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tert-Butylimido-λ^4-thio(phenylthio)methyl *p*-Tolyl Sulfone

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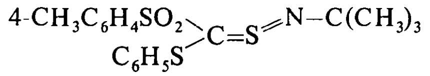
Abstract. $C_{18}H_{21}NO_2S_3$, M_r = 379·5, monoclinic, $P2_1/n$, a = 12·275 (4), b = 11·010 (6), c = 14·722 (5) Å, β = 96·37 (2)°, V = 1977·5 Å^3, Z = 4, D_c = 1·28 g cm^{-3}. Full-matrix least-squares refinement gave R = 0·051 and R_w = 0·053 for 217 parameters and 1410 significant reflexions [$I > 3\sigma(I)$]. C=S and S=N distances are 1·662 (7) and 1·534 (6) Å respectively; the mean C—S distance is 1·733 (7) Å.

Introduction. Holm, Boerma, Nilsson & Senning (1976) showed that crystalline α-chlorosulfenyl chlorides react with *tert*-butylamine to give thione-*S*-imides. The title compound with the formula



$$\begin{matrix}4\text{-}CH_3C_6H_4SO_2 \\ C_6H_5S\end{matrix}\!\!>\!C{=}S{=}N{-}C(CH_3)_3$$

was obtained by this reaction as yellow crystals with m.p. 118–120 °C. The crystals were kindly provided by A. Senning.

Table 1. *Fractional atomic coordinates* (×10^4) *and their standard deviations*

	x	y	z
S(1)	3115 (1)	4740 (2)	5684 (1)
S(2)	1830 (1)	4598 (2)	7275 (1)
S(3)	4032 (1)	3660 (2)	7498 (1)
O(1)	1516 (4)	5828 (4)	7049 (4)
O(2)	1994 (4)	4248 (5)	8223 (3)
N	5059 (4)	3625 (5)	6991 (4)
C(1)	3036 (4)	4294 (5)	6799 (4)
C(2)	6053 (5)	3015 (7)	7427 (5)
C(3)	6022 (6)	2696 (8)	8419 (6)
C(4)	6975 (6)	3855 (10)	7321 (7)
C(5)	6141 (8)	1836 (9)	6905 (7)
C(6)	850 (4)	3633 (5)	6677 (3)
C(7)	924 (5)	2391 (6)	6820 (5)
C(8)	199 (6)	1625 (7)	6355 (5)
C(9)	4330 (6)	2910 (7)	10716 (5)
C(10)	−708 (6)	3342 (8)	5592 (5)
C(11)	36 (6)	4092 (6)	6069 (5)
C(12)	3508 (6)	3760 (8)	10174 (5)
C(13)	3599 (4)	3428 (5)	5160 (4)
C(14)	3358 (5)	2253 (7)	5414 (5)
C(15)	3759 (6)	1274 (7)	4967 (5)
C(16)	4376 (7)	1461 (10)	4269 (6)
C(17)	4605 (7)	2619 (12)	4007 (6)
C(18)	4217 (6)	3610 (8)	4453 (5)

A crystal of dimensions 1·0 × 0·6 × 0·3 mm was mounted about *b*. Lattice type and space group were established from photographs taken with Cu and Mo radiation.

Intensities were measured out to $2\theta_{\max}$ = 40° with a Picker FACS-1 diffractometer. Monochromatic Mo $K\alpha$ radiation was used with a scintillation counter and a pulse-height analyzer. 1410 independent reflexions with $I > 3\sigma(I)$, according to counting statistics, were used in the subsequent calculations. No corrections were made for absorption.

The structure was determined with *MULTAN* (Germain, Main & Woolfson, 1971). Coordinates, anisotropic thermal parameters and a scale factor were refined by full-matrix least-squares calculations. The calculated positions of 18 H atoms were included in the structure-factor calculations but their coordinates and thermal parameters were not refined. The final R = 0·051 and R_w = 0·053 for 217 parameters. Atomic coordinates are listed in Table 1.*

Discussion. Bond distances are given in Table 2, and angles in Table 3. The numbering of atoms is shown in Fig. 1.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33699 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

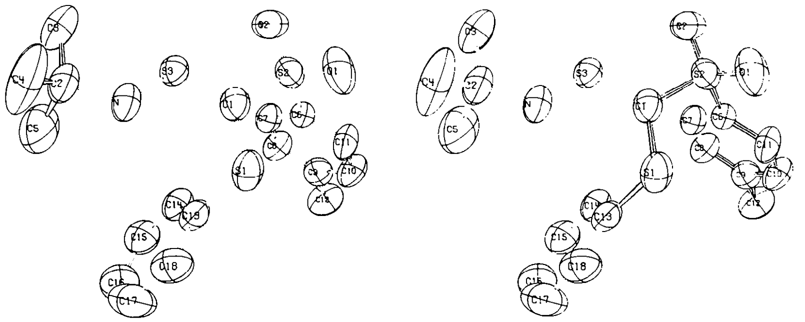


Fig. 1. A stereoscopic drawing of the molecule showing the numbering of the atoms.

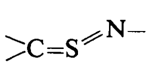
Table 2. *Bond lengths and their estimated standard deviations* (Å)

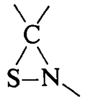
C(1)–S(1)	1·727 (7)	C(6)–C(7)	1·386 (9)
C(1)–S(2)	1·739 (7)	C(7)–C(8)	1·356 (9)
C(1)–S(3)	1·662 (7)	C(8)–C(9)	1·435 (10)
S(1)–C(13)	1·770 (7)	C(9)–C(10)	1·391 (11)
S(2)–C(6)	1·765 (7)	C(10)–C(11)	1·366 (10)
S(2)–O(1)	1·437 (5)	C(6)–C(11)	1·363 (9)
S(2)–O(2)	1·440 (5)	C(9)–C(12)	1·536 (10)
S(3)–N	1·534 (6)	C(13)–C(14)	1·388 (10)
N–C(2)	1·476 (9)	C(14)–C(15)	1·382 (10)
C(2)–C(3)	1·507 (11)	C(15)–C(16)	1·359 (12)
C(2)–C(4)	1·483 (11)	C(16)–C(17)	1·370 (13)
C(2)–C(5)	1·517 (12)	C(17)–C(18)	1·384 (12)
		C(13)–C(18)	1·369 (10)

Table 3. *Angles and their estimated standard deviations* (°)

C(1)–S(1)–C(13)	103·9 (3)	S(2)–C(6)–C(7)	119·3 (5)
O(1)–S(2)–O(2)	118·8 (4)	S(2)–C(6)–C(11)	121·0 (6)
O(1)–S(2)–C(1)	107·9 (3)	C(7)–C(6)–C(11)	119·7 (7)
O(1)–S(2)–C(6)	107·5 (3)	C(6)–C(7)–C(8)	120·7 (7)
O(2)–S(2)–C(1)	107·8 (3)	C(7)–C(8)–C(9)	120·4 (7)
O(2)–S(2)–C(6)	109·7 (3)	C(8)–C(9)–C(10)	117·0 (7)
C(1)–S(2)–C(6)	104·3 (3)	C(8)–C(9)–C(12)	121·3 (7)
N–S(3)–C(1)	107·3 (3)	C(10)–C(9)–C(12)	121·6 (7)
S(3)–N–C(2)	118·9 (5)	C(9)–C(10)–C(11)	121·2 (7)
S(1)–C(1)–S(2)	117·9 (4)	C(6)–C(11)–C(10)	121·0 (7)
S(1)–C(1)–S(3)	126·4 (4)	S(1)–C(13)–C(14)	123·4 (6)
S(2)–C(1)–S(3)	115·6 (4)	S(1)–C(13)–C(18)	117·1 (6)
N–C(2)–C(3)	114·8 (7)	C(14)–C(13)–C(18)	119·5 (7)
N–C(2)–C(4)	105·9 (7)	C(13)–C(14)–C(15)	120·2 (7)
N–C(2)–C(5)	105·7 (6)	C(14)–C(15)–C(16)	119·9 (9)
C(3)–C(2)–C(4)	110·4 (7)	C(15)–C(16)–C(17)	120·3 (9)
C(3)–C(2)–C(5)	107·6 (8)	C(16)–C(17)–C(18)	120·5 (8)
C(4)–C(2)–C(5)	112·4 (9)	C(13)–C(18)–C(17)	119·7 (8)

It has been proposed (Holm, Boerma, Nilsson & Senning, 1976) that thione-*S*-imides have a bent structure (*A*) similar to the structure of thione-*S*-oxides but a ring structure (*B*) might also be possible.


(*A*)


(*B*)

This structure determination has established the bent structure for thione-*S*-imides which consequently may exist in geometrical-isomeric forms. A bent structure for thione-*S*-imides has also been proposed on the basis of analytical and spectral data (Tangerman & Zwanenburg, 1977).

The C=S and S=N distances are 1·662 and 1·534 Å, respectively. In the crystal structure determination of 2-*tert*-butylimido-λ^4-thio-3,4-dihydro-3,3,5,8-tetramethyl-1(2*H*)-naphthalenone (Schepper, Nielsen & Norrestam, 1978) the corresponding distances were found to be 1·646 and 1·558 Å.

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