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N-(*tert*-Butyl)-*S*-(4-methylphenyl)thiohydroxylamine

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.004 Å
R factor = 0.044
wR factor = 0.114
Data-to-parameter ratio = 16.2

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

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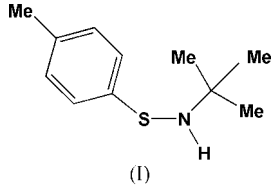
The title compound, $(CH_3)_3CN(H)–SC_6H_4CH_3$-4, is the first example of a simple alkylarylsulfenamide to be structurally characterized. Crystal packing consists of ribbons formed by N—H···S hydrogen bonds.

Comment

Sulfenamides, *R*N(H)–S*R*′ (*R*,*R*′ = alkyl or aryl), are compounds with simple structures yet remarkably rich chemistry. Their photolysis or chemical oxidation results in the formation of free radicals, some of which are persistent for long periods of time while others decay through complicated pathways (Miura, 1997). Deprotonation yields anions that act as ambidentate ligands towards transition (Hankin *et al.*, 1995, 1996*a*,*b*; Danopoulos *et al.*, 2000) and main-group metal ions (Mahmoudkhani *et al.*, 2003). There are only two compounds that have been structurally characterized previously, bearing the sulfenamide functional group as part of a more complex molecule (Lee *et al.*, 1995; Gotthardt *et al.*, 1987). However, the structures of the simplest members of the family were not examined until now, partly because of their low melting points. Highly pure (*p*-tolyl)(*tert*-butyl)sulfenamide, (I), solidifies just below room temperature, which allowed this study. The compound crystallizes in the monoclinic space group *C*2/*c* (No. 15).



(I)

The molecular structure is shown in Fig. 1. It contains an N atom as a chiral center, though the material is a racemic mixture. The C—N—S—C center adopts a *gauche* conformation with a torsion angle of −113.8 (2)°. The (N—)H atoms

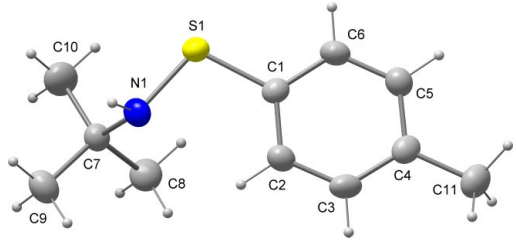


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

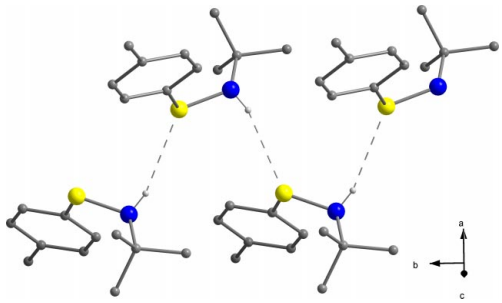


Figure 2
Representation of the ribbon structure of (I). The (C—)H atoms have been omitted for clarity.

are engaged in intermolecular hydrogen bonds with the S atoms, forming a ribbon, self-assembled along the *b* axis (Fig. 2).

Experimental

The synthesis of (I) was described previously (Mahmoudkhani *et al.*, 2003). Crystals were grown from the melt upon standing at room temperature overnight.

Crystal data

$C_{11}H_{17}NS$
$M_r = 195.32$
Monoclinic, $C2/c$
$a = 24.604\ (15)$ Å
$b = 6.267\ (4)$ Å
$c = 16.533\ (10)$ Å
$\beta = 115.533\ (9)^\circ$
$V = 2300\ (2)$ Å^3
$Z = 8$
$D_x = 1.128$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 833 reflections
$\theta = 1$–24°
$\mu = 0.24$ mm^{-1}
$T = 173\ (2)$ K
Needle, colorless
0.40 × 0.12 × 0.02 mm

Data collection

Bruker *P*4 CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
$T_{min} = 0.910$, $T_{max} = 0.996$
8741 measured reflections
2108 independent reflections
1359 reflections with $I > 2\sigma(I)$
$R_{int} = 0.066$
$\theta_{max} = 25.4^\circ$
$h = -27 \rightarrow 29$
$k = -7 \rightarrow 7$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.114$
$S = 1.01$
2108 reflections
130 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0538P)^2 + 0.5048P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.008$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—N1	1.694 (2)	N1—C7	1.486 (3)
S1—C1	1.776 (3)	N1—H1	0.82 (2)
N1—S1—C1	103.57 (11)	C7—N1—H1	111.4 (16)
C7—N1—S1	118.43 (16)	S1—N1—H1	110.4 (16)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···S1^{i}	0.82 (2)	2.75 (2)	3.523 (3)	159 (2)

Symmetry code: (i) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$.

The (N—)H atom was located from a difference Fourier map, whereas the (C—)H atoms were placed 0.98 Å from the parent atom with bond angles constrained to idealized values using the appropriate riding model and refined isotropically; U_{iso} values are in the range 0.020–0.073 Å^2.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART* and *SAINT* (Bruker, 1997); data reduction: *SAINT* and *SADABS* (Sheldrick, 2001); program(s) used to solve structure: *SHELXTL* (Bruker, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *DIAMOND* (Brandenburg, 2001); software used to prepare material for publication: *SHELXTL*.

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