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Dichloridobis(η^5-methylcyclopentadienyl)hafnium(IV)

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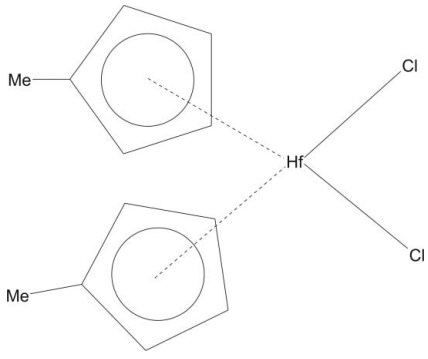
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.006 Å; R factor = 0.026; wR factor = 0.064; data-to-parameter ratio = 15.3.

The title compound, $[HfCl_2(C_6H_7)_2]$, has the Hf atom in a distorted pseudo-tetrahedral geometry. The molecule lies about a mirror plane.

Related literature

For dichloridobis(η^5-cyclopentadienyl)hafnium(IV), see Soloveichik *et al.* (1988), and for dichloridobis(η^5-ethylcyclopentadienyl)hafnium(IV), see Dong *et al.* (1982). For synthesis, see: Lappert *et al.* (1981).



Experimental

Crystal data

$[HfCl_2(C_6H_7)_2]$
M_r = 407.62
Orthorhombic, *Pnma*
a = 12.1368 (5) Å
b = 15.4218 (6) Å
c = 6.5656 (4) Å
V = 1228.89 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 8.89 mm^{-1}
T = 120 (2) K
0.16 × 0.14 × 0.1 mm

Data collection

Oxford Diffraction KM-4 CCD diffractometer
Absorption correction: analytical (*CrysAlis RED*; Oxford Diffraction, 2005)
T_{min} = 0.36, T_{max} = 0.469
5507 measured reflections
1132 independent reflections
1096 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.064
S = 1.11
1132 reflections
74 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 2.01 e Å^{-3}
$\Delta\rho_{min}$ = −0.66 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Hf1—Cl2	2.4286 (13)	Hf1—Cl1	2.4381 (11)
Cl2—Hf1—Cl1	92.97 (4)		

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2005); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2005); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2285).

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supplementary materials

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Dichloridobis(η^5-methylcyclopentadienyl)hafnium(IV)

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Comment

The discrete molecule has the Hf atom in a pseudotetrahedral geometry. The bond dimensions involving the metal atom are similar to those in [(η^5-C_5H_5)$_2$$HfCl_2$] (Soloveichik *et al.*, 1988) and [(η^5-$C_2H_5C_5H_4$)$_2$$HfCl_2$] (Dong *et al.*, 1982). The molecule lies about a mirror plane defined by the Hf—Cl1—Cl2 atoms.

Experimental

The compound was been obtained as a white powder in the reaction of ($CH_3C_5H_4$)Li with $HfCl_4$ in THF (Lappert *et al.*, 1981). Slow crystallization from THF at 203 K yielded colourless crystals.

Refinement

All H atoms were refined as riding on C atoms with aromatic C—H = 0.95 Å, methyl C—H = 0.98 Å, and U_{iso}(H) = 1.2U_{eq}(C) for CH groups, 1.5U_{eq}(C) for CH_3 groups. The final difference Fourier map had a large peak at about 1 Å from Hf1.

Figures

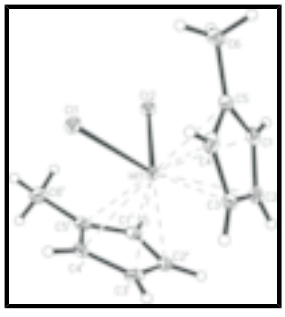

Fig. 1. The molecular structure of (I), with atom labels and 50% probability displacement ellipsoids for non-H atoms. Symmetry code i: *x*, 1/2 − *y*, *z*.

Dichloridobis(η^5-methylcyclopentadienyl)hafnium(IV)

Crystal data

$[HfCl_2(C_6H_7)_2]$	F_{000} = 768
M_r = 407.62	D_x = 2.203 Mg m^{-3}
Orthorhombic, *Pnma*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2n	Cell parameters from 7628 reflections

a = 12.1368 (5) Å	θ = 2.1–32.3º
b = 15.4218 (6) Å	μ = 8.89 mm^{-1}
c = 6.5656 (4) Å	T = 120 (2) K
V = 1228.89 (10) Å^3	Prism, colourless
Z = 4	0.16 × 0.14 × 0.1 mm

Data collection

Oxford Diffraction KM-4 CCD diffractometer	1132 independent reflections
Monochromator: graphite	1096 reflections with $I > 2\sigma(I)$
Detector resolution: 8.1883 pixels mm^{-1}	R_{int} = 0.028
T = 120(2) K	θ_{max} = 25.0º
0.75° ω scans	θ_{min} = 2.6º
Absorption correction: analytical (CrysAlis RED; Oxford Diffraction, 2005)	$h = -14 \rightarrow 14$
T_{min} = 0.36, T_{max} = 0.469	$k = -10 \rightarrow 18$
5507 measured reflections	$l = -7 \rightarrow 7$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.026	H-atom parameters constrained
$wR(F^2)$ = 0.064	$w = 1/[\sigma^2(F_o^2) + (0.051P)^2 + 0.3835P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.11	$(\Delta/\sigma)_{max}$ = 0.001
1132 reflections	$\Delta\rho_{max}$ = 2.01 e Å^{-3}
74 parameters	$\Delta\rho_{min}$ = −0.66 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}

Hf1	0.080494 (13)	0.25	0.90179 (3)	0.01576 (14)
Cl1	0.27234 (9)	0.25	1.01196 (18)	0.0244 (3)
Cl2	0.12988 (9)	0.25	0.5433 (2)	0.0231 (3)
C1	−0.0143 (3)	0.1133 (2)	0.7926 (5)	0.0254 (7)
H1	−0.0367	0.1071	0.6547	0.031*
C2	−0.0770 (2)	0.1505 (3)	0.9496 (8)	0.0265 (9)
H2	−0.1494	0.1732	0.9367	0.032*
C3	−0.0140 (3)	0.1482 (2)	1.1292 (6)	0.0266 (8)
H3	−0.0358	0.1699	1.2585	0.032*
C4	0.0869 (3)	0.1082 (4)	1.0834 (7)	0.0270 (12)
H4	0.1448	0.0974	1.1775	0.032*
C5	0.0882 (3)	0.0865 (3)	0.8746 (8)	0.0277 (11)
C6	0.1786 (3)	0.0414 (3)	0.7601 (6)	0.0409 (11)
H6A	0.2498	0.0554	0.8222	0.061*
H6B	0.1782	0.0606	0.6178	0.061*
H6C	0.1668	−0.0215	0.7655	0.061*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Hf1	0.01493 (18)	0.01262 (19)	0.01974 (19)	0	−0.00026 (5)	0
Cl1	0.0182 (5)	0.0260 (6)	0.0289 (7)	0	−0.0023 (5)	0
Cl2	0.0238 (6)	0.0233 (6)	0.0223 (5)	0	0.0005 (5)	0
C1	0.0314 (16)	0.0187 (17)	0.0262 (18)	−0.0100 (13)	−0.0001 (14)	0.0005 (14)
C2	0.019 (2)	0.017 (2)	0.043 (2)	−0.0055 (11)	0.0028 (14)	0.003 (2)
C3	0.0326 (19)	0.0187 (17)	0.0285 (18)	−0.0076 (15)	0.0119 (15)	−0.0009 (14)
C4	0.023 (2)	0.020 (3)	0.038 (3)	−0.0034 (13)	−0.0037 (12)	0.0115 (17)
C5	0.029 (2)	0.012 (2)	0.042 (3)	−0.0026 (13)	0.0113 (14)	0.0025 (17)
C6	0.042 (2)	0.016 (2)	0.065 (3)	0.0017 (14)	0.023 (2)	0.0012 (14)

Geometric parameters (Å, °)

Hf1—Cl2	2.4286 (13)	C2—C3	1.406 (7)
Hf1—Cl1	2.4381 (11)	C2—H2	0.95
Hf1—C3^{i}	2.451 (4)	C3—C4	1.404 (6)
Hf1—C2^{i}	2.472 (4)	C3—H3	0.95
Hf1—C4^{i}	2.492 (5)	C4—C5	1.411 (6)
Hf1—C1^{i}	2.507 (3)	C4—H4	0.95
Hf1—C5^{i}	2.529 (5)	C5—C6	1.502 (6)
C1—C2	1.404 (6)	C6—H6A	0.98
C1—C5	1.416 (5)	C6—H6B	0.98
C1—H1	0.95	C6—H6C	0.98
Cl2—Hf1—Cl1	92.97 (4)	C2—C1—C5	108.4 (4)
Cl2—Hf1—C3^{i}	134.90 (9)	C2—C1—H1	125.8
Cl1—Hf1—C3^{i}	105.43 (10)	C5—C1—H1	125.8
Cl2—Hf1—C2^{i}	108.30 (13)	C1—C2—C3	108.1 (3)
Cl1—Hf1—C2^{i}	134.50 (11)	C1—C2—H2	125.9

$C3^{i}$—Hf1—$C2^{i}$	33.18 (16)	C3—C2—H2	125.9
Cl2—Hf1—$C4^{i}$	117.13 (12)	C4—C3—C2	107.8 (4)
Cl1—Hf1—$C4^{i}$	80.11 (8)	C4—C3—H3	126.1
$C3^{i}$—Hf1—$C4^{i}$	32.99 (13)	C2—C3—H3	126.1
$C2^{i}$—Hf1—$C4^{i}$	54.43 (13)	C3—C4—C5	108.8 (3)
Cl2—Hf1—$C1^{i}$	80.56 (8)	C3—C4—H4	125.6
Cl1—Hf1—$C1^{i}$	121.53 (8)	C5—C4—H4	125.6
$C3^{i}$—Hf1—$C1^{i}$	54.62 (11)	C4—C5—C1	106.9 (4)
$C2^{i}$—Hf1—$C1^{i}$	32.76 (14)	C4—C5—C6	127.2 (4)
$C4^{i}$—Hf1—$C1^{i}$	54.05 (13)	C1—C5—C6	125.9 (4)
Cl2—Hf1—$C5^{i}$	85.55 (11)	C5—C6—H6A	109.5
Cl1—Hf1—$C5^{i}$	89.19 (9)	C5—C6—H6B	109.5
$C3^{i}$—Hf1—$C5^{i}$	54.69 (14)	H6A—C6—H6B	109.5
$C2^{i}$—Hf1—$C5^{i}$	54.44 (13)	C5—C6—H6C	109.5
$C4^{i}$—Hf1—$C5^{i}$	32.63 (15)	H6A—C6—H6C	109.5
$C1^{i}$—Hf1—$C5^{i}$	32.66 (12)	H6B—C6—H6C	109.5

Symmetry codes: (i) x, $-y+1/2$, z.

Fig. 1