(±)-*trans*-6,7-Dimethoxy-1-oxo-3-(2-thienyl)isochroman-4-carboxylic acid

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The title compound, $C_{16}H_{14}O_6S$, was synthesized by the reaction of 6,7-dimethoxyhomophthalic anhydride with thiophene-2-carbaldehyde in the presence of 4-(dimethylamino)pyridine (DMAP) as a basic catalyst. The thiophene ring of the title molecule is disordered over two sites with occupancies of 0.877 (3) and 0.123 (3). The disorder corresponds to an approximate 180° rotation of the thiophene ring with respect to the C—C bond linking it to the rest of the molecule. The six-membered ring of the 3,4-dihydro-isochromanone ring system is not planar [puckering parameters $Q_T = 0.571$ (2) Å, $\theta = 115.2$ (2)° and $\varphi = 99.1$ (2)°]. The benzene ring of the 3,4-dihydroisochromanone ring system makes dihedral angles of 75.0 (2) and 77.2 (5)° with the disordered thiophene rings. Intermolecular O—H···O and C—H···O hydrogen bonds, as well as C—H···π interactions, lead to the observed supramolecular structure.

Related literature

For details of the synthesis of the title compound, see: Bogdanov & Palamareva (2004). For the synthesis of new dihydroisocoumarins, see: Bogdanov *et al.* (2007*a,b*). For ring-puckering parameters, see: Cremer & Pople (1975).
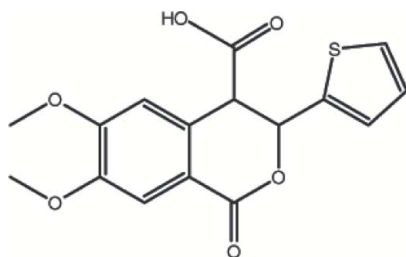
Experimental

Crystal data

$C_{16}H_{14}O_6S$	$\gamma = 72.958$ (6)°
$M_r = 334.34$	$V = 777.6$ (1) Å³
Triclinic, $P\bar{1}$	$Z = 2$
$a = 8.3369$ (6) Å	Mo $K\alpha$ radiation
$b = 8.4587$ (6) Å	$\mu = 0.24$ mm⁻¹
$c = 11.9143$ (9) Å	$T = 296$ K
$\alpha = 76.441$ (6)°	$0.53 \times 0.41 \times 0.21$ mm
$\beta = 81.127$ (6)°	

Data collection

Stoe IPDS II diffractometer	8329 measured reflections
Absorption correction: integration (X-RED32; Stoe & Cie, 2002)	3229 independent reflections
$T_{min} = 0.885$, $T_{max} = 0.952$	2663 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.030$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.113$	
$S = 1.07$	
3229 reflections	$\Delta\rho_{max} = 0.27$ e Å⁻³
219 parameters	$\Delta\rho_{min} = -0.31$ e Å⁻³
13 restraints	

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

D—H···A	D—H	H···A	D···A	D—H···A
O6—H6···O3[i]	0.86 (3)	1.84 (2)	2.658 (2)	159 (3)
C2—H2···O5[ii]	0.93	2.54	3.465 (2)	172
C10—H10···O6[iii]	0.98	2.54	3.475 (2)	159
C11—H11···Cg4[iv]	0.98	2.61	3.525 (2)	156

Symmetry codes: (i) $x, y+1, z$; (ii) $-x+1, -y+2, -z+1$; (iii) $-x, -y+2, -z+1$; (iv) $-x+1, -y+1, -z+1$. Cg4 is the centroid of the C1–C6 ring.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2117).

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supporting information

(±)-*trans*-6,7-Dimethoxy-1-oxo-3-(2-thienyl)isochroman-4-carboxylic acid

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S1. Comment

The title compound (I) was obtained as a part of a research project aimed at the synthesis of new dihydroisocoumarins with potential pharmacological activities (Bogdanov *et al.*, 2007*a*,*b*). (I) was synthesized by a one-pot reaction of 6,7-di-methoxyhomophthalic anhydride with thiophene-2-carboxaldehyde in the presence of DMAP as a basic catalyst (Bogdanov & Palamareva, 2004). The structure of (I) was determined by spectral methods (^{1}H NMR & IR) and microanalysis. In this paper, we report the X-ray crystallographic study of (I).

In the title molecule, (I), the thiophene ring is disordered over two sites and the major component of the disorder labelled with suffix A is shown in Fig. 1. The disorder corresponds to an approximate 180° rotation with respect to the C10—C13 bond. The six-membered ring (O4/C1/C6/C9–C11) of the 3,4-dihydroisochromanone ring system is not planar, showing the puckering parameters: $Q_T = 0.571$ (2) Å, $\theta = 115.2$ (2)° and $\varphi = 99.1$ (2)° (Cremer & Pople, 1975). The benzene ring (C1–C6) of the 3,4-dihydroisochromanone ring system encloses dihedral angles of 75.0 (2)° and 77.2 (5)° with the thiophene rings A(C13/C14A/C15/C16/S1A) and B (C13/C14B/C15/C16/S1B), respectively.

The crystal structure is realized by intermolecular O—H⋯O and C—H⋯O hydrogen bonds and C—H⋯π interactions (Table 1, Fig. 2).

S2. Experimental

Compound (I) was synthesized by the reaction of 6,7-dimethoxyhomophthalic anhydride (**1**) with thiophene-2-carbaldehyde (**2**) in the presence of DMAP as a basic catalyst (Bogdanov & Palamareva, 2004). To a mixture of **1** (0.33 g, 1.5 mmol) and **2** (0.15 ml, 1.65 mmol) in dry chloroform (5 ml) DMAP (0.18 g, 1.5 mmol) was added. The mixture was stirred at room temperature for 1 h. At the end of the reaction (monitored by TLC), the reaction mixture was extracted with 10% sodium hydrogen carbonate. The aqueous layer was further acidified (pH = 3) with 10% hydrochloric acid and extracted with ethyl acetate. The organic layer was dried (sodium sulfate), filtered and the solvent was then evaporated under reduced pressure giving (0.49 g, 98%) of a diastereomeric mixture of *cis-* and *trans*-(±)-6,7-dimethoxy-1-oxo-3-(thiophen-2-yl)isochroman-4-carboxylic acids, in a 30:70 ratio, favoring the *trans* diastereomer. Pure *trans*-diastereomer (I) was obtained by fractional crystallization of the residue from ethyl acetate. The product was characterized by ^{1}H NMR, IR spectra and elemental analysis. Single crystals were obtained by slow evaporation of a chloroform–ethyl acetate (3:1) solution of (I) at room temperature (m.p. 465–467 K). Analysis, calculated for $C_{16}H_{14}O_6S$ (334.34): C 57.48, H 4.22, O 28.71, S 9.59 (%); found: C 57.74, H 3.97, O 28.71, S 9.91 (%). IR (KBr) 1693 cm^{-1} (C═O), 1741 cm^{-1} (C═O). The ^{1}H NMR spectrum of (I) was obtained on a Bruker DRX-250 spectrometer at 250.13 MHz. Chemical shifts (δ) are expressed in parts per million (p.p.m.). ^{1}H NMR (250 MHz, DMSO-d$_6$) $\delta = 3.82$ (3H, s, –O—CH$_3$), 3.85 (3H, s, –O—CH$_3$), 4.43 (1H, d, $J = 3$ Hz, H-4), 6.29 (1H, d, $J = 3$ Hz, H-3), 6.96 (1H, dd, $J = 3.6$ and 5 Hz, Th—H), 7.08 (1H, s, Ph—H), 7.13 (1H, d, $J = 3.5$ Hz, Th—H), 7.37 (1H, s, Ph—H), 7.44 (1H, dd, $J = 1$ and 5 Hz, Th—H).

S3. Refinement

The H atom of the hydroxyl group was found from a difference Fourier map and refined freely [O6—H6 = 0.864 (3) Å]. H atoms bonded to C atoms were placed at calculated positions with the C—H distances in a range of 0.93–0.98 Å, and were included in the refinement in the riding-model approximation, with $U_{iso}(H) = 1.2$ or $1.5U_{eq}(C)$. The ratio of the refined site occupancies for the major and minor components of the disordered thiophene ring is 0.877 (3):0.123 (3). Similarity restraints were applied to the displacement parameters of the disordered atoms, and there were also geometrical restraints.
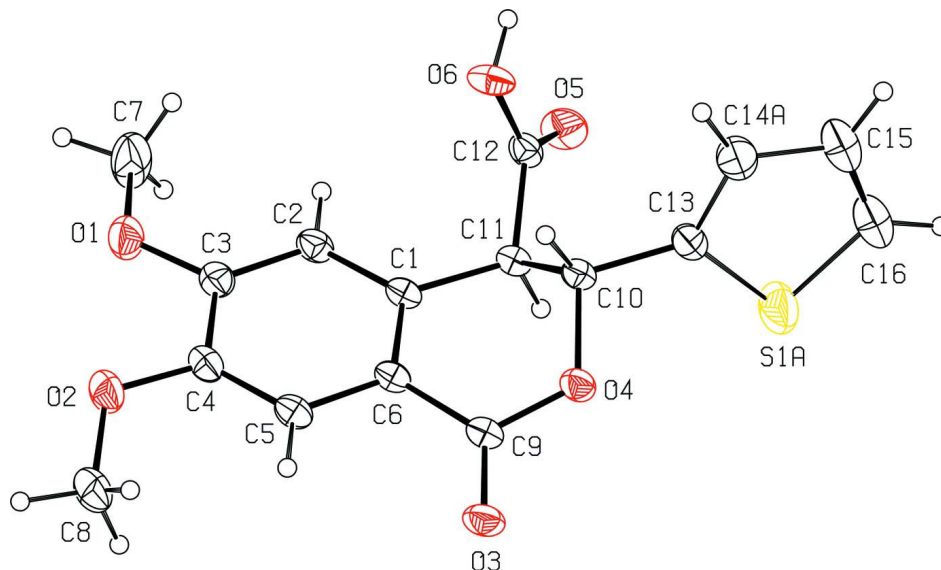
Figure 1

The title molecule of (I) with the atom-labelling scheme and displacement ellipsoids drawn at the 30% probability level. The minor component of the disorder has been omitted.
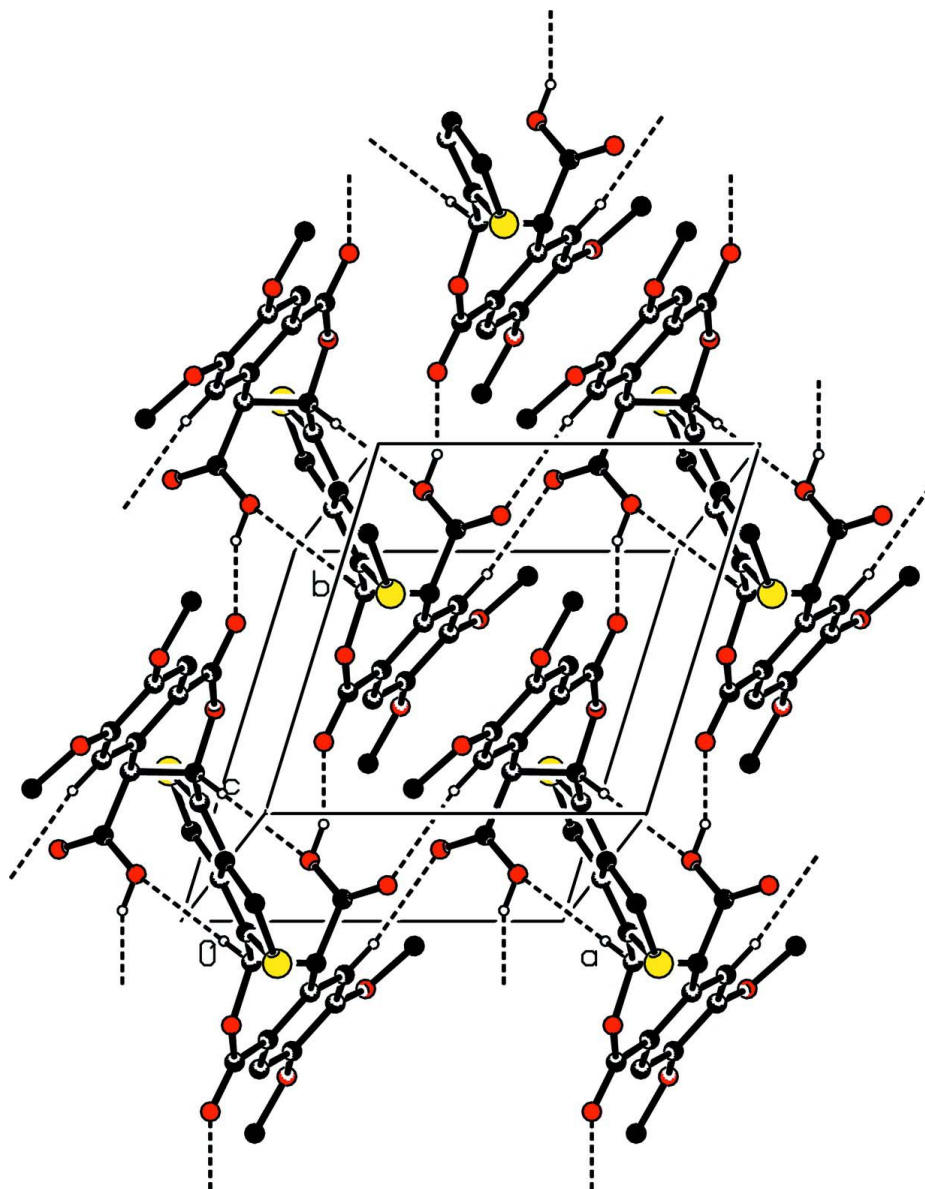
Figure 2

Perspective view of the intermolecular hdrogen bonding interactions in the structure of (I). H atoms not involved in hydrogen bonding and the minor component of the disordered thiophene ring have been omitted for clarity.

(±)-*trans*-6,7-Dimethoxy-1-oxo-3-(2-thienyl)isochroman-4-carboxylic acid

Crystal data

$C_{16}H_{14}O_6S$	$\gamma = 72.958\,(6)°$
$M_r = 334.34$	$V = 777.6\,(1)$ Å^3
Triclinic, $P\bar{1}$	$Z = 2$
Hall symbol: -P 1	$F(000) = 348$
$a = 8.3369\,(6)$ Å	$D_x = 1.428$ Mg m^{-3}
$b = 8.4587\,(6)$ Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$c = 11.9143\,(9)$ Å	Cell parameters from 16761 reflections
$\alpha = 76.441\,(6)°$	$\theta = 2.6–28.0°$
$\beta = 81.127\,(6)°$	$\mu = 0.24$ mm^{-1}

T = 296 K	$0.53 \times 0.41 \times 0.21$ mm
Prism, colourless	

Data collection

Stoe IPDS II diffractometer	T_{min} = 0.885, T_{max} = 0.952
	8329 measured reflections
Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus	3229 independent reflections
	2663 reflections with $I > 2\sigma(I)$
Plane graphite monochromator	R_{int} = 0.030
Detector resolution: 6.67 pixels mm⁻¹	θ_{max} = 26.5°, θ_{min} = 2.6°
ω scans	$h = -10 \rightarrow 10$
Absorption correction: integration	$k = -10 \rightarrow 10$
(*X-RED32*; Stoe & Cie, 2002)	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)]$ = 0.044	Hydrogen site location: inferred from neighbouring sites
$wR(F^2)$ = 0.113	
S = 1.07	H atoms treated by a mixture of independent and constrained refinement
3229 reflections	
219 parameters	$w = 1/[\sigma^2(F_o^2) + (0.0514P)^2 + 0.2422P]$
13 restraints	where $P = (F_o^2 + 2F_c^2)/3$
Primary atom site location: structure-invariant direct methods	$(\Delta/\sigma)_{max} < 0.001$
	$\Delta\rho_{max}$ = 0.27 e Å⁻³
	$\Delta\rho_{min}$ = −0.31 e Å⁻³

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R-factors wR and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -R-factor-obs *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 (Å²)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
S1A	0.17140 (13)	0.63559 (11)	0.85712 (7)	0.0759 (3)	0.877 (3)
O1	0.5188 (2)	0.77377 (19)	0.14713 (12)	0.0557 (5)	
O2	0.38518 (19)	0.54308 (18)	0.12791 (12)	0.0503 (5)	
O3	0.15691 (17)	0.32562 (15)	0.54690 (12)	0.0445 (4)	
O4	0.13106 (15)	0.53400 (15)	0.63275 (10)	0.0370 (4)	
O5	0.42866 (19)	0.91589 (18)	0.62175 (13)	0.0515 (5)	
O6	0.22136 (17)	1.01715 (16)	0.50460 (13)	0.0451 (4)	
C1	0.33543 (19)	0.67769 (19)	0.44799 (14)	0.0304 (4)	
C2	0.4244 (2)	0.7526 (2)	0.35183 (16)	0.0356 (5)	
C3	0.4377 (2)	0.7065 (2)	0.24602 (16)	0.0384 (5)	
C4	0.3637 (2)	0.5802 (2)	0.23513 (15)	0.0372 (5)	
C5	0.2797 (2)	0.5039 (2)	0.33076 (16)	0.0359 (5)	
C6	0.26476 (19)	0.55214 (19)	0.43731 (15)	0.0311 (5)	

C7	0.6107 (4)	0.8880 (4)	0.1547 (2)	0.0869 (13)	
C8	0.3476 (3)	0.3914 (3)	0.12059 (19)	0.0611 (8)	
C9	0.1816 (2)	0.4620 (2)	0.53946 (15)	0.0330 (5)	
C10	0.1347 (2)	0.7092 (2)	0.61942 (15)	0.0319 (5)	
C11	0.31183 (19)	0.72133 (19)	0.56665 (14)	0.0305 (4)	
C12	0.3300 (2)	0.8940 (2)	0.56779 (15)	0.0339 (5)	
C13	0.0822 (2)	0.7555 (2)	0.73510 (16)	0.0385 (5)	
C14A	−0.0344 (8)	0.8941 (8)	0.7626 (6)	0.0585 (17)	0.877 (3)
C15	−0.0506 (4)	0.9063 (4)	0.8781 (2)	0.0802 (8)	
C16	0.0534 (4)	0.7747 (4)	0.9384 (2)	0.0802 (8)	
S1B	−0.0621 (16)	0.9313 (15)	0.7433 (10)	0.058 (3)	0.123 (3)
C14B	0.144 (3)	0.692 (3)	0.8422 (15)	0.0802 (8)	0.123 (3)
H5	0.23240	0.41970	0.32480	0.0430*	
H6	0.227 (3)	1.112 (3)	0.516 (2)	0.064 (7)*	
H7A	0.69670	0.83190	0.20660	0.1050*	
H7B	0.53550	0.98290	0.18340	0.1050*	
H7C	0.66180	0.92640	0.07920	0.1050*	
H8A	0.23190	0.39800	0.14800	0.0730*	
H2	0.47510	0.83390	0.35870	0.0430*	
H8C	0.36690	0.37790	0.04140	0.0730*	
H10	0.05270	0.78130	0.56540	0.0380*	
H11	0.39450	0.63610	0.61480	0.0370*	
H14A	−0.09870	0.97510	0.70800	0.0700*	0.877 (3)
H15	−0.12440	0.99480	0.90910	0.0960*	
H16	0.06060	0.76040	1.01750	0.0960*	
H8B	0.41900	0.29650	0.16740	0.0730*	
H14B	0.23880	0.60080	0.85330	0.0960*	0.123 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1A	0.0947 (6)	0.0739 (6)	0.0417 (4)	0.0110 (4)	−0.0144 (3)	−0.0153 (3)
O1	0.0752 (10)	0.0589 (9)	0.0414 (8)	−0.0358 (8)	0.0138 (7)	−0.0161 (6)
O2	0.0680 (9)	0.0552 (8)	0.0366 (7)	−0.0240 (7)	−0.0013 (6)	−0.0192 (6)
O3	0.0519 (7)	0.0311 (6)	0.0557 (8)	−0.0171 (6)	0.0018 (6)	−0.0156 (6)
O4	0.0425 (6)	0.0332 (6)	0.0399 (7)	−0.0162 (5)	0.0033 (5)	−0.0132 (5)
O5	0.0596 (8)	0.0501 (8)	0.0586 (9)	−0.0260 (7)	−0.0186 (7)	−0.0145 (7)
O6	0.0488 (7)	0.0274 (6)	0.0646 (9)	−0.0079 (5)	−0.0164 (6)	−0.0154 (6)
C1	0.0262 (7)	0.0273 (7)	0.0391 (9)	−0.0044 (6)	−0.0033 (6)	−0.0128 (6)
C2	0.0346 (8)	0.0336 (8)	0.0430 (9)	−0.0126 (7)	0.0006 (7)	−0.0144 (7)
C3	0.0391 (9)	0.0362 (9)	0.0392 (9)	−0.0089 (7)	0.0020 (7)	−0.0112 (7)
C4	0.0379 (9)	0.0384 (9)	0.0370 (9)	−0.0062 (7)	−0.0049 (7)	−0.0152 (7)
C5	0.0342 (8)	0.0349 (8)	0.0437 (10)	−0.0099 (7)	−0.0054 (7)	−0.0157 (7)
C6	0.0264 (7)	0.0287 (8)	0.0395 (9)	−0.0046 (6)	−0.0035 (6)	−0.0128 (7)
C7	0.129 (3)	0.100 (2)	0.0588 (15)	−0.084 (2)	0.0344 (16)	−0.0281 (15)
C8	0.0914 (17)	0.0613 (13)	0.0447 (12)	−0.0308 (13)	−0.0033 (11)	−0.0266 (10)
C9	0.0298 (8)	0.0292 (8)	0.0422 (9)	−0.0064 (6)	−0.0041 (7)	−0.0131 (7)
C10	0.0324 (8)	0.0282 (8)	0.0373 (9)	−0.0086 (6)	−0.0015 (7)	−0.0113 (6)

C11	0.0296 (7)	0.0270 (7)	0.0365 (9)	−0.0060 (6)	−0.0041 (6)	−0.0108 (6)
C12	0.0349 (8)	0.0357 (8)	0.0364 (9)	−0.0137 (7)	0.0009 (7)	−0.0150 (7)
C13	0.0384 (9)	0.0389 (9)	0.0404 (10)	−0.0109 (7)	0.0015 (7)	−0.0148 (7)
C14A	0.066 (3)	0.053 (3)	0.047 (3)	0.001 (2)	−0.0090 (19)	−0.011 (2)
C15	0.1059 (16)	0.0819 (14)	0.0539 (11)	−0.0163 (11)	0.0061 (10)	−0.0355 (10)
C16	0.1059 (16)	0.0819 (14)	0.0539 (11)	−0.0163 (11)	0.0061 (10)	−0.0355 (10)
S1B	0.065 (4)	0.054 (5)	0.041 (4)	0.015 (3)	−0.008 (3)	−0.020 (4)
C14B	0.1059 (16)	0.0819 (14)	0.0539 (11)	−0.0163 (11)	0.0061 (10)	−0.0355 (10)

Geometric parameters (Å, º)

S1A—C13	1.702 (2)	C10—C13	1.487 (2)
S1A—C16	1.689 (3)	C10—C11	1.536 (2)
S1B—C15	1.584 (12)	C11—C12	1.515 (2)
S1B—C13	1.628 (13)	C13—C14B	1.379 (19)
O1—C7	1.423 (4)	C13—C14A	1.357 (7)
O1—C3	1.354 (2)	C14A—C15	1.387 (7)
O2—C8	1.431 (3)	C14B—C16	1.47 (2)
O2—C4	1.359 (2)	C15—C16	1.326 (4)
O3—C9	1.211 (2)	C2—H2	0.9300
O4—C9	1.344 (2)	C5—H5	0.9300
O4—C10	1.462 (2)	C7—H7A	0.9600
O5—C12	1.197 (2)	C7—H7B	0.9600
O6—C12	1.327 (2)	C7—H7C	0.9600
O6—H6	0.86 (3)	C8—H8A	0.9600
C1—C11	1.513 (2)	C8—H8B	0.9600
C1—C2	1.392 (2)	C8—H8C	0.9600
C1—C6	1.394 (2)	C10—H10	0.9800
C2—C3	1.384 (3)	C11—H11	0.9800
C3—C4	1.419 (2)	C14A—H14A	0.9300
C4—C5	1.371 (3)	C14B—H14B	0.9300
C5—C6	1.399 (2)	C15—H15	0.9300
C6—C9	1.469 (2)	C16—H16	0.9300
S1A···O4	3.0857 (15)	C3···H11^{viii}	3.0300
S1A···C8^i	3.589 (2)	C4···H11^{viii}	2.8300
S1B···O6^{ii}	3.308 (12)	C5···H8B	2.8100
S1B···O6	3.488 (13)	C5···H11^{viii}	2.7400
S1B···C12	3.571 (13)	C5···H8A	2.6600
S1B···C2^{ii}	3.530 (13)	C6···H10	2.7900
S1B···C3^{ii}	3.694 (13)	C6···H11^{viii}	2.8700
S1A···H8C^i	3.0000	C7···H2	2.5200
S1A···H11	3.1800	C8···H5	2.5100
O1···O2	2.581 (2)	C9···H11	2.9600
O2···O1	2.581 (2)	C9···H6^{iii}	2.95 (2)
O3···O6^{iii}	2.6575 (18)	C12···H2	2.6900
O3···C6^{iv}	3.350 (2)	H2···O6	2.8500
O3···O4^{iv}	3.2369 (19)	H2···C7	2.5200

O3···C9[iv]	3.051 (2)	H2···C12	2.6900
O4···O3[iv]	3.2369 (19)	H2···H7A	2.3800
O4···S1A	3.0857 (15)	H2···H7B	2.2400
O5···O6[v]	3.212 (2)	H2···O5[v]	2.5400
O5···O5[v]	3.138 (2)	H5···O3	2.6100
O5···C12[v]	3.244 (2)	H5···C8	2.5100
O5···C7[v]	3.382 (3)	H5···H8A	2.1600
O6···S1B[ii]	3.308 (12)	H5···H8B	2.4700
O6···C2	3.150 (2)	H5···O4[iv]	2.9200
O6···O3[vi]	2.6575 (18)	H6···O3[vi]	1.84 (2)
O6···S1B	3.488 (13)	H6···C9[vi]	2.95 (2)
O6···C13	3.385 (2)	H6···H10[ii]	2.5000
O6···O5[v]	3.212 (2)	H7A···C2	2.7800
O1···H8C[vii]	2.7500	H7A···H2	2.3800
O2···H8C[vii]	2.7700	H7B···C2	2.7100
O3···H6[iii]	1.84 (2)	H7B···H2	2.2400
O3···H10[iv]	2.8100	H7B···H8B[vi]	2.5200
O3···H5	2.6100	H7B···O5[v]	2.7400
O4···H5[iv]	2.9200	H7C···H15[x]	2.5600
O5···H2[v]	2.5400	H8A···C5	2.6600
O5···H7B[v]	2.7400	H8A···H5	2.1600
O6···H14A[ii]	2.8500	H8B···C5	2.8100
O6···H10	2.6700	H8B···H5	2.4700
O6···H2	2.8500	H8B···H7B[iii]	2.5200
O6···H10[ii]	2.5400	H8C···S1A[ix]	3.0000
C1···C1[viii]	3.545 (2)	H8C···O1[vii]	2.7500
C2···S1B[ii]	3.530 (13)	H8C···O2[vii]	2.7700
C2···C9[viii]	3.532 (2)	H10···O6	2.6700
C2···O6	3.150 (2)	H10···C6	2.7900
C3···S1B[ii]	3.694 (13)	H10···H14A	2.5700
C6···O3[iv]	3.350 (2)	H10···O3[iv]	2.8100
C7···O5[v]	3.382 (3)	H10···O6[ii]	2.5400
C8···S1A[ix]	3.589 (2)	H10···H6[ii]	2.5000
C9···C9[iv]	3.150 (2)	H11···S1A	3.1800
C9···C2[viii]	3.532 (2)	H11···C9	2.9600
C9···O3[iv]	3.051 (2)	H11···C1[viii]	3.1000
C12···O5[v]	3.244 (2)	H11···C3[viii]	3.0300
C12···S1B	3.571 (13)	H11···C4[viii]	2.8300
C12···C14A	3.527 (7)	H11···C5[viii]	2.7400
C13···O6	3.385 (2)	H11···C6[viii]	2.8700
C14A···C12	3.527 (7)	H14A···H10	2.5700
C1···H11[viii]	3.1000	H14A···O6[ii]	2.8500
C2···H7B	2.7100	H14A···C2[ii]	3.0600
C2···H14A[ii]	3.0600	H15···H7C[xi]	2.5600
C2···H7A	2.7800		
C13—S1A—C16	91.85 (12)	C13—C14A—C15	115.2 (5)
C13—S1B—C15	92.3 (7)	C13—C14B—C16	117.2 (17)

C3—O1—C7	117.71 (16)	C14A—C15—C16	110.8 (4)
C4—O2—C8	116.56 (15)	S1B—C15—C16	123.5 (5)
C9—O4—C10	117.75 (13)	S1A—C16—C15	113.17 (19)
C12—O6—H6	108.4 (17)	C14B—C16—C15	98.8 (9)
C6—C1—C11	116.64 (14)	C1—C2—H2	120.00
C2—C1—C6	119.26 (15)	C3—C2—H2	120.00
C2—C1—C11	124.09 (15)	C4—C5—H5	120.00
C1—C2—C3	120.20 (16)	C6—C5—H5	120.00
O1—C3—C2	124.71 (16)	O1—C7—H7A	110.00
C2—C3—C4	120.36 (16)	O1—C7—H7B	109.00
O1—C3—C4	114.93 (16)	O1—C7—H7C	109.00
C3—C4—C5	119.21 (16)	H7A—C7—H7B	109.00
O2—C4—C5	125.01 (16)	H7A—C7—H7C	109.00
O2—C4—C3	115.78 (16)	H7B—C7—H7C	109.00
C4—C5—C6	120.31 (16)	O2—C8—H8A	109.00
C5—C6—C9	118.97 (15)	O2—C8—H8B	109.00
C1—C6—C5	120.64 (16)	O2—C8—H8C	109.00
C1—C6—C9	120.30 (15)	H8A—C8—H8B	109.00
O4—C9—C6	118.04 (14)	H8A—C8—H8C	109.00
O3—C9—O4	117.07 (16)	H8B—C8—H8C	109.00
O3—C9—C6	124.89 (16)	O4—C10—H10	109.00
O4—C10—C13	107.21 (13)	C11—C10—H10	109.00
O4—C10—C11	107.57 (13)	C13—C10—H10	109.00
C11—C10—C13	115.54 (14)	C1—C11—H11	108.00
C1—C11—C10	106.68 (13)	C10—C11—H11	108.00
C10—C11—C12	109.46 (13)	C12—C11—H11	108.00
C1—C11—C12	115.26 (14)	C13—C14A—H14A	122.00
O6—C12—C11	112.16 (15)	C15—C14A—H14A	122.00
O5—C12—C11	123.52 (16)	C16—C14B—H14B	121.00
O5—C12—O6	124.30 (16)	C13—C14B—H14B	121.00
C10—C13—C14A	128.5 (3)	C16—C15—H15	125.00
S1A—C13—C10	122.57 (13)	S1B—C15—H15	112.00
S1A—C13—C14A	109.0 (3)	C14A—C15—H15	125.00
S1B—C13—C14B	107.5 (11)	C14B—C16—H16	138.00
S1B—C13—C10	118.1 (4)	S1A—C16—H16	123.00
C10—C13—C14B	133.8 (10)	C15—C16—H16	123.00
C16—S1A—C13—C14A	−0.8 (4)	O1—C3—C4—C5	179.60 (16)
C13—S1A—C16—C15	0.5 (3)	O2—C4—C5—C6	−179.91 (16)
C16—S1A—C13—C10	177.53 (18)	C3—C4—C5—C6	1.1 (3)
C7—O1—C3—C2	6.4 (3)	C4—C5—C6—C1	−0.3 (3)
C7—O1—C3—C4	−173.5 (2)	C4—C5—C6—C9	−176.89 (16)
C8—O2—C4—C3	165.72 (18)	C5—C6—C9—O4	−163.86 (15)
C8—O2—C4—C5	−13.3 (3)	C5—C6—C9—O3	17.3 (3)
C10—O4—C9—C6	10.7 (2)	C1—C6—C9—O4	19.5 (2)
C9—O4—C10—C13	−177.08 (14)	C1—C6—C9—O3	−159.38 (18)
C9—O4—C10—C11	−52.23 (19)	O4—C10—C11—C12	−171.59 (13)
C10—O4—C9—O3	−170.33 (15)	O4—C10—C13—S1A	50.92 (19)

C2—C1—C11—C10	144.44 (16)	C13—C10—C11—C1	−177.25 (14)
C2—C1—C6—C5	−1.3 (2)	O4—C10—C11—C1	63.07 (16)
C6—C1—C11—C12	−158.42 (15)	C11—C10—C13—C14A	109.1 (4)
C2—C1—C6—C9	175.27 (16)	C13—C10—C11—C12	−51.91 (19)
C11—C1—C2—C3	−179.11 (16)	O4—C10—C13—C14A	−131.1 (4)
C11—C1—C6—C5	179.76 (15)	C11—C10—C13—S1A	−68.96 (19)
C11—C1—C6—C9	−3.7 (2)	C1—C11—C12—O5	−123.00 (19)
C2—C1—C11—C12	22.7 (2)	C1—C11—C12—O6	58.6 (2)
C6—C1—C11—C10	−36.70 (19)	C10—C11—C12—O5	116.78 (19)
C6—C1—C2—C3	2.1 (3)	C10—C11—C12—O6	−61.66 (18)
C1—C2—C3—C4	−1.3 (3)	S1A—C13—C14A—C15	1.0 (6)
C1—C2—C3—O1	178.82 (17)	C10—C13—C14A—C15	−177.2 (3)
C2—C3—C4—C5	−0.3 (3)	C13—C14A—C15—C16	−0.7 (7)
C2—C3—C4—O2	−179.43 (16)	C14A—C15—C16—S1A	0.1 (5)
O1—C3—C4—O2	0.5 (2)		

Symmetry codes: (i) x, y, $z+1$; (ii) $-x$, $-y+2$, $-z+1$; (iii) x, $y-1$, z; (iv) $-x$, $-y+1$, $-z+1$; (v) $-x+1$, $-y+2$, $-z+1$; (vi) x, $y+1$, z; (vii) $-x+1$, $-y+1$, $-z$; (viii) $-x+1$, $-y+1$, $-z+1$; (ix) x, y, $z-1$; (x) $x+1$, y, $z-1$; (xi) $x-1$, y, $z+1$.

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
O6—H6···O3[vi]	0.86 (3)	1.84 (2)	2.658 (2)	159 (3)
C2—H2···O5[v]	0.93	2.54	3.465 (2)	172
C10—H10···O6[ii]	0.98	2.54	3.475 (2)	159
C11—H11···*Cg*4[viii]	0.98	2.61	3.525 (2)	156

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