bimetallic rhenium formyl to a hydride. Such a mechanism is not operative in the present case since (i) no iron formyl complex is detected at −80 °C and (ii) its radical chain decomposition would suppose the reduction of this neutral formyl complex to an anionic intermediate, a process that would require an extremely negative reduction potential (probably around −2.5 to −2.8 V vs. SCE).

Finally, note that coordinated CO is not reduced in the present ET mechanism. On the other hand, hydridocarbonyl species are currently postulated to form surface-bound formyl species in heterogeneous catalysis. An ET path for the hydride reduction of coordinated CO in the Fischer–Tropsch process may be significant insofar as ET through a metal surface should be facile.

Acknowledgment. We are grateful to Professor Louis Nadjo for EPR facilities (University of Paris VI) and to BASF (Ludwighafen, West Germany) for a generous gift of iron carbonyl.

Registry No. 1, 55904-75-1; 2, 84749-11-1; 3, 84365-32-2; 4, 88251-02-9; 5, 89530-20-1; CpFe(tripod)$^+$PF$_6^-$, 32843-42-8; [C$_5$Me$_5$Fe^I(dppe)(CO)], 89530-21-2.

Manipulation of Organoactinide Coordinative Unsaturation and Stereochemistry. Properties of Chelating Bis(polymethylcyclopentadienyl) Hydrocarbyls and Hydrides

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Received February 29, 1984

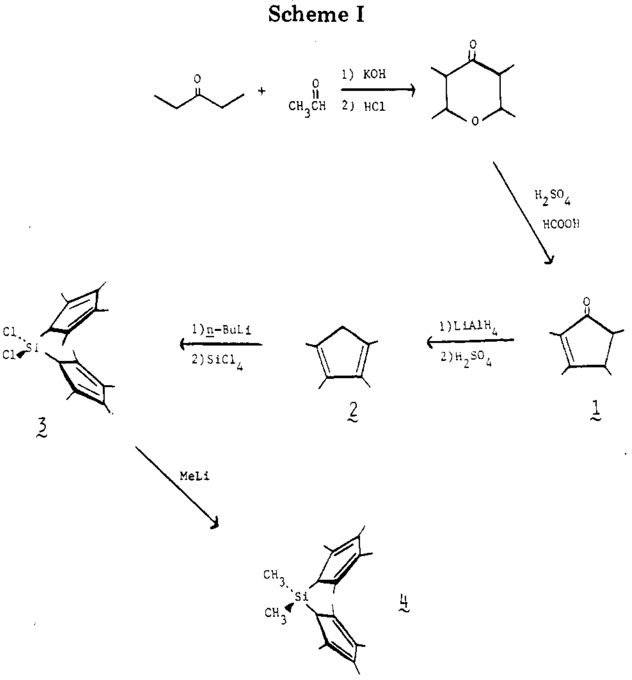
Summary: The synthesis of the chelating polymethylcyclopentadienyl ligand $(CH_3)_2Si[(CH_3)_4C_5]_2{}^{2-}$ is reported. Reaction with ThCl$_4$ followed by alkylation yields the new, thermally stable thorium hydrocarbyls $(CH_3)_2Si[(CH_3)_4C_5]_2ThR_2$, R = n-C$_4H_9$, CH$_2$C(CH$_3$)$_3$, CH$_2$Si(CH$_3$)$_3$, CH$_2C_6H_5$, and C$_6H_5$. The R = CH$_2$Si(CH$_3$)$_3$ complex has also been characterized by single-crystal X-ray diffraction. The most unusual feature of the molecular structure is the rather small (ring center of gravity)–Th–(ring center of gravity) angle of 118.4° and the rather large ring–ring dihedral angle of 105.4°. The hydrocarbyls undergo facile hydrogenolysis to yield the new thorium hydride $\{(CH_3)_2Si[(CH_3)_4C_5]_2ThH_2\}_x$. The infrared spectrum of this hydride lacks terminal Th–H stretching transitions. The new hydride catalyzes olefin hydrogenation at turnover frequencies significantly in excess of those for $\{Th[(CH_3)_5C_5]_2H_2\}_2$.

The advantageous characteristics of bis(pentamethylcyclopentadienyl) ligation (solubility, crystallizability, electron donation, and probable resistance to redistribution and certain thermal decomposition modes) have afforded major advances in organo f-element chemistry over bis(cyclopentadienyls).[1,2] However, as indicated by considerable structural data,[1-3] these improvements are realized at the expense of congesting the metal coordination sphere and possibly depressing reactivity. It would clearly be of interest to "open" the Cp′$_2$M < (Cp′ = η^5-(CH$_3$)$_5$C$_5$) geometry, and we report here[4] a chelating polymethylcyclopentadienyl ligand which, as illustrated by a new class of actinide hydrocarbyls and hydrides, offers one such approach to modifying coordinative saturation and reactivity. The unusual molecular structure of one member of the new series is also reported.

2,3,4,5-Tetramethylcyclopent-2-enone (1) was prepared by a modification of the Burger, Delay, and Mazenod procedure[5,6] (Scheme I) and converted straightforwardly to tetramethylcyclopentadiene (2). Reaction of the anion with SiCl$_4$ followed by methylation and workup afforded (CH$_3$)$_2$Si[(CH$_3$)$_4$C$_5$H]$_2$ (4, Me$_2$Si(Cp″H)$_2$) in good yield.[7a,b] The dilithio derivative of 4 (via n-C$_4$H$_9$Li metalation) can then be converted to thorium precursor complex 5 (eq 1).

$$\mathrm{Me_2SiCp''_2{}^{2-}} + \mathrm{ThCl_4} \xrightarrow{\mathrm{DME}} \underset{\mathbf{5}}{\text{“}\mathrm{Me_2SiCp''_2ThCl_2}\text{”}} + 2\mathrm{Cl^-} \tag{1}$$

Scheme I



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[7] (a) All new compounds gave expected spectral and analytical data; see supplementary material for details. (b) Yields in the ligand synthesis: 54% (2), 80% (3), 57% (4). (c) Flame tests indicate negligible Li in compounds 6–10.

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Complex **5** could not be freed of LiCl (elemental analysis) or DME (NMR, IR), suggesting an anionic, formally nine-coordinate formulation,[8] cf. $Li^+(THF)_2[CH_2(C_5H_4)_2]_2U_2Cl_5^-$.[9] Alkylation of **5** with a variety of sterically demanding lithium reagents[10] yields, after workup, colorless to pale yellow, crystalline, thermally stable,[11] extremely air-sensitive hydrocarbyls in 30–50% yield (eq 2). These new compounds were characterized by standard

$$\mathbf{5} + 2RLi \xrightarrow{DME} \begin{array}{c} (CH_3)_2SiCp''_2ThR_2 \\ \mathbf{6},\ R = n\text{-}C_4H_9 \\ \mathbf{7},\ R = CH_2C(CH_3)_3 \\ \mathbf{8},\ R = CH_2Si(CH_3)_3 \\ \mathbf{9},\ R = CH_2C_6H_5 \\ \mathbf{10},\ R = C_6H_5 \end{array} + 2LiCl \quad (2)$$

analytical/spectroscopic methods,[7a,c] and **8** was characterized by X-ray diffraction (vide infra). With smaller hydrocarbyl functionalities (e.g., R = CH_3) less tractable products, which appear to be $Me_2SiCp''_2Th(CH_3)_2Cl^-$ or $Me_2SiCp''_2Th(CH_3)_3^-$ complexes,[8b] are obtained (in contrast to $Cp'_2Th(CH_3)_2$ chemistry[10]).

Single crystals of **8** (from pentane at −30 °C) are at 20 ± 1 °C monoclinic of space group $P2_1/m$—C_{2h}^2 (No. 15) with a = 11.960 (5) Å, b = 11.270 (5) Å, c = 12.395 (6) Å, β = 99.32 (4)°, and Z = 2 [μ_a(Mo K$\bar{\alpha}$) = 48.0 mm^{-1}, d_{calcd} = 1.422 g cm^{-3}]. Three-dimensional X-ray diffraction data were collected for 3989 independent reflections having $2\theta_{MoK\alpha} < 55°$ (the equivalent of 1.0 limiting Cu K$\bar{\alpha}$ sphere) on a Nicolet P$\bar{1}$ autodiffractometer using graphite-monochromated Mo K$\bar{\alpha}$ radiation and full (0.90° wide) ω scans. The structure was solved by using the "heavy-atom" technique. The resulting structural parameters were reflected to convergence [R(unweighted, based on F) = 0.076 for 2586 independent absorption- and degradation-corrected reflections having $2\theta_{MoK\alpha} < 55°$ and $I > 3\sigma(I)$] by using cascade block-diagonal least-squares techniques with weights derived from counting statistics and anisotropic thermal parameters for all non-hydrogen atoms.[12] Calculations were performed on a Data General Eclipse S-200 computer using local versions of Nicolet EXTL and SHELXTL software.

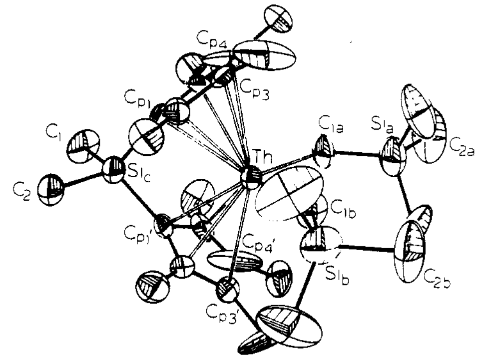

Figure 1. Perspective ORTEP drawing of the non-hydrogen atoms in $(CH_3)_2Si[(CH_3)_4C_5]_2Th[CH_2Si(CH_3)_3]_2$, **8**. All atoms are represented by thermal vibrational ellipsoids drawn to encompass 50% of the electron density. Individual bond lengths (Å) and angles (deg) of interest include the following: Th–C_{1a}, 2.54 (2); Th–C_{1b}, 2.48 (2); Th–C_{1a}–Si_a, 124 (1); Th–C_{1b}–Si_b, 150 (1); Th–C_{p1}, 2.686 (14); Th–C_{p2}, 2.744 (13); Th–C_{p3}, 2.875 (13); Th–C_{p4}, 2.917 (17); Th–C_{p5}, 2.733 (19); Th–Cg, 2.52 (···); Cp_1–Cp_2, 1.44 (2); Cp_1–Cp_5, 1.45 (2); Cp_2–Cp_3, 1.36 (2); Cp_3–Cp_4, 1.56 (3); Cp_4–Cp_5, 1.21 (3); Si_c–Cp_1, 1.88 (1); Th···Si_c, 3.465 (6); Si_a–C_{1a}, 1.81 (3); Si_b–C_{1b}, 1.90 (3); Cp_1–Si–Cp_1', 100.6 (9); Cg–Th–Cg′, 118.4 (...); Cg–Th–C_{1a}, 106.4 (...); Cg–Th–C_{1b}, 112.2 (...); and C_{1a}–Th–C_{1b}, 98.9 (8). Atoms denoted by a prime are related to those without a prime by the crystallographic mirror plane at (x, $^1/_4$, z).

The structure of **8** (Figure 1)[13] possesses crystallographic C_s—m site symmetry with Th, Si_c, C_1, C_2, C_{1a}, Si_a, C_{2a}, C_{1b}, Si_b, and C_{2b} lying in a mirror plane. The most striking feature is the severe distortion of the typical[1-3,14] organoactinide Cp'_2MX_2 coordination geometry (cf. $Cp'_2Th[CH_2Si(CH_3)_3]_2$,[14a] **11**) imposed by linking the Cp″ ligands. Thus, the dihedral angle between the C_5 ring mean planes has opened to 105.4° from 75° in **11** and the Cg–Th–Cg′ (Cg = ring center-of-gravity) angle has contracted to 118.4° (normally 135–138°[1-3,14,15]). Competition between maintaining normal η^5-Cp″ coordination and tetrahedral Si_c is evident in the large dispersion about the average Th–C(ring) distance 2.791 (15, 84, 126, 4) Å (cf. 2.81 (1, 1, 4, 10) Å in **11**[14a]) with the longest distances being to C(ring) atoms furthest from Si_c, the distorted Cp_1–Si_c–Cp'_1 angle of 100.6 (9)°, and the displacement of Si_c from each C_5 ring mean plane by 0.41 Å. Likely correlated with the dispersion in Th–C(ring) is a significant alternation in ring C–C contacts, ranging from 1.56 (3) Å for Cp_3–Cp_4 to 1.36 (2) and 1.21 (3) Å in Cp_2–Cp_3 and Cp_4–Cp_5, respectively; such distortions are not reasonably a consequence of Cp″–silyl functionalization alone.[16] Diminished ring methyl–$CH_2Si(CH_3)_3$ nonbonded interactions in **8** (vs. **11**) are suggested by displacements of C_{1a} and C_{1b} by +1.98 and −1.83 Å, respectively, from the Th, Si_c, Cp_1, Cp_1', Cg, Cg′ least-squares mean plane; the corresponding distances in **11** are +2.04 and −1.66 Å. However, the one large (150 (1)°) Th–C_{1b}–Si_b valence angle accompanied by a shortened (2.48 (2) Å) Th–C_{1b} bond distance is surprisingly reminiscent of the hydrocarbyl bonding pattern in **11**, which possesses corresponding parameters of 148.0 (7)° and 2.46 (1) Å.[14a] The C_{1a}–Th–C_{1b} angle in the two complexes is nearly identical: 98.9 (8)° (**8**) and 96.8 (4)° (**11**).

As regards chemical behavior, the new hydrocarbyls react rapidly with hydrogen (eq 3) to yield bright yellow

$$x\,Me_2SiCp''_2ThR_2 + x\,H_2 \xrightarrow{heptane} \underset{\mathbf{12}}{(Me_2SiCp''_2ThH_2)_x} + 2x\,RH \quad (3)$$

dihydride **12**.[7] While clearly a thorium dihydride[10] with magnetically equivalent hydrido ligands at room temper-

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(8) (a) NMR establishes the $Me_2SiCp''_2$:DME ratio as 2:1. (b) Full characterization of this complex is continuing.

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(10) General synthetic procedures: Fagan, P. J.; Manriquez, J. M.; Maatta, E. A.; Seyam, A. M.; Marks, T. J. *J. Am. Chem. Soc.* **1981**, *103*, 6650–6667.

(11) Compounds **6** and **7** undergo noticeable discoloration after ca. 1 week at ambient temperature. As judged by color and NMR, compounds **8**, **9**, and **10** are stable for weeks at room temperature.

(12) See supplementary material for details.

(13) The first number in parentheses following an averaged value of a bond length or angle is the root-mean-square estimated standard deviation of an individual datum. The second and third numbers, when given, are the average and maximum deviations from the averaged value, respectively. The fourth number represents the number of individual measurements that are included in the average value.

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ature (^1H NMR δ 18.35), low solubility in cyclohexane and reaction with benzene have so far precluded solution molecular weight measurements. Unlike formally analogous $[Cp'_2Th(\mu\text{-}H)H]_2$ (**13**),[10,15] the Th–H portion of the infrared spectrum of **12** ((Nujol mull, cm^{-1}) 1275 (mw), 1155 (w), 654 (m), 645 (m); **12**-*d*,[17] 904 (m), $\nu_{M\text{-}H}/\nu_{M\text{-}D} = 1.41$) *does not* exhibit detectable terminal Th–H stretching transitions, suggesting more extensive hydrogen bridging and an increased formal thorium coordination number. Preliminary reactivity comparisons between **12** and **13** have been sought in homogeneous olefin hydrogenation, for which **13** is moderately active.[10] Although mechanistic details have not as yet been fully elucidated, it is noteworthy that at room temperature (1 atm H_2 pressure), **12** is 13 times more active than **13** for 1-hexene hydrogenation (initial $N_t = 6.5\ h^{-1}$) and 21 times more active (initial $N_t = 2.5\ h^{-1}$)[18] for hydrogenating sterically more demanding *trans*-2-hexene.

The degree to which the present ligand linkage approach can modify Cp'_2M chemistry in 5f as well as other systems is under continuing investigation.

Acknowledgment. We thank the National Science Foundation (CHE8009060 and CHE8306255) for generous support of this research.

Supplementary Material Available: Spectroscopic and analytical data, a table of fractional atomic coordinates (Table I), and a table of anisotropic thermal parameters (Table II) for non-hydrogen atoms of $(CH_3)_2Si[(CH_3)_4C_5]_2Th[CH_2Si(CH_3)_3]_2$ (**8**) (5 pages). Ordering information is given on any current masthead page.

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(17) By carrying out eq 3 with D_2. Th–D modes other than at 904 cm^{-1} are obscured by $Me_2SiCp''_2$ vibrational transitions.

(18) In this case, catalyst lifetime is only ca. 8 h. The cause is under investigation.

Catalytic Halogen Exchange Mediated by the Dinuclear Gold(I) Complex $[Au(CH_2)_2PPh_2]_2$

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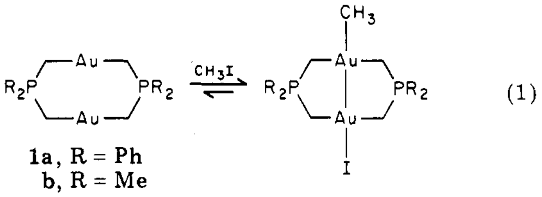
Summary: In addition to establishing the reversible nature of the oxidative addition of RX (R = CH_3, CD_3; X = Br, I) to $[Au(CH_2)_2PPh_2]_2$, we have observed that $[Au(CH_2)_2PPh_2]_2$ catalyzes halogen exchange between CH_3Br and CD_3I as well as between $[Au(CH_2)_2PPh_2]_2Br_2$ and CH_3I in $CDCl_3$. The results of attempted halogen exchange with a variety of alkyl halides suggests that a S_N2 process is likely via the formation of $[[Au(CH_2)_2PPh_2]_2CH_3]^+I^-$; thus the dinuclear gold(I) complex effects exchange by ionization of CH_3I.

While pursuing studies of the two-center two-electron oxidative addition of alkyl halides to the dinuclear gold(I) ylide complex[1] $[Au(CH_2)_2PPh_2]_2$, **1a**, we observed a catalytic halogen-exchange reaction mediated by **1a**. Many homogeneous catalytic reactions require sequential oxidative addition–reductive elimination cycles[2] in order to effect a given chemical transformation. However, oxidative addition–reductive elimination reactions have not been well studied for complexes containing more than one metal center.[2] Since we have observed[3] that $[Au(CH_2)_2PPh_2]_2(CH_3)I$, **2a**, is in equilibrium with CH_3I and **1a**, this system is particularly well suited for an investigation that addresses the role of the second metal center in the oxidative addition–reductive elimination cycle.

The dimers **1a** and **1b** were prepared according to established methods.[2b,3,4] The syntheses of **2a** and **2b** were carried out as described by Fackler[1] and Schmidbaur.[2b] Complexes **2c** and **2d** were obtained by the addition of excess CH_3Br (Matheson) and CD_3I (Aldrich) to **1a** in benzene, respectively. After 18 h of reaction at room temperature, the solvent was removed in vacuo, leaving a yellow powder,[5] **2c**, mp (uncorrected) 174 °C (loss of CH_3Br by reductive elimination), and the residue **1a**, mp 228–229 °C dec (lit.[3] mp 230–231 °C). The FT ^2H NMR spectra were obtained by using a Varian XL-200 spectrometer operating at 30.71 MHz. The $CHCl_3$ solvent used was distilled from P_4O_{10} before use. Chemical shifts were relative to internal $CDCl_3$ (δ 7.24). The ^1H NMR spectra were obtained by using a Varian EM 390 spectrometer (90 MHz), and chemical shifts are relative to internal Me_4Si (δ 0).

Our attention was drawn to the halide-exchange reaction when we observed[3a] the previously unreported[6] and possibly the first example[3b] of an intermolecular reversible two-center oxidative addition–reductive elimination equilibrium (eq 1). The reversibility of the reaction has been established by temperature-dependent NMR measurements.



1a, R = Ph
b, R = Me

2a, R = Ph
b, R = Me

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(1) Fackler, J. P., Jr.; Basil, J. D. *Organometallics* **1982**, *1*, 871. Structural characterization of **2c**, $[Au(CH_2)_2PPh]_2(CH_3)Br$, undertaken in our laboratory indicates the Au–Au bond to be 2.680 (3) Å. The Au–Au bond in **2b** is 2.663 (9) Å.

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(3) (a) Basil, J. D., Ph.D. Thesis, Case Western Reserve University, 1982. (b) R. J. Puddephatt has observed the reversible oxidative addition of MeI to the dinuclear Pt complex $[Pt(CH_3)_2(C_2H_5)_2P\text{–}CH_2\text{–}P(C_2H_5)_2]_2$, "but it occurs at only one metal center" giving a Pt(IV)/Pt(II) dimer (private communication).

(4) Schmidbaur, H. "Inorganic Synthesis"; Douglas, B. E., Ed., Wiley-Interscience: New York, 1978; Vol. XVIII, p 138.

(5) ^1H NMR of **3** (90 MHz, $CDCl_3$ solvent, relative Me_4Si internal) in equilibrium with **1a** and CH_3Br: **3**, C_6H_5, δ 7.59 (m) and 7.29 (m), Au–CH_2–P, δ 1.77 (d, J_{PH} = 10.8 Hz) and 1.42 (d, J_{PH} = 10.5 Hz), $AuCH_3$, δ 1.10 (t, J = <0.6 Hz); **1a**, C_6H_5, δ 7.55 (m) and 7.25 (m), δ 1.35 (d, J_{PH} = 12.9 Hz), CH_3Br, δ 2.63 (s).

(6) In Schmidbaur's original report of the oxidative addition of CH_3I to $[Au(CH_2)_2P(CH_3)_2]_2$ there is no mention that the ^1H NMR spectrum of this gold(II) dinuclear methyl iodide adduct shows it to be in equilibrium with the gold(I) dinuclear ylide and CH_3I. See ref 2b and: Schmidbaur, H. *Acc. Chem. Res.* **1975**, *8*, 62.

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