Synthesis and crystal structure of 2-(1,3-dithietan-2-ylidene)cyclohexane-1,3-dione

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The title compound, C_8H_8O_2S_2, contains a cyclohexane-1,3-dione ring, which has a twist-boat conformation. The C_2S_2 ring is close to planar (r.m.s. deviation = 0.023 Å) and the dihedral angle between the mean planes of the cyclohexane and 1,3-dithietane rings is 9.1 (3)°. Short intramolecular S⋅⋅⋅O contacts occur [2.719 (5) and 2.740 (5) Å]. In the crystal, the molecules are linked by weak C–H⋅⋅⋅S hydrogen bonds and short [3.165 (5) Å] S⋅⋅⋅O contacts, forming (010) layers. The prevalence of these interactions is illustrated by an analysis of the three-dimensional Hirshfeld surface and by two-dimensional fingerprint plots.

1. Chemical context

Ketene dithioacetals are useful intermediates in organic synthesis and have been used for the preparation of heterocyclic compounds (Kolb, 1990; Ila et al., 2001). The synthesis of trifluoromethyl ketene dithioacetals has applications in the field of pharmaceuticals and agrochemicals (Gouault-Bironneau et al., 2012; Timoshenko & Portella, 2009). The functionalization of ketene dithioacetals provides more powerful tools for the development of new intermediates (Wang et al., 2011; Gao et al., 2010; Hu et al., 2012). The direct formation of a C–C bond has been carried out by reacting a cyano ketene dithioacetal and Morita–Baylis–Hillman (MBH) alcohols resulting from the reaction of acrylonitrile and aryl aldehydes. This reaction led to the corresponding 1,4-pentadiene derivatives (Zhao et al., 2007). Fiala et al. (2007) have studied the inhibitive action of some synthetic ketene dithioacetal derivatives towards the corrosion of copper in aerated nitric acid solutions. They concluded that these compounds are good inhibitors of copper corrosion in this medium. In the present study, we report the synthesis, crystal structure and Hirshfeld surface analysis of the new title 1,3-dithian-2-ylidene derivative, C_8H_8O_2S_2, (I).

2. Structural commentary

In the molecular structure of (I), the cyclohexane and dithietane rings are linked by a C–C bond of 1.364 (8) Å (Fig. 1). The cyclohexane-1,3-dione ring adopts a twist-boat
conformation, as seen in related compounds (Kuppan Chandrakala et al., 2016; Liu et al., 2011). Atom C5 is displaced by 0.627 (8) Å with respect to the C2/C3/C4/C6/C7 mean plane, similar to the value observed for 2-[chloro(4-methoxyphenyl)methyl]-2-(4-methoxyphenyl)-5,5-dimethylcyclohexane-1,3-dione (Saloua Chelli et al., 2016). The largest endocyclic angle in the cyclohexane ring [C7—C2—C3 = 123.2 (6)°] is located opposite the dithietane ring and the largest exo-cyclic angle (C6—C7—O2) is 122.3 (5)°. A difference of 1.3° is observed between the angles located on either side of the C1C2 double bond. In the C2S2 ring, the C1—S1 and C1—S2 bond lengths are indistinguishable at 1.716 (6) Å whereas the C8—S1 and C8—S2 bond lengths differ slightly [1.819 (7) and 1.801 (7) Å, respectively].

The molecule has local Cs symmetry with a non-crystallographic mirror plane passing through atoms C8, C1, C2 and C5. The dihedral angle between the cyclohexane (all atoms) and dithietane rings is 9.1 (3)° and short intramolecular S1···O2 [2.719 (5) Å] and S2···O1 [2.740 (5) Å] contacts are observed (Fig. 1).

3. Supramolecular features

In the crystal, the molecules stack head-to-tail along the b-axis direction. The molecules are linked by C5—H5A···S2 hydrogen bonds (Table 1) and short [3.165 (5) Å] a van der Waals separation of 3.32 Å S2···O2n [symmetry code: (ii) $\frac{1}{2} - x, y, \frac{1}{2} - z$] contacts, forming (010) layers (Fig. 2).

4. Hirshfeld surface analysis

The nature of the intermolecular interactions in (I) has been computed by CrystalExplorer17.5 (Turner et al., 2017), using Hirshfeld surface (HS) analysis (Spackman & Jayatilaka, 2009) and two-dimensional fingerprint plots (McKinnon et al., 2007). The $d_{	ext{norm}}$ plot (Fig. 3) shows red spots corresponding to the C5—H5A···S2 hydrogen bond and short S2···O2 contact. A list of the relative percentage contributions of the close contacts to the HS of (I) are given in Table 2 and the overall two-dimensional fingerprint plot is shown in Fig. 4a. A contribution of 30.7% was found for the H···O/O···H interactions, representing the largest contribution; these contacts are represented by the spikes in the top left ($d_e > d_i, H···O, 14.3\%$) and bottom right ($d_e < d_i, O···H, 16.5\%$) of Fig. 4b. Interactions of the type H···H appear in the middle of the scattered points in the fingerprint plots with a pair of spikes at $d_e + d_i = 2.5 \text{ Å}$ and comprise 25.9% of the entire surface (Fig. 4c); the van der Waals radius for this interaction is 2.4 Å, which means it is a weak interaction. The S···H/H···S contacts (Fig. 4d), which account for 23.8% of the Hirshfeld surface, are displayed on the fingerprint plot as a pair of long

**Table 1**

Hydrogen-bond geometry (Å, °).

| D—H···A  | D—H | H···A | D···A | D—H···A |
|----------|------|-------|-------|----------|
| C5—H5A···S2i | 0.97 | 2.87  | 3.844 (8) | 178 |

Symmetry code: (i) $-x, -y + 2, z - \frac{1}{2}$

**Figure 1**

The molecular structure of (I) with displacement ellipsoids drawn at the 50% probability level. The short intramolecular S···O contacts are shown as dashed lines.

**Figure 2**

Structure of (I) viewed along the [010] direction, showing the infinite layers propagating parallel to the $ac$ plane. The C—H···S and short S···O contacts are shown as blue dashed lines.

**Figure 3**

Hirshfeld surface for (I) scaled from $-0.16$ (red) a.u. to $1.09$ (blue) a.u.
spikes at \(d_e + d_i = 2.7 \text{ Å}\). This distance differs by 0.3 Å from the sum of the van der Waals radii, which means it is the strongest interaction present. The \(\text{S} \cdots \text{C} \cdots \text{S}\) (4.0%, Fig. 4f) and \(\text{S} \cdots \text{O} \cdots \text{O} \cdots \text{S}\) (3.3%, Fig. 4g) contacts are seen as pairs of spikes at \(d_e + d_i = 3.2\) and 3.05 Å, respectively. These distances are shorter than the sums of the van der Waals radii of 3.5 and 3.32 Å, respectively. The \(\text{C} \cdots \text{O} \cdots \text{C}\) contacts make a contribution of 0.7% to the Hirshfeld surface (Fig. 4h), their interatomic distances \(d_e + d_i = 3.3 \text{ Å}\) being larger than the sum of the van der Waals radius (3.22 Å), so this interaction is very weak in this structure. The fingerprint plot corresponding to \(\text{C} \cdots \text{H} \cdots \text{C}\) contacts (Fig. 4e) shows a fin-like distribution of points with the edges at \(d_e + d_i = 2.8 \text{ Å}\).

5. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.43, last update March 2022; Groom et al., 2016) for the 1,3-dithietane fragment yielded three relevant hits. These are dispiro[1,3-dithietane-2,2’:4,2”-diadamantane] (CSD refcode AFECAP; Linden et al., 2002), trans-2,4-bis(isopropyl)-2,4-bis[(2-methyl-1-thioxo)propylsulfanyl]-1,3-dithietane (HUZHOZ; Mahjoub et al., 2003) and 2-(nitromethylene)-1,3-dithietane (WOCQEK; Shanmuga Sundara Raj et al., 2000): in these compounds the dithietane ring is planar. In (I), the angles \(\text{C}1 \cdots \text{S}1 \cdots \text{C}8\) and \(\text{S}1 \cdots \text{C}8 \cdots \text{S}2\) are 82.7 (3) and 93.6 (3)°, respectively, similar to the values observed for the aforementioned compounds, viz. 85.76 and 94.24°, 85.40 and 94.60°, 82.8 and 94.00° for AFECAP, HUZHOZ and WOCQEK, respectively. A search for the cyclohexane-1,3-dione fragment revealed over 30 hits. The most relevant structures are 2-(phenylaminomethylidene)cyclohexane-1,3-dione (ISUQAO; Kettmann et al., 2004), (E)-5,5-dimethyl-2-[3-(4- nitrophenyl)allylidene]cyclohexane-1,3-dione (VUGVUQ; Jae Kyun Lee et al., 2015), 2-[chloro(4-methoxyphenyl)methyl]-2-(4-methoxyphenyl)-5,5-dimethylcyclohexane-1,3-dione (TACZIJ; Saloua Chelli et al., 2016) and 2-[(1S*,2'S*)]-2-[(E)-2,4-dihydroxybenzylidene]amino)cyclohexyl)isoindoline-1,3-dione (EVABIN; Liu et al., 2011). The cyclohexane ring adopts a chair conformation in all five of these compounds, as in the title compound.

6. Synthesis and crystallization

Potassium carbonate (0.3 mol, 42 g) in DMF (50 ml) was well stirred at room temperature. To this mixture, cyclohexane-1,3-dione (0.1 mol) was added and the resultant solution stirred at room temperature for 20 min. Carbon disulfide (0.15 mol, 9.0 ml) was then added in one lot. The reaction mixture was stirred and kept for 10 min at room temperature. Diodomethane (0.12 mol) was added dropwise over 20 min and the reaction mixture stirred for 7 h at room temperature. Ice–water (500 ml) was added to the reaction mass, the solid was filtered and washed with water, dried and recrystallized from ethanol solution to give (I) in the form of colourless plates. Yield 81%; m.p. 487 K; UV (H2O) \(\lambda_{\text{max}}\), 335 nm (\(e\) 18760); IR (KBr, cm\(^{-1}\)) 1640 (C=O), \(^{1}H\text{NMR}\) (CDCl\(_3\)) \(\delta\) (ppm): 4.35 (s, 2H, CH\(_2\)-S), 2.52 (t, \(J = 6.5 \text{ Hz}\), 4H, CH\(_2\)−CH\(_2\)-CH\(_2\)-CH\(_2\)), 1.97 (q, \(J = 6.5 \text{ Hz}\), 2H, CH\(_2\)-CH\(_2\)-CH\(_2\)-CH\(_2\)), \(^{13}C\text{NMR}\) (CDCl\(_3\)) \(\delta\) (ppm): 197.28 (CO), 189.73 (C=O), 187.60; \(^{1}H\text{NMR}\) (CDCl\(_3\)) \(\delta\) (ppm): 128.5 (CH\(_2\)-S), 85.40 and 94.60°.

7. Refinement

Crystal data, data collection and structure refinement details for the title compound are summarized in Table 3. H atoms were positioned geometrically with C−H = 0.97 Å and refined as riding with \(U_{\text{iso}}(\text{H}) = 1.2U_{eq}\)(C).

Acknowledgements

We thank the Diffractometry Center of the University of Rennes 1 for collecting the X-ray diffraction data.

Table 2

| Contact type | Percentage contribution |
|-------------|------------------------|
| O · · · H/O/O/O | 30.7 |
| H · · · H | 25.9 |
| S · · · H/S · · · S | 23.8 |
| C · · · H/H · · · C | 11.6 |
| S · · · C/S · · · C | 4.0 |
| S · · · O/O · · · O | 3.3 |
| C · · · O/O · · · C | 0.7 |

Figure 4

Two-dimensional fingerprint plots for (I): (a) overall, and delineated into contributions from different contacts: (b) H · · · O/O · · · H, (c) H · · · H, (d) H · · · S/S · · · H, (e) C · · · H/H · · · C, (f) C · · · S/S · · · C, (g) O · · · S/S · · · O and (h) C · · · O/O · · · C.
Table 3

Experimental details.

| Crystal data | Chemical formula | C8H8O2S2 |
|--------------|------------------|----------|
| M            |                  | 200.28   |
| Crystal system, space group | Orthorhombic, Pca2, |
| Temperature (K) | 296 |
| a, b, c (Å) | 10.7521 (14), 5.5245 (9), 14.480 (2) |
| V (Å³) | 860.1 (2) |
| Z | 4 |
| Radiation type | Mo Kα |
| μ (mm⁻¹) | 0.57 |
| Crystal size (mm) | 0.13 × 0.06 × 0.01 |

Data collection

| Diffractometer | Bruker APEXII |
|----------------|---------------|
| Tmin, Tmax | 0.960, 0.994 |
| No. of measured, independent and observed | 3646, 1881, 1230 |
| Rint | 0.044 |
| Δρmax, Δρmin (e Å⁻³) | 0.34, -0.30 |
| Absolute structure | Flack x determined using 396 quotients [(I(Γ)−(I(Γ))]/[(I(Γ)+(I(Γ))] |
| Absolute structure parameter | 0.04 (8) |

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Synthesis and crystal structure of 2-(1,3-dithietan-2-ylidene)cyclohexane-1,3-dione

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Computing details

Data collection: SAINT (Bruker, 2014); cell refinement: APEX2 (Bruker, 2014); data reduction: SAINT (Bruker, 2014); program(s) used to solve structure: SHELXT (Sheldrick, 2015b); program(s) used to refine structure: SHELXL2014/7 (Sheldrick, 2015a); molecular graphics: ORTEP-3 for Windows (Farrugia, 2012); software used to prepare material for publication: WinGX publication routines (Farrugia, 2012).

2-(1,3-Dithietan-2-ylidene)cyclohexane-1,3-dione

Crystal data

C₈H₈O₂S₂

Mr = 200.28

Orthorhombic, Pca₂₁

Hall symbol: P 2c -2ac

a = 10.7521 (14) Å

b = 5.5245 (9) Å

c = 14.480 (2) Å

V = 860.1 (2) Å³

Z = 4

F(000) = 416

Dₐ = 1.547 Mg m⁻³

Mo Ka radiation, λ = 0.71073 Å

Cell parameters from 772 reflections

θ = 3.7–23.4°

µ = 0.57 mm⁻¹

T = 296 K

Plate, colorless

0.13 × 0.06 × 0.01 mm

Data collection

Bruker APEXII

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

CCD rotation images, thick slices scans

Absorption correction: multi-scan

(SADABS; Krause et al., 2015)

Tₘᵋᵣᵣᵦ = 0.960, Tₘᵋₓᵦ = 0.994

3646 measured reflections

1881 independent reflections

1230 reflections with I > 2σ(I)

Rₘᵋᵣᵦ = 0.044

θ max = 27.5°, θ min = 2.8°

hk0, l = −18→18

Refinement

Refinement on F²

Least-squares matrix: full

R[F² > 2σ(F²)] = 0.051

wR(F²) = 0.094

S = 1.00

1881 reflections

109 parameters

1 restraint

0 constraints

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
$w = 1/[\sigma^2(F_o^2) + (0.0343P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta \rho_{\text{max}} = 0.34 \text{ e Å}^{-3}$

$\Delta \rho_{\text{min}} = -0.29 \text{ e Å}^{-3}$

**Special details**

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

**Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)**

|      | x       | y       | z       | $U_{iso}^*/U_{eq}$ |
|------|---------|---------|---------|------------------|
| S1   | 0.27332 (13) | 0.3620 (3) | 0.53923 (11) | 0.0371 (5) |
| S2   | 0.16018 (17) | 0.5982 (3) | 0.67352 (10) | 0.0430 (5) |
| O1   | 0.0128 (4) | 0.9617 (7) | 0.6058 (4) | 0.0457 (16) |
| O2   | 0.2139 (4) | 0.5407 (8) | 0.3699 (3) | 0.0520 (19) |
| C1   | 0.1711 (5) | 0.5968 (10) | 0.5553 (4) | 0.0273 (19) |
| C2   | 0.1164 (5) | 0.7427 (12) | 0.4911 (4) | 0.0260 (17) |
| C3   | 0.0319 (5) | 0.9299 (10) | 0.5239 (5) | 0.034 (2) |
| C4   | -0.0307 (6) | 1.0849 (12) | 0.4519 (5) | 0.045 (2) |
| C5   | -0.0404 (7) | 0.9587 (14) | 0.3579 (6) | 0.060 (3) |
| C6   | 0.0814 (7) | 0.8675 (13) | 0.3269 (4) | 0.058 (3) |
| C7   | 0.1416 (6) | 0.7004 (12) | 0.3941 (4) | 0.034 (2) |
| C8   | 0.2644 (7) | 0.3440 (12) | 0.6645 (5) | 0.050 (3) |
| H4A  | -0.11352 | 1.12679 | 0.47307 | 0.0542* |
| H4B  | 0.01593 | 1.23405 | 0.44470 | 0.0542* |
| H5A  | -0.07254 | 1.07203 | 0.31262 | 0.0722* |
| H5B  | -0.09848 | 0.82483 | 0.36241 | 0.0722* |
| H6A  | 0.07063 | 0.78338 | 0.26865 | 0.0697* |
| H6B  | 0.13618 | 1.00398 | 0.31609 | 0.0697* |
| H8A  | 0.34374 | 0.37194 | 0.69448 | 0.0598* |
| H8B  | 0.22773 | 0.19389 | 0.68617 | 0.0598* |

**Atomic displacement parameters (Å²)**

|      | $U_{11}$  | $U_{22}$  | $U_{33}$  | $U_{12}$  | $U_{13}$  | $U_{23}$  |
|------|-----------|-----------|-----------|-----------|-----------|-----------|
| S1   | 0.0410 (8) | 0.0394 (9) | 0.0308 (8) | 0.0115 (8) | 0.0003 (9) | 0.0007 (9) |
| S2   | 0.0561 (11) | 0.0509 (10) | 0.0219 (7) | 0.0117 (9) | 0.0005 (8) | -0.0001 (10) |
| O1   | 0.052 (3) | 0.045 (3) | 0.040 (2) | 0.011 (2) | 0.006 (3) | -0.010 (3) |
| O2   | 0.070 (4) | 0.058 (3) | 0.028 (3) | 0.020 (3) | 0.008 (3) | -0.001 (2) |
| C1   | 0.026 (3) | 0.028 (4) | 0.028 (3) | -0.006 (2) | 0.002 (3) | 0.000 (3) |
| C2   | 0.027 (3) | 0.026 (3) | 0.025 (3) | 0.002 (3) | -0.001 (3) | 0.000 (3) |
| C3   | 0.026 (3) | 0.032 (4) | 0.044 (4) | -0.003 (3) | 0.001 (3) | 0.004 (3) |
| C4   | 0.039 (4) | 0.038 (4) | 0.059 (4) | 0.003 (3) | -0.007 (4) | 0.009 (4) |
| C5   | 0.064 (6) | 0.069 (6) | 0.048 (4) | 0.013 (4) | -0.014 (4) | 0.016 (5) |
| C6   | 0.061 (5) | 0.072 (5) | 0.041 (4) | 0.020 (4) | 0.010 (4) | 0.024 (4) |
### Geometric parameters (Å, °)

| Bond/Distance                      | Value                         |
|------------------------------------|-------------------------------|
| S1—C1                              | 1.716 (6)                     |
| S1—C8                              | 1.819 (7)                     |
| S2—C1                              | 1.716 (6)                     |
| S2—C8                              | 1.801 (7)                     |
| O1—C3                              | 1.216 (9)                     |
| O2—C7                              | 1.227 (8)                     |
| C1—C2                              | 1.364 (8)                     |
| C2—C3                              | 1.508 (9)                     |
| C1—S1—C8                          | 82.7 (3)                      |
| C1—S2—C8                          | 83.2 (3)                      |
| S1—C1—S2                          | 100.5 (3)                     |
| S1—C1—C2                          | 129.1 (5)                     |
| S2—C1—C2                          | 130.4 (5)                     |
| C1—C2—C3                          | 117.8 (5)                     |
| C1—C2—C7                          | 119.0 (6)                     |
| C3—C2—C7                          | 123.2 (6)                     |
| C1—C3—C2                          | 121.7 (6)                     |
| O1—C3—C2                          | 121.1 (5)                     |
| C2—C3—C4                          | 117.2 (6)                     |
| C3—C4—C5                          | 112.7 (6)                     |
| C4—C5—C6                          | 111.5 (6)                     |
| C5—C6—C7                          | 113.5 (6)                     |
| O2—C7—C6                          | 120.7 (6)                     |
| O2—C7—C6                          | 122.3 (5)                     |
| O1—C8—S2                          | 93.6 (3)                      |
| C3—C4—H4A                         | 109.00                        |
| C1—S1—C8—S2                       | 1.5 (3)                       |
| C8—S1—C1—C2                       | −179.6 (6)                    |
| C1—S1—C8—S2                       | −1.4 (3)                      |
| C8—S2—C1—C2                       | −1.5 (3)                      |
| C8—S2—C1—C2                       | 179.6 (6)                     |
| C1—S2—C8—S1                       | 1.4 (3)                       |
| C2—S1—C1—C2                       | −1.6 (9)                      |
| C1—S1—C2—C3                       | 179.8 (4)                     |
| C2—C1—C2—C3                       | 1.6 (9)                       |
| S1—C1—C2—C3                       | −179.8 (6)                    |

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C7—C2—C3—C4  -0.2 (8)  C5—C6—C7—O2  151.8 (6)

Hydrogen-bond geometry (Å, °)

| D—H···A   | D—H | H···A | D···A | D—H···A |
|-----------|------|-------|-------|---------|
| C5—H5A···S2i | 0.97 | 2.87  | 3.844 (8) | 178 |

Symmetry code: (i) −x, −y+2, z−1/2.