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.

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Registry No. 1, 55904-75-1; 2, 84749-11-1; 3, 84365-32-2; 4, 88251-02-9; 5, 89530-20-1; CpFe(tripod)⁺PF₆⁻, 32843-42-8; $[C_5Me_5Fe^{I}(dppe)(CO)]$, 89530-21-2.

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

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Summary: The synthesis of the chelating polymethylcyclopentadienyl ligand (CH₃)₂Si[(CH₃)₄C₅]₂²⁻ is reported. Reaction with ThCl₄ followed by alkylation yields the new, thermally stable thorium hydrocarbyls (CH₃)₂Si- $[(CH_3)_4C_5]_2ThR_2$, $R = n - C_4H_9$, $CH_2C(CH_3)_3$, $CH_2Si(CH_3)_3$, $CH_2C_6H_5$, and C_6H_5 . The R = $CH_2Si(CH_3)_3$ 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[(C- $H_{3}_{5}C_{5}]_{2}H_{2}_{2}$

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 consid-



erable 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 $Cp'_2M < (Cp' = \eta^5 - (CH_3)_5C_5)$ geometry, and we report here⁴ 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 SiCl₄ followed by methylation and workup afforded $(CH_3)_2Si[(CH_3)_4C_5H]_2$ (4, Me₂Si(Cp''H)₂) in good yield.^{7a,b} The dilithio derivative of 4 (via *n*-C₄H₉Li metalation) can then be converted to thorium precursor complex 5 (eq 1).

$$Me_{2}SiCp''_{2}^{2-} + ThCl_{4} \xrightarrow{DME} "Me_{2}SiCp''_{2}ThCl_{2}" + 2Cl^{-} 5$$
(1)

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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 reagents¹⁰ 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

5 + 2RLi
$$\xrightarrow{\text{DME}}$$
 (CH₃)₂SiCp"₂ThR₂ + 2LiCl (2)
6, R = *n*-C₄H₉
7, R = CH₂C(CH₃)₃
8, R = CH₂Si(CH₃)₃
9, R = CH₂C₆H₅
10, R = C₆H₅

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 $Cp'_{2}Th(CH_{3})_{2}$ chemistry¹⁰).

Single crystals of 8 (from pentane at -30 °C) are at 20 ± 1 °C monoclinic of space group $P2_1/m - C_{2h}$ (No. 15) with a = 11.960 (5) Å, b = 11.270 (5) Å, c = 12.395 (6) Å, $\beta = 99.32 \ (4)^{\circ}$, and $Z = 2 \ [\mu_a(Mo \ K\bar{\alpha}) = 48.0 \ mm^{-1}, \ d_{calcd}$ = 1.422 g cm^{-3}]. Three-dimensional X-ray diffraction data were collected for 3989 independent reflections having $2\theta_{MoK}\alpha < 55^{\circ}$ (the equivalent of 1.0 limiting Cu K $\bar{\alpha}$ sphere) on a Nicolet P1 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 $2\theta_{MoK}\alpha < 55^{\circ}$ 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.

The structure of 8 (Figure 1)¹³ possesses crystallographic C_s —*m* site symmetry with Th, Si_c, C₁, C₂, 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.

(12) See supplementary material for details



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-Cl_a , 1.84 (3); Si_c-Cy , 1.90 (3); Cp_3-Si_c-Cy , 1.00 6 (6); Cg-Th-Cg', 1.84 (4); $\begin{array}{l} Si_b-C_{1b}, 1.90 \ (3); \ Cp_1-Si-Cp_1', \ 100.6 \ (9); \ Cg-Th-Cg', \ 118.4 \ (...); \\ Cg-Th-C_{1a}, \ 106.4 \ (...); \ Cg-Th-C_{1b}, \ 112.2 \ (...); \ and \ C_{1a}-Th-C_{1b}, \end{array}$ 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).

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, Cp₁, Cp₁', 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

$$x \operatorname{Me_2SiCp''_2ThR_2} + x \operatorname{H_2} \xrightarrow{\operatorname{neptane}} (\operatorname{Me_2SiCp''_2ThH_2})_x + 2x \operatorname{RH} (3)$$

12

dihydride 12.7 While clearly a thorium dihydride¹⁰ with magnetically equivalent hydrido ligands at room temper-

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⁽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.

⁽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,

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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 $[Cp'_{2}Th(\mu-H)H]_{2}$ (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), $v_{M-H}/v_{M-D} = 1.41$) 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 $N_t = 6.5 \text{ h}^{-1}$) 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.

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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.

(17) By carrying out eq 3 with D_2 . Th-D modes other than at 904 cm⁻¹ are obscured by Me₂SiCp"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(CH₂)₂PPh₂]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-(CH₂)₂PPh₂]₂ catalyzes halogen exchange between CH₃Br and CD₃I as well as between [Au(CH₂)₂PPh₂]₂Br₂ and CH₃I in CDCl₃. 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 CH₃I.

While pursuing studies of the two-center two-electron oxidative addition of alkyl halides to the dinuclear gold(I) ylide complex¹ $[Au(CH_2)_2PPh_2]_2$, 1a, we observed a catalytic halogen-exchange reaction mediated by 1a. Many homogeneous catalytic reactions require sequential oxidative addition-reductive elimination cycles² 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 observed³ 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 Fackler¹ and Schmidbaur.^{2b} Complexes 2c and 2d were obtained by the addition of excess CH₃Br (Matheson) and CD₃I (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 CH₃Br 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 CHCl₃ 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 unreported⁶ 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.



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(6) In Schmidbaur's original report of the oxidative addition of CH₃I 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 CH₃I. See ref 2b and: Schmidbaur, H. Acc. Chem. Res. 1975, 8, 62.