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A Compound with a Hf-Hf Bond. The First Structurally Characterized Hafnium(III) Complex: $\text{Hf}_2\text{Cl}_6[(i\text{-Pr})_2\text{PCH}_2\text{CH}_2\text{P}(i\text{-Pr})_2]_2$

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Reduction of ZrCl_4 or HfCl_4 with Na/K alloy in the presence of the chelating phosphine 1,2-bis(diisopropylphosphino)ethane (dippe) gives the metal-metal-bonded dimers $\text{Zr}_2\text{Cl}_6(\text{dippe})_2$ and $\text{Hf}_2\text{Cl}_6(\text{dippe})_2$. These dark green compounds are diamagnetic as opposed to the previously reported paramagnetic titanium(III) dimer $\text{Ti}_2\text{Cl}_6(\text{dippe})_2$. The hafnium compound is the first molecular hafnium(III) species of any kind to be crystallographically characterized. The dimer is composed of two octahedral hafnium centers that are connected by two bridging chloride ligands: the Hf-Hf bond length is 3.099 (1) Å. Overall, the structure closely resembles analogous zirconium(III) dimers that have been described previously. Other distances and angles in the molecule are Hf-P = 2.746 (5) Å, Hf-Cl_a = 2.408 (4) Å, Hf-Cl_b = 2.517 (4) Å, Hf-Cl_b-Hf = 76.0 (1)°, Cl_b-Hf-Cl_b = 104.0 (1)°, Cl_a-Hf-Cl_a = 166.5 (1)°, and P-Hf-P = 76.7 (1)°. Crystal data for $\text{Hf}_2\text{Cl}_6(\text{dippe})_2\cdot\text{PhMe}$: orthorhombic, space group *Pbcn*, with *a* = 15.176 (2) Å, *b* = 14.503 (4) Å, *c* = 21.473 (4) Å, *V* = 4726 (4) Å³, *Z* = 4, *R_F* = 0.046, and *R_{wF}* = 0.053 for 179 variables and 1791 data with *I* > 2.58σ(*I*).

Introduction

The chemistry of hafnium is dominated by the +4 oxidation state.¹⁻³ The reduction potentials of hafnium are the most negative of all the transition elements and are significantly exceeded only by certain of the alkali metals, alkaline earth metals, and lanthanide metals.⁴ Accordingly, the lower oxidation states of hafnium are unusually difficult to prepare. A few high-temperature routes to the lower hafnium halides have been reported,⁵⁻⁷ and the only coordination complex of Hf^{III}, $\text{HfCl}_3(\text{pyridine})_4$, has never been described in the open literature.⁸ Only three organometallic derivatives of hafnium(III) have been isolated: $[\text{Cp}_2\text{Hf}(\text{PR}_2)_2]_2$,^{9,10} and $[\text{CpHf}(\mu\text{-}\eta^1, \eta^5\text{-C}_5\text{H}_4)(\text{PMe}_3)_2]_2$ ¹¹ have been formulated as dimers on the basis of their diamagnetic NMR behavior, while $[\text{C}_5\text{H}_3(t\text{-Bu})_2]_2\text{HfCl}$ is paramagnetic and has been formulated as a monomer.¹² Transient Hf^{III} monomers have been observed by EPR spectroscopy to result from the reduction of $(\text{C}_5\text{H}_4\text{SiMe}_3)_2\text{HfR}_2$ and $\text{Cp}_2\text{Hf}(\text{PR}_2)_2$ complexes with sodium naphthalene, but none of these species has been isolated.¹³⁻¹⁵ A few organohafnium complexes in oxidation states lower than +3 have also been reported, including anionic carbonyl complexes,^{16,17} several mono- and bis(cyclopentadienyl) compounds,¹⁸⁻²⁴ and some Hf⁰ phosphine and arene complexes.²⁵⁻²⁸

Table I. Crystallographic Data for $\text{Hf}_2\text{Cl}_6(\text{dippe})_2\cdot\text{PhMe}$ at -75 °C

$\text{C}_{35}\text{H}_{72}\text{Cl}_6\text{Hf}_2\text{P}_4$	mol wt = 1186.56
space group: <i>Pbcn</i>	$\lambda = 0.71073$ Å
<i>a</i> = 15.176 (2) Å	$\rho_{\text{calcd}} = 1.668$ g cm ⁻³
<i>b</i> = 14.503 (4) Å	$\mu_{\text{calcd}} = 48.51$ cm ⁻¹
<i>c</i> = 21.473 (4) Å	transm coeff: 0.306-0.826
<i>V</i> = 4726 (4) Å ³	<i>R_F</i> = 0.046
<i>Z</i> = 4	<i>R_{wF}</i> = 0.053

Table II. Atomic Coordinates for $\text{Hf}_2\text{Cl}_6(\text{dippe})_2\cdot\text{PhMe}$

	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>
Hf	0.08802 (4)	0.04629 (4)	0.01896 (3)
Cl(1)	-0.0491 (3)	0.0283 (3)	0.0835 (2)
Cl(2)	0.0516 (3)	0.1995 (3)	-0.0153 (2)
Cl(3)	0.1572 (3)	-0.0907 (3)	0.0598 (2)
P(1)	0.2525 (3)	0.0877 (3)	-0.0263 (3)
P(2)	0.1684 (3)	0.1324 (4)	0.1179 (2)
C(1A)	0.315 (2)	0.121 (2)	0.047 (1)
C(2A)	0.264 (1)	0.195 (2)	0.080 (1)
C(1B)	0.312 (2)	0.159 (2)	0.0345 (8)
C(2B)	0.2893 (7)	0.120 (2)	0.097 (1)
C(3)	0.255 (1)	0.177 (1)	-0.086 (1)
C(4)	0.348 (1)	0.221 (1)	-0.099 (1)
C(5)	0.211 (1)	0.147 (1)	-0.1449 (10)
C(6)	0.330 (1)	-0.001 (1)	-0.052 (1)
C(7)	0.283 (1)	-0.070 (1)	-0.0945 (10)
C(8)	0.382 (1)	-0.046 (1)	-0.001 (1)
C(9A)	0.208 (2)	0.053 (2)	0.179 (1)
C(9B)	0.185 (2)	0.069 (2)	0.1916 (9)
C(10A)	0.241 (3)	0.117 (3)	0.232 (2)
C(10B)	0.267 (2)	0.070 (2)	0.233 (2)
C(11A)	0.131 (2)	0.000 (3)	0.210 (2)
C(11B)	0.101 (2)	0.031 (2)	0.223 (2)
C(12A)	0.109 (2)	0.235 (1)	0.146 (2)
C(12B)	0.159 (1)	0.2582 (6)	0.129 (1)
C(13A)	0.030 (2)	0.222 (3)	0.190 (2)
C(13B)	0.059 (2)	0.266 (2)	0.144 (2)
C(14A)	0.155 (2)	0.320 (2)	0.174 (2)
C(14B)	0.216 (2)	0.293 (2)	0.183 (1)
C(15A)	0.502 (3)	0.279 (2)	0.233 (2)
C(16A)	0.499	0.207	0.190
C(17A)	0.499	0.116	0.210
C(18A)	0.500	0.096	0.273
C(19A)	0.502	0.167	0.317
C(20A)	0.503	0.259	0.297
C(21A)	0.502	0.378	0.211
C(15B)	0.505 (3)	0.191 (2)	0.276 (2)
C(16B)	0.505	0.157	0.215
C(17B)	0.502	0.063	0.204
C(18B)	0.499	0.001	0.254
C(19B)	0.499	0.034	0.315
C(20B)	0.502	0.129	0.326
C(21B)	0.508	0.293	0.288

We now describe the preparation and characterization of the first well-defined hafnium(III) coordination complex, Hf_2Cl_6 -

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Table III. Selected Bond Distances (Å) and Angles (deg) for $\text{Hf}_2\text{Cl}_6(\text{dippe})_2\cdot\text{PhMe}$

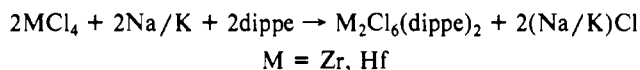
Bond Distances			
Hf-Hf'	3.099 (1)	P(1)-C(1A)	1.90 (2)
Hf-Cl(1)	2.513 (4)	P(1)-C(3)	1.82 (2)
Hf-Cl(1')	2.521 (4)	P(1)-C(6)	1.84 (2)
Hf-Cl(2)	2.405 (4)	P(2)-C(2A)	1.90 (2)
Hf-Cl(3)	2.411 (4)	P(2)-C(9A)	1.85 (3)
Hf-P(1)	2.744 (5)	P(2)-C(12A)	1.85 (2)
Hf-P(2)	2.749 (5)	C(1A)-C(2A)	1.50 (4)

Bond Angles			
Cl(2)-Hf-Cl(3)	166.5 (1)	Cl(1)-Hf-Cl(2)	94.3 (1)
Cl(1)-Hf-P(1)	166.1 (1)	Cl(1)-Hf-Cl(3)	94.3 (1)
Cl(1)-Hf-P(2)	166.6 (1)	Cl(2)-Hf-Cl(1')	94.3 (1)
P(1)-Hf-P(2)	76.7 (1)	Cl(2)-Hf-P(1)	84.2 (1)
Hf-Cl(1)-Hf'	76.0 (1)	Cl(2)-Hf-P(2)	85.4 (2)
Cl(1)-Hf-Cl(1')	104.0 (1)	Cl(3)-Hf-Cl(1')	93.8 (1)
Cl(1)-Hf-P(2)	89.3 (1)	Cl(3)-Hf-P(1)	85.0 (1)
Cl(1)-Hf-P(1)	89.9 (1)	Cl(3)-Hf-P(2)	84.3 (2)

(dippe)₂, where dippe is the bulky chelating ligand 1,2-bis(diisopropylphosphino)ethane. The X-ray crystal structure of this dimer is the first of a molecule that contains a Hf-Hf bond.

Results

Treatment of a toluene suspension of ZrCl_4 or HfCl_4 with sodium-potassium alloy in the presence of dippe yields a dark green or brown solution from which the trivalent dimer $\text{Zr}_2\text{Cl}_6(\text{dippe})_2$ or $\text{Hf}_2\text{Cl}_6(\text{dippe})_2$ may be isolated.



Sodium-potassium alloy is the preferred reducing agent, since HfCl_4 in particular reacts only slowly or not at all with other reductants such as sodium or sodium amalgam. The zirconium(III) and hafnium(III) complexes are diamagnetic as judged by their sharp ^1H NMR resonances. The number of phosphine ^1H NMR resonances (one backbone methylene PCH_2 signal, one isopropyl methine CHMe_2 signal, and two isopropyl methyl CHMe_2 signals) is consistent with a dimeric chloride-bridged structure in which the dippe ligands are trans to the bridging chlorides.

Discussion

X-ray Crystal Structure of $\text{Hf}_2\text{Cl}_6(\text{dippe})_2$. Single crystals of $\text{Hf}_2\text{Cl}_6(\text{dippe})_2\cdot\text{PhMe}$ were obtained by cooling saturated toluene solutions to -20°C . Crystal data are given in Table I, while final atomic coordinates and important bond distances and angles are given with estimated standard deviations in Tables II and III.

The structural analysis shows that crystals of $\text{Hf}_2\text{Cl}_6(\text{dippe})_2$ are composed of discrete chloride-bridged dimers that reside on crystallographic inversion centers (Figure 1). The molecules possess nearly ideal D_{2h} symmetry, which is broken only by the twisting of the dippe backbone. The unit cell also contains one disordered toluene molecule per dimer unit.

The hafnium centers are connected by two bridging chlorides, and the Hf-Hf bond length of 3.099 (1) Å is consistent with the presence of a metal-metal single bond. This distance is com-

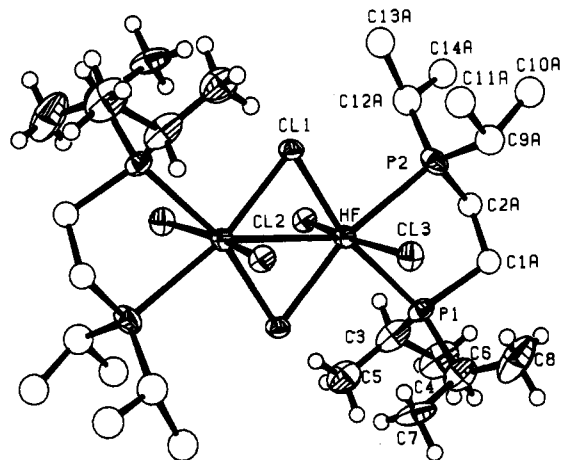


Figure 1. Molecular structure of $\text{Hf}_2\text{Cl}_6(\text{dippe})_2$. Only one orientation of the disorder involving the dippe carbon atoms is shown. Thermal ellipsoids are drawn at the 35% probability level.

parable to the Zr-Zr bond lengths of 3.104 (2) to 3.182 (1) Å in analogous $\text{Zr}_2\text{Cl}_6(\text{PR}_3)_4$ dimers (Table IV).^{29,30} The attractive force between the two metals is evident from the opening of the $\text{Cl}_b\text{-Hf-Cl}_b$ angles to ca. 103° from the octahedral value of 90° . By contrast, in the paramagnetic titanium(III) dimer $\text{Ti}_2\text{Cl}_6(\text{dippe})_2$,³¹ the $\text{Ti}\cdots\text{Ti}$ distance is much longer at 3.438 (2) Å and the $\text{Cl}_b\text{-Ti-Cl}_b$ angle of $91.4 (1)^\circ$ is very near the value for an ideal octahedron. The titanium complex is best considered to lack a metal-metal bond.

The bridging chloride groups in $\text{Hf}_2\text{Cl}_6(\text{dippe})_2$ possess acute Hf- Cl_b -Hf angles of $76.0 (1)^\circ$; this angle is the geometric complement of the $\text{Cl}_b\text{-Hf-Cl}_b$ angle due to the crystallographically imposed planarity of the Hf_2Cl_2 core. The bridging Hf- Cl_b distances of 2.517 (4) Å are significantly longer than the terminal Hf- Cl_t distances of 2.408 (4) Å, as expected. The terminal chloride ligands on each hafnium center are displaced from their ideal octahedral positions to minimize $\text{Cl}_t\cdots\text{Cl}_t$ nonbonded interactions between the terminal chloride ligands on the adjacent metal centers. The $\text{Cl}_t\text{-Hf-Cl}_t$ angle of $166.5 (1)^\circ$ is only slightly larger than the $162\text{--}165^\circ$ angles in the analogous zirconium(III) dimers. The $\text{Cl}_t\text{-Ti-Cl}_t$ angle of $169.6 (2)^\circ$ in $\text{Ti}_2\text{Cl}_6(\text{dippe})_2$ is more nearly linear due to the greater $\text{Ti}\cdots\text{Ti}$ separation and correspondingly reduced nonbonded repulsions between the ligand sets on the two metal centers.

Both the Hf- Cl_t and Hf- Cl_b distances are ca. 0.02 Å shorter than the corresponding Zr-Cl distances in the zirconium complexes due to the slightly smaller ionic radius of hafnium. For a presumably similar reason, the Hf-P distance is ca. 0.05 Å shorter than that in $\text{Zr}_2\text{Cl}_6(\text{dppe})_2$ (dppe = 1,2-bis(diphenylphosphino)ethane) although the presence of different phosphine ligands in the two complexes complicates quantitative comparisons. The P-Hf-P angle of $76.7 (1)^\circ$ in $\text{Hf}_2\text{Cl}_6(\text{dippe})_2$ is essentially identical with the analogous angle in $\text{Zr}_2\text{Cl}_6(\text{dppe})_2$; the bite angle of the phosphine in $\text{Ti}_2\text{Cl}_6(\text{dippe})_2$ of $78.6 (2)^\circ$ is slightly larger due to the smaller size of titanium and the correspondingly shorter Ti-P distances.

Concluding Remarks. The present results constitute the first structural study of a compound that contains a hafnium-hafnium bond. For the first time, there exists for every transition element in the periodic table a molecular compound in which metal-metal bonding has been demonstrated crystallographically.³² The Hf-Hf distance of 3.099 (1) Å in $\text{Hf}_2\text{Cl}_6(\text{dippe})_2$ is comparable to the

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Table IV. Selected Bond Distances (Å) and Angles (deg) for Group 4 $M_2Cl_6(PR_3)_4$ Complexes

compd	M-M	M-P	P-M-P	M-Cl _b	Cl _b -M-Cl _b	M-Cl _t	Cl _t -M-Cl _t	ref
Hf ₂ Cl ₆ (dippe) ₂	3.102 (3)	2.73 (1)	76.2 (5)	2.52 (1)	104.0 (4)	2.40 (1)	166.2 (4)	this work
Zr ₂ Cl ₆ (dppe) ₂ ^a	3.104 (2)	2.781 (3)	75.9 (1)	2.536 (3)	104.57 (8)	2.420 (3)	162.8 (1)	29
Zr ₂ Cl ₆ (PMe ₂ Ph) ₄	3.127 (1)	2.765 (3)	96.19 (8)	2.547 (3)	104.26 (8)	2.417 (3)	162.1 (1)	29
Zr ₂ Cl ₆ (PEt ₃) ₂	3.169 (1)	2.813 (2)	96.02 (6)	2.540 (2)	102.81 (5)	2.425 (2)	163.77 (6)	29
Zr ₂ Cl ₆ (PBu ₃) ₄	3.182 (1)	2.835 (2)	96.25 (5)	2.544 (2)	102.57 (5)	2.431 (2)	165.11 (6)	30
Ti ₂ Cl ₆ (dippe) ₂	3.438 (2)	2.652 (5)	78.6 (2)	2.461 (4)	91.4 (1)	2.312 (4)	169.6 (2)	31

^a Average of two independent molecules.

Hf-Hf distances of 3.159 Å in bulk hafnium metal.

The highly negative redox potentials of hafnium suggest that there should be a rich and varied chemistry of Hf₂Cl₆(dippe)₂ that involves reduction of substrate molecules and formation of Hf^{IV} products. Such reactivity has been demonstrated for some of the analogous Zr^{III} dimers,^{30,33} and we are currently pursuing a comparative study of the reactivity of the Hf^{III} oxidation state.

Experimental Section

All operations were carried out under vacuum or under argon. Toluene was distilled under nitrogen from sodium. The halides ZrCl₄ and HfCl₄ were obtained commercially and sublimed before use. The sodium/potassium alloy had a stoichiometry of NaK_{2.8} and was prepared according to a literature procedure.³⁴ The phosphine 1,2-bis(diisopropylphosphino)ethane was prepared as described previously.³⁵

Microanalyses were performed by Tom McCarthy of the University of Illinois Microanalytical Laboratory. The IR spectra were recorded as Nujol mulls on a Perkin-Elmer 599B instrument using CsI plates. The ¹H NMR data were recorded on a General Electric QE-300 spectrometer at 300 MHz. Chemical shifts are reported in δ units (positive shifts to high frequency) relative to TMS. Melting points were determined on a Thomas-Hoover Unimelt apparatus in sealed capillaries under argon.

Hexachlorobis[1,2-bis(diisopropylphosphino)ethane]dizirconium(III). To a suspension of ZrCl₄ (0.81 g, 3.5 mmol) and dippe (1.1 mL, 4.0 mmol) in toluene (80 mL) was added Na/K alloy (1.0 mL, 31 mmol). The solution turned pale green immediately; after being stirred for 16 h it was dark green-brown, and a green solid had separated. The solvent was removed under vacuum, and the residue was extracted with toluene (3 × 80 mL). The filtered extracts were concentrated to ca. 150 mL and cooled to -20 °C to give green microcrystals of the product. Yield: 0.56 g (35%). Mp: >190 °C. Although the crystals when originally isolated contain lattice toluene, the crystals readily desolvate. Anal. Calcd for Zr₂C₂₈H₆₄Cl₆P₄: C, 36.6; H, 7.01; Cl, 23.2. Found: C, 36.2; H, 6.98; Cl, 23.2. ¹H NMR (C₆D₆, 20 °C): δ 2.34 (d septets, *J*_{PH} = 7 Hz, *J*_{HH} = 7 Hz, CHMe₂), 1.68 (d, *J*_{PH} = 12 Hz, PCH₂), 1.49 (dd, *J*_{PH} = 14 Hz, *J*_{HH} = 7 Hz, CHMe₂), 1.27 (dd, *J*_{PH} = 12 Hz, *J*_{HH} = 7 Hz, CHMe₂). IR (Nujol, cm⁻¹): 2722 w, 1415 m, 1295 w, 1247 m, 1159 m, 1051 s, 962 w, 928 m, 886 m, 857 m, 816 m, 738 m, 729 m, 677 s, 655 m, 630 w, 611 m, 514 w, 466 w, 322 s.

Hexachlorobis[1,2-bis(diisopropylphosphino)ethane]dihafnium(III). To a suspension of HfCl₄ (0.25 g, 0.80 mmol) and dippe (0.25 mL, 0.90 mmol) in toluene (70 mL) was added Na/K alloy (0.3 mL, 9.0 mmol). The solution turned pale green immediately and after being stirred for 2 days was yellow-brown. The solution was filtered and cooled to -20 °C to yield dark green crystals. Yield: 0.25 g (57%). Mp: >190 °C. The isolated crystals contain variable amounts of lattice toluene; larger crystals generally retain more toluene. Anal. Calcd for Hf₂C₂₈H₆₄Cl₆P₄: C, 30.7; H, 5.89; Cl, 19.4. Found: C, 31.7; H, 6.05; Cl, 18.3. ¹H NMR (C₆D₆, 20 °C): δ 2.39 (d septets, *J*_{PH} = 7 Hz, *J*_{HH} = 7 Hz, CHMe₂), 1.68 (d, *J*_{PH} = 11 Hz, PCH₂), 1.51 (dd, *J*_{PH} = 14 Hz, *J*_{HH} = 7 Hz, CHMe₂), 1.28 (dd, *J*_{PH} = 13 Hz, *J*_{HH} = 7 Hz, CHMe₂). IR (Nujol, cm⁻¹): 1415 m, 1294 w, 1245 m, 1160 m, 1135 m, 1049 s, 928 m, 886 m, 864 m, 815 m, 728 m, 682 m, 674 m, 653 m, 630 w, 610 m, 591 w, 467 m, 296 s.

Crystallographic Studies. Complete details of the general crystallographic methods have been published previously.³⁶ Single crystals of Hf₂Cl₆(dippe)₂·PhMe, grown from toluene, were mounted on a thin glass fiber with oil (Paratone-N, Exxon) immediately after isolation to prevent

desolvation and were quickly cooled to -75 °C in a cold nitrogen gas stream on the diffractometer. Preliminary photographs gave rough cell dimensions, and standard peak search and automatic indexing procedures, followed by least-squares refinement using 25 reflections, yielded the cell dimensions given in Table I.

Data were collected in one octant of reciprocal space (-*h*, -*k*, -*l*), and additional low-angle data were collected in all eight octants (2.0° < 2θ < 9.0°) by using measurement parameters listed in Table I. Systematic absences for 0*kl* where *k* ≠ 2*n*, *h*0*l* where *l* ≠ 2*n*, and *hk*0 where *h* + *k* ≠ 2*n* were consistent only with space group *Pbcn*. The measured intensities were reduced to structure factor amplitudes and their esd's by correction for background, scan speed, and Lorentz and polarization effects. While corrections for crystal decay were unnecessary, absorption corrections were applied. Systematically absent reflections were deleted, and symmetry-equivalent reflections were averaged to yield the set of unique data. Only those data with *I* > 2.58σ(*I*) were used in the least-squares refinement.

The structure was solved by Patterson and weighted difference Fourier methods. The positions of the hafnium atoms were deduced from a sharpened Patterson map, and partial structure expansion gave positions for the chlorine and phosphorus atoms. Subsequent difference Fourier calculations revealed the positions of the remaining non-hydrogen atoms. The quantity minimized by the least-squares program was $\sum w(|F_o| - |F_c|)^2$, where $w = 0.90/(\sigma(F_o)^2 + (pF_o)^2)$. The analytical approximations to the scattering factors were used, and all structure factors were corrected for both the real and imaginary components of anomalous dispersion. The ethylene backbone and the two isopropyl groups on P(2) of the dippe ligand were disordered, as were the toluene solvate molecules. The relative site occupancy factors converged to 0.446 (5) for disorder "A" of the dippe ligand and to 0.260 (3) for disorder "A" of the toluene solvate molecules. In each dampened cycle of least-squares refinement, the bond lengths involving the disordered carbon atoms were refined with a common variable in groups as follows: [P(1)-C(1A), P(1)-C(1B), P(2)-C(2A), P(2)-C(2B)], [P(2)-C(9A), P(2)-C(9B), P(2)-C(12A), P(2)-C(12B)], [C(9A)-C(10A), C(9B)-C(10B), C(9A)-C(11A), C(9B)-C(11B), C(12A)-C(13A), C(12B)-C(13B), C(12A)-C(14A), C(12B)-C(14B)], and [C(1A)-C(2A), C(1B)-C(2B)]. The toluene solvate molecules, which were disordered about the 2-fold axis, were constrained to idealized geometries, and hydrogen atoms on ordered carbon atoms were included as fixed contributors in idealized locations with C-H = 0.95 Å. In the final cycle of least squares, all ordered non-hydrogen atoms were independently refined with anisotropic thermal coefficients, and common isotropic thermal parameters were varied for the hydrogen atoms and the disordered carbon atoms. Successful convergence was indicated by the maximum shift/error of 0.11 in the last cycle. Final refinement parameters are given in Table I. The largest peaks in the final difference Fourier map were located in the vicinity of the hafnium atom. A final analysis of variance between observed and calculated structure factors showed a slight inverse dependence on sin θ.

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Supplementary Material Available: Tables S1-S4, giving full crystallographic details, calculated hydrogen atom positions, anisotropic thermal parameters, and complete bond distances and angles for Hf₂Cl₆(dippe)₂·PhMe (3 pages); a table of final observed and calculated structure factors for Hf₂Cl₆(dippe)₂·PhMe (8 pages). Ordering information is given on any current masthead page.

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