Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

Dichloro{η^5:η^1-[2-(*p*-tolylsulfonamido)prop-1-yl]cyclopentadienyl}titanium

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The title compound, [$TiCl_2(C_{15}H_{17}NO_2S)$], has a five-coordinate titanium bonded to the N and one O atom of the sulfonamide tethered by a substituted two-carbon chain to a bound cyclopentadiene (assumed to occupy one position), and two Cl atoms. The Ti—N,O(sulfonamide) bond lengths are 1.994 (3) and 2.334 (3) Å, respectively. The Ti—O bond passes through one face of the approximate tetrahedron formed by the other four bonds.

Received 17 May 2001
Accepted 30 May 2001
Online 15 June 2001

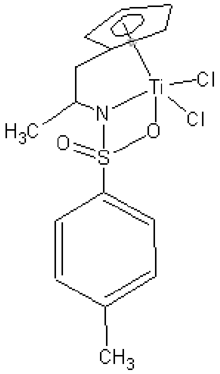
Key indicators

Single-crystal X-ray study
T = 168 K
Mean σ(C–C) = 0.007 Å
Disorder in main residue
R factor = 0.056
wR factor = 0.132
Data-to-parameter ratio = 17.4

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

Comment

The title compound, (I), is related to previously reported structures (Lensink, 1998; Lensink, Gainsford & Baxter, 2001; Lensink, Gainsford & Brandsma, 2001). The crystal structure consists of independent molecules (Fig. 1) of both enantiomorphs to make a racemic crystal with one significant intermolecular contact: C11—H11···Cl2^{i} is 2.80 Å for symmetry operation $\frac{3}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z$. The titanium has five-fold 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: 1.439 (3) Å; O2: 1.471 (3) Å] confirm the significant bonding of O2 to titanium; the Ti—C_g distance is 2.04 Å and lies within the narrow range 2.01–2.08 Å observed previously (Lensink, Gainsford & Baxter, 2001; Lensink, Gainsford & Brandsma, 2001; Allen & Kennard, 1993). The titanium coordination can best be described as being based on a tetrahedron formed by the cyclopentadiene, and the Cl1, Cl2 and N1 atoms with the Ti—O2 bond passing through the face formed by N1, Cl1 and Cl2; the angles subtended by O2 at the titanium to these three atoms are 64.50 (13), 79.65 (10) and 80.10 (9)°, respectively.



(I)

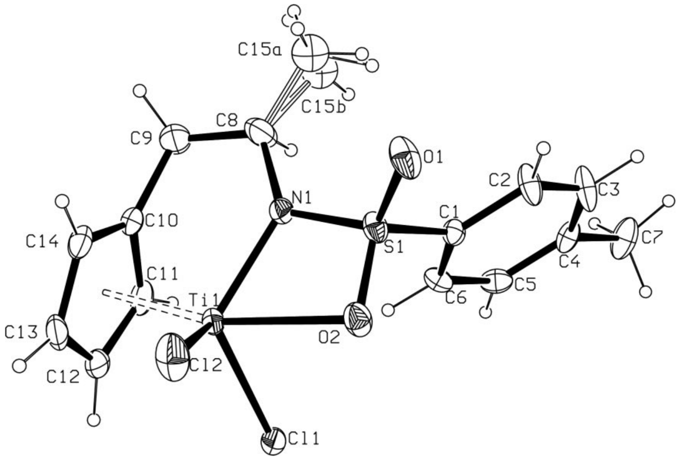


Figure 1
The molecular structure of (I) (Johnson, 1976; Farrugia, 1997). Displacement ellipsoids are drawn at the 20% probability level with the disordered methyl C atoms shown with three line bonds. H atoms (unlabelled) have arbitrary radii.

Table 1
Selected geometric parameters (Å, °).

Ti1—N1	1.994 (3)	S1—O1	1.439 (3)
Ti1—Cl1	2.2958 (16)	S1—O2	1.471 (3)
Ti1—Cl2	2.3055 (17)	S1—N1	1.608 (3)
Ti1—O2	2.334 (3)	S1—C1	1.759 (4)
Ti1—C14	2.339 (5)	C8—C15*B*	1.439 (16)
Ti1—C11	2.361 (5)	C8—C9	1.456 (7)
Ti1—C13	2.366 (4)	C8—C15*A*	1.472 (12)
Ti1—C12	2.368 (4)	C9—C10	1.497 (7)
Ti1—C10	2.378 (4)		
N1—Ti1—Cl1	114.78 (11)	O1—S1—N1	115.8 (2)
N1—Ti1—Cl2	115.24 (11)	O2—S1—N1	98.12 (18)
Cl1—Ti1—Cl2	109.16 (7)	O1—S1—C1	107.10 (19)
N1—Ti1—O2	64.50 (13)	S1—O2—Ti1	93.78 (15)
Cl1—Ti1—O2	79.65 (10)	C15*B*—C8—N1	127.2 (8)
Cl2—Ti1—O2	80.10 (9)	N1—C8—C15*A*	117.9 (7)
O1—S1—O2	117.5 (2)	C8—C9—C10	112.4 (4)
O2—S1—N1—C8	−179.0 (5)	S1—N1—C8—C15*A*	−21.5 (11)
O2—S1—C1—C6	59.4 (4)	C15*A*—C8—C9—C10	−155.8 (10)
S1—N1—C8—C9	−177.8 (4)	C8—C9—C10—C14	86.2 (7)

Experimental

The title compound was prepared from the stoichiometric reaction of $Ti(NMe_2)_4$ with *N*-(2-*p*-tolylsulfonamido-propyl)-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

$[TiCl_2(C_{15}H_{17}NO_2S)]$
$M_r = 394.16$
Monoclinic, $P2_1/n$
$a = 10.874$ (4) Å
$b = 13.517$ (5) Å
$c = 12.057$ (4) Å
$\beta = 101.717$ (5)°
$V = 1735.4$ (10) Å^3
$Z = 4$

$D_x = 1.509$ Mg m$^{-3$}
Mo $K\alpha$ radiation
Cell parameters from 1024 reflections
$\theta = 3.1$–25.9°
$\mu = 0.93$ mm^{-1}
$T = 168$ (2) K
Block, yellow
0.50 × 0.44 × 0.12 mm

Data collection

CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (Blessing, 1995)
$T_{\min} = 0.710$, $T_{\max} = 0.895$
21 781 measured reflections
3504 independent reflections

2182 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.078$
$\theta_{\max} = 26.3$°
$h = -13 \rightarrow 13$
$k = -16 \rightarrow 16$
$l = -14 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.132$
$S = 1.03$
3504 reflections
201 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0443P)^2 + 3.0084P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.009$
$\Delta\rho_{\max} = 0.52$ e Å^{-3}
$\Delta\rho_{\min} = -0.41$ e Å^{-3}

Atom C15 was disordered over two sites (*A* and *B*) with final occupancies 0.589:411 (17). All H atoms except those on methyl C atoms and C8 were constrained to an isotropic displacement parameter 1.2 times that of the equivalent *U* of their parent atom. This factor was 1.5 times for the other 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: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997) based on *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *CIFTAB* (Sheldrick, 1997).

We wish to thank Drs J. Wikaira and C. Richardson, University of Canterbury, for their assistance.

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