$$MA + I_2 \rightarrow IMA + I^- + H^+ \quad (3)$$

The rate-determining step is the enolization of malonic acid, which is followed by the fast reaction between the enol form and iodine. The rate law for this process is[9,10]

$$v_3 = -\frac{d[I_2]}{dt} = \frac{(4 \times 10^{-3})[MA][I_2]}{(1 \times 10^{-4}) + [I_2]} \quad (3')$$

The second component reaction occurs between chlorine dioxide and iodide ion.

$$ClO_2 + I^- \rightarrow \tfrac{1}{2}I_2 + ClO_2^- \quad (4)$$

We have redetermined the kinetics of this reaction, which was first studied by Fukutomi and Gordon.[11] Using a more modern stopped-flow instrument and conditions more appropriate for the oscillatory system, we obtained the following rate law, which is independent of both pH between 2.0 and 5.0 and ionic strength (0.1-1.0 M).

$$v_4 = 2d[I_2]/dt = (6 \times 10^3)[ClO_2^\bullet][I^-] \quad (4')$$

The third component reaction, between chlorite and iodide ions, was studied by Kern and Kim[12] and by De Meeus and Sigalla.[13]

$$ClO_2^- + 4I^- + 4H^+ \rightarrow Cl^- + 2I_2 + 2H_2O \quad (5)$$

The rate equation of Kern and Kim is

$$r_5 = (4.6 \times 10^2)[ClO_2^-][I^-][H^+] + (2.65 \times 10^{-3})[ClO_2^-][I_2]/[I^-] \quad (5')$$

The selfinhibition[14] of reaction 5 by iodide is much more important than the autocatalysis by iodine, because $[I_2]$ is nearly constant during an oscillatory period, while $[I^-]$ varies by several orders of magnitude. The second term of rate law (5') cannot be valid when the $[I^-]$ tends to zero, because this term becomes infinite, while the rate must go to zero if the concentration of one of the reactants vanishes. To alleviate this situation, we replace eq 5' by

$$r_3 = (4.6 \times 10^2)[ClO_2^-][I^-][H^+] + (2.65 \times 10^{-3})[ClO_2^-][I_2][I^-]/(u + [I^-]^2) \quad (5'')$$

with $u = 1 \times 10^{-13}$ M^2. The second term goes smoothly to zero as $[I^-] \rightarrow 0$, and gives Kern and Kim's expression (5') when $[I^-] \gg 10^{-6}$ M.

The simple model consisting of reactions 3-5 and the corresponding rate equations describes both the batch oscillations in the $ClO_2^\bullet$-I_2-MA system, as shown in Figure 2, and the oscillatory behavior of the $ClO_2^\bullet$-I^- reaction in a flow reactor. By treating the concentrations of I_2, MA, and $ClO_2^\bullet$ as constant, we obtain a two-variable ($[I^-]$, $[ClO_2^-]$) model, which gives good agreement with the observed dynamics.[15] Such a model is amenable to a variety of analytical techniques for studying both the temporal and the spatial behavior of this system. A more detailed analysis of the experimental kinetics and of the predictions of the model will be presented elsewhere.

Acknowledgment. We thank Patrick De Kepper for informing us about his discovery of Turing structures, and we thank Drs. De. Kepper and Harry Swinney for urging us to study this system. We also thank Robert Olsen and Kenneth Kustin for helpful discussions. This work was supported by the National Science Foundation (CHE-8800169) and by a U.S.-Hungarian cooperative grant from the NSF and the Hungarian Academy of Sciences.

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(15) The rate constant in eq 3' was increased to 7.5×10^{-3} M^{-1} s^{-1} for these calculations.

Chiral Pentaisopropyl-Cyclopentadienyl and Pentaisopentyl-Cyclopentadienyl Complexes: One-Pot Synthesis by Formation of 10 Carbon-Carbon Bonds from Pentamethylcobalticinium

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Received December 13, 1989

Following explosive developments in C_5Me_5-transition-metal chemistry, considerable interest has recently appeared for the design of new pentasubstituted Cp ligands[1] such as C_5Et_5[1a,b] (and C_5Et_4R[1c,d]), C_5Ph_5,[1e] $C_5(CH_2Ph)_5$,[1f] and C_5Me_4R (R = alkyl),[1g] and attempts to synthesize the $C_5(i\text{-}Pr)_5$ ligand have been published.[1h]

This report shows a one-pot route to the first pentaisoalkyl-cyclopentadienyl complexes involving the formation of 10 C-C bonds with the hope that, beyond their topological interest, these novel bulky ligands will also find future use in organometallic chemistry and catalysis.

We already know that the hexaalkylation of $[FeCp(C_6Me_6)]^+$ using a base and a halide proceeds according to eq 1, with replacement of one hydrogen by an alkyl substituent on each methyl group[2] (single branching).

$$[FeCp(C_6Me_6)]^+ \xrightarrow[RX\ (R = alkyl,\ allyl,\ benzyl)]{KOR'\ (R' = H,\ t\text{-}Bu),} [FeCp[C_6(CH_2R)_6]]^+ \quad (1)$$

We now find that 1,2,3,4,5-pentamethylcobalticinium (**1**)[3] reacts with excess (20 equiv) *t*-BuOK and CH_3I (THF, 60 °C, 14 h) to give pure 1,2,3,4,5-pentaisopropylcobalticinium (PF_6^- salt (**2**), Scheme I, double branching), as indicated by ^1H NMR of the crude reaction product.

Recrystallization from acetone/ethanol (1/1) gave an 81% yield of yellow crystals of **2**.[4] Its ^1H NMR spectrum ($CDCl_3$) shows two doublets at δ 1.17 and 1.51 ppm for the non-equivalent (endo and exo) methyl groups and only one C_5H_5 peak (δ 5.54 ppm) whereas the $[^1H]^{13}C$ spectrum ($CDCl_3$) also shows two methyl peaks at δ 21.4 and 24.3 ppm and only one methine peak at δ 25 ppm (Table I, supplementary material).[4] Thus, of the four possible pairs of enantiomers of variable energy, only the low-energy one, in which all the methine C-H bonds are pointing in the same direction, is observed. This is due to the "requirement that each isopropyl group interlocks exactly with its neighbours on either side" as stated in the case of hexaisopropylbenzene[5] (of which no

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(2) (a) Astruc, D. *Acc. Chem. Res.* **1986**, *19*, 377. (b) Moulines, F.; Astruc, D. *Angew. Chem., Int. Ed. Engl.* **1988**, *27*, 1347. (c) Moulines, F.; Astruc, D. *J. Chem. Soc., Chem. Commun.* **1989**, 614. (d) Astruc, D.; Hamon, J.-R.; Althoff, G.; Román, E.; Batail, P.; Michaud, P.; Mariot, J.-P.; Varret, F.; Cozak, D. *J. Am. Chem. Soc.* **1979**, *101*, 5445. (e) Hamon, J.-R.; Saillard, J.-Y.; Lebeuze, A.; Mc Glinchey, M.; Astruc, D. *J. Am. Chem. Soc.* **1982**, *104*, 7549. No. transition-metal complex is known with $C_5\text{-}i\text{-}Pr_5$.
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(4) See the analytical and spectroscopic (^1H and ^{13}C NMR) data for **2** and **3** and the mass spectrum of **4** in the supplementary material.

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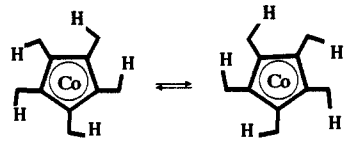
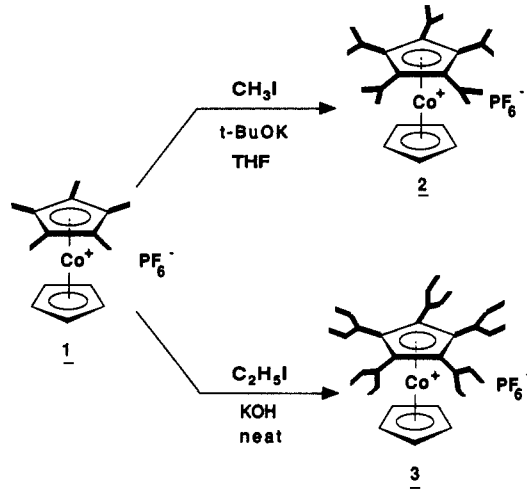


Figure 1. Clockwise and counterclockwise directionalities[7] of the methine C–H bonds in **2** and **3** whose interconversion is fast at high temperature only (see Figure 2). The methyl groups of the isopropyls in **2** and ethyl groups of the isopentyls in **3** are omitted for clarity. Co = $CpCo^+$.

Scheme I



metal complex is known). The metallocenic chirality[6,7] arises from the fact that all the methine C–H groups are pointing clockwise or counterclockwise around the ring with a slow interconversion of these enantiomers at 20 °C (Figure 1). However, interconversion becomes faster at higher temperature as shown by variable-temperature ^1H NMR up to 100 °C in *o*-$Cl_2C_6H_4$ (Figure 2). In the methyl region, the two doublets coalesce at T_c = 65 °C (80 MHz, $\Delta\nu$ = 27 Hz; $\Delta G^{\ddagger}$ = 17.1 ± 0.2 kcal mol^{-1}; 71.5 ± 0.8 kJ mol^{-1}) and only one doublet is observed at high temperature.

Extension of the hyperalkylation to longer alkyl halides bearing H's is impossible using *t*-BuOK because the dehydrohalogenation of RX is faster than the deprotonation of the sandwich complex.[2c] However, the reaction works if KOH is used instead of *t*-BuOK. It can be performed in DME at 80 °C (1 day), or even better (80 °C, 12 h) in the absence of solvent as exemplified with EtI (scheme I). Ion-pair exchange (eq 2) thus provides the driving force for deprotonation by naked, reactive OH^-, in the phase-transfer system.

$$[Co(C_5Me_5)Cp^+,PF_6^-] + [K^+,OH^-] \rightleftharpoons [Co(C_5Me_5)Cp^+,OH^-] + [K^+,PF_6^-] \quad (2)$$

The pure 1,2,3,4,5-pentaisopentylcobalticinium salt **3**[4] is obtained in 70% yield as yellow needles after recrystallization from

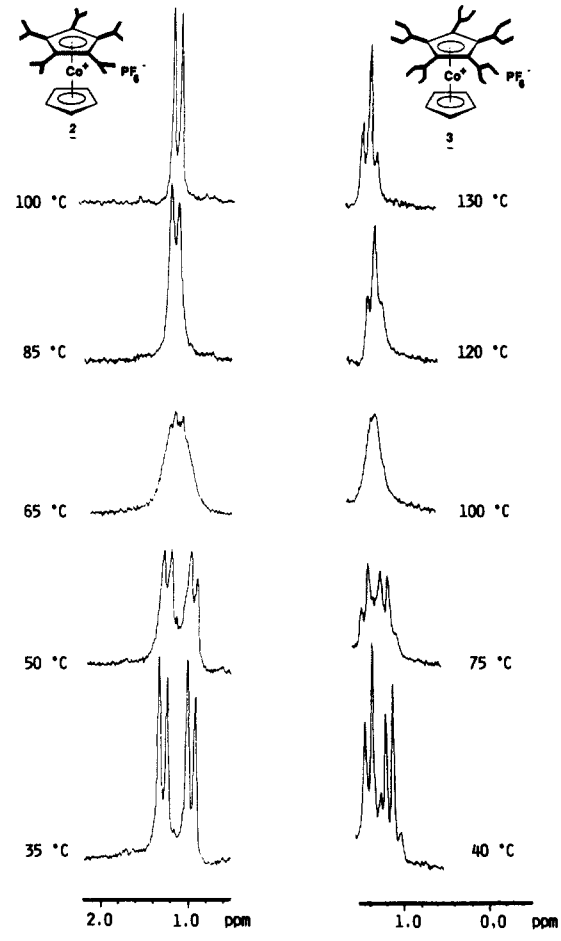


Figure 2. Variable-temperature ^1H NMR spectra of **2** and **3** in *o*-dichlorobenzene (80 MHz, the optimal frequency for **3**): methyl region. The full ^1H and ^{13}C NMR spectra at respectively 250 and 62.875 MHz (25 °C) are provided in the supplementary material. Molecular mechanism calculations have shown that stepwise, uncorrelated rotations are the rule with simple alkyl groups attached to planar frameworks.[7]

THF/ether, 1/1, at −40 °C. All the features of the ^1H and ^{13}C spectra at 20 °C (250 vs 62.875 MHz; supplementary material) show a C_5(isopentyl)$_5$ ligand structure bearing nonequivalent endo and exo ethyl groups, consistent with a blocked gear-meshed conformation. For instance, this metallocenic chirality[6,7] is indicated by the four well-separated multiplets of the diastereotopic methyl protons at 250 MHz. The interconversion of the clockwise and counterclockwise enantiomers[7] does occur at a higher temperature than for 2^+ but is nevertheless observed (Figure 2). The methyl groups coalesce at T_c = 100 °C (80 MHz, $\Delta\nu$ = 15 Hz, $\Delta G^{\ddagger}$ = 19.4 ± 0.2 kcal mol^{-1}; 81.0 ± 0.8 kJ mol^{-1}).

Information on the stereoelectronic properties of the new ligands is also provided by the electrochemical data. The E° values (Hg, DMF, *n*-Bu_4NBF_4 0.1 M, 20 °C, V vs ECS) of the complexes $CoCp(C_5R_5)^+PF_6^-$ are −0.89 (R = H),[7a,b] −1.17 (Me), −0.96 (*i*-Pr), and −1.00 (isopentyl) for the reversible $Co^{III} \rightleftharpoons Co^{II}$ couple and −1.88 (H),[7c,d] −2.19 (Me), −2.11 (*i*-Pr), and −2.10 (isopentyl) for the $Co^{II} \rightleftharpoons Co^{I}$ couple, chemically reversible for R = H, Me, and *i*-Pr at 25 °C and for R = isopentyl at −30 °C (i_a/i_c = 0.8; 400 mV s^{-1}) but irreversible for the latter at 25 °C (i_a/i_c = 0). Thus the electronic properties of C_5-*i*-Pr_5 and of C_5(isopentyl)$_5$ in **2** and **3** are intermediate between those of Cp and C_5Me_5. The C_5-*i*-Pr_5 ligand, unlike C_5(isopentyl)$_5$, stabilizes the 20-electron Co^I state. From a preparative point of view, **2** is instantaneously reduced to the stable complex $Co^{II}Cp(C_5$-*i*-$Pr_5)$ (**4**)[4] by using 1 equiv of the electron-reservoir complex $Fe^{I}CpC_6Me_6$[2a,9] in THF at 20 °C.

In conclusion, these facile, high-yield hyperalkylation reactions in a non-C_6 ring lead to the first perisoalkyl ring π-complexes and

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open a route to various hyperalkylations of cationic C_5Me_5 complexes and of other permethylated π ligands. The dramatic difference between the C_6Me_6 ligand in eq 1 (single branching) and the C_5Me_5 ligand (double branching) arises essentially because of the difference in steric bulk between the C_5 and C_6 rings whose internal angles are respectively 72° and 60°. These internal angles are also responsible for the large difference in rotational barriers of the *i*-Pr groups in **2** and C_6-*i*-Pr_6.[5c]

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Registry No. 1, 126580-33-4; **2**, 126580-35-6; **3**, 126580-37-8.

Supplementary Material Available: Experimental procedures for the synthesis of **2** and **3**, analytical and spectroscopic (^1H, ^{13}C NMR) data for **2** and **3**, mass spectral data and mass spectrum for **4**, and ^1H and [^1H]^{13}C NMR spectra for **2** and **3** (10 pages). Ordering information is given on any current masthead page.

A Direct Total Synthesis of (+)-Longifolene via an Intramolecular Diels-Alder Strategy

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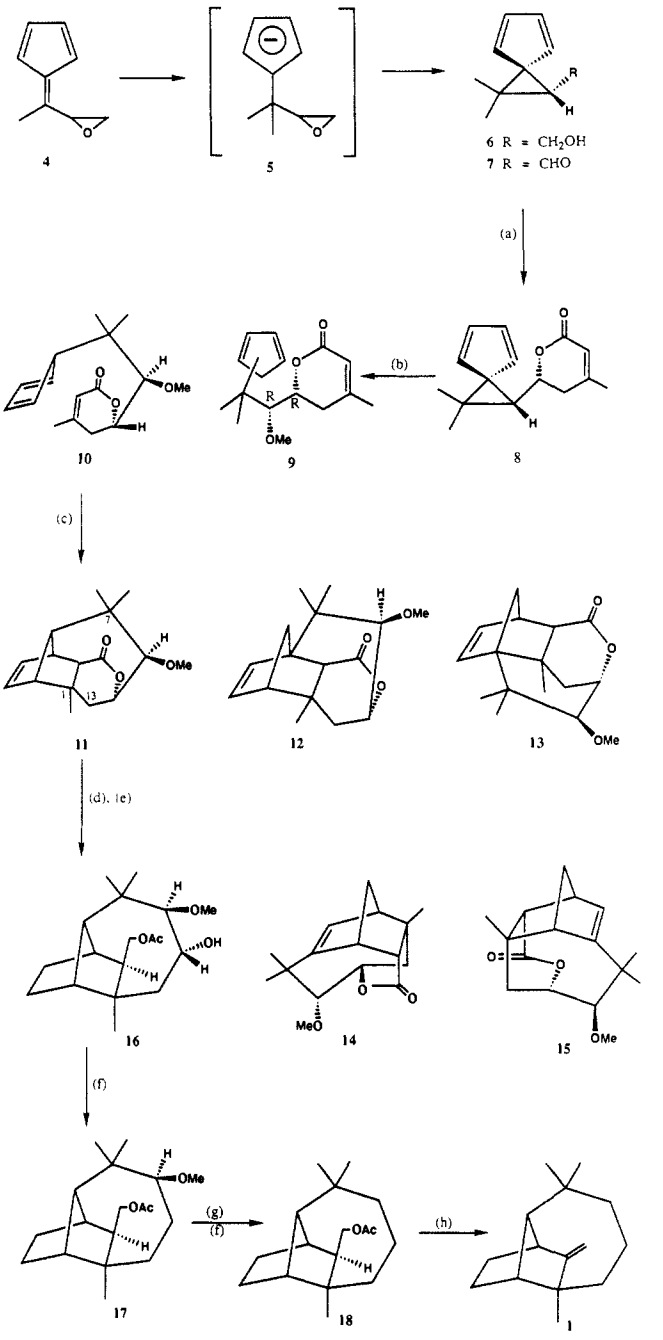
The topological framework represented by the sesquiterpene longifolene (**1**) continues to play an important and historic role in organic chemistry.[1-5] In particular, the longifolene skeleton has served as a subject for synthetic planning and strategy,[3a,5] and the total syntheses reported to date[3] reflect this diversity. From the standpoint of retrosynthetic analysis, the strategic double disconnection of **1** to a triene precursor of type **2** has considerable appeal and as such has been widely noted.[3a,4e,f,5] However, as an early approach to longifolene showed,[4e] the propensity of substituted cyclopentadienes to undergo facile 1,5-sigmatropic rearrangement prior to cyclization takes precedence. Thus for this strategy to succeed, either the rearrangement must be blocked,[6] conditions developed where cyclization can compete efficiently,[7] or alternatively constraints built into the system so the desired cyclization is the preferred one. Unfortunately, blocking the sigmatropic reaction is not necessarily straightforward, since even chlorine migrates prior to cyclization in a related case,[4f,8] although we have demonstrated that the cyclopropane moiety present in a spiro[2.4]heptadiene system can be used effectively for this purpose and as a latent carbon source.[6] Molecular models and molecular mechanics calculations suggest that if the dienophile

Scheme I[a]



[a] (a) LDA, THF, -40 °C, $Me_2C{=}CHCO_2Me$, $CdCl_2$, 30 min; 2 h, 0 °C, 73%. (b) $BF_3{\cdot}Et_2O$, MeOH, 4 h, 22 °C, 83%. (c) Toluene, microwave, sealed tube, 2.5 h, 97%. (d) H_2, 5% Pd/C, EtOAc, 30 psi, 4 h; $LiAlH_4$, ether, 0-22 °C, 4 h, 95%. (e) Ac_2O, pyridine, ether, 6 h, 0 °C, 74%. (f) ClC(=S)OPh, pyridine, CH_2Cl_2, 22 °C; nBu_3SnH, AIBN, toluene, 4 h, 110 °C, 71%. (g) NaI, Et_3N, Me_3SiCl, CH_2Cl_2, 22 °C, 1 h (f, 50%). (h) C_6H_6, flow system, 525 °C, 56%.

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