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Dichloro{*N*-[2-(η^5-indenyl)ethyl]-*p*-tolylsulfonamido-κ*N*}titanium

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The title compound, [$TiCl_2(C_{18}H_{17}NO_2S)$], has a Ti atom bonded tetrahedrally to the N atom of the sulfonamide, which is tethered by a substituted two-carbon chain to a bound indenyl, the indenyl and two Cl atoms. The Ti—N bond length is 1.954 (7) Å.

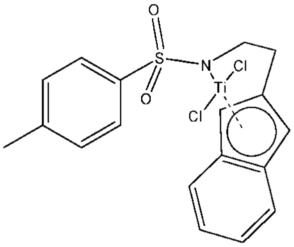
Key indicators

Single-crystal X-ray study
T = 163 K
Mean σ(C–C) = 0.010 Å
Disorder in main residue
R factor = 0.087
wR factor = 0.181
Data-to-parameter ratio = 10.3

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 *et al.*, 2001; Brandsma *et al.*, 2002). The racemic crystal structure consists of independent molecules (one enantiomer is shown in Fig. 1), with only weak intermolecular contacts, *e.g.* C9—H9*A*···Cl2($x-1, y, z$), with a C9···Cl2 distance of 3.482 (7) Å. The Ti atom has tetrahedral coordination through the N atom of the sulfonamide, the indenyl cyclopentadiene ring (assumed to occupy one coordination site) linked *via* an ethyl linkage to the nitrogen, and two Cl atoms [C_g—Ti1—N1 101.7 (5)°, where C_g is the centroid of C10—C14 ring]. The Ti—C_g distance is 2.057 (7) Å, within the normal range of 2.03–2.13 Å; the indenyl dimensions are normal (Allen & Kennard, 1993; *ConQuest*, 2001). The Ti—N distance of 1.954 (7) Å is indistinguishable from that found [1.963 (2) Å] in the analogous cyclopentadienyl compound dichloro[η^5-(2-*p*-toluenesulfonamidoethyl)cyclopentadienyl]titanium (Lensink, 1998).



(I)

Experimental

The title compound was prepared from the stoichiometric reaction of $Ti(NMe_2)_4$ with *N*-(2-*p*-tolylsulfonamidoethyl)indene (Lensink, 1995), 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

[$TiCl_2(C_{18}H_{17}NO_2S)$]
M_r = 430.19
Monoclinic, $P2_1/c$
a = 7.3761 (8) Å
b = 17.3967 (19) Å
c = 13.9155 (15) Å
β = 96.961 (3)°
V = 1772.5 (3) Å^3
Z = 4

D_x = 1.612 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2012 reflections
θ = 3.7–23.5°
μ = 0.91 mm^{-1}
T = 163 (2) K
Block, dark red
0.50 × 0.24 × 0.18 mm

Data collection

Siemens CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (Sheldrick, 1996; Blessing, 1995)
$T_{\min} = 0.700$, $T_{\max} = 0.848$
4699 measured reflections

2932 independent reflections
2208 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.047$
$\theta_{\max} = 26.3°$
$h = -9 \rightarrow 7$
$k = -19 \rightarrow 17$
$l = -17 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.087$
$wR(F^2) = 0.181$
$S = 1.17$
2932 reflections
284 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0635P)^2 + 3.4344P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.007$
$\Delta\rho_{\max} = 0.63$ e Å^{-3}
$\Delta\rho_{\min} = -0.51$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ti1—N1	1.954 (7)	S1—O1	1.437 (5)
Ti1—Cl1	2.2517 (19)	S1—N1	1.606 (5)
Ti1—Cl2	2.262 (2)	S1—C1	1.722 (17)
Ti1—C11	2.300 (6)	C14—C15	1.421 (10)
Ti1—C13	2.487 (7)	C15—C16	1.355 (10)
S1—O2	1.421 (5)	C16—C17	1.424 (11)
N1—Ti1—Cl1	111.43 (16)	Cl1—Ti1—Cl2	108.40 (8)
N1—Ti1—Cl2	112.32 (17)		

The completeness of data collection was relatively low, 0.816 for $\theta_{\max} = 26.3°$. Excessive buildup of ice, and subsequent loss of the crystal, restricted the collection to the first 360° run, with a small part of reciprocal space not sampled. There was still enough redundancy to permit adequate absorption corrections. The tolyl ring was refined in two orientations with unit total occupancy (see Fig. 1); primed and unprimed atom occupancies were 0.46 (2) and 0.54 (2), respectively. All H atoms, except those on methyl C atoms, were constrained to have $U_{\rm iso}$ values of 1.2 times $U_{\rm eq}$ 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: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP* in *PLATON* (Spek, 1990); software used to prepare material for publication: *SHELXL*97.

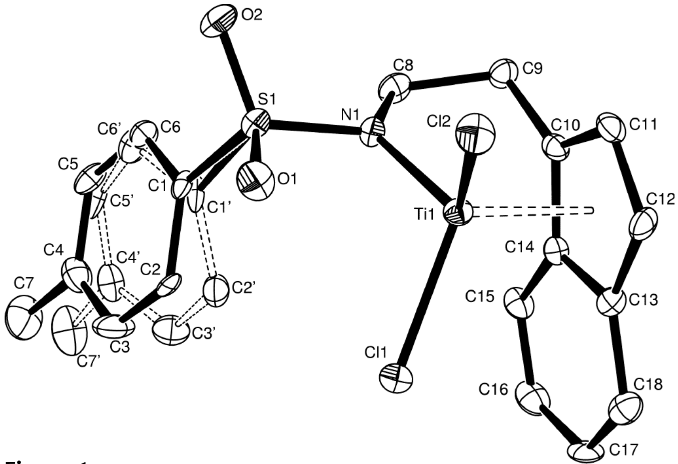


Figure 1
The molecular structure of (I). The alternative conformations of the tolyl ring are shown with solid and dotted bonds. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been excluded for clarity.

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