It is approximately symmetrical with respect to a pseudo-twofold axis running through atoms C2 and C7; $\Delta_2 = 11{\cdot}0$°.

Both N atoms in (VI) and (VII) have sp^3 hybridization. In (VI), both methylsulfonyl groups are equatorial; in (VII), on the contrary, both phenylsulfonyl groups are axial.

In (VI) the conformations of the O11, O12, C11 and O21, O22, C21 atoms with respect to C3, N4, C5 and C6, N7, C8, respectively, are staggered. The weighted least-squares plane calculated for the heterocyclic ring atoms is nearly parallel to the (100) plane, the angle formed by the normal to the best plane and the X axis being 13·6°. The planes of the heterocyclic rings of all the molecules in the crystal are nearly parallel. In (VII) the planes of the two benzene rings form an angle of 23·1 (3)°. The deviations of the S atoms from the benzene-ring planes are 0·13 Å for S1 and 0·04 Å for S2. The angles formed by the plane through the oxadiazecine ring with the planes of the benzene rings attached to S1 and S2 are 22·2 (1) and 19·9 (1)°, respectively.

This work was supported by project R.P.II.10 from the Polish Ministry of Science and Higher Education.

References

BRZEZIŃSKA, E., GLINKA, R. & SZADOWSKA, A. (1988). *Acta Pol. Pharm.* In the press.
BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1971). *ORFFE*. Report ORNL-TM-306. Oak Ridge National Laboratory, Tennessee, USA.
DUAX, W. L. & NORTON, D. A. (1975). *Atlas of Steroid Structure*, Vol. I. New York: IFI/Plenum.
SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Acta Cryst. (1988). C**44**, 2136–2138

Crystal Studies of Heterocyclic Compounds Containing One Oxygen and Two Nitrogen Atoms. VIII. *N,N'*-Ditosyl-7,8,9,14-tetrahydro-2*H*-dibenz[*c,h*][1,5,7]oxadiazecine

BY A. STĘPIEŃ, TOMASZ A. OLSZAK 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 C. BAVOUX

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

(*Received* 29 *October* 1987; *accepted* 20 *June* 1988)

Abstract. $C_{29}H_{28}N_2O_5S_2$, $M_r = 548{\cdot}7$, tetragonal, $P4_12_12$ or $P4_32_12$, $a = 10{\cdot}229$ (2), $c = 26{\cdot}047$ (3) Å, $V = 2725$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}337$ (1) g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 20{\cdot}01$ cm^{-1}, $F(000) = 1152$, room temperature, $R = 0{\cdot}0329$ for 1436 reflections with $I > 3\sigma(I)$. The molecule lies on a twofold axis. The oxadiazecine ring is in a chair conformation, and the tosyl substituents are axial. The two fused benzene rings are nearly parallel.

Introduction. The present paper is a continuation of the study of the relationship between the biological activity and the structure of heterocyclic compounds containing two N and one O atoms in a ring and, at the same time, the first study of a compound having two aromatic rings fused to this ten-membered heterocyclic ring. The title compound was obtained by condensation of *N,N'*-[oxybis(methylenedi-*o*-phenylene)]di-*p*-toluenesulfonamide with methylene bromide (Glinka & Walczyński, 1986). The structure was confirmed by elemental, IR, ^{1}H NMR and mass spectrometry analyses. The NMR spectrum displayed only one signal for the methylene group. The compound shows less neuroleptic activity than the nine-membered analogue, toxicity being much the same (Glinka, 1987).

Experimental. Colourless dipyramidal crystals were prepared by repeated recrystallization from ethanol at room temperature; crystal size 0·1 × 0·2 × 0·3 mm. Diffraction data measured on a CAD-4 diffractometer

0108-2701/88/122136-03$03.00

Table 1. *Final fractional coordinates* (× 10^4) *and equivalent isotropic temperature factors* (Å^2 × 10^4) *with e.s.d.'s in parentheses*

$U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33})$.

	x	y	z	U_{eq}
O1	−389 (2)	−389 (2)	5000	811 (17)
C2	−598 (4)	917 (4)	4819 (1)	745 (22)
C3	190 (3)	1208 (3)	4347 (1)	628 (18)
C4	1205 (3)	2107 (3)	4328 (1)	575 (16)
N5	1486 (2)	2902 (2)	4775 (1)	569 (13)
C6	2790 (3)	2790 (3)	5000	620 (19)
C7	1986 (3)	2226 (4)	3892 (1)	678 (20)
C8	1714 (4)	1461 (4)	3467 (1)	824 (26)
C9	686 (4)	611 (5)	3473 (1)	873 (26)
C10	−60 (4)	474 (4)	3905 (1)	775 (23)
S1	697 (1)	4294 (1)	4840 (0)	646 (4)
O11	−630 (2)	4031 (2)	4713 (1)	820 (15)
O12	1026 (3)	4785 (2)	5338 (1)	878 (19)
C11	1331 (3)	5387 (3)	4385 (1)	587 (17)
C12	933 (3)	5295 (3)	3874 (1)	659 (19)
C13	1463 (4)	6153 (3)	3516 (1)	720 (21)
C14	2371 (4)	7073 (3)	3654 (1)	752 (22)
C15	2757 (4)	7152 (4)	4160 (2)	853 (26)
C16	2238 (4)	6313 (4)	4525 (1)	744 (22)
C141	2977 (7)	7953 (5)	3245 (2)	1109 (37)

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

O1–C2	1·432 (4)	S1–N5	1·646 (2)
C2–C3	1·502 (4)	S1–O11	1·423 (2)
C3–C4	1·388 (4)	S1–O12	1·431 (2)
C4–N5	1·448 (3)	S1–C11	1·753 (3)
N5–C6	1·461 (3)	C11–C12	1·395 (4)
C4–C7	1·395 (4)	C12–C13	1·391 (4)
C7–C8	1·383 (5)	C13–C14	1·370 (5)
C8–C9	1·365 (5)	C14–C15	1·380 (5)
C9–C10	1·368 (5)	C15–C16	1·385 (5)
C10–C3	1·396 (4)	C11–C16	1·375 (4)
		C14–C141	1·526 (6)

C2–O1–C2^i	112·8 (4)	N5–S1–O11	106·3 (1)
O1–C2–C3	112·0 (3)	N5–S1–O12	106·3 (1)
C2–C3–C4	124·1 (3)	N5–S1–C11	107·5 (1)
C3–C4–N5	119·6 (3)	C11–S1–O11	108·5 (1)
C4–N5–C6	117·4 (2)	C11–S1–O12	107·6 (1)
N5–C6–N5^i	107·7 (4)	O11–S1–O12	120·1 (1)
C2–C3–C10	118·0 (3)	S1–C11–C12	119·6 (2)
N5–C4–C7	119·5 (3)	S1–C11–C16	120·6 (2)
C4–C3–C10	117·8 (3)	C16–C11–C12	119·7 (3)
C3–C4–C7	120·9 (3)	C11–C12–C13	118·9 (3)
C4–C7–C8	119·2 (3)	C12–C13–C14	121·5 (3)
C7–C8–C9	120·4 (4)	C13–C14–C15	119·0 (4)
C8–C9–C10	120·3 (4)	C14–C15–C16	120·6 (4)
C9–C10–C3	121·3 (4)	C15–C16–C11	120·3 (3)
S1–N5–C4	118·1 (2)	C141–C14–C13	119·8 (4)
S1–N5–C6	118·3 (2)	C141–C14–C15	121·1 (4)

Symmetry code: (i) $y, x, 1-z$.

using θ–2θ scan technique, graphite-monochromatized Cu $K\alpha$ radiation measured to $[(\sin\theta)/\lambda]_{\max} = 0{\cdot}62$ Å^{-1}, not corrected for absorption. Lattice parameters by least squares using 25 reflections in θ range 7–50°. Ranges of h, k, l : 0 to 12, 0 to 12, 0 to 32, respectively; standard reflection 215, counts variation 3·35%, $R_{\text{int}} = 0{\cdot}0452$. Of 1618 reflections, 1436 were considered observed by the criterion $I \geq 3\sigma(I)$. The structure was solved by Patterson and direct methods using *SHELX*76 (Sheldrick, 1976); refinement carried out by full-matrix least squares using F magnitudes, 229 parameters, with anisotropic thermal parameters for non-H atoms and isotropic for H atoms. All H atoms were located on a difference map. Final $R = 0{\cdot}0329$, unit weights. Max. shift/e.s.d. = 0·32; the largest peak on the final difference map was 0·13 e Å^{-3}. Scattering factors from *SHELX*76. The geometry of the molecule was calculated using *ORFFE*3 (Busing, Martin & Levy, 1971).

Discussion. The final positional parameters are listed in Table 1, interatomic distances and bond angles in Table 2.* Fig. 1 shows the structure of the molecule with the atom-numbering scheme.

The molecule lies on a special position: a crystallographic twofold axis runs through atoms O1 and C6. This confirms the NMR result for the methylene group. The oxadiazecine ring is in a chair conformation. It is approximately symmetrical with respect to a pseudo-mirror plane passing through the middle of C3—C4 and symmetry-related bonds and perpendicular to the twofold axis; the asymmetry parameter (Duax & Norton, 1975) $\Delta_m = 4{\cdot}8\ (5)°$. Thus, the planes of the two fused benzene rings are nearly parallel, the dihedral angle being 172·3 (2)°.

The tosyl substituents are axial. The planes of their benzene rings form a dihedral angle of 148·8 (2)° with the planes of the fused benzene rings.

This work was supported by project R.P.II.10 from the Polish Ministry of Science and Higher Education.

---

* Lists of structure factors, H-atom coordinates, bonds and angles involving H atoms, thermal parameters and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51165 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

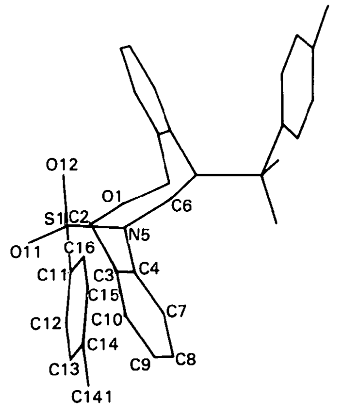

Fig. 1. The structure of the molecule with the atom-numbering scheme.

References

BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1971). *ORFFE*. Report ORNL-TM-306. Oak Ridge National Laboratory, Tennessee, USA.

DUAX, W. L. & NORTON, D. A. (1975). *Atlas of Steroid Structure*, Vol. 1. New York: IFI/Plenum.

GLINKA, R. (1987). 3rd Cyprus Conference on New Methods in Drug Research, Limassol, p. 38.

GLINKA, R. & WALCZYŃSKI, K. (1986). *Acta Pol. Pharm.* **43**, 32–35.

SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Acta Cryst. (1988). C**44**, 2138–2140

Studies on Modified Nucleic Acid Bases: Structure of 3-Isobutyl-1-methylxanthine

BY T. SRIKRISHNAN AND R. PARTHASARATHY*

Center for Crystallographic Research, Roswell Park Memorial Institute, Buffalo, New York 14263, *USA*

(*Received* 12 *April* 1988; *accepted* 18 *July* 1988)

Abstract. $C_{10}H_{14}N_4O_2$, $M_r = 222{\cdot}3$, monoclinic, $P2_1/c$, $a = 4{\cdot}882\,(3)$, $b = 8{\cdot}715\,(1)$, $c = 25{\cdot}955\,(3)$ Å, $\beta = 92{\cdot}28\,(1)°$, $V = 1103{\cdot}4\,(9)$ Å^3, $Z = 4$, $D_m = 1{\cdot}34$, $D_x = 1{\cdot}338$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}5418$ Å, $\mu = 7{\cdot}62$ cm^{-1}, $F(000) = 472$, $T = 294$ K, $R = 0{\cdot}058$ for 984 reflections, $I > 3\sigma(I)$. The isobutyl chain is oriented almost perpendicular to the xanthine ring [C(2)—N(3)—C(31)—C(32) ±99·8 (4)°]. The isobutyl chain torsion angles are N(3)—C(31)—C(32)—C(33) ±62·2 (4) and N(3)—C(31)—C(32)—C(34) ±174·1 (3)°. The structure forms self-paired dimers of xanthine bases with a pair of N—H···O and a pair of weaker C—H···N hydrogen bonds across centers of inversion. There is a partial stacking of the xanthine bases.

Introduction. We have been attempting to crystallize a number of modified bases and nucleosides in the hydrated forms with a view to discovering the underlying reasons that would lead to water molecules being 'sandwiched' between the bases (Parthasarathy, Srikrishnan, Ginell & Guru Row, 1982). In this line of investigation, we obtained crystals of some nucleic acid bases in the anhydrous forms as well. Crystals of 6-methyluracil (m^6U) and 3-isobutyl-1-methylxanthine were two such alkylated bases whose structures were investigated by us (Srikrishnan & Parthasarathy, 1984) with a view to understanding the hydrogen-bonding patterns of alkylated bases. This paper reports the crystal and molecular structure of 3-isobutyl-1-methylxanthine.

Experimental. Crystals were grown from aqueous methanol at room temperature as very fine needles; D_m by flotation (bromoform and benzene); unit-cell parameters on CAD-4 diffractometer using 25 reflections with $14 < \theta < 22°$; crystal of dimensions $0{\cdot}75 \times 0{\cdot}2 \times 0{\cdot}05$ mm, three-dimensional data ($2\theta < 154°$ for Cu $K\alpha$ radiation), $\omega/2\theta$ scan; scan widths calculated using the expression $(0{\cdot}8 + 0{\cdot}4\tan\theta)°$ and aperture widths using $(3{\cdot}0 + 1{\cdot}2\tan\theta)$ mm; max. time spent on any reflection measurement 100 s; faster scan used for strong reflections; intensities of three reflections measured after every hour of exposure; variation of intensities in these three reflections <6% during the course of data collection; crystal orientation checked after every 100 reflections; a total number of 2326 reflections measured out of which 984 were significant [$I > 3\sigma(I)$]; h 0–±6; k 0–11, l 0–32; Lorentz and polarization corrections applied; intensities of three reflections at $\chi \sim 90°$ were measured for different values of φ from 0 to 360° in steps of 10° and the resultant curve of transmission as a function of φ was used to calculate the absorption for all reflections; max. and min. transmission 0·99 and 0·86 with an average of 0·92. Structure solved using *MULTAN* (Germain, Main & Woolfson, 1971); 164 E values ($|E| > 1{\cdot}85$) used as input to *MULTAN* and the correct set with the figure of merit of 1·19 and residual of 0·135 gave all atoms in the molecule; refinement at first with isotropic thermal parameters and later with anisotropic thermal parameters led to an $R = 0{\cdot}089$; difference electron density maps revealed positions of all H atoms; final cycles of refinement with anisotropic thermal parameters for non-H atoms, isotropic for H atoms and extinction-parameter refinement ($g = 6{\cdot}6 \times 10^{-6}$) led to $R = 0{\cdot}058$ for 984 reflections; $wR = 0{\cdot}068$, $S = 2{\cdot}21$, $w[|F_o| - (1/k)|F_c|]^2$ minimized; $w = 4|F_o|^2/\sigma^2(|F_o|)^2$ and $\sigma^2(|F_o|)^2 = [\sigma^2(I) + p^2I^2]/\mathrm{Lp}$ where $p = 0{\cdot}05$, $\sigma(I)$ is standard deviation of intensity I based on counting statistics, k is scale factor; max. and av. $\Delta/\sigma = 0{\cdot}2$ and 0·02; final $|\Delta\rho| = 0{\cdot}2$ e Å^{-3}. Programs and atomic scattering factors as in Enraf–Nonius (1979) *SDP*; Fourier and torsion-angle programs by Dr S. T. Rao and *ORTEP* by Johnson (1965).

---

* To whom correspondence should be addressed.

0108-2701/88/122138-03$03.00