



Synthesis, molecular structure and Hirshfeld surface analysis of (4-methoxyphenyl)[2-(methylsulfanyl)-thiophen-3-yl]methanone

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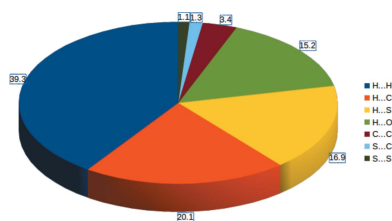
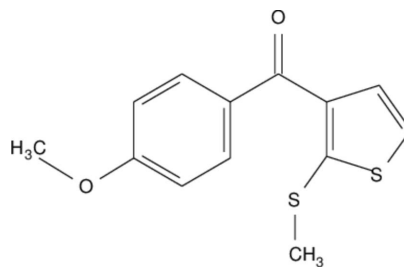
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The title compound, C₁₃H₁₂O₂S₂, crystallizes in the triclinic space group $P\bar{1}$. The molecular structure is substantially twisted, with a dihedral angle of 43.70 (2)° between the 2-(methylsulfanyl)thiophene and 4-methoxyphenyl rings. In the crystal, molecules are linked through C—H···O interactions and form a bifurcated layer stacking along the *b*-axis direction and enclosing $R_2^2(10)$ ring motifs. The phenyl rings are involved in π – π interactions with a centroid–centroid separation of 3.760 (2) Å. The Hirshfeld surfaces were studied and the contributions of the various intermolecular interactions were quantified.

1. Chemical context

Thiophenes are five-membered sulfur-containing heterocyclic compounds with important applications in areas such as agrochemistry, pharmaceuticals, molecular electronics, liquid crystalline materials and corrosion inhibition. Thiophenes are also important building blocks in organic synthesis. Their aromatic character gives enough stabilization to allow the manipulation of various substituents (Mishra *et al.*, 2011). α -Oxoketene thioacetals are powerful building blocks for the synthesis of numerous heterocyclic scaffolds, where the carbonyl carbon generally provides hard centers and the carbon-bearing methylsulfanyl group acts as a soft electrophilic center (Junjappa *et al.*, 1990). This synthetic building block was used for the synthesis of (4-methoxyphenyl) [2-(methylsulfanyl)thiophen-3-yl]methanone (Pradeepa Kumara *et al.*, 2016).



2. Structural commentary

In the title compound, the 2-(methylsulfanyl)thiophene and 4-methoxyphenyl aromatic rings are connected *via* a C(=O)—C methanone bridge (Fig. 1). The compound is

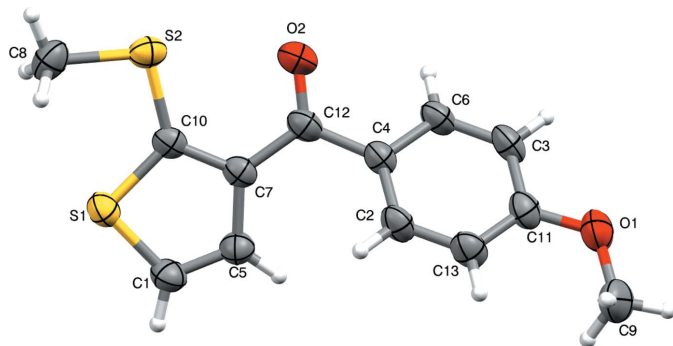


Figure 1
Molecular structure of the title compound, showing the atom-numbering scheme and displacement ellipsoids drawn at the 50% probability level.

substantially twisted about the methanone bridge as indicated by the dihedral angle of $43.70(2)^\circ$ between the thiophene (S1/C1/C5/C7/C10) and phenyl (C2–C6/C11/C13) rings. The methyl group at S2 is in a +syn-periplanar conformation, as indicated by the C8–S2–C10–S1 torsion angle of $6.09(16)^\circ$. However, in the related compound (4-fluorophenyl)[2-(methylsulfonyl)thiophen-3-yl]methanone (Nagaraju *et al.*, 2018), this group is in a -syn-periplanar conformation with a torsion angle of $-1.7(2)^\circ$. Atom C12 adopts a nearly trigonal geometry, as indicated by the bond angles C7–C12–O2 = $119.5(2)^\circ$, O2–C12–C4 = $119.2(2)^\circ$ and C4–C12–C7 = $121.3(2)^\circ$. The methoxy group attached at C11 is in a -anti-periplanar conformation [C3–C11–O1–C9 = $-176.9(2)^\circ$]. The bond lengths and angles are normal (Sreenatha *et al.*, 2017; Rajni Swamy *et al.*, 2014; Gopinath *et al.*, 2016).

3. Supramolecular features

The crystal structure features intermolecular hydrogen-bonding interactions of the type C9–H9A...O2 (Fig. 2, Table 1) and displays a bifurcated layer stacking along the *b*-axis direction through C6–H6...O2 interactions, which link inversion-related molecules into dimers enclosing an $R_2^2(10)$ ring motif. π - π stacking interactions are also observed between the phenyl rings (centroid Cg) of adjacent molecules [Cg...Cg(2 - *x*, -*y*, 1 - *z*) = $3.760(2)$ Å]. The packing of the title compound is illustrated in Fig. 3.

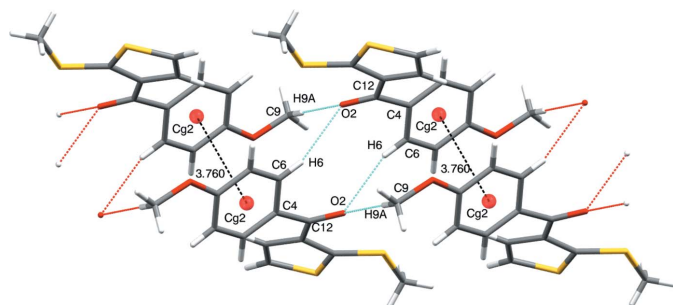


Figure 2
The $R_2^2(10)$ ring motif formed *via* intermolecular C6–H6...O2 hydrogen bonds (Table 1). The π - π interactions are also shown.

Table 1
Hydrogen-bond geometry (Å, °).

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
C6–H6...O2 ⁱ	0.93	2.48	3.374 (4)	161
C9–H9A...O2 ⁱⁱ	0.96	2.45	3.400 (4)	172

Symmetry codes: (i) $-x + 2, -y + 1, -z + 1$; (ii) $x, y - 1, z$.

4. Hirshfeld surfaces and 2D fingerprint plots

Hirshfeld surface (HS) analysis and the associated fingerprint plots (FP) (*CrystalExplorer*; Spackman & Jayatilaka, 2009) are useful tools for visualizing the types of intermolecular interactions present in a crystal structure and quantify their percentage contributions to the crystal packing. The 3D HS mapped over d_{norm} between -0.2106 a.u. (blue) and 1.2279 a.u. (red) is shown in Fig. 4. The area and volume of the HS are 287.29 Å² and 305.24 Å³, respectively. The deep-red spots on the d_{norm} surface are due to the presence of intermolecular C–H...O interactions (Sreenatha *et al.*, 2018). The 2D FP analysis (Fig. 5) shows that the H...H contacts make the highest contribution (39.3%) followed by the H...C/C...H contacts (20.1%), which are seen as a pair of blunt spikes in the region 1.2 Å < ($d_i + d_e$) < 1.75 Å. The H...S/S...H contacts make a contribution of 16.9% and appear as butterfly wings in the region 1.2 Å < ($d_i + d_e$) < 1.9 Å. The pair of sharp spikes is observed in the region 1.2 Å < ($d_i + d_e$) < 1.32 Å is due to the presence of H...O/O...H contacts (15.6% contribution). The C...C contacts (3.4% contribution) are visible as wings in almost the same region, 1.7 Å < ($d_i + d_e$) < 1.72 Å. The relative contributions of all the contacts to the Hirshfeld surface are depicted in Fig. 6.

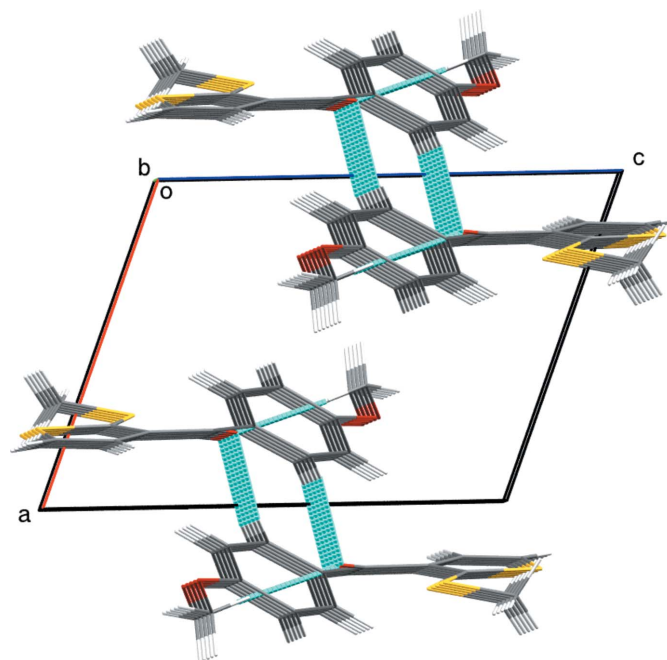


Figure 3
Packing for of the title compound viewed along the *b* axis.

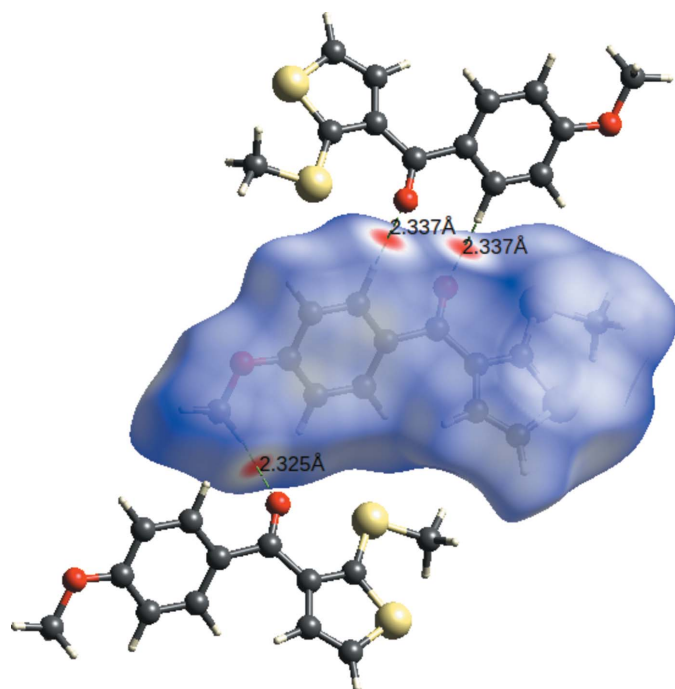


Figure 4
Hirshfeld surface for the title compound mapped over d_{norm} in the range -0.2106 to 1.2279 a.u. highlighting the C–H...O intermolecular interactions.

5. Database survey

A search for thiophene derivatives was carried out in the Cambridge Structural Database (CSD, Version 5.39, update of February 2018; Groom *et al.*, 2016). The most relevant compounds are 5-[bis(4-ethoxyphenyl)amino]thiophene-2-carbaldehyde (HOJCIU; Tan *et al.*, 2014) and 2-[4-(benzyloxy)phenyl]-5-(3,4-dimethoxyphenyl)-3,4-dimethylthiophene (ACETEI; Shi *et al.*, 2004), which are both non-planar. In ethyl 4-acetyl-5-anilino-3-methylthiophene-2-carboxylate (AFIGIH; Mabkhot *et al.*, 2013), the thiophene and phenyl rings make a dihedral angle of 36.81 (10°).

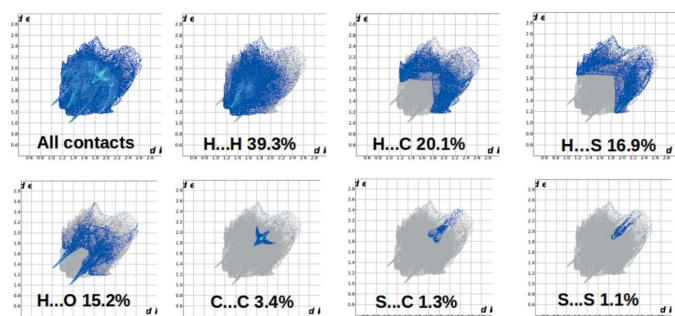


Figure 5
The full two-dimensional fingerprint plots for the title compound, showing (a) all interactions, and delineated into (b) H...H, (c) H...C/C...H, (d) H...S/S...H, (e) H...O/O...H, (f) C...C, (g) S...C/C...S and (h) S...S interactions. The d_i and d_e values are the closest internal and external distances (in Å) from given points on the Hirshfeld surface.

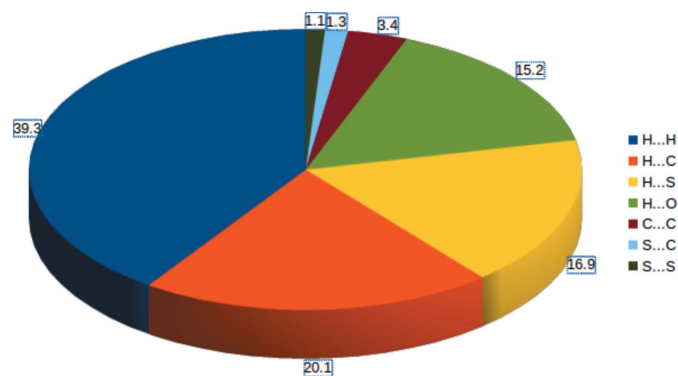


Figure 6
The relative contributions (%) to the Hirshfeld surface for the various contacts.

6. Synthesis and crystallization

To α -oxoketene dithioacetal (0.1 mol) and 1,4-dithiane-2,5-diol (0.05 mol) in dry ethanol (10 mL), anhydrous potassium carbonate (0.12 mol) was added. The reaction mixture was refluxed on a water bath for 30 minutes (the condenser being protected by a calcium chloride guard tube). After completion of the reaction (monitored by TLC), the catalyst was filtered off and washed with fresh ethanol. The combined ethanol solution was removed on a rotary evaporator to obtain a viscous liquid. The crude product was purified by column chromatography using silica gel with 5% ethyl acetate and petroleum ether to yield the title compound as a yellow solid product, which was recrystallized from dichloromethane solution. M.p. 489–493 K. IR (KBr) $\nu_{\text{max}} = 3449, 3079, 2923,$

Table 2
Experimental details.

Crystal data	
Chemical formula	$\text{C}_{13}\text{H}_{12}\text{O}_2\text{S}_2$
M_r	264.35
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	293
a, b, c (Å)	7.806 (4), 8.263 (3), 10.414 (6)
α, β, γ ($^\circ$)	97.260 (11), 109.65 (2), 93.79 (2)
V (Å ³)	623.3 (5)
Z	2
Radiation type	Mo $K\alpha$
μ (mm ⁻¹)	0.41
Crystal size (mm)	0.30 × 0.26 × 0.20
Data collection	
Diffractometer	Bruker APEX
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	2924, 2165, 1899
R_{int}	0.109
$(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹)	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.046, 0.128, 1.09
No. of reflections	2165
No. of parameters	157
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³)	0.37, -0.33

Computer programs: APEX2 (Bruker, 2006), SAINT (Bruker, 2006), SHELXS97 (Sheldrick, 2008), SHELXL2018 (Sheldrick, 2015), PLATON (Spek, 2009) and Mercury (Macrae *et al.*, 2008), PLATON (Spek, 2009).

2841, 1772, 1600, 1493, 1253, 1167, 1015, 842, 694, 550 cm^{-1} . ^1H NMR (300 MHz, CDCl_3): 7.79–7.77 (*m*, 2 H), 7.27–7.25 (*m*, 1H), 7.16–7.14 (*m*, 1H), 6.9–6.93 (*m*, 2H), 3.86 (*s*, 3H), 2.58 (*s*, 3H) ppm. ^{13}C NMR (75 MHz, CDCl_3): 188.86, 162.73, 151.33, 135.36, 131.60, 131.47, 130.24, 130.59, 122.02, 113.44, 55.37, 18.06. HRMS (ESI): calculated for $\text{C}_{13}\text{H}_{12}\text{O}_2\text{S}_2$ [$M + \text{H}$] $^+$ 265.0312; found 265.0407.

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All hydrogen atoms were placed at calculated positions and refined using a riding model with $\text{C}–\text{H} = 0.93 \text{ \AA}$ and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for aromatic ring atoms and with $\text{C}–\text{H} = 0.96 \text{ \AA}$ with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl groups.

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Acta Cryst. (2018). E74, 1800-1803 [https://doi.org/10.1107/S2056989018016043]

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Computing details

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT* (Bruker, 2006); data reduction: *SAINT* (Bruker, 2006); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2018* (Sheldrick, 2015); molecular graphics: *PLATON* (Spek, 2009) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *PLATON* (Spek, 2009).

(4-Methoxyphenyl)[2-(methylsulfanyl)thiophen-3-yl]methanone

Crystal data

$C_{13}H_{12}O_2S_2$

$M_r = 264.35$

Triclinic, $P\bar{1}$

$a = 7.806$ (4) Å

$b = 8.263$ (3) Å

$c = 10.414$ (6) Å

$\alpha = 97.260$ (11)°

$\beta = 109.65$ (2)°

$\gamma = 93.79$ (2)°

$V = 623.3$ (5) Å³

$Z = 2$

$F(000) = 276$

$D_x = 1.409$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2924 reflections

$\theta = 3.5$ – 25.0 °

$\mu = 0.41$ mm⁻¹

$T = 293$ K

Block, colourless

$0.30 \times 0.26 \times 0.20$ mm

Data collection

Bruker APEX
diffractometer

Radiation source: graphite

Detector resolution: 0.894 pixels mm⁻¹

SAINT (Bruker, 2006) [not correct; type of scans needed]

2924 measured reflections

2165 independent reflections

1899 reflections with $I > 2\sigma(I)$

$R_{int} = 0.109$

$\theta_{max} = 25.0$ °, $\theta_{min} = 3.5$ °

$h = -9 \rightarrow 9$

$k = -9 \rightarrow 9$

$l = -12 \rightarrow 11$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.046$

$wR(F^2) = 0.128$

$S = 1.09$

2165 reflections

157 parameters

0 restraints

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.072P)^2 + 0.1135P]$

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

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

$\Delta\rho_{max} = 0.37$ e Å⁻³

$\Delta\rho_{min} = -0.33$ e Å⁻³

Extinction correction: SHELXL2018
 (Sheldrick, 2015),
 $F_c^* = kF_c [1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.060 (18)

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.77364 (8)	0.32143 (7)	−0.09953 (6)	0.0412 (3)
S2	0.73606 (8)	0.60555 (7)	0.10012 (6)	0.0422 (3)
O1	0.7606 (3)	−0.1498 (2)	0.62854 (18)	0.0582 (5)
O2	0.7950 (3)	0.4642 (2)	0.33504 (18)	0.0601 (5)
C1	0.8084 (3)	0.1302 (3)	−0.0527 (3)	0.0450 (6)
H1	0.820956	0.039871	−0.110524	0.054*
C2	0.6715 (3)	0.0359 (3)	0.3139 (2)	0.0419 (6)
H2	0.607492	0.011357	0.219380	0.050*
C3	0.8621 (3)	0.1072 (3)	0.5930 (2)	0.0437 (6)
H3	0.927331	0.131023	0.687398	0.052*
C4	0.7766 (3)	0.1880 (3)	0.3679 (2)	0.0361 (5)
C5	0.8153 (3)	0.1275 (3)	0.0773 (2)	0.0402 (5)
H5	0.834586	0.034541	0.119774	0.048*
C6	0.8701 (3)	0.2215 (3)	0.5107 (2)	0.0407 (5)
H6	0.938609	0.323029	0.550038	0.049*
C7	0.7900 (3)	0.2813 (3)	0.1446 (2)	0.0352 (5)
C8	0.6913 (4)	0.6721 (3)	−0.0642 (3)	0.0525 (6)
H8A	0.594501	0.598579	−0.133310	0.079*
H8B	0.800026	0.672480	−0.087893	0.079*
H8C	0.655487	0.781032	−0.059371	0.079*
C9	0.6614 (5)	−0.3123 (4)	0.5746 (3)	0.0726 (9)
H9A	0.704580	−0.365458	0.505805	0.109*
H9B	0.533040	−0.303439	0.533903	0.109*
H9C	0.681017	−0.375822	0.648323	0.109*
C10	0.7680 (3)	0.4010 (3)	0.0595 (2)	0.0341 (5)
C11	0.7571 (3)	−0.0450 (3)	0.5371 (2)	0.0426 (5)
C12	0.7881 (3)	0.3201 (3)	0.2846 (2)	0.0397 (5)
C13	0.6597 (3)	−0.0792 (3)	0.3964 (2)	0.0459 (6)
H13	0.586967	−0.179094	0.358019	0.055*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0427 (4)	0.0471 (4)	0.0361 (4)	0.0025 (3)	0.0176 (3)	0.0050 (3)
S2	0.0402 (4)	0.0346 (4)	0.0488 (4)	−0.0026 (2)	0.0148 (3)	0.0010 (2)

O1	0.0729 (13)	0.0652 (12)	0.0423 (10)	0.0013 (10)	0.0272 (9)	0.0124 (8)
O2	0.0870 (15)	0.0460 (10)	0.0429 (10)	0.0040 (9)	0.0214 (9)	-0.0041 (8)
C1	0.0512 (14)	0.0421 (13)	0.0453 (13)	0.0072 (10)	0.0235 (11)	0.0008 (10)
C2	0.0356 (12)	0.0535 (14)	0.0302 (10)	-0.0047 (10)	0.0073 (9)	-0.0002 (9)
C3	0.0400 (12)	0.0615 (15)	0.0258 (10)	0.0005 (11)	0.0106 (9)	-0.0013 (10)
C4	0.0325 (11)	0.0448 (12)	0.0296 (10)	-0.0001 (9)	0.0115 (8)	0.0009 (9)
C5	0.0409 (12)	0.0393 (12)	0.0421 (12)	0.0079 (9)	0.0157 (10)	0.0067 (9)
C6	0.0375 (12)	0.0484 (13)	0.0311 (11)	-0.0036 (10)	0.0110 (9)	-0.0048 (9)
C7	0.0290 (10)	0.0398 (11)	0.0335 (11)	0.0008 (8)	0.0089 (8)	0.0007 (9)
C8	0.0504 (15)	0.0444 (13)	0.0623 (16)	-0.0009 (11)	0.0170 (12)	0.0173 (12)
C9	0.108 (3)	0.0627 (18)	0.0627 (18)	-0.0042 (17)	0.0516 (18)	0.0122 (14)
C10	0.0243 (10)	0.0389 (11)	0.0356 (11)	-0.0033 (8)	0.0096 (8)	-0.0005 (9)
C11	0.0407 (13)	0.0548 (14)	0.0372 (11)	0.0036 (11)	0.0204 (9)	0.0068 (10)
C12	0.0351 (12)	0.0443 (12)	0.0336 (11)	-0.0001 (9)	0.0080 (9)	-0.0026 (9)
C13	0.0409 (13)	0.0528 (14)	0.0387 (12)	-0.0097 (11)	0.0129 (10)	-0.0014 (10)

Geometric parameters (Å, °)

S1—C10	1.719 (2)	C4—C6	1.400 (3)
S1—C1	1.724 (3)	C4—C12	1.494 (3)
S2—C10	1.744 (2)	C5—C7	1.430 (3)
S2—C8	1.793 (3)	C5—H5	0.9300
O1—C11	1.360 (3)	C6—H6	0.9300
O1—C9	1.448 (4)	C7—C10	1.391 (3)
O2—C12	1.230 (3)	C7—C12	1.458 (3)
C1—C5	1.340 (4)	C8—H8A	0.9600
C1—H1	0.9300	C8—H8B	0.9600
C2—C13	1.379 (3)	C8—H8C	0.9600
C2—C4	1.394 (3)	C9—H9A	0.9600
C2—H2	0.9300	C9—H9B	0.9600
C3—C6	1.365 (3)	C9—H9C	0.9600
C3—C11	1.397 (4)	C11—C13	1.387 (3)
C3—H3	0.9300	C13—H13	0.9300
C10—S1—C1	92.19 (11)	S2—C8—H8A	109.5
C10—S2—C8	100.76 (12)	S2—C8—H8B	109.5
C11—O1—C9	117.8 (2)	H8A—C8—H8B	109.5
C5—C1—S1	111.72 (18)	S2—C8—H8C	109.5
C5—C1—H1	124.1	H8A—C8—H8C	109.5
S1—C1—H1	124.1	H8B—C8—H8C	109.5
C13—C2—C4	121.9 (2)	O1—C9—H9A	109.5
C13—C2—H2	119.0	O1—C9—H9B	109.5
C4—C2—H2	119.0	H9A—C9—H9B	109.5
C6—C3—C11	120.7 (2)	O1—C9—H9C	109.5
C6—C3—H3	119.6	H9A—C9—H9C	109.5
C11—C3—H3	119.6	H9B—C9—H9C	109.5
C2—C4—C6	117.6 (2)	C7—C10—S1	110.79 (16)
C2—C4—C12	124.3 (2)	C7—C10—S2	127.08 (17)

C6—C4—C12	118.1 (2)	S1—C10—S2	122.13 (14)
C1—C5—C7	113.6 (2)	O1—C11—C13	125.0 (2)
C1—C5—H5	123.2	O1—C11—C3	115.7 (2)
C7—C5—H5	123.2	C13—C11—C3	119.3 (2)
C3—C6—C4	121.0 (2)	O2—C12—C7	119.5 (2)
C3—C6—H6	119.5	O2—C12—C4	119.2 (2)
C4—C6—H6	119.5	C7—C12—C4	121.33 (19)
C10—C7—C5	111.7 (2)	C2—C13—C11	119.5 (2)
C10—C7—C12	120.6 (2)	C2—C13—H13	120.3
C5—C7—C12	127.7 (2)	C11—C13—H13	120.3
C10—S1—C1—C5	0.0 (2)	C8—S2—C10—S1	6.09 (16)
C13—C2—C4—C6	-0.5 (4)	C9—O1—C11—C13	2.7 (4)
C13—C2—C4—C12	-177.3 (2)	C9—O1—C11—C3	-176.9 (2)
S1—C1—C5—C7	0.8 (3)	C6—C3—C11—O1	179.4 (2)
C11—C3—C6—C4	-1.4 (4)	C6—C3—C11—C13	-0.2 (4)
C2—C4—C6—C3	1.7 (3)	C10—C7—C12—O2	-11.1 (3)
C12—C4—C6—C3	178.7 (2)	C5—C7—C12—O2	168.1 (2)
C1—C5—C7—C10	-1.4 (3)	C10—C7—C12—C4	168.49 (19)
C1—C5—C7—C12	179.4 (2)	C5—C7—C12—C4	-12.4 (4)
C5—C7—C10—S1	1.3 (2)	C2—C4—C12—O2	141.9 (3)
C12—C7—C10—S1	-179.42 (16)	C6—C4—C12—O2	-34.8 (3)
C5—C7—C10—S2	-179.26 (16)	C2—C4—C12—C7	-37.6 (3)
C12—C7—C10—S2	0.0 (3)	C6—C4—C12—C7	145.6 (2)
C1—S1—C10—C7	-0.78 (18)	C4—C2—C13—C11	-1.1 (4)
C1—S1—C10—S2	179.77 (14)	O1—C11—C13—C2	-178.2 (2)
C8—S2—C10—C7	-173.3 (2)	C3—C11—C13—C2	1.4 (4)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C6—H6...O2 ⁱ	0.93	2.48	3.374 (4)	161
C9—H9 <i>A</i> ...O2 ⁱⁱ	0.96	2.45	3.400 (4)	172

Symmetry codes: (i) $-x+2, -y+1, -z+1$; (ii) $x, y-1, z$.