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Structure of 4-Methyl-5-(2-pyrazinyl)-3*H*-1,2-dithiole-3-thione (Oltipraz), $C_8H_6N_2S_3$: A New Antischistosomal Drug*†

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Abstract. $M_r = 226\cdot342$, orthorhombic, *Pbca*, $a = 22\cdot904$ (1), $b = 22\cdot151$ (1), $c = 7\cdot4142$ (6) Å, $V = 3761\cdot6$ Å^3, $Z = 16$, $D_m = 1\cdot62$ (2), $D_x = 1\cdot598$ g cm^{-3}, Cu $K\alpha_1$ ($\lambda = 1\cdot54056$ Å), $\mu = 65\cdot14$ cm^{-1}, $F(000) = 1856$, $T = 296$ (1) K, $R(F) = 0\cdot034$ for 3152 counter data. The compound crystallized from an ethyl acetate solution. The two crystallographically independent molecules have corresponding molecular parameters closely resembling each other. Individual six- and five-membered rings are essentially planar for both molecules. Molecules are roughly planar, to within 0·108 (3) Å (for nonhydrogen atoms only) for one molecule and to within 0·151 (3) Å for the other. The angle between plane normals of the six- and five-membered rings is 3·71 (8)° for the former and 8·92 (9)° for the latter.

Introduction. Hycanthone and structurally analogous drugs have been known to be effective in the treatment of schistosomiasis, a slowly debilitating tropical disease (Bueding, Fisher & Bruce, 1973) that affects at least two million people in sections of Asia, Africa, Brazil, the Caribbean, and the Middle East. Structures of several of these drugs have been elucidated in our laboratory by X-ray diffraction (for example, see Wei, 1982).

Recently two new antischistosomal drugs have been developed. Unlike hycanthone, neither appears to have bad side effects. One of them is *p*-(*p*-nitroanilino)-phenyl isothiocyanate (amoscanate), the structure of which has been reported recently (Hardgrove, Einstein & Wei, 1983). The other, 4-methyl-5-(2-pyrazinyl)-3*H*-1,2-dithiole-3-thione (oltipraz), has been reported to act slowly on mice infected with *Schistosoma mansoni*, but eventually to attain parasitological cures by a reduction of the glutathione stores of the parasite (Bueding, Dolan & Leroy, 1982). It was also pointed out by the same authors that the activity of drugs of the oltipraz type has rather stringent structural requirements: the existence of thione sulfur and a diazine ring in the molecule is essential; further, the pyrazine and 1,2-dithiole rings have to be connected directly.

The X-ray investigation of the title compound was undertaken as part of our health-related program to provide the first detailed structural information regarding this new class of drugs.

Experimental. Maroon crystals of oltipraz grown at room temperature from an ethyl acetate solution of the specimen, kindly furnished by Professor Ernest Bueding of The Johns Hopkins University. Crystal density determinated by flotation in mixtures of bromoform and xylene. Space group and approximate unit-cell parameters established from Weissenberg and precession photographs. Data from a crystal of dimensions 0·18 × 0·16 × 0·48 mm, mounted on a Picker four-circle diffractometer. Lattice parameters refined by least-squares methods from angle measurements of 12 strong reflections in the 2θ range 102–129° (Busing, Ellison, Levy, King & Roseberry, 1968). Intensity data collected by θ–2θ step scans in the 2θ range 1–136°, divided into three separate subranges. Within any subrange the maximum variation for two standard reflections was less than 0·8%. Of the 3322 unique nonzero reflections, 167 reflections with F_o^2 less than $\sigma(F_o^2)$ and three reflections with $10|\Delta F^2|$ were excluded from the final least-squares refinement. Absorption corrections applied analytically by the method of Busing & Levy (1957), transmission range 0·294 to 0·452. No extinction corrections applied. Maximum mosaic spread of the crystal used estimated to be 0·44°.

Interpretation of a Patterson map was inconclusive. The structure was solved instead with *MULTAN* (Germain, Main & Woolfson, 1971). Preliminary refinement for the 26 nonhydrogen atoms was carried out using the block-diagonal least-squares program of Shiono (1971). Twelve H atoms, later obtained from a difference Fourier map, were also included in the refinement. The final four cycles of least-squares refinement on F^2 were carried out with the full-matrix program *ORXFLS*4 (Busing, Martin & Levy, 1962). Scattering factors were those of Cromer & Waber (1974); anomalous-scattering corrections for S (Cromer, 1974) were applied. Weights (w) used in the refinement were reciprocals of the variances $\sigma^2(F_o^2)$, which were estimated according to the empirical expression given elsewhere (Wei & Einstein, 1978). Thermal motions of nonhydrogen atoms were treated

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anisotropically and of H atoms isotropically. The final refinement resulted in $R(F)$, $R(F^2)$ and $R_w(F^2)$ values of 0·034, 0·041 and 0·093, respectively for 3152 reflections used. The standard deviation of an observation of unit weight, S, was 1·379. The data-to-variables ratio was 11·06. When all 3322 nonzero reflections were included, the $R(F)$ value was 0·037. $\Delta x_i/\sigma$ for all nonhydrogen atoms < 3%, $(\Delta x_i/\sigma)_{max}$ for H parameters = 18%; subsequent $\Delta\rho$ excursions $\leq 0·38$ e Å^{-3}.

Discussion. Positional parameters and isotropic temperature factors (or their equivalents) are listed in Table 1.*

There are two crystallographically independent molecules in this structure. The arrangement of these molecules in the asymmetric unit is shown in the [001] projection in Fig. 1. The structure consists of these molecules held together by van der Waals forces. The closest intermolecular contact between nonhydrogen atoms is 3·389 (1) Å for S(11)···S(32);† the closest such contact between nonhydrogen and H atoms is 2·70 (4) Å for N(21)···H(8*b*2).

The molecule can be formally considered to be derived by linking a pyrazine ring and a 4-methyl-1,2-dithiole-3-thione ring *via* the C(1)—C(7) bond, as shown in Fig. 2. Bond lengths, bond angles, and their corresponding e.s.d.'s calculated by the program *ORFFE*4 (Busing, Martin & Levy, 1964), are also given in the figure. The corresponding bond lengths and angles for the two independent molecules are remarkably similar, the maximum differences in bond lengths and angles being 0·015 (3) Å for C(5)—S(3) and 0·7 (3)° for C(5)—C(6)—C(7), respectively. These agreements make the apparent high precision of the molecular parameters even more credible.

The molecular parameters for the pyrazine rings are not much different from those reported for pyrazine (Wheatley, 1957) and pyrimidine (Wheatley, 1960). As expected, the angles at the N atoms range from 116·5 (3) to 117·2 (2)° [average of four values 116·7 (1)°], all less than 120°, in agreement with the observations made for the similar unsaturated heterocyclic systems containing N atoms (also see Foss & Tjomsland, 1958*b*). In contrast, the angles at the C atoms for the pyrazine rings are all greater than 120°, ranging from 120·5 (2) to 122·5 (3)°, the average of eight being 121·6 (1)°.

The five-membered heterocyclic ring system in this structure is nearly identical with that of 4-methyl-1,2-dithia-4-cyclopentene-3-thione (Kehl & Jeffrey, 1958; Jeffrey & Shiono, 1959). The fact that C(61)—

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38511 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

† The first number in parentheses designates the atom number; the second number, 1 or 2, represents either molecule 1 or molecule 2. When only one number is given in parentheses, it implies that particular atom in both molecule 1 and molecule 2.

Table 1. *Positional and isotropic thermal parameters*

The e.s.d.'s are given in parentheses in all tables and in the text. The digits in parentheses correspond to the least significant digits of the parameters. The equivalent isotropic temperature factors for C, N, S atoms were calculated from the corresponding anisotropic thermal parameters and unit-cell parameters by the relation $B_{eq} = \frac{4}{3}(\beta_{11}a^2 + \beta_{22}b^2 + \beta_{33}c^2)$ (Hamilton, 1959).

	x	y	z	B_{eq} or B(Å^2)
S(11)	−0·06660 (2)	−0·04940 (3)	0·17557 (9)	3·58 (2)
S(21)	−0·03615 (3)	−0·13568 (3)	0·14247 (10)	3·94 (2)
S(31)	0·08123 (3)	−0·17992 (3)	0·19333 (12)	5·08 (2)
N(11)	−0·05415 (9)	0·06838 (10)	0·2470 (3)	4·18 (6)
C(11)	−0·00128 (10)	0·04425 (10)	0·2750 (3)	3·21 (5)
C(21)	0·04420 (12)	0·07954 (13)	0·3360 (4)	4·39 (7)
N(21)	0·03766 (12)	0·13869 (11)	0·3702 (4)	5·18 (7)
C(31)	−0·01433 (16)	0·16145 (14)	0·3419 (5)	5·29 (9)
C(41)	−0·06034 (15)	0·12684 (14)	0·2810 (5)	5·14 (9)
C(51)	0·03556 (10)	−0·12204 (11)	0·1973 (3)	3·45 (6)
C(61)	0·04803 (10)	−0·05999 (11)	0·2395 (3)	3·30 (5)
C(71)	0·00167 (9)	−0·02114 (10)	0·2351 (3)	3·08 (5)
C(81)	0·10998 (12)	−0·04260 (15)	0·2789 (6)	5·09 (9)
S(12)	0·31030 (2)	0·24052 (3)	0·38369 (8)	3·28 (2)
S(22)	0·28551 (3)	0·32840 (3)	0·42472 (9)	3·45 (2)
S(32)	0·17415 (3)	0·37526 (3)	0·54324 (10)	4·14 (2)
N(12)	0·29037 (9)	0·12166 (9)	0·3845 (3)	3·94 (6)
C(12)	0·24300 (10)	0·14563 (10)	0·4622 (3)	3·21 (5)
C(22)	0·19941 (14)	0·10892 (12)	0·5329 (4)	4·58 (7)
N(22)	0·20197 (12)	0·04895 (10)	0·5217 (4)	5·22 (7)
C(32)	0·24815 (14)	0·02613 (13)	0·4401 (5)	5·20 (9)
C(42)	0·29207 (14)	0·06149 (13)	0·3723 (5)	4·95 (8)
C(52)	0·21578 (10)	0·31513 (10)	0·4998 (3)	3·07 (5)
C(62)	0·20054 (9)	0·25244 (10)	0·5142 (3)	2·96 (5)
C(72)	0·24348 (10)	0·21242 (10)	0·4664 (3)	2·97 (5)
C(82)	0·14016 (11)	0·23615 (13)	0·5756 (4)	3·93 (7)
H(21)*	0·079 (1)	0·063 (1)	0·368 (4)	4·3 (6)
H(31)	−0·022 (2)	0·201 (2)	0·367 (5)	6·7 (9)
H(41)	−0·095 (1)	0·142 (1)	0·265 (4)	5·4 (7)
H(8*a*1)	0·131 (2)	−0·069 (2)	0·267 (5)	6·0 (9)
H(8*b*1)	0·123 (2)	−0·008 (2)	0·205 (4)	6·7 (9)
H(8*c*1)	0·111 (2)	−0·028 (2)	0·401 (5)	7·4 (10)
H(22)	0·169 (2)	0·127 (2)	0·615 (6)	9·6 (13)
H(32)	0·254 (1)	−0·013 (2)	0·443 (5)	6·3 (8)
H(42)	0·320 (1)	0·048 (1)	0·325 (4)	4·8 (7)
H(8*a*2)	0·118 (1)	0·267 (1)	0·596 (4)	3·5 (6)
H(8*b*2)	0·125 (2)	0·209 (2)	0·496 (5)	6·5 (9)
H(8*c*2)	0·141 (1)	0·214 (1)	0·696 (4)	4·5 (6)

* Hydrogen atoms are numbered according to the C atoms to which they are attached.

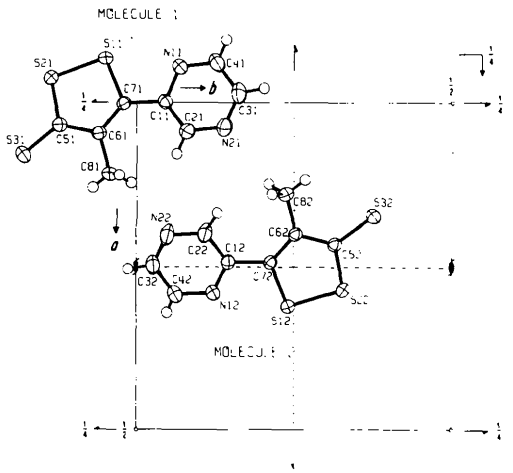


Fig. 1. Numbering scheme and arrangement of the two independent molecules in the asymmetric unit. For the sake of clarity, H atoms are not labeled. This and the other figures were prepared with the program *ORTEP*II (Johnson, 1976).

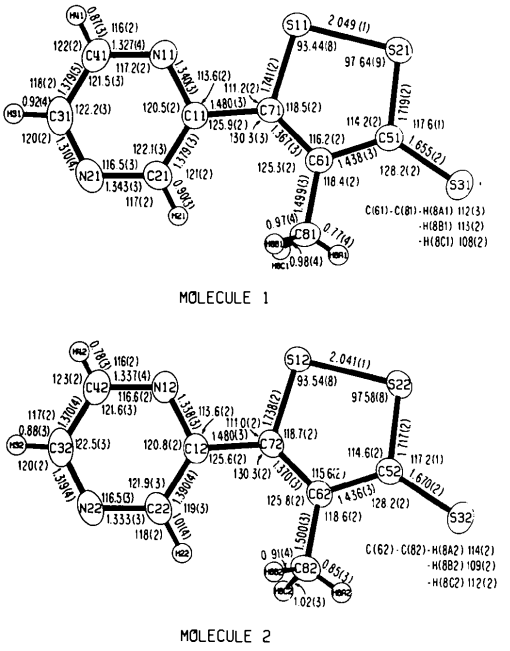


Fig. 2. Bond lengths (Å) and bond angles (°) for the two independent molecules. The close agreement of corresponding molecular parameters for the two molecules can be seen.

C(71) and C(62)—C(72) have double-bond character is evidenced by their bond lengths of 1·367 (3) and 1·370 (3) Å. The thione bond lengths, 1·655 (2) Å for molecule 1 and 1·670 (2) Å for molecule 2, are larger than the value expected for the double-bond length of 1·61 Å (Pauling, 1960), but they are comparable with those of 1·63 Å for 4-methyl-1,2-dithia-4-cyclopentene-3-thione (Jeffrey & Shiono, 1959), 1·650 Å for 3,4-dibenzyl-2-(*p*-methoxyphenyl)-1,3,4-thiadiazolidine-5-thione (Karle & Karle, 1965), 1·662 Å for tetraethylthiuram disulfide (Karle, Estlin & Britts, 1967), 1·65 (2) Å for *tert*-butyl *N,N*-dimethyltrithiopercarbamate (Mitchell, 1969), and 1·693 (5) and 1·675 (5) Å for tetraethylammonium cyanodithioformate (Engler, Dräger & Gattow, 1974). The S(11)—S(21) and S(12)—S(22) bond lengths of 2·049 (1) and 2·041 (1) Å are shorter than the length of 2·08 Å given by Pauling (1960) for an S—S single bond. (Pauling's equation, $D = D_1 - 0{\cdot}71 \log n$, results in 12% π-bond character if D_1 is taken as 2·08 Å.) However, the values obtained agree well with the 2·047 (7) Å for 4-methyl-1,2-dithia-4-cyclopentene-3-thione (Jeffrey & Shiono, 1959). Many other S—S bond lengths for noncyclic structures have been reported. A survey of these values reveals that they are in the range between 1·999 (5) Å for tetraethylthiuram disulfide (Karle, Estlin & Britts, 1967) and 2·083 (6) Å for 3,3′-trithiodi(2,4-pentanedione) (Power & Jones, 1971), depending upon different degrees of double-bond character arising from overlap of the $p\pi$ electron pairs of one divalent S atom with available 3*d* orbitals of a bond partner (Foss & Tjomsland, 1958*a*).

Table 2. *Equations of best molecular planes, and distances* (Å) *of atoms from these planes*

Root-mean-square deviations of the fitted atoms for planes (*a*), (*b*), (*c*) and (*d*) are 0·006 (1), 0·0008 (13), 0·0023 (7) and 0·009 (1) Å, respectively.

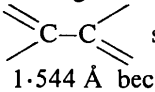
Calculations were performed with program *ORFFE*4 (Busing, Martin & Levy, 1964). *X*, *Y*, and *Z* represent direct-axis coordinates expressed in Å.

(*a*) $-0{\cdot}1945X - 0{\cdot}1907Y + 0{\cdot}9622Z = 1{\cdot}7561$
(*b*) $-0{\cdot}2536X - 0{\cdot}2101Y + 0{\cdot}9442Z = 1{\cdot}7263$
(*c*) $0{\cdot}3331X - 0{\cdot}0116Y + 0{\cdot}9428Z = 5{\cdot}0604$
(*d*) $0{\cdot}4733X - 0{\cdot}0356Y + 0{\cdot}8802Z = 5{\cdot}5492$

(*a*)		(*b*)		(*c*)		(*d*)	
S(11)	0·002 (1)	N(11)	−0·001 (2)	S(12)	−0·003 (1)	N(12)	0·012 (2)
S(21)	−0·005 (1)	C(11)	0·001 (2)	S(22)	0·003 (1)	C(12)	−0·013 (2)
C(51)	0·009 (1)	C(21)	−0·001 (2)	C(52)	−0·002 (1)	C(22)	0·004 (2)
C(61)	−0·008 (1)	N(21)	0·001 (2)	C(62)	−0·001 (2)	N(22)	0·006 (2)
C(71)	0·003 (1)	C(31)	−0·001 (2)	C(72)	0·003 (1)	C(32)	−0·008 (2)
S(31)*	0·021 (3)	C(41)	0·001 (2)	S(32)*	−0·031 (3)	C(42)	−0·002 (2)
C(81)*	−0·077 (5)	C(81)*	−0·215 (7)	C(82)*	−0·028 (4)	C(82)*	−0·460 (7)
H(8*b*1)*	−0·81 (3)			H(8*b*2)*	−0·69 (4)		
H(8*c*1)*	0·73 (4)			H(8*c*2)*	0·82 (3)		

* Atoms not included in the calculation of the least-squares plane.

The two endocyclic S—C bond lengths of a given ring in the present structure are noticeably different from each other: 1·741 (2) Å for S(11)—C(71) and 1·719 (2) Å for S(21)—C(51) in molecule 1; 1·738 (2) Å for S(12)—C(72) and 1·717 (2) Å for S(22)—C(52) in molecule 2. The phenomenon is similar to that observed by Jeffrey & Shiono (1959). Inasmuch as atoms C(7), S(1), S(2) and C(5) are coplanar or nearly so (Table 2), these differences in bond lengths require that the angles subtended at S(2) be greater than the angles subtended at S(1). Thus, the C(51)—S(21)—S(11) angle is greater than the C(71)—S(11)—S(21) angle by 4·2 (1)° in molecule 1, and the difference between the two corresponding C—S—S angles in molecule 2 is 4·0 (1)°.

The central C—C single-bond length in a conjugated system such as  should be less than the normal value of 1·544 Å because of partial double-bond character of the bond (Pauling, 1960). The observed values of 1·480 (3) Å for C(11)—C(71) and C(12)—C(72) are in accord with this estimation and in fact can be compared with 1·482 (1) Å obtained for tri(*p*-biphenyl)aminium perchlorate (Brown, Freeman & Walter, 1977).

In each molecule the five- and six-membered rings are essentially planar (Table 2). For molecule 1, the atoms of the 1,2-dithiole ring deviate from the best molecular plane (*a*) no more than 0·009 (1) Å; the atoms of pyrazine ring (*b*) deviate no more than 0·001 (2) Å. For molecule 2, the maximum deviations are 0·003 (1) and −0·013 Å [planes (*c*) and (*d*)], respectively for the two ring systems. The angle between plane normals for the five- and six-membered rings is 3·71 (8)° for molecule 1, whereas the corresponding value for molecule 2 is 8·92 (9)°. Alternatively this difference in the dihedral angle can be described in terms of the torsion angles S(11)—C(71)—

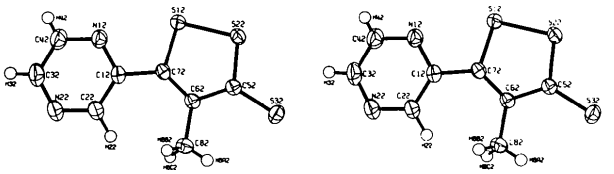


Fig. 3. Stereoscopic view of molecule 2. Thermal ellipsoids enclose 35% probability for non-H atoms.

C(11)—N(11) and S(12)—C(72)—C(12)—N(12): −3·7 (3)° for the former and −7·5 (3)° for the latter. The steric strain between H(21) [or H(22)] and the methyl group is somewhat relieved by the slight clockwise rotation of the whole five-membered ring around the C(11)—C(71) [or C(12)—C(72)] bond (to an extent not to interfere with the partial π character of this bond). The H(2)···C(8) separations are 2·53 (3) and 2·53 (4) Å in molecules 1 and 2. To achieve further the energy minimum, the methyl H atoms have rotated to positions which permit relatively facile interpenetration by the neighboring H(2), resulting in H(21)···H(8*b*1) and H(21)···H(8*c*1) distances of 2·24 (4) and 2·16 (4) Å in molecule 1; the corresponding values are 2·26 (5) and 2·12 (5) Å in molecule 2. Fig. 3 shows a stereoscopic view of molecule 2.

Relatively large deviations from the corresponding five-membered rings for the methyl carbons, C(81) and C(82) [−0·077 (5) and −0·028 (4) Å, respectively], can also be regarded as a consequence of the molecules alleviating the intramolecular strain. Despite the differences in molecular conformation arising also in part from possible packing effects, differences in the intramolecular contacts S(3)···C(8), N(1)···S(1) and C(2)···C(8) are 0·011 (3), 0·004 (3) and 0·018 (5) Å – not large enough to be significant.

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Structure of Dihydroxyacetic Acid (Glyoxylic Acid Monohydrate), $C_2H_4O_4$

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Abstract. $M_r = 92{\cdot}0$, triclinic, $P\bar{1}$, $a = 5{\cdot}833$ (4), $b = 5{\cdot}397$ (5), $c = 6{\cdot}586$ (6) Å, $\alpha = 97{\cdot}95$ (7), $\beta = 94{\cdot}71$ (7), $\gamma = 118{\cdot}26$ (6)°, $V = 178{\cdot}3$ Å^3, $Z = 2$, $D_m = 1{\cdot}70$, $D_x = 1{\cdot}72$ Mg m^{-3}, μ(Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å) $= 0{\cdot}19$ mm^{-1}, $F(000) = 96$, $T = 296 \pm 2$ K, final $R = 0{\cdot}040$ and $R_w = 0{\cdot}045$ for 690 non-zero reflexions. The X-ray analysis showed that glyoxylic acid monohydrate ($CHOCOOH.H_2O$)

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