Acta Cryst. (1984). C**40**, 1866–1869

Structure of Oleandrigenin Monohydrate, $C_{25}H_{37}O_6.H_2O$

BY KUANTEE GO AND GOPINATH KARTHA

Roswell Park Memorial Institute, 666 Elm Street, Buffalo, NY 14263, USA

(*Received* 9 *March* 1984; *accepted* 6 *June* 1984)

Abstract. $M_r = 451{\cdot}59$, monoclinic, $P2_1$, $a = 17{\cdot}878$ (1), $b = 7{\cdot}671$ (1), $c = 19{\cdot}531$ (3) Å, $\beta = 108{\cdot}26$ (1)°, $V = 2543{\cdot}6$ Å^3, $Z = 4$, $D_x = 1{\cdot}179$, $D_m = 1{\cdot}241$ Mg m^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}5418$ Å, $\mu = 0{\cdot}57$ mm^{-1}, $F(000) = 980$, room temperature, $R_w = 5{\cdot}5\%$ for 4608 reflections, 5025 unique reflections measured. There are two molecules of oleandrigenin and two molecules of water in the asymmetric unit. The *D* ring of the cardiac steroid has a 14β-envelope conformation for molecule (1), while in molecule (2) it has a 13α,14β-half-chair conformation. The lactone rings in the two independent molecules have slightly different positions relative to ring *D*. The hydroxyls in the aglycone along with the carbonyl O and the solvent O atoms are involved in an extensive hydrogen-bonding scheme stabilizing the structure.

Introduction. Oleandrigenin is the aglycone of oleandrin, a cardiac glycoside isolated from *Nerium oleander* (Neumann, 1937; Tschesche, 1937; Hesse, 1937). We have obtained oleandrigenin by acid hydrolysis of oleandrin (Neumann, 1937), a commercially available anhydrous oleandrin purchased from Sigma Chemical Co. Suitable crystals were obtained by recrystallization from ethanol and the structure was determined as an extension of our studies on the structure and conformation of cardiac glycosides and their genins.

Experimental. Crystal 0·3 × 0·4 × 0·5 mm. Enraf–Nonius CAD-4 automated diffractometer, Ni-filtered Cu $K\alpha$. Lattice dimensions by least-squares fit to a set of 25 reflections measured in θ range 9–36°. ω–2θ scans and integrated counts with $\theta < 77°$. 5025 independent reflections, 4608 with $I > 2\sigma(I)$. Three standard reflections (overall $\sigma = 0{\cdot}01$). Lp correction, empirical (one parameter, φ) absorption correction (range 0·95–1·08). Direct methods (*MULTAN*, Germain, Main & Woolfson, 1971). Anisotropic block-diagonal least-squares refinement for 64 non-hydrogen atoms; 78 H atoms (52 calculated, 26 from ΔF synthesis) included in the structure factor calculations but not refined; $R = 0{\cdot}050$, $R_w = 0{\cdot}055$ for 4608 reflections (except for the disordered solvents, which were refined isotropically, $\Delta/\sigma < 0{\cdot}2$); $\sum w(|F_o| - |F_c|)^2$ minimized where $w^{-1} = [\sigma^2(F) + (cF)^2]$; $\Delta\rho_{max} = 0{\cdot}2$ e Å^{-3}. f curves from *International Tables for X-ray Crystallography* (1962). Enraf–Nonius *SDP* package and local programs.

Discussion. The final parameters are given in Table 1;* standard deviations were calculated using Cruickshank's (1965) expressions.

Fig. 1 shows the numbering of the atoms and the ring designations. The *A* and *D* rings are *cis* with respect to the fused *B* and *C* rings, as are those of other cardiac active glycosides. The rings *A*, *B* and *C* have flattened chair conformations. The *D* ring has a distorted 14β-envelope conformation in molecule (1) [with pseudorotation parameters $\varphi_m = 40°$ and $\Delta = 28°$ (Altona, Geise & Romers, 1968)] and a distorted 13α,14β-half-chair conformation in molecule (2) (with $\varphi_m = 38°$ and $\Delta = 6°$).

The bond lengths and angles, not corrected for thermal vibration, are given in Table 2. The C(sp^3)–C(sp^3) bonds range from 1·507 (3) to 1·575 (3) Å with a mean value of 1·538 Å for both molecules. The longest bonds are C(9)–C(10) [1·567 (3) and 1·563 (2) Å for molecules (1) and (2) respectively] and C(13)–C(17) [1·575 (3) and 1·557 (3) Å

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, torsion angles and a stereoscopic view of the molecule have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39597 (32 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

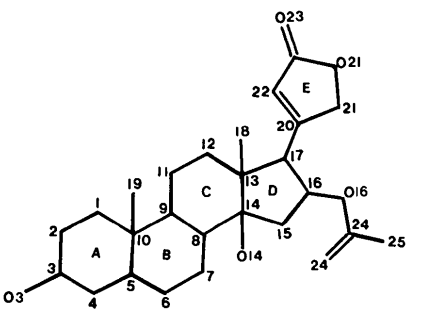

Fig. 1. Numbering of atoms and ring designations in oleandrigenin.

0108-2701/84/111866-04$01.50

respectively]. Unlike oleandrin, which has a long C(16)–C(17) bond of 1·568 (3) Å, this bond here has values of 1·549 (3) and 1·538 (3) Å – consistently shorter for both molecules in oleandrigenin.

Table 1. *Fractional coordinates* ($\times 10^4$) *and isotropic thermal parameters*

$B_{eq} = 8\pi^2 U_{eq}$ where $U_{eq} = \frac{1}{3}\sum_i \sum_j a^*_i a^*_j (\mathbf{a}_i . \mathbf{a}_j) U_{ij}$.

	x	y	z	B_{eq}(Å^2)
Molecule (1)				
C(1)	943 (1)	6056†	5611 (1)	3·4
C(2)	1312 (1)	7812 (4)	5589 (1)	4·2
C(3)	2034 (1)	8092 (4)	6243 (1)	4·3
C(4)	1855 (1)	7737 (4)	6940 (1)	3·7
C(5)	1462 (1)	5979 (3)	6955 (1)	2·9
C(6)	1286 (1)	5717 (4)	7668 (1)	3·4
C(7)	615 (1)	6909 (3)	7726 (1)	3·0
C(8)	−131 (1)	6667 (3)	7084 (1)	2·3
C(9)	49 (1)	6959 (3)	6366 (1)	2·4
C(10)	715 (1)	5697 (3)	6304 (1)	2·7
C(11)	−718 (1)	6837 (4)	5718 (1)	3·1
C(12)	−1341 (1)	8109 (3)	5807 (1)	3·0
C(13)	−1567 (1)	7756 (3)	6495 (1)	2·3
C(14)	−811 (1)	7824 (3)	7156 (1)	2·2
C(15)	−630 (1)	9757 (3)	7267 (1)	2·5
C(16)	−1435 (1)	10657 (3)	7090 (1)	2·6
C(17)	−2052 (1)	9377 (3)	6615 (1)	2·6
C(18)	−2024 (1)	6055 (3)	6404 (1)	3·2
C(19)	464 (1)	3779 (4)	6271 (1)	3·8
C(20)	−2764 (1)	8998 (3)	6848 (1)	2·9
C(21)	−2771 (1)	8319 (4)	7568 (1)	3·5
C(22)	−3490 (1)	9155 (4)	6428 (1)	4·1
C(23)	−4025 (1)	8616 (4)	6827 (1)	4·4
C(24)	−1376 (1)	12538 (3)	8073 (1)	3·0
C(25)	−1579 (2)	12703 (4)	8757 (1)	4·6
O(3)	2689 (1)	7016 (4)	6207 (1)	5·1
O(14)	−997 (1)	7285 (2)	7791 (1)	2·4
O(16)	−1616 (1)	11008 (2)	7751 (1)	2·9
O(21)	−3598 (1)	8129 (3)	7487 (1)	3·8
O(23)	−4738 (1)	8584 (4)	6633 (1)	6·7
O(24)	−1021 (1)	13588 (3)	7840 (1)	4·1
Molecule (2)				
C(1′)	2020 (1)	3579 (3)	10112 (1)	3·0
C(2′)	1288 (1)	4020 (3)	9478 (1)	3·2
C(3′)	801 (1)	2428 (4)	9178 (1)	3·4
C(4′)	1304 (1)	969 (4)	9021 (1)	3·2
C(5′)	2029 (1)	536 (3)	9668 (1)	2·8
C(6′)	2497 (1)	−981 (4)	9498 (1)	3·7
C(7′)	2896 (1)	−417 (3)	8934 (1)	3·3
C(8′)	3406 (1)	1209 (3)	9179 (1)	2·4
C(9′)	2942 (1)	2716 (3)	9382 (1)	2·3
C(10′)	2549 (1)	2147 (3)	9958 (1)	2·6
C(11′)	3473 (1)	4321 (3)	9609 (1)	3·1
C(12′)	3812 (1)	4856 (3)	9007 (1)	3·0
C(13′)	4309 (1)	3410 (3)	8810 (1)	2·5
C(14′)	3800 (1)	1721 (3)	8610 (1)	2·3
C(15′)	3238 (1)	2135 (3)	7855 (1)	2·8
C(16′)	3685 (1)	3384 (3)	7506 (1)	2·8
C(17′)	4441 (1)	3973 (3)	8090 (1)	2·7
C(18′)	5069 (1)	3159 (4)	9436 (1)	3·7
C(19′)	3162 (1)	1735 (4)	10689 (1)	4·1
C(20′)	5197 (1)	3428 (3)	7959 (1)	2·8
C(21′)	5528 (1)	1615 (4)	7972 (2)	4·2
C(22′)	5669 (1)	4523 (4)	7772 (1)	3·6
C(23′)	6324 (1)	3546 (4)	7659 (1)	3·8
C(24′)	3336 (2)	2445 (4)	6294 (1)	4·5
C(25′)	3616 (3)	1490 (7)	5754 (2)	7·1
O(3′)	435 (1)	1845 (3)	9692 (1)	4·3
O(14′)	4329 (1)	359 (2)	8560 (1)	3·0
O(16′)	3888 (1)	2531 (3)	6925 (1)	3·6
O(21′)	6246 (1)	1865 (3)	7795 (1)	4·4
O(23′)	6879 (1)	4044 (4)	7480 (2)	6·0
O(24′)	2682 (2)	3009 (5)	6187 (2)	7·4
Water oxygens				
O(*W*1)	4047 (1)	8117 (3)	7327 (1)	4·6
O(*W*2)	−295 (2)	−1216 (4)	9192 (1)	6·0

† Held fixed to define the origin in $P2_1$.

As shown in Fig. 2, the rings *A*, *B*, *C* and *D* of the two molecules in oleandrigenin and oleandrin are almost superimposable in spite of the differences in the orientations of the *E* ring and the acetate at C(16). It should be noted that oleandrin, which has a torsion angle C(13)–C(17)–C(20)–C(22) of 65·6 (5)°, has its acetate group under the lactone ring *E*, while oleandrigenin with this torsion angle −106·8 (5) and −128·3 (5)° has the acetate group between the OH substituent at C(14) and the lactone ring.

Table 3 lists C–H···O distances between the lactone and the OH group in the *D* ring of the steroid nucleus. Distances less than 3·22 Å are underlined. Owing to the presence of C(20)=C(22) and C(23)=O(23) in the lactone ring, C(17)–C(20), C(20)–C(21), C(22)–C(23) and C(21)–O(21) are shortened (Table 2). The short distances observed in Table 3 could be due to the interactions between the H and the π cloud created by the conjugated system.

A list of H bonds in oleandrigenin is given in Table 4. The bonds are also shown in Fig. 3 where they are indicated by dotted lines except for O(14)···O(24) which overlaps in the drawing. There are two molecules

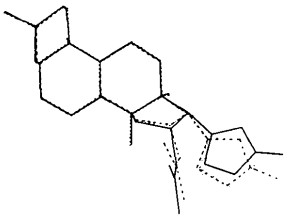

Fig. 2. Superposition of the fused *B* and *C* rings [best plane through atoms C(5), C(6), C(7), C(8), C(9), C(10), C(11), C(12), C(13) and C(14)] of molecules (1) and (2) of oleandrigenin and oleandrin indicated by ————, ·-·-·-·- and ····· respectively.

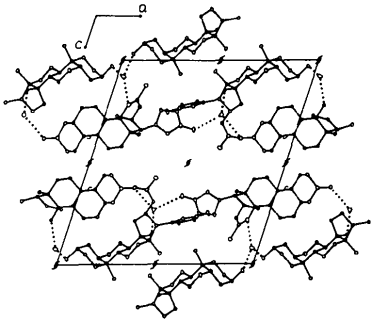

Fig. 3. Packing of molecules in the unit cell (viewed down **b**) with H bonding indicated by dotted lines.

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

	Molecule (1)	Molecule (2)		Molecule (1)	Molecule (2)		Molecule (1)	Molecule (2)
C(1)–C(2)	1·507 (4)	1·533 (3)	C(11)–C(12)	1·531 (3)	1·537 (3)	C(20)–C(22)	1·306 (3)	1·321 (3)
C(2)–C(3)	1·520 (4)	1·507 (3)	C(12)–C(13)	1·545 (2)	1·544 (3)	C(22)–C(23)	1·468 (3)	1·464 (3)
C(3)–C(4)	1·517 (3)	1·525 (3)	C(13)–C(14)	1·549 (2)	1·561 (3)	C(24)–C(25)	1·494 (3)	1·495 (5)
C(4)–C(5)	1·525 (3)	1·537 (3)	C(8)–C(14)	1·547 (2)	1·542 (2)	C(3) O(3)	1·452 (3)	1·431 (3)
C(5)–C(10)	1·545 (2)	1·543 (3)	C(14)–C(15)	1·519 (3)	1·536 (3)	C(14)–O(14)	1·441 (2)	1·433 (2)
C(10)–C(1)	1·556 (3)	1·539 (3)	C(15)–C(16)	1·536 (2)	1·536 (3)	C(16)–O(16)	1·449 (2)	1·452 (3)
C(5)–C(6)	1·533 (3)	1·530 (3)	C(16)–C(17)	1·549 (3)	1·538 (3)	C(21)–O(21)	1·444 (2)	1·444 (2)
C(6)–C(7)	1·541 (3)	1·549 (3)	C(17)–C(13)	1·575 (3)	1·557 (3)	C(23)–O(21)	1·331 (3)	1·333 (4)
C(7)–C(8)	1·529 (2)	1·530 (3)	C(10)–C(19)	1·534 (4)	1·536 (3)	C(24)–O(16)	1·337 (3)	1·317 (3)
C(8)–C(9)	1·552 (2)	1·545 (3)	C(13)–C(18)	1·521 (3)	1·529 (3)	C(24)–O(24)	1·200 (3)	1·201 (5)
C(9)–C(10)	1·567 (3)	1·563 (2)	C(17)–C(20)	1·509 (2)	1·512 (2)	C(23)–O(23)	1·212 (2)	1·212 (3)
C(9)–C(11)	1·550 (2)	1·533 (3)	C(20)–C(21)	1·504 (3)	1·508 (4)			
C(10)–C(1)–C(2)	114·8 (3)	114·9 (3)	C(14)–C(15)–C(16)	105·3 (3)	106·2 (3)	C(2)–C(3)–O(3)	111·3 (4)	108·4 (3)
C(1)–C(2)–C(3)	111·5 (3)	112·2 (3)	C(15)–C(16)–C(17)	106·9 (3)	108·2 (3)	C(4)–C(3)–O(3)	109·9 (3)	109·8 (3)
C(2)–C(3)–C(4)	111·5 (4)	111·6 (3)	C(16)–C(17)–C(13)	105·5 (3)	104·0 (3)	C(8)–C(14)–O(14)	108·2 (2)	108·9 (3)
C(3)–C(4)–C(5)	113·9 (3)	113·0 (3)	C(17)–C(13)–C(14)	103·1 (2)	104·2 (3)	C(13)–C(14)–O(14)	109·3 (2)	106·1 (3)
C(4)–C(5)–C(10)	113·1 (3)	112·6 (3)	C(20)–C(21)–O(21)	103·9 (3)	104·3 (3)	C(15)–C(14)–O(14)	104·6 (2)	109·4 (3)
C(5)–C(10)–C(1)	107·2 (3)	107·9 (3)	C(21)–O(21)–C(23)	109·5 (3)	109·5 (3)	C(8)–C(14)–C(15)	115·8 (3)	115·8 (3)
C(5)–C(6)–C(7)	112·1 (3)	110·3 (3)	O(21) C(23)–C(22)	108·8 (4)	109·1 (3)	C(15)–C(16)–O(16)	109·7 (3)	111·0 (3)
C(6)–C(7)–C(8)	111·5 (3)	111·6 (3)	C(23)–C(22)–C(20)	109·0 (4)	109·0 (3)	C(17)–C(16)–O(16)	109·6 (3)	109·6 (3)
C(7)–C(8)–C(9)	110·4 (3)	112·0 (3)	C(22)–C(20)–C(21)	108·7 (3)	108·1 (3)	C(13)–C(17)–C(20)	116·6 (3)	118·1 (3)
C(8)–C(9)–C(10)	111·0 (3)	111·9 (3)	C(22)–C(23)–O(23)	129·5 (5)	130·3 (4)	C(16)–C(17)–C(20)	116·8 (3)	114·7 (3)
C(9)–C(10)–C(5)	109·4 (3)	108·4 (3)	O(21)–C(23)–O(23)	121·6 (4)	120·5 (4)	C(17)–C(20)–C(21)	127·1 (3)	128·2 (3)
C(10)–C(5)–C(6)	111·0 (3)	112·4 (3)	C(1)–C(10)–C(19)	106·6 (3)	105·6 (3)	C(17)–C(20)–C(22)	124·0 (3)	123·6 (3)
C(8)–C(9)–C(11)	110·3 (3)	110·0 (3)	C(5)–C(10)–C(19)	109·5 (3)	110·4 (3)	C(16)–O(16)–C(24)	117·0 (3)	117·2 (3)
C(9)–C(11)–C(12)	111·0 (3)	110·5 (3)	C(9)–C(10)–C(19)	112·2 (3)	112·1 (3)	O(16)–C(24)–C(25)	111·3 (3)	111·5 (4)
C(11)–C(12)–C(13)	112·6 (3)	113·2 (3)	C(1)–C(10)–C(9)	111·7 (3)	112·6 (3)	O(16)–C(24)–O(24)	122·0 (4)	123·2 (5)
C(12)–C(13)–C(14)	108·8 (3)	108·8 (3)	C(12)–C(13)–C(18)	109·0 (3)	109·1 (3)	O(24)–C(24)–C(25)	125·9 (4)	125·2 (5)
C(13)–C(14)–C(8)	114·2 (3)	113·2 (3)	C(14)–C(13)–C(18)	115·0 (3)	113·4 (3)	C(4)–C(5)–C(6)	111·2 (3)	111·3 (3)
C(14)–C(8)–C(9)	113·0 (3)	113·4 (3)	C(17)–C(13)–C(18)	113·2 (3)	114·1 (3)	C(7)–C(8)–C(14)	111·4 (3)	110·1 (3)
C(13)–C(14)–C(15)	104·1 (2)	102·9 (3)	C(12)–C(13)–C(17)	107·3 (3)	106·7 (3)	C(10)–C(9)–C(11)	113·5 (3)	113·2 (3)

Table 3. C–H···O *distances* (Å) *and angles* (°) *in some cardiac active glycosides* (*distances less than* 3·220 Å *are underlined*)

	C(21)···O(14)	C(22)···O(14)	C(21)···O(16)	C(22)···O(16)	H···O	Angle C–H···O
Digitoxigenin[1]	4·763 Å	3·222	*	*	2·624	115·3
Digoxigenin[2]	3·322	4·636	*	*	2·456	138·3
Digoxin[3]	3·502	4·862	*	*	2·763	127·3
5β-Hydroxy-gitoxigenin[4]	5·401	4·037	3·978	3·122	3·067	95·6
Gitoxin[5]	2·821 (*a*)	4·483	3·208 (*b*)	3·471	2·327	107·6 (*a*)
					2·681	110·0 (*b*)
Digoxigenin monodigitoxoside[6]	2·101	4·378	*	*	2·466	128·3
Digoxigenin bisdigitoxoside[7]	3·682	4·922	*	*	3·052	119·7
Ouabain[8]	2·954	4·585	*	*	2·480	104·1
Ouabagenin[9]	3·189	4·637	*	*	2·698	112·3
Oleandrin[10]	4·722	3·049 (*a*)	3·849	2·993 (*b*)	2·470	117·3 (*a*)
					2·978	98·6 (*b*)
Oleandrigenin[11] [molecule (1)]	3·165 (*a*)	4·645	2·862 (*b*)	3·808	2·587	113·0 (*a*)
					2·242	115·0 (*b*)
Oleandrigenin[11] [molecule (2)]	2·895 (*a*)	4·541	3·085 (*b*)	3·452	2·443	104·2 (*a*)
					2·535	95·0 (*b*)

References: (1) Karle & Karle (1969); (2) Go & Kartha (1979); (3) Go, Kartha & Chen (1980); (4) Przybylska & Ahmed (1979); (5) Go & Kartha (1980); (6) Go & Kartha (1982); (7) Go & Kartha (1982); (8) Go & Kartha (1981); (9) Go & Kartha (1983); (10) Kartha & Go (1981); (11) this paper.

Note: where two distances for the same compound are underlined, (*a*) refers to the first set, (*b*) to the second.

* Not applicable.

Table 4. *Hydrogen bonding in oleandrigenin*

D–H···*A*	*D*···*A*	*D*–H	H···*A*	*D*–H···*A*	Symmetry operator of the second molecule		
O(*W*1)···O(3)	2·841 (3) Å	0·764 Å	2·186 Å	144·2°	*x*	*y*	*z*
O(*W*1)···O(23)	2·920 (3)	0·870	2·122	152·1	*x*−1	*y*	*z*
O(14′)···O(*W*1)	2·872 (3)	0·732	2·143	173·9	*x*	*y*−1	*z*
O(14)···O(24)	2·839 (3)	0·693	2·162	165·3	*x*	*y*−1	*z*
O(*W*2)···O(14)	2·869 (3)	0·664	2·245	158·2	*x*	*y*−1	*z*
O(3′)···O(*W*2)	2·716 (3)	0·662	2·074	163·8	*x*	*y*	*z*
O(*W*2)···O(3′)	2·714 (3)	0·827	2·075	133·6	−*x*	*y*−½	2−*z*

of oleandrigenin and two molecules of water in the asymmetric unit. In the difference Fourier maps, there are indications of additional disordered molecules of water between layers of oleandrigenin molecules along the *c* axis; but their locations are not very clear. This is also indicated in the density measurement where the observed value is significantly higher than the calculated value for two molecules of water in the asymmetric unit. The disordered water may serve in loosely holding the molecules in the *c*-axis direction through H bonds.

This work was supported by the New York State Department of Health.

References

ALTONA, C., GEISE, H. J. & ROMERS, C. (1968). *Tetrahedron*, **24**, 13–32.

CRUICKSHANK, D. W. J. (1965). In *Computing Methods in Crystallography*. Oxford: Pergamon Press.

GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

GO, K. & KARTHA, G. (1979). Unpublished results.

GO, K. & KARTHA, G. (1980). *Acta Cryst.* B**36**, 3034–3040.

GO, K. & KARTHA, G. (1981). *Cryst. Struct. Commun.* **10**, 1329–1334.

GO, K. & KARTHA, G. (1982). *Cryst. Struct. Commun.* **11**, 279–284, 285–290.

GO, K. & KARTHA, G. (1983). *Acta Cryst.* C**39**, 376–378.

GO, K., KARTHA, G. & CHEN, J. P. (1980). *Acta Cryst.* B**36**, 1811–1819.

HESSE, G. (1937). *Chem. Ber.* **70B**, 2264–2267.

International Tables for X-ray Crystallography (1962). Vol. III. Birmingham: Kynoch Press.

KARLE, I. L. & KARLE, J. (1969). *Acta Cryst.* B**25**, 434–442.

KARTHA, G. & GO, K. (1981). *Cryst. Struct. Commun.* **10**, 1323–1327.

NEUMANN, W. (1937). *Chem. Ber.* **70B**, 1547–1554.

PRZYBYLSKA, M. & AHMED, F. R. (1979). *Acta Cryst.* B**35**, 2436–2440.

TSCHESCHE, R. (1937). *Chem. Ber.* **70B**, 1554–1556.

Acta Cryst. (1984). C**40**, 1869–1871

Structure and Conformation of Sulfinpyrazone, $C_{23}H_{20}N_2O_3S$

BY KUANTEE GO AND GOPINATH KARTHA

Roswell Park Memorial Institute, 666 Elm Street, Buffalo, NY 14263, USA

(*Received* 9 *March* 1984; *accepted* 6 *June* 1984)

Abstract. (±)-1,2-Diphenyl-4-[2-(phenylsulfinyl)ethyl]-3,5-pyrazolidinedione: $M_r = 404{\cdot}5$, (±) form from ethyl acetate, monoclinic, $C2/c$, $a = 28{\cdot}349\ (3)$, $b = 5{\cdot}696\ (1)$, $c = 30{\cdot}799\ (3)$ Å, $\beta = 126{\cdot}07\ (1)°$, $V = 4019{\cdot}9$ Å^3, $Z = 8$, $D_x = 1{\cdot}337$ Mg m^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}5418$ Å, $\mu = 1{\cdot}556$ mm^{-1}, $F(000) = 1696$, room temperature, $R_w = 6{\cdot}4\%$ for 3019 reflections, 3799 unique reflections measured. The unit cell consists of four pairs of enantiomers. Each molecular location is occupied at random by either of a pair of almost superimposable diastereomers differing only in the configuration around the S atom. In the crystal, the populations of the diastereomers, however, are not equal; the molecule where S=O is *gauche* to the ethylpyrazolidinedione group is predominant. The two phenyl groups at the pyrazone make an angle of 86° with each other.

Introduction. Sulfinpyrazone (a derivative of phenylbutazone) has long been recognized as a potent uricosuric agent, but has recently been studied extensively as a platelet inhibitor and antithrombotic agent (Margulies, White & Sherry, 1980). We have obtained crystals of sulfinpyrazone and determined its crystal structure and found interesting conformations at the asymmetric S.

Experimental. Thin needle-shaped crystals were recrystallized from Anturane (in powder form supplied by Ciba Pharmaceutical Co.) in ethyl acetate. D_m not determined. Crystal $0{\cdot}05 \times 0{\cdot}2 \times 0{\cdot}75$ mm. Enraf–Nonius CAD-4 automated diffractometer, Ni-filtered Cu $K\alpha$. Lattice dimensions by least-squares fit to a set of 25 reflections measured in θ range 12–29°. ω–2θ scans and integrated counts with $\theta < 70°$. 3799 independent reflections, 3019 with $I > \sigma(I)$. Three standard reflections (overall $\sigma = 0{\cdot}03$). Lp corrections, empirical (one parameter, φ) absorption correction (range 0·93–1·15). Direct methods (*MULTAN*, Germain, Main & Woolfson, 1971). Anisotropic block-diagonal least-squares refinement for 29 non-hydrogen atoms; 20 calculated H atoms isotropic; final $R = 0{\cdot}059$, $R_w = 0{\cdot}064$ for 3019 reflections ($\Delta/\sigma < 0{\cdot}2$); $\sum w(|F_o| - |F_c|)^2$ minimized where $w^{-1} = [\sigma^2(F) + (cF)^2]$; $\Delta\rho_{max} = 0{\cdot}2$ e Å^{-3}. f curves from *International Tables for X-ray Crystallography* (1962). Enraf–Nonius *SDP* package and local programs.

0108-2701/84/111869-03$01.50