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## Key indicators

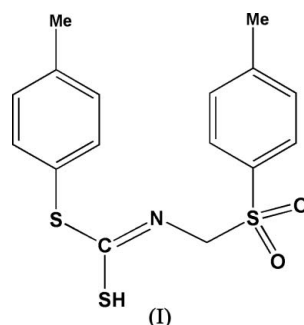
Single-crystal X-ray study  
 $T = 298\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$   
 $R$  factor = 0.056  
 $wR$  factor = 0.158  
Data-to-parameter ratio = 20.3For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.**(*E*)-1-Mercapto-1-(*p*-tolylsulfanyl)-*N*-(*p*-tosylmethyl)  
methanimine: a two-dimensional supramolecular  
network of  $\text{S}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\pi$  interactions**

In the crystal structure of the title compound,  $\text{C}_{16}\text{H}_{17}\text{NO}_2\text{S}_3$ , the dihedral angle between the mean planes of the two benzene rings is  $70.8(1)^\circ$ . There are intermolecular  $\text{S}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\pi$  interactions, and molecules are linked into a two-dimensional network. The layers are further linked together by weak  $\text{C}-\text{H}\cdots\text{O}$  interactions.

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## Comment

In previous papers, we have reported the structures of niobium complexes (Kakeya *et al.*, 2004, 2006). As part of a systematic investigation of their catalytic properties, we report here the crystal structure of the title compound, (I), which was obtained by a reaction of *p*-tosylmethyl isocyanide with a niobium complex. Aromatic sulfonates are used in many biological fields, and crystallographic studies of them have been undertaken in view of their biological importance (Manivannan *et al.*, 2005, and references therein).



The structure of (I), with the atom-numbering scheme, is shown in Fig. 1. Selected bond lengths are given in Table 1.

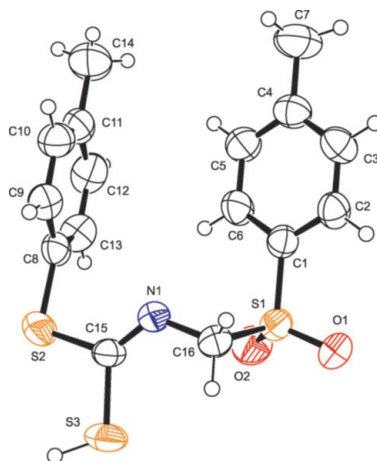
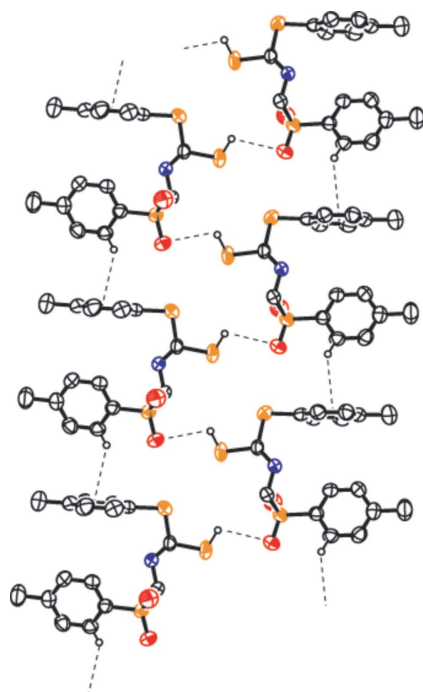
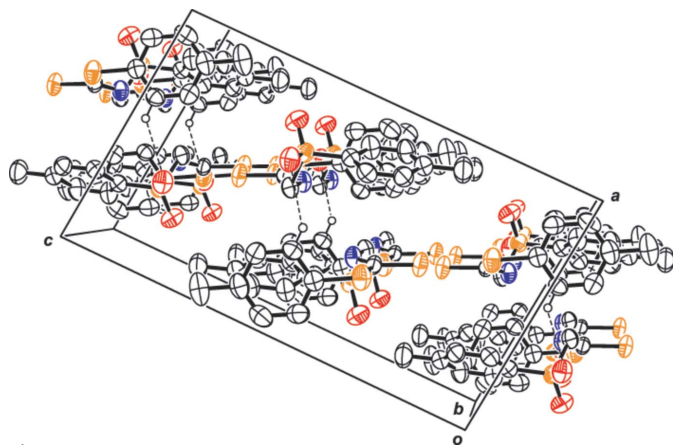


Figure 1

A perspective view of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

**Figure 2**

A view of the two-dimensional network of (I). Dashed lines indicate intralayer S—H...O and C—H... $\pi$  interactions. H atoms not involved in hydrogen bonding have been omitted.

**Figure 3**

A molecular packing diagram of (I). Dashed lines indicate interlayer C—H...O interactions. H atoms not involved in hydrogen bonding have been omitted.

The S—C $sp^2$  bond lengths agree with the normal S—C $sp^2$  bond length (1.75 Å; Allen *et al.*, 1987). Other S—C, S—O and S=O bond lengths are comparable to those found in related structures which contain *p*-toluenesulfonyl groups (Manivannan *et al.*, 2005). The dihedral angle between the mean planes of the two benzene rings is 70.8 (1)°. This non-coplanar orientation of the two benzene rings is similar to that in other aromatic sulfonates (Manivannan *et al.*, 2005).

There are two types of hydrogen-bonding interactions, *viz.* S—H...O and C—H... $\pi$  (Table 2), connecting neighbouring molecules and forming a two-dimensional network (Fig. 2). The layers are further linked by weak C—H...O interactions, as shown in Fig. 3.

## Experimental

To a mixture of [Nb<sub>2</sub>( $\mu$ -Cl)<sub>2</sub>Cl<sub>4</sub>( $\mu$ -Me<sub>2</sub>S)(Me<sub>2</sub>S)<sub>2</sub>] (Kakeya *et al.*, 2006) (0.1 g, 0.17 mmol) and *p*-tosylmethyl isocyanide (0.22 g, 1.1 mmol) in a 100 ml Schlenk tube under an argon stream was added dichloromethane (30 ml). The solution changed from purple to colourless after stirring for 5 min at room temperature. The resulting precipitates were removed by filtration, and the filtrate was concentrated to dryness, leaving a colourless powder. The crude product was washed with *n*-hexane (10 ml) three times and dried under reduced pressure. The compound thus obtained was recrystallized from CH<sub>2</sub>Cl<sub>2</sub>–*n*-hexane (1:7 *v/v*; yield 105 mg, 80%). IR (KBr,  $\nu$ , cm<sup>-1</sup>): 1727 (C=N). <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta$  7.5–7.0 (*m*, 8H, C<sub>6</sub>H<sub>4</sub>), 4.59 (*d*,  $J_{\text{HH}} = 7.0$  Hz, 2H, CH<sub>2</sub>), 2.45 (*s*, 1H, SH), 2.43 (*s*, 6H, CH<sub>3</sub>), 2.38 (*s*, 6H, CH<sub>3</sub>).

## Crystal data

C<sub>16</sub>H<sub>17</sub>NO<sub>2</sub>S<sub>3</sub>  
 $M_r = 351.52$   
 Monoclinic,  $P2_1/c$   
 $a = 10.2686$  (6) Å  
 $b = 9.5313$  (5) Å  
 $c = 17.5095$  (9) Å  
 $\beta = 95.708$  (2)°  
 $V = 1705.21$  (16) Å<sup>3</sup>  
 $Z = 4$

$D_x = 1.369$  Mg m<sup>-3</sup>  
 Mo K $\alpha$  radiation  
 Cell parameters from 2190 reflections  
 $\theta = 2.4$ – $24.3^\circ$   
 $\mu = 0.44$  mm<sup>-1</sup>  
 $T = 298$  (2) K  
 Plate, colourless  
 $0.25 \times 0.22 \times 0.08$  mm

## Data collection

Bruker SMART APEX CCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\text{min}} = 0.852$ ,  $T_{\text{max}} = 0.966$   
 12197 measured reflections

4071 independent reflections  
 2711 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.033$   
 $\theta_{\text{max}} = 27.9^\circ$   
 $h = -13 \rightarrow 9$   
 $k = -12 \rightarrow 12$   
 $l = -21 \rightarrow 23$

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.056$   
 $wR(F^2) = 0.158$   
 $S = 1.02$   
 4071 reflections  
 201 parameters  
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0824P)^2 + 0.2134P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.53$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.42$  e Å<sup>-3</sup>

**Table 1**

Selected bond lengths (Å).

C1—S1	1.758 (3)	C15—S2	1.741 (3)
C8—S2	1.768 (3)	C15—S3	1.761 (2)
C16—N1	1.445 (3)	O1—S1	1.430 (2)
C16—S1	1.796 (3)	O2—S1	1.430 (2)
C15—N1	1.250 (3)		

**Table 2**

Hydrogen-bond geometry (Å, °).

D—H...A	D—H	H...A	D...A	D—H...A
S3—H1A...O1 <sup>i</sup>	1.13 (4)	2.68 (3)	3.219 (2)	108 (2)
C13—H13...O1 <sup>ii</sup>	0.93	2.66	3.420 (4)	139
C2—H2...Cg1 <sup>iii</sup>	0.93	3.02	3.717 (2)	133

Symmetry codes: (i)  $-x + 1, y - \frac{1}{2}, -z + \frac{5}{2}$ ; (ii)  $-x + 1, -y + 1, -z + 2$ ; (iii)  $x, y + 1, z$ . Cg1 is the centroid of the C8—C13 ring.

The H atom attached to S3 was located in a difference map and its coordinates were refined, while  $U_{\text{iso}}(\text{H})$  was fixed at  $1.5U_{\text{eq}}(\text{S})$ . Other H atoms were placed in calculated positions, with C–H = 0.96 (for CH<sub>3</sub>), 0.97 (for CH<sub>2</sub>) or 0.93 Å (for aromatic), and refined using a riding model, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

Data collection: *SMART-W2K/NT* (Bruker, 2003); cell refinement: *SAINT-W2K/NT* (Bruker, 2003); data reduction: *SAINT-W2K/NT*; program(s) used to solve structure: *SHELXTL-NT* (Bruker, 2003); program(s) used to refine structure: *SHELXTL-NT*; molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL-NT*.

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