

3-Tosyl-1,2,3-benzoxathiazole 2,2-Dioxide

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Abstract. $C_{13}H_{11}NO_5S_2$, $M_r = 325.35$, triclinic, $P\bar{1}$, $a = 7.686$ (2), $b = 9.748$ (2), $c = 10.610$ (2) Å, $\alpha = 74.91$ (1), $\beta = 75.99$ (2), $\gamma = 64.88$ (2)°, $V = 686.9$ (5) Å³, $Z = 2$, $D_x = 1.573$ g cm⁻³, Cu $K\alpha$ radiation, $\lambda = 1.5418$ Å, $\mu = 36.1$ cm⁻¹, $F(000) = 336$, $T = 293$ K. Final $R = 0.048$ for 1777 observed reflections. This compound represents a new type of heterocycle: a cyclic sulfamate fused to a benzene ring. The five-membered ring is non-planar and the O–S–N bond angle is 95.0 (2)°.

Experimental. The title compound (Fig. 1) was prepared by the dropwise addition of triethylamine and sulfuryl chloride to *N*-(2-hydroxyphenyl)-4-toluene-sulfonamide, all dissolved in CH_2Cl_2 at 195 K. The mixture was allowed to warm to room temperature, water added and the organic layer concentrated under reduced pressure to yield the final product. Recrystallization from ethanol produced yellow needles. Intensity data collected on Enraf–Nonius CAD-4 diffractometer, graphite monochromator. Crystal dimensions 0.20 × 0.25 × 0.30 mm. Cell parameters measured on the diffractometer using 25 reflections in

the 2θ range 20–40°. Reduced cell: $a = 7.686$ (2), $b = 9.512$ (2), $c = 10.610$ (2) Å, $\alpha = 94.08$ (1), $\beta = 104.01$ (2), $\gamma = 111.90$ (2)°; transformation matrix from the cell used: $(-1,0,0/1,-1,0/0,0,1)$. Range of indices $-8 \leq h \leq 8$, $-10 \leq k \leq 10$, $0 \leq l \leq 11$ ($\theta < 60^\circ$). Three standards 04 $\bar{3}$, 330, $\bar{1}34$ measured after every 200 reflections showed a variation of 0.1%. Lorentz–polarization, semi-empirical absorption corrections (max. and min. corrections 0.992, 0.652) (North, Phillips & Mathews, 1968). 2064 unique reflections measured; 1777 observed reflections with $I > 3.0\sigma(I)$. Direct methods (*MULTAN*11/82; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) were used for structure determination. H atoms located by difference Fourier synthesis. Anisotropic full-matrix least-squares refinement (on F) for non-H atoms, isotropic for H atoms. $\sum w(|F_o| - |F_c|)^2$ minimized. $R = 0.048$, $wR = 0.048$, max. $\Delta/\sigma = 0.94$. Max. peak height in the final difference

Table 1. Fractional coordinates and equivalent isotropic temperature factors for non-H atoms with e.s.d.'s in parentheses

$$B_{eq} = \frac{1}{3} \sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	B_{eq} (Å ²)
S(1)	0.3601 (1)	0.0018 (1)	0.2518 (1)	3.28 (2)
S(2)	0.5568 (1)	0.2141 (1)	0.1240 (1)	4.10 (2)
O(1)	0.5024 (4)	0.3374 (3)	-0.0058 (3)	5.0 (1)
O(2)	0.7545 (4)	0.1177 (4)	0.1006 (3)	6.2 (1)
O(3)	0.4870 (4)	0.2904 (3)	0.2343 (3)	6.0 (1)
O(4)	0.2916 (4)	-0.0835 (3)	0.1991 (3)	3.9 (1)
O(5)	0.5326 (4)	-0.0696 (3)	0.3112 (3)	4.8 (1)
N(3)	0.4128 (4)	0.1260 (3)	0.1182 (3)	3.0 (1)
C(4)	0.2853 (5)	0.2211 (4)	0.0237 (3)	2.8 (1)
C(5)	0.1325 (5)	0.2030 (4)	-0.0057 (4)	3.4 (1)
C(6)	0.0352 (6)	0.3160 (5)	-0.1057 (4)	4.4 (1)
C(7)	0.0894 (7)	0.4349 (5)	-0.1717 (4)	4.6 (1)
C(8)	0.2441 (7)	0.4498 (5)	-0.1422 (4)	4.5 (1)
C(9)	0.3367 (6)	0.3409 (4)	-0.0447 (4)	3.6 (1)
C(10)	0.1691 (5)	0.1149 (4)	0.3567 (3)	3.1 (1)
C(11)	0.2137 (5)	0.1674 (4)	0.4501 (4)	3.8 (1)
C(12)	0.0639 (6)	0.2583 (5)	0.5299 (4)	4.1 (1)
C(13)	-0.1270 (6)	0.2988 (4)	0.5200 (4)	3.6 (1)
C(14)	-0.1676 (5)	0.2394 (4)	0.4288 (4)	3.6 (1)
C(15)	-0.0209 (5)	0.1484 (4)	0.3467 (3)	3.3 (1)
C(16)	-0.2871 (7)	0.4029 (5)	0.6067 (5)	5.2 (1)

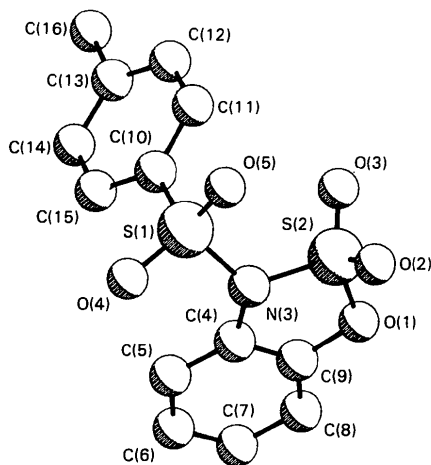


Fig. 1. Numbering of atoms and conformation of the molecule.

Fourier map $0.36 \text{ e} \text{ \AA}^{-3}$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Enraf-Nonius *SDP* (Frenz, 1984). Atomic parameters are given in Table 1; * bond distances, bond angles and relevant torsion angles are in Table 2.

Related literature. The O—S—N bond angle is in good agreement with those found in cyclic sulfates (Boer & Flynn, 1969; Boer, Flynn, Kaiser, Zaborsky, Tomalia, Young & Tong, 1968) and considerably smaller than that found in oxathiazines (Jansing & White, 1978; Mehdi & Rao, 1984).

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51473 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond distances (\AA), bond angles ($^\circ$) and selected torsion angles ($^\circ$); *e.s.d.*'s in parentheses

S(1)	O(4)	1.428 (4)	C(5)	C(6)	1.405 (5)		
S(1)	O(5)	1.425 (3)	C(6)	C(7)	1.362 (7)		
S(1)	N(3)	1.698 (3)	C(7)	C(8)	1.372 (8)		
S(1)	C(10)	1.755 (3)	C(8)	C(9)	1.359 (5)		
S(2)	O(1)	1.583 (3)	C(10)	C(11)	1.392 (7)		
S(2)	O(2)	1.407 (3)	C(10)	C(15)	1.380 (6)		
S(2)	O(3)	1.421 (3)	C(11)	C(12)	1.372 (5)		
S(2)	N(3)	1.684 (4)	C(12)	C(13)	1.372 (6)		
O(1)	C(9)	1.416 (6)	C(13)	C(14)	1.400 (7)		
N(3)	C(4)	1.430 (4)	C(13)	C(16)	1.506 (5)		
C(4)	C(5)	1.374 (7)	C(14)	C(15)	1.376 (5)		
C(4)	C(9)	1.364 (5)					
O(4)	S(1)	O(5)	122.0 (2)	C(5)	C(4)	C(9)	120.1 (3)
O(4)	S(1)	N(3)	103.9 (2)	C(4)	C(5)	C(6)	115.8 (4)
O(4)	S(1)	C(10)	109.5 (2)	C(5)	C(6)	C(7)	122.7 (5)

Table 2 (cont.)

O(5)	S(1)	N(3)	103.7 (2)	C(6)	C(7)	C(8)	120.7 (4)		
O(5)	S(1)	C(10)	109.8 (2)	C(7)	C(8)	C(9)	116.4 (4)		
N(3)	S(1)	C(10)	106.5 (1)	O(1)	C(9)	C(4)	112.5 (3)		
O(1)	S(2)	O(2)	108.4 (2)	O(1)	C(9)	C(8)	123.2 (4)		
O(1)	S(2)	O(3)	109.2 (2)	C(4)	C(9)	C(8)	124.3 (5)		
O(1)	S(2)	N(3)	95.0 (2)	S(1)	C(10)	C(11)	118.7 (3)		
O(2)	S(2)	O(3)	119.4 (2)	S(1)	C(10)	C(15)	119.9 (3)		
O(2)	S(2)	N(3)	111.2 (2)	C(11)	C(10)	C(15)	121.3 (3)		
O(3)	S(2)	N(3)	110.8 (2)	C(10)	C(11)	C(12)	118.5 (4)		
S(2)	O(1)	C(9)	111.2 (2)	C(11)	C(12)	C(13)	122.0 (5)		
S(1)	N(3)	S(2)	120.4 (2)	C(12)	C(13)	C(14)	118.2 (3)		
S(1)	N(3)	C(4)	124.7 (3)	C(12)	C(13)	C(16)	120.3 (5)		
S(2)	N(3)	C(4)	108.6 (2)	C(14)	C(13)	C(16)	121.5 (4)		
N(3)	C(4)	C(5)	129.2 (3)	C(13)	C(14)	C(15)	121.3 (4)		
N(3)	C(4)	C(9)	110.6 (4)	C(10)	C(15)	C(14)	118.6 (4)		
N(3)	S(2)	O(1)	C(9)	13.6 (3)	N(3)	C(4)	C(9)	O(1)	1.0 (4)
O(1)	S(2)	N(3)	C(4)	-12.9 (2)	C(10)	S(1)	N(3)	S(2)	82.0 (2)
S(2)	O(1)	C(9)	C(4)	-10.8 (4)	N(3)	S(1)	C(10)	C(11)	-88.7 (3)
S(2)	N(3)	C(4)	C(9)	8.4 (4)					

References

- BOER, F. P. & FLYNN, J. J. (1969). *J. Am. Chem. Soc.* **91**, 6604–6609.
- BOER, F. P., FLYNN, J. J., KAISER, E. T., ZABORSKY, O. R., TOMALIA, D. A., YOUNG, A. E. & TONG, Y. C. (1968). *J. Am. Chem. Soc.* **90**, 2970–2971.
- FRENZ, B. A. (1984). *Structure Determination Package*. College Station, Texas, USA, and Enraf-Nonius, Delft, The Netherlands. *International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- JANSING, J. A. & WHITE, J. G. (1978). *Acta Cryst.* **B34**, 2376–2378.
- MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1982). *MULTAN11/82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- MEHDI, S. & RAO, B. R. (1984). *Acta Cryst.* **C40**, 1057–1059.
- NORTH, A. C. T., PHILLIPS, D. C. & MATHEWS, F. S. (1968). *Acta Cryst.* **A24**, 351–359.