and **2c** are formed on carbonylation of **1a** and **2b**, respectively.

The structure of $Ru(\eta^2\text{-}C[S]SiMe_2OEt)Cl(CO)(PPh_3)_2$ (**1c**) has been determined by a single-crystal X-ray diffraction study,[12] and an ORTEP diagram is shown in Figure 1. The coordination geometry about ruthenium can be considered as a distorted octahedron with the two PPh_3 ligands axial and the carbonyl, chloride, and η^2-silathioacyl ligands all equatorial and coplanar with ruthenium. The Ru–C(1) distance of 1.978 (8) Å is slightly shorter than the Ru–C(sp^2) single-bond length of 2.056 (3) Å in $Ru(p\text{-tolyl})Cl(CO)(PPh_3)_2$,[13] and the C(1)–S distance of 1.637 (8) Å is midway between expected values for related carbon–sulfur double (1.55–1.56 Å) and single bonds (1.79–1.86 Å).[5a] The Ru–S distance of 2.545 (2) Å is longer than that typically found for Ru–S bonds.[14] These values can all be compared with those found in the η^1-arylthioacyl complex of osmium $Os(\eta^2\text{-}C[S]\text{-}p\text{-tolyl})(\eta^1\text{-}O_2CCF_3)(CO)(PPh_3)_2$.[5a] Here Os–C (1.91 (2) Å) and Os–S (2.513 (6) Å) are both shorter while C–S is longer (1.72 (2) Å) than the corresponding distances in **1c**.

The results provide a good illustration of the usefulness of the thiocarbonyl ligand in observing otherwise difficult migratory insertion reactions. Further elaboration of these group 8 silathioacyls through electrophilic addition to the S atom is under study.

Acknowledgment. We thank the University of Auckland Research Committee for partial support of this work through a grant-in-aid to W.R.R. and L.J.W.

Supplementary Material Available: Text giving elemental analysis and melting point data for compounds prepared in this paper and tables of crystal data (containing details of the data collection and structure solution), positional and thermal parameters, and bond distances and angles for **1c** (9 pages). Ordering information is given on any current masthead page.

OM920513H

---

(11) $Ru(SiMe_2OEt)Cl(CS)(PPh_3)_2$ (**1b**; 0.100 g) was dissolved in benzene (15 mL) at room temperature and CO gas at atmospheric pressure bubbled through the stirred solution for approximately 20 s. The resulting red solution was evaporated to a low volume under reduced pressure and hexane slowly added to effect crystallization. Recrystallization from benzene/hexane yielded red crystals of $Ru(\eta^2\text{-}C[S]\text{-}SiMe_2OEt)Cl(CO)(PPh_3)_2$ (**1c**; 0.083 g, 81%; mp 171–173 °C). Treatment of **1a** and **2b** in a similar manner yielded **1d** and **2c**, respectively.

(12) Red crystals of **1c** were grown from benzene/ethanol. Crystal data: $a = 35.285$ (7) Å, $b = 10.399$ (5) Å, $c = 24.116$ (4) Å, $\beta = 116.02$ (2)°, $Z = 8$, $D_{calc} = 1.370$ g cm^{-3}, space group $C2/c$. A total of 3495 reflections ($I > 3\sigma(I)$) were collected on a Nonius CAD-4 diffractometer at 293 K using Mo Kα radiation ($\lambda = 0.71069$). Least-squares refinement converged to $R(F) = 0.047$ and $R_w(F) = 0.045$.

(13) Rickard, C. E. F.; Roper, W. R.; Taylor, G. E.; Waters, J. M.; Wright, L. J. *J. Organomet. Chem.* **1990**, *389*, 375.

(14) See for example: Irvine, G. J.; Rickard, C. E. F.; Roper, W. R.; Wright, L. J. *J. Organomet. Chem.* **1990**, *387*, C9. Sellmann, D.; Knoch, F.; Wronna, C. *Angew. Chem., Int. Ed. Engl.* **1988**, *27*, 691. Draganjac, M.; Ruffing, C. J.; Rauchfuss, T. B. *Organometallics* **1985**, *4*, 1909. Mura, P.; Olby, B. G.; Robinson, S. D. *J. Chem. Soc., Dalton Trans.* **1985**, 2101.

Catalytic Activity of a Well-Defined Binuclear Ruthenium Alkylidene Complex[†]

Michel R. Gagné and Robert H. Grubbs*

The Arnold and Mabel Beckman Laboratory of Chemical Synthesis, Division of Chemistry and Chemical Engineering, California Institute of Technology, Pasadena, California 91125

Jerald Feldman

Central Research & Development Department, E. I. du Pont de Nemours & Company, Experimental Station, Wilmington, Delaware 19880-0328

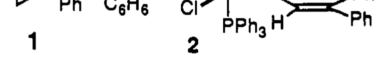
Joseph W. Ziller

Department of Chemistry, University of California at Irvine, Irvine, California 92717

Received August 21, 1992

Summary: The reaction of $[Cp^*RuCl]_4$ with 1,1-diphenylcyclopropene (**1**) gives the bridging vinyl carbene complex $[Cp^*RuCl]_2{=}CHCH{=}CPh_2$ (**4**) in moderate yield. This material selectively catalyzes the dimerization of **1** to the triene $Ph_2C{=}CHCH{=}CHCH{=}CPh_2$. Complex **4** polymerizes norbornene by a ring-opening metathesis process despite the bridging nature of the alkylidene.

Recent developments in our laboratories on the synthesis of well-defined, ruthenium-based olefin metathesis catalysts have stemmed from investigations of the reactivity of 1,1-diphenylcyclopropene (**1**) with ruthenium(II) sources, (e.g. eq 1).[1,2] In an effort to expand upon the base of ruthenium complexes capable of metathesis, the reactivity of $(Cp^*RuCl)_4$[3] ($Cp^* = \eta^5\text{-}C_5Me_5$; **3**) with **1**[4] was investigated.



(1)

Preparative-scale reaction of **3** with 8 equiv of **1** (2 equiv per Ru center) leads to clean formation of a crystalline

---

[†] Contribution No. 8680 from the California Institute of Technology; Contribution No. 6349 from Du Pont.

(1) Nguyen, S. T.; Johnson, L. K.; Grubbs, R. H.; Ziller, J. W. *J. Am. Chem. Soc.* **1992**, *114*, 3974–3975.

(2) 1,1-Diphenylcyclopropene has also been used to generate the following species. Titanium/zirconium vinylcarbenes: (a) Binger, P.; Müller, P.; Benn, R.; Mynott, R. *Angew. Chem., Int. Ed. Engl.* **1989**, *28*, 610–611. Tungsten vinylcarbenes: (b) Johnson, L. K. Ph.D. Thesis, California Institute of Technology, Pasadena, CA, 1992. (c) Johnson, L. K.; Grubbs, R. H.; Ziller, J. W. Manuscript in preparation.

(3) Fagan, P. J.; Mahoney, W. S.; Calabrese, J. C.; Williams, I. D. *Organometallics* **1990**, *9*, 1843–1852 and references therein.

(4) Binger, P. *Synthesis* **1974**, 190–192.

0276-7333/92/2311-3933$03.00/0

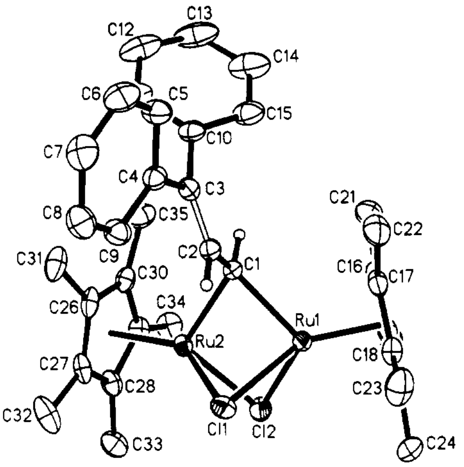
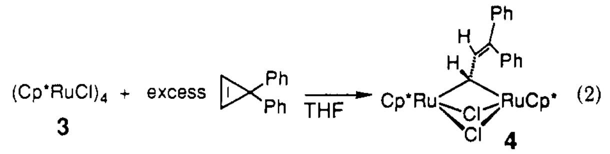


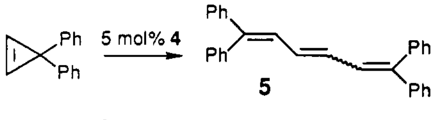
Figure 1. ORTEP diagram of 4. Ellipsoids are drawn at the 50% probability level. Relevant bond distances and angles are listed below. Hydrogen atoms have been omitted for clarity. Ru1–Ru2 = 2.621 (1) Å, Ru1–C1 = 2.105 (2) Å, Ru2–C1 = 2.117 (2) Å, C1–C2 = 1.454 (3) Å, C2–C3 = 1.361 (3) Å, Ru1–Cl1 = 2.509 (1) Å, Ru1–Cl2 = 2.410 (1) Å, Ru1–C1–Ru2 = 76.7 (1)°, Ru–Cl1–Ru2 = 62.9 (1)°.

yellow-green material which is soluble in THF, ether, and benzene and moderately soluble in pentane (eq 2). Extraction (2:1 ether/pentane) and recrystallization (2:1 pentane/ether) gives a 38% yield of 4. The ^{1}H NMR spectrum of this material shows one Cp* resonance (1.49 ppm) and characteristic α- and β-CH resonances at 13.32 ppm (d, J = 13.2 Hz) and 8.52 (d, J = 12.9 Hz), respectively, for the vinylcarbene moiety.[5] The doublets have J_{ab} coupling constants of 12–13 Hz, characteristic of the s-trans conformation of the vinylcarbene functionality.[6] Significantly, both the ^{1}H and ^{13}C NMR spectra show an upfield chemical shift of the vinylcarbene signal compared to signals for compound 2 (18.4 and 288.9 ppm, respectively) and its derivatives.[1] Spectral integration indicates 30 Cp* protons per alkylidene unit, suggesting at least a dinuclear species. Crystals suitable for X-ray analysis were grown from pentane at −40 °C. A low-temperature data set was collected and solved, leading to the structure shown in Figure 1,[7] confirming the connectivity of 4 in eq 2. The upfield shift of the bridging alkylidene compared to the shift for 2[8–10] is consistent with this also being the solution structure.

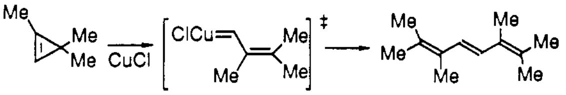


The reaction of 2 equiv of 1 with 3 in THF gives a ^{1}H NMR spectrum with three different alkylidene species in roughly equal amounts (13.32 ppm (d, J = 13.2 Hz) (4); 13.22 ppm (d, J = 13.2 Hz); 12.38 ppm (d, J = 12.3 Hz)). Addition of more 1 to an NMR solution of this mixture causes the downfield alkylidene resonance due to 4 to increase at the expense of the two upfield peaks. On the basis of the lower solubility of these upfield products and the presence of multiple Cp* resonances in the ^{1}H NMR spectrum, it is postulated that these alkylidenes are polynuclear species derived from incomplete breakdown of the parent (Cp*RuCl)$_4$ cubic core.

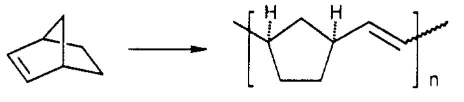
C_6D_6 and THF-d_8 solutions of 4 are capable of catalyzing the dimerization of 1 (eq 3).[11,12] In C_6D_6 using 5 mol % of 4, 1 is dimerized in 4–5 h at room temperature. NMR solutions containing the mixture of the three alkylidene species also catalyze the dimerization leading to the binuclear complex 4. Further breakdown to the mononuclear level is not detected spectroscopically, although its presence at small equilibrium concentrations (possibly contributing to catalysis) cannot be ruled out.



Literature precedent exists for the transition-metal-catalyzed dimerization of cyclopropenes.[13] In the copper(I) chloride catalyzed reaction (4), copper carbenes were implicated. A number of mechanistic scenarios are conceivable and include both mononuclear and binuclear metal centers.



4 reacts with strained bicyclic olefins such as norbornene to give the ring-opening metathesis (ROMP) product (eq 5). This process gives viscous, high-cis, high-molecular-weight polymer when only 10 equiv of norbornene is added, indicating that initiation is much slower than propagation.



In conclusion, we have shown that the Cp*-based class of ruthenium compounds discussed above activate 1,1-

---

(5) Spectroscopic data for 4: ^{1}H NMR (THF-d_8) 13.32 (d, J = 13.2 Hz, 1 H), 8.52 (d, J = 12.9 Hz, 1 H), 7.70 (d, J = 7.8 Hz, 2 H), 7.43 (m, 3 H), 7.31 (m, 2 H), 7.18 (m, 3 H), 1.49 (s, 30 H) ppm; ^{13}C NMR (THF-d_8) 194.9 (CH$_\alpha$), 153.0 (CH$_\beta$), 146.9 (C, Ph), 142.9 (C, Ph), 131.9 (CH, Ph), 128.90 (CH, Ph), 128.87 (CH, Ph), 128.73 (CH, Ph), 127.4 (CH, Ph), 126.9 (CPh$_2$), 126.5 (CH, Ph), 86.7 (C, Cp*), 10.45 (CH$_3$, Cp*) ppm. Anal. Calcd for $C_{35}H_{42}Cl_2Ru_2$: C, 57.14; H, 5.75. Found: C, 58.05, 57.73; H, 5.80, 5.90.

(6) J_{ab} couplings of 5–6 Hz have been observed in some tungsten vinylcarbenes and have been attributed to the s-cis conformation.[2b,c]

(7) Relevant X-ray structural information: triclinic, $P\bar{1}$, a = 10.4097 (12) Å, b = 10.9242 (14) Å, c = 15.964 (2) Å, α = 71.814 (11)°, β = 78.807 (10)°, γ = 66.959 (10)°; Z = 2; T = 158 K; R_F = 2.6%, R_{wF} = 3.8%, goodness of fit = 1.36. Hydrogen atoms were located in the difference Fourier map and were refined isotropically. Additional structural information is found in the supplementary material.

(8) (a) Hermann, W. A. *Adv. Organomet. Chem.* **1982**, *20*, 159–263. (b) Casey, C. P.; Audett, J. D. *Chem. Rev.* **1986**, *86*, 339–352. (c) Holton, J.; Lappert, M. F.; Pearce, R.; Yarrow, P. I. W. *Chem. Rev.* **1983**, *83*, 135–201.

(9) Experiments with [Cp*RuOMe]$_2$[9a,b] and [Cp*RuOC(CH$_3$)(CF$_3$)$_2$]$_2$ initially give vinylcarbenes (δ 12.9 (d, J = 12.3 Hz) and 12.9 (d, J = 13.0 Hz), respectively) which decompose in solution at room temperature ($t_{1/2}$ $\approx$ 2 h). The starting material [Cp*RuNHPh]$_2$[9c] gives an alkylidene which is stable and is tentatively thought to be isostructural with 4 (13.94 ppm (d, J = 13.2 Hz)); however, it does not polymerize norbornene. (a) Loren, S. D.; Campion, B. K.; Heyn, R. H.; Tilley, T. D. *J. Am. Chem. Soc.* **1989**, *111*, 4712–4718. (b) Kölle, U.; Kossakousky, J.; Buese, R. *J. Organomet. Chem.* **1989**, *378*, 449–455. (c) Blake, R. E., Jr.; Heyn, R. H.; Tilley, T. D. *Polyhedron* **1992**, *11*, 709–710.

(10) Attempts to break up 4 with 2 equiv of phosphine give the known Cp*Ru(PMe$_3$)$_2$Cl[9a] and a new alkylidene species that is phosphine free and resonates 2.3 ppm downfield of 4 (δ 15.59 (d, J = 9.6 Hz)). This solution does not polymerize norbornene faster than pure 4. Tilley, T. D.; Grubbs, R. H.; Bercaw, J. E. *Organometallics* **1984**, *3*, 274–278.

(11) Although only one isomer is formed (>95% by NMR), it is unclear whether it is in the cis or trans form.

(12) Crystalline yellow 5 was recrystallized from CH$_2$Cl$_2$ and washed (2×) with 1:1 CH$_2$Cl$_2$/petroleum ether to give an isolated yield of 86%. This material is highly fluorescent. ^{1}H NMR (CD$_2$Cl$_2$): 7.44 (m, 8 H), 7.25 (m, 12 H), 6.75 (dd, J = 7.7, 3.2 Hz), 6.52 (dd, J = 7.7, 3.2 Hz) ppm. ^{13}C NMR (CD$_2$Cl$_2$): 143.17, 142.37, 140.04, 132.91, 130.83, 128.63, 128.59, 128.49, 127.76, 127.69 ppm. Exact MS: calcd for $C_{30}H_{24}$ 384.1878 amu; found 384.1884 amu.

(13) Techl, H. H. *Chem. Ber.* **1964**, *97*, 2681–2688.

diphenylcyclopropene to form vinylcarbenes and that the resulting complexes do indeed react with strained olefins. Unfortunately, due to the bridging nature of the alkylidene, activity toward olefin metathesis is greatly diminished compared to that for **2**.

Acknowledgment. We thank Du Pont Chemicals and the NSF for financial support. In addition, we thank Dr. Paul Fagan of Du Pont CR&D for helpful discussions as well as a loan of starting material.

Supplementary Material Available: Text describing the X-ray diffraction study and tables of crystal data and data collection and refinement details, atomic coordinates, bond distances and angles, anisotropic thermal parameters, and hydrogen positions with isotropic *U* values (15 pages). Ordering information is given on any current masthead page.

OM920518E

An Unexpectedly Strong Chelate Effect in a Cobalt Half-Sandwich Complex with an Intramolecularly Coordinated C–C Double Bond

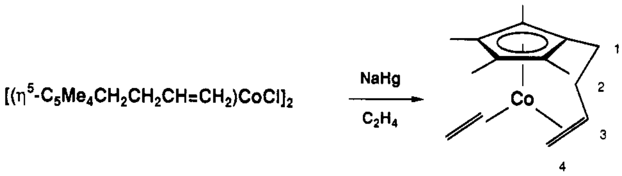
K. Heinz Zimmermann,[1a] Robert S. Pilato,[1b] István T. Horváth,[1b] and Jun Okuda*,[1a,c]
Anorganisch-chemisches Institut der Technischen Universität München, Lichtenbergstrasse 4,
D-8046 Garching, Federal Republic of Germany, and Corporate Research Laboratories,
Exxon Research and Engineering Company, Annandale, New Jersey 08801

Received August 20, 1992

Summary: A pronounced chelate effect is observed in the cobalt half-sandwich complex **1**, bearing an intramolecularly coordinated 3-butenyl side chain. The chelating C–C double bond is only slowly cleaved under ethylene pressure of 36 atm to form the bis(ethylene) complex **2**. Two-electron ligands CO, $P(OMe)_3$, and dimethyl fumarate give chelate complexes **3–5**, whereas the bidentate ligand 1,5-cyclooctadiene affords the "open-chain" complex **6**.

Polydentate ligands containing two types of inequivalent donors, one firmly bound to a metal center and another weakly bonded, which can temporarily block and protect a metal coordination site, hold promise as a novel class of ligands for homogeneous catalysts.[2] For example, phosphine ligands with tethered functional groups that form labile bonds to a transition-metal center have been reported in the literature.[2,3] Such ligands appear to allow intramolecular control of coordinative unsaturation. We have been investigating the possibility of extending the concept of these so-called "semilabile" ligands[2] to functionally substituted cyclopentadienyl ligands.[4] Due to the lability of the cobalt–ethylene bond, bis(olefin)cobalt complexes of the type $(\eta^5\text{-}C_5R_5)Co(\eta^2\text{-}C_2H_4)_2$ (R = H,[5a] Me[5b]) have been extensively used as a source for the 14-electron fragment $(\eta^5\text{-}C_5R_5)Co$, in particular as catalyst precursors for cobalt-mediated [2 + 2 + 2]-cycloaddition reactions.[6] We therefore sought to combine the anchoring effect of the cyclopentadienyl ligand with the lability of a C–C double bond and had previously described the design and synthesis of the 1-(3-butenyl)-2,3,4,5-tetramethylcyclopentadienyl ligand, in which an alkene functionality is tethered to the five-membered-ring ligand.[4,7,8] Herein, we report the synthesis and reactivity of the ethylene complex $(\eta^5{:}\eta^2\text{-}C_5Me_4CH_2CH_2CH{=}CH_2)Co(\eta^2\text{-}C_2H_4)$ (**1**) and the observation of an unexpected chelate effect of this ligand system.

Reduction of $[(\eta^5\text{-}C_5Me_4CH_2CH_2CH{=}CH_2)CoCl]_2$[4] by sodium amalgam under an atmosphere of ethylene affords the ethylene complex $(\eta^5{:}\eta^2\text{-}C_5Me_4CH_2CH_2CH{=}CH_2)Co(\eta^2\text{-}C_2H_4)$ (**1**)[9] in about 80% yield (eq 1). ^1H and ^{13}C NMR



1

(1)

---

[1] (a) Technische Universität München. (b) Exxon Research and Engineering Co. (c) New address: Department of Chemistry, State University of New York at Albany, Albany, NY 12222.

[2] Bader, A.; Lindner, E. *Coord. Chem. Rev.* **1991**, *108*, 27.

[3] Mason, M. R.; Verkade, J. G. *Organometallics* **1992**, *11*, 1514 and references therein.

[4] (a) Okuda, J.; Herdtweck, E.; Zimmermann, K. H. In *Organic Synthesis via Organometallics*; Dötz, K. H., Hoffmann, R. W., Eds.; Vieweg: Braunschweig, Germany, 1991; p 207. (b) Okuda, J.; Zimmermann, K. H.; Herdtweck, E. *Angew. Chem.* **1991**, *103*, 446; *Angew. Chem., Int. Ed. Engl.* **1992**, *30*, 430. (c) Okuda, J.; Zimmermann, K. H. *Chem. Ber.* **1992**, *125*, 637.

[5] (a) Jonas, K.; Deffense, E.; Habermann, D. *Angew. Chem.* **1983**, *95*, 729; *Angew. Chem., Int. Ed. Engl.* **1983**, *22*, 716; *Angew. Chem., Suppl.* **1983**, 1005. (b) Green, M. L. H.; Pardy, R. B. A. *J. Chem. Soc., Dalton Trans.* **1979**, 355. Beevor, R. G.; Frith, S. A.; Spencer, F. L. *J. Organomet. Chem.* **1981**, *221*, C25. Koelle, U.; Khouzami, F.; Fuss, B. *Angew. Chem.* **1982**, *94*, 132; *Angew. Chem., Int. Ed. Engl.* **1982**, *21*, 131; *Angew. Chem., Suppl.* **1982**, 232.

[6] (a) Vollhardt, K. P. C. *Angew. Chem.* **1984**, *96*, 525; *Angew. Chem., Int. Ed. Engl.* **1984**, *23*, 539. (b) Boennemann, H. *Angew. Chem.* **1985**, *97*, 264; *Angew. Chem., Int. Ed. Engl.* **1985**, *24*, 248.

[7] (a) Okuda, J.; Zimmermann, K. H. *J. Organomet. Chem.* **1988**, *344*, C1. (b) Okuda, J.; Zimmermann, K. H. *Chem. Ber.* **1989**, *122*, 1645; **1990**, *123*, 1641.

[8] (a) Miguel-Garcia, J. A.; Adams, H.; Bailey, N. A.; Maitlis, P. M. *J. Organomet. Chem.* **1991**, *413*, 427. (b) Lehmkuhl, H.; Naeser, J.; Mehler, G.; Keil, T.; Danowski, F.; Benn, R.; Mynott, R.; Schroth, G.; Gabor, B.; Krüger, C.; Betz, P. *Chem. Ber.* **1991**, *124*, 441. (c) Dötz, K. H.; Rott, J. *J. Organomet. Chem.* **1988**, *338*, C11. For a 3-butenyl-substituted carborane ligand, see: Delaney, M. S.; Knobler, C. B.; Hawthorne, M. F. *Inorg. Chem.* **1981**, *20*, 1341.

[9] $(\eta^5{:}\eta^2\text{-}C_5Me_4CH_2CH_2CH{=}CH_2)Co(\eta^2\text{-}C_2H_4)$ (**1**): Crude $[(\eta^5\text{-}C_5Me_4CH_2CH_2CH{=}CH_2)CoCl]_2$ (1.46 g, 5.4 mmol) was dissolved in 50 mL of THF and treated at −50 °C with 195 g of sodium amalgam (1% Na, 85 mmol). The reaction mixture was saturated with ethylene, warmed to room temperature, and vigorously stirred for 2 h under an atmosphere of ethylene. Evaporation of all volatiles followed by extraction with pentane, filtration through Kieselguhr, and concentration afforded after recrystallization 2.40 g of red brown crystals (76% based on $Li(C_5Me_4CH_2CH_2CH{=}CH_2)$): mp 83 °C; ^1H NMR (C_6D_6; 25 °C) δ 0.35 [d, $^3J(H,H)$ = 8.5 Hz, 1 Hz, C_2H_4], 0.50 [dd, $^3J(H,H)$ = 9.1 Hz, $^3J(H,H)$ = 13.0 Hz, 1 H, C_2H_4], 0.80 (s, 3 H, CH_3), 1.37 [dd, $^3J(H,H)$ = 9.1 Hz, $^3J(H,H)$ = 13.0 Hz, 1 H, C_2H_4], 1.53 [d, $^3J(H,H)$ = 8.5 Hz, 1 H, C_2H_4], 1.55 (m, 2 H, CH_2-1), 1.64 (s, 3 H, CH_3), 1.84 [d, $^3J(H,H)$ = 9.2 Hz, 1 H, CH_2-4 (cis)], 1.90 [d, $^3J(H,H)$ = 12.8 Hz, 1 H, CH_2-4 (trans)], 1.91 (s, 3 H, CH_3), 2.27 (m, 1 H, CH_2-2), 2.41 (s, 3 H, CH_3), 2.67 (m, 1 H, CH-3), 3.23 (m, 1 H, CH_2-2); ^{13}C NMR (C_6D_6; 25 °C) δ 7.13, 10.08 (double intensity), 11.33 [q, $^1J(C,H)$ = 126 Hz, CH_3], 19.88 [t, $^1J(C,H)$ = 127 Hz, C-1], 39.52 [t, $^1J(C,H)$ = 124 Hz, C-2], 43.45 [t, $^1J(C,H)$ = 153 Hz, C_2H_4], 44.29 [t, $^1J(C,H)$ = 153 Hz, C_2H_4], 46.01 [t, $^1J(C,H)$ = 150 Hz, C-4], 68.56 [d, $^1J(C,H)$ = 155 Hz, C-3], 91.42, 92.67, 92.72, 96.69, 103.23 (ring C); EI MS m/z 262 (11%, M^+), 234 (100%, $M^+ - C_2H_4$), 192 (75%, $M^+ - C_2H_4 - C_3H_6$). Anal. Calcd for $C_{15}H_{23}Co$: C, 68.69; H, 8.84; Co, 22.47. Found: C, 68.61; H, 8.80; Co, 21.80.

0276-7333/92/2311-3935$03.00/0