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1,3-Dithiolan-2-one

Eric Reinheimer, John Bacsa* and Kim R. Dunbar

Department of Chemistry, Texas A & M University, PO Box 30012, College Station, Texas 77842-3012, USA

Correspondence e-mail: jbacsa@mail.chem.tamu.edu

Key indicators

Single-crystal X-ray study
T = 110 K
Mean σ(C–C) = 0.002 Å
R factor = 0.020
wR factor = 0.052
Data-to-parameter ratio = 15.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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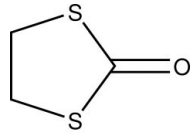
The title compound, $C_3H_4OS_2$, possesses pseudo-twofold symmetry and consists of a twisted five-membered ring of three C and two S atoms, with a ketone O atom in an equatorial position.

Comment

We report here, for the first time, the crystal and molecular structure of 1,3-dithiolan-2-one, (I). The molecule consists of a five-membered ring of three C and two S atoms, with a ketone O atom in an equatorial position (Fig. 1). Selected geometric parameters are given in Table 1.



(I)

Atoms O1, C1, S1 and S2 are in a distorted trigonal planar arrangement, but atoms C2 and C3 are in slightly distorted tetrahedral environments. The ring is in a twist (T) conformation, less typical of five-membered rings, with puckering parameters $\varphi = 127^\circ$ and $q = 0.431$ Å (Cremer & Pople, 1975). The puckering is best described by twisting the groups on C2 and C3 (Evans & Boeyens, 1989). The molecule has approximate C_2 symmetry. In the crystal structure, symmetry-related molecules are held together by very weak hydrogen bonds between the keto O atoms and the methylene H atoms (Fig. 2 and Table 2).

Experimental

The title compound, (I), was prepared by stirring a solution of vinylene trithiocarbonate (3.5 g) and mercuric acetate (19.4 g) in chloroform/acetic acid (3:1 v/v, 100 ml) under an atmosphere of N_2

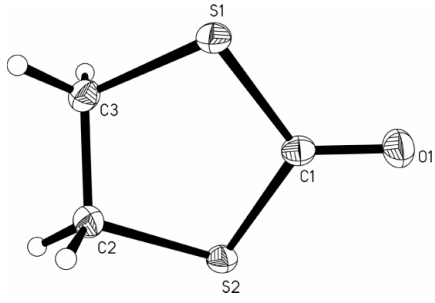


Figure 1
View of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as circles of arbitrary radius.

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for 12 h. The solution was filtered in air through celite and washed with chloroform. The organic phases were refluxed under N_2 with activated charcoal for 2 h. The solution was filtered and washed with aqueous $NaHCO_3$ and dried over $MgSO_4$. The solution was allowed to evaporate and large crystals grew in the solution over a period of 5 d.

Crystal data

$C_3H_4OS_2$
M_r = 120.18
Monoclinic, $P2_1/c$
a = 8.0397 (16) Å
b = 5.2020 (10) Å
c = 11.318 (2) Å
β = 90.426 (4)°
V = 473.31 (16) Å^3
Z = 4
D_x = 1.687 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3398 reflections
θ = 2.0–27.5°
μ = 0.96 mm^{-1}
T = 110 (2) K
Prism, light yellow
0.28 × 0.24 × 0.22 mm

Data collection

Bruker SMART 1K CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)
T_{min} = 0.775, T_{max} = 0.817
3916 measured reflections
1078 independent reflections
985 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 27.6°
$h = -10 \rightarrow 10$
$k = -5 \rightarrow 6$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.052
S = 1.02
1078 reflections
71 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0369P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C1	1.7725 (11)	S2—C2	1.8143 (12)
S1—C3	1.8128 (11)	O1—C1	1.2068 (14)
S2—C1	1.7779 (11)	C3—C2	1.5274 (15)
C1—S1—C3	96.60 (5)	S1—C1—S2	113.42 (6)
C1—S2—C2	96.57 (5)	C2—C3—S1	108.32 (7)
O1—C1—S1	124.10 (8)	C3—C2—S2	108.29 (8)
O1—C1—S2	122.48 (8)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2B···O1^{i}	0.965 (13)	2.635 (13)	3.3455 (14)	130.7 (10)
C3—H3B···O1^{ii}	0.947 (15)	2.613 (15)	3.3342 (14)	133.3 (12)
C3—H3A···O1^{iii}	0.948 (15)	2.635 (15)	3.5087 (15)	153.4 (11)

Symmetry codes: (i) $x, -\frac{1}{2}-y, \frac{1}{2}+z$; (ii) $x, \frac{1}{2}-y, \frac{1}{2}+z$; (iii) $-x, y-\frac{1}{2}, \frac{3}{2}-z$.

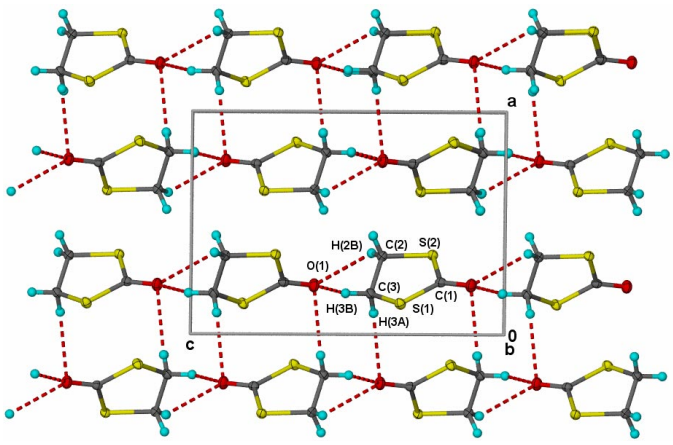

Figure 2
Projection of the molecular packing of (I) on the *ac* plane, showing the hydrogen bonding (dashed lines).

All the H atoms were located in difference electron-density maps and refined isotropically.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *X-SEED* (Barbour, 2001) and *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *PLATON* (Spek, 2003).

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