Although there is little difference in the C—S distances in the title compound, in compounds of this type asymmetric C—S distances are often observed with the shorter distance being to the C atom of the nitro-substituted ring (Kimura *et al.*, 1981; Korp *et al.*, 1978; Korp, Bernal & Martin, 1981). O···S—C angles close to 180° are generally observed and are 175·41 (8) and 175·31 (8)° for O(1)···S(1)—C(7) and O(2)···S(2)—C(13), respectively. A directional preference for atoms making close, non-bonded contacts with divalent sulfur has been suggested (Rosenfield, Parthasarathy & Dunitz, 1977). Nucleophiles (such as O in a nitro group) tend to approach S along the extension of one of the C—S bonds, which in the present case are the C(7)—S(1) and C(13)—S(2) bonds. It has been further suggested (Rosenfield *et al.*, 1977) that this directional preference is the result of an interaction between the nucleophile and a σ^* (C—S) orbital. Although the electron-withdrawing effects due to the nitro group cannot be overlooked, increasing the electron density in an anti-bonding orbital by such an interaction should result in a lengthening of the C—S bond and could explain the asymmetry in the C—S bond distances which is frequently observed.

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Structure of 5-(*p*-Methoxyphenyl)-3*H*-1,2-dithiole-3-thione, $C_{10}H_8OS_3$

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Abstract. $M_r = 240$, monoclinic, $P2_1/n$, $a = 10{\cdot}834$ (2), $b = 13{\cdot}446$ (2), $c = 7{\cdot}517$ (2) Å, $\beta = 104{\cdot}96$ (2)°, $V = 1057{\cdot}9$ Å^3, $Z = 4$, $D_m = 1{\cdot}50$, $D_x = 1{\cdot}51$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7093 Å, $\mu = 6{\cdot}39$ cm^{-1}, $F(000) = 496$, $T = 298$ K. Final $R = 0{\cdot}036$ for 1652 observed reflections. The molecule is composed of one methoxyphenyl ring and one five-membered heterocyclic ring containing an S—S single bond and an exocyclic C=S double bond. Each of the two individual rings is essentially planar, the angle between the two plane normals being 7·7 (1)°.

Introduction. Oltipraz, 4-methyl-5-(2-pyrazinyl)-3*H*-1,2-dithiole-3-thione, is a slow-acting schistosomidal drug which functions by reducing the glutathione stores of the worms (Bueding, Dolan & Leroy, 1982).

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Although some dithiolethiones analogous to oltipraz do not show antischistosomidal activity, they are of great interest because of their chemoprotective and antimutagenic activities.*

Following the X-ray structural report for oltipraz (Wei, 1983), the structure determinations of some representatives of this class of dithiolethiones have been carried out as part of our efforts to elucidate structure–function relationships.

Experimental. Orange-colored crystals grown at room temperature from an ethyl acetate solution. Crystal used: 0·15 × 0·22 × 0·61 mm. CAD-4 diffractometer. 25 reflections in 2θ range 22·5 to 31·86° used in refinement of cell parameters. D_m by flotation (KI/H_2O). $2\theta_{max} = 60°$, absorption correction according to ψ rotation. Ranges of h, k, l: −15 to 15, 0 to 18, 0 to 10, respectively. Three standard reflections monitored every 2 h: variation <4%. 3081 unique reflections, 1652 observed with $I \geq 3\sigma(I)$. $R(F) = 0{\cdot}036$, $wR(F) = 0{\cdot}029$, $S = 2{\cdot}423$. Weighting scheme from counting statistics. Structure solved by Patterson method. H atoms found from difference Fourier map included in final refinement. $(\Delta/\sigma)_{max} = 0{\cdot}02$. Peaks in final D map within $\pm 0{\cdot}3$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Computing programs: NRCC *SDP* PDP-11 package (Gabe & Lee, 1981) and *ORTEP* from Enraf–Nonius (1979) *SDP*.

Discussion. Positional parameters and equivalent isotropic temperature factors are listed in Table 1.† The molecular structure is shown in Fig. 1, in which bond lengths are also shown. The geometry of the methoxyphenyl ring is quite normal (see, for example, Wang, Lee & Yeh, 1984). The five-membered 1,2-dithiole-3-thione ring in the present molecule is similar to that of 4-methyl-5-(2-pyrazinyl)-3*H*-1,2-dithiole-3-thione (1) (Wei, 1983); the S(2)—S(3) distance is nearly identical with that of compound (1) and is within the range of S—S single bonds found in simple inorganic sulfur compounds, *e.g.* S_8 2·041–2·049 Å (Coppens, Yang, Blessing, Cooper & Larsen, 1977); $K_2S_4O_6$, $K_2S_3O_6$ 2·013–2·124 Å (Stewart & Szymański, 1979*a*,*b*). It is also similar to 2·036 Å in L-cysteinyl-L-cysteine (Capasso, Mattia, Mazzarella & Puliti, 1977) and can also be compared with 2·025, 2·102 Å found in $S_3(SO_2Ph)_2$ (Chen & Wang, 1984). This S—S bond length also follows the empirical relationship between the S—S bond length and the C—C—S—S torsion angle for aromatic disulfides (Higashi, Lundeen & Seff, 1978), where the S—S bond length is short (1·999–2·047 Å) when the C—C—S—S torsion angle is near 0°, and it is longer (2·059–2·108 Å) when the torsion angle is near 90°. The C(1)—S(2) and C(3)—S(3) distances are slightly shorter than S—C(sp^2) single bonds, *e.g.* 1·747 Å in $S_3(SO_2Ph)_2$ (Chen & Wang, 1984) and 1·789, 1·788 Å in $(PhS)_2$ (Sacerdoti & Gilli, 1975). Unlike those of (1), the two endocyclic S—C bond lengths are identical within their standard deviations. The C(1)—C(2) bond length is significantly shorter than that of (1), 1·436 (3) Å. These differences are probably due to the substitution of the methyl group at the C(2) position. The C(1)=S(1) double bond is comparable with those of 1·655 (2), 1·670 (2) Å for (1) and with 1·662 Å for tetraethylthiuram disulfide (Karle, Estlin & Britts, 1967).

In the title compound, the five- and six-membered rings are essentially planar with a maximum deviation of 0·006 Å for the former ($\chi^2 = 9{\cdot}27$) and 0·008 Å for the latter ($\chi^2 = 2{\cdot}89$). The methoxy O atom is 0·002 Å out of the six-membered plane and the S(1) atom is 0·02 Å out of the five-membered plane. The angle between the two plane normals is 7·7 (1)°.

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters*

$$B_{eq} = \tfrac{8}{3}\pi^2 \textstyle\sum_i \sum_j U_{ij} a^*_i a^*_j \mathbf{a}_i . \mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
S(1)	0·0090 (1)	0·3664 (1)	0·1881 (1)	5·50 (5)
S(2)	0·2170 (1)	0·2326 (1)	0·3568 (1)	4·25 (4)
S(3)	0·2093 (1)	0·0812 (1)	0·3799 (1)	4·03 (3)
C(1)	0·0588 (2)	0·2504 (2)	0·2418 (4)	3·7 (1)
C(2)	−0·0109 (2)	0·1609 (2)	0·2092 (4)	3·5 (1)
C(3)	0·0488 (2)	0·0732 (2)	0·2663 (3)	3·0 (1)
C(4)	−0·0088 (2)	−0·0258 (2)	0·2444 (4)	3·0 (1)
C(5)	−0·1336 (2)	−0·0396 (2)	0·1360 (5)	3·5 (1)
C(6)	−0·1895 (2)	−0·1323 (2)	0·1181 (4)	4·0 (1)
C(7)	−0·1208 (2)	−0·2144 (2)	0·2044 (4)	3·6 (1)
C(8)	0·0028 (2)	−0·2023 (2)	0·3102 (4)	3·9 (1)
C(9)	0·0573 (2)	−0·1089 (2)	0·3285 (4)	3·9 (1)
C(10)	−0·1121 (3)	−0·3904 (2)	0·2500 (5)	5·3 (1)
O	−0·1835 (2)	−0·3036 (1)	0·1778 (3)	4·9 (1)

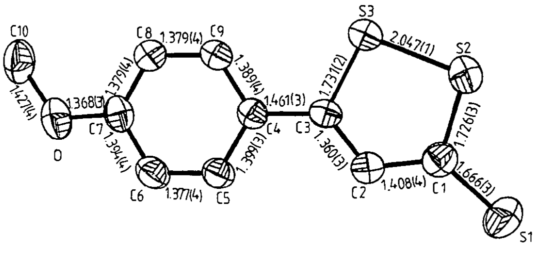

Fig. 1. *ORTEP* drawing with 50% probability in thermal motion.

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* Private communication to CHW from Professor Ernest Bueding, Johns Hopkins University, June, 1982.

† Lists of H parameters, anisotropic temperature factors and structure factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42204 (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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(Acétamido-3 phényl-2 propylidène)-4,4′ Bis(acétate de phényle), $C_{27}H_{27}NO_5$

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(*Reçu le* 3 *décembre* 1984, *accepté le* 26 *avril* 1985)

Abstract. $M_r = 445{\cdot}5$, orthorhombic, $Pbca$, $a = 9{\cdot}458\,(2)$, $b = 32{\cdot}090\,(4)$, $c = 16{\cdot}359\,(2)$ Å, $V = 4965\,(2)$ Å^3, $Z = 8$, $D_x = 1{\cdot}19$ Mg m^{-3}, $\lambda(\mathrm{Cu}\ K\bar{\alpha}) = 1{\cdot}5424$ Å, $\mu = 0{\cdot}30$ mm^{-1}, $F(000) = 1728$, $T = 293$ K, $R = 0{\cdot}053$ for 2212 observations. The relative angles between the aromatic rings are similar to those observed for triphenylethylene derivatives but the general shape of the molecule is very different. There are no unusual bond distances or angles. There is an intermolecular hydrogen bond [N···O(acetamido) 2·817 (7) Å].

Introduction. De nombreux dérivés du triphényl-ethylène, par exemple le clomiphène, chloro-2 [*p*-(diéthylamino-2 éthoxy)phényl]-1 diphényl-1,2 éthylène (Ernst & Hite, 1976), ou le tamoxifène [*p*-(diméthylamino-2 éthoxy)phényl]-1 *trans*-diphényl-1,2 butène-1 (Précigoux, Courseille, Geoffre & Hospital, 1979), sont connus pour leurs propriétés anti-estrogènes et sont utilisés en clinique en tant qu'inducteurs de l'ovulation (Klopper & Hall, 1971) ou dans le traitement de cancers hormono dépendants (Legha & Carter, 1976). D'autre part, parmi les dérivés du triphényléthylène, certains sont de bons inhibiteurs de la prostaglandine synthétase tandis que les dérivés tels que le composé titre, dont la double liaison centrale est saturée, s'avèrent plutôt être de mauvais inhibiteurs (Gilbert, Miquel, Précigoux, Hospital, Raynaud, Michel & Crastes de Paulet, 1983).

L'analyse cristallographique de ce composé a été entreprise afin d'observer les modifications conformationnelles du squelette triphényléthylène introduites par la saturation de la double liaison centrale.

Partie expérimentale. Cristallisation par évaporation d'une solution (1/1) méthanol/propanol-2. Cristal de dimensions 0,1 × 0,3 × 0,3 mm. Diffractomètre Nonius CAD-4; monochromateur de graphite. Paramètres de la maille affinés par moindres carrés en utilisant 25 réflexions ($\theta_{\max} = 54°$). Corrections de Lorentz et de polarisation, absorption ignorée. 4213 réflexions indépendantes mesurées pour $2 \le 2\theta \le 140°$, $0 \le h \le 11$,