Supplementary Material (ESI) for New Journal of Chemistry

Supporting Information
X-Ray Data

Organometallic Compounds Containing new Hf–Ga and Hf–In Bonds: $Cp_2Hf(ER)_2$ (Cp = C_5H_5; E = Ga, In; R = 2,6-(2,4,6-*i*-$Pr_3C_6H_2)_2C_6H_3$-)

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Data Collection Compound 1: $Cp_2Hf(GaR)_2$

Experimental

Data Collection

Black-purple crystals were mounted and sealed in a glass capillary under nitrogen. The X-ray intensity data were measured at room temperature on a Bruker SMART APEX II X-ray diffractometer system with graphite-monochromated Mo Kα radiation (λ = 0.71073 Å) ω-scan technique. The data were collected in a hemisphere of data in 1664 frames with 10 second exposure times. Crystallographic data: $C_{86}H_{118}OGa_2Hf$: a = 15.879(4) Å, b = 17.556(4) Å, c = 29.381(6) Å, α= 90°, β = 90° , γ = 90°, V = 8191(3) Å^3, Z = 4, F.W. = 1485.74, μ = 1.958 mm^{-1}, d = 1.205 g/cm^3, F(000) = 3104.

Data Reduction [1]

Of the 7217 unique reflections collected, 3120 were observed ($I > 2\ \sigma(I)$). The linear absorption coefficient for Mo Kα radiation is 1.958 mm^{-1}. The data were corrected for Lorentz and polarization effects and integrated with the manufacturer's SAINT software. Absorption corrections were applied with the SADABS.

Structure Solution and Refinement [2]

Subsequent solution and refinement was performed using the SHELXTL 6.1[3] solution package operating on a Pentium computer. The structure was solved by direct methods using the SHELXTL 6.1 Software Package. Non-hydrogen atomic scattering factors were taken from the literature tabulations.[4] The heavy atom positions were determined using direct methods employing the SHELXTL routine methods. The remaining non-hydrogen atoms were located from successive difference Fourier map calculations. The Cp and discrete solvent ether molecule (in half) are found disordered in adjacent positions in two sets labeled as C(37), C(38), C(39), C(40), C(41) (one set) and C(37'), C(38'), C(39'), C(40'), C(41') (another set) for the Cp group, and as C(42), C(43) (one set), and C(42'), C(43') (another set) for solvent ether, respectively. Each of these two sets is divided using the PART command and the corresponding hydrogen atoms are added in the correct calculated positions. The occupancies of all atoms for both disordered Cp and half a solvent ether molecule have been set at 0.50 (requested by the referee). In the final cycles of each refinement, the non-hydrogen atoms were refined anisotropically except for the half a discrete disordered solvent ether molecule in which the positions and temperature factors of atoms are constrained (see explanation below*). Hydrogen atom positions were calculated and allowed to ride on the carbon to which they are bonded assuming a C–H bond length of 0.95 Å. Hydrogen atom temperature factors were fixed at 1.10 times the isotropic temperature factor of the C-atom to which they are bonded. The crystal system of compound is orthorhombic, space group Pbcn (No. 60) and the final residual values based

on 431 variable parameters and 3120 observed reflections ($I > 2\sigma(I)$) are $R1 = 0.0614$, $wR2 = 0.1563$, and those for all unique reflections are $R1 = 0.1602$, $wR2 = 0.2307$. The goodness-of-fit indicator for all data is 1.013. Peaks on the final difference map ranged from 0.979 to -2.310e/Å^3, which are of no chemical significance.

Summary
The compound crystallizes in orthorhombic, space group Pbcn (No. 60). The asymmetric unit contains half a molecule in the form of $C_{41}H_{54}GaHf_{0.5}\cdot 0.5O(C_2H_5)_2$ with the Hf atom located at a special position with a two-fold axis symmetry. The whole molecule is in the form of $C_{82}H_{108}Ga_2Hf\cdot O(C_2H_5)_2$ in which the Hf atom is coordinated by two Cp and two Ga atoms bonded with m-terphenyl ligands. Structure solution, refinement and the calculation of derived results were performed using the SHELXTL 6.1[3] package of computer programs. Neutral atom scattering factors were those of Cromer and Waber,[4] and the real and imaginary anomalous dispersion corrections were those of Cromer.[5]

References

1. Data Reduction:

Intensity

$I = [S - B/R]\cdot V$

Standard Deviation in Intensity

$s(I) = [S + B/R^2]^{1/2}\cdot V$

Structure Factor

$F = (I/Lp)^{1/2}$

Standard Deviation in Structure Factor

$\sigma(F) = \sigma(I)/(2\cdot F\cdot Lp)$

Where: S = total scan count
B = sum of background counts
R = ratio of background counting time to scan counting time
V = scan rate
Lp = Lorentz-polarization correction

2. Least-Squares Refinement:

Weighting Scheme

$w = 1/[\sigma^2(F_o) + 0.0954*P)^2 + 34.2000*P]$ where $P = [\mathrm{Max}(F_o^2, 0) + 2*F_c^2]/3$;

Residuals

R-factors:

$R = \Sigma\,||F_o| - |F_c||/\Sigma|F_o|$;

Weighted R-factor on F^2:

$wR2 = [\Sigma[w\,(F_o^2 - F_c^2)^2] / \Sigma[w(F_o^2)^2]]^{1/2}$;

Goodness of Fit Indicator:

goodness-of-fit $= [\Sigma[w\,(F_o^2 - F_c^2)^2] /(N_{observns} - N_{params})\,]^{1/2}$.

3. Sheldrick, G.M. *SHELXTL 6.1*, *Crystallographic Computing System*; Siemens Analytical X-Ray Instruments: Madison, WI, 2000.

4. Cromer, D.T. and Waber, J.T. International Tables for X-ray Crystallography, Vol. IV, Table 2.2B, The Kynoch Press, Birmingham England, 1974.

5. Cromer, D.T. International Tables for X-ray Crystallography, Vol. IV, Table 2.3.1, The Kynoch Press, Birmingham England, 1974.

*The remaining electron densities of the half a discrete solvent ether molecule in the difference Fourier map are barely invisible due to reflection intensities from it being very weak since diffraction data were taken at ambient temperature. Attempts to establish a disordered model for half an ether molecule with fully anisotropic refinement have been made unsuccessfully, resulting in either explosion of model or unbearably high displacement parameters (far beyond reasonable values) even with appropriate restraints. In order to make chemical sense for solvent ether molecule, the disordered model has to be constrained and refined only isotropically.

Table 1. Crystal data and structure refinement for Compound **1**.

Identification code	Compound **1**
Empirical formula	C86 H118 Ga2 Hf O
Formula weight	1485.74
Temperature	273(2) K
Wavelength	0.71073 A
Crystal system, space group	Orthorhombic, Pbcn
Unit cell dimensions	a = 15.879(4) A alpha = 90 deg.
	b = 17.556(4) A beta = 90 deg.
	c = 29.381(6) A gamma = 90 deg.

Volume	8191(3) A^3
Z, Calculated density	4, 1.205 Mg/m^3
Absorption coefficient	1.958 mm^-1
F(000)	3104
Crystal size	0.20 x 0.10 x 0.05 mm
Theta range for data collection	2.22 to 25.00 deg.
Limiting indices	-18<=h<=14, -20<=k<=20, -34<=l<=34
Reflections collected / unique	41723 / 7217 [R(int) = 0.1355]
Completeness to theta = 25.00	99.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9084 and 0.6955
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	7217 / 141 / 431

Goodness-of-fit on F^2	1.013
Final R indices [I>2sigma(I)]	R1 = 0.0614, wR2 = 0.1563
R indices (all data)	R1 = 0.1602, wR2 = 0.2307
Largest diff. peak and hole	0.979 and -2.310 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for compound **1**.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Hf(1)	0	2157(1)	7500	52(1)
Ga(1)	39(1)	1206(1)	6814(1)	67(1)
C(1)	129(7)	602(6)	6230(3)	50(3)
C(2)	-543(7)	153(6)	6081(4)	52(3)
C(3)	-473(8)	-343(7)	5729(4)	67(4)
C(4)	306(7)	-404(7)	5490(4)	65(3)
C(5)	963(7)	56(7)	5618(4)	58(3)
C(6)	888(7)	565(6)	5983(4)	52(3)
C(7)	1617(7)	1089(7)	6069(4)	54(3)
C(8)	1577(8)	1827(8)	5894(4)	69(4)
C(9)	2296(9)	2295(7)	5955(5)	80(4)
C(10)	2992(8)	2070(9)	6172(5)	75(4)
C(11)	2989(8)	1352(9)	6348(4)	74(4)
C(12)	2318(8)	841(7)	6299(4)	62(3)
C(13)	2403(8)	54(8)	6483(5)	70(3)
C(14)	2992(9)	-448(8)	6202(6)	110(5)

C(15)	2723(11)	5(9)	6972(5)	117(6)
C(16)	3752(11)	2621(11)	6223(7)	126(7)
C(17)	4405(12)	2461(13)	5918(7)	170(10)
C(18)	3849(12)	2917(12)	6652(7)	187(12)
C(19)	849(9)	2104(8)	5612(5)	84(4)
C(20)	534(13)	2876(10)	5762(7)	151(8)
C(21)	1072(13)	2114(11)	5106(6)	146(8)
C(22)	-1409(7)	254(7)	6300(4)	56(3)
C(23)	-1927(9)	836(9)	6141(5)	76(4)
C(24)	-2724(9)	899(9)	6325(5)	89(5)
C(25)	-3011(8)	423(10)	6655(5)	87(5)
C(26)	-2498(8)	-137(9)	6791(5)	79(4)
C(27)	-1690(7)	-252(7)	6625(4)	66(3)
C(28)	-1160(9)	-921(9)	6776(6)	99(5)
C(29)	-1505(12)	-1631(9)	6540(8)	156(9)
C(30)	-1155(12)	-1029(14)	7286(7)	176(11)
C(31)	-3917(11)	480(13)	6872(7)	131(7)
C(32)	-4022(13)	1261(13)	7075(9)	198(13)
C(33)	-4538(12)	115(18)	6592(10)	252(17)
C(34)	-1686(9)	1355(11)	5744(6)	97(5)
C(35)	-1803(15)	2211(11)	5842(7)	164(9)
C(36)	-2119(12)	1127(10)	5309(6)	134(7)
C(37)	-960(30)	3230(20)	7435(17)	83(8)
C(38)	-1140(30)	2900(30)	7865(13)	80(8)
C(39)	-1500(30)	2170(30)	7751(17)	81(8)
C(40)	-1520(30)	2090(30)	7290(19)	82(8)
C(41)	-1200(30)	2730(30)	7089(16)	90(9)

C(37')	-1030(30)	3180(30)	7659(18)	87(8)
C(38')	-1340(30)	2490(30)	7895(14)	84(8)
C(39')	-1560(30)	1960(30)	7564(18)	91(8)
C(40')	-1420(40)	2240(40)	7143(15)	88(9)
C(41')	-1070(30)	2980(30)	7193(17)	83(8)
O(1)	0	5000	5000	150
C(42)	-738	4632	5124	160
C(43)	-1389	4442	5386	160
C(42')	-466	4513	4738	160
C(43')	-1241	4307	4901	160

Table 3. Bond lengths [A] and angles [deg] for compound **1**.

Hf(1)-C(41')#1	2.41(5)
Hf(1)-C(37)#1	2.44(4)
Hf(1)-C(37')#1	2.48(4)
Hf(1)-C(40)#1	2.49(5)
Hf(1)-C(38)#1	2.47(3)
Hf(1)-C(38')#1	2.49(4)
Hf(1)-C(40')#1	2.49(6)
Hf(1)-C(39)#1	2.49(5)
Hf(1)-C(41)#1	2.48(5)
Hf(1)-C(39')#1	2.51(5)
Hf(1)-Ga(1)	2.6199(13)
Hf(1)-Ga(1)#1	2.6199(13)
Ga(1)-C(1)	2.022(10)
C(1)-C(2)	1.396(14)
C(1)-C(6)	1.408(14)
C(2)-C(3)	1.358(15)
C(2)-C(22)	1.527(15)
C(3)-C(4)	1.426(16)
C(4)-C(5)	1.372(15)
C(5)-C(6)	1.401(14)
C(6)-C(7)	1.500(15)
C(7)-C(12)	1.374(16)
C(7)-C(8)	1.394(16)

C(8)-C(9)	1.418(17)
C(8)-C(19)	1.504(18)
C(9)-C(10)	1.336(18)
C(10)-C(11)	1.364(18)
C(10)-C(16)	1.555(18)
C(11)-C(12)	1.400(16)
C(12)-C(13)	1.490(17)
C(13)-C(14)	1.528(17)
C(13)-C(15)	1.524(17)
C(16)-C(18)	1.37(2)
C(16)-C(17)	1.40(2)
C(19)-C(21)	1.53(2)
C(19)-C(20)	1.510(19)
C(22)-C(27)	1.379(16)
C(22)-C(23)	1.393(17)
C(23)-C(24)	1.380(18)
C(23)-C(34)	1.53(2)
C(24)-C(25)	1.358(19)
C(25)-C(26)	1.339(19)
C(25)-C(31)	1.577(19)
C(26)-C(27)	1.387(17)
C(27)-C(28)	1.512(18)
C(28)-C(30)	1.51(2)
C(28)-C(29)	1.53(2)
C(31)-C(33)	1.44(3)
C(31)-C(32)	1.50(3)
C(34)-C(36)	1.51(2)

C(34)-C(35)	1.54(2)
C(37)-C(41)	1.40(5)
C(37)-C(38)	1.42(3)
C(38)-C(39)	1.44(5)
C(39)-C(40)	1.36(5)
C(40)-C(41)	1.37(6)
C(37')-C(41')	1.41(4)
C(37')-C(38')	1.48(5)
C(38')-C(39')	1.39(5)
C(39')-C(40')	1.35(5)
C(40')-C(41')	1.41(6)
O(1)-C(42')#2	1.36788(18)
O(1)-C(42')	1.36789(17)
O(1)-C(42)#2	1.3869(2)
O(1)-C(42)	1.3869(2)
C(42)-C(43)	1.3313(2)
C(42')-C(43')	1.3691(2)

C(41')#1-Hf(1)-C(37)#1	20.3(11)
C(41')#1-Hf(1)-C(37')#1	33.6(10)
C(37)#1-Hf(1)-C(37')#1	15.7(9)
C(41')#1-Hf(1)-C(40)#1	41.8(15)
C(37)#1-Hf(1)-C(40)#1	54.0(17)
C(37')#1-Hf(1)-C(40)#1	56.1(17)
C(41')#1-Hf(1)-C(38)#1	47.9(14)
C(37)#1-Hf(1)-C(38)#1	33.6(9)
C(37')#1-Hf(1)-C(38)#1	18.5(10)

C(40)#1-Hf(1)-C(38)#1	55.0(15)
C(41')#1-Hf(1)-C(38')#1	55.3(14)
C(37)#1-Hf(1)-C(38')#1	47.1(12)
C(37')#1-Hf(1)-C(38')#1	34.7(12)
C(40)#1-Hf(1)-C(38')#1	45.6(15)
C(38)#1-Hf(1)-C(38')#1	18.4(11)
C(41')#1-Hf(1)-C(40')#1	33.5(13)
C(37)#1-Hf(1)-C(40')#1	49.6(15)
C(37')#1-Hf(1)-C(40')#1	55.6(17)
C(40)#1-Hf(1)-C(40')#1	12.4(12)
C(38)#1-Hf(1)-C(40')#1	59.3(17)
C(38')#1-Hf(1)-C(40')#1	53.8(15)
C(41')#1-Hf(1)-C(39)#1	55.2(17)
C(37)#1-Hf(1)-C(39)#1	54.2(16)
C(37')#1-Hf(1)-C(39)#1	45.9(14)
C(40)#1-Hf(1)-C(39)#1	31.7(12)
C(38)#1-Hf(1)-C(39)#1	33.7(11)
C(38')#1-Hf(1)-C(39)#1	17.1(11)
C(40')#1-Hf(1)-C(39)#1	42.3(14)
C(41')#1-Hf(1)-C(41)#1	13.3(12)
C(37)#1-Hf(1)-C(41)#1	33.0(11)
C(37')#1-Hf(1)-C(41)#1	44.3(14)
C(40)#1-Hf(1)-C(41)#1	32.1(13)
C(38)#1-Hf(1)-C(41)#1	55.4(14)
C(38')#1-Hf(1)-C(41)#1	58.2(17)
C(40')#1-Hf(1)-C(41)#1	21.7(14)
C(39)#1-Hf(1)-C(41)#1	53.3(16)

C(41')#1-Hf(1)-C(39')#1	54.0(16)
C(37)#1-Hf(1)-C(39')#1	59.5(15)
C(37')#1-Hf(1)-C(39')#1	55.3(15)
C(40)#1-Hf(1)-C(39')#1	19.2(11)
C(38)#1-Hf(1)-C(39')#1	47.0(12)
C(38')#1-Hf(1)-C(39')#1	32.2(11)
C(40')#1-Hf(1)-C(39')#1	31.4(11)
C(39)#1-Hf(1)-C(39')#1	15.4(10)
C(41)#1-Hf(1)-C(39')#1	47.9(14)
C(41')#1-Hf(1)-Ga(1)	130.9(11)
C(37)#1-Hf(1)-Ga(1)	122.7(10)
C(37')#1-Hf(1)-Ga(1)	107.6(13)
C(40)#1-Hf(1)-Ga(1)	97.8(11)
C(38)#1-Hf(1)-Ga(1)	89.2(12)
C(38')#1-Hf(1)-Ga(1)	76.7(11)
C(40')#1-Hf(1)-Ga(1)	110.0(12)
C(39)#1-Hf(1)-Ga(1)	75.9(11)
C(41)#1-Hf(1)-Ga(1)	128.1(14)
C(39')#1-Hf(1)-Ga(1)	80.3(12)
C(41')#1-Hf(1)-Ga(1)#1	96.4(12)
C(37)#1-Hf(1)-Ga(1)#1	116.6(12)
C(37')#1-Hf(1)-Ga(1)#1	128.5(11)
C(40)#1-Hf(1)-Ga(1)#1	78.5(13)
C(38)#1-Hf(1)-Ga(1)#1	133.4(10)
C(38')#1-Hf(1)-Ga(1)#1	121.8(13)
C(40')#1-Hf(1)-Ga(1)#1	74.7(13)
C(39)#1-Hf(1)-Ga(1)#1	104.9(12)

C(41)#1-Hf(1)-Ga(1)#1	84.4(11)
C(39')#1-Hf(1)-Ga(1)#1	89.7(11)
Ga(1)-Hf(1)-Ga(1)#1	100.76(6)
C(1)-Ga(1)-Hf(1)	171.6(3)
C(2)-C(1)-C(6)	117.9(9)
C(2)-C(1)-Ga(1)	120.4(8)
C(6)-C(1)-Ga(1)	121.4(8)
C(3)-C(2)-C(1)	122.5(10)
C(3)-C(2)-C(22)	118.0(10)
C(1)-C(2)-C(22)	119.5(10)
C(2)-C(3)-C(4)	119.7(11)
C(5)-C(4)-C(3)	118.7(11)
C(6)-C(5)-C(4)	121.4(11)
C(5)-C(6)-C(1)	119.7(10)
C(5)-C(6)-C(7)	117.0(10)
C(1)-C(6)-C(7)	123.1(10)
C(12)-C(7)-C(8)	120.8(11)
C(12)-C(7)-C(6)	120.9(11)
C(8)-C(7)-C(6)	118.2(11)
C(7)-C(8)-C(9)	117.1(13)
C(7)-C(8)-C(19)	122.6(12)
C(9)-C(8)-C(19)	120.0(13)
C(10)-C(9)-C(8)	123.7(13)
C(9)-C(10)-C(11)	116.9(12)
C(9)-C(10)-C(16)	120.4(16)
C(11)-C(10)-C(16)	122.8(15)
C(10)-C(11)-C(12)	123.7(13)

C(7)-C(12)-C(11)	117.7(12)
C(7)-C(12)-C(13)	123.1(12)
C(11)-C(12)-C(13)	119.2(13)
C(12)-C(13)-C(14)	113.1(12)
C(12)-C(13)-C(15)	115.1(12)
C(14)-C(13)-C(15)	105.8(12)
C(18)-C(16)-C(17)	125.6(18)
C(18)-C(16)-C(10)	114.2(15)
C(17)-C(16)-C(10)	112.9(14)
C(8)-C(19)-C(21)	111.2(14)
C(8)-C(19)-C(20)	112.5(13)
C(21)-C(19)-C(20)	110.6(14)
C(27)-C(22)-C(23)	120.9(12)
C(27)-C(22)-C(2)	120.5(10)
C(23)-C(22)-C(2)	118.4(11)
C(24)-C(23)-C(22)	118.0(14)
C(24)-C(23)-C(34)	118.8(14)
C(22)-C(23)-C(34)	123.0(13)
C(23)-C(24)-C(25)	122.6(14)
C(26)-C(25)-C(24)	117.5(13)
C(26)-C(25)-C(31)	118.7(16)
C(24)-C(25)-C(31)	123.8(16)
C(25)-C(26)-C(27)	124.4(14)
C(22)-C(27)-C(26)	116.7(12)
C(22)-C(27)-C(28)	121.6(11)
C(26)-C(27)-C(28)	121.7(13)
C(27)-C(28)-C(30)	113.1(14)

C(27)-C(28)-C(29)	107.4(14)
C(30)-C(28)-C(29)	110.5(16)
C(33)-C(31)-C(32)	124(2)
C(33)-C(31)-C(25)	111.4(18)
C(32)-C(31)-C(25)	108.6(17)
C(36)-C(34)-C(23)	112.0(15)
C(36)-C(34)-C(35)	111.3(15)
C(23)-C(34)-C(35)	114.1(15)
C(41)-C(37)-C(38)	110(4)
C(37)-C(38)-C(39)	104(4)
C(40)-C(39)-C(38)	110(4)
C(39)-C(40)-C(41)	109(5)
C(40)-C(41)-C(37)	108(5)
C(41')-C(37')-C(38')	104(4)
C(39')-C(38')-C(37')	108(4)
C(40')-C(39')-C(38')	111(4)
C(39')-C(40')-C(41')	108(4)
C(37')-C(41')-C(40')	110(4)
C(42')#2-O(1)-C(42')	180.0
C(42')#2-O(1)-C(42)#2	53.099(10)
C(42')-O(1)-C(42)#2	126.901(10)
C(42')#2-O(1)-C(42)	126.901(10)
C(42')-O(1)-C(42)	53.099(10)
C(42)#2-O(1)-C(42)	180.0
C(43)-C(42)-O(1)	157.623(4)
O(1)-C(42')-C(43')	117.031(11)

```
Symmetry transformations used to generate equivalent atoms:

#1 -x,y,-z+3/2    #2 -x,-y+1,-z+1
```

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for Compound **1**.

The anisotropic displacement factor exponent takes the form:

-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
Hf(1)	67(1)	46(1)	44(1)	0	6(1)	0
Ga(1)	70(1)	66(1)	66(1)	-7(1)	5(1)	-1(1)
C(1)	57(8)	42(5)	50(6)	-10(5)	-3(6)	4(6)
C(2)	50(7)	54(7)	52(7)	-1(6)	4(6)	-1(6)
C(3)	57(8)	70(9)	75(9)	-5(7)	-7(7)	-19(7)
C(4)	58(7)	65(8)	70(8)	-13(7)	13(6)	2(6)
C(5)	41(6)	70(8)	65(8)	-4(7)	13(6)	-2(6)
C(6)	50(7)	57(7)	51(7)	-7(6)	-8(6)	11(6)
C(7)	46(7)	57(8)	60(8)	-6(7)	11(6)	-12(6)
C(8)	56(8)	73(9)	79(9)	2(8)	-1(7)	-21(7)
C(9)	94(11)	60(9)	86(10)	-5(7)	28(9)	-22(8)
C(10)	50(8)	88(11)	87(10)	-30(9)	7(7)	-22(8)
C(11)	57(8)	92(11)	71(9)	-18(8)	0(7)	-1(8)
C(12)	57(8)	59(8)	70(9)	-12(7)	9(7)	-14(7)
C(13)	54(8)	82(9)	76(9)	-2(8)	4(7)	1(7)
C(14)	91(11)	85(11)	153(16)	-11(11)	-2(11)	13(9)
C(15)	149(16)	102(12)	101(13)	21(11)	-19(12)	6(12)

C(16)	92(13)	147(17)	138(17)	-62(14)	25(12)	-59(12)
C(17)	121(16)	220(20)	170(20)	-39(18)	61(15)	-119(17)
C(18)	136(17)	230(30)	190(20)	-120(20)	28(16)	-111(17)
C(19)	87(10)	65(9)	99(11)	22(8)	-13(9)	-11(8)
C(20)	143(17)	120(16)	190(20)	-22(15)	-75(17)	38(13)
C(21)	168(19)	180(20)	89(13)	13(13)	-35(13)	-43(15)
C(22)	42(6)	55(7)	70(8)	-13(6)	-4(6)	-1(6)
C(23)	60(9)	94(11)	74(10)	-6(9)	-3(8)	-4(9)
C(24)	67(10)	102(12)	98(11)	-3(10)	-3(9)	33(9)
C(25)	39(8)	126(14)	96(12)	-10(11)	17(8)	2(9)
C(26)	51(8)	109(12)	79(10)	-2(9)	8(8)	-14(8)
C(27)	48(7)	69(8)	80(9)	-1(7)	8(7)	-7(6)
C(28)	60(9)	94(12)	142(15)	30(11)	32(9)	4(9)
C(29)	152(18)	74(12)	240(30)	10(15)	43(18)	-14(12)
C(30)	118(16)	270(30)	144(17)	119(19)	17(14)	68(17)
C(31)	81(12)	160(20)	148(18)	7(15)	19(12)	39(13)
C(32)	160(20)	170(20)	260(30)	80(20)	140(20)	79(18)
C(33)	47(11)	400(50)	310(40)	80(30)	-13(18)	-30(20)
C(34)	63(10)	126(15)	102(13)	18(12)	-5(9)	32(10)
C(35)	250(30)	115(18)	129(17)	37(14)	11(18)	12(18)
C(36)	159(18)	151(17)	93(13)	11(12)	23(13)	47(14)
C(37)	103(16)	82(13)	65(19)	-7(15)	14(18)	34(12)
C(38)	95(17)	75(18)	69(13)	-12(14)	34(15)	41(15)
C(39)	71(16)	104(18)	66(17)	-7(16)	13(17)	16(15)
C(40)	57(15)	124(17)	64(18)	6(17)	-4(18)	-7(14)
C(41)	95(18)	110(20)	69(13)	2(14)	-3(15)	2(17)
C(37')	107(17)	84(15)	71(19)	-9(16)	20(20)	42(14)

C(38')	79(17)	100(20)	69(14)	-4(15)	26(14)	35(17)
C(39')	77(15)	121(16)	70(20)	6(15)	9(19)	-3(14)
C(40')	89(18)	106(19)	69(15)	8(16)	-12(17)	-2(16)
C(41')	99(18)	81(18)	68(16)	3(15)	-3(17)	26(15)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for compound **1**.

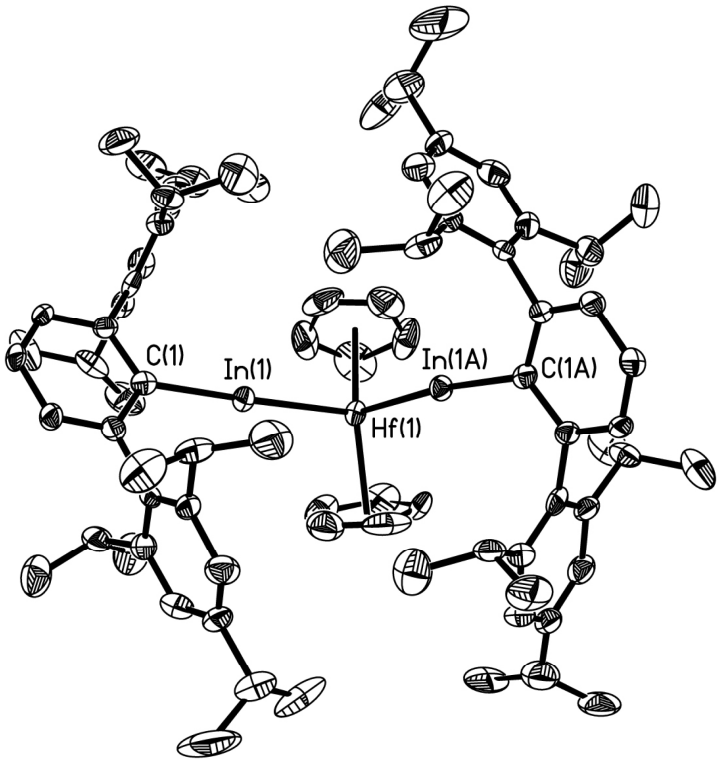
	x	y	z	U(eq)
H(3)	-930	-641	5643	80
H(4)	367	-748	5252	78
H(5)	1469	29	5459	70
H(9)	2282	2786	5836	96
H(11)	3460	1192	6511	88
H(13)	1843	-181	6476	84
H(14A)	3010	-950	6331	164
H(14B)	3548	-232	6203	164
H(14C)	2789	-476	5895	164
H(15A)	2742	-519	7065	176
H(15B)	2350	282	7169	176
H(15C)	3277	220	6989	176
H(16)	3514	3069	6071	151

H(17A)	4172	2294	5633	255
H(17B)	4757	2068	6041	255
H(17C)	4734	2912	5869	255
H(18A)	3305	3010	6783	280
H(18B)	4156	3388	6634	280
H(18C)	4153	2562	6838	280
H(19)	386	1741	5652	100
H(20A)	64	3023	5577	227
H(20B)	977	3244	5731	227
H(20C)	362	2850	6075	227
H(21A)	592	2277	4933	219
H(21B)	1234	1611	5011	219
H(21C)	1531	2459	5055	219
H(24)	-3077	1282	6219	107
H(26)	-2696	-471	7012	95
H(28)	-579	-838	6674	119
H(29A)	-1177	-2066	6627	234
H(29B)	-1472	-1565	6216	234
H(29C)	-2081	-1707	6627	234
H(30A)	-812	-1461	7362	265
H(30B)	-1720	-1114	7391	265
H(30C)	-930	-581	7429	265
H(31)	-3880	146	7139	158
H(32A)	-3537	1383	7256	297
H(32B)	-4516	1269	7264	297
H(32C)	-4081	1629	6836	297
H(33A)	-4349	-387	6513	378

H(33B)	-4621	408	6320	378
H(33C)	-5060	80	6756	378
H(34)	-1082	1279	5693	116
H(35A)	-1499	2345	6113	246
H(35B)	-2390	2319	5884	246
H(35C)	-1591	2502	5591	246
H(36A)	-1938	1456	5067	201
H(36B)	-2717	1170	5346	201
H(36C)	-1976	610	5236	201
H(37)	-726	3712	7390	100
H(38)	-1044	3105	8152	96
H(39)	-1691	1815	7960	97
H(40)	-1711	1657	7137	98
H(41)	-1158	2821	6778	108
H(37')	-856	3638	7787	105
H(38')	-1379	2421	8208	100
H(39')	-1781	1480	7624	109
H(40')	-1525	1997	6869	106
H(41')	-895	3290	6954	99
H(42A)	-1061	4767	4856	192
H(42B)	-548	4118	5060	192
H(43A)	-1736	4079	5229	240
H(43B)	-1714	4889	5454	240
H(43C)	-1186	4221	5664	240
H(42C)	-139	4053	4692	192
H(42D)	-545	4747	4442	192
H(43D)	-1507	3967	4689	240

H(43E)	-1583	4753	4938	240
H(43F)	-1176	4057	5189	240

Data Collection Compound 2: $Cp_2Hf(InR)_2$



Molecular structure of compound 2

Experimental

Data Collection

Black-blue crystals were mounted and sealed in a glass capillary under nitrogen. The X-ray intensity data were measured at room temperature on a Bruker SMART APEX II X-ray diffractometer system with graphite-monochromated Mo Kα radiation (λ = 0.71073 Å) ω-scan technique. The data were collected in a hemisphere of data in 1664 frames with 10 second exposure times. Crystallographic data: $C_{86}H_{118}OIn_2Hf$: a = 15.748(4) Å, b = 17.855(5) Å, c = 29.708(8) Å, α= 90°, β = 90°, γ = 90°, V = 8354(4) Å^3, Z = 4, F.W. = 1575.94, μ = 1.827 mm-1, d = 1.253 g/cm^3, F(000) = 3248.

Data Reduction [1]

Of the 7353 unique reflections collected, 2891 were observed ($I > 2\ \sigma(I)$). The linear absorption coefficient for Mo Kα radiation is 1.827 mm^{-1}. The data were corrected for

Lorentz and polarization effects and integrated with the manufacturer's SAINT software. Absorption corrections were applied with the SADABS.

Structure Solution and Refinement [2]

Subsequent solution and refinement was performed using the SHELXTL 6.1[3] solution package operating on a Pentium computer. The structure was solved by direct methods using the SHELXTL 6.1 Software Package. Non-hydrogen atomic scattering factors were taken from the literature tabulations.[4] The heavy atom positions were determined using direct methods employing the SHELXTL routine methods. The remaining non-hydrogen atoms were located from successive difference Fourier map calculations. The Cp and discrete solvent ether molecule (in half) are found disordered in adjacent positions in two sets labeled as C(37), C(38), C(39), C(40), C(41) (one set) and C(37'), C(38'), C(39'), C(40'), C(41') (another set) for the Cp group, and as C(42), C(43) (one set), and C(42'), C(43') (another set) for solvent ether, respectively. Each of these two sets is divided using the PART command and the corresponding hydrogen atoms are added in the correct calculated positions. The occupancies of all atoms for both disordered Cp and half a solvent ether molecule have been set at 0.50 (requested by the referee). In the final cycles of each refinement, the non-hydrogen atoms were refined anisotropically except for the half a discrete disordered solvent ether molecule in which the positions and temperature factors of atoms are constrained (see explanation below*). Hydrogen atom positions were calculated and allowed to ride on the carbon to which they are bonded assuming a C–H bond length of 0.95 Å. Hydrogen atom temperature factors were fixed at 1.10 times the isotropic temperature factor of the C-atom to which they are bonded. The crystalline compound is orthorhombic, space group Pbcn (No. 60) and the final residual values based on 431 variable parameters and 2891 observed reflections ($I > 2\,\sigma(I)$) are $R1 = 0.0653$, $wR2 = 0.1452$, and those for all unique reflections are $R1 = 0.1882$, $wR2 = 0.2080$. The goodness-of-fit indicator for all data is 1.010. Peaks on the final difference map ranged from 0.771 to -2.770e/Å^3, which are of no chemical significance.

Summary

The compound crystallizes in orthorhombic, space group Pbcn (No. 60). The asymmetric unit contains half a molecule in the form of $C_{41}H_{54}InHf_{0.5}\cdot 0.5O(C_2H_5)_2$ with the Hf atom located at a special position with a two-fold axis symmetry. The whole molecule is in the form of $C_{82}H_{108}In_2Hf\cdot O(C_2H_5)_2$ in which the Hf atom is coordinated by two Cp and two In atoms bonded with m-terphenyl ligands. Structure solution, refinement and the calculation of derived results were performed using the SHELXTL 6.1[3] package of computer programs. Neutral atom scattering factors were those of Cromer and Waber,[4] and the real and imaginary anomalous dispersion corrections were those of Cromer.[5]

References

1. Data Reduction:

Intensity

$I = [S - B/R]\cdot V$

Standard Deviation in Intensity

$s(I) = [S + B/R^2]^{1/2}\cdot V$

Structure Factor

$F = (I/Lp)^{1/2}$

Standard Deviation in Structure Factor

$\sigma(F) = \sigma(I)/(2\cdot F\cdot Lp)$

Where: S = total scan count
B = sum of background counts
R = ratio of background counting time to scan counting time
V = scan rate
Lp = Lorentz-polarization correction

2. Least-Squares Refinement:

Weighting Scheme

$w = 1/[\sigma^2(F_o) + 0.0954*P)^2 + 34.2000*P]$ where $P = [\mathrm{Max}(F_o^2, 0) + 2*F_c^2]/3$;

Residuals

R-factors:

$R = \Sigma\,||F_o| - |F_c||/\Sigma|F_o|$;

Weighted R-factor on F^2:

$wR2 = [\Sigma[w\,(F_o^2 - F_c^2)^2] / \Sigma[w(F_o^2)^2]]^{1/2}$;

Goodness of Fit Indicator:

goodness-of-fit $= [\Sigma[w\,(F_o^2 - F_c^2)^2] /(N_{observns} - N_{params})\,]^{1/2}$.

3. Sheldrick, G.M. *SHELXTL 6.1*, *Crystallographic Computing System*; Siemens Analytical X-Ray Instruments: Madison, WI, 2000.

4. Cromer, D.T. and Waber, J.T. International Tables for X-ray Crystallography, Vol. IV, Table 2.2B, The Kynoch Press, Birmingham England, 1974.

5. Cromer, D.T. International Tables for X-ray Crystallography, Vol. IV, Table 2.3.1, The Kynoch Press, Birmingham England, 1974.

*The remaining electron densities of the half a discrete solvent ether molecule in the difference Fourier map are barely invisible due to reflection intensities from it being very weak since diffraction data were taken at ambient temperature. Attempts to establish a disordered model for half an ether molecule with fully anisotropic refinement have been made unsuccessfully, resulting in either explosion of model or unbearably high displacement parameters (far beyond reasonable values) even with appropriate restraints. In order to make chemical sense for solvent ether molecule, the disordered model has to be constrained and refined only isotropically.

Table 1. Crystal data and structure refinement for compound **2**.

Identification code	compound **2**
Empirical formula	C86 H118 Hf In2 O
Formula weight	1575.94
Temperature	273(2) K
Wavelength	0.71073 A
Crystal system, space group	Orthorhombic, Pbcn
Unit cell dimensions	a = 15.748(4) A alpha = 90 deg. b = 17.855(5) A beta = 90 deg. c = 29.708(8) A gamma = 90 deg.
Volume	8354(4) A^3
Z, Calculated density	4, 1.253 Mg/m^3
Absorption coefficient	1.827 mm^-1
F(000)	3248

Crystal size	0.07 x 0.07 x 0.06 mm
Theta range for data collection	2.20 to 25.00 deg.
Limiting indices	-18<=h<=18, -21<=k<=21, -35<=l<=35
Reflections collected / unique	64198 / 7353 [R(int) = 0.2256]
Completeness to theta = 25.00	99.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.8983 and 0.8828
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	7353 / 150 / 431
Goodness-of-fit on F^2	1.010
Final R indices [I>2sigma(I)]	R1 = 0.0653, wR2 = 0.1452
R indices (all data)	R1 = 0.1882, wR2 = 0.2080
Largest diff. peak and hole	0.771 and -2.770 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for compound **2**.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Hf(1)	0	2298(1)	7500	46(1)
In(1)	24(1)	1254(1)	6812(1)	49(1)
C(1)	140(9)	561(7)	6206(4)	45(3)
C(2)	-537(9)	115(8)	6056(4)	51(4)
C(3)	-450(9)	-361(8)	5707(4)	52(4)
C(4)	320(9)	-397(8)	5476(5)	51(4)
C(5)	992(8)	50(8)	5590(4)	52(4)
C(6)	907(8)	533(8)	5951(4)	48(3)
C(7)	1635(10)	1055(9)	6060(5)	50(4)
C(8)	1608(10)	1783(10)	5889(5)	61(5)
C(9)	2321(11)	2244(9)	5965(5)	72(5)
C(10)	2999(11)	2002(11)	6201(5)	71(5)
C(11)	3015(10)	1300(10)	6363(5)	68(5)
C(12)	2342(11)	798(11)	6307(5)	65(5)
C(13)	2397(9)	11(9)	6476(6)	65(4)
C(14)	3026(10)	-457(10)	6220(6)	108(7)

C(15)	2616(14)	-38(11)	6968(6)	116(8)
C(16)	3743(17)	2566(19)	6284(8)	128(9)
C(17)	4243(19)	2667(18)	5922(9)	240(20)
C(18)	3775(15)	2879(14)	6691(9)	172(12)
C(19)	882(10)	2072(9)	5608(6)	70(5)
C(20)	580(13)	2841(10)	5742(7)	127(8)
C(21)	1144(14)	2042(11)	5113(6)	123(8)
C(22)	-1410(8)	234(9)	6286(4)	49(4)
C(23)	-1919(11)	840(10)	6142(6)	62(5)
C(24)	-2696(11)	924(11)	6354(6)	79(6)
C(25)	-2972(10)	433(11)	6691(6)	72(5)
C(26)	-2465(11)	-134(11)	6798(6)	82(6)
C(27)	-1677(9)	-261(9)	6601(5)	57(4)
C(28)	-1158(10)	-942(10)	6737(6)	78(5)
C(29)	-1527(13)	-1660(10)	6505(7)	122(8)
C(30)	-1129(14)	-1083(12)	7248(7)	132(9)
C(31)	-3830(13)	532(16)	6920(8)	132(9)
C(32)	-3891(14)	1318(13)	7138(8)	157(11)
C(33)	-4509(13)	236(19)	6640(12)	236(18)
C(34)	-1674(11)	1333(12)	5747(6)	80(6)
C(35)	-1743(15)	2157(12)	5862(7)	124(9)
C(36)	-2168(14)	1137(11)	5340(6)	118(8)
C(37)	-940(30)	3370(16)	7474(17)	70(7)
C(38)	-1140(30)	3010(30)	7878(12)	76(8)
C(39)	-1520(40)	2300(30)	7791(15)	75(9)
C(40)	-1560(40)	2240(20)	7320(15)	76(8)
C(41)	-1260(40)	2880(30)	7121(10)	78(8)

C(37')	-1030(30)	3282(19)	7670(15)	80(9)
C(38')	-1300(30)	2640(30)	7909(10)	75(9)
C(39')	-1570(30)	2090(20)	7606(15)	72(8)
C(40')	-1450(40)	2380(30)	7171(13)	80(9)
C(41')	-1080(40)	3070(30)	7198(12)	85(9)
O(1)	0	5000	5000	150
C(42)	-738	4632	5124	160
C(43)	-1389	4442	5386	160
C(42')	-466	4513	4738	160
C(43')	-1241	4307	4901	160

Table 3. Bond lengths [A] and angles [deg] for compound **2**.

Hf(1)-C(41')#1	2.37(6)
Hf(1)-C(37)#1	2.42(4)
Hf(1)-C(40')#1	2.49(6)
Hf(1)-C(38')#1	2.46(5)
Hf(1)-C(38)#1	2.47(4)
Hf(1)-C(39')#1	2.52(5)
Hf(1)-C(37')#1	2.45(5)
Hf(1)-C(40)#1	2.51(5)
Hf(1)-C(39)#1	2.54(5)
Hf(1)-C(41)#1	2.51(6)
Hf(1)-In(1)	2.7665(10)
Hf(1)-In(1)#1	2.7665(10)
In(1)-C(1)	2.192(13)
C(1)-C(2)	1.403(17)
C(1)-C(6)	1.428(17)
C(2)-C(3)	1.348(18)
C(2)-C(22)	1.549(17)
C(3)-C(4)	1.394(17)
C(4)-C(5)	1.367(17)
C(5)-C(6)	1.382(17)
C(6)-C(7)	1.515(19)
C(7)-C(8)	1.40(2)
C(7)-C(12)	1.41(2)

C(8)-C(9)	1.41(2)
C(8)-C(19)	1.51(2)
C(9)-C(10)	1.35(2)
C(10)-C(11)	1.34(2)
C(10)-C(16)	1.56(3)
C(11)-C(12)	1.40(2)
C(12)-C(13)	1.50(2)
C(13)-C(14)	1.50(2)
C(13)-C(15)	1.51(2)
C(16)-C(17)	1.34(3)
C(16)-C(18)	1.33(3)
C(19)-C(20)	1.51(2)
C(19)-C(21)	1.53(2)
C(22)-C(27)	1.355(18)
C(22)-C(23)	1.414(19)
C(23)-C(24)	1.38(2)
C(23)-C(34)	1.52(2)
C(24)-C(25)	1.40(2)
C(25)-C(26)	1.33(2)
C(25)-C(31)	1.52(2)
C(26)-C(27)	1.39(2)
C(27)-C(28)	1.52(2)
C(28)-C(30)	1.54(2)
C(28)-C(29)	1.57(2)
C(31)-C(33)	1.46(3)
C(31)-C(32)	1.55(3)
C(34)-C(36)	1.48(2)

C(34)-C(35)	1.51(2)
C(37)-C(38)	1.40(3)
C(37)-C(41)	1.46(3)
C(38)-C(39)	1.41(3)
C(39)-C(40)	1.40(3)
C(40)-C(41)	1.37(3)
C(37')-C(38')	1.40(3)
C(37')-C(41')	1.45(3)
C(38')-C(39')	1.41(3)
C(39')-C(40')	1.40(3)
C(40')-C(41')	1.38(3)
O(1)-C(42')#2	1.3786(2)
O(1)-C(42')	1.3786(2)
O(1)-C(42)#2	1.3850(3)
O(1)-C(42)	1.3850(3)
C(42)-C(43)	1.3312(2)
C(42')-C(43')	1.3636(3)

C(41')#1-Hf(1)-C(37)#1	24.1(13)
C(41')#1-Hf(1)-C(40')#1	32.9(9)
C(37)#1-Hf(1)-C(40')#1	51.9(14)
C(41')#1-Hf(1)-C(38')#1	56.2(12)
C(37)#1-Hf(1)-C(38')#1	46.3(12)
C(40')#1-Hf(1)-C(38')#1	54.3(13)
C(41')#1-Hf(1)-C(38)#1	49.4(12)
C(37)#1-Hf(1)-C(38)#1	33.3(8)
C(40')#1-Hf(1)-C(38)#1	58.7(15)

C(38')#1-Hf(1)-C(38)#1	16.2(10)
C(41')#1-Hf(1)-C(39')#1	55.3(12)
C(37)#1-Hf(1)-C(39')#1	61.6(13)
C(40')#1-Hf(1)-C(39')#1	32.5(9)
C(38')#1-Hf(1)-C(39')#1	32.9(9)
C(38)#1-Hf(1)-C(39')#1	46.0(13)
C(41')#1-Hf(1)-C(37')#1	35.0(9)
C(37)#1-Hf(1)-C(37')#1	14.7(10)
C(40')#1-Hf(1)-C(37')#1	55.3(12)
C(38')#1-Hf(1)-C(37')#1	33.3(9)
C(38)#1-Hf(1)-C(37')#1	18.9(10)
C(39')#1-Hf(1)-C(37')#1	55.2(11)
C(41')#1-Hf(1)-C(40)#1	40.8(15)
C(37)#1-Hf(1)-C(40)#1	55.3(11)
C(40')#1-Hf(1)-C(40)#1	12.2(10)
C(38')#1-Hf(1)-C(40)#1	45.9(13)
C(38)#1-Hf(1)-C(40)#1	53.6(12)
C(39')#1-Hf(1)-C(40)#1	20.4(10)
C(37')#1-Hf(1)-C(40)#1	54.8(15)
C(41')#1-Hf(1)-C(39)#1	56.8(15)
C(37)#1-Hf(1)-C(39)#1	55.6(11)
C(40')#1-Hf(1)-C(39)#1	43.1(13)
C(38')#1-Hf(1)-C(39)#1	18.0(11)
C(38)#1-Hf(1)-C(39)#1	32.7(9)
C(39')#1-Hf(1)-C(39)#1	15.3(10)
C(37')#1-Hf(1)-C(39)#1	45.7(13)
C(40)#1-Hf(1)-C(39)#1	32.3(9)

C(41')#1-Hf(1)-C(41)#1	11.3(13)
C(37)#1-Hf(1)-C(41)#1	34.3(9)
C(40')#1-Hf(1)-C(41)#1	22.2(13)
C(38')#1-Hf(1)-C(41)#1	57.3(13)
C(38)#1-Hf(1)-C(41)#1	54.2(11)
C(39')#1-Hf(1)-C(41)#1	48.9(12)
C(37')#1-Hf(1)-C(41)#1	42.9(16)
C(40)#1-Hf(1)-C(41)#1	31.7(9)
C(39)#1-Hf(1)-C(41)#1	53.7(12)
C(41')#1-Hf(1)-In(1)	131.6(10)
C(37)#1-Hf(1)-In(1)	123.3(11)
C(40')#1-Hf(1)-In(1)	108.4(11)
C(38')#1-Hf(1)-In(1)	78.0(10)
C(38)#1-Hf(1)-In(1)	89.9(12)
C(39')#1-Hf(1)-In(1)	78.1(9)
C(37')#1-Hf(1)-In(1)	108.8(10)
C(40)#1-Hf(1)-In(1)	96.7(10)
C(39)#1-Hf(1)-In(1)	74.8(10)
C(41)#1-Hf(1)-In(1)	126.9(9)
C(41')#1-Hf(1)-In(1)#1	97.2(12)
C(37)#1-Hf(1)-In(1)#1	121.2(11)
C(40')#1-Hf(1)-In(1)#1	76.1(10)
C(38')#1-Hf(1)-In(1)#1	123.1(11)
C(38)#1-Hf(1)-In(1)#1	133.7(10)
C(39')#1-Hf(1)-In(1)#1	90.3(10)
C(37')#1-Hf(1)-In(1)#1	130.2(10)
C(40)#1-Hf(1)-In(1)#1	80.1(10)

C(39)#1-Hf(1)-In(1)#1	105.4(12)
C(41)#1-Hf(1)-In(1)#1	87.7(11)
In(1)-Hf(1)-In(1)#1	95.27(4)
C(1)-In(1)-Hf(1)	171.2(3)
C(2)-C(1)-C(6)	117.0(12)
C(2)-C(1)-In(1)	121.2(10)
C(6)-C(1)-In(1)	121.8(10)
C(3)-C(2)-C(1)	121.7(13)
C(3)-C(2)-C(22)	121.0(13)
C(1)-C(2)-C(22)	117.1(12)
C(2)-C(3)-C(4)	119.7(13)
C(5)-C(4)-C(3)	121.7(12)
C(4)-C(5)-C(6)	118.7(13)
C(5)-C(6)-C(1)	121.1(13)
C(5)-C(6)-C(7)	118.6(12)
C(1)-C(6)-C(7)	120.3(12)
C(8)-C(7)-C(12)	121.2(16)
C(8)-C(7)-C(6)	118.1(14)
C(12)-C(7)-C(6)	120.7(15)
C(7)-C(8)-C(9)	117.3(16)
C(7)-C(8)-C(19)	123.0(14)
C(9)-C(8)-C(19)	119.6(16)
C(10)-C(9)-C(8)	121.8(16)
C(11)-C(10)-C(9)	120.0(16)
C(11)-C(10)-C(16)	122(2)
C(9)-C(10)-C(16)	118(2)
C(10)-C(11)-C(12)	122.8(17)

C(11)-C(12)-C(7)	116.9(17)
C(11)-C(12)-C(13)	121.2(16)
C(7)-C(12)-C(13)	121.8(16)
C(14)-C(13)-C(12)	113.0(15)
C(14)-C(13)-C(15)	108.0(15)
C(12)-C(13)-C(15)	113.2(15)
C(17)-C(16)-C(18)	130(2)
C(17)-C(16)-C(10)	114(2)
C(18)-C(16)-C(10)	116(2)
C(8)-C(19)-C(20)	113.9(15)
C(8)-C(19)-C(21)	108.4(15)
C(20)-C(19)-C(21)	111.8(15)
C(27)-C(22)-C(23)	122.2(14)
C(27)-C(22)-C(2)	119.4(14)
C(23)-C(22)-C(2)	118.4(13)
C(24)-C(23)-C(22)	116.5(16)
C(24)-C(23)-C(34)	121.0(16)
C(22)-C(23)-C(34)	122.3(16)
C(23)-C(24)-C(25)	122.2(17)
C(26)-C(25)-C(24)	117.5(16)
C(26)-C(25)-C(31)	120.9(18)
C(24)-C(25)-C(31)	121.5(19)
C(25)-C(26)-C(27)	124.0(18)
C(22)-C(27)-C(26)	117.5(16)
C(22)-C(27)-C(28)	122.6(14)
C(26)-C(27)-C(28)	119.8(15)
C(27)-C(28)-C(30)	114.2(15)

C(27)-C(28)-C(29)	109.7(14)
C(30)-C(28)-C(29)	108.2(16)
C(33)-C(31)-C(25)	111(2)
C(33)-C(31)-C(32)	122(2)
C(25)-C(31)-C(32)	110(2)
C(36)-C(34)-C(23)	111.1(16)
C(36)-C(34)-C(35)	112.2(17)
C(23)-C(34)-C(35)	111.8(16)
C(38)-C(37)-C(41)	105(2)
C(39)-C(38)-C(37)	111(3)
C(38)-C(39)-C(40)	106(3)
C(39)-C(40)-C(41)	110(3)
C(40)-C(41)-C(37)	108(2)
C(38')-C(37')-C(41')	105(2)
C(39')-C(38')-C(37')	110(3)
C(40')-C(39')-C(38')	107(3)
C(39')-C(40')-C(41')	110(3)
C(40')-C(41')-C(37')	108(3)
C(42')#2-O(1)-C(42')	180.0
C(42')#2-O(1)-C(42)#2	53.432(12)
C(42')-O(1)-C(42)#2	126.568(12)
C(42')#2-O(1)-C(42)	126.568(12)
C(42')-O(1)-C(42)	53.432(12)
C(42)#2-O(1)-C(42)	180.0
C(43)-C(42)-O(1)	157.326(5)
C(43')-C(42')-O(1)	116.493(12)

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Symmetry transformations used to generate equivalent atoms:

#1 -x,y,-z+3/2    #2 -x,-y+1,-z+1
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Table 4. Anisotropic displacement parameters (A^2 x 10^3) for compound **2**.

The anisotropic displacement factor exponent takes the form:

-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
Hf(1)	56(1)	43(1)	40(1)	0	7(1)	0
In(1)	53(1)	49(1)	44(1)	-6(1)	3(1)	1(1)
C(1)	46(10)	41(6)	47(7)	13(5)	1(7)	0(7)
C(2)	42(9)	66(10)	44(8)	5(8)	1(7)	-11(8)
C(3)	51(9)	58(10)	46(9)	-16(8)	1(8)	-11(8)
C(4)	59(9)	52(9)	43(8)	-24(7)	3(7)	-1(7)
C(5)	35(8)	76(11)	44(8)	-15(8)	9(6)	0(8)
C(6)	41(8)	55(9)	48(8)	5(7)	2(7)	5(7)
C(7)	57(10)	56(10)	37(9)	-12(8)	20(8)	-2(8)
C(8)	55(10)	72(12)	55(10)	-17(9)	14(8)	-25(10)
C(9)	76(13)	73(12)	67(11)	6(9)	12(10)	-20(11)
C(10)	58(11)	93(14)	61(11)	-9(10)	13(9)	-22(10)
C(11)	38(10)	90(14)	75(11)	-4(11)	1(9)	-5(10)
C(12)	42(10)	89(14)	66(11)	0(10)	2(9)	5(11)
C(13)	37(9)	74(12)	85(12)	13(10)	-5(9)	-10(8)
C(14)	72(13)	101(15)	151(19)	-17(14)	20(13)	28(12)

C(15)	140(20)	100(17)	108(17)	28(14)	-2(15)	20(15)
C(16)	120(20)	160(20)	99(18)	-20(17)	3(16)	-73(19)
C(17)	220(30)	370(50)	130(20)	-10(30)	20(20)	-230(40)
C(18)	130(20)	180(30)	210(30)	-60(20)	-40(20)	-86(19)
C(19)	61(11)	60(10)	89(12)	36(9)	-31(10)	-29(8)
C(20)	128(19)	91(17)	160(20)	8(15)	-58(17)	25(14)
C(21)	160(20)	118(18)	90(15)	2(13)	-52(15)	-28(16)
C(22)	36(8)	75(10)	36(7)	-8(7)	-3(6)	-12(8)
C(23)	55(12)	76(13)	56(11)	7(10)	-4(9)	2(10)
C(24)	60(13)	88(16)	88(13)	6(12)	-18(11)	6(11)
C(25)	46(10)	103(15)	68(11)	9(10)	17(9)	3(10)
C(26)	63(12)	99(16)	85(14)	13(12)	4(11)	-9(11)
C(27)	43(9)	78(11)	48(8)	-6(8)	6(7)	-10(8)
C(28)	44(10)	79(12)	113(15)	16(11)	17(10)	2(9)
C(29)	140(20)	68(14)	160(20)	7(14)	32(17)	9(14)
C(30)	122(19)	160(20)	111(16)	60(16)	4(15)	34(17)
C(31)	68(14)	190(30)	140(20)	-5(19)	34(15)	13(17)
C(32)	120(20)	140(20)	210(30)	30(20)	110(20)	46(17)
C(33)	40(13)	340(50)	330(40)	50(40)	30(20)	-10(20)
C(34)	59(11)	119(17)	63(12)	20(12)	-9(9)	23(12)
C(35)	170(30)	95(19)	111(17)	11(15)	17(17)	-15(17)
C(36)	160(20)	121(18)	69(13)	13(12)	11(14)	25(16)
C(37)	96(17)	53(12)	61(18)	6(15)	20(19)	49(11)
C(38)	91(19)	69(19)	68(12)	6(15)	30(17)	35(17)
C(39)	70(18)	86(18)	70(16)	9(17)	21(18)	20(16)
C(40)	66(17)	94(16)	68(17)	20(16)	10(20)	9(14)
C(41)	80(20)	84(19)	69(11)	20(14)	0(16)	17(17)

C(37')	101(18)	70(16)	70(19)	8(15)	40(20)	36(15)
C(38')	72(19)	80(20)	70(12)	9(14)	31(15)	23(18)
C(39')	52(14)	90(16)	70(20)	20(14)	9(19)	18(14)
C(40')	80(19)	87(18)	73(14)	18(15)	11(18)	15(16)
C(41')	100(20)	80(18)	70(15)	15(15)	14(18)	18(16)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for compound **2**.

	x	y	z	U(eq)
H(3)	-901	-665	5620	62
H(4)	378	-732	5239	61
H(5)	1497	29	5428	62
H(9)	2323	2728	5849	86
H(11)	3494	1141	6518	81
H(13)	1837	-218	6435	79
H(14A)	3020	-959	6335	162
H(14B)	3584	-248	6254	162
H(14C)	2875	-462	5907	162
H(15A)	2212	246	7139	174
H(15B)	3175	160	7016	174
H(15C)	2600	-552	7062	174

H(16)	4108	2156	6387	500(500)
H(17A)	4059	2343	5683	360
H(17B)	4820	2552	6000	360
H(17C)	4207	3179	5825	360
H(18A)	3329	2677	6875	258
H(18B)	3703	3411	6664	258
H(18C)	4314	2774	6827	258
H(19)	404	1727	5647	84
H(20A)	119	2990	5550	190
H(20B)	1038	3192	5712	190
H(20C)	390	2832	6049	190
H(21A)	685	2218	4929	184
H(21B)	1279	1535	5033	184
H(21C)	1633	2354	5067	184
H(24)	-3046	1319	6270	94
H(26)	-2648	-466	7019	99
H(28)	-574	-870	6631	94
H(29A)	-1200	-2089	6593	183
H(29B)	-1499	-1602	6184	183
H(29C)	-2107	-1728	6594	183
H(30A)	-794	-1521	7308	199
H(30B)	-1696	-1158	7358	199
H(30C)	-880	-658	7395	199
H(31)	-3808	188	7177	158
H(32A)	-3394	1408	7317	235
H(32B)	-4387	1342	7325	235
H(32C)	-3930	1691	6906	235

H(33A)	-5044	303	6790	354
H(33B)	-4415	-288	6587	354
H(33C)	-4517	498	6357	354
H(34)	-1076	1232	5680	96
H(35A)	-1402	2263	6122	187
H(35B)	-2324	2279	5924	187
H(35C)	-1545	2450	5612	187
H(36A)	-1995	1452	5095	177
H(36B)	-2762	1210	5398	177
H(36C)	-2067	622	5264	177
H(37)	-655	3824	7440	84
H(38)	-1047	3201	8164	91
H(39)	-1697	1951	8001	90
H(40)	-1759	1823	7165	91
H(41)	-1266	2988	6814	94
H(37')	-867	3740	7789	96
H(38')	-1292	2596	8221	90
H(39')	-1788	1618	7679	86
H(40')	-1597	2134	6905	96
H(41')	-892	3362	6957	101
H(42A)	-1068	4761	4859	192
H(42B)	-542	4128	5061	192
H(43A)	-1737	4082	5233	240
H(43B)	-1720	4879	5453	240
H(43C)	-1179	4228	5661	240
H(42C)	-549	4742	4445	192
H(42D)	-132	4063	4693	192

H(43D)	-1508	3970	4693	240
H(43E)	-1589	4744	4938	240
H(43F)	-1171	4063	5186	240