(*E*)-1-(2-Thienyl)-3-(3,4,5-trimethoxy-phenyl)prop-2-en-1-one[1]

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The molecule of the title heteroaryl chalcone, $C_{16}H_{16}O_4S$, which consists of substituted thiophene and benzene rings bridged by the prop-2-en-1-one group, is slightly twisted. The dihedral angle between the thiophene and 3,4,5-trimethoxy-phenyl rings is 12.18 (4)°. The three methoxy groups have two different conformations; two methoxy groups are coplanar [C—O—C—C torsion angles = −1.38 (12) and 0.47 (12)°] whereas the third is (−)-synclinal with the benzene ring. In the crystal structure, adjacent molecules are linked in a face-to-side manner into chains along the *c* axis by weak C—H⋯O(enone) interactions. These chains are stacked along the *b* axis by weak C—H⋯O(methoxy) interactions.

Related literature

For bond-length data, see: Allen *et al.* (1987). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For related structures, see: Chantrapromma *et al.* (2009); Patil *et al.* (2006; 2007); Suwunwong *et al.* (2009*a*,*b*). For background to and applications of chalcones, see: Dimmock *et al.* (1999); Go *et al.* (2005); Jung *et al.* (2008); Ni *et al.* (2004); Patil *et al.* (2007); Patil & Dharmaprakash (2008). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer, (1986).
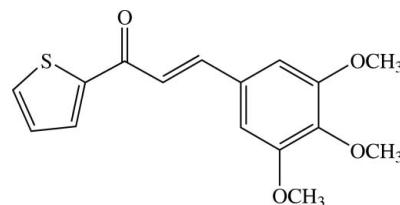
Experimental

Crystal data

$C_{16}H_{16}O_4S$	V = 1413.73 (7) Å^3
M_r = 304.36	Z = 4
Orthorhombic, $Pna2_1$	Mo $K\alpha$ radiation
a = 25.3323 (8) Å	μ = 0.24 mm^{-1}
b = 3.9816 (1) Å	T = 100 K
c = 14.0163 (4) Å	$0.58 \times 0.31 \times 0.21$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer	56940 measured reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	7416 independent reflections
	7177 reflections with $I > 2\sigma(I)$
T_{min} = 0.872, T_{max} = 0.951	R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033	H-atom parameters constrained
$wR(F^2)$ = 0.099	$\Delta\rho_{max}$ = 0.66 e Å^{-3}
S = 1.10	$\Delta\rho_{min}$ = −0.54 e Å^{-3}
7416 reflections	Absolute structure: Flack (1983), 3588 Friedel pairs
193 parameters	Flack parameter: 0.04 (4)
1 restraint	

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C1—H1A⋯O1^i	0.93	2.52	3.1827 (14)	129
C15—H15C⋯O3^{ii}	0.96	2.39	3.3340 (11)	169

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2627).

organic compounds

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supporting information

(*E*)-1-(2-Thienyl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one

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S1. Comment

Chalcone or 1,3-diaryl-2-propen-1-one, originally isolated from natural sources, and its derivatives are known to display a variety of biological activities, demonstrating analgesic, anti-inflammatory, antibacterial and antimycotic properties (Dimmock *et al.*, 1999; Go *et al.*, 2005; Ni *et al.*, 2004). Moreover synthetic chalcones have also been found to be non-linear optical (NLO) (Patil & Dharmaprakash, 2008) and electro-active fluorescent materials (Jung *et al.*, 2008). We have previously reported the synthesis and crystal structures of chalcone derivatives (Chantrapromma *et al.*, 2009; Suwunwong *et al.*, 2009*a*, *b*). Our research into the NLO and biological properties of chalcone derivatives led us to synthesize the title heteroaryl chalcone (I). (I) crystallizes in the non-centrosymmetric orthorhombic space group *Pna*2₁ and should therefore exhibit second-order nonlinear optical properties.

   The molecule of the title heteroaryl chalcone (Fig. 1) exists in an *E* configuration with respect to the C6═C7 double bond [1.3437 (11) Å] with a C5–C6–C7–C8 torsion angle 176.81 (8)°. The whole molecule is twisted as shown by the interplanar angle between thiophene and 3,4,5-trimethoxyphenyl rings being 12.18 (4)°. The propenone unit (C5—C7/O1) is also twisted with the O1–C5–C6–C7 torsion angle 10.94 (15)°. The three substituted methoxy groups of 3,4,5-trimethoxyphenyl unit have two different orientations: two methoxy groups (at the C10 and C12 positions) are co-planar with the phenyl ring with torsion angles C14–O2–C10–C9 = -1.38 (12)° and C16–O4–C12–C13 = 0.47 (12)° whereas the one at C11 is (-)-*syn*-clinally attached with the C15–O3–C11–C12 torsion angle -76.76 (10)°. In the structure, weak intramolecular C7—H7A⋯O1 and C15—H15B⋯O4 interactions generate S(5) and S(6) ring motifs, respectively (Bernstein *et al.*, 1995). The bond distances have normal values (Allen *et al.*, 1987) and bond lengths and angles are comparable with closely related structures (Chantrapromma *et al.*, 2009; Patil *et al.*, 2006; 2007; Suwunwong *et al.*, 2009*a*, *b*).

   In the crystal packing, the adjacent molecules are linked in a face-to-side manner into chains along the *c* axis through the enone unit by weak C1—H1A⋯O1 interactions (Fig. 2, Table 1). Weak C15—H15C⋯O3 interactions involving one of methoxy groups further stack these chains along the *b* axis (Fig. 3, Table 1).

S2. Experimental

The title compound was synthesized by the condensation of 3,4,5-trimethoxybenzaldehyde (0.40 g, 2 mmol) with 2-acetylthiophene (0.35 ml, 2 mmol) in ethanol (30 ml) in the presence of 30% NaOH (aq) (5 ml). After stirring for 3 h in ice bath at 278 K, the resulting pale yellow solid was collected by filtration, washed with distilled water, dried in air and purified by repeated recrystallization from acetone (72% yield). Pale yellow block-shaped single crystals of the title compound suitable for *x*-ray structure determination were recrystalized from acetone/ethanol (1:1 *v*/*v*) by the slow evaporation of the solvent at room temperature after several days, Mp. 420–421 K.

S3. Refinement

All H atoms were placed in calculated positions, with C—H = 0.93 Å, U_{iso} = 1.2U_{eq}(C) for aromatic and CH and C—H = 0.96 Å, U_{iso} = 1.5U_{eq}(C) for CH$_3$ atoms. A rotating group model was used for the methyl groups. The highest residual electron density peak is located at 0.22 Å from C3 and the deepest hole is located at 0.20 Å from S1.
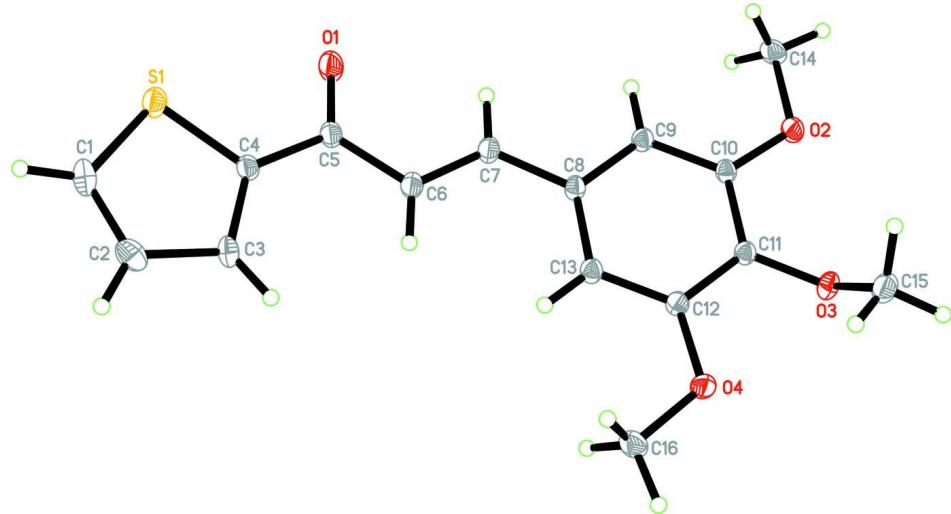
Figure 1

The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme.
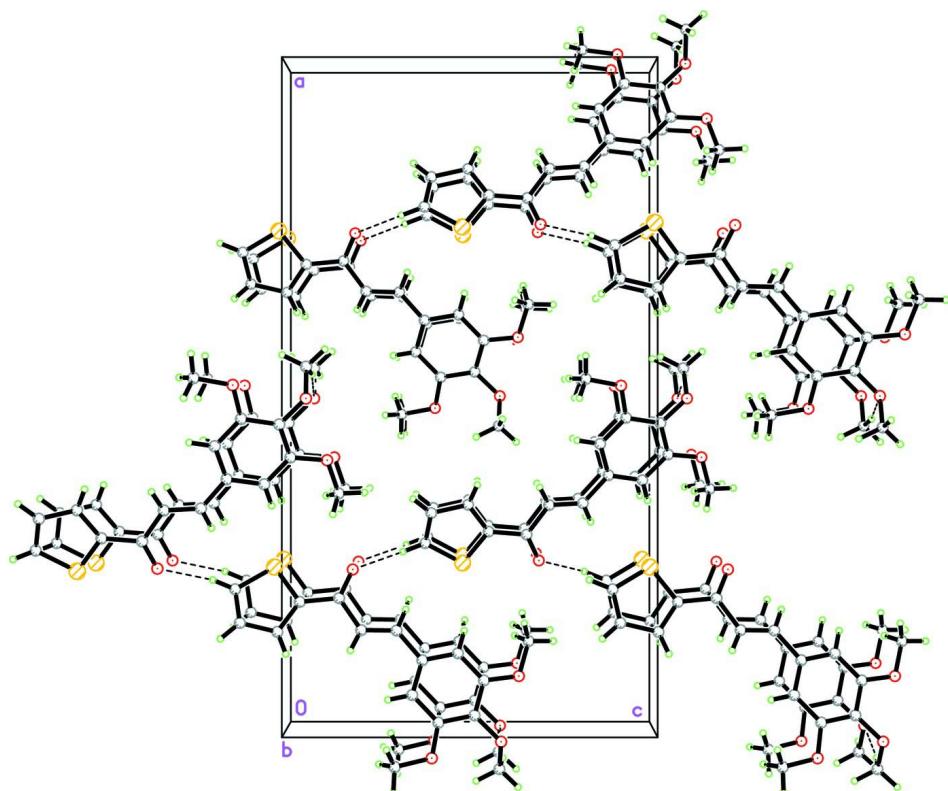
Figure 2

The crystal packing of the title compound viewed along the *b* axis, showing chains running along the *c* axis. Weak C—H···O interactions are shown as dashed lines.
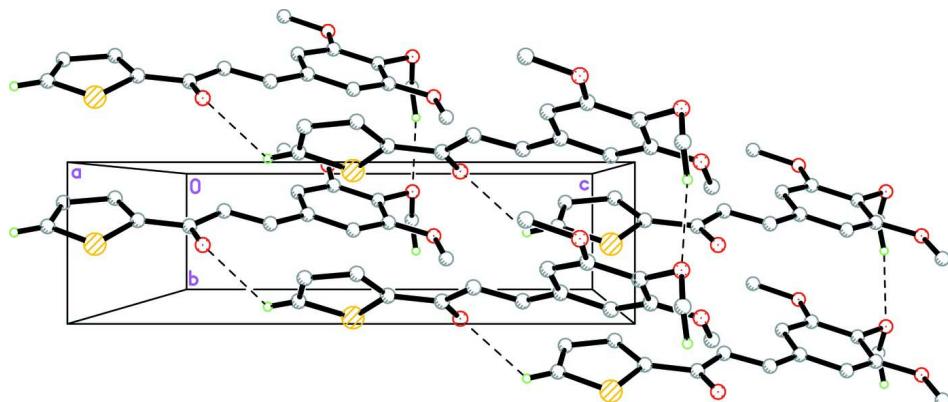
Figure 3

The crystal packing of the title compound viewed along the *a* axis, showing chains stacking along the *b* axis. Weak C—H···O interactions are shown as dashed lines and hydrogen atoms not involved in C—H···O interactions were omitted for clarity.

(*E*)-1-(2-Thienyl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one

Crystal data

$C_{16}H_{16}O_4S$	$D_x = 1.430$ Mg m^{-3}
$M_r = 304.36$	Melting point = 420–421 K
Orthorhombic, *Pna*2$_1$	Mo *K*α radiation, $\lambda = 0.71073$ Å
Hall symbol: P 2c -2n	Cell parameters from 7416 reflections
$a = 25.3323$ (8) Å	$\theta = 2.2$–37.5°
$b = 3.9816$ (1) Å	$\mu = 0.24$ mm^{-1}
$c = 14.0163$ (4) Å	$T = 100$ K
$V = 1413.73$ (7) Å^3	Block, pale yellow
$Z = 4$	0.58 × 0.31 × 0.21 mm
$F(000) = 640$	

Data collection

Bruker APEXII CCD area-detector diffractometer	56940 measured reflections
Radiation source: sealed tube	7416 independent reflections
Graphite monochromator	7177 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.028$
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	$\theta_{max} = 37.5°$, $\theta_{min} = 2.2°$
	$h = -43 \rightarrow 43$
$T_{min} = 0.872$, $T_{max} = 0.951$	$k = -6 \rightarrow 6$
	$l = -23 \rightarrow 24$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.033$	$w = 1/[\sigma^2(F_o^2) + (0.0672P)^2 + 0.1429P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.099$	
$S = 1.10$	$(\Delta/\sigma)_{max} = 0.001$
7416 reflections	$\Delta\rho_{max} = 0.66$ e Å^{-3}
193 parameters	$\Delta\rho_{min} = -0.54$ e Å^{-3}
1 restraint	
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 3588 Friedel pairs
Secondary atom site location: difference Fourier map	Absolute structure parameter: 0.04 (4)

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 120.0 (1) K.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'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	0.249987 (8)	0.46794 (6)	0.47989 (2)	0.01892 (5)

O1	0.25370 (3)	0.4617 (3)	0.68801 (7)	0.02618 (18)
O2	0.40965 (3)	0.51095 (18)	1.13056 (5)	0.01662 (11)
O3	0.49824 (2)	0.83176 (19)	1.08244 (5)	0.01615 (10)
O4	0.51691 (3)	1.02802 (19)	0.90201 (5)	0.01753 (11)
C1	0.28105 (4)	0.5872 (3)	0.37853 (6)	0.02121 (16)
H1A	0.2679	0.5465	0.3177	0.025*
C2	0.32771 (4)	0.7503 (3)	0.39595 (7)	0.02174 (16)
H2A	0.3493	0.8331	0.3477	0.026*
C3	0.34021 (3)	0.7815 (2)	0.49530 (5)	0.01517 (12)
H3A	0.3702	0.8844	0.5200	0.018*
C4	0.29890 (3)	0.6283 (2)	0.55061 (6)	0.01440 (12)
C5	0.29401 (3)	0.5880 (2)	0.65390 (6)	0.01617 (13)
C6	0.33914 (3)	0.6919 (2)	0.71401 (6)	0.01613 (13)
H6A	0.3662	0.8202	0.6878	0.019*
C7	0.34122 (3)	0.6018 (2)	0.80632 (6)	0.01549 (13)
H7A	0.3122	0.4836	0.8294	0.019*
C8	0.38341 (3)	0.6666 (2)	0.87483 (5)	0.01329 (11)
C9	0.37539 (3)	0.5559 (2)	0.96864 (6)	0.01372 (11)
H9A	0.3441	0.4479	0.9849	0.016*
C10	0.41415 (3)	0.6073 (2)	1.03751 (5)	0.01261 (11)
C11	0.46146 (3)	0.7683 (2)	1.01284 (5)	0.01296 (11)
C12	0.46951 (3)	0.8767 (2)	0.91840 (5)	0.01333 (12)
C13	0.43078 (3)	0.8266 (2)	0.84955 (6)	0.01399 (12)
H13A	0.4362	0.8985	0.7872	0.017*
C14	0.36101 (4)	0.3538 (2)	1.15750 (6)	0.01816 (14)
H14A	0.3619	0.3001	1.2243	0.027*
H14B	0.3563	0.1516	1.1212	0.027*
H14C	0.3322	0.5043	1.1452	0.027*
C15	0.54355 (3)	0.6178 (2)	1.07845 (8)	0.02027 (15)
H15A	0.5681	0.6817	1.1274	0.030*
H15B	0.5601	0.6391	1.0172	0.030*
H15C	0.5329	0.3890	1.0881	0.030*
C16	0.52661 (4)	1.1439 (3)	0.80695 (6)	0.01969 (15)
H16A	0.5598	1.2597	0.8049	0.030*
H16B	0.4989	1.2941	0.7880	0.030*
H16C	0.5276	0.9555	0.7642	0.030*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01673 (9)	0.02390 (10)	0.01613 (9)	−0.00032 (6)	−0.00311 (6)	0.00034 (9)
O1	0.0181 (3)	0.0441 (5)	0.0164 (3)	−0.0106 (3)	−0.0019 (2)	0.0083 (3)
O2	0.0169 (2)	0.0225 (3)	0.0104 (2)	−0.0028 (2)	0.00034 (19)	0.00240 (19)
O3	0.0152 (2)	0.0203 (3)	0.0130 (2)	−0.00066 (19)	−0.00317 (18)	−0.0026 (2)
O4	0.0148 (2)	0.0241 (3)	0.0136 (2)	−0.0053 (2)	0.0005 (2)	0.0021 (2)
C1	0.0243 (4)	0.0266 (4)	0.0127 (3)	0.0053 (3)	−0.0033 (3)	−0.0005 (3)
C2	0.0220 (4)	0.0247 (4)	0.0185 (3)	0.0027 (3)	0.0059 (3)	0.0038 (3)
C3	0.0144 (3)	0.0203 (3)	0.0108 (3)	0.0051 (2)	−0.0017 (2)	−0.0003 (2)

C4	0.0135 (3)	0.0168 (3)	0.0129 (3)	0.0002 (2)	−0.0012 (2)	0.0020 (2)
C5	0.0142 (3)	0.0215 (3)	0.0128 (3)	−0.0013 (3)	−0.0015 (2)	0.0025 (2)
C6	0.0147 (3)	0.0199 (3)	0.0138 (3)	−0.0014 (2)	−0.0017 (2)	0.0012 (2)
C7	0.0134 (3)	0.0206 (3)	0.0125 (3)	−0.0006 (2)	−0.0014 (2)	0.0006 (2)
C8	0.0123 (3)	0.0168 (3)	0.0108 (2)	0.0004 (2)	−0.0006 (2)	−0.0002 (2)
C9	0.0125 (3)	0.0169 (3)	0.0117 (3)	−0.0006 (2)	−0.0001 (2)	0.0002 (2)
C10	0.0128 (3)	0.0149 (3)	0.0101 (3)	0.0006 (2)	0.0003 (2)	0.0001 (2)
C11	0.0130 (3)	0.0153 (3)	0.0105 (2)	−0.0002 (2)	−0.0003 (2)	−0.0003 (2)
C12	0.0127 (3)	0.0157 (3)	0.0116 (3)	−0.0003 (2)	0.0010 (2)	−0.0007 (2)
C13	0.0129 (3)	0.0178 (3)	0.0113 (3)	0.0000 (2)	0.0002 (2)	0.0001 (2)
C14	0.0186 (3)	0.0206 (4)	0.0152 (3)	−0.0016 (3)	0.0039 (2)	0.0030 (3)
C15	0.0174 (3)	0.0188 (3)	0.0246 (4)	0.0001 (3)	−0.0066 (3)	−0.0001 (3)
C16	0.0171 (3)	0.0252 (4)	0.0168 (3)	−0.0013 (3)	0.0032 (3)	0.0051 (3)

Geometric parameters (Å, º)

S1—C1	1.6921 (11)	C7—C8	1.4598 (11)
S1—C4	1.7104 (8)	C7—H7A	0.9300
O1—C5	1.2347 (11)	C8—C9	1.4015 (11)
O2—C10	1.3642 (10)	C8—C13	1.4040 (11)
O2—C14	1.4325 (11)	C9—C10	1.3920 (11)
O3—C11	1.3725 (10)	C9—H9A	0.9300
O3—C15	1.4304 (12)	C10—C11	1.4023 (11)
O4—C12	1.3630 (10)	C11—C12	1.4073 (11)
O4—C16	1.4312 (11)	C12—C13	1.3905 (11)
C1—C2	1.3704 (15)	C13—H13A	0.9300
C1—H1A	0.9300	C14—H14A	0.9600
C2—C3	1.4335 (13)	C14—H14B	0.9600
C2—H2A	0.9300	C14—H14C	0.9600
C3—C4	1.4382 (12)	C15—H15A	0.9600
C3—H3A	0.9300	C15—H15B	0.9600
C4—C5	1.4619 (12)	C15—H15C	0.9600
C5—C6	1.4792 (12)	C16—H16A	0.9600
C6—C7	1.3437 (11)	C16—H16B	0.9600
C6—H6A	0.9300	C16—H16C	0.9600
C1—S1—C4	92.57 (5)	O2—C10—C9	124.24 (7)
C10—O2—C14	116.54 (7)	O2—C10—C11	115.83 (7)
C11—O3—C15	114.04 (7)	C9—C10—C11	119.93 (7)
C12—O4—C16	116.78 (7)	O3—C11—C10	119.29 (7)
C2—C1—S1	112.61 (7)	O3—C11—C12	120.91 (7)
C2—C1—H1A	123.7	C10—C11—C12	119.73 (7)
S1—C1—H1A	123.7	O4—C12—C13	124.62 (7)
C1—C2—C3	113.88 (8)	O4—C12—C11	114.94 (7)
C1—C2—H2A	123.1	C13—C12—C11	120.43 (7)
C3—C2—H2A	123.1	C12—C13—C8	119.54 (7)
C2—C3—C4	109.02 (8)	C12—C13—H13A	120.2
C2—C3—H3A	125.5	C8—C13—H13A	120.2

C4—C3—H3A	125.5	O2—C14—H14A	109.5
C3—C4—C5	129.94 (7)	O2—C14—H14B	109.5
C3—C4—S1	111.91 (6)	H14A—C14—H14B	109.5
C5—C4—S1	118.14 (6)	O2—C14—H14C	109.5
O1—C5—C4	119.89 (8)	H14A—C14—H14C	109.5
O1—C5—C6	122.19 (8)	H14B—C14—H14C	109.5
C4—C5—C6	117.90 (7)	O3—C15—H15A	109.5
C7—C6—C5	120.26 (8)	O3—C15—H15B	109.5
C7—C6—H6A	119.9	H15A—C15—H15B	109.5
C5—C6—H6A	119.9	O3—C15—H15C	109.5
C6—C7—C8	127.94 (8)	H15A—C15—H15C	109.5
C6—C7—H7A	116.0	H15B—C15—H15C	109.5
C8—C7—H7A	116.0	O4—C16—H16A	109.5
C9—C8—C13	120.22 (7)	O4—C16—H16B	109.5
C9—C8—C7	117.10 (7)	H16A—C16—H16B	109.5
C13—C8—C7	122.68 (7)	O4—C16—H16C	109.5
C10—C9—C8	120.15 (7)	H16A—C16—H16C	109.5
C10—C9—H9A	119.9	H16B—C16—H16C	109.5
C8—C9—H9A	119.9		

C4—S1—C1—C2	−0.66 (8)	C14—O2—C10—C11	178.47 (7)
S1—C1—C2—C3	0.53 (11)	C8—C9—C10—O2	179.44 (8)
C1—C2—C3—C4	−0.06 (11)	C8—C9—C10—C11	−0.40 (12)
C2—C3—C4—C5	178.30 (9)	C15—O3—C11—C10	106.33 (9)
C2—C3—C4—S1	−0.43 (9)	C15—O3—C11—C12	−76.76 (10)
C1—S1—C4—C3	0.62 (7)	O2—C10—C11—O3	−2.98 (11)
C1—S1—C4—C5	−178.27 (7)	C9—C10—C11—O3	176.88 (8)
C3—C4—C5—O1	176.49 (10)	O2—C10—C11—C12	−179.93 (7)
S1—C4—C5—O1	−4.85 (13)	C9—C10—C11—C12	−0.07 (12)
C3—C4—C5—C6	−5.52 (14)	C16—O4—C12—C13	0.47 (12)
S1—C4—C5—C6	173.14 (7)	C16—O4—C12—C11	−179.67 (8)
O1—C5—C6—C7	10.94 (15)	O3—C11—C12—O4	3.51 (11)
C4—C5—C6—C7	−166.99 (8)	C10—C11—C12—O4	−179.59 (7)
C5—C6—C7—C8	176.81 (8)	O3—C11—C12—C13	−176.63 (8)
C6—C7—C8—C9	177.83 (9)	C10—C11—C12—C13	0.27 (12)
C6—C7—C8—C13	−3.34 (14)	O4—C12—C13—C8	179.86 (8)
C13—C8—C9—C10	0.69 (12)	C11—C12—C13—C8	0.01 (12)
C7—C8—C9—C10	179.55 (8)	C9—C8—C13—C12	−0.49 (12)
C14—O2—C10—C9	−1.38 (12)	C7—C8—C13—C12	−179.29 (8)

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1A···O1i	0.93	2.52	3.1827 (14)	129
C7—H7A···O1	0.93	2.48	2.8243 (12)	102
C15—H15B···O4	0.96	2.49	3.0396 (13)	116
C15—H15C···O3ii	0.96	2.39	3.3340 (11)	169

| C7—H7*A*···O1 | 0.93 | 2.48 | 2.8243 (12) | 102 |
| C15—H15*B*···O4 | 0.96 | 2.49 | 3.0396 (13) | 116 |

Symmetry codes: (i) −*x*+1/2, *y*+1/2, *z*−1/2; (ii) *x*, *y*−1, *z*.