

Related literature. Two-dimensional NMR analysis of this compound shows a preferred orientation of the C4-methoxy group towards the H atom of C3 (Cox, Prieto, Retamozo & Rodriguez, 1989). Details for the isolation are given by Takeda (1941), Diment, Ritchie & Taylor (1967), Calderwood & Fish (1966), Fish & Waterman (1971), Benages, Juarez, Albonico, Urzua & Cassels (1974) and Torres & Cassels (1978). For other spectroscopic data see Mitscher, Bathala, Clark & Beal (1975).

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Crystal Studies of Heterocyclic Compounds Containing One Oxygen and Two Nitrogen Atoms. XIII. 5,6,7,8,9,10-Hexahydro-4*H*-*N,N'*-ditosyl-1-oxa-5,9-diazadibenzo[*b,k*]cyclododecene

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Abstract. $C_{31}H_{32}N_2O_5S_2$, $M_r = 576.7$, monoclinic, $P2_1/c$, $a = 11.358$ (4), $b = 21.199$ (6), $c = 12.23$ (3) Å, $\beta = 96.14$ (2)°, $V = 2928.4$ (1.6) Å³, $Z = 4$, $D_x = 1.307$ (1) g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.54178$ Å, $\mu = 18.83$ cm⁻¹, $F(000) = 1216$. Diffractometer data at room temperature, $R = 0.044$ for 3760 reflections with $I > 2.5\sigma(I)$. The 12-membered heterocycle may be considered as consisting of two parts, in chair-like and boat-like conformations respectively, while the tosyl groups are in an *exo,exo* conformation. The heterocycles are arranged along the x axis.

Experimental. The title compound was obtained by condensation of the disodium salt of bis[2-(ditosylaminomethyl)phenyl] ether with 1,3-bis(tosyloxy)propane. The formula was confirmed by MS, IR and NMR spectra.

Colourless crystals from ethanol at room temperature; crystal size $0.1 \times 0.2 \times 0.3$ mm, Stoe diffractometer using θ – 2θ scan technique; unit-cell parameters from 21 reflections, $\theta_{\text{max}} = 20^\circ$, Cu $K\alpha$ radiation, range of h , k and l $0 \rightarrow 12$, $0 \rightarrow 23$, $-13 \rightarrow 13$ respectively. Total of 4345 unique reflections measured to $(\sin \theta)/\lambda$

Table 1. Final positional and equivalent isotropic thermal parameters ($\times 10^4$) with e.s.d.'s in parentheses
$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	x	y	z	$U_{eq} (\text{\AA}^2)$
O1	2843 (2)	10164 (1)	-926 (2)	691 (13)
C1	2509 (3)	11114 (2)	558 (3)	614 (20)
N1	1811 (2)	10725 (1)	1249 (2)	564 (14)
C2	2109 (3)	10049 (2)	1412 (3)	558 (18)
C3	1233 (3)	9617 (1)	748 (3)	512 (17)
C4	1666 (3)	8938 (2)	761 (3)	550 (18)
N2	2772 (2)	8874 (1)	212 (2)	487 (12)
C5	2576 (3)	8852 (2)	-1001 (3)	517 (17)
S1	989 (1)	11066 (0)	2067 (1)	573 (4)
O101	530 (2)	11633 (1)	1535 (2)	725 (14)
O102	184 (2)	10599 (1)	2399 (2)	714 (14)
C101	1895 (3)	11294 (1)	3255 (2)	496 (16)
C102	2406 (3)	11889 (2)	3328 (3)	599 (19)
C103	3082 (3)	12068 (2)	4278 (3)	653 (21)
C104	3270 (3)	11673 (2)	5166 (3)	614 (19)
C105	2787 (3)	11068 (2)	5055 (3)	690 (22)
C106	2113 (3)	10881 (2)	4122 (3)	623 (20)
C111	3914 (4)	11878 (3)	6239 (4)	919 (30)
S2	3705 (1)	8352 (0)	792 (1)	595 (4)
O201	4708 (2)	8355 (1)	184 (2)	774 (15)
O202	3840 (2)	8481 (1)	1946 (2)	768 (15)
C201	3008 (3)	7603 (1)	603 (3)	567 (17)
C202	2256 (3)	7402 (2)	1339 (3)	623 (20)
C203	1715 (3)	6817 (2)	1186 (3)	707 (22)
C204	1949 (3)	6433 (2)	325 (3)	722 (22)
C205	2689 (4)	6648 (2)	-413 (4)	845 (27)
C206	3226 (4)	7235 (2)	-285 (3)	749 (23)
C222	1417 (6)	5779 (2)	214 (5)	1039 (35)
C11	2167 (3)	10668 (1)	-1353 (3)	560 (18)
C12	1650 (3)	10678 (2)	-2422 (3)	682 (22)
C13	948 (4)	11190 (2)	-2784 (4)	804 (26)
C14	765 (3)	11672 (2)	-2073 (4)	793 (26)
C15	1253 (3)	11648 (2)	-1002 (3)	669 (21)
C16	1979 (3)	11144 (1)	-610 (3)	541 (17)
C21	3544 (3)	9844 (2)	-1606 (2)	577 (18)
C22	3466 (3)	9191 (1)	-1603 (2)	516 (16)
C23	4193 (3)	8869 (2)	-2264 (3)	666 (22)
C24	4935 (3)	9183 (2)	-2899 (3)	803 (26)
C25	5004 (3)	9831 (2)	-2864 (3)	800 (26)
C26	4313 (4)	10158 (2)	-2202 (3)	726 (24)

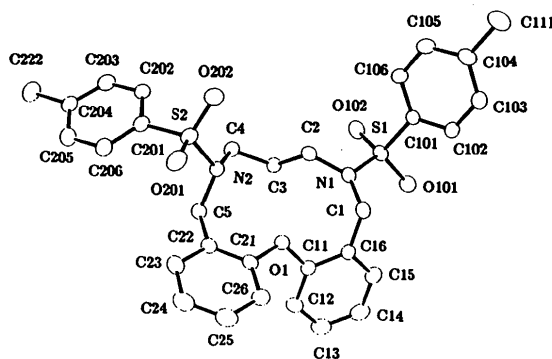
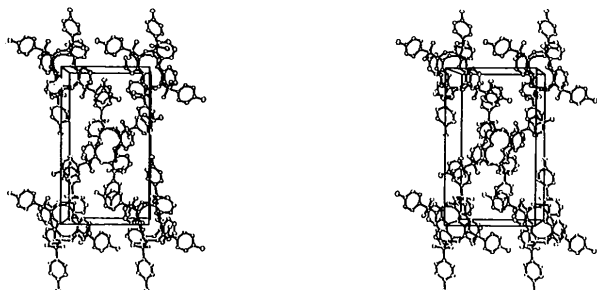


Fig. 1. Atomic numbering scheme.

Fig. 2. Stereoview of the molecular packing, viewed down the *a* axis.Table 2. Interatomic distances (\AA), angles ($^\circ$) and selected torsion angles ($^\circ$) with e.s.d.'s in parentheses

O1	C11	1.384 (4)	C15	C16	1.404 (5)				
O1	C21	1.390 (4)	C16	C11	1.389 (4)				
C1	N1	1.472 (5)	C21	C22	1.387 (4)				
C1	C16	1.492 (4)	C22	C23	1.394 (5)				
C2	N1	1.481 (4)	C23	C24	1.377 (6)				
C2	C3	1.523 (4)	C24	C25	1.377 (7)				
C3	C4	1.519 (4)	C25	C26	1.375 (6)				
C4	N2	1.492 (4)	C26	C21	1.368 (5)				
C5	N2	1.478 (4)	C101	C102	1.387 (4)				
C5	C22	1.497 (4)	C102	C103	1.377 (5)				
S1	N1	1.611 (3)	C103	C104	1.371 (5)				
S1	O101	1.437 (2)	C104	C105	1.395 (5)				
S1	O102	1.435 (2)	C105	C106	1.363 (5)				
S1	C101	1.756 (3)	C106	C101	1.377 (4)				
S2	N2	1.639 (2)	C104	C111	1.499 (5)				
S2	O201	1.425 (3)	C201	C202	1.374 (5)				
S2	O202	1.430 (2)	C202	C203	1.387 (5)				
S2	C201	1.778 (3)	C203	C204	1.379 (5)				
C11	C12	1.376 (5)	C204	C205	1.374 (6)				
C12	C13	1.390 (6)	C205	C206	1.388 (6)				
C13	C14	1.372 (7)	C206	C201	1.381 (5)				
C14	C15	1.368 (6)	C204	C222	1.514 (6)				
C11	O1	C21	118.8 (2)	C1	C16	C11	121.4 (3)		
C16	C1	N1	112.7 (3)	C1	C16	C15	121.4 (3)		
C1	N1	C2	119.4 (3)	O1	C21	C22	116.4 (3)		
C1	N1	S1	119.3 (2)	O1	C21	C26	121.3 (3)		
C2	N1	S1	119.3 (2)	C26	C21	C22	122.2 (3)		
N1	C2	C3	112.5 (2)	C21	C22	C23	116.4 (3)		
C2	C3	C4	111.8 (3)	C22	C23	C24	121.8 (4)		
C3	C4	N2	111.7 (3)	C23	C24	C25	120.1 (4)		
C4	N2	C5	114.2 (2)	C24	C25	C26	119.1 (4)		
C4	N2	S2	113.9 (2)	C25	C26	C21	120.3 (4)		
C5	N2	S2	115.7 (2)	C5	C22	C21	121.8 (3)		
N2	C5	C22	116.3 (2)	C5	C22	C23	121.6 (3)		
N1	S1	C101	108.0 (1)	S1	C101	C102	120.5 (2)		
N1	S1	O101	107.5 (1)	S1	C101	C106	120.3 (2)		
N1	S1	O102	107.2 (1)	C106	C101	C102	119.2 (3)		
O101	S1	O102	119.6 (1)	C101	C102	C103	119.8 (3)		
C101	S1	O101	107.2 (1)	C102	C103	C104	121.7 (3)		
C101	S1	O102	106.9 (1)	C103	C104	C105	117.3 (3)		
N2	S2	C201	106.7 (1)	C104	C105	C106	121.9 (3)		
N2	S2	O201	106.8 (1)	C105	C106	C101	120.0 (3)		
N2	S2	O202	107.1 (1)	C111	C104	C103	122.4 (4)		
O201	S2	O202	120.5 (1)	C111	C104	C105	120.3 (3)		
C201	S2	O201	107.8 (2)	S2	C201	C202	119.5 (3)		
C201	S2	O202	107.3 (2)	S2	C201	C206	119.5 (3)		
O1	C11	C12	122.3 (3)	C206	C201	C202	121.0 (3)		
O1	C11	C16	115.6 (3)	C201	C202	C203	119.1 (3)		
C16	C11	C12	122.0 (3)	C202	C203	C204	120.8 (4)		
C11	C12	C13	119.2 (4)	C203	C204	C205	119.2 (3)		
C12	C13	C14	120.0 (4)	C204	C205	C206	121.0 (4)		
C13	C14	C15	120.4 (4)	C205	C206	C201	118.9 (4)		
C14	C15	C16	121.2 (4)	C222	C204	C203	120.1 (4)		
C15	C16	C11	117.2 (3)	C222	C204	C205	120.7 (4)		
O1	C11	C16	C1	1.8 (5)	C1	N1	S1	O102	164.5 (2)
C11	C16	C1	N1	-82.5 (4)	C1	N1	S1	C101	-80.7 (2)
C16	C1	N1	C2	93.1 (3)	C3	C2	N1	S1	91.8 (3)
C1	N1	C2	C3	-104.7 (3)	C2	N1	S1	O101	-161.7 (2)
N1	C2	C3	C4	169.7 (3)	C2	N1	S1	O102	-32.0 (3)
C2	C3	C4	N2	-64.2 (4)	C2	N1	S1	C101	82.9 (2)
C3	C4	N2	C5	-80.1 (3)	C3	C4	N2	S2	144.0 (2)
C4	N2	C5	C22	141.6 (3)	C4	N2	S2	O201	-179.3 (2)
N2	C5	C22	C21	-69.7 (4)	C4	N2	S2	O202	-49.1 (2)
C5	C22	C21	O1	6.8 (4)	C4	N2	S2	C201	65.6 (3)
C22	C21	O1	C11	-131.9 (3)	C22	C5	N2	S2	-83.3 (3)
C21	O1	C11	C16	-148.9 (3)	C5	N2	S2	O201	45.5 (2)
C16	C1	N1	S1	-103.3 (3)	C5	N2	S2	O202	175.7 (2)
C1	N1	S1	O101	34.8 (2)	C5	N2	S2	C201	-69.6 (3)

$= 0.56 \text{ \AA}^{-1}$, data not corrected for absorption, standard reflections 080 and 006, maximum change of 3.1% and 2.5% respectively, 3760 reflections with $I > 2.5\sigma(I)$ used in calculations; solution by direct methods using *SHELX86* (Sheldrick, 1986), refinement using *SHELX76* (Sheldrick, 1976), H atoms located from difference Fourier map, refinement by full-matrix least-squares procedure on *F* magnitudes,

342 parameters. Refinement to final $R = 0.044$, $S = 1.0614$, unit weights. Largest peak on a final difference Fourier map 0.22 , lowest trough $-0.26 \text{ e } \text{Å}^{-3}$, $(\Delta/\sigma)_{\text{max}} = 0.29$. Scattering factors from *SHELX76*. The molecule and the numbering scheme are shown in Fig. 1, molecular packing in Fig. 2. Positional parameters and equivalent values of the anisotropic temperature factors for the non-H atoms are given in Table 1,* interatomic distances, angles and selected torsion angles in Table 2.

Related literature. The paper is a continuation of the study of the structure–biological activity relationship of oxazadibenzocycloalkenes. The title compound shows

* 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 51743 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

neuroleptic activity (Glinka, 1986). Related studies: Stępień, Wajzman, Grabowski, Glinka & Perrin (1987), Olszak, Stępień, Wajzman, Grabowski, Glinka & Lecocq (1987).

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Structure of (+)-(1*R*,2*S*,3*S*)-3-Benzoyl-6,6-dimethyl-2-bicyclo[3.1.1]heptanecarboxylic Acid

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Abstract. $\text{C}_{17}\text{H}_{20}\text{O}_3$, $M_r = 272.35$, monoclinic, C_2 , $a = 20.324$ (3), $b = 6.779$ (1), $c = 13.890$ (2) Å, $\beta = 126.72$ (1)°, $V = 1534.1$ (5) Å³, $Z = 4$, $D_x = 1.18 \text{ Mg m}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.0744 \text{ mm}^{-1}$, $F(000) = 584$, $T = 296 \text{ K}$, final $R_F = 0.042$ for 976 unique observed reflections. Pairs of molecules are held together across the twofold axis by the formation of two intermolecular hydrogen bonds between the carboxyl groups: the $\text{O}(1)\cdots\text{O}(2')$ distance is 2.628 (3) Å and the $\text{O}(1)\text{—H}\cdots\text{O}(2')$ angle 156 (6)°. The cyclohexane ring has an unusual conformation: five C atoms [C(1)–C(5)] are coplanar within the experimental uncertainty, with the sixth C atom [C(6)] 1.1 Å out of this plane. The carboxyl and benzoyl groups are in *trans* positions with regard to the cyclohexane ring, as suggested by ¹H NMR data.

Experimental. Colourless crystal obtained by evaporation of an ether solution, $0.60 \times 0.50 \times 0.35 \text{ mm}$, mounted on a glass fibre, Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized $\text{Mo } K\alpha$, ω - 2θ method, lattice parameters from 25 reflections ($8 < \theta < 13^\circ$), three standard reflections measured every hour, no loss of intensity, 1600 measured reflections (h 0→24, k 0→8, l -16→16) with $\theta < 25^\circ$, 1463 independent, 976 with $I > 3\sigma(I)$, Lp correction, no absorption correction, direct methods, refinement by full-matrix least squares using $w = 4F_o^2/[(\sigma I)^2 + (pF_o^2)^2]$ and F ; all non-H atoms anisotropic. H atom for the carboxylic group [H(10), involved in hydrogen bonding] found on difference map and refined, all other H atoms included in the refinement at calculated positions ($\text{C—H} = 0.95 \text{ Å}$, $B = 5 \text{ Å}^2$) and not refined