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Structure of a Tosyl Derivative of Morpholine

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Abstract. 4-Methylphenyl morpholino sulfone, $C_{11}H_{15}NO_3S$, $M_r = 241{\cdot}3$, monoclinic, $P2_1/c$, $a = 8{\cdot}0230\,(9)$, $b = 18{\cdot}476\,(2)$, $c = 8{\cdot}8106\,(9)$ Å, $\beta = 112{\cdot}320\,(8)°$, $V = 1208{\cdot}2\,(2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}33$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 2{\cdot}5$ cm^{-1}, $F(000) = 512$, $T = 293\,(2)$ K, $R(F) = 0{\cdot}049$ for 1192 independent reflections with $I > 3\sigma(I)$. The molecular geometry is nearly identical to that previously reported for 4-aminophenyl morpholino sulfone; however, the crystal packing in the present compound is different due to the absence of intermolecular hydrogen-bonding interactions.

Experimental. The compound was isolated as a by-product of a reaction of 2-(2-aminoethoxy)ethanol with tosyl chloride in dimethylformamide. The crystal used for data collection was a colorless columnar prism; approximate dimensions 0·15 × 0·20 × 0·40 mm. Unit-cell parameters were derived from a least-squares analysis of 25 reflections, in the range $19 < \theta < 22°$, automatically centered on a Rigaku AFC-5R X-ray diffractometer equipped with a 12 kW rotating anode Mo source and a graphite monochromator. Intensity data were collected with the $\omega/2\theta$-scan technique; scan speed 16° min^{-1} in ω; up to three identical scans for weak data. The intensities of three standards monitored at 150 reflection intervals throughout data collection were constant within intensity statistics. A total of 2378 X-ray diffraction intensities measured to $(\sin\theta)_{\max}/\lambda = 0{\cdot}5947$ Å^{-1} in the range $0 \le h \le 10$, $0 \le k \le 22$, $-10 \le l \le 10$, were averaged to yield 2217 unique data including those with $F_o^2 \le 0$ [$R_{\rm int}(F^2) = 0{\cdot}028$]. No absorption correction was made. The structure was solved with the direct-methods program *MITHRIL* (Gilmore, 1983). The S, N, O, and phenyl C atoms were located from an E map. The remaining non-H atoms were found on an electron density map produced by the program *DIRDIF* (Beurskens *et al.*, 1984). All calculations were performed on a DEC MicroVAXII computer using the *TEXSAN* series of programs (Molecular Structure Corporation, 1987). Scattering factors and anomalous-dispersion terms were taken from standard sources (Cromer & Waber, 1974; Cromer & Ibers, 1974). H atoms were located on a difference electron density map calculated using 2133 observations with $I > 0$. They were included in subsequent refinements as fixed isotropic scatterers with thermal parameters equal to 1·2 times the $B_{\rm eq}$ of the attached C atom. The final refinement on F_o, based on 146 variables, including anisotropic thermal parameters for all non-H atoms, and 1192 observations [$I > 3\sigma(I)$], resulted in residuals $R = 0{\cdot}049$ and $wR = 0{\cdot}065$. The weights are given by $1/\sigma^2(F_o)$ where $\sigma^2(F_o) = 1/(4F_o^2)[C + \frac{1}{4}(t_c/t_b)^2(b_1 + b_2) + (pI)^2]$ {C = total counts, t_c = time spent counting peak intensity, t_b = time spent counting one side of background, b_1 and b_2 = high- and low-angle background counts, $p = 0{\cdot}05$, I = intensity $[C - \frac{1}{2}(t_c/t_b)(b_1 + b_2)]$}. The largest Δ/σ value in the final cycle is 0·03 while the extreme peaks in the final difference electron density map are 0·16 and −0·21 e Å^{-3}. The error in an observation of unit weight is 1·60.[*]

Final atomic fractional coordinates and equivalent isotropic thermal parameters for the non-H atoms are listed in Table 1. Bond distances and angles appear in Table 2. Fig. 1 shows an *ORTEP* diagram of the molecule with the labeling scheme. A stereo diagram of the unit cell showing the molecular packing is given in Fig. 2.

Related literature. The metrical data presented in Table 2 are entirely regular and in excellent agreement with those of the related compound 4-aminophenyl morpholino sulfone (Gridunova, Lindeman, Shklover, Struchkov & Chayanov, 1984). The molecule displays the same stereochemistry in which the morpholine ring adopts a chair conformation with the morpholino O atom pointing towards the phenyl ring. The crystal structure of the 4-methylphenyl compound is distinguished by a markedly different packing scheme. The 4-aminophenyl morpholino sulfone molecules are oriented head-to-tail, affording close (*i.e.* hydrogen-bonding) contacts between the amino protons and the sulfonyl and morpholino O atoms of neighboring molecules.

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[*] Lists of structure amplitudes, anisotropic thermal parameters, and calculated H-atom positions have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51980 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Positional parameters and equivalent isotropic thermal parameters for 4-methylphenyl morpholino sulfone*

$B_{eq} = \frac{4}{3}[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + (2ab\cos\gamma)\beta_{12} + (2ac\cos\beta)\beta_{13} + (2bc\cos\alpha)\beta_{23}]$.

	x	y	z	B_{eq}(Å^2)
S	0·7936 (1)	0·13250 (7)	0·9038 (1)	4·88 (5)
O(1)	0·6438 (4)	0·1722 (2)	0·9070 (4)	6·9 (2)
O(2)	0·8702 (4)	0·0766 (2)	1·0219 (4)	6·7 (2)
O(3)	1·2212 (6)	0·2817 (3)	0·8964 (4)	8·5 (2)
N	0·9550 (4)	0·1919 (2)	0·9301 (4)	4·4 (1)
C(1)	0·7369 (5)	0·0954 (2)	0·7064 (5)	3·9 (2)
C(2)	0·6107 (6)	0·1295 (3)	0·5736 (6)	5·3 (2)
C(3)	0·5726 (7)	0·1020 (3)	0·4193 (6)	6·2 (2)
C(4)	0·6575 (7)	0·0408 (3)	0·3939 (6)	5·4 (2)
C(5)	0·7833 (7)	0·0079 (3)	0·5294 (7)	5·8 (2)
C(6)	0·8247 (6)	0·0343 (2)	0·6853 (6)	5·0 (2)
C(7)	0·6139 (8)	0·0114 (4)	0·2238 (7)	8·9 (3)
C(8)	1·1288 (6)	0·1610 (3)	0·9408 (6)	5·6 (2)
C(9)	1·2696 (6)	0·2195 (4)	1·0026 (7)	7·5 (3)
C(10)	1·057 (1)	0·3098 (3)	0·8910 (7)	7·8 (3)
C(11)	0·9030 (6)	0·2566 (3)	0·8232 (6)	5·5 (2)

Table 2. *Bond distances* (Å) *and angles* (°) *for 4-methylphenyl morpholino sulfone*

S	O(1)	1·417 (3)	C(1)	C(6)	1·381 (5)
S	O(2)	1·429 (3)	C(2)	C(3)	1·374 (6)
S	N	1·645 (3)	C(3)	C(4)	1·381 (7)
S	C(1)	1·760 (4)	C(4)	C(5)	1·378 (7)
O(3)	C(10)	1·404 (7)	C(4)	C(7)	1·505 (7)
O(3)	C(9)	1·440 (6)	C(5)	C(6)	1·375 (6)
N	C(8)	1·476 (5)	C(8)	C(9)	1·508 (7)
N	C(11)	1·481 (5)	C(10)	C(11)	1·510 (7)
C(1)	C(2)	1·375 (5)			

O(1)	S	O(2)	119·5 (2)	C(6)	C(1)	S	119·8 (3)
O(1)	S	N	106·3 (2)	C(3)	C(2)	C(1)	119·2 (4)
O(1)	S	C(1)	108·5 (2)	C(2)	C(3)	C(4)	121·7 (4)
O(2)	S	N	106·4 (2)	C(5)	C(4)	C(3)	117·7 (4)
O(2)	S	C(1)	108·5 (2)	C(5)	C(4)	C(7)	121·4 (5)
N	S	C(1)	107·0 (2)	C(3)	C(4)	C(7)	120·9 (5)
C(10)	O(3)	C(9)	109·6 (4)	C(6)	C(5)	C(4)	121·9 (4)
C(8)	N	C(11)	112·5 (3)	C(5)	C(6)	C(1)	118·9 (4)
C(8)	N	S	115·2 (3)	N	C(8)	C(9)	107·9 (4)
C(11)	N	S	115·6 (3)	O(3)	C(9)	C(8)	110·9 (4)
C(2)	C(1)	C(6)	120·6 (4)	O(3)	C(10)	C(11)	112·8 (5)
C(2)	C(1)	S	119·5 (3)	N	C(11)	C(10)	106·5 (4)

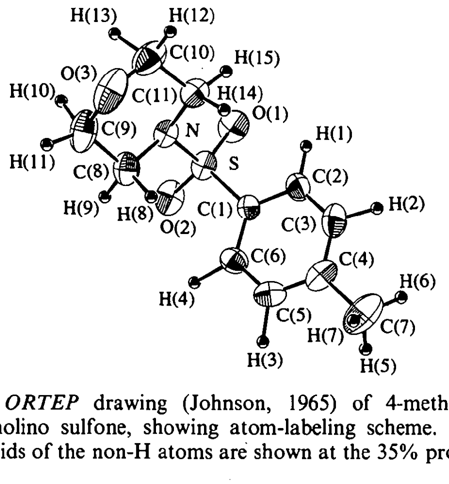

Fig. 1. *ORTEP* drawing (Johnson, 1965) of 4-methylphenyl morpholino sulfone, showing atom-labeling scheme. Thermal ellipsoids of the non-H atoms are shown at the 35% probability level.

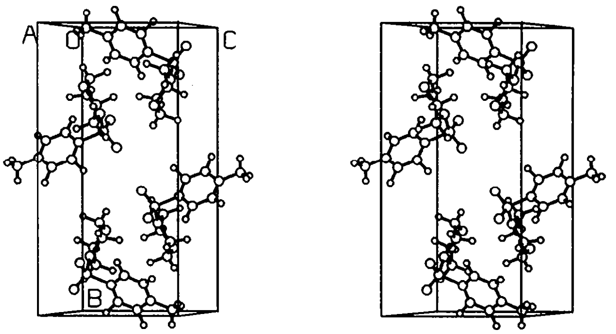

Fig. 2. Stereo packing diagram of the unit cell of 4-methylphenyl morpholino sulfone.

The result is a non-centrosymmetric (space group $P2_1$, $Z = 2$) arrangement in which the phenyl rings are face-to-face along the [100] direction. In the absence of such energetically favorable hydrogen-bonding interactions, the 4-methylphenyl morpholino sulfone molecules pack in a centrosymmetric arrangement ($P2_1/c$, $Z = 4$) with the morpholine rings face-to-face and the phenyl rings pointing away from each other (Fig. 2).

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