Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

μ-Oxido-bis({η^5-1-[3,5-bis(trifluoromethyl)phenyl]-2,3,4,5-tetramethylcyclopentadienyl}dichlorido-titanium(IV))

Qiao-Lin Wu, Ling Ye, Bao Li and Ying Mu*

Key Laboratory for Supramolecular Structure and Materials of the Ministry of Education, School of Chemistry, Jilin University, Changchun 130012, People's Republic of China

Correspondence e-mail: ymu@jlu.edu.cn

Received 11 March 2007
Accepted 20 March 2007

Key indicators

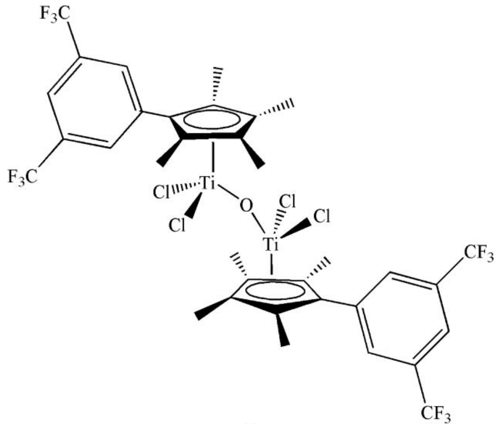
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.049
wR factor = 0.137
Data-to-parameter ratio = 15.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The central bridging O atom of the title homodinuclear titanocene complex, [$Ti_2(C_{17}H_{15}F_6)_2Cl_4O$], lies on a crystallographic inversion center. The Ti^{IV} ion has a distorted tetrahedral coordination geometry defined by two Cl atoms, a cyclopendienyl ring and the O atom.

Comment

Titanocene compounds such as Cp′$TiCl_3$ (Cp′ = cyclopentadienyl or substituted cyclopentadienyl) have received much interest due to their application as active catalysts for styrene polymerization and research shows that catalytic performance improves by ligand modification (Ishihara *et al.*, 1986; Xu, 1998; Lee *et al.*, 2001). Our research efforts are focused on cyclopentadienyl titanium derivatives with different substitutents on the Cp ring, these substituents having varying electronic and steric effects. These types of titanium complexes are sensitive to moisture and readily hydrolyse to produce the corresponding dinuclear compounds in solution. Here we present the crystal structure of one such dinuclear compound, (I).



(I)

The molecular structure of (I) (Fig. 1) is centrosymmetric, the Ti^{IV} ion having a distorted tetrahedral coordination geometry defined by a substituted cyclopentadienyl group, two chlorine atoms, and a bridging O atom. The Ti$\cdots$*Cg* distance (*Cg* is the centroid of atoms C1–C5) of 2.044 (5) Å; other key bond lengths and angles are listed in Table 1 are comparable to those in the structure of [(2,3,4,5-tetramethyl-1-phenylcyclopentadienyl)titanium(IV) dichloride]$_2$(μ-O) (Björgvinsson *et al.*, 1997). The Ti—O—Ti angle is exactly linear by virtue of the inversion center. The dihedral angle between the Cp ring and the attached benzene ring is 58.0 (3)°, thus avoiding steric interactions. The two Cp rings and the two

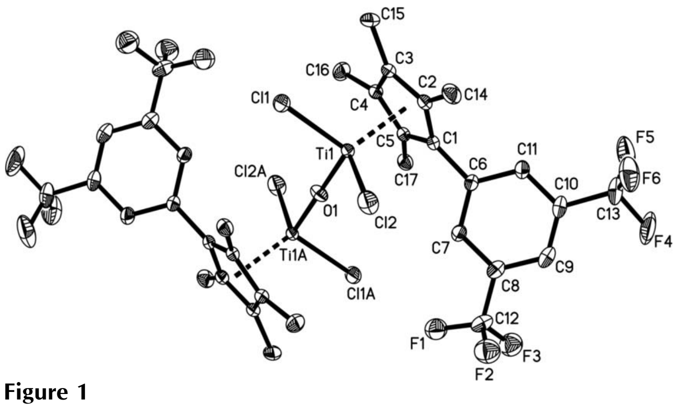


Figure 1

View of the molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. All H atoms have been omitted for clarity. The minor component of the disordered trifluoromethyl goup is not shown. [Symmetry code: (A) $1 - x$, $2 - y$, $1 - z$.]

benzene rings are parallel, again because of the crystallographic symmetry.

Experimental

A mixture of Et_2O (15 ml) and distilled H_2O (15 µl) was added at 258 K to solid {2,3,4,5-tetramethyl-1-[3,5-bis(trifluoromethylphenyl)]cyclopentadienyl}titanium(IV) trichloride (463.5 mg, 1.00 mmol), and the resulting solution was warmed to room temperature and stirred for 36 h. The solvent was removed *in vacuo* and the resulting residue was extracted with a mixed solvent of CH_2Cl_2 and *n*-hexane (1:1, 25 ml). The solution was concentrated to give a red solid (yield 711.0 mg, 81.5%). Red crystals of the title complex suitable for X-ray diffraction were obtained by recrystallization of (I) from a mixture of CH_2Cl_2 and *n*-hexane (1:3) at room temperature.

Crystal data

$[Ti_2(C_{17}H_{15}F_6)_2Cl_4O]$
$M_r = 920.18$
Triclinic, $P\overline{1}$
$a = 8.6637$ (17) Å
$b = 10.551$ (2) Å
$c = 11.630$ (2) Å
$\alpha = 78.63$ (3)°
$\beta = 71.28$ (3)°
$\gamma = 69.52$ (3)°
$V = 938.9$ (4) Å^3
$Z = 1$
Mo $K\alpha$ radiation
$\mu = 0.80$ mm^{-1}
$T = 293$ (2) K
0.28 × 0.24 × 0.19 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.808$, $T_{max} = 0.863$
9309 measured reflections
4265 independent reflections
3604 reflections with $I > 2\sigma(I)$
$R_{int} = 0.017$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.137$
$S = 1.06$
4265 reflections
273 parameters
54 restraints
H-atom parameters constrained
$\Delta\rho_{max} = 1.02$ e Å^{-3}
$\Delta\rho_{min} = -0.77$ e Å^{-3}

Table 1

Selected geometric parameters (Å, °).

Ti1—O1	1.8077 (8)	Ti1—C5	2.352 (3)
Ti1—Cl1	2.2481 (11)	Ti1—C1	2.363 (3)
Ti1—Cl2	2.2492 (11)	Ti1—C3	2.402 (3)
Ti1—C4	2.344 (3)	Ti1—C2	2.412 (3)
O1—Ti1—Cl1	103.97 (5)	Cl1—Ti1—Cl2	103.15 (6)
O1—Ti1—Cl2	102.20 (4)	Ti1—O1—Ti1^{i}	180

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

C-bound H atoms were positioned geometrically, with C—H = 0.93–0.98 Å, and allowed to ride on their parent atoms, with U_{iso}(H) = 1.2 U_{eq}(C) for benzene or $1.5U_{eq}$(C) for methyl H atoms. The F atoms attached to C12 are disordered over two sites with refined occupancies of 0.553 (12) and 0.447 (12), respectively, for the major and minor components. The highest peak in the final difference map is 0.92 Å from atom F2′.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC and Rigaku, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXL97*.

We thank the National Natural Science Foundation of China (grant Nos. 20674024 and 20374023).

References

Björgvinsson, M., Halldorsson, S., Arnason, I., Magull, J. & Fenske, D. (1997). *J. Organomet. Chem.* **544**, 207–215.
Bruker (1997). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Ishihara, N., Seimiya, T., Kuramoto, M. & Uoi, M. (1986). *Macromolecules*, **19**, 2464–2465.
Lee, B. Y., Han, J. W., Seo, H., Lee, I. S. & Chung, Y. K. (2001). *J. Organomet. Chem.* **627**, 233–238.
Rigaku (1998). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC and Rigaku (2002). *CrystalStructure*. Rigaku/MSC Inc., The Woodlands, Texas, USA, and Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Xu, G. (1998). *Macromolecules*, **31**, 586–591.

Acta Cryst. (2007). E**63**, m1194–m1195 Wu *et al.* • $[Ti_2(C_{17}H_{15}F_6)_2Cl_4O]$ **m1195**