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Crystal Studies of Heterocyclic Compounds Containing One Oxygen and Two Nitrogen Atoms. X. *N,N'*-Ditosyl-6,7,8,13-tetrahydrodibenz[*b,g*][1,4,6]oxadiazonine

BY A. STĘPIEŃ, U. DEREWENDA AND M. J. GRABOWSKI

Department of Crystallography, Institute of Chemistry, University of Łódź, Nowotki 18, 91-416 *Łódź, Poland*

R. GLINKA

Institute of Chemistry and Technology of Drugs, School of Medicine, Narutowicza 120*a*, 90-145 *Łódź, Poland*

AND A. THOZET

Laboratoire de Minéralogie–Cristallographie associé au CNRS (UA 805), *Université Claude Bernard Lyon* I, 43 *Boulevard du* 11 *Novembre* 1918, 69622 *Villeurbanne CEDEX, France*

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Abstract. $C_{28}H_{26}N_2O_5S_2$, $M_r = 534{\cdot}6$, monoclinic, $P2_1/c$, $a = 13{\cdot}734\ (6)$, $b = 30{\cdot}818\ (9)$, $c = 13{\cdot}303\ (7)$ Å, $\beta = 110{\cdot}87\ (3)°$, $V = 5261\ (4)$ Å^3, $Z = 8$, $D_x = 1{\cdot}350\ (1)$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}54178$ Å, $\mu = 20{\cdot}61$ cm^{-1}, $F(000) = 2240$, room temperature, $R = 0{\cdot}0587$ for 4018 reflections with $I > 3\sigma(I)$. There are two different molecules in the asymmetric unit: molecule 1 is in an *exo,exo* modification and molecule 2 in an *exo,endo* modification. The nine-membered ring is in a quasi-chair conformation for both molecules.

Introduction. This paper is a continuation of the study of the structure–biological-activity relationship of dibenzoxadiazacycloalkanes. The title compound was obtained by reaction of the sodium salt of *N*-{2-[2-(*p*-toluenesulfonamide)methoxy]phenyl}toluenesulfonamide with methylene dibromide. The formula was confirmed by mass spectrometry and IR and NMR spectra (Glinka, 1985). The compound shows weak neuroleptic activity (Glinka, 1986).

Experimental. Light-yellow crystals from ethanol, room temperature; crystal dimensions $0{\cdot}1 \times 0{\cdot}2 \times 0{\cdot}4$ mm. CAD-4 diffractometer using θ–2θ scan technique; unit-cell parameters from 25 reflections in the θ range 9·0–46·3°; graphite-monochromatized Cu $K\alpha$ radiation; range of h, k, and l: 0 to 15, 0 to 37 and −15 to 15, respectively. 7199 independent reflections measured to $(\sin\theta)/\lambda = 0{\cdot}63$ Å^{-1}, data not corrected for absorption, $R_{int} = 0{\cdot}0477$; standard reflection 372, maximum change 2·2%. 4018 reflections with $I > 3\sigma(I)$ used in calculations. Solution by direct methods using *SHELX*76 (Sheldrick, 1976); all H atoms located from a difference map; refinement by a full-matrix least-squares procedure on F magnitudes (875 parameters); each symmetry-independent molecule was refined in a separate block. Because of high thermal vibrations (or disorder) of atoms of the benzene rings of both tosyl groups, in last two refinement cycles constraints were applied to the geometry of these rings. Refinement to final $R = 0{\cdot}0587$, $S = 2{\cdot}26$, unit weights; largest peak on final difference map 0·36 e Å^{-3}; ratio of max. shift/e.s.d. = 0·74; scattering factors from *SHELX*76.

Discussion. The final atomic coordinates are listed in Table 1,* bond lengths and angles in Table 2. A view of the molecular structure with the atomic numbering scheme is shown in Fig. 1. The geometry of the molecule was calculated using the *ORFFE*3 program (Busing, Martin & Levy, 1971).

There are two different molecules in the asymmetric unit. The difference is mainly in the orientation of the tosyl groups: in molecule 1 they are oriented away from the heterocyclic ring while in molecule 2 the orientation

* 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 51166 (33 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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of the N1 tosyl group is changed significantly: torsion angle C31–S2–N1–C14 is −74·7 (5)° for molecule 1 and 77·6 (4)° for molecule 2. It follows that the N1 tosyl group of molecule 2 is oriented towards the heterocyclic ring (see Fig. 1). Thus we observe the *exo,exo* conformation in molecule 1 and the *exo,endo* conformation in molecule 2. Such a conformation for molecule 2 ensures closer packing of the molecules in the crystal.

The nine-membered ring is in a quasi-chair conformation in both molecules and is approximately

Table 1. *Final positional* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^4$) *with e.s.d.'s in parentheses*

Asterisks indicate the atoms of molecule 1.

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

	x	y	z	U_{eq}
O1*	5502 (6)	1767 (2)	4819 (5)	955 (66)
N1*	7142 (5)	1283 (2)	6118 (5)	706 (58)
C5*	6920 (7)	1149 (3)	7047 (7)	621 (80)
S2*	8187 (2)	1539 (1)	6189 (2)	707 (17)
O21*	8566 (5)	1729 (2)	7244 (6)	1107 (68)
O22*	7949 (5)	1804 (2)	5255 (6)	1090 (67)
C1*	4940 (8)	1186 (4)	3519 (8)	791 (88)
C2*	5108 (9)	786 (4)	3208 (9)	898 (104)
C3*	5946 (9)	537 (4)	3798 (8)	792 (96)
C4*	6632 (8)	710 (3)	4747 (8)	716 (83)
C14*	6473 (7)	1110 (3)	5089 (6)	641 (72)
C13*	5621 (8)	1353 (3)	4472 (7)	691 (77)
C32*	9428 (7)	824 (3)	6903 (6)	807 (89)
C33*	10152 (8)	524 (3)	6826 (7)	889 (96)
C34*	10534 (7)	534 (3)	5994 (8)	813 (96)
C35*	10195 (8)	860 (4)	5218 (7)	929 (109)
C36*	9468 (8)	1161 (3)	5288 (7)	919 (103)
C31*	9094 (6)	1153 (3)	6123 (7)	750 (80)
C37*	11303 (10)	226 (4)	5937 (12)	1168 (135)
N2*	5864 (5)	1244 (2)	6937 (5)	655 (57)
C10*	4638 (9)	1804 (4)	5243 (10)	773 (102)
S1*	5053 (2)	851 (1)	6930 (2)	692 (18)
O11*	5224 (5)	517 (2)	6266 (4)	757 (50)
O12*	4041 (4)	1040 (2)	6672 (5)	901 (57)
C6*	5982 (9)	1828 (3)	8232 (9)	863 (101)
C7*	5770 (10)	2236 (4)	8511 (11)	968 (115)
C8*	5173 (10)	2503 (4)	7703 (12)	1010 (124)
C9*	4816 (8)	2368 (3)	6630 (11)	893 (103)
C11*	5021 (7)	1952 (3)	6324 (8)	794 (83)
C12*	5634 (7)	1683 (3)	7197 (8)	745 (77)
C22*	5156 (7)	854 (3)	9025 (7)	794 (94)
C23*	5506 (8)	696 (3)	10071 (7)	869 (115)
C24*	6135 (8)	328 (3)	10333 (6)	829 (94)
C25*	6429 (7)	127 (3)	9554 (7)	800 (93)
C26*	6094 (7)	288 (3)	8511 (6)	713 (84)
C21*	5456 (6)	654 (3)	8242 (5)	632 (82)
C27*	6490 (12)	143 (5)	11427 (9)	1311 (146)
O1	855 (4)	1348 (2)	2611 (4)	761 (47)
N1	521 (4)	758 (2)	931 (4)	566 (47)
C5	988 (7)	941 (2)	203 (6)	604 (63)
S2	−704 (2)	625 (1)	532 (2)	707 (17)
O21	−1099 (4)	675 (2)	−621 (4)	812 (45)
O22	−764 (4)	219 (2)	1015 (5)	962 (55)
C1	2231 (6)	900 (3)	3795 (6)	713 (68)
C2	2770 (7)	520 (3)	3986 (7)	756 (74)
C3	2524 (7)	205 (3)	3204 (7)	726 (77)
C4	1756 (6)	274 (2)	2205 (6)	646 (68)
C14	1237 (5)	661 (2)	2004 (5)	560 (56)
C13	1450 (6)	976 (2)	2802 (6)	631 (63)
C32	−1616 (7)	1397 (3)	561 (7)	911 (91)
C33	−2091 (8)	1700 (3)	1023 (10)	1150 (124)
C34	−2294 (9)	1604 (4)	1940 (10)	1269 (146)
C35	−2010 (9)	1199 (5)	2429 (8)	1253 (151)
C36	−1518 (7)	903 (3)	1991 (7)	1014 (104)
C31	−1314 (6)	995 (3)	1075 (6)	749 (73)
C37	−2817 (15)	1900 (8)	2434 (16)	2285 (268)
N2	1529 (4)	1356 (2)	608 (4)	529 (44)
C10	1372 (9)	1748 (3)	2505 (8)	742 (85)
S1	2702 (1)	1429 (1)	613 (2)	622 (14)
O11	3123 (4)	1790 (2)	1320 (4)	750 (42)
O12	3207 (4)	1016 (2)	840 (4)	774 (43)
C6	319 (7)	1896 (3)	−535 (7)	769 (78)
C7	−304 (8)	2256 (3)	−652 (10)	1010 (101)
C8	−361 (9)	2452 (4)	247 (12)	1139 (124)
C9	196 (8)	2297 (3)	1284 (10)	946 (101)
C11	827 (6)	1931 (3)	1422 (7)	713 (72)
C12	888 (6)	1738 (2)	500 (6)	637 (64)
C22	2381 (7)	1270 (2)	−1489 (6)	713 (78)
C23	2261 (7)	1386 (3)	−2531 (6)	742 (81)
C24	2344 (6)	1816 (3)	−2790 (5)	732 (73)
C25	2560 (8)	2129 (2)	−1986 (6)	875 (87)
C26	2672 (7)	2014 (2)	−946 (6)	778 (79)
C21	2600 (5)	1583 (2)	−680 (5)	582 (57)
C27	2241 (10)	1932 (4)	−3897 (9)	1051 (121)

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

	Molecule 1	Molecule 2
C11–C12	1·431 (12)	1·391 (13)
C12–C6	1·361 (15)	1·406 (11)
C6–C7	1·370 (17)	1·375 (14)
C7–C8	1·370 (17)	1·367 (21)
C8–C9	1·397 (20)	1·402 (17)
C9–C11	1·402 (15)	1·395 (13)
S1–N2	1·643 (7)	1·624 (6)
S1–O11	1·429 (6)	1·438 (5)
S1–O12	1·432 (6)	1·429 (5)
S1–C21	1·742 (7)	1·741 (7)
C21–C22	1·392 (14)	1·395 (10)
C22–C23	1·388 (13)	1·383 (11)
C23–C24	1·393 (14)	1·385 (11)
C24–C25	1·384 (14)	1·391 (11)
C25–C26	1·389 (12)	1·383 (12)
C21–C26	1·394 (11)	1·388 (10)
C24–C27	1·475 (15)	1·472 (14)
C5–N2	1·435 (13)	1·482 (9)
N2–C12	1·459 (11)	1·446 (9)
O1–C10	1·490 (18)	1·454 (12)
C10–C11	1·420 (16)	1·478 (12)
C13–C14	1·387 (12)	1·390 (10)
C14–C4	1·357 (14)	1·366 (11)
C4–C3	1·386 (13)	1·388 (10)
C3–C2	1·374 (15)	1·373 (13)
C2–C1	1·346 (17)	1·361 (13)
C1–C13	1·379 (12)	1·393 (10)
S2–N1	1·611 (8)	1·627 (6)
S2–O21	1·437 (8)	1·441 (5)
S2–O22	1·425 (8)	1·423 (6)
S2–C31	1·748 (9)	1·719 (9)
C31–C32	1·405 (14)	1·406 (13)
C32–C33	1·388 (12)	1·400 (16)
C33–C34	1·384 (16)	1·376 (20)
C34–C35	1·397 (14)	1·396 (19)
C35–C36	1·390 (16)	1·382 (18)
C31–C36	1·379 (15)	1·372 (14)
C34–C37	1·441 (18)	1·455 (28)
C5–N1	1·435 (13)	1·452 (11)
N1–C14	1·451 (10)	1·447 (8)
O1–C13	1·386 (12)	1·379 (9)

	Molecule 1	Molecule 2
N2–S1–O11	105·9 (4)	106·1 (3)
N2–S1–O12	107·5 (4)	106·5 (3)
N2–S1–C21	106·2 (3)	107·6 (3)
C21–S1–O11	107·8 (4)	107·9 (3)
C21–S1–O12	108·4 (4)	108·3 (3)
O11–S1–O12	120·3 (4)	119·9 (3)
S1–C21–C26	119·0 (7)	121·6 (5)
S1–C21–C22	121·5 (6)	119·6 (5)
C26–C21–C22	119·5 (7)	118·7 (6)
C21–C22–C23	120·4 (8)	120·8 (6)
C22–C23–C24	119·9 (10)	120·4 (7)
C23–C24–C25	119·8 (8)	119·1 (7)
C24–C25–C26	120·4 (8)	120·6 (7)
C25–C26–C21	120·0 (8)	120·5 (6)
C27–C24–C23	121·2 (10)	119·6 (8)
C27–C24–C25	119·0 (10)	121·2 (8)
S1–N2–C5	120·5 (6)	119·3 (5)
S1–N2–C12	118·8 (7)	117·1 (5)
C11–C12–C6	121·6 (9)	122·0 (7)
C12–C6–C7	122·9 (10)	119·7 (10)
C6–C7–C8	117·6 (12)	119·1 (10)
C7–C8–C9	121·2 (12)	121·9 (11)
C8–C9–C11	122·3 (10)	120·1 (12)
C9–C11–C12	114·5 (9)	117·3 (8)
C6–C12–N2	121·1 (8)	119·0 (8)
C9–C11–C10	123·3 (9)	120·8 (10)
C5–N2–C12	117·5 (7)	117·3 (5)
N2–C12–C11	117·3 (8)	119·0 (6)
C12–C11–C10	122·2 (9)	121·9 (8)
C11–C10–O1	110·5 (9)	108·7 (7)
N2–C5–N1	111·7 (7)	112·1 (6)
N1–S2–O21	105·3 (5)	105·9 (3)
N1–S2–O22	107·9 (4)	107·2 (3)
N1–S2–C31	107·4 (4)	107·3 (3)
C31–S2–O21	108·1 (4)	108·7 (3)
C31–S2–O22	107·0 (5)	106·1 (4)
O21–S2–O22	120·6 (4)	121·0 (3)
S2–C31–C36	120·3 (7)	121·4 (7)
S2–C31–C32	120·3 (8)	118·9 (7)
C36–C31–C32	119·4 (8)	119·7 (9)
C31–C32–C33	118·7 (9)	118·5 (9)
C32–C33–C34	122·0 (8)	121·2 (10)
C33–C34–C35	119·0 (9)	119·7 (12)
C34–C35–C36	119·3 (10)	119·4 (12)
C35–C36–C31	121·6 (8)	121·5 (10)
C37–C34–C33	121·5 (10)	123·6 (14)
C37–C34–C35	119·5 (11)	116·7 (15)
S2–N1–C5	123·2 (5)	122·1 (4)
S2–N1–C14	119·2 (7)	122·0 (5)
C13–C14–C4	119·7 (7)	120·4 (6)
C14–C4–C3	121·1 (9)	119·2 (7)
C4–C3–C2	117·7 (10)	121·0 (8)
C3–C2–C1	122·5 (9)	119·7 (7)
C2–C1–C13	119·3 (9)	120·4 (8)
C1–C13–C14	119·7 (9)	119·2 (7)
C4–C14–N1	121·8 (7)	119·8 (6)
C1–C13–O1	121·9 (8)	121·4 (7)
C5–N1–C14	116·7 (7)	115·6 (6)
N1–C14–C13	118·4 (8)	119·6 (6)
C14–C13–O1	118·4 (7)	119·3 (6)
C13–O1–C10	113·6 (8)	116·4 (7)

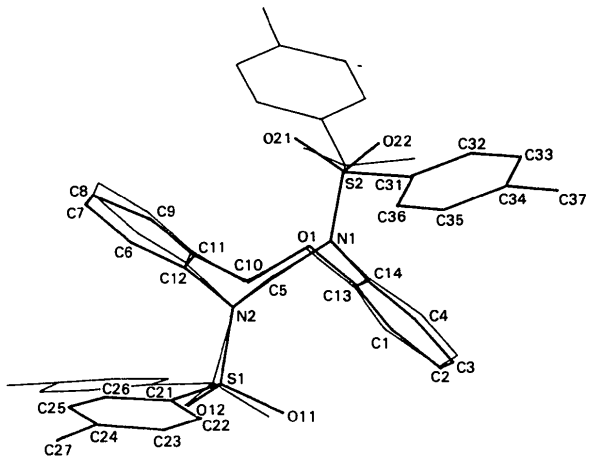

Fig. 1. Comparison of the structures of molecule 1 (bold line) and molecule 2 (fine line). The coincidence of the two molecules was obtained by fitting together the heterocyclic rings of the two molecules by least squares. The atomic numbering scheme is given for molecule 1.

symmetrical with respect to a pseudo-twofold axis running through C5 and the middle of the O1–C10 bond; the asymmetry parameters (Duax & Norton, 1975) $\Delta_2 = 23{\cdot}8$ (6) and 25·0 (5)° for molecules 1 and 2, respectively. The planes of the fused benzene rings form an angle of 24·5 (3)° in 1 and 25·1 (3)° in 2.

The N1 and N2 atoms show different behaviour in the two molecules. The tendency to form pyramidal bonds by the N2 atoms is more pronounced than for N1, particularly for N2 of the second molecule. The sums of the bond angles about the N atoms are: 359·1 (11)° for N1* and 359·7 (9)° for N1 and 356·8 (12)° for N2* and 353·7 (9)° for N2. The distance of N1 from the plane passing through C5, S2 and C14 is 0·082 (12) and 0·047 (14) Å, while that of N2 from the plane passing through C5, S1 and C12 is 0·156 (10) and 0·222 (12) Å, for molecules 1 and 2, respectively.

There are van der Waals molecular contacts only.

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Structure of 5-(Trifluoromethyl)-2′-deoxyuridine

BY J. N. LOW AND P. TOLLIN

Department of Applied Physics, Electronics and Manufacturing Engineering, University of Dundee, Dundee DD1 4HN, Scotland

AND R. ALAN HOWIE

Department of Chemistry, University of Aberdeen, Meston Walk, Old Aberdeen AB9 2UE, Scotland

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Abstract. $C_{10}H_{11}F_3N_2O_5$, $M_r = 296{\cdot}02$u, orthorhombic, $P2_12_12$, $a = 5{\cdot}618$ (4), $b = 23{\cdot}91$ (2), $c = 8{\cdot}897$ (5) Å, $U = 1195{\cdot}1$ Å^3, $Z = 4$, $D_x = 1{\cdot}65$ Mg m^{-3}, Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}15$ mm^{-1}, $F(000) = 608$, $T = 293$ K. $R = 0{\cdot}063$ for 615 unique observed $[F > 5\sigma(F)]$ reflections. The molecule is *anti*, $\chi = -143$ (1)°. The sugar pucker is ${}^2_1T$ with $P = 161$ (1)° and $\psi_m = 42$ (1)°. Atom O5′ is statistically disordered so that the conformation about the C4′–C5′ bond is either $+sc$ or ap, with $\gamma = 67$ (1) and 158 (1)° respectively.

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Introduction. We have determined the crystal and molecular structure of the title compound as part of our continuing program of investigation of modified nucleosides. Another crystal form of this compound has been reported by Tench (1972). This crystallized in space group $P2_1$ with cell dimensions $a = 8{\cdot}97$, $b = 5{\cdot}40$, $c = 12{\cdot}39$ Å, $\beta = 97{\cdot}0$°. The cell volume, 596 Å^3, is almost exactly half that of the present unit cell. No further information with regard to this structure is contained within the Cambridge Structural Database (Elder, Hull, Machin & Mills, 1981).