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### Structures of Two Isomers of Phenyl-3*H*-1,2-dithiole-3-thione\*†

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**Abstract.** Structures of two isomeric compounds are reported: 4-phenyl-3*H*-1,2-dithiole-3-thione (4-PDT) and 5-phenyl-3*H*-1,2-dithiole-3-thione (5-PDT). Both have formula  $C_6H_5C_3HS_3$ ,  $M_r = 210.339$ , X-ray data collected with  $\lambda(Mo Ka) = 0.7093 \text{ \AA}$  at 296 (1) K. The former belongs to the monoclinic space group  $P2_1/c$ ,  $a = 17.827 (4)$ ,  $b = 5.731 (1)$ ,  $c = 9.272 (2) \text{ \AA}$ ,  $\beta = 91.93 (2)^\circ$ ,  $V = 946.8 (3) \text{ \AA}^3$ ,  $Z = 4$ ,  $D_m = 1.47 (1)$ ,  $D_x = 1.475 \text{ g cm}^{-3}$ ,  $\mu = 6.93 \text{ cm}^{-1}$ ,  $F(000) = 432$ . Final  $R(F) = 0.031$  for 2255 counter data with  $F_o^2 \geq 2\sigma(F_o^2)$ . The latter crystallizes in the orthorhombic space group  $Pbca$ ,  $a = 19.949 (6)$ ,  $b = 12.188 (3)$ ,  $c = 7.544 (2) \text{ \AA}$ ,  $V = 1834.2 (9) \text{ \AA}^3$ ,  $Z = 8$ ,  $D_m = 1.51 (1)$ ,  $D_x = 1.523 \text{ g cm}^{-3}$ ,  $\mu = 7.16 \text{ cm}^{-1}$ ,  $F(000) = 864$ . Final  $R(F) = 0.031$  for 1633 data with  $F_o^2 \geq 2\sigma(F_o^2)$ . The molecules are composed of a phenyl ring and a five-membered heterocyclic ring, the difference between the two isomers being the positions of the C atom of the 3*H*-1,2-dithiole-3-thione ring to which the phenyl ring is linked. The two molecules have bond lengths and angles resembling each other and other compounds containing 3*H*-1,2-dithiole-3-thione as a building unit. The phenyl rings and the five-membered-ring systems are both planar to within 0.01  $\text{\AA}$  in both structures, the angle between the two plane normals being 67.40 (9)° for 4-PDT and 29.36 (8)° for 5-PDT.

**Introduction.** The title compounds, 4-PDT and 5-PDT, contain 3*H*-1,2-dithiole-3-thione as a building unit which was considered by Bueding, Dolan & Leroy (1982) to be essential for the antischistosomal drug 4-methyl-5-(2-pyrazinyl)-3*H*-1,2-dithiole-3-thione (oltipraz) to function. In our previous studies, it was shown that the pyrazine ring is linked to 4-methyl-3*H*-1,2-dithiole-3-thione via a C–C bond to yield oltipraz (Wei, 1983), or to 4-methyl-3*H*-1,2-dithiol-3-one to form an oltipraz analog (Wei, 1985a). On the other hand, the 3*H*-1,2-dithiole-3-thione ring could be linked to a *p*-methoxyphenyl group, yielding 5-(*p*-methoxyphenyl)-3*H*-1,2-dithiole-3-thione (Wang, Lin & Wei, 1985). The present paper reports the structures in which a phenyl ring replaces an H atom of the 3*H*-1,2-dithiole-3-thione ring at two different positions. Thus, crystallographic elucidation of the stereochemistry of a variety of different compounds containing the six- and five-membered-ring systems afford considerable stereochemical insight into the molecular parameters, including the angle between plane normals for the two ring systems.

**Experimental.** Maroon crystals of 4-PDT were obtained from toluene solution and orange crystals of 5-PDT from ethyl acetate solution of the corresponding specimen, kindly furnished by Professor Ernest Bueding of The Johns Hopkins University. Crystal densities determined by flotation in mixtures of 2-bromobutyric acid and 1,2-dichloroethane. In each case, space group and approximate cell parameters established from Weissenberg and precession photographs. For 4-PDT,

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data were obtained from a rod-like crystal of dimensions  $0.27 \times 0.35 \times 0.74$  mm; for 5-PDT, from a crystal, having an elongated irregular hexagonal cross section, of dimensions  $0.10 \times 0.27 \times 0.63$  mm, mounted on a Picker four-circle diffractometer. For each compound, lattice parameters were refined by the

Table 1. Positional and isotropic thermal parameters for 4-PDT

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 and S atoms were calculated from the corresponding anisotropic thermal parameters and unit-cell parameters by the relation  $B_{eq} = \frac{4}{3}(\beta_{11}a^2 + \beta_{22}b^2 + \beta_{33}c^2 + 2\beta_{13}ac\cos\beta)$  (Hamilton, 1959).

|       | <i>x</i>    | <i>y</i>     | <i>z</i>    | $B_{eq}$ or $B(\text{\AA}^2)$ |
|-------|-------------|--------------|-------------|-------------------------------|
| S(1)  | 0.41905 (2) | -0.31559 (9) | 0.44137 (4) | 3.81 (1)                      |
| S(2)  | 0.44961 (2) | -0.03101 (8) | 0.32295 (4) | 3.60 (1)                      |
| S(3)  | 0.35409 (3) | 0.26269 (8)  | 0.13821 (5) | 4.00 (1)                      |
| C(3)  | 0.3612 (1)  | 0.0425 (3)   | 0.2555 (1)  | 3.01 (3)                      |
| C(4)  | 0.3033 (1)  | -0.1082 (3)  | 0.3064 (2)  | 3.25 (3)                      |
| C(5)  | 0.3266 (1)  | -0.2855 (3)  | 0.3939 (2)  | 3.62 (4)                      |
| C(6)  | 0.2235 (1)  | -0.0705 (3)  | 0.2629 (2)  | 4.07 (4)                      |
| C(7)  | 0.1864 (1)  | 0.1253 (5)   | 0.3100 (4)  | 7.16 (9)                      |
| C(8)  | 0.1121 (2)  | 0.1634 (6)   | 0.2702 (6)  | 9.51 (14)                     |
| C(9)  | 0.0747 (1)  | 0.0062 (6)   | 0.1806 (5)  | 8.45 (11)                     |
| C(10) | 0.1108 (2)  | -0.1849 (7)  | 0.1331 (4)  | 8.23 (11)                     |
| C(11) | 0.1858 (1)  | -0.2290 (5)  | 0.1750 (3)  | 5.84 (7)                      |
| H(5)* | 0.294 (1)   | -0.394 (4)   | 0.435 (2)   | 4.8 (5)                       |
| H(7)  | 0.219 (2)   | 0.241 (6)    | 0.376 (5)   | 12.2 (12)                     |
| H(8)  | 0.085 (2)   | 0.291 (7)    | 0.291 (5)   | 13.7 (14)                     |
| H(9)  | 0.027 (2)   | 0.030 (6)    | 0.156 (3)   | 9.7 (9)                       |
| H(10) | 0.085 (2)   | -0.290 (6)   | 0.083 (4)   | 9.4 (10)                      |
| H(11) | 0.208 (2)   | -0.355 (5)   | 0.142 (3)   | 7.3 (8)                       |

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

Table 2. Positional and isotropic thermal parameters for 5-PDT

The equivalent isotropic temperature factors for C and 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).

|       | <i>x</i>    | <i>y</i>    | <i>z</i>      | $B_{eq}$ or $B(\text{\AA}^2)$ |
|-------|-------------|-------------|---------------|-------------------------------|
| S(1)  | 0.64616 (3) | 0.30649 (4) | 0.04307 (8)   | 2.79 (2)                      |
| S(2)  | 0.74128 (3) | 0.27023 (4) | -0.04029 (8)  | 2.86 (2)                      |
| S(3)  | 0.79761 (3) | 0.05197 (5) | -0.09609 (10) | 3.85 (2)                      |
| C(3)  | 0.7331 (1)  | 0.1285 (2)  | -0.0332 (3)   | 2.56 (5)                      |
| C(4)  | 0.6687 (1)  | 0.0938 (2)  | 0.0218 (3)    | 2.60 (5)                      |
| C(5)  | 0.6214 (1)  | 0.1704 (2)  | 0.0597 (3)    | 2.37 (5)                      |
| C(6)  | 0.5516 (1)  | 0.1494 (2)  | 0.1144 (3)    | 2.56 (5)                      |
| C(7)  | 0.5353 (1)  | 0.0545 (2)  | 0.2070 (3)    | 3.30 (6)                      |
| C(8)  | 0.4697 (1)  | 0.0373 (2)  | 0.2591 (4)    | 4.02 (7)                      |
| C(9)  | 0.4204 (1)  | 0.1121 (2)  | 0.2204 (4)    | 4.12 (7)                      |
| C(10) | 0.4360 (1)  | 0.2051 (2)  | 0.1269 (4)    | 4.14 (7)                      |
| C(11) | 0.5012 (1)  | 0.2243 (2)  | 0.0738 (3)    | 3.33 (6)                      |
| H(4)* | 0.659 (1)   | 0.019 (2)   | 0.021 (3)     | 3.0 (5)                       |
| H(7)  | 0.569 (1)   | 0.002 (2)   | 0.234 (3)     | 3.9 (6)                       |
| H(8)  | 0.463 (1)   | -0.027 (2)  | 0.312 (4)     | 4.6 (6)                       |
| H(9)  | 0.378 (1)   | 0.100 (2)   | 0.262 (4)     | 4.1 (6)                       |
| H(10) | 0.406 (2)   | 0.253 (3)   | 0.100 (4)     | 6.1 (8)                       |
| H(11) | 0.511 (1)   | 0.284 (2)   | 0.011 (4)     | 4.0 (6)                       |

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

least-squares method from angle measurements of 12 strong reflections in the  $2\theta$  range  $48\text{--}53^\circ$  for 4-PDT and  $41\text{--}51^\circ$  for 5-PDT (Busing, Ellison, Levy, King & Roseberry, 1968). Intensity data were collected by  $\theta\text{--}\theta$  step scans to  $2\theta = 60^\circ$  for 4-PDT ( $-24 \leq h \leq 24$ ,  $0 \leq k \leq 8$ ,  $0 \leq l \leq 12$ ) and to  $2\theta = 55^\circ$  for 5-PDT ( $0 \leq h \leq 25$ ,  $0 \leq k \leq 15$ ,  $0 \leq l \leq 9$ ). Strong low-angle reflections were remeasured by  $\omega$  scans. Of 2618 unique nonzero reflections measured, 2255 reflections with  $F_o^2 \geq 2\sigma(F_o^2)$  were used for the final refinement for 4-PDT; 1633 reflections with the same criteria were used for 5-PDT out of 1968 data. In both cases, absorption corrections were applied by the method of Busing & Levy (1957), transmission range 0.777 to 0.840 for 4-PDT and 0.856 to 0.953 for 5-PDT. No extinction corrections were applied. Maximum mosaic spread of the crystals used was estimated to be  $0.5^\circ$ .

Both structures were solved by the program *MULTAN* (Germain, Main & Woolfson, 1971). By iterated least-squares refinements [by the program *ORXFLS4* (Busing, Martin & Levy, 1962)] and difference-Fourier syntheses [by the program *ORFFP3* (Levy, 1977)] all atoms including H atoms were eventually located and refined. The function minimized in the least-squares refinement was  $\sum w|F_o^2 - sF_c^2|^2$ , where the weights  $w$  are reciprocals of  $\sigma^2(F_o^2) + (0.04F_o^2)^2$  and  $s$  is a scale factor. Scattering factors were those of Cromer & Waber (1974), anomalous-scattering corrections for S ( $f' = 0.110$ ,  $f'' = 0.124$ ) from Cromer (1974). The least-squares refinements were carried out until  $\Delta/\sigma$  for non-H atoms all became less than 0.038 and maximum  $\Delta/\sigma$  for H parameters were down to 0.098 for 4-PDT; the corresponding values for 5-PDT were  $< 0.02$  and 0.001. Final  $R(F)$ ,  $R(F^2)$ ,  $wR(F^2)$  and  $S$  were 0.031, 0.044, 0.079 and 1.361 for 4-PDT, and 0.031, 0.042, 0.072 and 1.150 for 5-PDT, respectively. The final difference-Fourier maps showed a maximum  $\Delta\rho$  of  $0.31 \text{ e \AA}^{-3}$  for 4-PDT and  $0.30 \text{ e \AA}^{-3}$  for 5-PDT. With all 2618 unique nonzero reflections included for 4-PDT and 1968 included for 5-PDT,  $R(F) = 0.038$  for the former and 0.042 for the latter.

**Discussion.** Positional parameters and isotropic temperature factors (or their equivalents) are listed for 4-PDT in Table 1, and those for 5-PDT are listed in Table 2.\*

Molecular configurations for 4-PDT and 5-PDT are shown in Fig. 1(a) and (b), respectively. Bond lengths, bond angles and their corresponding e.s.d.'s, calculated by the program *ORFFE4* (Busing, Martin & Levy,

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43223 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

1964), are also shown. The two isomeric molecules have bond lengths and angles closely resembling each other and other compounds containing 3*H*-1,2-dithiole-3-thione as a building unit. As can be seen in Fig. 1, the structural formulae for these two compounds are identical except that atom C(6) of the phenyl rings is linked *via* a C(6)–C(4) bond in (a) as compared with a C(6)–C(5) bond in (b). In each case, the structure is made up of the corresponding molecules held together by van der Waals forces. In 4-PDT, the closest intermolecular contact for non-H atoms is 3.374 (1) Å for S(1)…S(3); the closest such contact between non-H and H atoms is 3.00 (2) Å for S(3)…H(5). The corresponding closest contacts in 5-PDT are 3.363 (1) and 2.90 (2) Å for S(1)…S(3) and C(9)…H(4), respectively.

E.s.d.'s of bond lengths and angles involving C atoms are the order of 0.006 Å and 0.3° for 4-PDT; 0.004 Å

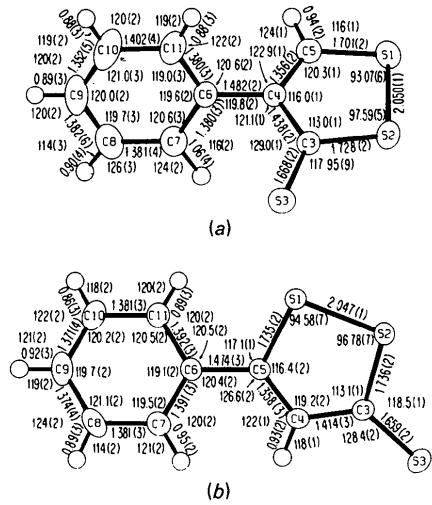


Fig. 1. Numbering scheme, bond lengths (Å) and bond angles (°) for (a) 4-PDT and (b) 5-PDT. Figs. 1 and 2 were prepared with the program ORTEPII (Johnson, 1976) in which thermal ellipsoids enclose 35% probability for non-H atoms.

and 0.2° for 5-PDT. The phenyl rings as well as the five-membered heterocyclic ring systems are planar to within 0.01 Å. Geometries of 3*H*-1,2-dithiole-3-thione in both compounds are not unlike those found in the analogous compounds containing this group of atoms (see below). However, the angle between plane normals for the six- and five-membered rings of 4-PDT is 67.40 (9)° as compared with 29.36 (8)° for 5-PDT. The measure of the twist between these two ring systems can also be expressed in terms of the torsion angle. For 4-PDT, the torsion angle C(5)–C(4)–C(6)–C(11) is –66.9 (3)° and in 5-PDT the torsion angle S(1)–C(5)–C(6)–C(11) is 29.6 (3)°. The situation can better be shown in the stereoscopic views of the molecules of Fig. 2.

So far structures of seven compounds containing 3*H*-1,2-dithiole-3-thione (or analogous 3*H*-1,2-dithiol-3-one) have been determined. Five of them are connected to six-membered aromatic rings. It is of interest to compare some of their bond lengths and the angles between plane normals for six- and five-membered rings, as shown in Table 3. It should be noted that the S–S bonds are all less than the 2.08 Å given by Pauling (1960) for an S–S single bond. Furthermore, the central C–C single bonds connecting the six- and five-membered rings are in a conjugated system, and hence the range of 1.46–1.48 Å observed in Table 3 represents partial double-bond character of the bond (Pauling, 1960). Despite the fact that the two endocyclic S–C bond distances are different in 4-PDT and nearly identical in 5-PDT (Fig. 1), it is noteworthy that the angle subtended at S(2) is larger than that subtended at S(1), as also observed in all the compounds listed in Table 3. For compounds (b), (c) and (d), angles between plane normals for the six- and five-membered rings are all less than 10°, while those angles in the present structures are much larger. As was argued in the case of olitipraz (Wei, 1983), a small dihedral angle of this kind would result in too close contacts between S(3)…H(7) and H(5)…H(11) in 4-PDT and between S(1)…H(11) and H(4)…H(7) in 5-PDT. In order to achieve the most stable conformations in the solid state, the five-membered ring is rotated around the central C–C bond to afford enough van der Waals separations between these atoms. The resulting intramolecular contacts are 3.32 (2), 3.05 (5), 2.73 (3) and 2.42 (5) Å for S(3)…H(7), H(5)…H(11), S(1)…H(11) and H(4)…H(7), respectively.

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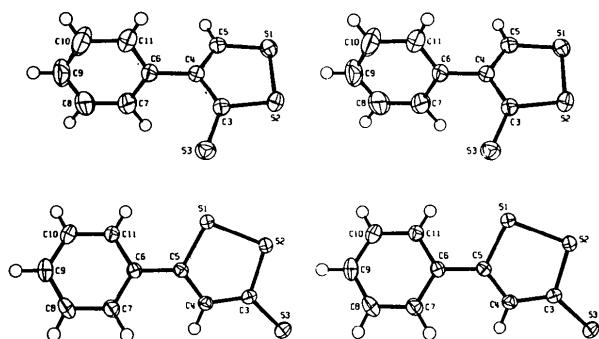


Fig. 2. Stereoscopic views of molecules 4-PDT (top) and 5-PDT (bottom).

Table 3. Comparison of some molecular parameters for compounds containing 3*H*-1,2-dithiole-3-thione or 3*H*-1,2-dithiol-3-one

|   | S-S       | C-S<br>(thione) | C-C<br>(central) | Angle between<br>plane normals<br>for six- and<br>five-membered<br>rings |
|---|-----------|-----------------|------------------|--|
| 4-Methyl-3 <i>H</i> -1,2-di-thiole-3-thione*                    | 2.047 Å   | 1.627 Å         | —                | —  |
| 4-Methyl-5-(2-pyrazinyl)-3 <i>H</i> -1,2-dithiole-3-thione†     | 2.049 (1) | 1.655 (2)       | 1.480 (3) Å      | 3.71 (8)°  |
| 5-( <i>p</i> -Methoxyphenyl)-3 <i>H</i> -1,2-dithiole-3-thione‡ | 2.041 (1) | 1.670 (2)       | 1.480 (3)        | 8.92 (9)   |
| 4-Methyl-5-(2-pyrazinyl)-3 <i>H</i> -1,2-dithiol-3-one§         | 2.047 (1) | 1.666 (3)       | 1.461 (3)        | 7.7 (1)  |
| 3 <i>H</i> -1,2-Dithiole-3-thione¶                              | 2.035 (1) | —               | 1.476 (4)        | 4.6 (1)  |
| This work: 4-PDT  | 2.038 (1) |                 | 1.477 (4)        | 9.6 (1)  |
| This work: 5-PDT  | 2.050 (3) | 1.688 (3)       | —                | —  |
|   | 2.050 (1) | 1.668 (2)       | 1.482 (2)        | 67.40 (9)  |
|   | 2.047 (1) | 1.659 (2)       | 1.474 (3)        | 29.36 (8)  |

\* Jeffrey & Shiono (1959). † Wei (1983). ‡ Wang, Lin & Wei (1985). § Wei (1985a). ¶ Wei (1985b).

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## Structure of Ammonium Methanesulfonate\*

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**Abstract.**  $\text{CH}_3\text{O}_3\text{S}^-\text{NH}_4^+$ ,  $M_r = 113.138$ , monoclinic,  $C2/m$ ,  $a = 7.4993 (6)$ ,  $b = 7.2882 (6)$ ,  $c = 9.2342 (8)$  Å,  $\beta = 93.341 (7)^\circ$ ,  $V = 503.85 (7)$  Å<sup>3</sup>,  $Z = 4$ ,  $D_m = 1.50 (2)$  by flotation,  $D_x = 1.491$  g cm<sup>-3</sup>,  $\lambda(\text{Cu } \text{K}\alpha_1) = 1.54056$  Å,  $\mu = 47.07$  cm<sup>-1</sup>,  $F(000) = 240$ ,  $T = 296 (1)$  K. Final  $R(F) = 0.045$  for 476 counter data with  $F_o^2 \geq \sigma(F_o^2)$ . Seven particularly strong reflections significantly affected by isotropic extinction also omitted from refinements. The methanesulfonate anion as well as tetrahedral ammonium cation are located in the unit cell so as to possess crystallographic

$C_s$  symmetry in each case. All molecular parameters are similar to those obtained for other related compounds. Each of three O atoms for the anion is hydrogen-bonded to a neighboring H atom of the cation and forms an infinite H-bonding chain around the crystallographic twofold screw axis at  $x = \frac{1}{4}$  (or  $\frac{3}{4}$ ) and  $z = 0$ .

**Introduction.** Further attempts to crystallize the methanesulfonate salt of an antischistosomal drug, IA-4 *N*-oxide (Hulbert, Bueding & Hartman, 1974), containing a chlorobenzothiopyranoidazole fragment (Patterson, Capell & Walker, 1960), have led to an accidental preparation of yellowish thin-plate crystals which were subsequently characterized by X-ray analysis as the

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