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Key indicators

Single-crystal X-ray study

T = 170 K

Mean $\sigma(\text{C}-\text{C}) = 0.007 \text{ \AA}$

Disorder in main residue

R factor = 0.052

wR factor = 0.140

Data-to-parameter ratio = 17.6

For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Bis(μ -N-(3-[3-indenyl]propyl)-p-toluenesulfamido-
N,O,O')-dimethylamidodichlorotitanium)

The title compound, $[\text{Ti}(\text{C}_{21}\text{H}_{26}\text{N}_2\text{O}_2\text{S})\text{Cl}_2]$, crystallizes as a centrosymmetric dimer, with an eight-membered ring derived from the monomer sub-units by the formation of two Ti—(N,O)—S—O head-to-tail sequences around a crystallographic inversion centre. The titanium atoms each have a distorted octahedral geometry through the nitrogen and one oxygen of the sulfonamido group [Ti1—O1, Ti—N1 2.280 (3), 2.091 (3) Å], one oxygen from the adjacent sulfonamide [Ti—O2 2.170 (3) Å], a dimethylamido nitrogen and two chlorides.

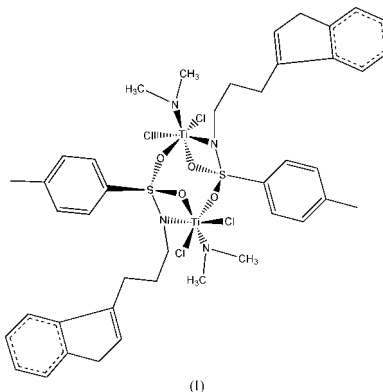
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Comment

The title compound, which is related to previously reported structures (Lensink, 1998, Lensink *et al.*, 2001), crystallizes as a centrosymmetric dimer with an eight-membered ring derived from the monomer sub-units by the formation of two Ti—(N,O)—S—O head-to-tail sequences around a crystallographic inversion centre (Fig. 1, Table 1). There are no significant intermolecular contacts in the crystal structure. The titanium atoms have a distorted octahedral geometry through the nitrogen and one oxygen of the sulfonamido group [Ti1—O1, Ti—N1 2.280 (3), 2.091 (3) Å], one oxygen from the adjacent sulfonamide [Ti—O2 2.170 (3) Å], a dimethylamido nitrogen and two chlorides. A similar ring structure has been reported for the yttrium compound, bis(μ_2 -*trans*-1,2-bis(2,4,6-tri-isopropylbenzene-sulfonamidato)cyclohexane-*N,N',O,O',O''*)-bis(methylsilyl)-amido-yttrium(III)) (Goerlitzer *et al.*, 1998).



The Ti—N2 bond distance of 1.860 (4) Å is consistent with $N(p\pi)-M(d\pi)$ interaction expected for a dimethylamide, and the Ti—Cl bond distances and relevant geometric parameters are similar to those found in dichloro-(4-methyl-2-(tri methylsilylamino)pyridine-*N,N'*)-dimethylamino-dimethyl-amido-titanium (Fuhrmann *et al.*, 1996). The S—O bond

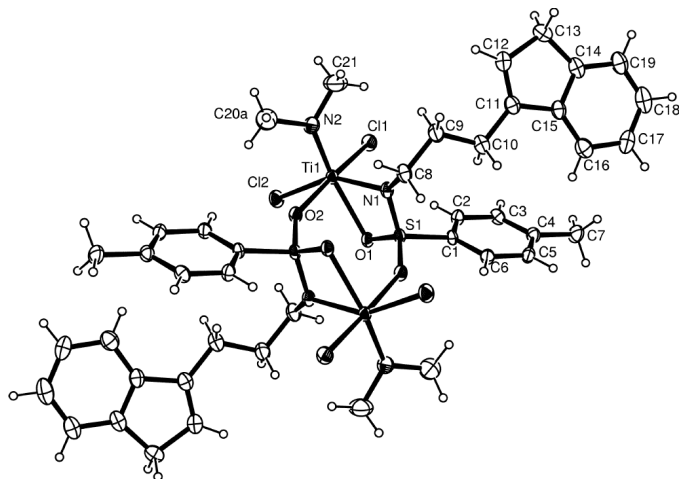


Figure 1
Molecular structure of $[\text{Ti}(\text{C}_{21}\text{H}_{26}\text{N}_2\text{O}_2\text{S})\text{Cl}_2]$ (Farrugia, 1997). Displacement ellipsoids are drawn at the 30% probability level. H atoms have arbitrary radii.

distances [1.471 (3), 1.477 (3) Å] reflect the equivalent dative binding of the O atoms to the titanium centres (Lensink *et al.*, 2001). By comparison with the free 3-[3-indenyl]propyl group in *N*-(3-(3-indenyl)propyl)benzylammonium bromide (Groux *et al.*, 1999), it appears that the total geometry of the group is unaffected by its link to the complex, with the only significant difference being close to the nitrogen N1, with an N1—C8—C9—C10 torsion angle 74.8 (5)° in the complex, compared with −54.4 (3)° in the free group.

Experimental

A solution of $\text{C}_9\text{H}_7(\text{CH}_2)_3\text{N}(\text{H})\text{SO}_2\text{C}_6\text{H}_4\text{CH}_3$ (0.13 g, 0.40 mmol) in benzene-*d*₆ (2 ml) was added dropwise to a solution of $\text{Ti}(\text{NMe}_2)_4$ (89 mg, 0.40 mmol) dissolved in benzene-*d*₆ (3 ml), turning the mixture from yellow to orange. The mixture was refluxed over a period of 4 days. Subsequently Me_3SiCl (108 mg, 0.99 mmol) was slowly added. The mixture was stirred for 20 h, resulting in a dark-brown solution. Recrystallization from a CH_2Cl_2 /pentane mixture resulted in crystals suitable for X-ray analysis. Yield: 20 mg (10%).

Crystal data

$[\text{Ti}(\text{C}_{21}\text{H}_{26}\text{NO}_2\text{S})\text{Cl}_2]$
 $M_r = 489.30$
 Triclinic, $P\bar{1}$
 $a = 7.688$ (3) Å
 $b = 10.122$ (5) Å
 $c = 16.212$ (7) Å
 $\alpha = 94.808$ (6)°
 $\beta = 100.973$ (5)°
 $\gamma = 109.200$ (5)°
 $V = 1154.9$ (9) Å³

$Z = 2$
 $D_x = 1.407$ Mg m^{−3}
 Mo $K\alpha$ radiation
 Cell parameters from 6100 reflections
 $\theta = 2.6$ – 25.8 °
 $\mu = 0.71$ mm^{−1}
 $T = 170$ (2) K
 Block, orange-brown
 $0.36 \times 0.20 \times 0.16$ mm

Data collection

Siemens CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (Blessing, 1995)
 $T_{\min} = 0.393$, $T_{\max} = 0.892$
 14834 measured reflections

4641 independent reflections
 2627 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.078$
 $\theta_{\max} = 26.4$ °
 $h = -9 \rightarrow 4$
 $k = -12 \rightarrow 12$
 $l = -20 \rightarrow 20$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.052$
 $wR(F^2) = 0.140$
 $S = 0.94$
 4641 reflections
 264 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0747P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.73$ e Å^{−3}
 $\Delta\rho_{\min} = -0.54$ e Å^{−3}

Table 1

Selected geometric parameters (Å, °).

Ti1—N2	1.860 (4)	Ti1—Cl2	2.3196 (14)
Ti1—N1	2.091 (3)	S1—O1	1.471 (3)
Ti1—O2	2.170 (3)	S1—O2 ⁱ	1.477 (3)
Ti1—O1	2.280 (3)	S1—N1	1.566 (3)
Ti1—Cl1	2.3010 (16)	S1—C1	1.776 (4)
N2—Ti1—N1	100.84 (15)	C8—N1—Ti1	134.6 (3)
Cl1—Ti1—Cl2	95.64 (5)	S1—N1—Ti1	99.79 (16)
C8—N1—S1	122.1 (3)		

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

All H atoms except those on methyl C atoms were constrained with a riding model, with an isotropic thermal parameter 1.2 times that of the equivalent U of their parent atom. Atom C20 was disordered over two sites (*a/b*), with final occupancies 0.78 (1)/0.22(1) and a common U of 0.069 Å².

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); software used to prepare material for publication: *SHELXL97*.

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