

Dichloro[(3-methyl-2-*p*-tolylsulfonamidobutyl)- κ^2 N,O: η^5 -cyclopentadienyl]titanium

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Key indicators

Single-crystal X-ray study

$T = 163$ K

Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å

R factor = 0.021

wR factor = 0.056

Data-to-parameter ratio = 17.7

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

The title compound, $[\text{TiCl}_2(\text{C}_{17}\text{H}_{21}\text{NO}_2\text{S})]$, has a five-coordinate Ti atom bonded to the N and one O atom of the sulfonamide, which is tethered by a substituted two-carbon chain to a bound cyclopentadiene, the cyclopentadiene and two Cl atoms. The Ti–N/O(sulfonamide) bond lengths are 2.0095 (19) and 2.2819 (13) Å, respectively, with the Ti–O bond through one face of the approximate tetrahedron formed by the other four bonds about the titanium.

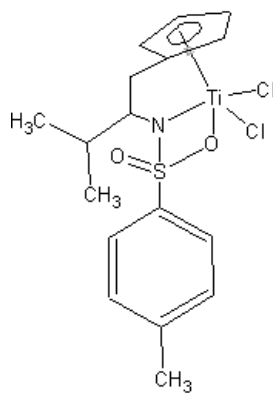
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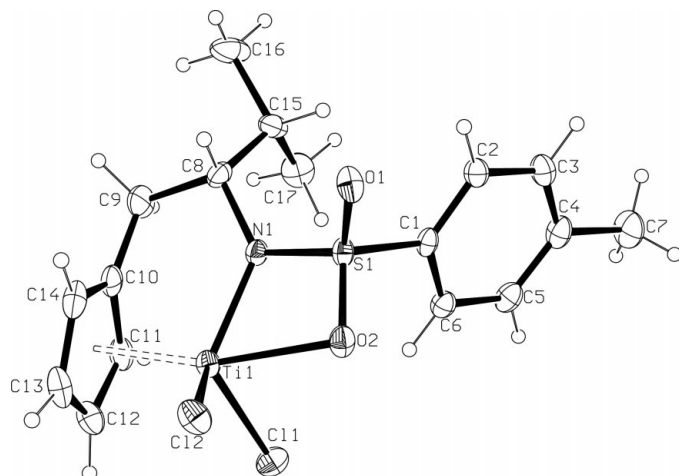
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Comment

The title compound, (I), is related to previously reported structures (Lensink *et al.*, 2001; Lensink, 1998). The crystal structure consists of independent molecules of the absolute configuration shown (Fig. 1) with only one weak intermolecular contact $\text{C13}-\text{H13}\cdots\text{O1}(x-\frac{1}{2}, -\frac{3}{2}-y, -2-z)$ of 2.48 Å. The Ti atom has fivefold coordination through the N and one O atom of the sulfonamide, a cyclopentadiene (assumed to occupy one coordination site) linked *via* an ethyl linkage to the nitrogen, and two Cl atoms. The S–O bonds [O1: 1.431 (1); O2: 1.471 (1) Å] confirm the significant bonding of atom O2 to the titanium in comparison with the free ligand (Gainsford & Lensink, 1996). The Ti–C_g distance is 2.04 Å, within the narrow range 2.01–2.08 Å observed previously (Lensink *et al.*, 2001; Allen & Kennard, 1993; Cambridge Structural Database, 2000). The Ti coordination geometry can best be described as based on a tetrahedron formed by the cyclopentadiene, Cl1, Cl2 and N1, with the Ti–O2 bond through the face formed by N1, Cl1 and Cl2; the angles subtended by O2 at the Ti atom to these three atoms are 65.33 (5), 83.39 (4) and 77.39 (4)°, respectively.



(I)


Figure 1

The molecular structure of (I) (Farrugia, 1997). Displacement ellipsoids are drawn at the 30% probability level. H atoms (unlabelled) have arbitrary radii.

Experimental

The title compound was prepared from the stoichiometric reaction of $\text{Ti}(\text{NMe}_2)_4$ with *N*-(3-methyl-2-*p*-tolylsulfonamidobutyl)cyclopentadiene followed by treatment with excess Me_3SiCl . Crystals were obtained from dichloromethane/pentane. Details and spectroscopic data will be reported in a future publication.

Crystal data

$[\text{TiCl}_2(\text{C}_{17}\text{H}_{21}\text{NO}_2\text{S})]$	Mo $K\alpha$ radiation
$M_r = 422.21$	Cell parameters from 8192 reflections
Orthorhombic, $P2_12_12_1$	$\theta = 2.7\text{--}26.4^\circ$
$a = 8.8053(18) \text{ \AA}$	$\mu = 0.85 \text{ mm}^{-1}$
$b = 13.885(3) \text{ \AA}$	$T = 163(2) \text{ K}$
$c = 15.647(3) \text{ \AA}$	Block, yellow
$V = 1913.0(7) \text{ \AA}^3$	$0.60 \times 0.54 \times 0.53 \text{ mm}$
$Z = 4$	
$D_x = 1.466 \text{ Mg m}^{-3}$	

Data collection

Siemens SMART CCD area-detector diffractometer	3902 independent reflections
φ and ω scans	3769 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (Blessing, 1995)	$R_{\text{int}} = 0.018$
$T_{\text{min}} = 0.571$, $T_{\text{max}} = 0.639$	$\theta_{\text{max}} = 26.5^\circ$
10 578 measured reflections	$h = -11 \rightarrow 10$
	$k = -17 \rightarrow 17$
	$l = -7 \rightarrow 19$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0315P)^2 + 0.3134P]$
$R[F^2 > 2\sigma(F^2)] = 0.021$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.056$	$(\Delta/\sigma)_{\text{max}} = 0.032$
$S = 1.06$	$\Delta\rho_{\text{max}} = 0.20 \text{ e \AA}^{-3}$
3902 reflections	$\Delta\rho_{\text{min}} = -0.17 \text{ e \AA}^{-3}$
220 parameters	Absolute structure: Flack (1983)
H-atom parameters constrained	Flack parameter = 0.01 (2)

Table 1

 Selected geometric parameters (\AA , $^\circ$).

Ti1—N1	2.0095 (15)	Ti1—C10	2.3686 (19)
Ti1—O2	2.2819 (13)	Ti1—C13	2.3784 (19)
Ti1—C11	2.2895 (6)	S1—O1	1.4309 (14)
Ti1—C12	2.3150 (6)	S1—O2	1.4709 (14)
Ti1—C11	2.3491 (19)	S1—N1	1.6075 (14)
Ti1—C14	2.3520 (19)	S1—C1	1.7582 (16)
Ti1—C12	2.365 (2)	N1—C8	1.488 (2)
N1—Ti1—O2	65.33 (5)	O1—S1—N1	113.93 (8)
N1—Ti1—C11	110.15 (4)	O2—S1—N1	98.15 (8)
O2—Ti1—C11	83.39 (4)	S1—O2—Ti1	94.33 (6)
N1—Ti1—C12	122.15 (4)	C8—N1—S1	120.35 (12)
O2—Ti1—C12	77.39 (4)	C8—N1—Ti1	132.89 (11)
C11—Ti1—C12	107.44 (2)	N1—C8—C15	114.63 (14)
N1—Ti1—C11	90.95 (6)	N1—C8—C9	104.30 (15)
O2—Ti1—C11	148.02 (6)	C10—C9—C8	110.44 (16)
N1—S1—O2—Ti1	-7.96 (7)	O2—S1—C1—C2	-137.71 (14)
C1—S1—O2—Ti1	-124.24 (7)	S1—N1—C8—C15	59.59 (19)
O2—S1—N1—C8	166.12 (13)	N1—C8—C9—C10	24.2 (2)
C1—S1—N1—C8	-81.90 (15)	C8—C9—C10—C11	-103.7 (2)

All H atoms, except those on methyl C atoms, were constrained to an isotropic displacement parameter 1.2 times that of the equivalent U of their parent atom. The factor was 1.5 times for the methyl H atoms.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1996) and *SADABS* (Sheldrick, 1996); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) in *WinGX*; software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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References

- Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 1, 31–37.
- Blessing, R. H. (1995). *Acta Cryst.* **A51**, 33–38.
- Cambridge Structural Database (2000). *ConQuest*. Version 1.0. Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge, England.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Gainsford, G. J. & Lensink, C. L. (1996). *Acta Cryst.* **C52**, 472–474.
- Lensink, C. (1998). *J. Organomet. Chem.* **553**, 387–392.
- Lensink, C., Gainsford, G. J. & Baxter, N. I. (2001). *Acta Cryst.* **C57**, 366–367.
- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
- Siemens (1996). *SMART* and *SAINT*. Versions 4.0. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.