absorption, maximum values of h,k,l were 21,13,9 respectively, standard reflection 212 with mean variation 0·6%, solution by direct methods using *SHELX*76 (Sheldrick, 1976), H atoms found on difference map, refinement by full-matrix least squares (F magnitudes, 282 parameters), final $R = 0.0362$ for 1766 reflections with $I > 3\sigma(I)$, $wR = 0.0403$ where $w = 1.0000\sigma^2(F) + 0.0054(F^2)$; max. shift/e.s.d. = 0·20, largest peak on final difference map was $0.17\,e\,Å^{-3}$. Atomic scattering factors those of *SHELX*.

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

The molecules are in general positions (the numbering of the atoms is shown in Fig. 1). All the rings of the molecule are planar [the maximum distance from the plane is 0·006 (4) Å]. The oxadiazole and the neighbouring phenyl rings are almost coplanar [2·7 (3) and 0·9 (3)°]. The oxadiazole rings form an angle of 18·7 (3)°. Therefore, the whole molecule is nearly planar. The distances and angles between the C4, O1 and C5 atoms are typical for ethers. The distances between the middles of the rings are within the limits of
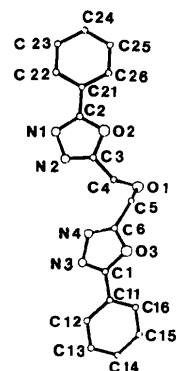

Fig. 1. The atom-numbering scheme.

3·85–4·37 Å. Hence, it seems that all four rings of the molecule can take part in intermolecular charge-transfer interactions, though this is not confirmed by the colour of the crystals.

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* 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 44146 (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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Crystal Studies of Heterocyclic Compounds Containing One Oxygen and Two Nitrogen Atoms. IV. *N,N'*-Ditosylperhydro-1,4,6-oxadiazocine

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Abstract. $C_{19}H_{24}N_2O_5S_2$, $M_r = 424.6$, monoclinic, $P2_1/n$ (non-standard group), $a = 12.523\,(4)$, $b = 5.374\,(1)$, $c = 30.128\,(9)$ Å, $\beta = 100.49\,(3)°$, $V = 1993.8\,(16)$ Å^3, $Z = 4$, $\mu = 25.8$ cm^{-1}, $D_x = 1.414$ g cm^{-3}. Diffractometer data at room temperature, $\lambda(\text{Cu } K\alpha) = 1.54178$ Å. $F(000) = 896$; final

$R = 0.0565$ for 2371 reflections with $I > 3\sigma(I)$. The oxadiazocine ring is in the crown conformation. There are no unusual bond lengths or angles.

Introduction. The title compound was obtained by condensation of N,N'-ditosyl-3-oxa-1,5-pentane-diamine with dibromomethane (Krakowiak, 1982), m.p. 367–368 K. The interest in this compound arises from its properties of forming complexes with metals. The complexed metals can be transported into lipophilic phases *via* natural and artificial membranes (Auto-renkollektiv, 1982, 1983) and this property of the compound makes it very similar to antibiotics.

Experimental. The compound was recrystallized from methanol. The dimensions of the needle-shaped crystals were $0.5 \times 0.1 \times 0.1$ mm. Diffraction data measured on a CAD-4 diffractometer, lattice parameters by

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

$$U_{eq} = \tfrac{1}{3}(U_{11} + U_{22} + U_{33} + 2U_{13}\cos\beta).$$

	x	y	z	$U_{eq}(\text{Å}^2)$
O1	534 (2)	−1223 (6)	1573 (1)	849 (22)
C2	1399 (4)	180 (11)	1820 (2)	745 (30)
C3	2462 (4)	−1197 (11)	1891 (2)	736 (30)
N4	3178 (3)	−608 (7)	1572 (1)	648 (20)
C5	2975 (5)	−1756 (11)	1135 (2)	760 (32)
N6	2017 (3)	−813 (7)	839 (1)	655 (20)
C7	959 (5)	−2072 (13)	829 (2)	829 (36)
C8	251 (4)	−778 (13)	1109 (2)	841 (35)
S1	4182 (1)	1250 (2)	1746 (0)	776 (8)
O11	3757 (3)	3383 (6)	1950 (2)	1132 (29)
O12	4762 (3)	1574 (9)	1389 (1)	1232 (31)
C11	5017 (3)	−340 (8)	2184 (1)	606 (24)
C12	5580 (4)	−2433 (9)	2089 (2)	677 (27)
C13	6257 (4)	−3631 (9)	2432 (2)	727 (29)
C14	6385 (3)	−2857 (8)	2874 (1)	631 (24)
C15	5808 (4)	−780 (9)	2962 (2)	746 (29)
C16	5133 (4)	461 (9)	2620 (2)	748 (30)
C141	7110 (5)	−4182 (16)	3251 (2)	884 (41)
S2	2149 (1)	948 (2)	413 (0)	628 (6)
O21	1099 (2)	1848 (6)	235 (1)	791 (20)
O22	3013 (3)	2638 (6)	563 (1)	763 (19)
C21	2557 (3)	−1052 (8)	12 (1)	587 (22)
C22	3638 (4)	−1282 (10)	−7 (2)	834 (34)
C23	3957 (5)	−2928 (11)	−312 (2)	931 (38)
C24	3221 (5)	−4332 (9)	−591 (2)	805 (31)
C25	2140 (5)	−4082 (10)	−565 (2)	874 (34)
C26	1802 (4)	−2451 (10)	−262 (2)	758 (30)
C241	3585 (12)	−6103 (18)	−926 (3)	1252 (67)

Table 2. *Selected bond lengths (Å), bond angles (°) and torsion angles (°)*

O1—C2	1.415 (5)	O1—C8	1.400 (5)
C2—C3	1.505 (7)	C7—C8	1.501 (8)
C3—N4	1.462 (5)	N6—C7	1.484 (6)
N4—C5	1.434 (6)	C5—N6	1.450 (6)
N4—S1	1.617 (3)	N6—S2	1.626 (3)
S1—O11	1.448 (4)	S2—O21	1.412 (3)
S1—O12	1.414 (3)	S2—O22	1.422 (3)
S1—C11	1.750 (4)	S2—C21	1.761 (4)
C11—C12	1.385 (6)	C21—C22	1.371 (6)
C12—C13	1.372 (6)	C22—C23	1.387 (7)
C13—C14	1.374 (6)	C23—C24	1.357 (7)
C14—C15	1.381 (6)	C24—C25	1.376 (7)
C15—C16	1.380 (6)	C25—C26	1.386 (7)
C16—C11	1.365 (6)	C26—C21	1.363 (6)
C14—C141	1.500 (7)	C24—C241	1.516 (8)
O1—C2—C3	112.7 (4)	O1—C8—C7	114.5 (5)
C2—C3—N4	115.7 (4)	N6—C7—C8	113.2 (4)
C3—N4—C5	118.9 (4)	C5—N6—C7	119.2 (4)
N4—C5—N6	113.8 (4)	C2—O1—C8	117.7 (4)
S1—N4—C3	117.0 (3)	S2—N6—C7	117.9 (3)
S1—N4—C5	124.1 (3)	S2—N6—C5	119.9 (3)
N4—S1—O11	107.6 (2)	N6—S2—O21	106.1 (2)
N4—S1—O12	107.8 (2)	N6—S2—O22	107.5 (2)
N4—S1—C11	105.4 (2)	N6—S2—C21	105.6 (2)
C11—S1—O11	106.4 (2)	C21—S2—O21	108.4 (2)
C11—S1—O12	108.3 (2)	C21—S2—O22	108.0 (2)
O11—S1—O12	120.4 (3)	O21—S2—O22	120.3 (2)
S1—C11—C12	119.9 (3)	S2—C21—C22	119.7 (3)
S1—C11—C16	121.1 (3)	S2—C21—C26	119.8 (3)
C16—C11—C12	119.1 (4)	C26—C21—C22	120.4 (4)
C11—C12—C13	119.5 (4)	C21—C22—C23	119.4 (5)
C12—C13—C14	122.3 (4)	C22—C23—C24	121.3 (5)
C13—C14—C15	117.3 (4)	C23—C24—C25	118.4 (5)
C14—C15—C16	121.1 (4)	C24—C25—C26	121.4 (5)
C15—C16—C11	120.6 (4)	C25—C26—C21	119.1 (5)
C141—C14—C13	122.6 (5)	C241—C24—C23	120.5 (7)
C141—C14—C15	120.1 (5)	C241—C24—C25	121.1 (7)
O1—C2—C3—N4	97.3 (5)	C5—N6—C7—C8	99.4 (6)
C2—C3—N4—C5	−80.4 (6)	N6—C7—C8—O1	−78.4 (6)
C3—N4—C5—N6	71.7 (6)	C7—C8—O1—C2	81.1 (6)
N4—C5—N6—C7	−91.9 (6)	C8—O1—C2—C3	−97.3 (6)

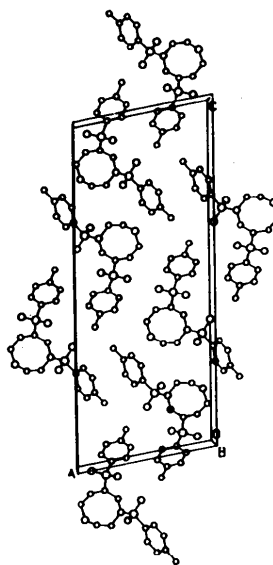Fig. 1. The structure of the molecule.

Fig. 2. A view of the molecular packing along **b**.

least-squares method using 25 reflections with $\theta_{max} \leq 51\cdot7°$; 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 SHELX76 (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 $R = 0\cdot0565$; $wR = 0\cdot0639$ where $w = k/[\sigma^2(F) + pF^2]$ with $p = 0\cdot000104$. Max. shift/e.s.d. $= 0\cdot8$, largest peak on the final difference map was $0\cdot4$ 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.*

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* 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: $C_{m1} = 2\cdot91°$ (the mirror plane passes through N6 and C2), $C_{m2} = 3\cdot12°$ (mirror plane passes through N4 and C8) and $C_2 = 3\cdot24°$ (twofold axis is perpendicular to the ring). The mean value of the torsion 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 $0\cdot007$ (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, $P\bar{1}$, $a = 12\cdot756$ (3), $b = 9\cdot950$ (3), $c = 13\cdot566$ (3) Å, $\alpha = 90\cdot49$ (1), $\beta = 118\cdot04$ (1), $\gamma = 90\cdot04$ (1)°, $V = 1519\cdot7$ (7) Å^3, $Z = 2$, $D_x = 1\cdot291$ g cm^{-3}, λ(Cu $K\alpha$) $= 1\cdot54178$ Å, $\mu = 18\cdot25$ cm^{-1}, $F(000) = 624$, room temperature, $R = 0\cdot052$ 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-