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5,6-Dihydrothieno[2,3-*d*][1,3]dithiol-2-one

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The title compound, $C_5H_4OS_3$, contains discrete molecules, which are linked into sheets by a combination of short S···S contacts [S···S = 3.512 (1) and 3.586 (1) Å] and C—H···O hydrogen bonds.

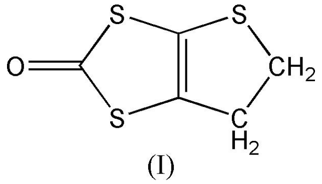
Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.039
wR factor = 0.099
Data-to-parameter ratio = 17.4

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

Comment

The title compound, (I), has been used successfully as a precursor in the synthesis of the π electronic donor BET-TTF [bis(ethylenethio)tetrathiafulvalene; Pérez-Benítez *et al.*, 1999]. This organic TTF-type donor has been used as a precursor of new molecular materials with interesting conducting properties (Rovira, 1999). More recently, this ketone has also been used as a precursor in the synthesis of new transition metal bisdithiothiophene compounds, either by direct hydrolytic cleavage with potassium methoxide in CH_3OH solution, to form the 2,3-dihydro-5,6-thiophenedithiolate ligand, or by being aromatized with DDQ (2,3-dichloro-5,6-dicyanobenzoquinone), to give 5,6-thieno[2,3-*d*]-1,3-dithiol-2-one. The latter is used as a precursor in the syntheses of complexes with the bis-2,3-thiophenedithiolate (tpdt) ligand. The Au(α-tpdt)$_2$ species, which uses this ketone as a precursor, is one of the first materials, based on a neutral species, exhibiting metallic type behaviour (Belo, Alves, Lopes *et al.*, 2001; Belo, Alves, Rabaça *et al.*, 2001). As already seen in the precursor ketone, the S atoms present in the molecule, especially the thiophene S atom, are able to establish contacts with neighbouring species. This situation gives rise to extra intermolecular S···S contacts, which are very important in the control of the crystal structure and the resulting electronic properties in molecular materials. The crystal structure and supramolecular arrangement of 5,6-dihydrothieno[2,3-*d*]-1,3-dithiol-2-one, (I), are reported here.



(I)

The molecular structure of (I) is illustrated in Fig. 1. Selected bond distances and angles are given in Table 1. The molecule is almost planar; however, atoms S3 and C4 of the thiophene ring deviate by 0.179 (3) and 0.153 (4) Å, respectively, from the least-squares plane through the other non-H atoms in the molecule.

In the crystal structure of (I), the hydrogen bonds and S···S short contacts play an important role in controlling the supramolecular assembly of the molecules. Details are given in

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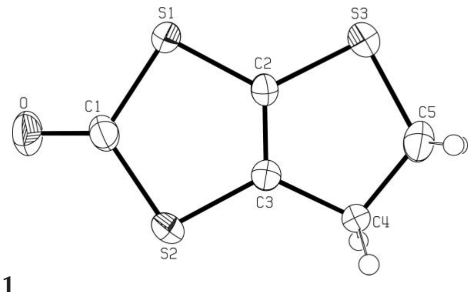

Figure 1
The molecular structure of (I), showing the atom-labelling scheme and displacement ellipsoids at the 50% probability level.

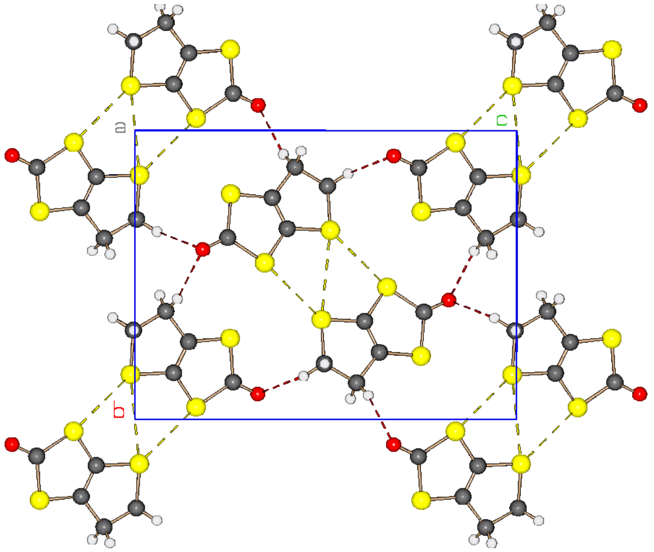

Figure 2
A packing diagram of (I), showing the intermolecular contacts within a layer. Dashed lines indicate the S···S short contacts and the C—H···O hydrogen bonds between the dimers of molecules (see Tables 1 and 2 for details).

Tables 1 and 2, and Fig. 2. The short contacts between neighbouring molecules [S1···S3^{i} = 3.512 (1) Å and S3···S3^{i} = 3.586 (1) Å] create centrosymmetric dimers. These are interconnected by hydrogen bonds in the *bc* plane, forming a layer-like structure.

Experimental

The title compound, (I), was synthesized according to literature methods (Pérez-Benítez *et al.*, 1999). Suitable colourless needle-shaped crystals were obtained by slow evaporation and cooling of hexane solutions.

Crystal data

$C_5H_4OS_3$
$M_r = 176.26$
Monoclinic, $P2_1/c$
$a = 4.0743$ (7) Å
$b = 11.154$ (2) Å
$c = 14.778$ (4) Å
$\beta = 94.651$ (17)°
$V = 669.4$ (2) Å^3
$Z = 4$

$D_x = 1.749$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 8\text{–}12°$
$\mu = 1.01$ mm^{-1}
$T = 294$ (2) K
Needle, colourless
0.38 × 0.16 × 0.08 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.825$, $T_{max} = 0.949$
1476 measured reflections
1423 independent reflections
1132 reflections with $I > 2\sigma(I)$

$R_{int} = 0.020$
$\theta_{max} = 27.0°$
$h = -5 \rightarrow 5$
$k = 0 \rightarrow 14$
$l = 0 \rightarrow 18$
5 standard reflections every 200 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.099$
$S = 1.09$
1423 reflections
82 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0384P)^2 + 1.0429P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.46$ e Å^{-3}
$\Delta\rho_{min} = -0.35$ e Å^{-3}

Table 1
Selected geometric parameters (Å).

C1—O	1.201 (4)	S3—C2	1.740 (3)
C1—S2	1.774 (3)	S3—C5	1.804 (4)
C1—S1	1.781 (3)	C2—C3	1.337 (4)
S1—C2	1.732 (3)	C3—C4	1.559 (4)
S2—C3	1.737 (3)	C4—C5	1.543 (4)
S1···S3^{i}	3.586 (1)	S3···S3^{ii}	3.512 (1)

Symmetry codes: (i) $-x + 1, -y + 1, -z + 1$.

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4A···O^{ii}	0.97	2.58	3.517 (4)	162
C5—H5A···O^{iii}	0.97	2.70	3.578 (5)	150

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

All of the H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.97 Å, and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1994); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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