

(E)-1-(2,5-Dichloro-3-thienyl)-3-[4-(dimethylamino)phenyl]prop-2-en-1-one

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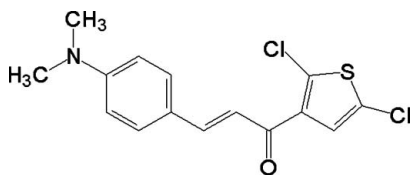
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 Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.038; wR factor = 0.109; data-to-parameter ratio = 17.1.

In the title compound, $\text{C}_{15}\text{H}_{13}\text{Cl}_2\text{NOS}$, the benzene and thiophene rings make a dihedral angle of $10.8(1)^\circ$. The dimethylamino substituent and the α,β -unsaturated carbonyl group are almost coplanar with respect to the aromatic ring, forming dihedral angles of $4.73(3)^\circ$ and $5.0(2)^\circ$, respectively. In the crystal structure, molecules are connected into two-dimensional layers by weak $\text{C}-\text{H}\cdots\text{Cl}$ hydrogen bonds and $\text{C}-\text{Cl}\cdots\text{O}$ [$\text{Cl}\cdots\text{O} = 3.073(2)$ Å] interactions. These layers are stacked with short $\text{C}(\text{methyl})-\text{H}\cdots\pi$ contacts between the layers.

Related literature

For applications of chalcone derivatives, see: Indira *et al.* (2002); Sarojini *et al.* (2006); Tomar *et al.* (2007).



Experimental

Crystal data

 $\text{C}_{15}\text{H}_{13}\text{Cl}_2\text{NOS}$
 $M_r = 326.22$
 Triclinic, $P\bar{1}$
 $a = 7.2637(9)$ Å
 $b = 8.1136(9)$ Å

 $c = 13.478(2)$ Å
 $\alpha = 89.011(9)^\circ$
 $\beta = 79.71(1)^\circ$
 $\gamma = 73.07(1)^\circ$
 $V = 747.2(2)$ Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.57$ mm⁻¹
 $T = 295$ K
 $0.6 \times 0.3 \times 0.3$ mm

Data collection

 Oxford Diffraction Xcalibur diffractometer with an Eos detector
 Absorption correction: multi-scan (*CrysAlis PRO*; Oxford)

 Diffraction, 2009)
 $T_{\min} = 0.785$, $T_{\max} = 1.000$
 8710 measured reflections
 3152 independent reflections
 2403 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.018$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.109$
 $S = 1.10$
 3152 reflections

 184 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.37$ e Å⁻³
 $\Delta\rho_{\min} = -0.40$ e Å⁻³
Table 1

Hydrogen-bond geometry (Å, °).

 C_g is the centroid of the phenyl ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C16}-\text{H16B}\cdots\text{Cl5}^i$	0.96	2.72	3.664 (2)	168
$\text{C16}-\text{H16A}\cdots\text{Cg}^{ii}$	0.96	3.01	3.899 (3)	155

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *Stereochemical Workstation Operation Manual* (Siemens, 1989) and *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2193).

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supporting information

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(E)-1-(2,5-Dichloro-3-thienyl)-3-[4-(dimethylamino)phenyl]prop-2-en-1-one**Grzegorz Dutkiewicz, C. S. Chidan Kumar, H. S. Yathirajan, B. Narayana and Maciej Kubicki****S1. Comment**

Chalcones derivatives are known for their interesting pharmacological activities. Radical quenching properties of the phenolic groups present in many chalcones have raised interest in using the compounds themselves or chalcone rich plant extracts as drugs or food preservatives. Apart from being biologically important compounds, chalcone derivatives show non-linear optical properties with excellent blue light transmittance and good crystallizability (Indira *et al.*, 2002; Sarojini *et al.*, 2006). They provide a necessary configuration to show NLO property with two planar rings connected by a conjugated double bond. Synthesis and antimicrobial evaluation of new chalcones containing a 2,5-dichlorothiophene moiety is reported (Tomar *et al.*, 2007). Here, we report the synthesis and crystal structure of the new chalcone derivative, (2E)-1-(2,5-dichlorothiophen-3-yl)-3-(4-dimethylamino-phenyl)prop-2-en-1-one (**I**, Scheme 1).

The molecule as a whole does not deviate significantly from planarity (Fig. 1). Dihedral angles between the constituent planar fragments are relatively small. The two ring planes of the phenyl and thiophene groups make a dihedral angle of 10.8 (1)°. The dimethylamino substituent and the α,β -unsaturated carbonyl moiety are inclined with respect to the phenyl ring plane by 4.73 (3)° and 5.0 (2)°, respectively. The bond lengths pattern within the C(=O)—C=C- fragment shows significant conjugation with shorter formal single bonds compared to formal double bonds that are longer than typical values.

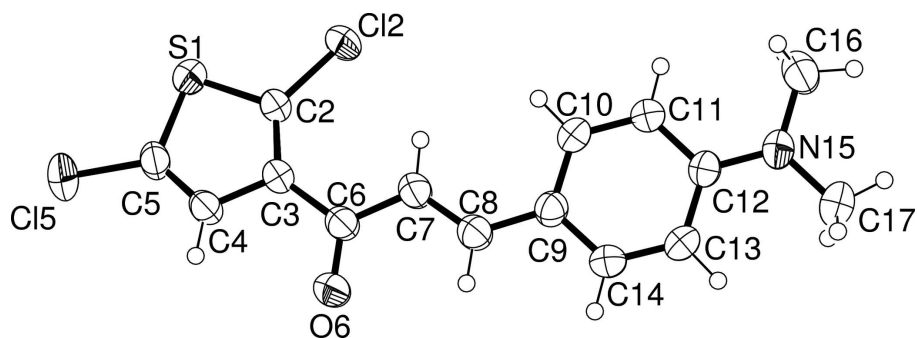
In the crystal structure an intermolecular C—H...Cl hydrogen bond (H...Cl distance 2.72 Å, C—H...Cl angle 168°) and C—Cl...O interactions connect the molecules into approximately planar layers parallel to (101) (Fig. 2). The chlorine oxygen interaction also is almost linear (C2—Cl2...O6 angle of 167.6 (8)°) and relatively short (Cl2...O6 3.073 (2) Å). These layers are stacked on each other showing additional intermolecular C—H... π interactions with H16A...Cg distance of 3.01 Å (Cg is the centroid of the phenyl ring).

S2. Experimental

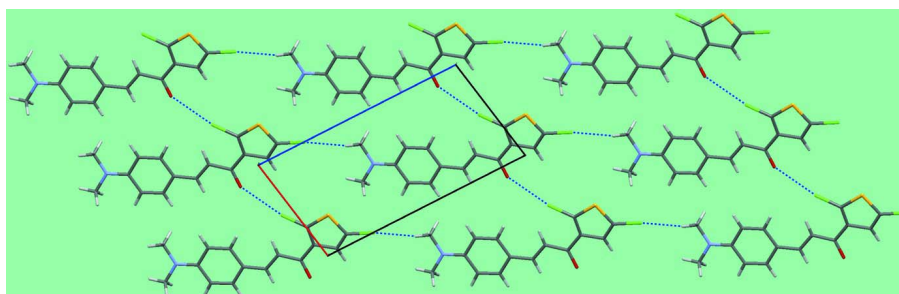
1-(2,5-Dichlorothiophen-3-yl)ethanone (1.95 g, 0.01 mol) was mixed with 4-dimethylamino-benzaldehyde (1.49 g, 0.01 mol) and dissolved in ethanol (30 ml). 3 ml of KOH (50%) was added to this solution. The reaction mixture was stirred for 6 hours. The resulting crude solid was filtered, washed successively with distilled water and finally recrystallized from ethanol (95%) to give the pure chalcone. Crystals suitable for x-ray diffraction studies were grown by slow evaporation of solution in toluene (M.P.: 358 K).

S3. Refinement

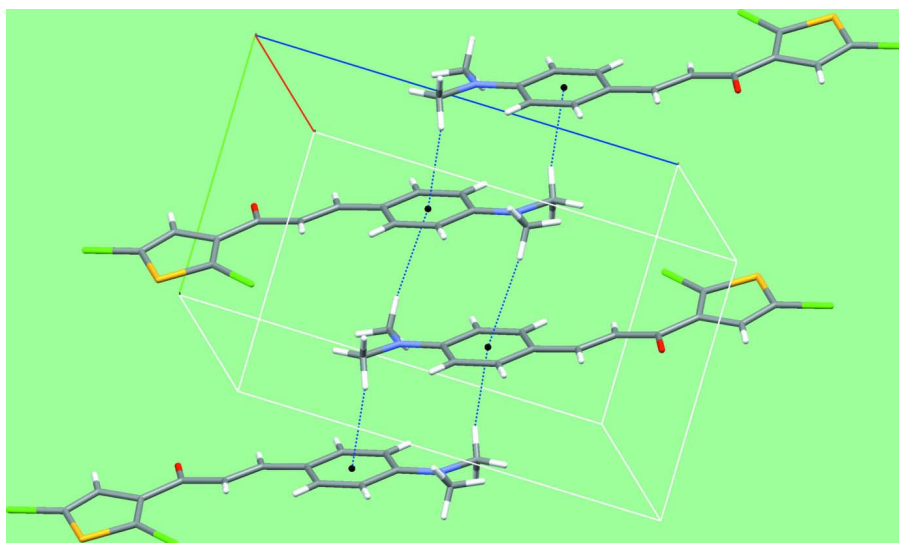
H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C—H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ for phenyl hydrogen and olefinic CH groups and with 0.96 Å and $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{C})$ for CH₃ groups.

**Figure 1**

Molecular structure of (**I**), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

**Figure 2**

Crystal packing of (**I**) viewed along the *b* axis. C—H...Cl hydrogen bonds and C—Cl...O interactions are shown as dashed lines.

**Figure 3**

C—H... π interactions in the stack of molecules (**I**).

(E)-1-(2,5-Dichloro-3-thienyl)-3-[4-(dimethylamino)phenyl]prop-2-en-1-one*Crystal data*C₁₅H₁₃Cl₂NOS $M_r = 326.22$ Triclinic, $P\bar{1}$

Hall symbol: -P 1

 $a = 7.2637$ (9) Å $b = 8.1136$ (9) Å $c = 13.478$ (2) Å $\alpha = 89.011$ (9)° $\beta = 79.71$ (1)° $\gamma = 73.07$ (1)° $V = 747.2$ (2) Å³ $Z = 2$ $F(000) = 336$ $D_x = 1.450$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 5611 reflections

 $\theta = 2.6$ – 28.2 ° $\mu = 0.57$ mm⁻¹ $T = 295$ K

Block, yellow

 $0.6 \times 0.3 \times 0.3$ mm*Data collection*

Oxford Diffraction Xcalibur

diffractometer with an Eos detector

Radiation source: Enhance (Mo) X-ray Source

Graphite monochromator

Detector resolution: 16.1544 pixels mm⁻¹ ω scan

Absorption correction: multi-scan

(CrysAlis PRO; Oxford Diffraction, 2009)

 $T_{\min} = 0.785$, $T_{\max} = 1.000$

8710 measured reflections

3152 independent reflections

2403 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.018$ $\theta_{\max} = 28.3$ °, $\theta_{\min} = 2.6$ ° $h = -9 \rightarrow 9$ $k = -10 \rightarrow 10$ $l = -17 \rightarrow 17$ *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.038$ $wR(F^2) = 0.109$ $S = 1.10$

3152 reflections

184 parameters

0 restraints

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

 $w = 1/[\sigma^2(F_o^2) + (0.0546P)^2 + 0.1725P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.001$ $\Delta\rho_{\max} = 0.37$ e Å⁻³ $\Delta\rho_{\min} = -0.40$ e Å⁻³*Special details*

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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 (Å²)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.67788 (8)	0.11979 (8)	1.11606 (4)	0.05238 (18)
Cl2	0.49256 (8)	0.25700 (9)	0.94627 (4)	0.0620 (2)
C2	0.6887 (3)	0.2294 (3)	1.00611 (14)	0.0407 (4)

C3	0.8555 (3)	0.2780 (2)	0.98033 (14)	0.0383 (4)
C4	0.9777 (3)	0.2234 (3)	1.05438 (15)	0.0431 (5)
H4A	1.0980	0.2443	1.0507	0.052*
C15	1.00237 (10)	0.05512 (9)	1.23148 (4)	0.0699 (2)
C5	0.9014 (3)	0.1398 (3)	1.12908 (15)	0.0454 (5)
C6	0.9255 (3)	0.3667 (3)	0.89011 (15)	0.0432 (5)
O6	1.0970 (2)	0.3669 (2)	0.87491 (13)	0.0636 (5)
C7	0.7927 (3)	0.4499 (3)	0.82226 (16)	0.0473 (5)
H7A	0.6621	0.4519	0.8378	0.057*
C8	0.8548 (3)	0.5232 (3)	0.73845 (16)	0.0456 (5)
H8A	0.9845	0.5242	0.7283	0.055*
C9	0.7452 (3)	0.6010 (2)	0.66156 (15)	0.0412 (4)
C10	0.5490 (3)	0.6086 (3)	0.66361 (15)	0.0435 (5)
H10A	0.4820	0.5665	0.7188	0.052*
C11	0.4534 (3)	0.6765 (3)	0.58656 (15)	0.0435 (5)
H11A	0.3233	0.6795	0.5908	0.052*
C12	0.5478 (3)	0.7420 (2)	0.50101 (14)	0.0399 (4)
C13	0.7440 (3)	0.7332 (3)	0.49862 (16)	0.0489 (5)
H13A	0.8125	0.7735	0.4432	0.059*
C14	0.8364 (3)	0.6662 (3)	0.57683 (16)	0.0500 (5)
H14A	0.9662	0.6641	0.5731	0.060*
N15	0.4527 (3)	0.8088 (2)	0.42422 (14)	0.0527 (5)
C16	0.2469 (4)	0.8283 (3)	0.4313 (2)	0.0669 (7)
H16A	0.2267	0.7165	0.4308	0.100*
H16B	0.2015	0.8906	0.3749	0.100*
H16C	0.1756	0.8906	0.4929	0.100*
C17	0.5519 (4)	0.8642 (4)	0.33356 (17)	0.0657 (7)
H17A	0.5962	0.9594	0.3497	0.098*
H17B	0.4636	0.8996	0.2868	0.098*
H17C	0.6623	0.7707	0.3037	0.098*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0501 (3)	0.0691 (4)	0.0431 (3)	-0.0253 (3)	-0.0097 (2)	0.0124 (3)
C12	0.0468 (3)	0.0961 (5)	0.0581 (4)	-0.0360 (3)	-0.0242 (3)	0.0189 (3)
C2	0.0371 (10)	0.0488 (11)	0.0378 (10)	-0.0123 (9)	-0.0117 (8)	0.0027 (8)
C3	0.0376 (10)	0.0399 (10)	0.0387 (10)	-0.0103 (8)	-0.0124 (8)	0.0024 (8)
C4	0.0383 (10)	0.0475 (12)	0.0464 (11)	-0.0125 (9)	-0.0160 (9)	0.0042 (9)
C15	0.0750 (4)	0.0881 (5)	0.0490 (3)	-0.0178 (3)	-0.0300 (3)	0.0209 (3)
C5	0.0479 (11)	0.0516 (12)	0.0372 (10)	-0.0104 (9)	-0.0162 (9)	0.0060 (9)
C6	0.0400 (10)	0.0458 (11)	0.0467 (11)	-0.0139 (9)	-0.0138 (9)	0.0059 (9)
O6	0.0461 (9)	0.0881 (12)	0.0681 (11)	-0.0317 (8)	-0.0226 (8)	0.0325 (9)
C7	0.0437 (11)	0.0525 (12)	0.0500 (12)	-0.0165 (10)	-0.0170 (9)	0.0138 (10)
C8	0.0436 (11)	0.0482 (12)	0.0490 (12)	-0.0165 (9)	-0.0139 (9)	0.0072 (9)
C9	0.0448 (11)	0.0398 (11)	0.0408 (10)	-0.0144 (9)	-0.0091 (8)	0.0057 (8)
C10	0.0447 (11)	0.0474 (12)	0.0389 (10)	-0.0165 (9)	-0.0046 (8)	0.0086 (9)
C11	0.0382 (10)	0.0496 (12)	0.0436 (11)	-0.0148 (9)	-0.0078 (8)	0.0083 (9)

C12	0.0451 (11)	0.0370 (10)	0.0379 (10)	-0.0119 (8)	-0.0085 (8)	0.0044 (8)
C13	0.0474 (12)	0.0568 (13)	0.0456 (12)	-0.0226 (10)	-0.0054 (9)	0.0145 (10)
C14	0.0404 (11)	0.0612 (14)	0.0528 (13)	-0.0217 (10)	-0.0098 (9)	0.0138 (10)
N15	0.0540 (11)	0.0618 (12)	0.0466 (10)	-0.0205 (9)	-0.0163 (8)	0.0205 (8)
C16	0.0601 (15)	0.0778 (17)	0.0707 (16)	-0.0228 (13)	-0.0302 (13)	0.0236 (13)
C17	0.0734 (17)	0.0800 (17)	0.0454 (13)	-0.0247 (14)	-0.0132 (12)	0.0191 (12)

Geometric parameters (Å, °)

S1—C2	1.717 (2)	C10—C11	1.371 (3)
S1—C5	1.717 (2)	C10—H10A	0.9300
C12—C2	1.7175 (19)	C11—C12	1.411 (3)
C2—C3	1.366 (3)	C11—H11A	0.9300
C3—C4	1.433 (3)	C12—N15	1.364 (3)
C3—C6	1.489 (3)	C12—C13	1.401 (3)
C4—C5	1.333 (3)	C13—C14	1.371 (3)
C4—H4A	0.9300	C13—H13A	0.9300
C15—C5	1.718 (2)	C14—H14A	0.9300
C6—O6	1.226 (2)	N15—C17	1.439 (3)
C6—C7	1.462 (3)	N15—C16	1.443 (3)
C7—C8	1.335 (3)	C16—H16A	0.9600
C7—H7A	0.9300	C16—H16B	0.9600
C8—C9	1.444 (3)	C16—H16C	0.9600
C8—H8A	0.9300	C17—H17A	0.9600
C9—C14	1.391 (3)	C17—H17B	0.9600
C9—C10	1.403 (3)	C17—H17C	0.9600
C2—S1—C5	89.86 (10)	C10—C11—C12	121.54 (19)
C3—C2—S1	113.70 (14)	C10—C11—H11A	119.2
C3—C2—C12	130.86 (16)	C12—C11—H11A	119.2
S1—C2—C12	115.43 (11)	N15—C12—C13	121.88 (18)
C2—C3—C4	110.04 (18)	N15—C12—C11	121.47 (19)
C2—C3—C6	130.73 (17)	C13—C12—C11	116.65 (18)
C4—C3—C6	119.18 (17)	C14—C13—C12	120.89 (19)
C5—C4—C3	113.12 (18)	C14—C13—H13A	119.6
C5—C4—H4A	123.4	C12—C13—H13A	119.6
C3—C4—H4A	123.4	C13—C14—C9	123.04 (19)
C4—C5—S1	113.29 (15)	C13—C14—H14A	118.5
C4—C5—C15	127.12 (17)	C9—C14—H14A	118.5
S1—C5—C15	119.59 (13)	C12—N15—C17	121.73 (19)
O6—C6—C7	121.58 (19)	C12—N15—C16	121.05 (19)
O6—C6—C3	117.89 (17)	C17—N15—C16	117.22 (19)
C7—C6—C3	120.54 (17)	N15—C16—H16A	109.5
C8—C7—C6	121.49 (19)	N15—C16—H16B	109.5
C8—C7—H7A	119.3	H16A—C16—H16B	109.5
C6—C7—H7A	119.3	N15—C16—H16C	109.5
C7—C8—C9	127.96 (19)	H16A—C16—H16C	109.5
C7—C8—H8A	116.0	H16B—C16—H16C	109.5

C9—C8—H8A	116.0	N15—C17—H17A	109.5
C14—C9—C10	116.05 (18)	N15—C17—H17B	109.5
C14—C9—C8	120.06 (18)	H17A—C17—H17B	109.5
C10—C9—C8	123.80 (18)	N15—C17—H17C	109.5
C11—C10—C9	121.81 (18)	H17A—C17—H17C	109.5
C11—C10—H10A	119.1	H17B—C17—H17C	109.5
C9—C10—H10A	119.1		
C5—S1—C2—C3	0.36 (17)	C6—C7—C8—C9	176.1 (2)
C5—S1—C2—C12	179.45 (13)	C7—C8—C9—C14	-177.7 (2)
S1—C2—C3—C4	-0.3 (2)	C7—C8—C9—C10	-1.0 (4)
C12—C2—C3—C4	-179.25 (16)	C14—C9—C10—C11	0.0 (3)
S1—C2—C3—C6	176.89 (17)	C8—C9—C10—C11	-176.8 (2)
C12—C2—C3—C6	-2.0 (4)	C9—C10—C11—C12	0.1 (3)
C2—C3—C4—C5	0.1 (3)	C10—C11—C12—N15	179.72 (19)
C6—C3—C4—C5	-177.47 (18)	C10—C11—C12—C13	0.3 (3)
C3—C4—C5—S1	0.1 (2)	N15—C12—C13—C14	179.7 (2)
C3—C4—C5—C15	-179.88 (15)	C11—C12—C13—C14	-0.9 (3)
C2—S1—C5—C4	-0.28 (18)	C12—C13—C14—C9	1.0 (4)
C2—S1—C5—C15	179.74 (14)	C10—C9—C14—C13	-0.5 (3)
C2—C3—C6—O6	-166.3 (2)	C8—C9—C14—C13	176.4 (2)
C4—C3—C6—O6	10.7 (3)	C13—C12—N15—C17	4.0 (3)
C2—C3—C6—C7	13.5 (3)	C11—C12—N15—C17	-175.4 (2)
C4—C3—C6—C7	-169.47 (18)	C13—C12—N15—C16	-175.5 (2)
O6—C6—C7—C8	3.0 (3)	C11—C12—N15—C16	5.2 (3)
C3—C6—C7—C8	-176.8 (2)		

Hydrogen-bond geometry (\AA , $^\circ$)

Cg is the centroid of the phenyl ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C16—H16B \cdots C15 ⁱ	0.96	2.72	3.664 (2)	168
C16—H16A \cdots Cg ⁱⁱ	0.96	3.01	3.899 (3)	155

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