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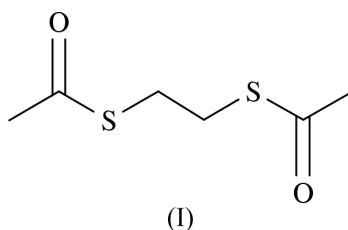
## Key indicators

Single-crystal X-ray study  
 $T = 183\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$   
 $R$  factor = 0.040  
 $wR$  factor = 0.118  
Data-to-parameter ratio = 20.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

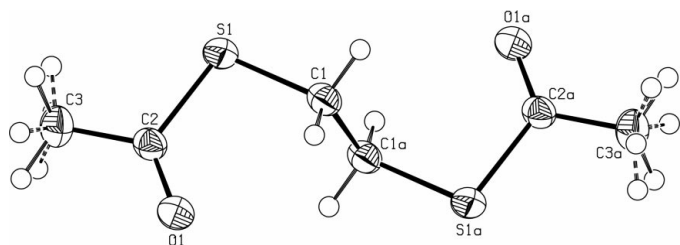
## Ethanedithiol diacetate

Ethane-1,2-diyl *S,S'*-bis(thioacetate),  $\text{H}_3\text{CC}(\text{O})\text{SCH}_2\text{CH}_2\text{S}-\text{C}(\text{O})\text{CH}_3$  or  $\text{C}_6\text{H}_{10}\text{O}_2\text{S}_2$ , forms centrosymmetric molecules in the solid state and the molecular structure determined by X-ray crystallography is in good agreement with that obtained by density functional geometry optimization. The planarity of the  $\text{O}=\text{C}-\text{S}-\text{C}$  fragment, which is also found in structures of other thioacetates, is attributed to a strong  $n_p(\text{S})-\pi^*(\text{C}-\text{O})$  orbital interaction.Received 8 March 2001  
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## Comment

Thioesters are very important acetylating agents in biochemical processes as well as in many chemical transformations (Nicolaou, 1977; Hirama *et al.*, 1979; Zheng *et al.*, 1999). We obtained ethanedithiol diacetate,  $\text{H}_3\text{CC}(\text{O})\text{SCH}_2\text{CH}_2\text{S}-\text{C}(\text{O})\text{CH}_3$ , (I), as a by-product in the synthesis of ethanedithiol monoacetate,  $\text{HSCH}_2\text{CH}_2\text{SC}(\text{O})\text{CH}_3$ , (II), according to a literature procedure (Wiesler *et al.*, 1996). Crystals of (I) precipitated at 278 K from its solution in (II). They were washed with cold petroleum ether and identified and checked for purity by  $^1\text{H}$  NMR (Bauer *et al.*, 1965). One of them was selected for single-crystal X-ray diffraction.

The molecular structure of (I) as found in the solid state is depicted in Fig. 1. Table 1 shows selected structural parameters from the XRD experiment in comparison with those obtained by a density functional (DF) geometry optimization. Apart from the two  $\text{S}-\text{C}$  and the  $\text{C}2-\text{C}3$  distances, the DF structural parameters agree quite well with the experimental ones, in spite of the fact that the former refer to an isolated molecule and the latter do not (see Table 1). We attribute the differences in the bond distances mentioned to the chosen level of theory and the agreement among most of the above values to the absence of significant intermolecular interactions in the crystal. The structural parameters of (I) agree well with those found for other compounds exhibiting an *S*-acetyl moiety (Evans *et al.*, 1999; Divjakovic *et al.*, 1992; Huber *et al.*, 1984; Kiel *et al.*, 1974; Mackay *et al.*, 1992; Mattes *et al.*, 1977; Mattes & Waldmann, 1983). This implies that the *S*-acetyl fragment is a relatively rigid structural unit. An analysis of the bonding situation in terms of natural bond orbitals (Reed *et*



**Figure 1**  
ORTEP (Johnson, 1976) view of the centrosymmetric molecule of (I). Displacement ellipsoids are shown at the 50% probability level. H atoms are shown as spheres of fixed radius. The methyl group is disordered (0.45/0.55).



**Figure 2**  
Scheme of orbital interaction within the  $-C(O)-S$  moiety of (I). See text for details.

*al.*, 1988) reveals a strong delocalization of electron density within the  $O=C-S$  fragment. The  $p$ -type lone pair of the S atom interacts strongly with the  $\pi^*(C-O)$  orbital (see Fig. 2a) and the  $p$ -type lone pair of the O-atom interacts strongly with the  $\sigma^*(S-C)$  orbital (see Fig. 2b). The  $n_p(S)-\pi^*(C-O)$  interaction explains very well the nearly planar conformation of the  $C1-S1-C2-O1$  moiety.

## Experimental

### Crystal data

$C_6H_{10}O_2S_2$	$D_x = 1.385 \text{ Mg m}^{-3}$
$M_r = 178.26$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 2083 reflections
$a = 5.1677 (6) \text{ \AA}$	$\theta = 3.3-28.3^\circ$
$b = 7.1944 (9) \text{ \AA}$	$\mu = 0.56 \text{ mm}^{-1}$
$c = 11.6869 (15) \text{ \AA}$	$T = 183 (2) \text{ K}$
$\beta = 100.449 (2)^\circ$	Block, colourless
$V = 427.30 (9) \text{ \AA}^3$	$1.26 \times 0.37 \times 0.30 \text{ mm}$
$Z = 2$	

### Data collection

Smart CCD diffractometer	$R_{\text{int}} = 0.101$
$\theta$ and $\varphi$ scans	$\theta_{\text{max}} = 28.3^\circ$
2629 measured reflections	$h = -6 \rightarrow 6$
1042 independent reflections	$k = -9 \rightarrow 5$
968 reflections with $I > 2\sigma(I)$	$l = -15 \rightarrow 15$

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0565P)^2 + 0.0812P]$
$R[F^2 > 2\sigma(F^2)] = 0.040$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.118$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.10$	$\Delta\rho_{\text{max}} = 0.45 \text{ e \AA}^{-3}$
1042 reflections	$\Delta\rho_{\text{min}} = -0.25 \text{ e \AA}^{-3}$
50 parameters	Extinction correction: <i>SHELXL97</i>
H-atom parameters constrained	Extinction coefficient: 0.19 (2)

**Table 1**

Comparison of selected structural parameters ( $\text{\AA}$ ,  $^\circ$ ) from the molecular structures of (I) in the solid state (XRD) and from density functional (DF) geometry optimization [B3LYP/6-311+G(2d,p)] (Frisch *et al.*, 1995).

	XRD	DF
S1—C1	1.806 (2)	1.833
S1—C2	1.772 (1)	1.798
C1—C1a	1.516 (3)	1.522
C2—C3	1.494 (2)	1.511
O1—C2	1.208 (2)	1.206
C2—S1—C1	100.7 (1)	100.2
C1a—C1—S1	112.1 (1)	112.3
O1—C2—C3	124.0 (1)	123.5
O1—C2—S1	122.4 (1)	123.1
C3—C2—S1	113.6 (1)	113.5
C2—S1—C1—C1a	80.3 (1)	82.0
C1—S1—C2—O1	1.5 (2)	0.2
C1—S1—C2—C3	-179.1 (1)	-179.7

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP* (Johnson, 1976).

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