

# (*E*)-1-(5-Chlorothiophen-2-yl)-3-(2,4-dimethylphen-yl)prop-2-en-1-one

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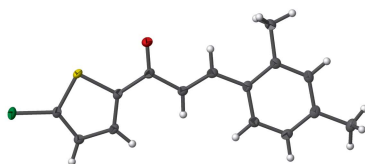
Keywords: crystal structure; bis-chalcone; hydrogen bonds.

CCDC reference: 1522076

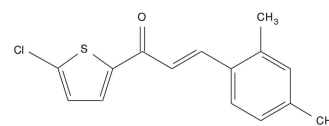
Structural data: full structural data are available from [iucrdata.iucr.org](http://iucrdata.iucr.org)

In the title compound, C<sub>15</sub>H<sub>13</sub>ClOS, the olefinic double bond adopts an *E* configuration. The molecule is nearly planar, as seen by the dihedral angle of 9.07 (8)° between the thiophene and phenyl rings. The *trans* configuration of the C=C double bond in the central enone group is confirmed by the C–C=C–C torsion angle of 177.6 (2)°. In the crystal, molecules are linked by weak C–H···O and C–H···S hydrogen bonds, forming chains propagating along the *c* axis.

## 3D view



## Chemical scheme



## Structure description

Chalcones form the central cores for the construction of variety of bioactive molecules (Naveen *et al.*, 2016). The most commonly employed method for the synthesis of chalcones involves the condensation of an aromatic aldehyde and an aromatic ketone in the presence of aqueous alkaline bases (Mahapatra *et al.*, 2015). In view of the broad spectrum of applications associated with chalcones and as a part of our ongoing work on such molecules (Tejkiran *et al.*, 2016), we report here the synthesis and crystal structure of the title compound.

The title molecule (Fig. 1) is nearly planar, with a dihedral angle of 9.07 (8)° between the thiophene and phenyl rings that are bridged by the olefinic double bond. This value is less than the value of 19.13 (15)° reported earlier between the aromatic rings in the related chalcone derivative (*E*)-3-(2,3-dichlorophenyl)-1-(4-fluorophenyl)prop-2-en-1-one (Naveen *et al.*, 2016). The *trans* configuration about the C6=C7 double bond in the central enone group is confirmed by the C5–C6=C7–C8 torsion angle, 177.6 (2)°. The

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C3-H3\cdots O1^i$	0.93	2.48	3.400 (3)	169
$C3-H3\cdots S1^i$	0.93	2.90	3.459 (2)	120

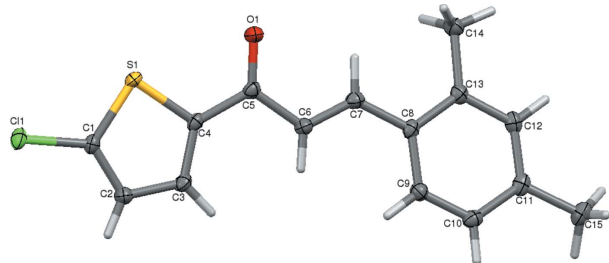
Symmetry code: (i)  $x, -y + \frac{1}{2}, z + \frac{1}{2}$ .

carbonyl group at C5 lies in the plane of the olefinic double bond and thiophene ring as indicated by the  $S1-C4-C5-O1$  [ $1.2(3)^\circ$ ] and  $O1-C5-C6-C7$  [ $-4.7(3)^\circ$ ] torsion angles.

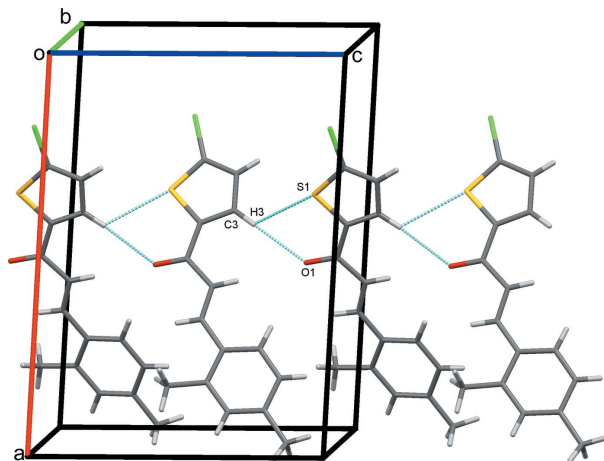
In the crystal, the molecules are linked *via* weak  $C-H\cdots O$  and  $C-H\cdots S$  hydrogen bonds, forming chains propagating along the  $c$  axis (Table 1 and Fig. 2).

### Synthesis and crystallization

A mixture of 1-(5-chlorothiophen-2-yl)ethanone (5 mmol), 2,4-dimethylbenzaldehyde (5 mmol) and potassium hydroxide (5 mmol) in 95% ethyl alcohol (25 ml) was stirred at room temperature for 3 h. The progress of the reaction was monitored by TLC. After completion, the reaction mixture was poured in to ice-cold water and kept in the refrigerator overnight. The solid that formed was filtered, and washed with cold methanol (5%) to obtain the crude product. Pure green



**Figure 1**  
The molecular structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.



**Figure 2**  
Packing of the molecules viewed along the  $b$  axis, with hydrogen bonds drawn as dashed lines.

**Table 2**  
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{13}ClOS$
$M_r$	276.77
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
$a, b, c$ (Å)	15.588 (2), 7.4306 (11), 11.4165 (17)
$\beta$ (°)	94.293 (3)
$V$ (Å <sup>3</sup> )	1318.6 (3)
$Z$	4
Radiation type	Cu $K\alpha$
$\mu$ (mm <sup>-1</sup> )	3.90
Crystal size (mm)	0.29 × 0.26 × 0.23
Data collection	
Diffractometer	Bruker X8 Proteum
Absorption correction	Multi-scan (SADABS; Bruker, 2013)
$T_{min}, T_{max}$	0.397, 0.467
No. of measured, independent and observed [ $I > 2\sigma(I)$ ] reflections	9075, 2149, 2120
$R_{int}$	0.050
$(\sin \theta/\lambda)_{max}$ (Å <sup>-1</sup> )	0.584
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.054, 0.148, 1.11
No. of reflections	2149
No. of parameters	165
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å <sup>-3</sup> )	0.50, -0.87

Computer programs: APEX2 and SAINT (Bruker, 2013), SHELXS97 and SHELXL97 (Sheldrick, 2008), Mercury (Macrae *et al.*, 2008).

crystals of the title compound were obtained by crystallization from methanol by the slow evaporation technique, m.p. 98–100°C.

### Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

### Acknowledgements

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## full crystallographic data

*IUCrData* (2016). **1**, x161974 [https://doi.org/10.1107/S241431461601974X]

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**(E)-1-(5-Chlorothiophen-2-yl)-3-(2,4-dimethylphenyl)prop-2-en-1-one***Crystal data*

C<sub>15</sub>H<sub>13</sub>ClOS

$M_r = 276.77$

Monoclinic,  $P2_1/c$

Hall symbol: -P 2ybc

$a = 15.588$  (2) Å

$b = 7.4306$  (11) Å

$c = 11.4165$  (17) Å

$\beta = 94.293$  (3)°

$V = 1318.6$  (3) Å<sup>3</sup>

$Z = 4$

$F(000) = 576$

$D_x = 1.394$  Mg m<sup>-3</sup>

Cu  $K\alpha$  radiation,  $\lambda = 1.54178$  Å

Cell parameters from 2120 reflections

$\theta = 5.7$ – $64.2$ °

$\mu = 3.90$  mm<sup>-1</sup>

$T = 296$  K

Rectangle, green

$0.29 \times 0.26 \times 0.23$  mm

*Data collection*

Bruker X8 Proteum  
diffractometer

Radiation source: Bruker MicroStar microfocus  
rotating anode

Helios multilayer optics monochromator

Detector resolution: 18.4 pixels mm<sup>-1</sup>

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan  
(SADABS; Bruker, 2013)

$T_{\min} = 0.397$ ,  $T_{\max} = 0.467$

9075 measured reflections

2149 independent reflections

2120 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.050$

$\theta_{\max} = 64.2$ °,  $\theta_{\min} = 5.7$ °

$h = -18 \rightarrow 17$

$k = -7 \rightarrow 8$

$l = -12 \rightarrow 13$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.054$

$wR(F^2) = 0.148$

$S = 1.11$

2149 reflections

165 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

$w = 1/[\sigma^2(F_o^2) + (0.1085P)^2 + 0.5955P]$

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

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

$\Delta\rho_{\max} = 0.50$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.87$  e Å<sup>-3</sup>

*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 esds 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  $F^2 > 2\sigma(F^2)$  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 ( $\text{\AA}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.22467 (3)	0.94779 (7)	0.40476 (4)	0.0210 (2)
S1	0.39687 (3)	0.81834 (7)	0.35033 (4)	0.0179 (2)
O1	0.57439 (10)	0.7025 (2)	0.32391 (14)	0.0240 (5)
C1	0.32622 (13)	0.8762 (3)	0.45189 (19)	0.0163 (6)
C2	0.36001 (14)	0.8625 (3)	0.56508 (19)	0.0181 (6)
C3	0.44560 (15)	0.8010 (3)	0.5701 (2)	0.0174 (6)
C4	0.47497 (13)	0.7695 (3)	0.46152 (19)	0.0155 (6)
C5	0.56000 (14)	0.7119 (3)	0.42881 (19)	0.0170 (6)
C6	0.62517 (14)	0.6691 (3)	0.5239 (2)	0.0172 (6)
C7	0.70203 (14)	0.6053 (3)	0.49961 (19)	0.0179 (7)
C8	0.77481 (14)	0.5622 (3)	0.58247 (19)	0.0163 (7)
C9	0.77421 (14)	0.6023 (3)	0.7026 (2)	0.0187 (6)
C10	0.84389 (15)	0.5652 (3)	0.7802 (2)	0.0192 (7)
C11	0.91834 (14)	0.4879 (3)	0.7412 (2)	0.0185 (6)
C12	0.91894 (14)	0.4460 (3)	0.6227 (2)	0.0185 (7)
C13	0.84913 (14)	0.4818 (3)	0.5421 (2)	0.0173 (6)
C14	0.85383 (15)	0.4288 (3)	0.4153 (2)	0.0218 (7)
C15	0.99642 (15)	0.4547 (3)	0.8251 (2)	0.0232 (7)
H2	0.33040	0.89010	0.63050	0.0220*
H3	0.47900	0.78340	0.64010	0.0210*
H6	0.61300	0.68640	0.60160	0.0210*
H7	0.71000	0.58610	0.42070	0.0210*
H9	0.72560	0.65510	0.73040	0.0220*
H10	0.84140	0.59190	0.85950	0.0230*
H12	0.96760	0.39200	0.59600	0.0220*
H14A	0.90930	0.37800	0.40460	0.0330*
H14B	0.84510	0.53310	0.36630	0.0330*
H14C	0.81000	0.34140	0.39420	0.0330*
H15A	1.02790	0.35360	0.79840	0.0350*
H15B	0.97840	0.43010	0.90200	0.0350*
H15C	1.03260	0.55950	0.82810	0.0350*

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Cl1	0.0149 (4)	0.0250 (4)	0.0222 (4)	0.0028 (2)	-0.0046 (2)	0.0022 (2)
S1	0.0155 (4)	0.0266 (4)	0.0112 (4)	0.0009 (2)	-0.0024 (2)	-0.0011 (2)
O1	0.0188 (9)	0.0397 (10)	0.0134 (9)	0.0036 (7)	0.0011 (6)	-0.0022 (6)
C1	0.0136 (10)	0.0163 (11)	0.0185 (11)	-0.0008 (8)	-0.0014 (8)	0.0001 (8)

C2	0.0175 (11)	0.0218 (11)	0.0151 (11)	0.0002 (9)	0.0013 (8)	-0.0008 (9)
C3	0.0175 (11)	0.0199 (11)	0.0141 (11)	-0.0009 (8)	-0.0025 (9)	0.0031 (8)
C4	0.0152 (11)	0.0147 (10)	0.0160 (11)	-0.0026 (8)	-0.0022 (8)	0.0004 (8)
C5	0.0171 (11)	0.0177 (10)	0.0159 (11)	-0.0031 (8)	-0.0011 (9)	-0.0018 (8)
C6	0.0171 (11)	0.0201 (11)	0.0142 (11)	-0.0006 (8)	0.0006 (9)	-0.0006 (8)
C7	0.0202 (12)	0.0187 (11)	0.0148 (11)	-0.0020 (9)	0.0014 (9)	0.0005 (9)
C8	0.0155 (12)	0.0150 (11)	0.0182 (11)	-0.0034 (8)	-0.0005 (9)	0.0016 (8)
C9	0.0179 (11)	0.0194 (11)	0.0189 (11)	-0.0003 (8)	0.0027 (9)	-0.0007 (9)
C10	0.0221 (12)	0.0192 (11)	0.0161 (11)	-0.0040 (8)	0.0001 (9)	0.0001 (8)
C11	0.0182 (11)	0.0131 (10)	0.0237 (12)	-0.0046 (8)	-0.0021 (9)	0.0036 (9)
C12	0.0155 (11)	0.0143 (11)	0.0258 (13)	-0.0005 (8)	0.0016 (9)	0.0009 (8)
C13	0.0175 (11)	0.0143 (10)	0.0202 (12)	-0.0035 (8)	0.0022 (9)	0.0001 (8)
C14	0.0204 (12)	0.0241 (12)	0.0211 (12)	0.0015 (8)	0.0032 (10)	-0.0031 (9)
C15	0.0228 (12)	0.0198 (12)	0.0258 (13)	-0.0007 (9)	-0.0060 (10)	0.0011 (9)

*Geometric parameters (Å, °)*

C11—C1	1.718 (2)	C11—C15	1.512 (3)
S1—C1	1.713 (2)	C12—C13	1.397 (3)
S1—C4	1.730 (2)	C13—C14	1.507 (3)
O1—C5	1.237 (3)	C2—H2	0.9300
C1—C2	1.362 (3)	C3—H3	0.9300
C2—C3	1.407 (3)	C6—H6	0.9300
C3—C4	1.373 (3)	C7—H7	0.9300
C4—C5	1.468 (3)	C9—H9	0.9300
C5—C6	1.465 (3)	C10—H10	0.9300
C6—C7	1.337 (3)	C12—H12	0.9300
C7—C8	1.457 (3)	C14—H14A	0.9600
C8—C9	1.404 (3)	C14—H14B	0.9600
C8—C13	1.411 (3)	C14—H14C	0.9600
C9—C10	1.377 (3)	C15—H15A	0.9600
C10—C11	1.397 (3)	C15—H15B	0.9600
C11—C12	1.389 (3)	C15—H15C	0.9600
C1—S1—C4	90.50 (10)	C1—C2—H2	124.00
C11—C1—S1	119.33 (13)	C3—C2—H2	124.00
C11—C1—C2	127.02 (17)	C2—C3—H3	123.00
S1—C1—C2	113.63 (16)	C4—C3—H3	123.00
C1—C2—C3	111.2 (2)	C5—C6—H6	120.00
C2—C3—C4	113.4 (2)	C7—C6—H6	120.00
S1—C4—C3	111.27 (16)	C6—C7—H7	116.00
S1—C4—C5	118.28 (16)	C8—C7—H7	116.00
C3—C4—C5	130.4 (2)	C8—C9—H9	119.00
O1—C5—C4	119.7 (2)	C10—C9—H9	119.00
O1—C5—C6	122.6 (2)	C9—C10—H10	120.00
C4—C5—C6	117.65 (19)	C11—C10—H10	120.00
C5—C6—C7	120.4 (2)	C11—C12—H12	119.00
C6—C7—C8	127.6 (2)	C13—C12—H12	119.00

C7—C8—C9	121.7 (2)	C13—C14—H14A	110.00
C7—C8—C13	120.0 (2)	C13—C14—H14B	110.00
C9—C8—C13	118.3 (2)	C13—C14—H14C	109.00
C8—C9—C10	121.6 (2)	H14A—C14—H14B	109.00
C9—C10—C11	120.7 (2)	H14A—C14—H14C	109.00
C10—C11—C12	118.0 (2)	H14B—C14—H14C	109.00
C10—C11—C15	120.9 (2)	C11—C15—H15A	109.00
C12—C11—C15	121.1 (2)	C11—C15—H15B	110.00
C11—C12—C13	122.5 (2)	C11—C15—H15C	109.00
C8—C13—C12	118.9 (2)	H15A—C15—H15B	109.00
C8—C13—C14	121.6 (2)	H15A—C15—H15C	109.00
C12—C13—C14	119.4 (2)	H15B—C15—H15C	109.00
<hr/>			
C4—S1—C1—C11	179.60 (15)	C6—C7—C8—C9	-6.7 (4)
C4—S1—C1—C2	0.98 (19)	C6—C7—C8—C13	174.8 (2)
C1—S1—C4—C3	-0.93 (18)	C7—C8—C9—C10	-178.5 (2)
C1—S1—C4—C5	-178.14 (18)	C13—C8—C9—C10	0.1 (3)
C11—C1—C2—C3	-179.26 (17)	C7—C8—C13—C12	178.4 (2)
S1—C1—C2—C3	-0.8 (3)	C7—C8—C13—C14	-3.5 (3)
C1—C2—C3—C4	0.0 (3)	C9—C8—C13—C12	-0.2 (3)
C2—C3—C4—S1	0.7 (3)	C9—C8—C13—C14	177.9 (2)
C2—C3—C4—C5	177.5 (2)	C8—C9—C10—C11	0.8 (3)
S1—C4—C5—O1	1.2 (3)	C9—C10—C11—C12	-1.5 (3)
S1—C4—C5—C6	-179.34 (16)	C9—C10—C11—C15	177.2 (2)
C3—C4—C5—O1	-175.4 (2)	C10—C11—C12—C13	1.4 (3)
C3—C4—C5—C6	4.1 (4)	C15—C11—C12—C13	-177.3 (2)
O1—C5—C6—C7	-4.7 (3)	C11—C12—C13—C8	-0.5 (3)
C4—C5—C6—C7	175.9 (2)	C11—C12—C13—C14	-178.7 (2)
C5—C6—C7—C8	177.6 (2)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C3—H3 $\cdots$ O1 <sup>i</sup>	0.93	2.48	3.400 (3)	169
C3—H3 $\cdots$ S1 <sup>i</sup>	0.93	2.90	3.459 (2)	120

Symmetry code: (i)  $x, -y+3/2, z+1/2$ .