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.

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Structure and Conformation of Sulfinpyrazone, C23H20N2O3S

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Abstract. ($\pm$)-1,2-Diphenyl-4-[2-(phenylsulfinyl)ethyl]-3,5-pyrazolidinedione: $M_r = 404 \cdot 5$, ($\pm$) 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 $0 \cdot 93 - 1 \cdot 15$). 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.

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

Fig. 1 shows the molecular structure and numbering of the atoms. The 1,2-diphenyl rings in sulfinpyrazone make an angle of 86° with each other. Kodama, Zushi, Nishihata & Nishio (1980) studied the preferred conformations of some alkyl 1-phenylethyl sulfoxides, and observed that, in the most populated conformation, the achiral alkyl group is oriented *gauche* to the phenyl and *anti* to the methyl group, irrespective of the nature

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, torsion angles and short intermolecular distances less than 3·5 Å have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39570 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

$$B_{eq} = 8\pi^2 U_{eq} \text{ where } U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j a_i^* a_j^* (\mathbf{a}_i.\mathbf{a}_j)U_{ij}.$$

	x	y	z	$B_{eq}(\text{Å}^2)$
S	3473 (1)	1740 (2)	609 (1)	3·8
O(1)†	4024 (1)	439 (6)	896 (1)	5·2
O(1')†	2895 (2)	685 (10)	318 (2)	4·2
O(3)	3271 (1)	8509 (4)	−573 (1)	3·5
O(5)	4735 (1)	2731 (4)	88 (1)	4·2
N(1)	3961 (1)	3983 (4)	−745 (1)	2·6
N(2)	3555 (1)	5894 (4)	−942 (1)	2·5
C(3)	3608 (1)	7025 (4)	−517 (1)	2·7
C(4)	4124 (1)	5990 (5)	−5 (1)	2·9
C(5)	4324 (1)	4036 (5)	−188 (1)	3·0
C(6)	4000 (1)	5235 (5)	393 (1)	3·0
C(7)	3467 (1)	3652 (5)	135 (1)	3·4
C(1A)	4165 (1)	3346 (4)	−1056 (1)	2·5
C(2A)	4112 (1)	4876 (5)	−1431 (1)	2·9
C(3A)	4350 (1)	4267 (5)	−1701 (1)	3·7
C(4A)	4645 (1)	2190 (6)	−1590 (1)	3·8
C(5A)	4692 (1)	662 (5)	−1219 (1)	3·4
C(6A)	4441 (1)	1199 (5)	−956 (1)	2·8
C(1B)	2985 (1)	5446 (4)	−1434 (1)	2·5
C(2B)	2695 (1)	3372 (5)	−1497 (1)	3·1
C(3B)	2140 (1)	3059 (5)	−1967 (1)	3·8
C(4B)	1894 (1)	4753 (6)	−2361 (1)	4·1
C(5B)	2185 (1)	6787 (5)	−2293 (1)	3·8
C(6B)	2738 (1)	7139 (5)	−1826 (1)	3·1
C(1C)	3569 (1)	3924 (6)	1072 (1)	3·7
C(2C)	3120 (1)	5426 (6)	939 (1)	4·2
C(3C)	3209 (1)	7117 (8)	1303 (1)	5·5
C(4C)	3732 (1)	7224 (10)	1795 (1)	7·3
C(5C)	4164 (2)	5708 (10)	1928 (1)	9·1
C(6C)	4088 (1)	4032 (10)	1568 (1)	6·7

† O(1) has a weight of 0·75 and O(1') a weight of 0·25.
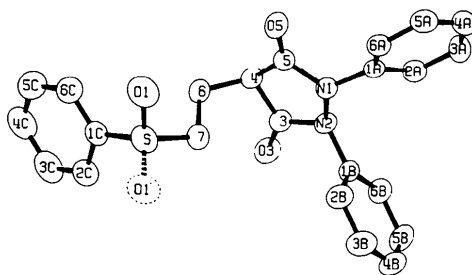
Fig. 1. Molecular structure of sulfinpyrazone.

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

N(1)−N(2)	1·433 (2)	C(3A)−C(4A)	1·373 (4)
N(2)−C(3)	1·385 (2)	C(4A)−C(5A)	1·381 (3)
N(1)−C(5)	1·388 (2)	C(5A)−C(6A)	1·390 (2)
C(3)−C(4)	1·504 (3)	C(6A)−C(1A)	1·386 (3)
C(4)−C(5)	1·502 (3)	C(1B)−C(2B)	1·386 (3)
C(4)−C(6)	1·524 (2)	C(2B)−C(3B)	1·386 (3)
C(6)−C(7)	1·521 (3)	C(3B)−C(4B)	1·376 (4)
S−C(7)	1·813 (2)	C(4B)−C(5B)	1·366 (4)
S−O(1)	1·465 (3)	C(5B)−C(6B)	1·384 (3)
S−O(1')	1·456 (5)	C(6B)−C(1B)	1·373 (3)
S−C(1C)	1·790 (3)	C(1C)−C(2C)	1·381 (3)
C(3)−O(3)	1·209 (2)	C(2C)−C(3C)	1·383 (5)
C(5)−O(5)	1·211 (3)	C(3C)−C(4C)	1·360 (4)
N(1)−C(1A)	1·429 (2)	C(4C)−C(5C)	1·350 (8)
N(2)−C(1B)	1·447 (2)	C(5C)−C(6C)	1·381 (8)
C(1A)−C(2A)	1·383 (3)	C(6C)−C(1C)	1·363 (3)
C(2A)−C(3A)	1·386 (2)		
N(2)−N(1)−C(5)	109·5 (3)	N(1)−C(1A)−C(2A)	120·8 (3)
N(2)−N(1)−C(1A)	116·8 (2)	N(1)−C(1A)−C(6A)	118·3 (3)
C(5)−N(1)−C(1A)	122·3 (3)	C(2A)−C(1A)−C(6A)	120·8 (3)
N(1)−N(2)−C(3)	109·9 (2)	C(1A)−C(2A)−C(3A)	119·4 (3)
N(1)−N(2)−C(1B)	115·6 (2)	C(2A)−C(3A)−C(4A)	120·5 (4)
C(3)−N(2)−C(1B)	120·2 (3)	C(3A)−C(4A)−C(5A)	119·7 (4)
N(2)−C(3)−C(4)	107·8 (3)	C(4A)−C(5A)−C(6A)	120·8 (3)
N(2)−C(3)−O(3)	123·6 (3)	C(5A)−C(6A)−C(1A)	118·7 (3)
C(4)−C(3)−O(3)	128·5 (3)	N(2)−C(1B)−C(2B)	120·2 (3)
C(3)−C(4)−C(5)	104·3 (3)	N(2)−C(1B)−C(6B)	118·3 (3)
C(3)−C(4)−C(6)	114·2 (3)	C(2B)−C(1B)−C(6B)	121·5 (3)
C(5)−C(4)−C(6)	114·2 (3)	C(1B)−C(2B)−C(3B)	118·1 (3)
N(1)−C(5)−C(4)	108·1 (3)	C(2B)−C(3B)−C(4B)	120·3 (4)
N(1)−C(5)−O(5)	124·2 (3)	C(3B)−C(4B)−C(5B)	120·8 (4)
C(4)−C(5)−O(5)	127·6 (3)	C(4B)−C(5B)−C(6B)	119·7 (4)
C(4)−C(6)−C(7)	112·2 (3)	C(5B)−C(6B)−C(1B)	119·4 (3)
C(6)−C(7)−S	113·6 (3)	S−C(1C)−C(2C)	120·9 (3)
C(7)−S−C(1C)	98·6 (2)	S−C(1C)−C(6C)	118·8 (4)
C(7)−S−O(1)	107·0 (2)	C(2C)−C(1C)−C(6C)	120·2 (4)
O(1)−S−C(1C)	105·2 (2)	C(1C)−C(2C)−C(3C)	119·6 (4)
C(7)−S−O(1')	106·2 (4)	C(2C)−C(3C)−C(4C)	119·5 (5)
O(1')−S−C(1C)	111·4 (4)	C(3C)−C(4C)−C(5C)	120·5 (7)
O(1)−S−O(1')	125·2 (5)	C(4C)−C(5C)−C(6C)	120·9 (8)
		C(5C)−C(6C)−C(1C)	119·0 (7)

of the alkyl group or the configuration of the sulfoxide. In their conformational study of some 2-thio derivatives of 2-phenylethanol, Alcudia, Brunet, Garcia Ruano, Rodriguez & Sanchez (1982) concluded that the steric interactions are primary factors for the conformational stability control while electrostatic interactions are secondary. Our results show that in sulfinpyrazone the sulfoxide of the tricoordinated S exists in two configurations; that in which S=O is *gauche* to the ethylpyrazolidine group is three times more populated than the alternate *anti* form. Since the intermolecular distance between the two O(1') is short [2·82 (1) Å], it appears that steric interactions could be responsible for this preference. On the other hand, O(1) is in the plane of S−C(1C)=C(6C) [the torsion angle O(1)=S−C(1C)=C(6C) is −178° while O(1')=S−C(1C)=C(6C) is 139°] owing to electrostatic interactions in the conjugated system. A combination of steric and electrostatic interactions may give rise to the two alternate positions. We have tried to confirm this by also considering the space groups C2 and Cc during the X-ray refinement. In all three cases (C2/c, C2 and Cc), the electron density map clearly indicated the presence of two alternate positions for O in which the S=O that is *gauche* to the ethylpyrazone is three times more populated than that which is *anti*.
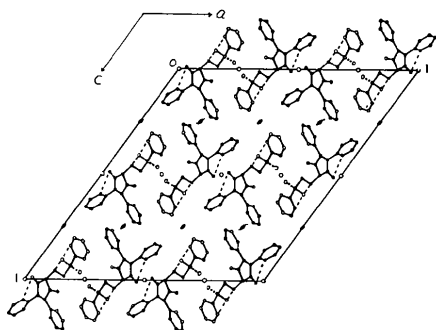
Fig. 2. Packing of molecules in the unit cell (viewed down **b**). Dotted lines indicate alternate positions. Dashed lines indicate the three intramolecular short contacts.

Bond lengths and angles, given in Table 2, are mostly within the range of standard values (Sutton, 1965). However, rather large differences are seen for the bonds C(3C)—C(4C), C(4C)—C(5C) and C(6C)—C(1C) of the ring C, but these are presumably due to the larger positional errors near the disordered region where the two alternate configurations around the S atom have to be accommodated.

Fig. 2 (a projection along **b**) shows the packing of the molecules in the unit cell. S=O(1') bonds are shown by dotted lines. Short contacts C(6)···O(1) [3·122 (4)], C(6C)···O(1) [2·839 (6)] and C(6A)—O(5) [2·940 (2) Å] are shown by dashed lines. These short

contacts may be responsible for the differences in the torsion angles around the junction between the phenyls and the pyrazone: C(5)—N(1)—C(1A)—C(2A) is −121·7 (4) and C(3)—N(2)—C(1B)—C(6B) 88·6 (4)°, a difference of 33°. As a result, phenyl ring A makes an angle of 46·3 (5)° and ring B an angle of 110·4 (5)° with the pyrazone ring. There are some intermolecular short contacts such as C(5A)···O(1) and C(6A)···O(5) with distances of 3·221 (2) and 3·203 (3) Å respectively, which may be responsible for loosely holding the molecules in the crystal.

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5,11-Dimethyl-6H-pyrido[4,3-b]carbazole 2-Oxide Trihydrate (Ellipticine N-Oxide Trihydrate), C₁₇H₁₄N₂O.3H₂O

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Abstract. $M_r = 316·4$, monoclinic, $P2_1/c$, $a = 12·229$ (3), $b = 6·962$ (3), $c = 18·946$ (6) Å, $\beta = 106·67$ (3)°, $V = 1545$ (1) Å³, $Z = 4$, $D_x = 1·360$ (1), $D_m = 1·35$ (1) g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0·7107$ Å, $\mu = 0·91$ cm⁻¹, $F(000) = 672$, $T = 295$ (1) K. Final $R = 0·045$ for 887 observed reflections. This is a new compound from the oxidation of ellipticine with hydrogen peroxide. The structural parameters are, in general, normal and are compared with those in ellipticine and related compounds. The organic molecule, except for the methyl H atoms, is nearly planar with the maximum deviation of any non-H atom

from the best least-squares plane being 0·048 (6) Å for a methyl C atom. The three waters of hydration are involved in a hydrogen-bonding network with each other, the N-oxide O, and the H of the NH group. The N-oxide O acts as the acceptor for three water H atoms.

Introduction. Ellipticine, a naturally occurring alkaloid, is known to exhibit antitumor activity *in vitro* because of its ability to intercalate with DNA (Le Pecq, Dat-Xuong, Gosse & Paoletti, 1974). The planar ring system is known to insert between the DNA strands with overlap between the heterocyclic ring system and