Chloro(η^5-cyclopentadienyl){[η^5:η^1-2-(*p*-tolyl-sulfonamido)ethyl]cyclopentadienyl}titanium

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Key indicators

Single-crystal X-ray study

T = 149 K

Mean σ(C–C) = 0.006 Å

R factor = 0.044

wR factor = 0.097

Data-to-parameter ratio = 11.4

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

The title compound, [TiCl(C$_5$H$_5$)(C$_{14}$H$_{15}$NO$_2$)], has a four-coordinate Ti atom tetrahedrally bonded to the N atom of the sulfonamide moiety, which is tethered by a substituted two-carbon chain to a bound cyclopentadiene ring, another cyclopentadiene ligand and one Cl atom. The Ti—N,Cl bond lengths are 2.123 (3) and 2.3250 (13) Å, respectively.

Comment

The title compound, (I), is related to previously reported structures (Lensink, Gainsford & Baxter, 2001; Lensink, Gainsford & Brandsma, 2001; Lensink, 1998). The crystal structure of (I) consists of independent molecules (Fig. 1), with only a few weak intermolecular contacts, *e.g.* C17—H17···O1(x−1/2, 1/2−y, z−1/2), with a C17···O1 distance of 3.422 (5) Å.

(I)
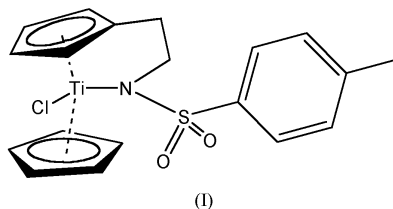
The Ti atom has tetrahedral coordination through the N atom of the sulfonamide moiety, a cyclopentadiene (assumed to occupy one coordination site) linked *via* an ethyl linkage to the nitrogen, one chloride and another cyclopentadiene. The Ti—C$_g$ distances (C$_g$ being the cyclopentadienyl centres) are 2.061 (2) and 2.063 (2) Å, within the narrow range 2.01–2.08 Å observed previously (Lensink, Gainsford & Baxter, 2001; Lensink, Gainsford & Brandsma, 2001; Allen & Kennard, 1993; *ConQuest*, 2001), with a C$_g$—Ti—C$_g$′ angle of 131.37 (9)°. The Ti—N distance of 2.123 (3) Å is shorter than in two related compounds: 2.437 Å in (η^5-cyclopentadienyl){η^5-[(2-*N*,*N*-dimethylamino)ethyl]tetramethylcyclopentadienyl]chlorotitanium (CSD refcode NOGYAJ; Beckhaus *et al.*, 1997) and 2.428 Å in chloro(η^5-diaminoethylcyclopentadienyl-N)(η^5-dimethylaminoethyl)cyclopentadienyl)titanium(III) (REXGOQ; Qian *et al.*, 1997), but longer than the Ti—N(imino) distance found in (η^5-2-isopropyliminoethylcyclopentadienyl)dichlorotitanium (NOMSUD; Sinnema *et al.*, 1997). The Ti—Cl distance is within the range found for other titanium complexes with bound cyclopentadienes (2.29–2.50 Å; *Conquest*, 2001): 2.325 (1) Å compared with 2.461, 2.483 and 2.275/2.300 Å in NOGYAJ, REXGOQ and NOMSUD, respectively. The lengthening of the Ti—N bonds in these four compounds parallels the change from trigonal

planar to trigonal pyramidal geometry of their N atoms [here N1 is 0.277 (4) Å from the plane of Ti1, S1 and C8]. A more dominant factor for the change compared to that in NOMSUD is the increased intramolecular crowding: between the sulfonamide O atoms and cyclopentadienyl ring here, involving the two methyl groups bound to the nitrogen in NOGYAG and REXGOQ, and also the methyl groups on the cyclopentadienyl in REXGOQ.

Experimental

The title compound, (I), was prepared from the stoichiometric reaction of $(C_{14}H_{15}NO_2S)TiCl_2$ with NaC_5H_5. Crystals were obtained from dichloromethane/pentane. Details and spectroscopic data will be reported in a future publication.

Crystal data

$[TiCl(C_5H_5)(C_{14}H_{15}NO_2S)]$	$D_x = 1.585$ Mg m^{-3}
$M_r = 409.77$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 8192
$a = 7.9620$ (18) Å	reflections
$b = 18.734$ (2) Å	$\theta = 2.9$–24.4°
$c = 11.6482$ (15) Å	$\mu = 0.79$ mm^{-1}
$\beta = 98.678$ (11)°	$T = 149$ (2) K
$V = 1717.5$ (5) Å^3	Needle, yellow
$Z = 4$	$0.40 \times 0.10 \times 0.04$ mm

Data collection

Siemens CCD area-detector diffractometer	2551 independent reflections
	1746 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.062$
Absorption correction: multi-scan (Sheldrick, 1996; Blessing, 1995)	$\theta_{max} = 26.3°$
	$h = -9 \rightarrow 9$
$T_{min} = 0.743$, $T_{max} = 0.969$	$k = -23 \rightarrow 23$
6268 measured reflections	$l = -12 \rightarrow 10$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.044$	$w = 1/[\sigma^2(F_o^2) + (0.039P)^2]$
$wR(F^2) = 0.097$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.97$	$(\Delta/\sigma)_{max} < 0.001$
2551 reflections	$\Delta\rho_{max} = 0.29$ e Å^{-3}
223 parameters	$\Delta\rho_{min} = -0.31$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ti1—N1	2.123 (3)	S1—N1	1.602 (4)
Ti1—Cl1	2.3250 (13)	S1—C1	1.779 (4)
S1—O1	1.441 (3)	N1—C8	1.491 (5)
S1—O2	1.444 (3)		
N1—Ti1—Cl1	99.91 (9)	C8—N1—Ti1	114.2 (3)
C8—N1—S1	112.7 (2)	S1—N1—Ti1	125.25 (19)

The completeness of data collection was relatively low, 0.730 for $\theta_{max} = 26.3°$. Excessive build-up of ice, coupled with subsequent loss of the crystal, restricted the collection to the initial 360° scan, leaving
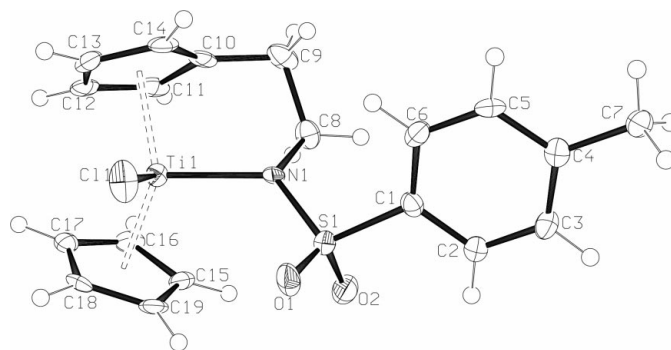

Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

part of the reciprocal space not sampled. Sufficient redundancy of data permitted adequate absorption corrections. All H atoms, except those on methyl C atoms, were constrained to U_{iso} 1.2 times U_{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*-3 in *WinGX* (Farrugia, 1997, 1999); software used to prepare material for publication: *SHELXL*97.

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