MARSDEN, C. J. & SHELDRICK, G. M. (1971). *J. Mol. Struct.* **10**, 413–418.
MAVRIDIS, A. & MOUSTAKALI-MAVRIDIS, I. (1977). *Acta Cryst.* B**33**, 3612–3615.
NES, G. J. H. VAN & VAN BOLHUIS, F. (1979). *Acta Cryst.* B**35**, 2580–2593.
PAULING, L. (1960). *The Nature of the Chemical Bond*, 3rd ed. Ithaca, New York: Cornell Univ. Press.
SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.
STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

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Structure of Oxodipine:* a New Calcium Antagonist

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Abstract. $C_{19}H_{21}NO_6$, $M_r = 359{\cdot}4$, monoclinic, $P2_1/c$, $a = 15{\cdot}019$ (7), $b = 8{\cdot}285$ (2), $c = 14{\cdot}651$ (6) Å, $\beta = 102{\cdot}6$ (2)°, $V = 1779$ (2) Å^3, $Z = 4$, $D_x = 1{\cdot}342$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 7{\cdot}95$ cm^{-1}, $F(000) = 760$, $T = 293$ K, $R = 0{\cdot}065$ for 2531 observed reflections. The dihydropyridine ring presents a flat-boat conformation. The pentagonal heterocycle has an envelope conformation with the flap at C10. The O—C—C—O part of the five-membered ring is planar and coplanar with the phenyl ring. The methylenedioxyphenyl moiety is nearly perpendicular to the dihydropyridine ring.

Introduction. The vasodilating effect of calcium antagonists finds clinical application, especially in the treatment of oxygen-deficiency diseases of the heart, such as angina pectoris. In recent years, the study of 4-aryldihydropyridinedicarboxylates has undergone a great development; these 4-aryl derivatives are highly active calcium antagonists: oxodipine, studied here, belongs to this group, hence the importance of its structural study.

Experimental. Light-yellow, acicular crystals were kindly supplied by Dr L. Veiga (Departamento de Farmacia Galénica, Universidad Complutense, Madrid). A suitable crystal of approximate size 0·37 × 0·27 × 0·33 mm was mounted on a Philips PW 1100 automatic four-circle diffractometer. Cell dimensions obtained by least-squares refinement from 83 reflections with $12 < 2\theta < 87°$. Intensity data collected for $2 < \theta < 65°$ using graphite-monochromatized Cu $K\alpha$ radiation and ω–2θ-scan technique; two standard reflections (202, $\bar{2}0\bar{2}$) measured every 90 min showed no significant variation in intensity; Lorentz and polarization corrections; no correction for absorption 3385 ($-17 < h < 17$, $0 < k < 10$, $0 < l < 17$) data measured with $\theta_{max} = 65°$. 2531 observed data with $I > 2\sigma(I)$. Structure determined by direct methods, using *MITHRIL* (Gilmore, 1983); all non-H atoms were located in this way. Successive isotropic refinement cycles (on F) showed large temperature values for atoms C19, C23, C24 and O25, which suggests a certain disorder. However, a careful inspection of the Fourier map revealed no alternative peaks for the atoms in question. The positions of the H atoms associated with the disordered C atoms were calculated geometrically; the remaining H atoms were located from a difference map and included in the refinement with the same isotropic temperature factors as the atoms to which they are bonded. Several cycles of full-matrix, mixed least-squares refinement of all non-H atoms treated anisotropically, H atoms fixed, were performed; at this stage, an empirical weighting scheme (Martínez-Ripoll & Cano, 1975) was applied to give similar values of $w\Delta^2$ over ranges of $\sin\theta/\lambda$ and F_o. Final $R = 0{\cdot}065$, $wR = 0{\cdot}083$, $S = 8{\cdot}5$; $(\Delta/\sigma)_{max} = 0{\cdot}1$; max. height in final difference Fourier synthesis 0·74 e Å^{-3}, min. height −0·41 e Å^{-3}; atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Calculations carried out using *XRAY*70 (Stewart, Kundell & Baldwin, 1970) and *PARST* (Nardelli, 1983).

Discussion. Final parameters are given in Table 1.† The identification of the atoms and a perspective molecular drawing of oxodipine are shown in Fig. 1. Bond lengths

* Ethyl methyl 2,6-dimethyl-4-(2,3-methylenedioxyphenyl)-1,4-dihydro-3,5-pyridinedicarboxylate.

† 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 43205 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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and bond angles are given in Table 2. The molecule shows close conformational similarity to 3,5-diacetyl-2,6-dimethyl-4-(3-pyridyl)-1,4-dihydropyridine (Krajewski, Urbanczyk-Lipkowska & Gluzinski, 1977), diethyl 2,6-dimethyl-4-phenyl-1,4-dihydro-3,5-pyridinedicarboxylate (Hempel & Gupta, 1978) and diethyl 2,4,6-trimethyl-1,4-dihydro-3,5-pyridinedicarboxylate (Fortier, Fraser, Moore, Park, Whitney & Marks, 1985); N1 and C4 lie at the same side with respect to the plane formed by C2, C3, C5 and C6, so the dihydropyridine ring has a flat-boat conformation; all alkyl and alkoxycarbonyl substituents are on the opposite side. From this result and those of previous works (Karle, 1961; Lenstra, Petit, Dommisse & Alderweireldt, 1979) one can deduce that C4-unsubstituted dihydropyridines are planar, while the C4-substituted ones adopt flat-boat conformations (Krajewski *et al.*, 1977; Hempel & Gupta, 1978; Fortier *et al.*, 1985). The five-membered ring is an almost perfect envelope with the flap at C10; this ring is coplanar with the phenyl one [dihedral angle 2·4 (1)°]; the methylenedioxyphenyl moiety is nearly perpendicular to the dihydropyridine ring [88·38 (9)°]. As a consequence of disorder, the C23—C24 distance appears considerably shortened [1·273 (12) Å]. The remaining interatomic distances and angles are close to those found in the works previously mentioned.

The molecules are packed forming chains along **a** (Fig. 2); these chains are held together by the hydrogen bond N1···O20(x, $-y+\frac{1}{2}$, $z+\frac{1}{2}$) of 3·105 (4) Å [similar values found in Eggleston, Chodosh, Jain, Kaiser & Ackerman (1985) and Lee, Lee, Juang & Chung (1985)] and by van der Waals forces.

From the structural study of oxodipine and according to the work of Bossert, Meyer & Wehinger (1981), this compound presents all the structural characteristics that confer an optimum biological activity on this type of compound.

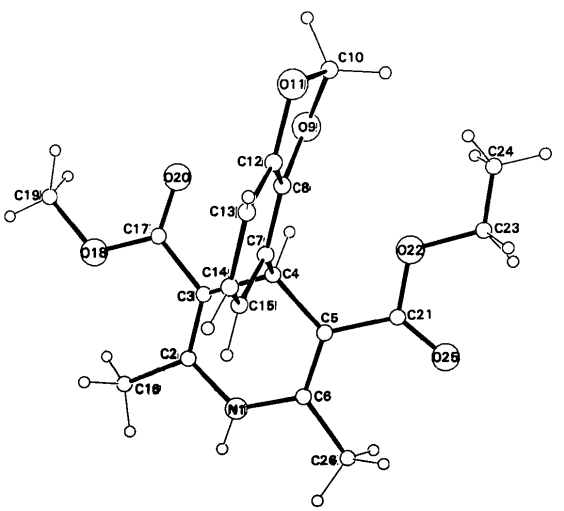

Fig. 1. A view of the molecule showing the atomic numbering.

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å² × 10⁴)

$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j(\cos a_i, a_j)$.

	x	y	z	U_{eq}
N1	0·2023 (2)	0·2876 (4)	0·8924 (2)	507 (9)
C2	0·1383 (2)	0·2753 (4)	0·8096 (2)	447 (9)
C3	0·1604 (2)	0·3217 (3)	0·7289 (2)	392 (9)
C4	0·2531 (2)	0·3965 (3)	0·7282 (2)	384 (8)
C5	0·3211 (2)	0·3671 (4)	0·8203 (2)	444 (9)
C6	0·2935 (2)	0·3214 (4)	0·8982 (2)	470 (10)
C7	0·2421 (2)	0·5761 (4)	0·7073 (2)	396 (8)
C8	0·2632 (2)	0·6474 (4)	0·6304 (2)	433 (9)
O9	0·2956 (2)	0·5746 (3)	0·5592 (2)	586 (8)
C10	0·3216 (3)	0·7061 (6)	0·5073 (3)	677 (14)
O11	0·2741 (2)	0·8450 (4)	0·5270 (2)	700 (10)
C12	0·2507 (2)	0·8105 (4)	0·6110 (2)	534 (11)
C13	0·2177 (3)	0·9128 (5)	0·6680 (3)	659 (14)
C14	0·1966 (3)	0·8444 (5)	0·7470 (3)	645 (13)
C15	0·2079 (2)	0·6813 (4)	0·7664 (2)	508 (10)
C16	0·0489 (3)	0·2076 (6)	0·8222 (3)	657 (14)
C17	0·1012 (2)	0·3070 (4)	0·6367 (2)	445 (9)
O18	0·0130 (2)	0·2858 (5)	0·6361 (2)	775 (11)
C19	−0·0477 (3)	0·2615 (9)	0·5464 (3)	1003 (23)
O20	0·1272 (2)	0·3173 (4)	0·5646 (1)	589 (8)
C21	0·4178 (2)	0·3921 (5)	0·8232 (3)	620 (13)
O22	0·4339 (2)	0·4207 (5)	0·7397 (2)	803 (12)
C23	0·5287 (3)	0·4473 (12)	0·7371 (5)	1225 (32)
C241	0·5431 (5)	0·4426 (15)	0·6547 (7)	1575 (47)
O25	0·4802 (2)	0·3863 (7)	0·8908 (2)	1096 (17)
C26	0·3514 (3)	0·2978 (5)	0·9940 (2)	618 (12)

Table 2. *Bond distances* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

N1—C2	1·376 (4)	C8—C12	1·386 (5)
N1—C6	1·381 (4)	O9—C10	1·431 (5)
C2—C3	1·353 (4)	C10—O11	1·416 (6)
C2—C16	1·503 (5)	O11—C12	1·381 (5)
C3—C4	1·526 (4)	C12—C13	1·358 (6)
C3—C17	1·450 (4)	C13—C14	1·386 (6)
C4—C5	1·523 (4)	C14—C15	1·384 (5)
C4—C7	1·520 (4)	C17—O18	1·334 (4)
C5—C6	1·351 (5)	C17—O20	1·207 (4)
C5—C21	1·458 (5)	O18—C19	1·441 (4)
C6—C26	1·495 (4)	C21—O22	1·320 (5)
C7—C8	1·370 (4)	C21—O25	1·206 (4)
C7—C15	1·403 (5)	O22—C23	1·450 (6)
C8—O9	1·382 (4)	C23—C24	1·273 (12)

C2—N1—C6	124·1 (2)	C7—C8—O9	128·0 (3)
N1—C2—C16	113·0 (3)	O9—C8—C12	109·0 (3)
N1—C2—C3	119·4 (3)	C8—O9—C10	104·5 (3)
C3—C2—C16	127·6 (3)	O9—C10—O11	107·6 (3)
C2—C3—C17	124·9 (3)	C10—O11—C12	104·6 (3)
C2—C3—C4	121·3 (2)	C8—C12—O11	109·6 (3)
C4—C3—C17	113·7 (2)	O11—C12—C13	128·2 (3)
C3—C4—C7	109·8 (2)	C8—C12—C13	122·3 (3)
C3—C4—C5	111·4 (2)	C12—C13—C14	116·1 (4)
C5—C4—C7	110·9 (2)	C13—C14—C15	122·0 (4)
C4—C5—C21	118·3 (3)	C7—C15—C14	121·7 (3)
C4—C5—C6	121·5 (3)	C3—C17—O20	123·9 (3)
C6—C5—C21	120·2 (3)	C3—C17—O18	115·0 (2)
N1—C6—C5	119·3 (3)	O18—C17—O20	121·0 (3)
C5—C6—C26	127·4 (3)	C17—O18—C19	117·0 (3)
N1—C6—C26	113·3 (3)	C5—C21—O25	127·5 (3)
C4—C7—C15	121·5 (3)	C5—C21—O22	112·4 (3)
C4—C7—C8	123·6 (3)	O22—C21—O25	120·1 (3)
C8—C7—C15	114·9 (3)	C21—O22—C23	115·6 (3)
C7—C8—C12	123·0 (3)	O22—C23—C24	113·1 (6)

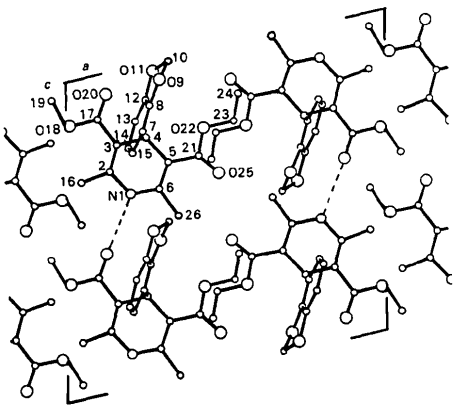

Fig. 2. Molecular packing viewed down **b**; hydrogen bonds are marked with dashed lines.

References

BOSSERT, F., MEYER, H. & WEHINGER, E. (1981). *Angew. Chem. Int. Ed. Engl.* **20**, 762–769.

EGGLESTON, D. S., CHODOSH, D. F., JAIN, T., KAISER, C. & ACKERMAN, D. M. (1985). *Acta Cryst.* C**41**, 76–82.

FORTIER, S., FRASER, M. E., MOORE, N. J., PARK, Y. S., WHITNEY, R. A. & MARKS, G. S. (1985). *Acta Cryst.* C**41**, 411–413.

GILMORE, C. J. (1983). *MITHRIL. A Computer Program for the Automatic Solution of Crystal Structures from X-ray Data.* Univ. of Glasgow, Scotland.

HEMPEL, A. & GUPTA, M. P. (1978). *Acta Cryst.* B**34**, 3815–3817.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

KARLE, I. L. (1961). *Acta Cryst.* **14**, 497–502.

KRAJEWSKI, J., URBANCZYK-LIPKOWSKA, Z. & GLUZINSKI, P. (1977). *Acta Cryst.* B**33**, 2967–2969.

LEE, T.-J., LEE, T.-Y., JUANG, W.-B. & CHUNG, C.-S. (1985). *Acta Cryst.* C**41**, 1596–1598.

LENSTRA, A. T. H., PETIT, G. H., DOMMISSE, R. A. & ALDERWEIRELDT, F. C. (1979). *Bull. Soc. Chim. Belg.* **88**(3), 133–141.

MARTÍNEZ-RIPOLL, M. & CANO, F. H. (1975). *PESOS.* Instituto 'Rocasolano', CSIC, Madrid, Spain.

NARDELLI, M. (1983). *Comput. Chem.* **7**, 95–98.

STEWART, J. M., KUNDELL, F. A. & BALDWIN, J. C. (1970). The *XRAY*70 system. Computer Science Center, Univ. of Maryland, College Park, Maryland.

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Structure of 3,7-Dibromo-10-ethylphenothiazine

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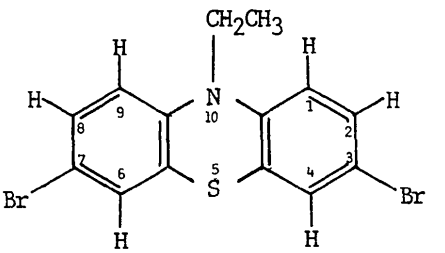
Abstract. $C_{14}H_{11}Br_2NS$, $M_r = 385{\cdot}1$, triclinic, $P\bar{1}$, $a = 8{\cdot}403\ (2)$, $b = 11{\cdot}405\ (2)$, $c = 15{\cdot}807\ (3)$ Å, $\alpha = 73{\cdot}93\ (1)$, $\beta = 102{\cdot}28\ (2)$, $\gamma = 83{\cdot}56\ (1)°$, $V = 1398{\cdot}6\ (4)$ Å^3, $Z = 4$, $D_x = 1{\cdot}829$ g cm^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}70926$ Å, $\mu = 57{\cdot}86$ cm^{-1}, $F(000) = 752$, $T = 295$ K. Final $R = 0{\cdot}047$ for 3060 observed reflections. The folding angle between the benzo planes is 153·6 (2) and 145·3 (2)° for the two crystallographically independent molecules. The ethyl groups occupy quasi-equatorial positions relative to the central ring in both molecules; however, the configuration of the ethyl group is different in the two molecules.

Introduction. This paper reports the synthesis and structure of 3,7-dibromo-10-ethylphenothiazine (I) and is part of our continuing structural studies of substituted phenothiazines (Jovanovic, Biehl, de Meester & Chu, 1984). The objective of these studies is to determine the effect of the different substituents on the conformation and configuration of the phenothiazine ring system.



(I)

Experimental. The title compound was prepared by addition of 672 mg (2·1 mmol) of pyridium hydrobromide perbromide to a stirred solution of 227 mg

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