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Structure of 2-Methoxy-3-methyl-3-phenylcyclobutanol *p*-Toluenesulfonate

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Abstract. $C_{19}H_{22}O_4S$, $M_r = 346{\cdot}45$, monoclinic, $C2/c$, $a = 25{\cdot}032\ (4)$, $b = 7{\cdot}9807\ (7)$, $c = 22{\cdot}073\ (3)$ Å, $\beta = 124{\cdot}926\ (8)°$, $V = 3615{\cdot}4\ (7)$ Å^3, $Z = 8$, $D_x = 1{\cdot}273$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 1{\cdot}88$ cm^{-1}, $F(000) = 1472$, $T = 298\ (2)$ K, $R = 0{\cdot}048$ for 2584 unique observed reflections. The methoxy and methyl substituents to the cyclobutane ring are *trans* and diaxial. The phenyl and toluenesulfonate substituents are *cis* and diequatorial. The puckering angle of the cyclobutane as described by the dihedral angle across the molecule is 152·1 (4)°. The endocyclic torsion angles in the four-membered ring are sequentially 19·9 (1), −19·4 (1), 19·9 (1) and −19·9 (1)°. Endocyclic cyclobutane bond angles are 89·7 (2), 87·0 (1), 88·2 (2) and 88·1 (2)°. Of the four C—C bond lengths in the cyclobutane ring, three are nearly equal [1·547 (3) Å ave.] while one is significantly shorter [1·511 (3) Å]. Presumably this short bond length is due to torsional strain in the more substituted portion of the ring.

Experimental. The title compound is a tosylate derivative of a product generated from the intramolecular rearrangement of an oxonium ylide. The structure determination was undertaken to determine the relative configuration of the methoxy group (Roskamp & Johnson, 1986). Colorless square rods, 0·36 × 0·48 × 0·36 mm, Nicolet *R*3 diffractometer, lattice parameters from 25 high-angle reflections ($2\theta > 20°$) constrained monoclinic, no absorption corrections or corrections for secondary extinction, $\theta/2\theta$ scans, $3 \le 2\theta \le 52°$, $0 \le h \le 31$, $0 \le k \le 10$, $-28 \le l \le 28$, three standard reflections fluctuated 2%, 4074 total reflections, 3583 unique, 2584 observed with $I_o \ge 2\sigma(I)$, direct-methods *SHELXTL* (Sheldrick, 1978), full-matrix refinement *via SHELX*76 (Sheldrick, 1976) on *F*'s minimizing $\sum w(F_o - |F_c|)^2$, all non-hydrogen atoms anisotropic, H atoms placed in observed positions and refined isotropically. For observed reflections $R = 0{\cdot}048$, $wR = 0{\cdot}024$, $S = 3{\cdot}8$, $w = (\sigma_F)^{-2}$. Including unobserved reflections: $R = 0{\cdot}069$, $wR = 0{\cdot}024$. In a

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters*

$$U_{\text{eq}} = \tfrac{1}{3}\textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j .$$

	x	y	z	U_{eq}(Å^2)
S1	0·42353 (4)	0·53084 (8)	0·08681 (5)	0·0680 (5)
O1	0·45267 (8)	0·3821 (2)	0·1290 (1)	0·093 (2)
O2	0·36408 (8)	0·5202 (2)	0·0144 (1)	0·090 (1)
O3	0·41299 (7)	0·6439 (2)	0·13760 (8)	0·065 (1)
O4	0·32575 (7)	0·7360 (2)	0·17049 (7)	0·0474 (9)
C1	0·4806 (1)	0·6439 (3)	0·0814 (1)	0·049 (1)
C2	0·5389 (1)	0·6912 (3)	0·1447 (1)	0·059 (2)
C3	0·5847 (1)	0·7743 (3)	0·1403 (2)	0·061 (2)
C4	0·5735 (1)	0·8094 (3)	0·0729 (1)	0·057 (2)
C5	0·5145 (1)	0·7642 (4)	0·0102 (2)	0·070 (2)
C6	0·4681 (1)	0·6822 (4)	0·0139 (1)	0·066 (2)
C7	0·6247 (2)	0·8952 (6)	0·0683 (3)	0·090 (3)
C8	0·3785 (1)	0·8009 (3)	0·1079 (1)	0·052 (2)
C9	0·3200 (1)	0·8268 (3)	0·1128 (1)	0·041 (1)
C10	0·34347 (9)	1·0098 (2)	0·1354 (1)	0·042 (1)
C11	0·4113 (1)	0·9528 (3)	0·1564 (2)	0·058 (2)
C12	0·3092 (1)	1·1226 (4)	0·0668 (2)	0·060 (2)
C13	0·3390 (1)	1·0882 (3)	0·1941 (1)	0·045 (1)
C14	0·2810 (1)	1·0824 (3)	0·1884 (1)	0·059 (2)
C15	0·2758 (2)	1·1644 (4)	0·2406 (2)	0·078 (2)
C16	0·3278 (2)	1·2511 (4)	0·2977 (2)	0·084 (3)
C17	0·3845 (2)	1·2586 (4)	0·3030 (2)	0·079 (2)
C18	0·3908 (1)	1·1781 (3)	0·2521 (1)	0·061 (2)
C19	0·2962 (2)	0·5754 (4)	0·1479 (2)	0·077 (3)

Table 2. *Bond lengths* (Å) *and angles* (°)

S1—O1	1·425 (2)	C5—C6	1·378 (3)
S1—O2	1·433 (2)	C8—C9	1·544 (3)
S1—O3	1·574 (1)	C8—C11	1·511 (3)
S1—C1	1·750 (2)	C9—C10	1·547 (3)
O3—C8	1·448 (3)	C10—C11	1·550 (3)
O4—C9	1·399 (2)	C10—C12	1·534 (3)
O4—C19	1·419 (4)	C10—C13	1·501 (2)
C1—C2	1·376 (3)	C13—C14	1·384 (3)
C1—C6	1·369 (2)	C13—C18	1·390 (3)
C2—C3	1·375 (3)	C14—C15	1·396 (3)
C3—C4	1·376 (2)	C15—C16	1·372 (5)
C4—C5	1·374 (4)	C16—C17	1·359 (5)
C4—C7	1·507 (5)	C17—C18	1·380 (2)

S1—O3—C8	118·2 (1)	C5—C4—C7	121·2 (2)
S1—C1—C2	120·3 (1)	C8—C9—C10	87·0 (2)
S1—C1—C6	120·1 (2)	C8—C11—C10	88·1 (2)
O1—S1—O2	119·7 (1)	C9—O4—C19	112·8 (1)
O1—S1—O3	104·10 (8)	C9—C8—C11	89·7 (2)
O1—S1—C1	110·0 (1)	C9—C10—C11	88·2 (2)
O2—S1—O3	109·75 (9)	C9—C10—C12	110·0 (2)
O2—S1—C1	108·74 (9)	C9—C10—C13	118·0 (1)
O3—S1—C1	103·25 (9)	C10—C13—C14	120·0 (2)
O3—C8—C9	115·4 (2)	C10—C13—C18	121·2 (2)
O3—C8—C11	115·6 (2)	C11—C10—C12	111·4 (1)
O4—C9—C8	112·7 (2)	C11—C10—C13	118·4 (2)
O4—C9—C10	111·9 (1)	C12—C10—C13	109·4 (2)
C1—C2—C3	120·2 (2)	C13—C14—C15	119·8 (2)
C1—C6—C5	119·7 (2)	C13—C18—C17	120·6 (2)
C2—C1—C6	119·5 (2)	C14—C13—C18	118·6 (2)
C2—C3—C4	120·9 (2)	C14—C15—C16	120·5 (3)
C3—C4—C5	118·1 (2)	C15—C16—C17	119·8 (2)
C3—C4—C7	120·7 (2)	C16—C17—C18	120·6 (3)
C4—C5—C6	121·5 (2)		

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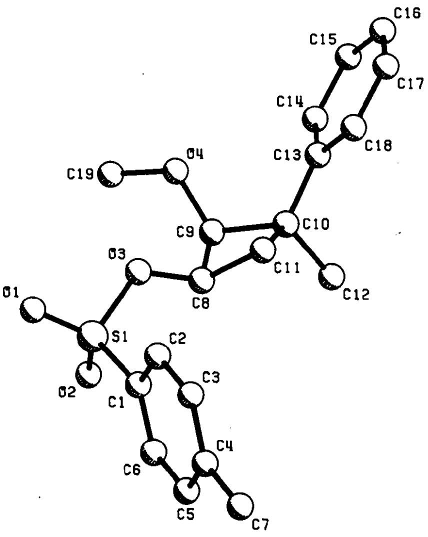

Fig. 1. A diagram of the title molecule.

final cycle $(\Delta/\sigma)_{\max} < 0{\cdot}1$ for non-hydrogen atoms, $(\Delta\rho)_{\max} = 0{\cdot}24$, $(\Delta\rho)_{\min} = -0{\cdot}19$ e Å^{-3}. Neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974). Table 1* gives the atomic coordinates and Table 2 lists bond lengths and angles. A view of the molecule is shown in Fig. 1.

Related literature. The synthesis has been published (Roskamp & Johnson, 1986). Similar cyclobutanesulfonates for structural comparisons have been published, *e.g.* Decesare, Corbel, Durst & Blount (1981) and Petty, Ikeda, Samuelson, Boriack, Onan, McPhail & Meinwald (1978). A discussion of the stereochemistry of cyclobutane has been given by Moriarty (1974).

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43967 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 2-Amino-5-{3-[3-(1-piperidinylmethyl)phenoxy]propylamino}-1,3,4-thiadiazole Hydrochloride (TAS)

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Abstract. $C_{17}H_{26}N_5OS^+.Cl^-$, $M_r = 383{\cdot}96$, monoclinic, *C*2/*c*, $a = 18{\cdot}050\,(4)$, $b = 22{\cdot}844\,(2)$, $c = 11{\cdot}783\,(2)$ Å, $\beta = 127{\cdot}33\,(1)°$, $V = 3863{\cdot}4\,(12)$ Å^3, $Z = 8$, $D_x = 1{\cdot}32$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}7107$ Å, $\mu = 0{\cdot}31$ mm^{-1}, $F(000) = 1632$, $T = 293$ K, final $R = 0{\cdot}053$ for 2832 unique observed reflections. The Cl atom is located with equal occupancy (0·5) at two special positions, and protonation occurs at the N atom

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