

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Pentacarbonyl-2 κ^5 C-chlorido-1 κ Cl-bis[1(η^5)-cyclopentadienyl](μ -1-oxido-ethylene-1:2 κ^2 O:C)chromium(0)-zirconium(IV)

Catharine Esterhuysen,^{a*} Lizette Retief,^{a‡} Gert J. Kruger,^b Stephanie Cronje^a and Helgard G. Raubenheimer^a

^aDepartment of Chemistry and Polymer Science, University of Stellenbosch, Private Bag X1, Matieland 7602, South Africa, and ^bDepartment of Chemistry and Biochemistry, University of Johannesburg, P O Box 524, Auckland Park, Johannesburg, 2006, South Africa

Correspondence e-mail: ce@sun.ac.za

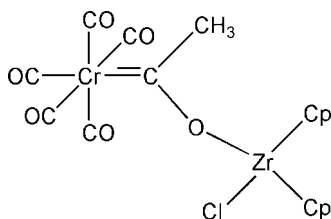
Received 8 December 2008; accepted 15 December 2008

Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.011$ Å; R factor = 0.039; wR factor = 0.109; data-to-parameter ratio = 14.6.

The title compound, $[\text{CrZr}(\text{C}_5\text{H}_5)_2(\text{C}_2\text{H}_3\text{O})\text{Cl}(\text{CO})_5]$, consists of two metal centres, with a (pentacarbonylchromium)-oxymethylcarbene group coordinating as a monodentate ligand to the zirconocene chloride. π -Delocalization through the $\text{Zr}-\text{O}-\text{C}=\text{Cr}$ unit is indicated by a short $\text{Zr}-\text{O}$ distance [2.041 (3) Å] and a nearly linear $\text{Zr}-\text{O}-\text{C}$ angle [170.5 (3)°]. Molecules are aligned with their molecular planes (through Zr, Cl, carbene and Cr) parallel to the ab plane. $\text{C}-\text{H}\cdots\text{Cl}$ interactions result in zigzag chains of molecules propagating parallel to the b axis.

Related literature

For related literature regarding catalytic data of the title compound, see: Sinn *et al.* (1980); Luruli *et al.* (2004, 2006). For other cases of anionic Fischer-type carbenes being used as monodentate ligands, see: Barluenga & Fañanás (2000). For comparable structures, see: Esterhuysen, Nel & Cronje (2008); Esterhuysen, Neveling *et al.* (2008).



[‡] Currently at The Coaching Café, 6 Bright Str, Somerset West, 7130, South Africa.

Experimental

Crystal data

$[\text{CrZr}(\text{C}_5\text{H}_5)_2(\text{C}_2\text{H}_3\text{O})\text{Cl}(\text{CO})_5]$
 $M_r = 491.94$
 Monoclinic, $P2_1/c$
 $a = 12.7395$ (7) Å
 $b = 12.1117$ (6) Å
 $c = 12.7859$ (7) Å
 $\beta = 100.826$ (5)°
 $V = 1937.71$ (18) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 1.27$ mm⁻¹
 $T = 173$ (2) K
 $0.30 \times 0.28 \times 0.08$ mm

Data collection

Philips PW1100 diffractometer
 Absorption correction: ψ scan
 (North *et al.*, 1968)
 $T_{\text{min}} = 0.68$, $T_{\text{max}} = 0.88$
 3423 measured reflections
 3423 independent reflections
 2332 reflections with $I > 2\sigma(I)$
 3 standard reflections
 every 50 reflections
 intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.109$
 $S = 1.06$
 3423 reflections
 235 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.55$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.58$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{Cl16}-\text{H16}\cdots\text{Cl1}^i$	0.95	2.74	3.581 (8)	149

Symmetry code: (i) $-x + 1, y - \frac{1}{2}, -z + \frac{3}{2}$.

Data collection: *PWPC* (Gomm, 1998); cell refinement: *PWPC*; data reduction: *Xtal3.4* (Hall *et al.*, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001; Atwood & Barbour, 2003); software used to prepare material for publication: *publCIF* (Westrip, 2009).

We thank the NRF, the University of Stellenbosch and the University of Johannesburg for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2691).

References

- Atwood, J. L. & Barbour, L. J. (2003). *Cryst. Growth Des.* **3**, 3–8.
 Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
 Barluenga, J. & Fañanás, F. J. (2000). *Tetrahedron*, **56**, 4597–4628.
 Esterhuysen, C., Nel, I. B. J. & Cronje, S. (2008). *Acta Cryst.* **E64**, m1150.
 Esterhuysen, C., Neveling, A., Luruli, N., Kruger, G. J. & Cronje, S. (2008). *Acta Cryst.* **E64**, m1252.
 Gomm, M. (1998). *PWPC*. Institut für Angewandte Physik, Erlangen, Germany.
 Hall, S. R., King, G. S. D. & Stewart, J. M. (1995). Editors. *Xtal3.4 Reference Manual*. University of Western Australia: Lamb, Perth.
 Luruli, N., Grumel, V., Brüll, R., Du Toit, A., Pasch, H., Van Reenen, A. J. & Raubenheimer, H. G. (2004). *J. Polym. Sci. [A1]*, pp. 5121–5133.
 Luruli, N., Heinz, L. C., Grumel, V., Brüll, R., Pasch, H. & Raubenheimer, H. G. (2006). *Polymer*, **47**, 56–66.
 North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Sinn, H., Kaminsky, W., Vollmer, H. J. & Woldt, R. (1980). *Angew. Chem. Int. Ed. Engl.* **19**, 390–392.
 Westrip, S. P. (2009). *publCIF*. In preparation.

supplementary materials

Acta Cryst. (2009). E65, m125 [doi:10.1107/S1600536808042621]

Pentacarbonyl-2 κ^5 C-chlorido-1 κ Cl-bis[1(η^5)-cyclopentadienyl](μ -1-oxidoethylene-1:2 κ^2 O:C)chromium(0)zirconium(IV)

C. Esterhuysen, L. Retief, G. J. Kruger, S. Cronje and H. G. Raubenheimer

Comment

Since Cp₂TiCl₂ was shown to polymerize ethylene when activated by methylaluminumoxane, MAO (Sinn *et al.*, 1980), derivatives of this compound have been synthesized where a Cl ligand was replaced by a monodentate anionic Fischer-type carbene ligand (Barluenga and Fañanás, 2000). We have shown that zirconocene equivalents of this family of homogeneous catalysts, Cp₂Zr(Cl)OC(*R*)M(CO)₅ (where M = W or Cr), catalyze the oligomerization of 1-pentene, as well as the copolymerization of ethene and 1-pentene, in the presence of MAO (Luruli *et al.*, 2004; Luruli *et al.*, 2006). Herein we report the crystal structure of the title zirconocene complex, (I).

In the molecular structure the Zr—O and O—C distances are similar to those found in the equivalent tungsten pentacarbonyl complex (Esterhuysen, Nel & Cronje, 2008). The Zr—O—C angle, on the other hand, is less linear than the previously published tungsten structure [177.4 (7)°], but similar to the hafnocene complex W(CO)₅C(C₆H₅)OHf(C₅H₅)₂Cl (Esterhuysen, Neveling *et al.*, 2008), where the Hf—O—C angle deviates slightly more from linearity [171.4 (3)°]. These results are indicative of π delocalization through the Zr—O—C = W unit.

Molecules are linked by C—H \cdots Cl interactions into zigzag chains along the *b* axis. All molecules in a chain point in the same direction, with their molecular planes parallel. Neighbouring chains in the *a*-direction have the same orientation, thus forming a layer parallel to the *ab*-plane. Molecules in neighbouring layers in the *c*-direction have alternating orientations.

Experimental

A solution of LiCH₃ (11 ml, 1.5M in diethylether, 16.5 mmol) in 10 ml diethylether was added to a well stirred suspension of Cr(CO)₆ (3.30 g, 15.0 mmol) in 100 ml of diethylether over the period of 1.5 h. The mixture was stripped of solvent *in vacuo*. The residue was dried for 3 h, extracted with cold (273 K), degassed water (1 \times 40 ml, 2 \times 20 ml) and the formed solution filtered. The aqueous solution was treated with a solution of [NEt₄]Cl (2.49 g, 15 mmol) in cold, degassed water (4 ml) and the formed precipitate was isolated and dried overnight *in vacuo*. The precipitate was dissolved in warm CH₂Cl₂ (5 ml) layered with pentane and cooled to 258 K to yield yellow crystals of (CO)₅Cr{=C(Me)O}[NEt₄]. A solution of 0.61 g (2.0 mmol) of the product in 30 ml of CH₂Cl₂ was added to a solution of Cp₂ZrCl₂ (0.58 g, 2.0 mmol) in 70 ml of diethylether at 233 K over a period of 40 min. AgBF₄ (0.39 g, 2.0 mmol) was then added to the mixture and stirred for an hour at 233 K. After reaching room temperature the solvent was removed *in vacuo* and the residue extracted in 5 portions of 10 ml of toluene. The extract was filtered, and the filtrate dried over anhydrous MgSO₄. The solution was layered with pentane and kept at 258 K to yield orange crystals suitable for X-ray diffraction analysis.

Refinement

H atoms were positioned geometrically, with C—H = 0.95 Å and 0.98 Å, and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2$ or $1.5U_{\text{eq}}(\text{C})$. Large anisotropy on atoms C16 and C17 suggests the presence of disorder in the C13–C17 Cp ring, however this could not be modeled. Highest peak: 1.03 Å from Zr1; deepest hole: 1.04 Å from Zr1.

Figures

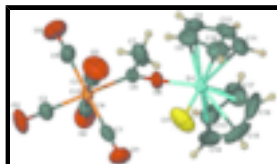


Fig. 1. The molecular structure of (I) showing the atomic labelling scheme and displacement ellipsoids drawn at the 50% probability level.

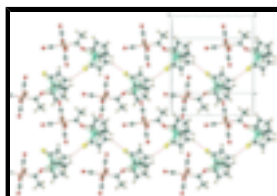


Fig. 2. A portion of the packing diagram showing zigzag chains of molecules forming a layer perpendicular to the *c* axis.

Pentacarbonyl-2κ⁵C-chlorido-1κCl-bis[1(η⁵)-cyclopentadienyl](μ-1-oxidoethylene-1:2κ²O:C)chromiumzirconium

Crystal data

[CrZr(C₅H₅)₂(C₂H₃O)Cl(CO)₅]

$M_r = 491.94$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 12.7395$ (7) Å

$b = 12.1117$ (6) Å

$c = 12.7859$ (7) Å

$\beta = 100.826$ (5)°

$V = 1937.71$ (18) Å³

$Z = 4$

$F_{000} = 976$

$D_x = 1.686$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 48 reflections

$\theta = 2$ – 17°

$\mu = 1.27$ mm⁻¹

$T = 173$ (2) K

Plate, orange

$0.30 \times 0.28 \times 0.08$ mm

Data collection

Philips PW1100
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 173$ (2) K

ω - 2θ scans

Absorption correction: ψ scan

$R_{\text{int}} = 0.0000$

$\theta_{\text{max}} = 25.0^\circ$

$\theta_{\text{min}} = 2.3^\circ$

$h = -15 \rightarrow 14$

$k = 0 \rightarrow 14$

$l = 0 \rightarrow 15$

(North *et al.*, 1968)

$T_{\min} = 0.68$, $T_{\max} = 0.88$

3423 measured reflections

3423 independent reflections

2332 reflections with $I > 2\sigma(I)$

3 standard reflections

every 50 reflections

intensity decay: none

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.039$

$wR(F^2) = 0.109$

$S = 1.06$

3423 reflections

235 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.053P)^2]$$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.55 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.58 \text{ e } \text{\AA}^{-3}$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Zr1	0.67690 (3)	0.45977 (3)	0.79870 (3)	0.03838 (16)
Cr2	0.82371 (6)	0.82936 (6)	0.79664 (6)	0.0441 (2)
Cl1	0.51592 (14)	0.56328 (16)	0.80604 (18)	0.1054 (7)
O1	0.6062 (3)	0.7896 (4)	0.6561 (3)	0.0785 (12)
O2	0.7878 (4)	1.0760 (3)	0.7936 (4)	0.1005 (16)
O3	0.7167 (4)	0.7914 (4)	0.9852 (4)	0.1065 (17)
O4	0.9358 (4)	0.8437 (4)	0.6086 (4)	0.0886 (14)
O5	1.0309 (4)	0.8682 (5)	0.9517 (4)	0.1162 (19)
O6	0.7769 (3)	0.5916 (3)	0.8006 (3)	0.0532 (9)
C1	0.6880 (4)	0.8052 (4)	0.7069 (4)	0.0492 (12)
C2	0.8007 (5)	0.9823 (5)	0.7944 (4)	0.0631 (15)
C3	0.7572 (5)	0.8072 (5)	0.9152 (5)	0.0627 (15)
C4	0.8933 (4)	0.8394 (4)	0.6786 (5)	0.0566 (13)

supplementary materials

C5	0.9534 (5)	0.8515 (5)	0.8931 (5)	0.0689 (16)
C6	0.8496 (4)	0.6625 (4)	0.7962 (4)	0.0449 (11)
C7	0.9547 (4)	0.6101 (5)	0.7899 (6)	0.086 (2)
H7A	1.0080	0.6678	0.7871	0.129*
H7B	0.9469	0.5645	0.7256	0.129*
H7C	0.9780	0.5639	0.8528	0.129*
C8	0.8066 (5)	0.4232 (6)	0.9667 (5)	0.0733 (18)
H8	0.8737	0.4599	0.9772	0.088*
C9	0.7144 (6)	0.4611 (5)	0.9977 (4)	0.0769 (19)
H9	0.7065	0.5287	1.0332	0.092*
C10	0.6349 (5)	0.3814 (6)	0.9672 (4)	0.0758 (18)
H10	0.5635	0.3850	0.9787	0.091*
C11	0.6779 (6)	0.2982 (5)	0.9184 (5)	0.0762 (19)
H11	0.6414	0.2335	0.8893	0.091*
C12	0.7831 (6)	0.3228 (5)	0.9179 (5)	0.0711 (17)
H12	0.8315	0.2779	0.8887	0.085*
C13	0.7269 (6)	0.4442 (6)	0.6179 (5)	0.081 (2)
H13	0.7843	0.4867	0.6012	0.097*
C14	0.6228 (6)	0.4758 (6)	0.6006 (5)	0.084 (2)
H14	0.5943	0.5445	0.5727	0.100*
C15	0.5653 (7)	0.3857 (10)	0.6326 (6)	0.119 (3)
H15	0.4901	0.3814	0.6282	0.142*
C16	0.6386 (11)	0.3060 (7)	0.6711 (6)	0.124 (4)
H16	0.6228	0.2365	0.6989	0.149*
C17	0.7361 (9)	0.3420 (7)	0.6632 (5)	0.106 (3)
H17	0.8011	0.3027	0.6854	0.127*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zr1	0.0412 (3)	0.0306 (2)	0.0410 (3)	-0.0002 (2)	0.00175 (18)	0.0023 (2)
Cr2	0.0464 (5)	0.0299 (4)	0.0544 (5)	0.0011 (3)	0.0054 (4)	0.0004 (3)
Cl1	0.0717 (11)	0.0871 (13)	0.1666 (19)	0.0361 (10)	0.0464 (12)	0.0408 (12)
O1	0.048 (2)	0.097 (3)	0.084 (3)	0.002 (2)	-0.005 (2)	0.002 (2)
O2	0.121 (4)	0.035 (2)	0.140 (4)	0.013 (2)	0.013 (3)	-0.005 (2)
O3	0.134 (5)	0.122 (4)	0.076 (3)	0.023 (4)	0.053 (3)	0.009 (3)
O4	0.086 (3)	0.096 (3)	0.094 (3)	-0.016 (3)	0.042 (3)	-0.007 (3)
O5	0.077 (3)	0.118 (4)	0.132 (4)	-0.015 (3)	-0.033 (3)	-0.002 (4)
O6	0.052 (2)	0.0333 (18)	0.073 (2)	-0.0062 (16)	0.0079 (17)	-0.0001 (16)
C1	0.058 (3)	0.039 (3)	0.054 (3)	0.008 (2)	0.018 (3)	0.005 (2)
C2	0.074 (4)	0.041 (3)	0.071 (4)	0.004 (3)	0.007 (3)	-0.003 (3)
C3	0.076 (4)	0.055 (3)	0.057 (3)	0.008 (3)	0.010 (3)	-0.005 (3)
C4	0.054 (3)	0.045 (3)	0.071 (4)	-0.008 (3)	0.014 (3)	0.000 (3)
C5	0.061 (4)	0.052 (3)	0.088 (4)	-0.007 (3)	-0.002 (3)	-0.003 (3)
C6	0.044 (3)	0.036 (3)	0.052 (3)	0.000 (2)	0.003 (2)	0.002 (2)
C7	0.048 (3)	0.049 (3)	0.160 (7)	0.008 (3)	0.016 (4)	0.004 (4)
C8	0.073 (4)	0.076 (4)	0.058 (4)	-0.007 (4)	-0.020 (3)	0.016 (3)
C9	0.115 (6)	0.071 (4)	0.041 (3)	0.015 (4)	0.004 (3)	-0.011 (3)

C10	0.083 (5)	0.099 (5)	0.047 (3)	-0.006 (4)	0.019 (3)	0.015 (3)
C11	0.124 (6)	0.049 (3)	0.050 (4)	-0.013 (4)	0.002 (4)	0.014 (3)
C12	0.084 (5)	0.065 (4)	0.061 (4)	0.026 (4)	0.005 (3)	0.020 (3)
C13	0.104 (6)	0.093 (5)	0.048 (3)	-0.021 (4)	0.019 (4)	-0.003 (3)
C14	0.100 (5)	0.094 (5)	0.048 (3)	-0.010 (5)	-0.007 (3)	0.023 (3)
C15	0.104 (6)	0.185 (10)	0.053 (4)	-0.072 (7)	-0.021 (4)	-0.008 (5)
C16	0.232 (12)	0.088 (6)	0.047 (4)	-0.088 (8)	0.009 (6)	-0.022 (4)
C17	0.188 (10)	0.076 (5)	0.058 (4)	0.042 (6)	0.032 (5)	-0.012 (4)

Geometric parameters (Å, °)

Zr1—O6	2.041 (3)	C6—C7	1.498 (7)
Zr1—C11	2.4205 (16)	C7—H7A	0.9800
Zr1—C16	2.463 (7)	C7—H7B	0.9800
Zr1—C17	2.470 (6)	C7—H7C	0.9800
Zr1—C12	2.476 (5)	C8—C12	1.373 (8)
Zr1—C11	2.483 (5)	C8—C9	1.387 (9)
Zr1—C15	2.490 (6)	C8—H8	0.9500
Zr1—C8	2.492 (5)	C9—C10	1.400 (9)
Zr1—C9	2.500 (5)	C9—H9	0.9500
Zr1—C10	2.503 (5)	C10—C11	1.354 (9)
Zr1—C14	2.504 (6)	C10—H10	0.9500
Zr1—C13	2.517 (6)	C11—C12	1.374 (9)
Cr2—C2	1.875 (6)	C11—H11	0.9500
Cr2—C5	1.885 (6)	C12—H12	0.9500
Cr2—C3	1.889 (6)	C13—C14	1.358 (9)
Cr2—C4	1.892 (6)	C13—C17	1.362 (9)
Cr2—C1	1.910 (6)	C13—H13	0.9500
Cr2—C6	2.048 (5)	C14—C15	1.417 (10)
O1—C1	1.135 (6)	C14—H14	0.9500
O2—C2	1.146 (6)	C15—C16	1.368 (13)
O3—C3	1.130 (6)	C15—H15	0.9500
O4—C4	1.131 (6)	C16—C17	1.338 (12)
O5—C5	1.141 (7)	C16—H16	0.9500
O6—C6	1.271 (5)	C17—H17	0.9500
O6—Zr1—C11	97.24 (10)	C3—Cr2—C6	87.6 (2)
O6—Zr1—C16	130.2 (3)	C4—Cr2—C6	87.8 (2)
C11—Zr1—C16	110.7 (3)	C1—Cr2—C6	88.58 (19)
O6—Zr1—C17	100.8 (3)	C6—O6—Zr1	170.5 (3)
C11—Zr1—C17	134.4 (2)	O1—C1—Cr2	178.0 (5)
C16—Zr1—C17	31.5 (3)	O2—C2—Cr2	179.2 (6)
O6—Zr1—C12	104.4 (2)	O3—C3—Cr2	178.3 (6)
C11—Zr1—C12	133.79 (17)	O4—C4—Cr2	178.7 (5)
C16—Zr1—C12	85.4 (3)	O5—C5—Cr2	177.9 (6)
C17—Zr1—C12	80.9 (2)	O6—C6—C7	112.5 (4)
O6—Zr1—C11	132.73 (19)	O6—C6—Cr2	123.2 (3)
C11—Zr1—C11	106.9 (2)	C7—C6—Cr2	124.3 (4)
C16—Zr1—C11	77.9 (3)	C6—C7—H7A	109.5
C17—Zr1—C11	90.6 (3)	C6—C7—H7B	109.5

supplementary materials

C12—Zr1—C11	32.2 (2)	H7A—C7—H7B	109.5
O6—Zr1—C15	123.2 (2)	C6—C7—H7C	109.5
C11—Zr1—C15	82.4 (3)	H7A—C7—H7C	109.5
C16—Zr1—C15	32.1 (3)	H7B—C7—H7C	109.5
C17—Zr1—C15	52.7 (3)	C12—C8—C9	107.3 (6)
C12—Zr1—C15	116.3 (3)	C12—C8—Zr1	73.3 (3)
C11—Zr1—C15	100.2 (3)	C9—C8—Zr1	74.2 (3)
O6—Zr1—C8	79.46 (18)	C12—C8—H8	126.3
C11—Zr1—C8	119.22 (18)	C9—C8—H8	126.3
C16—Zr1—C8	117.0 (3)	Zr1—C8—H8	118.2
C17—Zr1—C8	105.2 (3)	C8—C9—C10	107.4 (6)
C12—Zr1—C8	32.1 (2)	C8—C9—Zr1	73.5 (3)
C11—Zr1—C8	53.3 (2)	C10—C9—Zr1	73.9 (3)
C15—Zr1—C8	148.3 (3)	C8—C9—H9	126.3
O6—Zr1—C9	89.1 (2)	C10—C9—H9	126.3
C11—Zr1—C9	87.62 (17)	Zr1—C9—H9	118.3
C16—Zr1—C9	130.9 (3)	C11—C10—C9	107.9 (6)
C17—Zr1—C9	133.9 (2)	C11—C10—Zr1	73.4 (3)
C12—Zr1—C9	53.1 (2)	C9—C10—Zr1	73.6 (3)
C11—Zr1—C9	53.1 (2)	C11—C10—H10	126.0
C15—Zr1—C9	147.1 (3)	C9—C10—H10	126.0
C8—Zr1—C9	32.3 (2)	Zr1—C10—H10	118.8
O6—Zr1—C10	121.38 (19)	C10—C11—C12	108.6 (6)
C11—Zr1—C10	81.00 (17)	C10—C11—Zr1	75.1 (3)
C16—Zr1—C10	103.6 (3)	C12—C11—Zr1	73.6 (3)
C17—Zr1—C10	121.9 (3)	C10—C11—H11	125.7
C12—Zr1—C10	52.9 (2)	C12—C11—H11	125.7
C11—Zr1—C10	31.5 (2)	Zr1—C11—H11	117.6
C15—Zr1—C10	114.7 (3)	C8—C12—C11	108.7 (6)
C8—Zr1—C10	53.5 (2)	C8—C12—Zr1	74.6 (3)
C9—Zr1—C10	32.5 (2)	C11—C12—Zr1	74.2 (3)
O6—Zr1—C14	90.23 (19)	C8—C12—H12	125.7
C11—Zr1—C14	85.7 (2)	C11—C12—H12	125.7
C16—Zr1—C14	53.6 (3)	Zr1—C12—H12	117.5
C17—Zr1—C14	52.9 (3)	C14—C13—C17	109.2 (7)
C12—Zr1—C14	133.6 (2)	C14—C13—Zr1	73.8 (4)
C11—Zr1—C14	130.8 (2)	C17—C13—Zr1	72.2 (4)
C15—Zr1—C14	33.0 (2)	C14—C13—H13	125.4
C8—Zr1—C14	153.9 (3)	C17—C13—H13	125.4
C9—Zr1—C14	173.1 (2)	Zr1—C13—H13	120.3
C10—Zr1—C14	146.9 (2)	C13—C14—C15	106.2 (7)
O6—Zr1—C13	78.71 (18)	C13—C14—Zr1	74.9 (3)
C11—Zr1—C13	115.91 (19)	C15—C14—Zr1	73.0 (3)
C16—Zr1—C13	52.3 (3)	C13—C14—H14	126.9
C17—Zr1—C13	31.7 (2)	C15—C14—H14	126.9
C12—Zr1—C13	108.2 (2)	Zr1—C14—H14	117.5
C11—Zr1—C13	122.3 (2)	C16—C15—C14	107.0 (8)
C15—Zr1—C13	52.6 (3)	C16—C15—Zr1	72.9 (4)
C8—Zr1—C13	122.5 (3)	C14—C15—Zr1	74.1 (4)

C9—Zr1—C13	154.5 (3)	C16—C15—H15	126.5
C10—Zr1—C13	153.4 (2)	C14—C15—H15	126.5
C14—Zr1—C13	31.4 (2)	Zr1—C15—H15	118.6
C2—Cr2—C5	89.1 (2)	C17—C16—C15	108.9 (8)
C2—Cr2—C3	93.4 (2)	C17—C16—Zr1	74.5 (4)
C5—Cr2—C3	88.0 (3)	C15—C16—Zr1	75.0 (5)
C2—Cr2—C4	91.1 (2)	C17—C16—H16	125.5
C5—Cr2—C4	91.6 (3)	C15—C16—H16	125.5
C3—Cr2—C4	175.4 (2)	Zr1—C16—H16	116.8
C2—Cr2—C1	91.2 (2)	C16—C17—C13	108.7 (9)
C5—Cr2—C1	176.2 (2)	C16—C17—Zr1	74.0 (5)
C3—Cr2—C1	88.2 (2)	C13—C17—Zr1	76.1 (4)
C4—Cr2—C1	92.2 (2)	C16—C17—H17	125.7
C2—Cr2—C6	178.9 (2)	C13—C17—H17	125.6
C5—Cr2—C6	91.2 (2)	Zr1—C17—H17	116.3

Hydrogen-bond geometry (\AA , $^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C16—H16 \cdots C11 ⁱ	0.95	2.74	3.581 (8)	149

Symmetry codes: (i) $-x+1, y-1/2, -z+3/2$.

Fig. 1

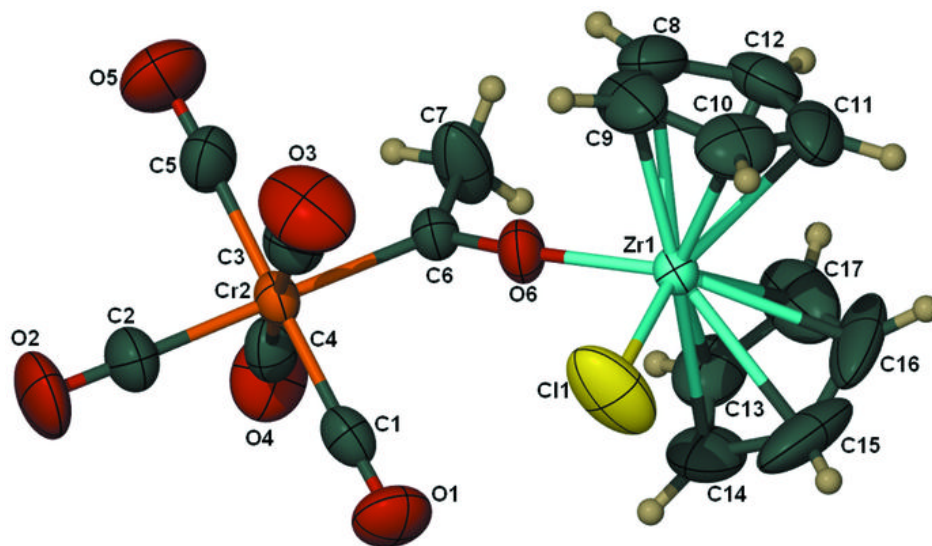


Fig. 2

