (**5**)

| entry | R | FG-R | $R_L$ | $R_S$ | product 1 | cis:trans ratio[b] | yield (%)[a] |
|---|---|---|---|---|---|---|---|
| 1 | H | Bu | c-Hex | H | **1a** | 80:20 | 76 |
| 2 | H | $PhCH_2$ | Ph | H | **1b** | 92:8 | 78 |
| 3 | H | $NC(CH_2)_3$ | Ph | H | **1c** | 90:10 | 75 |
| 4 | H | $BuC{\equiv}C(CH_2)_2$ | Ph | H | **1d** | 95:5 | 76 |
| 5 | H | $EtO_2C(CH_2)_3$ | Ph | H | **1e** | 95:5[c] | 85 |
| 6 | H | $EtO_2C(CH_2)_3$ | $(CH_2)_5$ | | **1f** | | 68 |
| 7 | H | $Cl(CH_2)_4$ | Ph | $CH_3$ | **1g** | 100:0[c] | 82 |
| 8 | $EtO_2C$ | $EtO_2C(CH_2)_3$ | $PhCH_2CH_2$ | H | **1h** | 85:15 | 86 |
| 9 | $EtO_2C$ | $NC(CH_2)_3$ | c-Hex | H | **1i** | 95:5 | 93 |
| 10 | Bu | c-Hex | Ph | H | **1j** | 75:25 | 60 |
| 11 | c-Hex | Bu | Ph | H | **1j** | 98:2[c] | 67 |
| 12 | Bu | Ph | Ph | H | **1k** | 60:40 | 78 |
| 13 | Ph | Bu | Ph | H | **1k** | 98:2 | 85 |

[a] All yields refer to isolated yields of analytically pure products. [b] Crude GC ratios. [c] Relative stereochemistry determined by $^1H$ NMR NOE experiments or X-ray analysis (see supplementary material).

**Scheme I**



synthesis of highly functionalized $\alpha$-methylene-$\gamma$-butyrolactones **1** starting from an acetylenic ester **2**, a readily prepared zinc–copper reagent[3] **3**, an aldehyde or a ketone **4**, and (iodomethyl)zinc iodide[4] (**5**) (Scheme I and Table I).

The carbocupration of acetylenic esters[5] **2** with (FG-R)Cu(CN)ZnX (FG = functional group) provides stereoselectively the alkenylcopper **6** (THF, $-50$ °C, 3 h). The reaction mixture is then treated with a mixture of a carbonyl compound **4** (0.7 equiv) and (iodomethyl)zinc iodide in THF (2 equiv). The vinylic copper reagent **6** is not reactive enough to add to an aldehyde or ketone **4**; however, it is readily homologated ($-30$ to 0 °C) by a methylene unit[6] with (iodomethyl)zinc iodide (**5**), affording in situ a highly reactive polyfunctional allylic zinc and copper species **7**, which reacts stereoselectively with the carbonyl compound **4**, giving, after workup, the $\alpha$-methylene-$\gamma$-butyrolactone **1**. The groups R of the alkyne, FG-R of the zinc–copper organometallic, and $R_L$ of the aldehyde can bear a wide range of functionalities (ester, nitrile, halide, triple bond), allowing a unique approach to new $\alpha$-methylene-$\gamma$-butyrolactones[7] **1**. A cis relationship is always found between the substituents $R_L$ and FG-R in the major diastereoisomer[8] of **1**, which is compatible with the chair transition state[9] **8**. The stereochemistry of lactone **1** depends on the stereochemistry of the vinylic copper **6**. If the carbocupration can be performed at low temperature (below $-30$ °C), the resulting syn-adduct **6** is configurationally stable at this reaction temperature and affords, after methylene homologation and reaction with a carbonyl compound, the cis lactone as the major diastereoisomer (entries 1–9). Thus, the addition of BuCu(CN)Li to ethyl 3-cyclohexylpropiolate provides stereospecifically the alkenyl copper **6j** (R = c-Hex, FG-R = Bu), as confirmed by low-temperature iodolysis ($-30$ °C) giving only (*E*)-ethyl 3-cyclohexyl-2-iodo-2-heptenoate. The methylene homologation of **6j** followed by reaction with PhCHO gives the lactone **1j** (cis:trans, 98:2; entry 11). However, if the alkenylcopper **6j** obtained as described previously is allowed to warm to $-10$ °C, a mixture of (*E*)- and (*Z*)-alkenyl iodides is obtained after iodolysis (*E*:*Z* = 75:25). Also the carbocupration of ethyl 2-heptynoate with c-HexCu(CN)ZnI·2LiCl occurs only above $-10$ °C and leads under these reaction conditions to the same *E*/*Z* mixture of the alkenylcopper **6j** (*E*:*Z* = 75:25 as shown by iodolysis). After methylene homologation and reaction with benzaldehyde, the butyrolactone **1j** is produced as a 75:25 cis–trans mixture (entry 10). Similar behavior is observed in the addition of PhCu(CN)Li and BuCu(CN)Li respectively to ethyl 2-heptynoate (entry 12) and ethyl 3-phenylpropiolate (entry 13).

The intramolecular version of the reaction is also possible, and the addition of the keto-substituted copper[10] reagent **9** to ethyl propiolate and diethyl acetylenedicarboxylate provides, after the addition of $(ICH_2)_2Zn$[11] and benzaldehyde (THF, $-35$°, 0.5 h), the bicyclic $\alpha$-methylene lactones **10a** and **10b** in respectively 76% and 83% yields (eq 1).

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(7) Typical procedure: preparation of 4-(3-cyanopropyl)-4,5-dihydro-3-methylene-5-phenyl-2(3*H*)-furanone (**1c**). A THF solution of 4-iodobutyronitrile (1.38 g, 7.1 mmol in 4 mL of THF) was added at 25 °C to zinc dust (1.3 g, 20 mmol pretreated with 1,2-dibromoethane (0.3 g, 1.5 mmol) and $Me_3SiCl$ (0.1 mL)).[5] An exothermic reaction occurred, and the reaction mixture was stirred for 1.5 h at 40 °C. The excess of zinc was allowed to settle, and the supernatant solution was added to a solution of CuCN (0.635 g, 7.1 mmol) and LiCl (0.6 g, 14 mmol) in THF (10 mL) at $-10$ °C. After 5 min, the reaction mixture was cooled to $-60$ °C, ethyl propiolate (0.588 g, 6 mmol) was added, and the mixture was stirred for 4 h between $-60$ and $-40$ °C. A solution of PhCHO (0.53 g, 5 mmol) in THF (2 mL) was added, followed by the dropwise addition of a THF solution of (iodomethyl)zinc iodide (ca. 10 mmol) prepared from $CH_2I_2$ (3.75 g, 14 mmol) and zinc foil (0.91 g, 14 mmol) in THF (8 mL) at 25–26 °C (3 h).[5] The reaction mixture was allowed to warm to 0 °C and was stirred for 0.5 h at this temperature. After the usual workup, the residual oil was purified by flash chromatography, affording **1c** as a cis–trans mixture (90:10, 0.9 g, 75% yield).

(8) The relative stereochemistry of the $\alpha$-methylene-$\gamma$-butyrolactone substituents has been established by comparisons with the literature, $^1H$ NMR NOE experiments, or X-ray structure determinations (**1g** and **1j**; see supplementary material).

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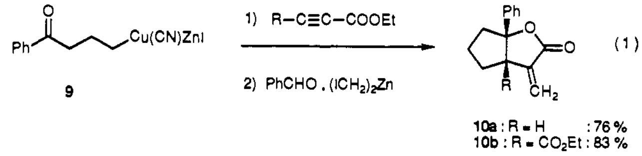

In conclusion, we have developed a new, efficient one-pot synthesis of polyfunctionalized α-methylene-γ-butyrolactones starting from readily available acetylenic esters, $ICH_2ZnI$, (FG-R)Cu(CN)ZnX, and an aldehyde or ketone. Applications to the synthesis of natural products are currently underway.

**Acknowledgment**. We thank the donors of the Petroleum Research Fund, administered by the American Chemical Society, for support of this research.

**Supplementary Material Available:** Spectral data for **1a–k** and **10a,b** and X-ray crystallographic analyses of **1g** and **1j** (52 pages). Ordering information is given on any current masthead page.

## Preparation, Characterization, and Sequential Transformation of Dicarbide Cluster Compounds with Permetalated Ethyne, Ethene, and Ethane Structures

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The structures and properties of the $C_2$ ligand incorporated in polymetallic systems have been studied as models for elementary species on a heterogeneous catalyst surface, and several coordination modes have been reported.[1] Although ethynediyldimetal complexes (MC≡CM) are expected to serve as versatile starting compounds, this route has been frustrated by their limited accessibility. We[2a,b] and other groups[3] recently developed a preparative method for MC≡CM via deprotonation of $[M_2(\mu\text{-}C\equiv CH)]^+$. Herein we report the utility of Fp*C≡CFp* (**1**)[2a,b] [Fp* = $Cp^*Fe(CO)_2$; Cp* = $\eta^5\text{-}C_5Me_5$] as a building block for a series of *dicarbide* cluster compounds.

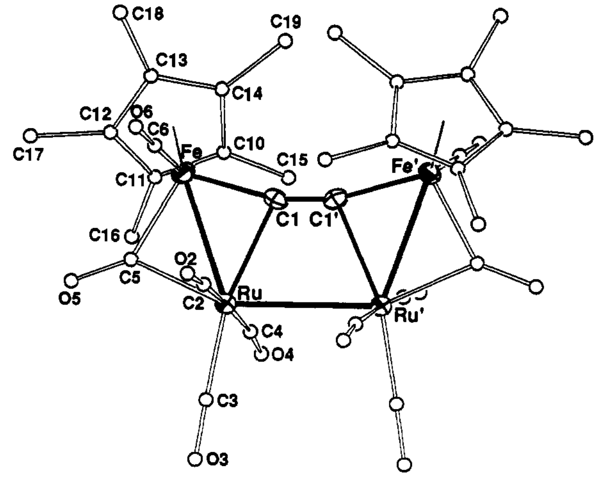


**Figure 1**. Molecular structure of **2**. Selected bond lengths (Å) and angles (deg): Ru–Ru, 2.963 (2); Ru–Fe, 2.733 (2); Ru–C1, 2.204 (7); Fe–C1, 1.946 (7); C1–C1′, 1.24 (1); Fe–Ru–Ru′, 110.61 (4), C1–Ru–Ru′, 66.5 (2); Fe–Ru–C1, 44.9 (2), Ru–Fe–C1, 53.0 (2); Ru–C1–Fe, 82.1 (2); Ru–C1–C1′, 112.0 (2); Fe–C1–C1′, 165.5 (2).

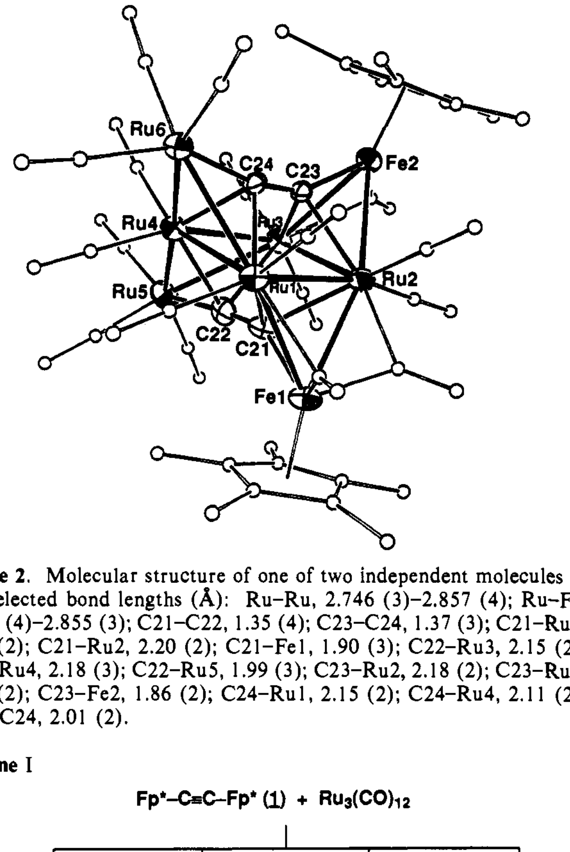
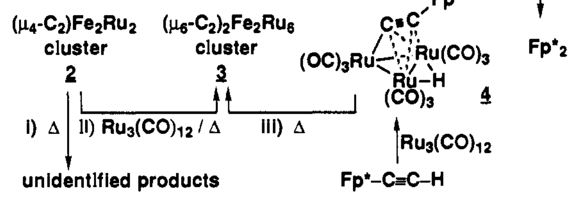


**Figure 2**. Molecular structure of one of two independent molecules of **3**. Selected bond lengths (Å): Ru–Ru, 2.746 (3)–2.857 (4); Ru–Fe, 2.687 (4)–2.855 (3); C21–C22, 1.35 (4); C23–C24, 1.37 (3); C21–Ru1, 2.25 (2); C21–Ru2, 2.20 (2); C21–Fe1, 1.90 (3); C22–Ru3, 2.15 (2); C22–Ru4, 2.18 (3); C22–Ru5, 1.99 (3); C23–Ru2, 2.18 (2); C23–Ru3, 2.17 (2); C23–Fe2, 1.86 (2); C24–Ru1, 2.15 (2); C24–Ru4, 2.11 (2); Ru6–C24, 2.01 (2).

**Scheme I**



Treatment of **1** with $Ru_3(CO)_{12}$ in refluxing benzene afforded a mixture of products, from which a tetranuclear dicarbide cluster, $(\mu_4\text{-}C_2)(Cp^*Fe)_2Ru_2(CO)_8(\mu\text{-}CO)_2$ (**2**), and an octanuclear bis-

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