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)-3H-1,2-dithiole-3-thione, C_{10}H_{8}OS_{3}

BY YU WANG* AND H. C. LIN

Department of Chemistry, National Taiwan University, Taipei, Taiwan

AND CHIN HSUAN WEI

Biology Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee 37831, USA

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Abstract. \( M_r = 240 \), monoclinic, \( P2_1/n \), \( a = 10.834 (2) \), \( b = 13.446 (2) \), \( c = 7.517 (2) \ \text{Å} \), \( \beta = 104.96 (2) \), \( V = 1057.9 \ \text{Å}^3 \), \( Z = 4 \), \( D_m = 1.50 \), \( D_0 = 1.51 \ \text{g cm}^{-3} \), \( \lambda(\text{Mo } K\alpha) = 0.7093 \ \text{Å} \), \( \mu = 6.39 \ \text{cm}^{-1} \), \( F(000) = 496 \), \( T = 298 \ \text{K} \). Final \( R = 0.036 \) 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)-3H-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).
Although some dithiolethiones analogous to oltipraz do not show antischistosomidal activity, they are of great interest because of their chemoprotective and anti-mutagenic 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 x 0.22 x 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/H2O). \( 2\theta_{\text{max}} = 60° \), 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 \( I > 3\sigma(I) \).<br>

In the title compound, the five- and six-membered rings are essentially planar with a maximum deviation of 0.006 Å for the former (\( \chi^2 = 9.27 \)) and 0.008 Å for the latter (\( \chi^2 = 2.89 \)). The methoxy O atom is 0.002 Å out of the five-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

<table>
<thead>
<tr>
<th></th>
<th>x</th>
<th>y</th>
<th>z</th>
<th>Beq(Å²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S(1)</td>
<td>0.00358 (1)</td>
<td>0.03644 (1)</td>
<td>0.01881 (1)</td>
<td>5.5 (5)</td>
</tr>
<tr>
<td>S(2)</td>
<td>0.2190 (1)</td>
<td>0.2352 (1)</td>
<td>0.03586 (1)</td>
<td>4.25 (4)</td>
</tr>
<tr>
<td>S(3)</td>
<td>0.2079 (1)</td>
<td>0.0312 (1)</td>
<td>0.03799 (1)</td>
<td>4.03 (3)</td>
</tr>
<tr>
<td>C(1)</td>
<td>0.0588 (2)</td>
<td>0.2504 (2)</td>
<td>0.02418 (4)</td>
<td>3.7 (1)</td>
</tr>
<tr>
<td>C(2)</td>
<td>-0.0108 (2)</td>
<td>0.2009 (2)</td>
<td>0.01026 (2)</td>
<td>2.5 (1)</td>
</tr>
<tr>
<td>C(3)</td>
<td>0.0488 (2)</td>
<td>0.0732 (2)</td>
<td>0.2663 (3)</td>
<td>3.0 (1)</td>
</tr>
<tr>
<td>C(4)</td>
<td>0.0110 (2)</td>
<td>0.2052 (2)</td>
<td>0.2444 (3)</td>
<td>3.0 (1)</td>
</tr>
<tr>
<td>C(5)</td>
<td>-0.0136 (1)</td>
<td>0.3936 (1)</td>
<td>0.1360 (5)</td>
<td>3.5 (1)</td>
</tr>
<tr>
<td>C(6)</td>
<td>-0.1895 (2)</td>
<td>-0.1323 (2)</td>
<td>0.1181 (4)</td>
<td>4.0 (1)</td>
</tr>
<tr>
<td>C(7)</td>
<td>-0.1208 (1)</td>
<td>0.2141 (4)</td>
<td>0.2404 (4)</td>
<td>3.6 (1)</td>
</tr>
<tr>
<td>C(8)</td>
<td>0.0108 (2)</td>
<td>-0.2023 (2)</td>
<td>0.3102 (4)</td>
<td>3.9 (1)</td>
</tr>
<tr>
<td>C(9)</td>
<td>0.0573 (2)</td>
<td>0.0108 (2)</td>
<td>0.3285 (4)</td>
<td>3.9 (1)</td>
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<tr>
<td>C(10)</td>
<td>0.0258 (2)</td>
<td>-0.3904 (2)</td>
<td>0.2500 (5)</td>
<td>5.3 (1)</td>
</tr>
<tr>
<td>O</td>
<td>-0.1835 (2)</td>
<td>0.3036 (1)</td>
<td>0.1778 (3)</td>
<td>4.9 (1)</td>
</tr>
</tbody>
</table>

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

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* Private communication to CHW from Professor Ernest Blessing, 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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References


(Acétamido-3 phényl-2 propylidène)-4,4’ Bis(acétate de phényle), C_{27}H_{27}NO_{5}

PAR GILLES PRÉCIGOUX ET MICHEL HOSPITAL

Laboratoire de Cristallographie et Physique Cristalline associé au CNRS, 351 cours de la Libération, 33405 Talence, France

ET JEAN-FRANÇOIS MIQUEL ET JACQUES GILBERT

Centre d'Etude et de Recherche de Chimie Organique Appliquée, 2 rue H. Dunant, 94320 Thiais, France

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

Abstract. \( M_r = 445.5 \), orthorhombic, \( Pbc\alpha \), \( a = 9.458 (2), b = 32.090 (4), c = 16.359 (2) \AA, \( V = 4965 (2) \AA^3, Z = 8, D_x = 1.19 \text{ Mg m}^{-3}, \lambda (\text{Cu} K\alpha) = 1.5424 \text{Å}, \mu = 0.30 \text{ mm}^{-1}, F(000) = 1728, T = 293 \text{ K}, R = 0.053 \) 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 \[ \text{N}...\text{O(acetamido)} \approx 2.817 (7) \text{ Å}. \]

Introduction. De nombreux dérivés du triphényl-éthylène, par exemple le clomiphène, chloro-2 \([p-(\text{diéthylamino-2 éthoxy})\text{phényl}]\) diphényl-1,2 éthylène (Ernst & Hite, 1976), ou le tamoxifène \([p-(\text{diméthylamino-2 éthoxy})\text{phényl}]\) trans-diphényl-1,2 buténe-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 triphényl-éthylène, 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 triphényl-éthylène 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. Diffactomètre Nonius CAD-4; monochromateur de graphite. Paramètres de la maille affinés par moindres carrés en utilisant 25 réflexions \( \theta_{\text{max}} = 54^\circ \). Corrections de Lorentz et de polarisation, absorption ignorée. 4213 réflexions indépendantes mesurées pour \( 2 \leq 2\theta \leq 140^\circ, 0 \leq h \leq 11, \)

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