least-squares method using 25 reflections with $\theta_{max} \leq 51{\cdot}7°$; data collected to $2\theta_{max} = 146°$, not corrected for absorption. Ranges of h, k, l: -15 to 15, 0 to 6, 0 to 37 respectively, standard reflection 422, counts variation 0·6% (if variation was greater than 3% the orientation matrix was recalculated); of 3335 independent reflections, 2388 were accepted as observed by the criterion $I \geq 3\sigma(I)$. The structure was solved by Patterson and direct methods using *SHELX*76 (Sheldrick, 1976), the refinement carried out by full-matrix least squares using F magnitudes, 349 parameters; all H atoms located by a difference map. Final $R = 0{\cdot}0565$; $wR = 0{\cdot}0639$ where $w = k/[\sigma^2(F) + pF^2]$ with $p = 0{\cdot}000104$. Max. shift/e.s.d. = 0·8, largest peak on the final difference map was 0·4 e Å^{-3}. Atomic scattering factors those of *SHELX*.

Discussion. The final positional parameters are listed in Table 1, bond lengths, bond angles and torsion angles in Table 2.*

* 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 44147 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The oxadiazocine ring is in a crown conformation (see Fig. 1). It is pseudosymmetric; the asymmetry coefficients are: $C_{m1} = 2{\cdot}91°$ (the mirror plane passes through N6 and C2), $C_{m2} = 3{\cdot}12°$ (mirror plane passes through N4 and C8) and $C_2 = 3{\cdot}24°$ (twofold axis is perpendicular to the ring). The mean value of the torison angles in the heterocyclic ring is 87·20°. Both N atoms are supposed to be in sp^2 hybridization. The bond lengths and angles are typical. The phenyl rings are planar [maximum distance from the best planes is 0·007 (5) Å] and form an angle of 57·1 (3)°. There are van der Waals intermolecular interactions only. The molecular packing is shown in Fig. 2.

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Crystal Studies of Heterocyclic Compounds Containing One Oxygen and Two Nitrogen Atoms. V. 5,6,7,8,9,10-Hexahydro-5,10-ditosyl-16-oxa-5,10-diazadibenzo[*c*,*k*]cyclotridecene

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Abstract. $C_{32}H_{34}N_2O_5S_2$, $M_r = 590$, triclinic, $P\bar{1}$, $a = 12{\cdot}756$ (3), $b = 9{\cdot}950$ (3), $c = 13{\cdot}566$ (3) Å, $\alpha = 90{\cdot}49$ (1), $\beta = 118{\cdot}04$ (1), $\gamma = 90{\cdot}04$ (1)°, $V = 1519{\cdot}7$ (7) Å^3, $Z = 2$, $D_x = 1{\cdot}291$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 18{\cdot}25$ cm^{-1}, $F(000) = 624$, room temperature, $R = 0{\cdot}052$ for 3878 reflexions with $I > 3\sigma(I)$. The thirteen-membered ring is in a twist conformation and is approximately symmetrical with respect to a pseudo-twofold axis running through the O atom.

Introduction. The present paper is the continuation of the study of the relationship between the biological activity and the structure of heterocyclic rings con-

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Table 1. *Final positional and thermal parameters* ($\times 10^4$) *with e.s.d.'s in parentheses*

$U_{iso} = \frac{1}{3}\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{iso}(Å^2)
O8	5976 (2)	2492 (4)	5000 (3)	895 (22)
S1	10162 (1)	2306 (1)	8047 (1)	613 (6)
N1	9629 (3)	3200 (3)	6900 (2)	546 (18)
O1	10668 (3)	1147 (3)	7819 (2)	811 (20)
O2	9233 (3)	2178 (3)	8330 (2)	769 (20)
N6	7719 (3)	1787 (3)	3092 (2)	530 (18)
O3	5897 (2)	2813 (3)	1681 (2)	756 (19)
O4	7850 (3)	3860 (3)	2175 (2)	784 (20)
S2	7116 (1)	2694 (1)	1954 (1)	586 (6)
C5	9011 (3)	1536 (4)	3575 (3)	513 (21)
C4	9637 (3)	1755 (4)	4839 (3)	486 (20)
C3	9798 (3)	3242 (4)	5160 (3)	500 (20)
C2	10429 (3)	3466 (4)	6424 (3)	515 (21)
C9	7103 (4)	2530 (5)	5992 (4)	764 (29)
C7	6107 (4)	2467 (5)	4014 (4)	786 (31)
C23	6392 (5)	143 (5)	−585 (4)	922 (36)
C22	6281 (4)	933 (5)	195 (4)	834 (32)
C24	7386 (4)	182 (5)	−718 (3)	735 (29)
C21	7227 (3)	1754 (4)	897 (3)	553 (22)
C26	8212 (4)	1829 (5)	756 (3)	716 (28)
C25	8314 (4)	1041 (5)	−27 (4)	807 (31)
C241	7515 (5)	−701 (6)	−1560 (4)	1103 (42)
C14	13106 (4)	4823 (5)	10707 (3)	696 (28)
C11	11328 (3)	3234 (4)	9110 (3)	544 (22)
C12	11086 (4)	4063 (5)	9789 (4)	826 (32)
C16	12445 (4)	3178 (5)	9228 (3)	708 (28)
C15	13346 (4)	3976 (5)	10041 (4)	765 (30)
C13	11994 (5)	4837 (5)	10598 (4)	898 (35)
C141	14071 (5)	5729 (6)	11554 (4)	1024 (39)
C46	7119 (4)	−564 (5)	2928 (4)	701 (28)
C44	5816 (5)	−1325 (7)	3618 (5)	1036 (42)
C45	6511 (5)	−1594 (5)	3125 (5)	922 (37)
C43	5688 (4)	−5 (6)	3892 (4)	901 (36)
C42	6296 (3)	1052 (5)	3707 (3)	640 (26)
C41	7011 (3)	753 (4)	3232 (3)	558 (23)
C32	9187 (4)	5578 (4)	7070 (4)	728 (29)
C31	8782 (3)	4255 (4)	6764 (3)	576 (23)
C34	7193 (5)	6319 (7)	6391 (5)	1045 (43)
C33	8386 (5)	6593 (5)	6876 (5)	972 (39)
C36	7574 (4)	3950 (5)	6280 (3)	674 (27)
C35	6805 (4)	5021 (7)	6106 (4)	905 (37)

taining one O and two N atoms and fused with two aromatic rings. The title compound was obtained by Glinka (Glinka & Dalczynski, 1986) as a result of the condensation of bis(2-tosylaminobenzyl) ether with butanediol-1,4-ditosyl ester. The formula was confirmed by MS, IR and NMR spectra. Its pharmaceutical activity is less than that of the nine-membered analogue, toxicity being much the same as for the dibenzo-oxadiazonine systems.

Experimental. Colourless, acicular crystals from ethanol, dimensions 0·2 × 0·3 × 1 mm, room temperature. CAD-4 diffractometer using θ–2θ scan technique; unit-cell parameters from 25 reflexions in the θ range 11·5–31·7°, graphite-monochromatized Cu $K\alpha$ radiation, range of h, k and l −15 to 13, −12 to 12, 0 to 15 respectively; total of 6464 independent reflexions measured to $(\sin\theta)/\lambda = 0{\cdot}63$ Å^{-1}, data not corrected for absorption, $R_{int} = 0{\cdot}0421$; standard reflection $\bar{1}\bar{1}5$, maximum change 0·2%. 3884 reflexions with $I > 3\sigma(I)$ used in calculations, solution by direct methods using *SHELX*76 (Sheldrick, 1976), all H atoms located from difference maps, refinement by full-matrix least-squares procedure on F magnitudes (506 parameters) to final $R = 0{\cdot}052$, $S = 0{\cdot}89$, unit weights; largest peak on final difference map 0·29 e Å^{-3}, ratio of max. shift/e.s.d. = 0·996, scattering factors from *SHELX*76.

Table 2. *Interatomic distances* (Å) *and bond angles* (°)

C9–O8	1·435 (4)	C241–C24	1·504 (8)
C7–O8	1·423 (7)	C21–C26	1·358 (7)
N1–S1	1·645 (3)	C25–C26	1·370 (8)
O1–S1	1·423 (4)	C15–C14	1·366 (8)
O2–S1	1·413 (4)	C13–C14	1·355 (8)
C11–S1	1·761 (3)	C141–C14	1·517 (6)
C2–N1	1·467 (6)	C12–C11	1·370 (7)
C31–N1	1·456 (5)	C16–C11	1·359 (7)
S2–N6	1·643 (3)	C13–C12	1·389 (6)
C5–N6	1·482 (5)	C15–C16	1·400 (6)
C41–N6	1·439 (6)	C45–C46	1·386 (8)
S2–O3	1·425 (3)	C41–C46	1·397 (6)
S2–O4	1·428 (3)	C45–C44	1·363 (11)
C21–S2	1·766 (5)	C43–C44	1·392 (9)
C5–C4	1·527 (5)	C42–C43	1·399 (8)
C3–C4	1·526 (5)	C41–C42	1·374 (7)
C2–C3	1·527 (5)	C31–C32	1·400 (6)
C36–C9	1·510 (7)	C33–C32	1·374 (8)
C42–C7	1·516 (7)	C31–C36	1·394 (6)
C22–C23	1·374 (8)	C33–C34	1·371 (9)
C24–C23	1·363 (9)	C35–C34	1·368 (9)
C21–C22	1·392 (6)	C35–C36	1·394 (8)
C25–C24	1·397 (6)		

C7–O8–C9	112·0 (4)	O1–S1–N1	105·5 (2)
O2–S1–N1	106·3 (2)	O2–S1–O1	120·6 (2)
C11–S1–N1	107·8 (2)	C11–S1–O1	107·5 (2)
C11–S1–O2	108·5 (2)	C2–N1–S1	117·0 (2)
C31–N1–S1	118·0 (3)	C31–N1–C2	116·1 (3)
C5–N6–S2	116·3 (3)	C41–N6–S2	118·5 (2)
C41–N6–C5	117·0 (3)	O3–S2–N6	105·3 (2)
O4–S2–N6	106·5 (2)	O3–S2–O4	120·8 (2)
C21–S2–N6	106·9 (2)	C21–S2–O3	109·1 (2)
C21–S2–O4	107·4 (2)	C4–C5–N6	111·1 (4)
C3–C4–C5	112·2 (3)	C2–C3–C4	112·4 (3)
C3–C2–N1	111·2 (3)	C36–C9–O8	111·1 (4)
C42–C7–O8	111·8 (4)	C24–C23–C22	121·7 (4)
C21–C22–C23	119·0 (5)	C25–C24–C23	118·4 (5)
C241–C24–C23	121·6 (4)	C241–C24–C25	119·9 (6)
C22–C21–S2	119·0 (4)	C26–C21–S2	121·1 (3)
C26–C21–C22	119·9 (4)	C25–C26–C21	120·6 (4)
C26–C25–C24	120·2 (5)	C13–C14–C15	118·9 (4)
C141–C14–C15	120·3 (5)	C141–C14–C13	120·7 (5)
C43–C44–C45	119·8 (6)	C41–C46–C45	119·6 (5)
C12–C13–C14	121·4 (5)	C16–C15–C14	120·4 (5)
C15–C16–C11	119·8 (5)	C13–C12–C11	119·3 (5)
C16–C11–C12	120·1 (4)	C16–C11–S1	120·6 (3)
C12–C11–S1	119·2 (3)	C44–C45–C46	120·3 (5)
C42–C43–C44	121·0 (6)	C43–C42–C7	118·4 (5)
C41–C42–C7	123·5 (4)	C41–C42–C43	118·1 (5)
C46–C41–N6	118·5 (4)	C42–C41–N6	120·4 (4)
C42–C41–C46	121·1 (4)	C33–C32–C31	119·8 (4)
C32–C31–N1	119·4 (4)	C36–C31–N1	119·7 (4)
C36–C31–C32	120·8 (4)	C35–C34–C33	119·4 (6)
C34–C33–C32	120·4 (5)	C31–C36–C9	122·6 (4)
C35–C36–C9	120·6 (4)	C35–C36–C31	116·8 (4)
C36–C35–C34	122·8 (5)		

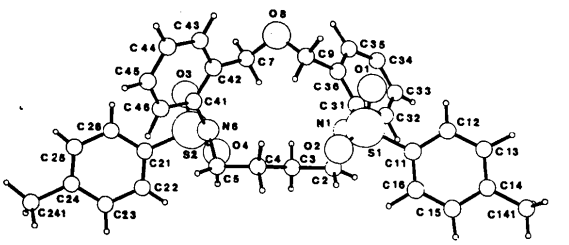

Fig. 1. The structure of the molecule viewed down the y axis with the numbering scheme.

Discussion. The final positional parameters are listed in Table 1,* and bond lengths and bond angles are given in Table 2. Fig. 1 shows the structure of the molecule with the numbering scheme. The geometry of the molecule was calculated using *FFE*3 (Busing, Martin & Levy, 1971).

The thirteen-membered ring is in a twist conformation. The ring is approximately symmetrical with respect to a pseudo-twofold axis running through O8 and the middle of the C3–C4 bond; the asymmetry coefficient $\Delta_2 = 0{\cdot}35°$, $\Delta_2 = [\sum(\varphi_i - \varphi'_i)^2/n(n-1)]^{1/2}$, where φ_i and φ'_i are torsional angles of approximately symmetrical atoms.

The fused ring planes form a dihedral angle of 46·9 (1)°, while the tosyl substituent planes (excluding the O atoms) form a dihedral angle of 11·2 (1)°. There are van der Waals molecular contacts only.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44075 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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The Structure of 2,2′-Bis(tosylmethylphenyl) Ether

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Abstract. $C_{28}H_{26}O_5S_2$, $M_r = 506{\cdot}64$, triclinic, $P\bar{1}$, $a = 9{\cdot}243$ (3), $b = 10{\cdot}439$ (3), $c = 13{\cdot}646$ (3) Å, $\alpha = 92{\cdot}79$ (4), $\beta = 99{\cdot}36$ (4), $\gamma = 101{\cdot}39$ (4)°, $V = 1269{\cdot}1$ (7) Å^3, $Z = 2$, $D_x = 1{\cdot}326$ g cm^{-3}, $\mu = 20{\cdot}87$ cm^{-1}. Diffractometer data collected at room temperature [λ(Cu $K\alpha$) = 1·54178 Å] gave 4386 independent reflections with $I > 3\sigma(I)$, $F(000) = 532$, final $R = 0{\cdot}0570$. The molecule is not a symmetrical one; the angles formed by the four six-membered planar rings are different.

Introduction. Condensation of 2,2′-bis(bromoethylphenyl) ether with *N,N′*-ditosylhydrazine produces *N,N′*-ditosyltetrahydrodibenzo[*b,h*][1,5,6]oxadiazonine. The structure of this product has been established by elementary and spectral (IR, NMR, MS) analyses. Its melting point is 416–417 K. The compound was found to be unstable. During its decomposition with the evolution of nitrogen a product characterized by a melting point of 406–408 K was obtained. On the basis of elemental analysis which excluded the presence of nitrogen, IR and NMR studies, identification as 2,2′-bis(tosylmethylphenyl) ether was proposed (Glinka, 1981).

Experimental. Yellowish crystals grown from methanol at room temperature, $\mu r = 0{\cdot}21$, cell parameters and intensity data measured on a CAD-4 diffractometer using θ–2θ scan technique; lattice parameters refined by least-squares method using 25 reflections with $\theta_{max} = 50{\cdot}8°$; total of 4390 independent reflections measured to $(\sin\theta)/\lambda = 0{\cdot}63$ Å^{-1}, values of h,k,l were −11 to 11, −12 to 12, 0 to 15 respectively, data not corrected for absorption, standard reflection $\bar{2}\bar{2}2$, mean variation 0·6%; solution by direct method using *SHELX*76 (Sheldrick, 1976), all non-H atoms found on

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