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## 1-(1,3-Dithian-2-yl)-2-phenylethanone

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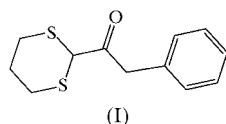
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The title molecule, C<sub>12</sub>H<sub>14</sub>OS<sub>2</sub>, has approximate C<sub>s</sub> symmetry. The dithiane ring adopts a chair conformation with the acetyl substituent in an axial orientation. Weak intermolecular C—H···O hydrogen bonds link molecules into a chain along the y axis.

## Comment

1-(1,3-Dithian-2-yl)-2-phenylethanone, (I), was first synthesized by Tolstikov *et al.* (1989). In the course of our studies of synthetic tropane analogues (Tavasli, 1999), this compound was obtained by a different route and its structure is presented here.



The dithiane ring adopts the chair conformation with the phenylacetyl substituent in an axial orientation. The carbonyl group is directed towards the dithiane S atoms and the C=C double bond has an almost ideally staggered orientation between the C2—S bonds; the S1—C2—C7—O and S3—C2—C7—O torsion angles are  $-71.5$  (1) and  $58.7$  (1)°, respectively. The molecule has approximate (local) C<sub>s</sub> symmetry. A survey of the April 2000 release of the Cambridge Structural Database (Allen & Kennard, 1993) revealed 31 structures with a dithiane ring monosubstituted at the 2-position, all of which displayed a chair conformation of the ring. The substituent was axial in ten compounds, equatorial in 20 and in one, namely bis(1,3-dithian-2-yl)methanol, both orientations were observed simultaneously (Bulman-Page *et al.*, 1987). It is

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noteworthy that an electronegative atom bonded to the  $\alpha$ -atom of the side chain usually favours the axial orientation, as in (I). The gas-phase electron diffraction study of unsubstituted dithiane (Adams & Bartell, 1977) also revealed the chair conformation and bond lengths [average C—S 1.812 (3) and C—C 1.533 (2) Å] very close to those observed in (I) [average C—S 1.817 (5) and C—C 1.519 (2) Å]. The torsion angles in the gas phase, S—C—S—C 61.4, C—S—C—C 58.3 and S—C—C—C 64.1°, differ slightly from those in (I), which average 53.1, 56.9 and 68.0°, respectively, obviously due to steric repulsion between the ring and the substituent.

Molecules related by the *b*-glide plane, *i.e.* the symmetry operation  $(\frac{3}{2} - x, \frac{1}{2} + y, z)$ , form a very weak C2—H···O hydrogen bond [C—H 0.95 (2), C···O 3.220 (2) and H···O(2) 2.39 Å; angles C—H—O 146 (2) and H—O—C 136 (1)°].

## Experimental

A 1.6 M solution of *n*-butyllithium (0.5 g, 7.3 mmol) in hexane was added dropwise at 213 K to a solution of 1,3-dithiane (0.8 g, 7 mmol) in THF (25 ml) and stirred for 2.5 h at 253 K. *N*-Methyl-*N*-methoxyphenylacetamide was prepared according to Sibous & Tipping (1993) and added to the mixture at 213 K (1.5 g, 8.4 mmol, in 5 ml of THF). The reaction was stirred for 18 h at 213 K to room temperature, then quenched with water. Organic products were extracted into CH<sub>2</sub>Cl<sub>2</sub>, washed with HCl, saturated NaHCO<sub>3</sub> and water, dried over MgSO<sub>4</sub>, concentrated *in vacuo* and purified over alumina, giving (I) as a white solid. X-ray quality crystals were obtained from ethyl acetate/petrol (3:7).

## Crystal data

C<sub>12</sub>H<sub>14</sub>OS<sub>2</sub>  
M<sub>r</sub> = 238.35  
Orthorhombic, *Pbca*  
a = 11.735 (1) Å  
b = 9.775 (1) Å  
c = 20.695 (2) Å  
V = 2373.9 (4) Å<sup>3</sup>  
Z = 8  
D<sub>x</sub> = 1.334 Mg m<sup>-3</sup>

Mo K $\alpha$  radiation  
Cell parameters from 411 reflections  
 $\theta$  = 10–20°  
 $\mu$  = 0.419 mm<sup>-1</sup>  
T = 150 (2) K  
Block, colourless  
0.40 × 0.33 × 0.22 mm

## Data collection

SMART 1K CCD area-detector diffractometer  
 $\omega$  scans  
Absorption correction: by integration (*XPREP/SHELXTL*; Bruker, 1997);  $R_{\text{int}} = 0.029$  before correction  
 $T_{\text{min}} = 0.878$ ,  $T_{\text{max}} = 0.927$

10 906 measured reflections  
2846 independent reflections  
2533 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.025$   
 $\theta_{\text{max}} = 28^\circ$   
 $h = -16 \rightarrow 14$   
 $k = -13 \rightarrow 12$   
 $l = -28 \rightarrow 14$

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.030$   
 $wR(F^2) = 0.080$   
S = 1.094  
2804 reflections  
192 parameters  
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0273P)^2 + 1.3282P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.27 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.26 \text{ e \AA}^{-3}$

**Table 1**

Selected geometric parameters (Å, °).

S1—C2	1.8143 (14)	C4—C5	1.520 (2)
S1—C6	1.8165 (15)	C5—C6	1.518 (2)
C2—C7	1.528 (2)	C7—O	1.213 (2)
C2—S3	1.8113 (15)	C7—C8	1.511 (2)
S3—C4	1.824 (2)	C8—C9	1.509 (2)
C2—S1—C6	101.14 (7)	S3—C2—S1	115.12 (7)
C7—C2—S3	113.13 (9)	C2—S3—C4	102.13 (7)
C7—C2—S1	109.88 (9)		

Refined C—H distances are in the range 0.92 (2)–0.98 (2) Å.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1996); program(s) used to solve structure: *SHELXTL* (Bruker, 1997); program(s) used to refine

structure: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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