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Table 4	Heck	reaction	with	TPPTS	39	and 3b [a]
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No.	R	Ligand	Yield of 9 [%] (<i>E</i> : <i>Z</i>) [b]	cat. [mol%]	TON [c]
1	COCH,	 3a	98 (89:11)	1	98
2	COCH,	3b	80 (95:5)	1	80
3	COCH	TPPTS	79 (92:8)	1	79
4	NO ₂	3a	85 (95:5)	0.1	850
5	NO,	3b	88 (95:5)	0.1	880
6	NO,	TPPTS	78 (94:6)	1	78

[a] 8 (15 mmol), 7 (22.5 mmol, 1.5 equiv), NaOAc \cdot 3 H₂O (16.5 mmol, 1.1 equiv), Pd(OAc)₂, Pd:L ratio 1:3, xylene (10 mL), ethylene glycol (10 mL), T = 130 °C, t = 20 h. [b] Yield of isolated product. [c] TON = turnover number: mole (product) per mole (catalyst).

achieved with as little as 0.1 mol% catalyst, as demonstrated by the treatment of 1-bromo-4-nitrobenzene with styrene. The improvement attained in the results for the new carbohydratephosphane ligands, as compared to those for TPPTS, is attributed to the greater concentration of catalyst in the nonpolar phase. The extent of the influence of electronic effects of the substituents on the ligand is currently under investigation.

Experimental Section

Compound 2 (1 equiv) and TBAHS (0.5 equiv) were dissolved in CH_2Cl_2 (ca. 0.4 M solution) and treated with the same volume of 1 M NaOH solution. After 5 min, 1 (5 equiv) was added and after 1 h the mixture was diluted with the 8-fold amount of ethyl acetate. The organic phase was washed with 1 M NaOH solution (2 ×), with water (2 ×), and with saturated NaCl solution (2 ×). After the organic phase had been dried over MgSO₄, the solvents were evaporated, and the residue taken up in methanol and treated with a little 1 M sodium methanolate solution. After 30 min the mixture was diluted with methanol, and the solution neutralized with acidic ion exchanger (Amberlyst 15). The ion eachanger was filtered off, the solvent evaporate ed, and the residue purified by column chromatography (silica gel 60) (CHCl₃/ MeOH/hexAne 6/1/1, v/v/v).

3a: C,H,N analysis calcd for $C_{26}H_{28}NO_6P \cdot 1.5 H_2O: C 61.41$, H 6.14, N 2.75; found: C 61.48, H 6.06, N 2.67; ¹H NMR (360 MHz, $[D_6]DMSO, 25^{\circ}C): \delta = 7.79$ (d, ³/(H,H) = 8.8 Hz, 1 H), 7.37 (m, 6H), 7.18 (m, 6H), 6.99 (d, ³/(H,H) = 8.1 Hz, 2 H), 5.09 (dd, ³/(H,H) = 5.2, ³/(H,H) = 13.2 Hz, 2 H), 5.00 (d, ³/(H,H) = 8.4 Hz, 1 H), 4.59 (m, 1 H), 3.68 (m, 2 H), 3.37 (s, 3 H), 3.18 (m, 1 H), 1.79 (s, 3 H); ¹³C NMR (90 MHz, $[D_6]DMSO, 25^{\circ}C): \delta = 169.3, 158.2, 137.2 (d, ¹/(C,P) = 11 Hz), 135.1 (d, ²/(C,P) = 11 Hz), 131.5 (d, ¹/(C,P) = 10 Hz), 132.9 (d, ²/(C,P) = 19 Hz), 128.7, 128.7 (d, ³/(C,P) = 11 Hz), 116.6 (d, ³/(C,P) = 8 Hz), 98.6, 77.2, 74.0, 70.3, 60.7, 55.4, 23.0; ³¹P NMR (101 MHz, <math>[D_6]DMSO, 25^{\circ}C): \delta = -2.7; IR (KBr): v = 3275, 3069, 2925, 1653, 1558, 1540, 1496, 1435, 1374, 1237, 1077, 825, 743, 696 cm⁻¹.$

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Activity of $[Sm(C_5Me_5)_3]$ in Ethylene Polymerization and Synthesis of $[U(C_5Me_5)_3]$, the First Tris(pentamethylcyclopentadienyl) 5f-Element Complex**

William J. Evans,* Kevin J. Forrestal, and Joseph W. Ziller

For decades it was thought that molecules of formula $[M(C_5Me_5)_3]$ were too sterically crowded to exist. Indeed, no examples had been found despite extensive work with a variety of C_5Me_5/M combinations. However, in 1991 the first tris(pentamethylcyclopentadienyl) complex was made simply by mixing the divalent complex $[Sm(C_5Me_5)_2]$ and cyclooctatetraene [Eq. (a)].^{[11} An improved synthesis of $[Sm(C_5Me_5)_3]$ was subsequently developed with the Sm^{II} precursor $[Sm(C_5Me_5)_2(Et_2O)]$ and $[Pb(C_5Me_5)_2].^{[21]}$

 $2[\operatorname{Sm}(\operatorname{C}_{5}\operatorname{Me}_{5})_{2}] + \operatorname{C}_{8}\operatorname{H}_{8} \longrightarrow [\operatorname{Sm}(\operatorname{C}_{5}\operatorname{Me}_{5})_{3}] + [(\operatorname{C}_{5}\operatorname{Me}_{5})\operatorname{Sm}(\operatorname{C}_{8}\operatorname{H}_{8})]$ (a)

The isolation of $[Sm(C_5Me_5)_3]$ implied that other $[M(C_5Me_5)_3]$ complexes should exist if M is of similar or greater size than Sm and if synthetic routes are available. However, to date the only successful syntheses of $[Sm(C_5Me_5)_3]$ have involved Sm^{II} precursors.^[2, 3] Since Sm^{II} has rather unique reactivity, ^[4] these routes are not available to any other metal. This is unfortunate since the interesting chemistry already shown for $[Sm(C_5Me_5)_3]$ (for example, the synthesis of complexes of thermally stable nonclassical carbonium ions) is incentive to make other $[M(C_5Me_5)_3]$ initiates ethylene polymerization and that this observation suggested a new route to $[Sm(C_5Me_5)_3]$, which can be applied to uranium to make $[U(C_5Me_5)_3]$.

 $[Sm(C_5Me_5)_3]$ polymerizes ethylene at 344 kPa in toluene in 1 hour to form polyethylene, whose molecular weight is too high to be studied by NMR or field desorption mass spectroscopy (FD-MS).^[6] The catalysis of ethylene polymerization by this sterically crowded molecule was unexpected since it contains

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none of the usual hydride, alkyl, or Sm^{II} initiating sites.^[6] The most likely route is via the η^1 -C₅Me₅ intermediate **A** [Eq. (b)], but no evidence for the η^1 structure was obtained by low temper-



ature NMR spectroscopy (-80° C in toluene). However, reaction of $[Sm(C_5Me_5)_3]$ with H₂ formed $[\{Sm(C_5Me_5)_2(\mu-H)\}_2]$ and C₅Me₅H [Eq. (c)], which is analogous to reactions of σ -bound alkyl complexes $[Sm(C_5Me_5)_2R]$ with H₂,⁽⁷¹ and represents a rare example of the hydrogenolysis of a pentamethyl-cyclopentadienyl ligand.

$$2[Sm(C_{5}Me_{5})_{3}] + 2H_{2} \longrightarrow [\{(C_{5}Me_{5})_{2}Sm(\mu-H)\}_{2}] + 2C_{5}Me_{5}H$$
(c)

If intermediate A is accessible with $[Sm(C_5Me_5)_3]$, β -hydrogen elimination could form $[{Sm(C_5Me_5)_2(\mu-H)}_2]$ and tetramethylfulvalene. Since this was not observed, we examined the reverse reaction and found that $[Sm(C_5Me_5)_3]$ could be obtained in greater than 90% yield from tetramethylfulvalene and $[{Sm(C_5Me_5)_2(\mu-H)}_2]$ [Eq. (d)].



This synthetic technique was applied to uranium to obtain the first tris(pentamethylcyclopentadienyl) complex of a 5f-element. $[U(C_5Me_5)_2H(dmpe)]^{[8]}$ reacted with tetramethylfulvalene to form $[U(C_5Me_5)_3]$ in 50% yield, which was characterized by elemental and chemical analysis, NMR and IR spectroscopy, magnetic susceptibility, and X-ray crystallography^[9] (Figure 1). The 36 Hz line width of the ¹H NMR C₅Me₅-ligated U^{III} complexes.^[8, 10, 11] Furthermore, reaction with D₂O exclusively formed D₂ as expected for a U^{III} complex.

 $[U(C_5Me_5)_3]$ is isostructural with $[Sm(C_5Me_5)_3]^{[1]}$ and has the same crystallographic $\overline{6}$ symmetry at the uranium center, which leads to only three unique U-C bond lengths and a rigorous 120° Cn-U-Cn angle (Cn = ring centroid). The average U-C bond length (2.84(4) Å) is equivalent within experimental error to that in $[Sm(C_5Me_5)_3]$ (2.82(5) Å). The Shannon radius for U^{III} is 0.067 Å larger than that of Sm^{III}.^[12] As in $[Sm(C_5Me_5)_3]$, the rings in $[U(C_5Me_5)_3]$ are oriented steric to minimize interactions. The progression U-C1>U-C2>U-C3 shows that each ring is tipped away from the uranium atom such that the U-Cn-C1 angle (94.1°) is larger than the idealized 90° and also larger than U-Cn-C2 (89.8°) and U-Cn-C3 (88.2°). The methyl groups are bent away



Figure 1. Molecular structure of $[U(C_5Me_5)_3]$. Thermal ellipsoids are drawn at the 50% probability level. Selected distances [Å]: U-C1 2.920(4), U-C2 2.840(3), U-C3 2.813(3), U-Cn 2.581.

from the center of the molecule, and the methyl carbon atoms lie out of the plane of the ring carbon atoms by 0.18 (C5) to 0.51 Å (C4). This methyl group displacement causes the Cn-C(ring)-C(methyl) angles to deviate from 180°; the angle containing C1 is distorted the most (Cn-C1-C4 162.5°, Cn-C2-C5 170.9°, Cn-C3-C6 167.3°).

Although tris(cyclopentadienyl) 5f-element complexes have been known for years,^[13] only a few containing sterically demanding η^5 -cyclopentadienyl ligands have been characterized by crystallography: [Th{1,3-(Me_3Si)_2C_5H_3}],^[14] [U(Me_3SiC_5H_4)_3],^[15] and [U(C_5Me_4H)_3].^[16] [U(C_5Me_5)_3] is the first example of a fully permethylated analog. Comparison of this structure with other C₅Me_5-ligated U^{III} complexes is limited to just three crystallographically characterized examples: [{U(C₅Me_5)_2(μ -Cl)}_3],^[10] [U(C_5Me_5)_2H(dmpe)],^[8] and [U(C₅Me_5)(C_8H_8)(4,4'-Me_2-bpy)].^[11] The average U–C distance in [U(C₅Me_5)_3] is much larger than in these compounds (2.77(1), 2.79(3), and 2.752 Å, respectively). Interestingly, the U–Cn distance to the C₅Me_5 ring (2.58 Å) is only slightly longer than that to the C₅Me_4H ring in [U(C₅Me_4H)_3(CO)] (2.53 Å).^[16]

The synthesis of $[U(C_5Me_5)_3]$ will allow the reactivity of $[M(C_5Me_5)_3]$ complexes of 5f and 4f elements to be compared. Preliminary studies have shown that $[U(C_5Me_5)_3]$ also initiates polymerization of ethylene to high molecular weight, but does not form the same CO reaction product as generated by $[Sm(C_5Me_5)_3]$.^[5] Extension of this synthetic technique to other metals should provide further examples.

Experimental Section

 $[Sm(C_5Me_5)_3]$: Addition of tetramethylfulvalene [17] (3 mg, 0.024 mmol) to $[{Sm(C_5Me_5)_2(\mu-H)}_2]$ [18] (10 mg, 0.012 mmol) in C_6D_6 (1 mL) in an argon glovebox caused an instantaneous color change of the solution from orange to dark brown. The product was identified by ¹H NMR spectroscopy as $[Sm(C_5Me_5)_3]$ and isolated by removal of solvent (13 mg, 95%).

[U(C₅Me₅)₃]: Addition of tetramethylfulvalene [17] (74.0 mg. 0.55 mmol) to [U(C₅Me₅)₂H(dmpe)] [8] (240 mg, 0.37 mmol) in toluene (5 mL) in an argon glovebox caused an immediate color change of the solution from black to brown. The solvent was removed to give a brown powder, and crystallization from hot toluene afforded very dark brown crystals (118 mg, 50%). ¹H NMR (500 MHz, 25°C, C₆D₆): $\delta = -0.93$. A frequent by-product of this reaction has a ¹H NMR resonance at $\delta = 7.89$, but this material has not yet been identified. ¹³C NMR (500 MHz, 25°C, C₆D₆): $\delta = 324.8$, -86.7. Magnetic susceptibility: $\chi_m = 4.0 \times$

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 $10^{-3} \text{ m}^3 \text{ mol}^{-1}$, $\mu_{err} = 3.1 \,\mu_B$; IR (KBr): $\tilde{\nu} = 2967$ (s), 2894 (s), 2869 (s), 1437 (m), 1370 (m), 1012 (w), 800 (w) cm⁻¹; elemental analysis calcd for $C_{30}H_{45}U$: C 56.00, H 7.00, U 37.00; found: C 55.53, H 7.21, U 37.40. Decomposition of $[U(C_5Me_5)_3]$ (330 mg, 0.513 mmol) with D₂O in a vessel connected to a Toepler pump forms D₂ (0.219 mmol, 86%).

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Stabilization of Iron Clusters by Polyolato Ligands and Calcium Ions: An Fe₁₄ Oxocluster from Aqueous Alkaline Solution

Joachim Burger and Peter Klüfers*

The oxoiron(III) core of ferritin and the iron(III) mineral ferrihydrite, $Fe_5O_3(OH)_0$, appear to adopt a similar structure.^[1] This structure has still not been resolved, and even the coordination number of the iron atoms is a matter of debate.^[2] Hexagonal densest packing (hdp) is assumed for the oxygen atoms, thus explaining the transformation paths into the thermodynamically stable hdp phases goethite, α -FeO(OH) (diaspore structure), and hematite, α -Fe₂O₃ (corundum structure).^[3] The high activation energy of the formation of crystalline phases indicates a fundamental structural reorganization in the course of the phase transition; this is apparently the reason for the metastability of noncrystalline ferric oxides/hydroxides.^[4] We report herein on an oxoiron(III) cluster, which is formed in aqueous solution, and which is stabilized by polyolato ligands and calcium ions. Hematite fragments that are considered to be characteristic for the ferrihydrite structure are combined in the sense of a microtwin; thus, the formation of a crystalline bulk phase is not possible by mere nucleus growth.

The nonchiral sugar alcohol erythritol (meso-butane-1,2,3,4tetraol) and ferric nitrate in the molar ratio 3:1 form green-yellow solutions on addition of aqueous sodium hydroxide solution $(c(Fe^{III}) = 0.25, c(OH^{-}) = 2.5 \text{ mol } L^{-1})$. Such greenish ferrate solutions may also be prepared by using simple diols in place of the tetraol.^[5] In the course of a few months, solutions containing erythritol that have been slowly enriched with calcium ions turn brown, thus indicating increasing aggregation of ferric species ("RT-solution"). Ion chromatography reveals formiate and further carboxylates due to incomplete exclusion of oxygen. However, partial oxidation is not a basic requirement for aggregation; a brown coloration can also be achieved within a few hours when the NaOH-containing solutions are heated to 55 °C.^[6] Subsequent addition of calcium nitrate causes calcium hydroxide to initially precipitate, but on continued stirring, redissolution takes place ("55 °C-solution"). Light scattering experiments on the RT-solution revealed particles of a hydrodynamic radius of about 2 nm; the particle radii ranged between 1 and 10 nm. Analysis of the 55 °C-solution revealed a wider distribution of the particle radii.^[7] Solely in terms of the hydrodynamic radius, the oxoiron(III) cation described in the following is a typical, but not the only ferric species, in the brown solutions.

Half a year after the preparation several brown, well-formed tetragonal crystals formed in the carboxylate-containing RT-solution. X-ray crystal structure analysis^[8] reveals the formula **1**

 $[Ca_{12}(H_2O)_{24}Fe_{14}(\mu_3-O)_{12}(\mu_2-OH)_4(OH)_{12}(LH_{-3})_4(LH_{-2})_4]X_6+8H_2O-1$

(L = erythritol, X = formiate and other oxidation products of erythritol;⁽⁹⁾ μ_n refers exclusively to Fe, not to Ca).

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