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Crystal Studies of Heterocyclic Compounds Containing One Oxygen and Two Nitrogen Atoms. IX. 2,7,8,9,10,15-Hexahydro-*N,N'*-ditosyldibenzo[*c,i*][1,5,8]oxadiazacycloundecene

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Abstract. $C_{30}H_{30}N_2O_5S_2$, $M_r = 562·7$, monoclinic, $A2/a$, $a = 9·607(1)$, $b = 13·368(2)$, $c = 21·293(3)$ Å, $\beta = 91·21(1)$°, $V = 2734·0(6)$ Å^3, $Z = 4$, $D_x = 1·3669(3)$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 20·06$ cm^{-1}, $F(000) = 1184$, room temperature, $R = 0·050$ for 2010 reflections with $I > 3\sigma(I)$. The molecule lies in a special position: the twofold axis runs through the O atom. The eleven-membered ring is in a quasi-chair conformation. The fused aromatic rings form a dihedral angle of 13·9 (3)° and the benzene rings of the tosyl groups form a dihedral angle of 15·2 (4)°. The sum of the angles at N is 351·8 (2)°.

Experimental. Light yellow prismatic crystals from ethanol, room temperature: crystal size 0·2 × 0·2 × 0·3 mm. CAD-4 diffractometer using θ–2θ scan technique; unit-cell parameters from 25 reflections in the θ range 8·5–40·7°; graphite-monochromatized Cu $K\alpha$ radiation, range of h, k and l 0 to 11, 0 to 15, −26 to 26 respectively; total of 2412 independent reflections measured to $(\sin\theta)/\lambda = 0·63$ Å^{-1}, data not corrected for absorption, $R_{\rm int} = 0·0521$; standard reflection 355, maximum change 2·1%; 2010 reflections with $I > 3\sigma(I)$ used in calculations; solution by direct methods using *SHELX76* (Sheldrick, 1976), all H atoms located from a difference map, refinement by full-matrix least-squares procedure on F magnitudes (237 parameters)

Table 1. *Final fractional coordinates* (×10^4) *and equivalent isotropic temperature factors* (×10^4) *with e.s.d.'s in parentheses*

For non-H atoms $U_{\rm eq} = \frac{1}{3}(U_{11}+U_{22}+U_{33}U_{13}\cos\beta)$.

	x	y	z	$U_{\rm eq}$(Å^2)
O3	2500	9316 (2)	0	590 (22)
C1	2560 (4)	8703 (3)	550 (2)	539 (22)
C11	4164 (4)	7191 (2)	707 (1)	445 (18)
C12	3965 (4)	8223 (2)	636 (2)	482 (20)
C13	5156 (5)	8820 (3)	618 (2)	606 (24)
C14	6467 (5)	8435 (3)	682 (2)	672 (27)
C15	6651 (5)	7416 (3)	769 (2)	651 (27)
C16	5498 (4)	6800 (3)	788 (2)	538 (22)
N1	2988 (3)	6521 (2)	678 (1)	445 (15)
C2	3080 (4)	5649 (2)	248 (2)	450 (20)
S1	2090 (1)	6352 (1)	1322 (0)	479 (04)
O1	930 (2)	5733 (2)	1136 (1)	597 (15)
O2	1842 (3)	7324 (2)	1578 (1)	634 (17)
C21	3141 (4)	5690 (2)	1867 (1)	467 (19)
C22	3000 (4)	4664 (3)	1925 (2)	531 (22)
C23	3824 (4)	4159 (3)	2362 (2)	588 (24)
C24	4793 (4)	4656 (3)	2733 (2)	564 (22)
C25	4904 (5)	5692 (3)	2668 (2)	631 (25)
C26	4089 (4)	6203 (3)	2239 (2)	583 (24)
C241	5711 (6)	4099 (5)	3193 (2)	759 (32)

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

O3–C1	1·430 (4)	N1–C11	1·443 (4)
C1–C12	1·502 (5)	N1–C2	1·485 (4)
C11–C12	1·400 (4)	C21–C22	1·384 (5)
C12–C13	1·396 (5)	C22–C23	1·384 (5)
C13–C14	1·365 (6)	C23–C24	1·379 (5)
C14–C15	1·385 (6)	C24–C25	1·396 (5)
C15–C16	1·381 (5)	C25–C26	1·372 (5)
C11–C16	1·391 (5)	C21–C26	1·377 (5)
C2–C2$^{\rm i}$	1·520 (7)	C24–C241	1·502 (6)
S1–N1	1·649 (3)	Si–C21	1·761 (3)
S1–O1	1·437 (2)	S1–O2	1·432 (2)

C1–O3–C1$^{\rm i}$	110·0 (4)	O3–C1–C12	111·4 (3)
C1–C12–C11	123·6 (3)	C1–C12–C13	119·2 (3)
C11–C12–C13	117·1 (3)	C12–C13–C14	122·5 (4)
C13–C14–C15	119·9 (4)	C14–C15–C16	119·3 (4)
C11–C16–C15	120·6 (4)	C12–C11–C16	120·4 (3)
N1–C11–C12	120·2 (3)	N1–C11–C16	119·4 (3)
C2–N1–C11	117·1 (3)	N1–C2–C2$^{\rm i}$	112·1 (3)
C2–N1–S1	116·4 (2)	C11–N1–S1	118·2 (2)
N1–S1–C21	108·3 (1)	C21–S1–O1	108·8 (2)
N1–S1–O1	105·5 (1)	C21–S1–O2	107·6 (2)
N1–S1–O2	106·7 (1)	O1–S1–O2	119·6 (2)
S1–C21–C22	120·1 (3)	S1–C21–C26	119·5 (3)
C26–C21–C22	120·5 (4)	C21–C22–C23	119·2 (4)
C22–C23–C24	121·3 (4)	C23–C24–C25	118·2 (4)
C24–C25–C26	121·1 (4)	C21–C26–C25	119·7 (4)
C241–C24–C23	121·0 (4)	C241–C24–C25	120·7 (4)

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

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to final $R = 0.0501$, $S = 1.75$, unit weights; largest peak on final difference map 0·30 e Å^{-3}, ratio of max. shift/e.s.d. = 0·49, scattering factors from *SHELX*76. The geometry of the molecule was calculated using *ORFFE* (Busing, Martin & Levy, 1971). Atomic coordinates are given in Table 1, interatomic distances and angles in Table 2.* Atom-numbering scheme is shown in Fig. 1.

Related literature. In previous studies (Stępień, Wajsman, Grabowski, Glinka & Perrin, 1987; Olszak, Stępień, Wajsman, Grabowski, Glinka & Lecocq, 1987) were presented details of the properties and structures of related compounds.

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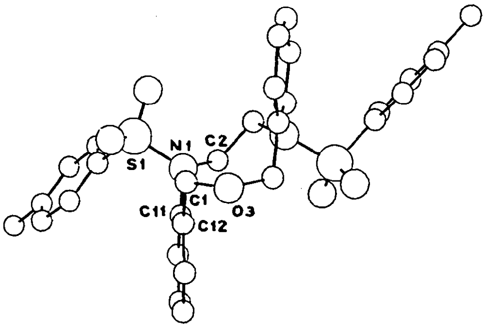

Fig. 1. The structure of the molecule with selected atom numbering.

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* Lists of structure factors, H-atom coordinates, thermal parameters and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51158 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Stiripentol

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Abstract. 4,4-Dimethyl-1-(3,4-methylenedioxyphenyl)-1-penten-3-ol, $C_{14}H_{18}O_3$, $M_r = 234.3$, monoclinic, $P2_1/c$, $a = 15.667$ (4), $b = 6.120$ (2), $c = 14.533$ (7) Å, $\beta = 116.39$ (3)°, $V = 1248$ (2) Å^3, $Z = 4$, $D_x = 1.247$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $\mu = 0.081$ mm^{-1}, $F(000) = 504$, $T = 294$ (1) K, $R = 0.032$ for 1240 independent reflections. The bond lengths and angles agree with corresponding values in related compounds. The nine-membered methylenedioxyphenyl ring is approximately planar. The dihedral angle between its least-squares plane and that of the ethylenic bond measures 11·0 (2)°. The molecules form layers parallel to the (100) face. Racemic arrangement within the crystal consists of 'dimers' in which the chiral molecules are linked together by two centrosymmetric weak O—H···O hydrogen bonds [3·115 (2) Å, 166 (2)°] or strong van der Waals interactions.

Partie expérimentale. Cristal parallélépipédique: 0,16 × 0,25 × 0,30 mm. Dimensions de la maille déterminées avec 25 réflexions telles que $5{,}80 \leq \theta \leq 13{,}40°$. Diffractomètre Enraf–Nonius CAD-4. $0{,}039 \leq (\sin\theta)/\lambda \leq 0{,}572$ Å^{-1}; $-17 \leq h \leq 16$, $0 \leq k \leq 6$, $0 \leq l \leq 16$. Réflexions de contrôle de l'intensité: 700, 006 et 020.

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