Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Chloridobis(η^5-cyclopentadienyl)-(4-methoxyphenethyl)zirconium(IV)

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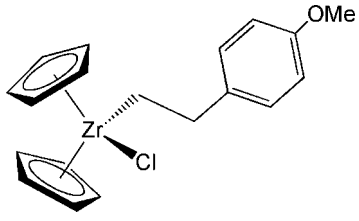
Received 14 October 2007; accepted 16 October 2007

Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; disorder in main residue; R factor = 0.022; wR factor = 0.059; data-to-parameter ratio = 15.4.

In the structure of the title compound, $[Zr(C_5H_5)_2(C_9H_{11}O)Cl]$, the Zr atom is in a pseudo-tetrahedral coordination environment and is coordinated by two η^5-cyclopentadienyl (Cp) rings, a chloride ion and a 4-methoxyphenethyl group. The 4-methoxyphenethyl ligand is disordered over two positions; the site occupancy factors are *ca* 0.6 and 0.4.

Related literature

For related structures, see: Chirik *et al.* (1999); Grimmond *et al.* (2000); Lappert *et al.* (1997).



Experimental

Crystal data

$[Zr(C_5H_5)_2(C_9H_{11}O)Cl]$
M_r = 392.03
Monoclinic, $P2_1/n$
a = 6.8095 (7) Å
b = 12.3823 (12) Å
c = 20.645 (2) Å
β = 93.198 (1)°
V = 1738.0 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.79 mm^{-1}
T = 173 (2) K
0.04 × 0.03 × 0.03 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.879, T_{max} = 0.977
21010 measured reflections
4215 independent reflections
3945 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.059
S = 1.06
4215 reflections
273 parameters
32 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

We are grateful to NSERC (Canada), the Canada Research Chairs Programme (Canada), CFI (Canada), FQRNT (Québec), Mount-Allison University and Université Laval for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2378).

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  doi:10.1107/S1600536807050775

supplementary materials

Acta Cryst. (2007). E**63**, m2790 [**doi:10.1107/S1600536807050775**]

Chloridobis(η^5-cyclopentadienyl)(4-methoxyphenethyl)zirconium(IV)

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Comment

In the complex $Cp_2ZrCl(CH_2CH_2\text{-}4\text{-}C_6H_4OMe)$ (**1**), the observed Zr—Cl bond length of 2.4508 (5) Å is consistent with the lengths observed in previously reported structures of $Cp_2ZrCl(R)$ complexes (average 2.445 Å) (Chirik *et al.*, 1999; Lappert *et al.*, 1997; Grimmond *et al.*, 2000). The Zr—Csp^3 distance was found to be 2.2816 (15) Å, which compares to the distance observed for the analogue $Cp_2ZrCl(cyclo\text{-}C_5H_9)$ (2.276 (5) Å) (Chirik *et al.*, 1999) and the average C—C distance in the six-membered ring is 1.386 Å. The methoxy group is bent out of the plane formed by the adjacent aromatic carbons by 8.0 (6)° with C—O distances of 1.370 (5) Å and 1.430 (8) Å to C17 and C19, respectively. The 4-methoxyphenethyl ligand, was disordered over two positions, Fig. 1. A slight rotation of 7.7° around C12 shifts the position of the aryl moiety in the second disorder component, with the methoxy group rotated by 179.6°.

Experimental

A 1 ml toluene solution of 4-vinyl anisole (158 mg; 1.18 mmol) was added to a 1 ml toluene suspension of Cp_2ZrHCl (302 mg, 1.17 mmol) at room temperature. The resulting solution was stirred for 30 min while it quickly turned from cloudy white to red. The reaction mixture was filtered to afford a clear dark red solution which was cooled to −30°C and filtered again. The solvent was removed *in vacuo* and a $CDCl_3$ solution afforded red-orange crystals of quality suitable for X-ray determination (147 mg; 32% yield).

Refinement

All H-atoms were placed at idealized positions and refined using a riding model with d(C—H) = 0.95 Å, U_{iso}=1.2U_{eq} (C) for aromatic 0.99 Å, U_{iso} = 1.2U_{eq} (C) for CH_2, 0.98 Å, U_{iso} = 1.5U_{eq} (C) for CH_3 atoms. The 4-methoxyphenethyl ligand was disordered over two positions with occupancies that refined to 0.56:0.44. In the final refinement the occupancies were fixed at 0.50.

Figures

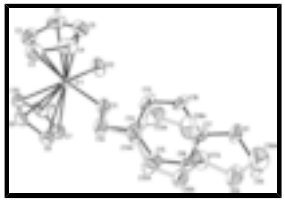


Fig. 1. The molecular structure of **1** showing the numbering scheme adopted. Anisotropic atomic displacement ellipsoids for the non-hydrogen atoms are shown at the 50% probability level. Hydrogen atoms were removed for clarity. Bonds to atoms in the two disorder components of the 4-methoxyphenethyl ligand are drawn as full lines and open double lines respectively.

Chloridobis(η^5-cyclopentadienyl)(4-methoxyphenethyl)zirconium(IV)

Crystal data

$[Zr(C_5H_5)_2(C_9H_{11}O)Cl]$	F_{000} = 800
M_r = 392.03	D_x = 1.498 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 7049 reflections
a = 6.8095 (7) Å	θ = 2.6–28.1°
b = 12.3823 (12) Å	μ = 0.79 mm^{-1}
c = 20.645 (2) Å	T = 173 (2) K
β = 93.198 (1)°	Block, orange
V = 1738.0 (3) Å^3	0.04 × 0.03 × 0.03 mm
Z = 4	

Data collection

Bruker SMART APEXII CCD diffractometer	4215 independent reflections
Radiation source: fine-focus sealed tube	3945 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.024
T = 173(2) K	θ_{max} = 28.2°
ω scans	θ_{min} = 1.9°
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)	h = −8→8
T_{min} = 0.879, T_{max} = 0.977	k = −16→16
21010 measured reflections	l = −27→27

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.022	H-atom parameters constrained
$wR(F^2)$ = 0.059	$w = 1/[\sigma^2(F_o^2) + (0.027P)^2 + 0.7941P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.06	$(\Delta/\sigma)_{max}$ = 0.001
4215 reflections	$\Delta\rho_{max}$ = 0.43 e Å^{-3}
273 parameters	$\Delta\rho_{min}$ = −0.34 e Å^{-3}
32 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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}	Occ. (<1)
Zr1	0.468663 (19)	0.587021 (11)	0.190854 (6)	0.02628 (5)	
Cl2	0.70826 (7)	0.47764 (4)	0.25495 (2)	0.05049 (12)	
C1	0.3455 (3)	0.40828 (14)	0.14762 (10)	0.0497 (5)	
H1	0.3594	0.3464	0.1746	0.060*	
C2	0.1864 (3)	0.47838 (17)	0.14413 (9)	0.0481 (4)	
H2	0.0729	0.4729	0.1687	0.058*	
C3	0.2207 (3)	0.55795 (17)	0.09870 (9)	0.0483 (4)	
H3	0.1350	0.6161	0.0868	0.058*	
C4	0.4032 (3)	0.53761 (16)	0.07354 (8)	0.0489 (5)	
H4	0.4630	0.5792	0.0414	0.059*	
C5	0.4828 (3)	0.44500 (16)	0.10407 (10)	0.0498 (4)	
H5	0.6064	0.4130	0.0967	0.060*	
C6	0.3718 (3)	0.64765 (16)	0.30112 (9)	0.0490 (4)	
H6	0.3965	0.6011	0.3373	0.059*	
C7	0.4989 (3)	0.72725 (16)	0.27985 (9)	0.0445 (4)	
H7	0.6258	0.7437	0.2987	0.053*	
C8	0.4069 (3)	0.77817 (14)	0.22615 (9)	0.0411 (4)	
H8	0.4596	0.8359	0.2023	0.049*	
C9	0.2244 (3)	0.72982 (15)	0.21356 (9)	0.0422 (4)	
H9	0.1314	0.7488	0.1795	0.051*	
C10	0.2010 (3)	0.64864 (16)	0.25954 (10)	0.0475 (4)	
H10	0.0902	0.6026	0.2622	0.057*	
C11	0.7008 (2)	0.68241 (14)	0.13947 (8)	0.0381 (4)	
H11A	0.7863	0.7152	0.1745	0.046*	
H11B	0.7824	0.6276	0.1187	0.046*	
C12	0.6661 (3)	0.77161 (15)	0.08869 (10)	0.0462 (4)	
H12A	0.5509	0.7527	0.0595	0.055*	
H12B	0.6370	0.8403	0.1107	0.055*	
C13	0.8621 (6)	0.7152 (3)	−0.00156 (19)	0.0355 (9)	0.50
H13	0.7675	0.6596	−0.0094	0.043*	0.50
C14	0.8408 (19)	0.7859 (12)	0.0500 (6)	0.036 (3)	0.50
C15	0.9837 (9)	0.8657 (5)	0.0579 (3)	0.0372 (12)	0.50
H15	0.9716	0.9159	0.0923	0.045*	0.50

C16	1.1410 (6)	0.8770 (3)	0.0198 (2)	0.0313 (8)	0.50
H16	1.2356	0.9325	0.0278	0.038*	0.50
C17	1.1571 (6)	0.8043 (4)	−0.0311 (2)	0.0254 (8)	0.50
C18	1.0171 (5)	0.7236 (3)	−0.04151 (15)	0.0328 (7)	0.50
H18	1.0281	0.6741	−0.0763	0.039*	0.50
O1	1.3041 (3)	0.8057 (2)	−0.07376 (12)	0.0411 (5)	0.50
C19	1.4382 (13)	0.8946 (7)	−0.0718 (4)	0.0430 (15)	0.50
H19A	1.3641	0.9625	−0.0746	0.064*	0.50
H19B	1.5231	0.8896	−0.1085	0.064*	0.50
H19C	1.5192	0.8927	−0.0311	0.064*	0.50
C13A	0.9084 (7)	0.7584 (4)	−0.0056 (2)	0.0495 (11)	0.50
H13A	0.8288	0.7049	−0.0271	0.059*	0.50
C14A	0.8482 (18)	0.8024 (13)	0.0515 (6)	0.0327 (18)	0.50
C15A	0.9705 (8)	0.8793 (5)	0.0809 (3)	0.0402 (12)	0.50
H15A	0.9313	0.9122	0.1197	0.048*	0.50
C16A	1.1438 (6)	0.9102 (3)	0.0572 (2)	0.0478 (9)	0.50
H16A	1.2274	0.9600	0.0803	0.057*	0.50
C17A	1.1958 (7)	0.8678 (4)	−0.0010 (2)	0.0414 (11)	0.50
C18A	1.0815 (8)	0.7900 (5)	−0.0326 (3)	0.0488 (16)	0.50
H18A	1.1203	0.7587	−0.0719	0.059*	0.50
O1A	1.3740 (4)	0.9094 (2)	−0.02203 (14)	0.0538 (7)	0.50
C19A	1.4326 (19)	0.8715 (9)	−0.0808 (5)	0.061 (3)	0.50
H19D	1.4171	0.7928	−0.0825	0.091*	0.50
H19E	1.5710	0.8901	−0.0855	0.091*	0.50
H19F	1.3514	0.9045	−0.1162	0.091*	0.50

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zr1	0.02377 (8)	0.02842 (8)	0.02714 (8)	0.00189 (5)	0.00585 (5)	0.00335 (5)
Cl2	0.0438 (2)	0.0560 (3)	0.0516 (3)	0.0183 (2)	0.00319 (19)	0.0185 (2)
C1	0.0717 (14)	0.0336 (9)	0.0441 (10)	−0.0136 (8)	0.0070 (9)	−0.0004 (7)
C2	0.0427 (9)	0.0601 (12)	0.0420 (9)	−0.0165 (9)	0.0083 (7)	−0.0152 (8)
C3	0.0528 (11)	0.0543 (11)	0.0364 (9)	0.0034 (9)	−0.0093 (8)	−0.0098 (8)
C4	0.0732 (13)	0.0477 (10)	0.0268 (8)	−0.0117 (9)	0.0131 (8)	−0.0037 (7)
C5	0.0550 (11)	0.0429 (10)	0.0530 (11)	0.0026 (9)	0.0172 (9)	−0.0154 (8)
C6	0.0672 (12)	0.0495 (10)	0.0317 (8)	0.0131 (9)	0.0154 (8)	−0.0012 (7)
C7	0.0359 (8)	0.0531 (10)	0.0440 (9)	0.0009 (8)	−0.0028 (7)	−0.0168 (8)
C8	0.0454 (9)	0.0318 (8)	0.0471 (9)	−0.0001 (7)	0.0108 (7)	−0.0060 (7)
C9	0.0347 (8)	0.0442 (9)	0.0474 (9)	0.0124 (7)	−0.0010 (7)	−0.0114 (8)
C10	0.0386 (9)	0.0468 (10)	0.0596 (11)	−0.0075 (8)	0.0246 (8)	−0.0179 (9)
C11	0.0266 (7)	0.0433 (9)	0.0453 (9)	0.0009 (6)	0.0086 (6)	0.0112 (7)
C12	0.0313 (8)	0.0430 (9)	0.0653 (12)	0.0007 (7)	0.0114 (8)	0.0188 (9)
C13	0.0319 (19)	0.041 (2)	0.0337 (17)	−0.0203 (16)	0.0040 (13)	−0.0003 (17)
C14	0.032 (4)	0.035 (4)	0.038 (4)	−0.007 (3)	−0.007 (3)	0.014 (2)
C15	0.045 (2)	0.032 (2)	0.036 (3)	0.0011 (17)	0.014 (2)	−0.006 (2)
C16	0.034 (2)	0.0268 (16)	0.033 (3)	−0.0097 (15)	0.0089 (16)	−0.0079 (17)
C17	0.026 (2)	0.0254 (16)	0.0252 (16)	−0.0052 (17)	0.0027 (17)	0.0009 (13)

C18	0.0356 (17)	0.0353 (16)	0.0279 (15)	−0.0214 (14)	0.0042 (12)	−0.0070 (12)
O1	0.0364 (12)	0.0470 (13)	0.0417 (12)	−0.0142 (11)	0.0195 (10)	−0.0041 (10)
C19	0.033 (2)	0.048 (3)	0.050 (3)	−0.015 (2)	0.015 (2)	0.010 (3)
C13A	0.048 (3)	0.054 (3)	0.046 (2)	−0.014 (2)	−0.010 (2)	0.006 (2)
C14A	0.029 (4)	0.034 (4)	0.036 (4)	0.003 (2)	0.011 (2)	0.011 (2)
C15A	0.038 (2)	0.038 (3)	0.046 (3)	−0.0049 (17)	0.015 (2)	0.000 (2)
C16A	0.045 (2)	0.051 (2)	0.049 (2)	−0.0112 (17)	0.0115 (18)	−0.0041 (19)
C17A	0.043 (3)	0.046 (2)	0.036 (3)	0.005 (2)	0.0075 (18)	0.007 (2)
C18A	0.046 (3)	0.070 (4)	0.031 (2)	0.006 (3)	0.004 (3)	0.005 (2)
O1A	0.0480 (16)	0.0639 (18)	0.0509 (16)	−0.0081 (13)	0.0136 (13)	0.0004 (13)
C19A	0.060 (4)	0.066 (5)	0.057 (4)	0.001 (3)	0.013 (3)	−0.004 (3)

Geometric parameters (Å, °)

Zr1—C11	2.2816 (15)	C12—C14	1.480 (9)
Zr1—Cl2	2.4508 (5)	C12—C14A	1.542 (8)
Zr1—C9	2.4897 (16)	C12—H12A	0.9900
Zr1—C10	2.4905 (16)	C12—H12B	0.9900
Zr1—C2	2.4958 (18)	C13—C18	1.379 (4)
Zr1—C3	2.4985 (18)	C13—C14	1.392 (6)
Zr1—C1	2.5130 (18)	C13—H13	0.9500
Zr1—C4	2.5138 (17)	C14—C15	1.390 (6)
Zr1—C5	2.5161 (18)	C15—C16	1.371 (5)
Zr1—C8	2.5187 (17)	C15—H15	0.9500
Zr1—C6	2.5197 (17)	C16—C17	1.392 (5)
Zr1—C7	2.5281 (17)	C16—H16	0.9500
C1—C2	1.387 (3)	C17—O1	1.370 (5)
C1—C5	1.408 (3)	C17—C18	1.390 (4)
C1—H1	0.9500	C18—H18	0.9500
C2—C3	1.389 (3)	O1—C19	1.430 (8)
C2—H2	0.9500	C19—H19A	0.9800
C3—C4	1.396 (3)	C19—H19B	0.9800
C3—H3	0.9500	C19—H19C	0.9800
C4—C5	1.403 (3)	C13A—C14A	1.382 (6)
C4—H4	0.9500	C13A—C18A	1.387 (6)
C5—H5	0.9500	C13A—H13A	0.9500
C6—C7	1.398 (3)	C14A—C15A	1.383 (6)
C6—C10	1.407 (3)	C15A—C16A	1.357 (5)
C6—H6	0.9500	C15A—H15A	0.9500
C7—C8	1.394 (3)	C16A—C17A	1.375 (5)
C7—H7	0.9500	C16A—H16A	0.9500
C8—C9	1.390 (3)	C17A—C18A	1.379 (6)
C8—H8	0.9500	C17A—O1A	1.409 (5)
C9—C10	1.398 (3)	C18A—H18A	0.9500
C9—H9	0.9500	O1A—C19A	1.382 (10)
C10—H10	0.9500	C19A—H19D	0.9800
C11—C12	1.532 (2)	C19A—H19E	0.9800
C11—H11A	0.9900	C19A—H19F	0.9800
C11—H11B	0.9900		

C11—Zr1—Cl2	94.56 (4)	C4—C5—H5	126.4
C11—Zr1—C9	101.96 (6)	C1—C5—H5	126.4
Cl2—Zr1—C9	136.25 (4)	Zr1—C5—H5	118.3
C11—Zr1—C10	130.85 (6)	C7—C6—C10	107.89 (17)
Cl2—Zr1—C10	110.37 (5)	C7—C6—Zr1	74.25 (10)
C9—Zr1—C10	32.60 (7)	C10—C6—Zr1	72.56 (10)
C11—Zr1—C2	129.64 (6)	C7—C6—H6	126.1
Cl2—Zr1—C2	112.71 (5)	C10—C6—H6	126.1
C9—Zr1—C2	86.98 (6)	Zr1—C6—H6	119.0
C10—Zr1—C2	79.21 (6)	C8—C7—C6	108.07 (16)
C11—Zr1—C3	100.19 (7)	C8—C7—Zr1	73.60 (10)
Cl2—Zr1—C3	137.12 (5)	C6—C7—Zr1	73.59 (10)
C9—Zr1—C3	79.37 (6)	C8—C7—H7	126.0
C10—Zr1—C3	89.58 (7)	C6—C7—H7	126.0
C2—Zr1—C3	32.30 (7)	Zr1—C7—H7	118.7
C11—Zr1—C1	121.13 (7)	C9—C8—C7	108.14 (16)
Cl2—Zr1—C1	84.49 (5)	C9—C8—Zr1	72.74 (10)
C9—Zr1—C1	118.63 (7)	C7—C8—Zr1	74.34 (10)
C10—Zr1—C1	103.40 (7)	C9—C8—H8	125.9
C2—Zr1—C1	32.15 (7)	C7—C8—H8	125.9
C3—Zr1—C1	53.46 (7)	Zr1—C8—H8	118.9
C11—Zr1—C4	76.60 (7)	C8—C9—C10	108.50 (16)
Cl2—Zr1—C4	117.56 (5)	C8—C9—Zr1	75.03 (10)
C9—Zr1—C4	105.65 (7)	C10—C9—Zr1	73.73 (10)
C10—Zr1—C4	121.81 (7)	C8—C9—H9	125.7
C2—Zr1—C4	53.49 (6)	C10—C9—H9	125.7
C3—Zr1—C4	32.35 (7)	Zr1—C9—H9	117.4
C1—Zr1—C4	53.53 (6)	C9—C10—C6	107.40 (17)
C11—Zr1—C5	88.61 (7)	C9—C10—Zr1	73.67 (10)
Cl2—Zr1—C5	86.99 (5)	C6—C10—Zr1	74.84 (10)
C9—Zr1—C5	133.07 (7)	C9—C10—H10	126.3
C10—Zr1—C5	132.67 (7)	C6—C10—H10	126.3
C2—Zr1—C5	53.65 (6)	Zr1—C10—H10	117.3
C3—Zr1—C5	53.71 (7)	C12—C11—Zr1	127.38 (11)
C1—Zr1—C5	32.52 (7)	C12—C11—H11A	105.5
C4—Zr1—C5	32.39 (7)	Zr1—C11—H11A	105.5
C11—Zr1—C8	77.25 (6)	C12—C11—H11B	105.5
Cl2—Zr1—C8	118.69 (5)	Zr1—C11—H11B	105.5
C9—Zr1—C8	32.23 (6)	H11A—C11—H11B	106.0
C10—Zr1—C8	53.71 (6)	C14—C12—C11	111.0 (8)
C2—Zr1—C8	118.74 (7)	C11—C12—C14A	114.9 (8)
C3—Zr1—C8	103.82 (6)	C14—C12—H12A	109.4
C1—Zr1—C8	150.75 (7)	C11—C12—H12A	109.4
C4—Zr1—C8	118.94 (6)	C14A—C12—H12A	112.8
C5—Zr1—C8	151.21 (7)	C14—C12—H12B	109.4
C11—Zr1—C6	119.27 (7)	C11—C12—H12B	109.4
Cl2—Zr1—C6	82.92 (5)	C14A—C12—H12B	101.7
C9—Zr1—C6	53.63 (6)	H12A—C12—H12B	108.0
C10—Zr1—C6	32.60 (7)	C18—C13—C14	121.7 (5)

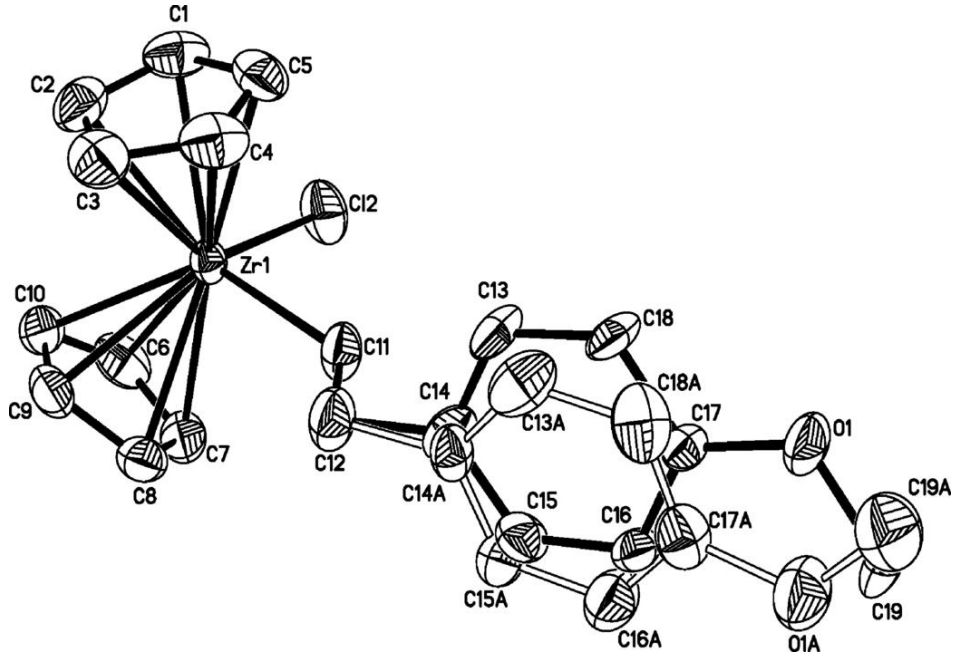
C2—Zr1—C6	105.96 (7)	C18—C13—H13	119.2
C3—Zr1—C6	122.08 (7)	C14—C13—H13	119.2
C1—Zr1—C6	118.97 (7)	C15—C14—C13	115.8 (7)
C4—Zr1—C6	154.39 (7)	C15—C14—C12	126.8 (5)
C5—Zr1—C6	150.92 (7)	C13—C14—C12	117.3 (6)
C8—Zr1—C6	53.29 (6)	C16—C15—C14	124.6 (5)
C11—Zr1—C7	87.24 (7)	C16—C15—H15	117.7
Cl2—Zr1—C7	87.85 (5)	C14—C15—H15	117.7
C9—Zr1—C7	53.38 (6)	C15—C16—C17	117.7 (4)
C10—Zr1—C7	53.72 (6)	C15—C16—H16	121.2
C2—Zr1—C7	132.91 (6)	C17—C16—H16	121.2
C3—Zr1—C7	132.57 (6)	O1—C17—C18	115.4 (4)
C1—Zr1—C7	151.06 (7)	O1—C17—C16	124.7 (4)
C4—Zr1—C7	150.49 (7)	C18—C17—C16	119.9 (4)
C5—Zr1—C7	173.10 (6)	C13—C18—C17	120.2 (3)
C8—Zr1—C7	32.06 (6)	C13—C18—H18	119.9
C6—Zr1—C7	32.17 (7)	C17—C18—H18	119.9
C2—C1—C5	108.02 (17)	C17—O1—C19	118.7 (4)
C2—C1—Zr1	73.24 (10)	C14A—C13A—C18A	122.1 (5)
C5—C1—Zr1	73.86 (10)	C14A—C13A—H13A	119.0
C2—C1—H1	126.0	C18A—C13A—H13A	119.0
C5—C1—H1	126.0	C13A—C14A—C15A	116.5 (6)
Zr1—C1—H1	118.8	C13A—C14A—C12	127.8 (6)
C1—C2—C3	108.57 (17)	C15A—C14A—C12	115.7 (5)
C1—C2—Zr1	74.60 (11)	C16A—C15A—C14A	123.3 (5)
C3—C2—Zr1	73.96 (11)	C16A—C15A—H15A	118.3
C1—C2—H2	125.7	C14A—C15A—H15A	118.3
C3—C2—H2	125.7	C15A—C16A—C17A	118.7 (4)
Zr1—C2—H2	117.7	C15A—C16A—H16A	120.7
C2—C3—C4	108.06 (18)	C17A—C16A—H16A	120.7
C2—C3—Zr1	73.74 (10)	C16A—C17A—C18A	120.9 (4)
C4—C3—Zr1	74.43 (11)	C16A—C17A—O1A	113.4 (4)
C2—C3—H3	126.0	C18A—C17A—O1A	125.7 (4)
C4—C3—H3	126.0	C17A—C18A—C13A	118.5 (5)
Zr1—C3—H3	117.8	C17A—C18A—H18A	120.8
C3—C4—C5	108.05 (17)	C13A—C18A—H18A	120.8
C3—C4—Zr1	73.22 (10)	C19A—O1A—C17A	116.3 (6)
C5—C4—Zr1	73.90 (10)	O1A—C19A—H19D	109.5
C3—C4—H4	126.0	O1A—C19A—H19E	109.5
C5—C4—H4	126.0	H19D—C19A—H19E	109.5
Zr1—C4—H4	118.8	O1A—C19A—H19F	109.5
C4—C5—C1	107.29 (18)	H19D—C19A—H19F	109.5
C4—C5—Zr1	73.71 (10)	H19E—C19A—H19F	109.5
C1—C5—Zr1	73.62 (10)		
C11—Zr1—C1—C2	−116.08 (12)	C11—Zr1—C7—C8	70.15 (11)
Cl2—Zr1—C1—C2	151.90 (11)	Cl2—Zr1—C7—C8	164.83 (10)
C9—Zr1—C1—C2	11.17 (14)	C9—Zr1—C7—C8	−36.96 (11)
C10—Zr1—C1—C2	42.23 (13)	C10—Zr1—C7—C8	−77.80 (12)
C3—Zr1—C1—C2	−37.06 (11)	C2—Zr1—C7—C8	−75.67 (14)

C4—Zr1—C1—C2	−77.61 (13)	C3—Zr1—C7—C8	−31.16 (15)
C5—Zr1—C1—C2	−115.06 (18)	C1—Zr1—C7—C8	−120.63 (16)
C8—Zr1—C1—C2	7.3 (2)	C4—Zr1—C7—C8	13.94 (19)
C6—Zr1—C1—C2	73.14 (13)	C6—Zr1—C7—C8	−115.07 (16)
C7—Zr1—C1—C2	76.52 (18)	C11—Zr1—C7—C6	−174.78 (12)
C11—Zr1—C1—C5	−1.03 (16)	Cl2—Zr1—C7—C6	−80.10 (11)
Cl2—Zr1—C1—C5	−93.04 (13)	C9—Zr1—C7—C6	78.11 (12)
C9—Zr1—C1—C5	126.23 (13)	C10—Zr1—C7—C6	37.27 (12)
C10—Zr1—C1—C5	157.29 (13)	C2—Zr1—C7—C6	39.40 (15)
C2—Zr1—C1—C5	115.06 (18)	C3—Zr1—C7—C6	83.90 (14)
C3—Zr1—C1—C5	78.00 (14)	C1—Zr1—C7—C6	−5.6 (2)
C4—Zr1—C1—C5	37.45 (12)	C4—Zr1—C7—C6	129.01 (15)
C8—Zr1—C1—C5	122.38 (15)	C8—Zr1—C7—C6	115.07 (16)
C6—Zr1—C1—C5	−171.80 (12)	C6—C7—C8—C9	−0.6 (2)
C7—Zr1—C1—C5	−168.42 (13)	Zr1—C7—C8—C9	65.41 (12)
C5—C1—C2—C3	0.5 (2)	C6—C7—C8—Zr1	−66.06 (13)
Zr1—C1—C2—C3	66.74 (13)	C11—Zr1—C8—C9	139.23 (12)
C5—C1—C2—Zr1	−66.22 (14)	Cl2—Zr1—C8—C9	−132.54 (10)
C11—Zr1—C2—C1	86.76 (14)	C10—Zr1—C8—C9	−37.34 (11)
Cl2—Zr1—C2—C1	−30.54 (12)	C2—Zr1—C8—C9	10.78 (13)
C9—Zr1—C2—C1	−170.19 (12)	C3—Zr1—C8—C9	41.70 (12)
C10—Zr1—C2—C1	−138.27 (13)	C1—Zr1—C8—C9	6.34 (19)
C3—Zr1—C2—C1	115.02 (17)	C4—Zr1—C8—C9	72.60 (13)
C4—Zr1—C2—C1	77.77 (12)	C5—Zr1—C8—C9	76.84 (17)
C5—Zr1—C2—C1	37.20 (11)	C6—Zr1—C8—C9	−78.21 (12)
C8—Zr1—C2—C1	−175.93 (11)	C7—Zr1—C8—C9	−115.19 (16)
C6—Zr1—C2—C1	−119.44 (12)	C11—Zr1—C8—C7	−105.58 (11)
C7—Zr1—C2—C1	−140.02 (12)	Cl2—Zr1—C8—C7	−17.35 (12)
C11—Zr1—C2—C3	−28.26 (15)	C9—Zr1—C8—C7	115.19 (16)
Cl2—Zr1—C2—C3	−145.56 (11)	C10—Zr1—C8—C7	77.85 (12)
C9—Zr1—C2—C3	74.79 (12)	C2—Zr1—C8—C7	125.97 (11)
C10—Zr1—C2—C3	106.71 (13)	C3—Zr1—C8—C7	156.89 (11)
C1—Zr1—C2—C3	−115.02 (17)	C1—Zr1—C8—C7	121.53 (14)
C4—Zr1—C2—C3	−37.25 (12)	C4—Zr1—C8—C7	−172.21 (11)
C5—Zr1—C2—C3	−77.82 (13)	C5—Zr1—C8—C7	−167.97 (13)
C8—Zr1—C2—C3	69.05 (13)	C6—Zr1—C8—C7	36.98 (11)
C6—Zr1—C2—C3	125.54 (12)	C7—C8—C9—C10	0.25 (19)
C7—Zr1—C2—C3	104.96 (13)	Zr1—C8—C9—C10	66.72 (12)
C1—C2—C3—C4	−0.1 (2)	C7—C8—C9—Zr1	−66.47 (12)
Zr1—C2—C3—C4	67.09 (13)	C11—Zr1—C9—C8	−40.62 (12)
C1—C2—C3—Zr1	−67.17 (13)	Cl2—Zr1—C9—C8	69.19 (13)
C11—Zr1—C3—C2	158.26 (12)	C10—Zr1—C9—C8	114.85 (16)
Cl2—Zr1—C3—C2	50.06 (14)	C2—Zr1—C9—C8	−170.55 (12)
C9—Zr1—C3—C2	−101.35 (13)	C3—Zr1—C9—C8	−138.91 (12)
C10—Zr1—C3—C2	−70.20 (12)	C1—Zr1—C9—C8	−176.48 (11)
C1—Zr1—C3—C2	36.89 (12)	C4—Zr1—C9—C8	−119.86 (11)
C4—Zr1—C3—C2	114.62 (18)	C5—Zr1—C9—C8	−140.06 (11)
C5—Zr1—C3—C2	77.61 (13)	C6—Zr1—C9—C8	77.07 (12)
C8—Zr1—C3—C2	−122.51 (12)	C7—Zr1—C9—C8	36.76 (11)

C6—Zr1—C3—C2	−67.41 (14)	C11—Zr1—C9—C10	−155.46 (11)
C7—Zr1—C3—C2	−106.08 (13)	Cl2—Zr1—C9—C10	−45.66 (13)
C11—Zr1—C3—C4	43.64 (13)	C2—Zr1—C9—C10	74.61 (12)
Cl2—Zr1—C3—C4	−64.56 (14)	C3—Zr1—C9—C10	106.25 (12)
C9—Zr1—C3—C4	144.04 (13)	C1—Zr1—C9—C10	68.68 (12)
C10—Zr1—C3—C4	175.19 (13)	C4—Zr1—C9—C10	125.29 (11)
C2—Zr1—C3—C4	−114.62 (18)	C5—Zr1—C9—C10	105.09 (13)
C1—Zr1—C3—C4	−77.73 (13)	C8—Zr1—C9—C10	−114.85 (16)
C5—Zr1—C3—C4	−37.01 (12)	C6—Zr1—C9—C10	−37.78 (11)
C8—Zr1—C3—C4	122.88 (12)	C7—Zr1—C9—C10	−78.09 (12)
C6—Zr1—C3—C4	177.97 (11)	C8—C9—C10—C6	0.24 (19)
C7—Zr1—C3—C4	139.31 (12)	Zr1—C9—C10—C6	67.83 (12)
C2—C3—C4—C5	−0.4 (2)	C8—C9—C10—Zr1	−67.58 (12)
Zr1—C3—C4—C5	66.24 (14)	C7—C6—C10—C9	−0.6 (2)
C2—C3—C4—Zr1	−66.64 (13)	Zr1—C6—C10—C9	−67.03 (12)
C11—Zr1—C4—C3	−135.71 (13)	C7—C6—C10—Zr1	66.39 (12)
Cl2—Zr1—C4—C3	136.12 (11)	C11—Zr1—C10—C9	32.48 (15)
C9—Zr1—C4—C3	−36.83 (13)	Cl2—Zr1—C10—C9	148.16 (10)
C10—Zr1—C4—C3	−5.67 (15)	C2—Zr1—C10—C9	−101.45 (12)
C2—Zr1—C4—C3	37.18 (12)	C3—Zr1—C10—C9	−70.67 (12)
C1—Zr1—C4—C3	77.48 (13)	C1—Zr1—C10—C9	−122.80 (11)
C5—Zr1—C4—C3	115.08 (18)	C4—Zr1—C10—C9	−67.64 (12)
C8—Zr1—C4—C3	−68.73 (13)	C5—Zr1—C10—C9	−106.41 (12)
C6—Zr1—C4—C3	−4.0 (2)	C8—Zr1—C10—C9	36.90 (10)
C7—Zr1—C4—C3	−77.13 (17)	C6—Zr1—C10—C9	113.72 (16)
C11—Zr1—C4—C5	109.21 (13)	C7—Zr1—C10—C9	76.96 (12)
Cl2—Zr1—C4—C5	21.04 (13)	C11—Zr1—C10—C6	−81.23 (14)
C9—Zr1—C4—C5	−151.91 (12)	Cl2—Zr1—C10—C6	34.44 (12)
C10—Zr1—C4—C5	−120.75 (13)	C9—Zr1—C10—C6	−113.72 (16)
C2—Zr1—C4—C5	−77.90 (13)	C2—Zr1—C10—C6	144.83 (13)
C3—Zr1—C4—C5	−115.08 (18)	C3—Zr1—C10—C6	175.62 (13)
C1—Zr1—C4—C5	−37.60 (12)	C1—Zr1—C10—C6	123.48 (12)
C8—Zr1—C4—C5	176.19 (11)	C4—Zr1—C10—C6	178.65 (11)
C6—Zr1—C4—C5	−119.06 (17)	C5—Zr1—C10—C6	139.88 (12)
C7—Zr1—C4—C5	167.79 (12)	C8—Zr1—C10—C6	−76.82 (12)
C3—C4—C5—C1	0.7 (2)	C7—Zr1—C10—C6	−36.75 (11)
Zr1—C4—C5—C1	66.51 (14)	Cl2—Zr1—C11—C12	179.89 (16)
C3—C4—C5—Zr1	−65.79 (13)	C9—Zr1—C11—C12	−40.86 (18)
C2—C1—C5—C4	−0.8 (2)	C10—Zr1—C11—C12	−58.06 (19)
Zr1—C1—C5—C4	−66.57 (13)	C2—Zr1—C11—C12	55.21 (19)
C2—C1—C5—Zr1	65.81 (13)	C3—Zr1—C11—C12	40.31 (18)
C11—Zr1—C5—C4	−66.77 (12)	C1—Zr1—C11—C12	93.57 (17)
Cl2—Zr1—C5—C4	−161.41 (12)	C4—Zr1—C11—C12	62.62 (17)
C9—Zr1—C5—C4	38.37 (16)	C5—Zr1—C11—C12	93.02 (17)
C10—Zr1—C5—C4	83.40 (15)	C8—Zr1—C11—C12	−61.71 (17)
C2—Zr1—C5—C4	77.35 (13)	C6—Zr1—C11—C12	−95.68 (17)
C3—Zr1—C5—C4	36.96 (12)	C7—Zr1—C11—C12	−92.50 (17)
C1—Zr1—C5—C4	114.11 (19)	Zr1—C11—C12—C14	−159.3 (3)
C8—Zr1—C5—C4	−6.9 (2)	Zr1—C11—C12—C14A	−166.5 (3)

C6—Zr1—C5—C4	128.99 (15)	C18—C13—C14—C15	1(2)
C11—Zr1—C5—C1	179.12 (13)	C18—C13—C14—C12	178.3 (9)
Cl2—Zr1—C5—C1	84.48 (13)	C11—C12—C14—C15	−100.5 (17)
C9—Zr1—C5—C1	−75.75 (16)	C14A—C12—C14—C15	22 (9)
C10—Zr1—C5—C1	−30.72 (17)	C11—C12—C14—C13	82.1 (14)
C2—Zr1—C5—C1	−36.77 (13)	C14A—C12—C14—C13	−156 (12)
C3—Zr1—C5—C1	−77.15 (14)	C13—C14—C15—C16	−1(2)
C4—Zr1—C5—C1	−114.11 (19)	C12—C14—C15—C16	−178.5 (11)
C8—Zr1—C5—C1	−121.05 (15)	C14—C15—C16—C17	0.9 (14)
C6—Zr1—C5—C1	14.9 (2)	C15—C16—C17—O1	179.3 (5)
C11—Zr1—C6—C7	5.98 (14)	C15—C16—C17—C18	−0.3 (7)
Cl2—Zr1—C6—C7	97.26 (11)	C14—C13—C18—C17	−0.1 (12)
C9—Zr1—C6—C7	−77.26 (12)	O1—C17—C18—C13	−179.7 (4)
C10—Zr1—C6—C7	−115.04 (17)	C16—C17—C18—C13	−0.1 (7)
C2—Zr1—C6—C7	−151.08 (12)	C18—C17—O1—C19	171.6 (6)
C3—Zr1—C6—C7	−120.21 (12)	C16—C17—O1—C19	−8.0 (8)
C1—Zr1—C6—C7	176.93 (11)	C18A—C13A—C14A—C15A	0(2)
C4—Zr1—C6—C7	−117.70 (16)	C18A—C13A—C14A—C12	−176.8 (12)
C5—Zr1—C6—C7	167.85 (13)	C14—C12—C14A—C13A	29 (9)
C8—Zr1—C6—C7	−36.85 (11)	C11—C12—C14A—C13A	90.0 (18)
C11—Zr1—C6—C10	121.02 (12)	C14—C12—C14A—C15A	−147 (12)
Cl2—Zr1—C6—C10	−147.71 (12)	C11—C12—C14A—C15A	−86.3 (15)
C9—Zr1—C6—C10	37.78 (11)	C13A—C14A—C15A—C16A	−2(2)
C2—Zr1—C6—C10	−36.05 (13)	C12—C14A—C15A—C16A	175.2 (9)
C3—Zr1—C6—C10	−5.18 (15)	C14A—C15A—C16A—C17A	4.0 (14)
C1—Zr1—C6—C10	−68.04 (14)	C15A—C16A—C17A—C18A	−4.4 (8)
C4—Zr1—C6—C10	−2.7 (2)	C15A—C16A—C17A—O1A	177.3 (5)
C5—Zr1—C6—C10	−77.11 (18)	C16A—C17A—C18A—C13A	2.5 (8)
C8—Zr1—C6—C10	78.19 (12)	O1A—C17A—C18A—C13A	−179.4 (5)
C7—Zr1—C6—C10	115.04 (17)	C14A—C13A—C18A—C17A	0.0 (14)
C10—C6—C7—C8	0.8 (2)	C16A—C17A—O1A—C19A	−178.9 (7)
Zr1—C6—C7—C8	66.07 (12)	C18A—C17A—O1A—C19A	3.0 (9)
C10—C6—C7—Zr1	−65.27 (12)		

Fig. 1


C1
C5
C2
C4
C3
Cl2
Zr1
C10
C13
C18
C11
C6
C18A
C14
C13A
C17
O1
C9
C7
C12
C19A
C14A
C15
C16
C17A
C8
C15A
C19
C16A
O1A