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5,6-Dihydro-2*H*-1,3-dithiolo[4,5-*b*][1,4]-dioxine-2-thione

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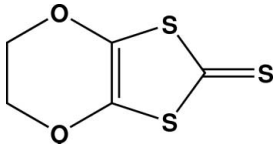
Key indicators: single-crystal X-ray study; T = 223 K; mean σ(C–C) = 0.003 Å; R factor = 0.031; wR factor = 0.083; data-to-parameter ratio = 14.5.

The title molecule, $C_5H_4O_2S_3$, consists of a planar [mean deviation = 0.020 (1) Å] 1,3-dithiole-2-thione unit with an ethylenedioxy group in the 4,5-positions. The dioxine ring is in a twist-chair conformation.

Related literature

For related structures, see: Kanchanadevi *et al.* (2010); Rizvi *et al.* (2010); Suzuki *et al.* (1989); Xu *et al.* (2009); Sugumar *et al.* (2008). For the synthesis of the title compound, see: Hartke & Lindenblatt (1990); Suzuki *et al.* (1989).



Experimental

Crystal data

$C_5H_4O_2S_3$
M_r = 192.26
Monoclinic, $P2_1/n$
a = 5.4645 (8) Å
b = 13.430 (2) Å
c = 9.8189 (16) Å
β = 91.294 (3)°
V = 720.41 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.96 mm^{-1}
T = 223 K
0.55 × 0.20 × 0.20 mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan (*REQAB*; Jacobson, 1998)
T_{min} = 0.605, T_{max} = 0.826
3605 measured reflections
1332 independent reflections
1199 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.083
S = 1.17
1332 reflections
92 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AA2007).

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supporting information

Acta Cryst. (2011). E**67**, o1404 [doi:10.1107/S1600536811017417]

5,6-Dihydro-2*H*-1,3-dithiolo[4,5-*b*][1,4]dioxine-2-thione

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S1. Comment

The title compound is a precursor to ethylenedioxotetrathiafulvalene(EDO-TTF). EDO-TTF has attracted much interest due to ultrafast gigantic photo-response in organic salts $(EDO\text{-}TTF)_2PF_6$.

In the title compound (Fig.1), the five-membered ring and attached S and O atoms are essentially coplanar, mean deviation from the mean plane 0.020 (1) Å. The C—S bond lengths range from 1.728 to 1.748 Å, it is smaller than that typical of C—S bond lengths, 1.82 Å, suggesting a degree of conjugation in the dithiole-2-thione system. The dioxine ring is in a twist-chair conformation.

S2. Experimental

The $C_5H_4S_3O_2$ was prepared according to the literature (Suzuki *et al.*, 1989).Yellow crystals were obtained from slow evaporation of a dichloromethane solution at room temperature.

S3. Refinement

The H atoms were included at geometrically idealized positions and refined in riding-model approximation with the following constraints: C—H distances were set to 0.98 Å for methylene H-atoms, with U_{iso}(H) = 1.2(C) U_{eq} of the carrier atoms.

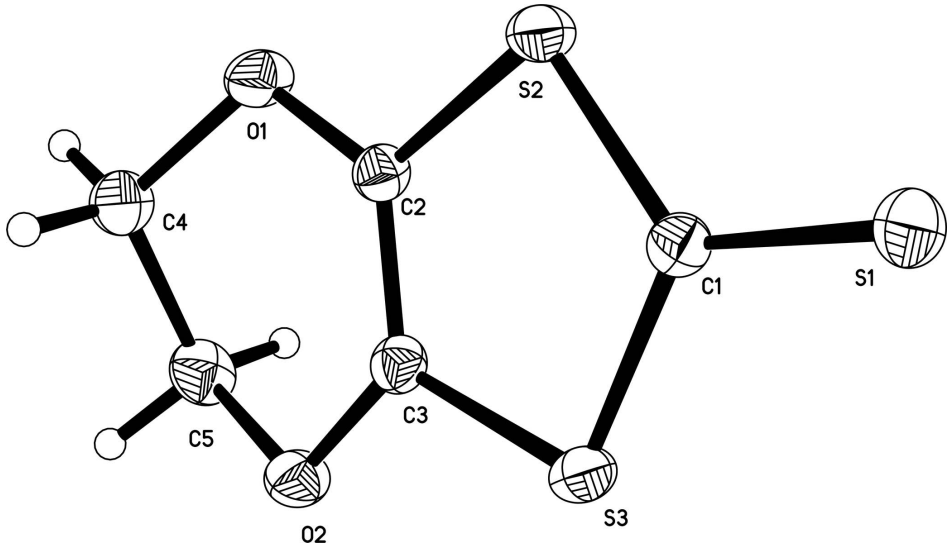

Figure 1

The molecular structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level.

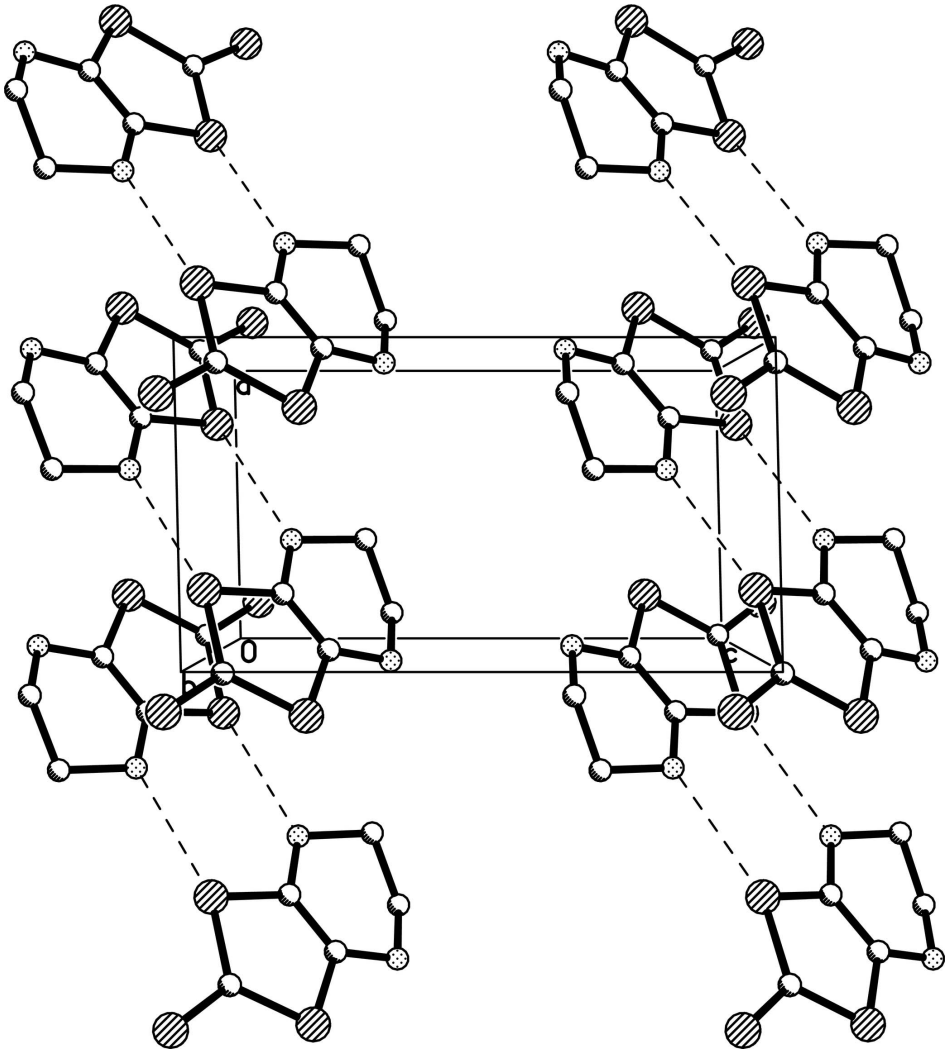

Figure 2

The crystal packing diagram view along the crystallographic *b* axis.

5,6-Dihydro-2*H*-1,3-dithiolo[4,5-*b*][1,4]dioxine-2-thione

Crystal data

$C_5H_4O_2S_3$
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c = 9.8189 (16) Å
β = 91.294 (3)°
V = 720.41 (19) Å^3
Z = 4
$F(000)$ = 392
D_x = 1.773 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71075 Å
Cell parameters from 3311 reflections
θ = 3.0–27.5°
μ = 0.96 mm^{-1}
T = 223 K
Block, yellow
0.55 × 0.20 × 0.20 mm

Data collection

Rigaku Saturn diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 14.63 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (REQAB; Jacobson, 1998)
T_{min} = 0.605, T_{max} = 0.826
3605 measured reflections
1332 independent reflections
1199 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 25.5°, θ_{min} = 3.0°
h = −6→6
k = −14→16
l = −11→9

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.083
S = 1.17
1332 reflections
92 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0486P)^2 + 0.013P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
S1	1.15131 (11)	0.20574 (4)	1.05803 (6)	0.0394 (2)
S2	0.77835 (10)	0.36220 (4)	0.99990 (5)	0.03292 (19)
S3	1.18427 (10)	0.33798 (4)	0.81841 (6)	0.03387 (19)
O1	0.6189 (3)	0.51707 (11)	0.85167 (16)	0.0381 (4)
O2	1.0238 (3)	0.49167 (11)	0.66988 (16)	0.0367 (4)
C1	1.0418 (4)	0.29782 (14)	0.9629 (2)	0.0282 (5)
C2	0.7954 (4)	0.44643 (15)	0.8651 (2)	0.0276 (4)
C3	0.9812 (4)	0.43492 (14)	0.7810 (2)	0.0280 (5)
C4	0.6183 (4)	0.55689 (18)	0.7147 (2)	0.0412 (6)
H4A	0.5199	0.6179	0.7115	0.049*
H4B	0.5419	0.5086	0.6523	0.049*
C5	0.8699 (5)	0.57960 (16)	0.6687 (2)	0.0413 (6)
H5A	0.8607	0.6069	0.5761	0.050*
H5B	0.9437	0.6303	0.7286	0.050*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0427 (4)	0.0365 (3)	0.0392 (4)	0.0112 (2)	0.0020 (3)	0.0078 (2)
S2	0.0318 (3)	0.0340 (3)	0.0333 (3)	0.0071 (2)	0.0086 (2)	0.0068 (2)
S3	0.0289 (3)	0.0393 (3)	0.0337 (3)	0.0073 (2)	0.0071 (2)	0.0031 (2)
O1	0.0372 (8)	0.0368 (8)	0.0406 (9)	0.0126 (7)	0.0095 (7)	0.0107 (7)
O2	0.0364 (8)	0.0400 (9)	0.0340 (9)	0.0041 (7)	0.0079 (7)	0.0105 (6)
C1	0.0288 (11)	0.0264 (11)	0.0295 (11)	0.0008 (8)	0.0001 (9)	−0.0033 (8)
C2	0.0276 (10)	0.0260 (10)	0.0293 (11)	0.0013 (8)	0.0010 (8)	0.0018 (8)
C3	0.0256 (10)	0.0289 (10)	0.0296 (11)	−0.0021 (8)	−0.0003 (9)	0.0015 (8)
C4	0.0417 (13)	0.0424 (12)	0.0394 (13)	0.0116 (11)	0.0019 (10)	0.0134 (10)
C5	0.0499 (14)	0.0354 (12)	0.0387 (13)	0.0029 (10)	0.0026 (11)	0.0091 (10)

Geometric parameters (Å, º)

S1—C1	1.654 (2)	O2—C5	1.450 (3)
S2—C1	1.725 (2)	C2—C3	1.332 (3)
S2—C2	1.746 (2)	C4—C5	1.488 (3)
S3—C1	1.720 (2)	C4—H4A	0.9800
S3—C3	1.744 (2)	C4—H4B	0.9800
O1—C2	1.357 (2)	C5—H5A	0.9800
O1—C4	1.447 (3)	C5—H5B	0.9800
O2—C3	1.356 (2)		
C1—S2—C2	96.07 (10)	O1—C4—C5	112.07 (18)
C1—S3—C3	96.32 (9)	O1—C4—H4A	109.2
C2—O1—C4	109.56 (15)	C5—C4—H4A	109.2
C3—O2—C5	110.75 (15)	O1—C4—H4B	109.2
S1—C1—S3	122.39 (12)	C5—C4—H4B	109.2
S1—C1—S2	123.28 (13)	H4A—C4—H4B	107.9
S3—C1—S2	114.33 (12)	O2—C5—C4	111.73 (18)
C3—C2—O1	124.84 (18)	O2—C5—H5A	109.3
C3—C2—S2	116.70 (16)	C4—C5—H5A	109.3
O1—C2—S2	118.45 (14)	O2—C5—H5B	109.3
C2—C3—O2	125.52 (18)	C4—C5—H5B	109.3
C2—C3—S3	116.52 (16)	H5A—C5—H5B	107.9
O2—C3—S3	117.97 (14)		
C3—S3—C1—S1	−177.35 (13)	O1—C2—C3—S3	−179.51 (15)
C3—S3—C1—S2	2.19 (13)	S2—C2—C3—S3	−0.7 (2)
C2—S2—C1—S1	177.05 (14)	C5—O2—C3—C2	11.6 (3)
C2—S2—C1—S3	−2.48 (13)	C5—O2—C3—S3	−168.64 (15)
C4—O1—C2—C3	17.2 (3)	C1—S3—C3—C2	−0.90 (19)
C4—O1—C2—S2	−161.53 (15)	C1—S3—C3—O2	179.31 (15)
C1—S2—C2—C3	1.93 (18)	C2—O1—C4—C5	−45.5 (3)
C1—S2—C2—O1	−179.19 (16)	C3—O2—C5—C4	−39.8 (2)
O1—C2—C3—O2	0.3 (3)	O1—C4—C5—O2	59.5 (3)

S2—C2—C3—O2	179.05 (15)