

$> 3\sigma(I)$. Structure solved by direct methods, using *RANTAN* (Yao, 1981), refined by full-matrix least-squares based upon F with weights $w = 4F_o^2[\sigma^2(I) + (0.02F_o^2)]^{-1}$ using the Enraf-Nonius package *SDP/VAX* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), anomalous coefficients of Cromer (1974). Non-H atoms refined anisotropically; H atoms located from difference maps and refined isotropically. Final $R = 0.040$ (0.047 for all data), $wR = 0.056$, $S = 3.057$ for 273 variables. Maximum shift 0.02σ in the final cycle, max. residual density 0.13, min. $-0.15 \text{ e } \text{\AA}^{-3}$, extinction coefficient (Larson, 1969) $g = 1.16(5) \times 10^{-6}$, where the correction factor $(1 + gI_c)^{-1}$ was applied to F_c ; maximum correction 33.8% for the 024 reflection.

Coordinates* and equivalent isotropic thermal parameters are given in Table 1; bond distances and angles are given in Table 2. The molecule is illustrated in Fig. 1, and the unit cell is shown in Fig. 2.

* Lists of H-atom positional parameters, bond distances and angles involving H, torsion angles, least-squares planes, anisotropic thermal parameters, and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54144 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. Structures with 2-arylbenzofuran skeleton: 2-(2'-hydroxy-4'-methoxyphenyl)benzofuran-6-ol: Jurd & Wong (1984); albanol A methyl ether: Rama Rao, Deshpande, Shastri, Tavale & Dhaneshwar (1983); daphnodorin A pentamethyl ether: Baba, Takeuchi, Doi, Inoue & Kozawa (1986).

References

- BABA, K., TAKEUCHI, K., DOI, M., INOUE, M. & KOZAWA, M. (1986). *Chem. Pharm. Bull.* **34**, 1540–1545.
- CROMER, D. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.3.1. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2B. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- FRENZ, B. A. & OKAYA, Y. (1980). *Enraf-Nonius Structure Determination Package*. Enraf-Nonius, Delft, The Netherlands.
- JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- JURD, L. & WONG, R. Y. (1984). *Aust. J. Chem.* **37**, 1127–1133.
- LARSON, A. C. (1969). *Crystallographic Computing*, edited by F. R. AHMED, S. R. HALL & C. P. HUBER, pp. 291–294. Copenhagen: Munksgaard.
- RAMA RAO, A. V., DESHPANDE, V. H., SHASTRI, R. K., TAVALE, S. S. & DHANESHWAR, N. N. (1983). *Tetrahedron Lett.* **24**, 3013–3016.
- YAO, J. (1981). *Acta Cryst.* **A37**, 642–644.

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Crystal Studies of Heterocyclic Compounds Containing One Oxygen and Two Nitrogen Atoms. XVI. 6,8-Ditosyl-6,7,8,9-tetrahydrobenzo[*b,h*][1,4,6]oxadiazonine

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Abstract. $\text{C}_{28}\text{H}_{26}\text{N}_2\text{O}_5\text{S}_2$, $M_r = 534.7$, orthorhombic, $P2_12_12_1$, $a = 8.939(1)$, $b = 11.67(1)$, $c = 24.968(2) \text{ \AA}$, $V = 2604.6(6) \text{ \AA}^3$, $Z = 4$, $D_x = 1.364(1) \text{ g cm}^{-3}$, $\lambda(\text{Cu } K\alpha) = 1.54178 \text{ \AA}$, $\mu = 20.82 \text{ cm}^{-1}$, $F(000) = 1120$, room temperature, $R = 0.0395$ for 3767 reflections with $I > 3\sigma(I)$. The nine-membered heterocyclic ring is in a chair-like conformation. There is an *exo, exo* conformation of tosyl groups.

Experimental. Colourless crystals $0.2 \times 0.3 \times 0.4 \text{ mm}$ from ethanol at room temperature. CAD-4 diffractometer using θ - 2θ scan technique, graphite-

monochromatized $\text{Cu } K\alpha$ radiation, data not corrected for absorption. Unit-cell parameters from 25 reflections in the θ range 16.4 – 45.0° . Total of 4558 independent reflections measured to $(\sin\theta)/\lambda = 0.63 \text{ \AA}^{-1}$, $R_{\text{int}} = 3.52\%$, range of h, k, l : -10 to $10, 0$ to $12, 0$ to 26 , respectively; standard reflection 1,3,10, max. change 2.4%, 3767 reflections with $I > 3\sigma(I)$ used in calculation. Solution by direct methods using *SHELX76* (Sheldrick, 1976), all H atoms located from a difference map, refinement by a full-matrix least-squares procedure on F magnitudes, 438 parameters. Refinement to final $R = 0.0395$, unit weights, $S = 0.880$. Absolute configuration of the

Table 1. Final fractional coordinates ($\times 10^4$) and isotropic temperature factors ($\text{\AA}^2 \times 10^4$) with e.s.d.'s in parentheses

$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
O1	-2608 (3)	849 (2)	1168 (1)	509 (7)
C2	-2050 (4)	1025 (3)	656 (1)	455 (11)
C3	-1079 (4)	1925 (3)	587 (1)	474 (10)
N4	-581 (3)	2553 (2)	1048 (1)	500 (9)
C5	836 (4)	2217 (3)	1297 (2)	548 (13)
N6	768 (3)	1004 (2)	1436 (1)	479 (9)
C7	-217 (4)	678 (4)	1893 (1)	570 (14)
C8	-1234 (4)	-312 (3)	1780 (1)	555 (12)
C9	-2384 (4)	-227 (3)	1407 (1)	513 (11)
C31	-499 (5)	2174 (4)	83 (2)	580 (14)
C32	-971 (5)	1512 (4)	-351 (1)	667 (16)
C33	-1944 (5)	629 (4)	-284 (1)	669 (16)
C34	-2522 (4)	377 (3)	222 (1)	572 (13)
C41	-3369 (5)	-1097 (4)	1321 (2)	654 (13)
C42	-3230 (6)	-2112 (4)	1597 (2)	821 (18)
C43	-2092 (6)	-2221 (4)	1974 (2)	856 (21)
C44	-1134 (5)	-1336 (4)	2058 (2)	758 (17)
S1	-1608 (1)	3595 (1)	1285 (0)	551 (3)
O11	-1158 (3)	3745 (3)	1834 (1)	779 (10)
O12	-3135 (3)	3340 (2)	1159 (1)	674 (10)
C11	-1099 (4)	4839 (3)	935 (1)	506 (12)
C12	-1622 (5)	4987 (3)	415 (1)	579 (13)
C13	-1225 (5)	5970 (3)	142 (2)	623 (15)
C14	-343 (5)	6809 (3)	376 (2)	707 (17)
C15	147 (6)	6624 (4)	899 (2)	850 (20)
C16	-220 (5)	5660 (4)	1171 (2)	719 (17)
C141	33 (10)	7874 (5)	66 (4)	1140 (33)
S2	2281 (1)	244 (1)	1336 (0)	504 (2)
O21	2948 (3)	662 (2)	849 (1)	681 (10)
O22	1865 (3)	-921 (2)	1382 (1)	670 (9)
C21	3550 (4)	545 (3)	1861 (1)	436 (10)
C22	3566 (4)	-156 (3)	2308 (2)	582 (14)
C23	4615 (5)	67 (4)	2711 (2)	686 (15)
C24	5602 (4)	957 (4)	2664 (2)	645 (15)
C25	5547 (4)	1657 (4)	2218 (2)	594 (13)
C26	4517 (4)	1448 (3)	1814 (1)	512 (12)
C241	6765 (8)	1165 (9)	3100 (3)	1043 (32)

Table 2. Interatomic distances (\AA) and bond angles ($^\circ$)

C2	C34	1.387 (4)	C9	C41	1.361 (6)		
C33	C34	1.396 (4)	C41	C42	1.376 (7)		
C32	C33	1.359 (7)	C42	C43	1.392 (7)		
C31	C32	1.396 (6)	C43	C44	1.358 (7)		
C3	C31	1.392 (6)	C8	C44	1.385 (6)		
N4	S1	1.635 (3)	N6	S2	1.637 (3)		
S1	O11	1.439 (3)	S2	O21	1.439 (3)		
S1	O12	1.432 (3)	S2	O22	1.414 (3)		
S1	C11	1.755 (3)	S2	C21	1.769 (3)		
C11	C12	1.391 (4)	C21	C22	1.384 (5)		
C11	C16	1.372 (6)	C21	C26	1.368 (5)		
C12	C13	1.381 (5)	C22	C23	1.400 (7)		
C13	C14	1.386 (6)	C23	C24	1.368 (6)		
C14	C15	1.394 (7)	C24	C25	1.382 (7)		
C15	C16	1.354 (7)	C25	C26	1.387 (5)		
C14	C141	1.502 (8)	C24	C241	1.525 (9)		
O1	C2	1.388 (4)	O1	C9	1.405 (4)		
C2	C3	1.373 (5)	C8	C9	1.391 (4)		
C3	N4	1.435 (4)	C7	N6	1.491 (4)		
N4	C5	1.465 (5)	C5	N6	1.459 (4)		
C8	C7	1.497 (6)					
O1	C9	C41	120.5 (3)	O1	C2	C34	122.0 (3)
C9	C41	C42	120.3 (4)	C2	C34	C33	118.7 (3)
C41	C42	C43	118.9 (4)	C32	C33	C34	120.5 (3)
C42	C43	C44	119.7 (5)	C31	C32	C33	121.2 (3)
C43	C44	C8	122.6 (4)	C3	C31	C32	118.3 (4)
C44	C8	C9	116.4 (3)	C2	C3	C31	120.6 (3)
C8	C9	C41	122.1 (3)	C3	C2	C34	120.7 (3)
C7	C8	C44	122.2 (3)	N4	C3	C31	120.2 (3)
S2	N6	C5	117.1 (2)	S1	N4	C5	122.1 (2)
S2	N6	C7	117.8 (2)	S1	N4	C3	119.7 (2)
N6	S2	O21	106.7 (1)	N4	S1	O11	106.2 (2)
N6	S2	O22	106.9 (2)	N4	S1	O12	107.5 (1)
N6	S2	C21	108.0 (1)	N4	S1	C11	106.8 (1)
C21	S2	O21	107.0 (2)	C11	S1	O11	107.5 (2)
C21	S2	O22	107.4 (1)	C11	S1	O12	108.1 (1)
O21	S2	O22	120.2 (1)	O11	S1	O12	120.1 (2)
S2	C21	C22	119.1 (3)	S1	C11	C12	118.7 (3)
S2	C21	C26	119.7 (2)	S1	C11	C16	120.8 (3)
C22	C21	C26	121.2 (3)	C12	C11	C16	120.5 (3)
C21	C22	C23	118.5 (4)	C11	C12	C13	118.5 (3)
C22	C23	C24	120.8 (4)	C12	C13	C14	121.7 (4)
C23	C24	C25	119.7 (4)	C13	C14	C15	117.7 (4)
C24	C25	C26	120.4 (4)	C14	C15	C16	121.5 (4)
C21	C26	C25	119.5 (3)	C11	C16	C15	120.2 (4)
C241	C24	C23	120.0 (5)	C141	C14	C13	119.6 (5)
C241	C24	C25	120.4 (5)	C141	C14	C15	122.7 (6)
O1	C9	C8	117.0 (3)	O1	C2	C3	117.1 (2)
C7	C8	C9	121.3 (3)	C2	C3	N4	119.1 (3)
N6	C7	C8	114.3 (2)	N4	C5	N6	109.0 (3)
C5	N6	C7	117.0 (3)	C3	N4	C5	118.2 (3)
C2	O1	C9	118.2 (3)				

Table 3. Selected torsion angles ($^\circ$)

C2	O1	C9	C8	100.8 (4)	C9	O1	C2	C3	-123.4 (3)
O1	C9	C8	C7	-4.1 (5)	O1	C2	C3	N4	6.2 (5)
C5	N6	C7	C8	131.9 (3)	C2	C3	N4	C5	94.4 (4)
N4	C5	N6	C7	-71.7 (4)	C3	N4	C5	N6	-55.6 (4)
N6	C7	C8	C9	-66.7 (4)					

Table 1* lists the fractional coordinates, Table 2 the interatomic distances and angles and Table 3 gives selected torsion angles. Fig. 1 shows the atom-numbering scheme in the title compound.

Related literature. The paper is a continuation of the study of the structure–biological-activity relationship of dibenzooxadiazacycloalkanes. The title compound was obtained by Glinka (1982). The formula was

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54349 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

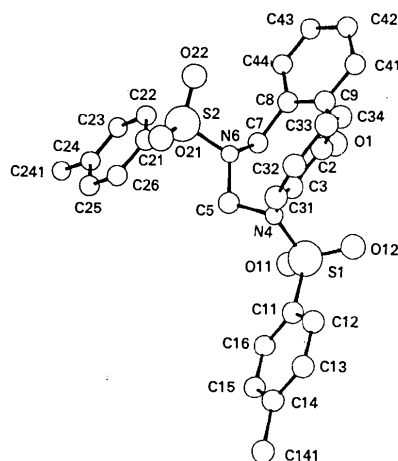


Fig. 1. The structure of the molecule with the atom-numbering scheme.

molecule was established; the final R value for the enantiomorphic form is 0.0505. Max. shift/e.s.d. = 0.14; the largest peak on a final difference map is 0.28 e \AA^{-3} . Complex scattering factors from *SHELX76*.

confirmed by MS, IR and NMR spectra (Glinka, 1985). The compound shows neuroleptic activity (Glinka, 1986). Previous papers (Stępień, Wajzman, Grabowski, Glinka & Perrin, 1987; Olszak, Stępień, Wajzman, Grabowski, Glinka & Lecocq, 1987) give details of the properties and structure of related compounds.

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References

- GLINKA, R. (1982). *Pol. J. Chem.* **56**, 1139–1144.
 GLINKA, R. (1985). *Acta Pol. Pharm.* **42**, 539–544.
 GLINKA, R. (1986). 9th International Conference on Medicinal Chemistry, West Berlin.
 OLSZAK, T. A., STĘPIEŃ, A., WAJSMAN, E., GRABOWSKI, M. J., GLINKA, R. & LECOCQ, S. (1987). *Acta Cryst.* **C43**, 2169–2171.
 SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
 STĘPIEŃ, A., WAJSMAN, E., GRABOWSKI, M. J., GLINKA, R. & PERRIN, M. (1987). *Acta Cryst.* **C43**, 2164–2165.

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Crystal Studies of Heterocyclic Compounds Containing One Oxygen and Two Nitrogen Atoms. XVII. *N,N'*-Bis(tosyl)perhydro-1,5,7-oxadiazecine

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Abstract. $C_{27}H_{28}N_2O_5S_2$, $M_r = 452.6$, orthorhombic, $Pnaa$, $a = 11.814$ (2), $b = 18.257$ (3), $c = 20.683$ (3) Å, $V = 4461$ (1) Å³, $Z = 8$, $D_m = 1.327$, $D_x = 1.348$ (1) g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.54178$ Å, $\mu = 24.1$ cm⁻¹, $F(000) = 1920$, room temperature, $R = 0.0512$ for 3533 reflections with $I > 3\sigma(I)$. The ten-membered perhydrooxadiazecine ring is in a boat-chair-boat conformation. The ring is approximately symmetrical with respect to a pseudo-twofold axis; the asymmetry parameter [Duax & Norton (1975). *Atlas of Steroid Structure*, Vol. I. New York: IFI/Plenum] $\Delta_2 = 9.9^\circ$. Both tosyl groups are axial. The weighted least-squares planes of both benzene rings form an angle of 86.3 (1) $^\circ$.

Experimental. This paper is a continuation of the study of the structure-biological-activity relationship of perhydrooxadiazecine derivatives that show neuroleptic activity.

Colourless, thick tabular crystals from ethanol at room temperature. D_m measured by flotation. The

specimen used for X-ray work, $0.2 \times 0.2 \times 0.3$ mm, was cut from a larger crystal. Diffraction data measured on a CAD-4 diffractometer using $\theta-2\theta$ scan technique, graphite-monochromatized Cu $K\alpha$ radiation. Unit-cell parameters obtained by least squares using setting angles of 22 reflections with $\theta_{\max} = 40.7^\circ$. Data collected in range $2.1 < \theta < 73.1^\circ$; not corrected for absorption; standard reflection 533, maximum change 9.9%; range of h , k and l : 0 to 14, 0 to 22, 0 to 25, respectively. Of 4244 independent reflections 3533 were considered observed by the criterion $I > 3\sigma(I)$ and used in calculations. Solution by direct methods using *SHELX76* (Sheldrick, 1976), H atoms located from difference Fourier map, refinement by full-matrix least-squares procedure on F magnitudes, 383 parameters. Refinement to final $R = 0.0512$, $S = 1.546$, unit weights. Max. shift/e.s.d. = 0.57; largest peaks, 0.62 and 0.60 e Å⁻³, on a final difference map near S atom (1.06 Å); the next peak is 0.45 e Å⁻³. Scattering factors from *SHELX76*. The geometry of the