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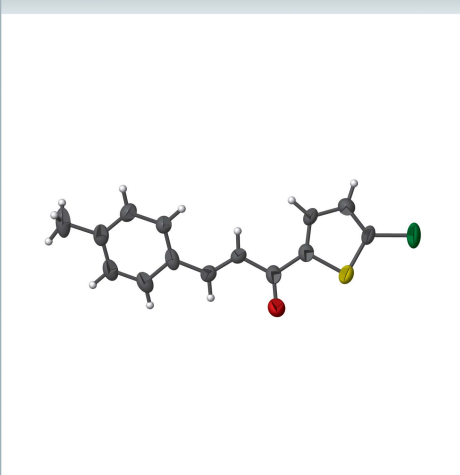
# (*E*)-1-(5-Chlorothiophen-2-yl)-3-(*p*-tolyl)prop-2-en-1-one

Karthik Kumara,<sup>a</sup> S. Naveen,<sup>b</sup> M. G. Prabhudeva,<sup>c</sup> K. Ajay Kumar,<sup>c</sup> N. K. Lokanath<sup>a\*</sup> and Ismail Warad<sup>d\*</sup>

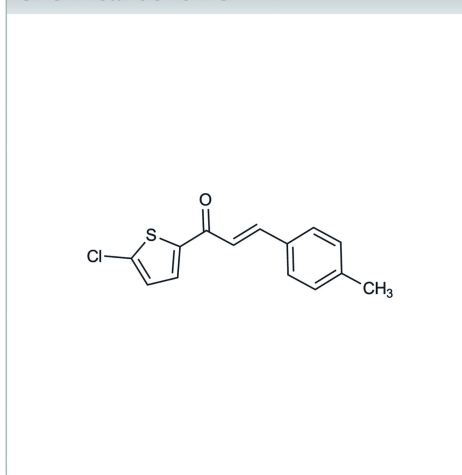
<sup>a</sup>Department of Studies in Physics, University of Mysore, Manasagangotri, Mysuru 570 006, India, <sup>b</sup>Institution of Excellence, University of Mysore, Manasagangotri, Mysuru 570 006, India, <sup>c</sup>Department of Chemistry, Yuvaraja's College, University of Mysore, Mysuru 570 005, India, and <sup>d</sup>Department of Chemistry, Science College, An-Najah National University, PO Box 7, Nablus, West Bank, Palestinian Territories. \*Correspondence e-mail: lokanath@physics.uni-mysore.ac.in, khalil.i@najah.edu

In the title compound, C<sub>14</sub>H<sub>11</sub>ClOS, the *trans* conformation of the C=C double bond in the central enone group is confirmed by the C—C=C—C torsion angle of 178.3 (4)°. The molecule is non-planar as seen by the dihedral angle of 22.6 (2)° between the chlorothiophene and the *p*-toluene rings. In the crystal, molecules are linked by pairs of C—H... $\pi$  interactions, forming inversion dimers. There are no other significant intermolecular interactions present.

## 3D view



## Chemical scheme



## Structure description

The usual method for the synthesis of chalcones involves the condensation of an aromatic aldehyde and aromatic ketone in the presence of aqueous alkaline bases (Naveen *et al.*, 2016a). Chalcones and their derivatives demonstrate a wide range of biological activities such as antidiabetic, antineoplastic, antitubercular, antiarrhythmic, hypnotic, antiangiogenic, antiprotozoal, antibacterial, antisteroidal, cardioprotective. 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; Naveen *et al.*, 2016b), we report herein the synthesis and crystal structure of the title compound.

The molecular structure of the title compound is shown in Fig. 1. The molecule is non-planar, with a dihedral angle of 22.6 (2)° between the chlorothiophene and *p*-toluene rings that are bridged by the olefinic double bond. This value is in good agreement with 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.*, 2016b). The *trans* conformation about the C6=C7 double bond in the

**Table 1**Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ ). $C_g$  is the centroid of the C8–C13 ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C14-H14A\cdots C_g^i$	0.96	2.77	3.576 (6)	141

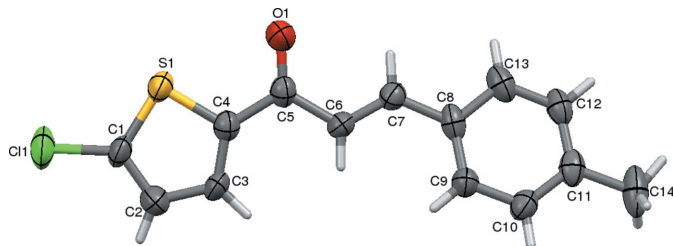
Symmetry code: (i)  $-x+2, -y-1, -z+1$ .

central enone group is confirmed by the  $C5-C6=C7-C8$  torsion angle of  $178.3 (4)^\circ$ . The carbonyl group at C5 lies in the plane of the olefinic double bond and chloro-thiophene rings as indicated by the  $O1-C5-C4-C3$  and  $O1-C5-C6-C7$  torsion angle values of  $177.9 (5)^\circ$  and  $-11.7 (6)^\circ$ , respectively.

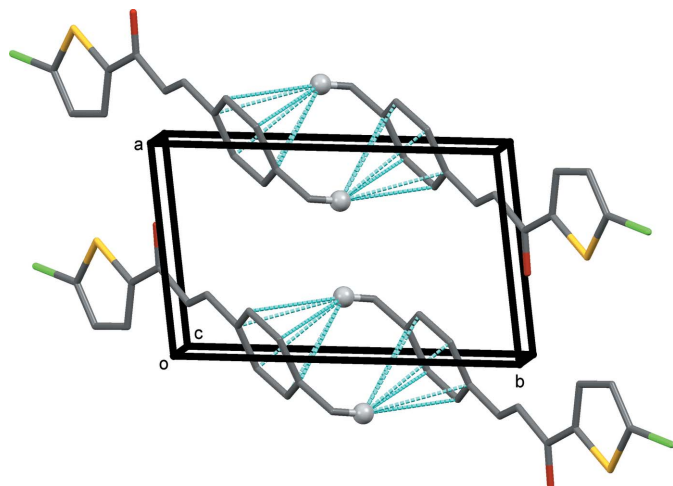
In the crystal, molecules are linked by pairs of  $C-H\cdots\pi$  interactions, forming inversion dimers (Table 1 and Fig. 2). There are no other significant intermolecular interactions present.

### Synthesis and crystallization

A mixture of 4-methylbenzaldehyde (5 mmol), 5-chloro-2-acetylthiophene (5 mmol) and sodium 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 of the reaction, the mixture was poured into ice-

**Figure 1**

The molecular structure of the title compound, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

**Figure 2**

A view along the  $c$  axis of the crystal packing of the title compound. The  $C-H\cdots\pi$  interactions (Table 1) are represented by dashed lines, and only H atom H14A (grey ball) has been included.

**Table 2**

Experimental details.

Crystal data	
Chemical formula	$C_{14}H_{11}ClOS$
$M_r$	262.75
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	293
$a, b, c$ ( $\text{\AA}$ )	6.000 (2), 9.718 (4), 10.936 (4)
$\alpha, \beta, \gamma$ ( $^\circ$ )	94.268 (17), 93.83 (3), 96.97 (2)
$V$ ( $\text{\AA}^3$ )	629.3 (4)
$Z$	2
Radiation type	Mo $K\alpha$
$\mu$ ( $\text{mm}^{-1}$ )	0.45
Crystal size (mm)	$0.39 \times 0.31 \times 0.27$
Data collection	
Diffractometer	Rigaku Saturn724+
Absorption correction	Multi-scan (NUMABS; Rigaku, 1999)
$T_{\min}, T_{\max}$	0.972, 0.976
No. of measured, independent and observed [ $I > 2\sigma(I)$ ] reflections	3635, 2808, 1279
$R_{\text{int}}$	0.071
$(\sin \theta/\lambda)_{\text{max}}$ ( $\text{\AA}^{-1}$ )	0.651
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.067, 0.264, 0.90
No. of reflections	2808
No. of parameters	155
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ ( $e \text{\AA}^{-3}$ )	0.51, $-0.65$

Computer programs: *CrystalClear* (Rigaku, 2011), *SHELXS97* and *SHELXL97* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2008).

cold water and kept in the refrigerator overnight. The solid formed was filtered, and washed with cold hydrochloric acid (5%). Block-like colourless crystals of the title compound were obtained by crystallization from methanol by the slow evaporation technique (yield 84%, m.p. 403–405 K).

### Refinement

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

### Acknowledgements

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## full crystallographic data

*IUCrData* (2017). **2**, x170038 [<https://doi.org/10.1107/S2414314617000384>]

**(*E*)-1-(5-Chlorothiophen-2-yl)-3-(*p*-tolyl)prop-2-en-1-one**

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**(*E*)-1-(5-Chlorothiophen-2-yl)-3-(*p*-tolyl)prop-2-en-1-one***Crystal data*

$C_{14}H_{11}ClOS$

$M_r = 262.75$

Triclinic,  $P\bar{1}$

Hall symbol:  $-P\ 1$

$a = 6.000\ (2)\ \text{\AA}$

$b = 9.718\ (4)\ \text{\AA}$

$c = 10.936\ (4)\ \text{\AA}$

$\alpha = 94.268\ (17)^\circ$

$\beta = 93.83\ (3)^\circ$

$\gamma = 96.97\ (2)^\circ$

$V = 629.3\ (4)\ \text{\AA}^3$

$Z = 2$

$F(000) = 272$

$D_x = 1.387\ \text{Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 3635 reflections

$\theta = 3.4\text{--}27.6^\circ$

$\mu = 0.45\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Block, colourless

$0.39 \times 0.31 \times 0.27\ \text{mm}$

*Data collection*

Rigaku Saturn724+

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution:  $18.4\ \text{pixels mm}^{-1}$

profile data from  $\omega$ -scans

Absorption correction: multi-scan

(NUMABS; Rigaku, 1999)

$T_{\min} = 0.972$ ,  $T_{\max} = 0.976$

3635 measured reflections

2808 independent reflections

1279 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.071$

$\theta_{\max} = 27.6^\circ$ ,  $\theta_{\min} = 3.4^\circ$

$h = -7 \rightarrow 7$

$k = -11 \rightarrow 12$

$l = -13 \rightarrow 14$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.067$

$wR(F^2) = 0.264$

$S = 0.90$

2808 reflections

155 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

$w = 1/[\sigma^2(F_o^2) + (0.1466P)^2]$

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

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

$\Delta\rho_{\max} = 0.51\ \text{e \AA}^{-3}$

$\Delta\rho_{\min} = -0.65\ \text{e \AA}^{-3}$

*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 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	0.6471 (3)	0.42181 (15)	−0.15570 (14)	0.0791 (6)
S1	0.5125 (2)	0.22896 (12)	0.03032 (11)	0.0490 (4)
O1	0.4218 (6)	0.0428 (4)	0.2208 (3)	0.0609 (12)
C1	0.7155 (8)	0.3040 (5)	−0.0540 (4)	0.0473 (16)
C2	0.9174 (9)	0.2628 (5)	−0.0289 (5)	0.0566 (17)
C3	0.9106 (8)	0.1657 (5)	0.0608 (4)	0.0494 (17)
C4	0.7015 (7)	0.1373 (4)	0.1020 (4)	0.0421 (14)
C5	0.6217 (8)	0.0486 (4)	0.1973 (4)	0.0435 (14)
C6	0.7863 (7)	−0.0282 (4)	0.2624 (4)	0.0415 (12)
C7	0.7370 (8)	−0.0886 (4)	0.3637 (4)	0.0423 (12)
C8	0.8780 (9)	−0.1707 (4)	0.4362 (4)	0.0463 (14)
C9	1.0772 (8)	−0.2111 (5)	0.3982 (4)	0.0475 (17)
C10	1.2012 (8)	−0.2947 (5)	0.4676 (4)	0.0499 (17)
C11	1.1293 (9)	−0.3381 (4)	0.5784 (4)	0.0485 (16)
C12	0.9276 (9)	−0.2947 (5)	0.6170 (4)	0.0516 (16)
C13	0.8017 (9)	−0.2132 (5)	0.5487 (4)	0.0525 (14)
C14	1.2604 (11)	−0.4281 (5)	0.6534 (5)	0.075 (2)
H2	1.04670	0.29450	−0.06610	0.0680*
H3	1.03520	0.12540	0.08890	0.0600*
H6	0.92600	−0.03400	0.23170	0.0500*
H7	0.59690	−0.07760	0.39200	0.0510*
H9	1.12970	−0.18200	0.32510	0.0570*
H10	1.33340	−0.32170	0.43930	0.0600*
H12	0.87730	−0.32190	0.69120	0.0620*
H13	0.66870	−0.18680	0.57640	0.0630*
H14A	1.26880	−0.51450	0.60630	0.1130*
H14B	1.18680	−0.44620	0.72680	0.1130*
H14C	1.40970	−0.38140	0.67500	0.1130*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Cl1	0.1032 (13)	0.0772 (10)	0.0688 (10)	0.0388 (9)	0.0066 (9)	0.0402 (8)
S1	0.0518 (8)	0.0535 (7)	0.0465 (7)	0.0221 (6)	0.0015 (6)	0.0126 (5)
O1	0.063 (2)	0.071 (2)	0.057 (2)	0.0232 (19)	0.0186 (19)	0.0236 (18)
C1	0.054 (3)	0.047 (2)	0.046 (3)	0.024 (2)	0.000 (2)	0.013 (2)
C2	0.048 (3)	0.066 (3)	0.060 (3)	0.009 (3)	0.011 (2)	0.024 (3)
C3	0.045 (3)	0.056 (3)	0.054 (3)	0.025 (2)	0.005 (2)	0.019 (2)
C4	0.050 (3)	0.040 (2)	0.037 (2)	0.009 (2)	0.002 (2)	0.0042 (18)

C5	0.055 (3)	0.041 (2)	0.036 (2)	0.011 (2)	0.003 (2)	0.0051 (18)
C6	0.035 (2)	0.047 (2)	0.045 (2)	0.0122 (19)	0.004 (2)	0.009 (2)
C7	0.041 (2)	0.041 (2)	0.046 (2)	0.0092 (19)	0.002 (2)	0.0063 (19)
C8	0.069 (3)	0.034 (2)	0.034 (2)	0.000 (2)	−0.002 (2)	0.0073 (17)
C9	0.049 (3)	0.052 (3)	0.045 (3)	0.014 (2)	0.006 (2)	0.013 (2)
C10	0.046 (3)	0.052 (3)	0.053 (3)	0.013 (2)	−0.003 (2)	0.009 (2)
C11	0.061 (3)	0.036 (2)	0.045 (3)	−0.002 (2)	−0.011 (2)	0.0065 (19)
C12	0.065 (3)	0.050 (3)	0.037 (2)	−0.004 (2)	−0.005 (2)	0.012 (2)
C13	0.069 (3)	0.048 (2)	0.038 (2)	−0.005 (2)	0.004 (2)	0.008 (2)
C14	0.115 (5)	0.048 (3)	0.056 (3)	−0.006 (3)	−0.031 (3)	0.018 (2)

*Geometric parameters (Å, °)*

Cl1—C1	1.720 (5)	C11—C12	1.409 (7)
S1—C1	1.703 (5)	C11—C14	1.498 (7)
S1—C4	1.710 (4)	C12—C13	1.383 (7)
O1—C5	1.239 (6)	C2—H2	0.9300
C1—C2	1.339 (7)	C3—H3	0.9300
C2—C3	1.410 (7)	C6—H6	0.9300
C3—C4	1.365 (6)	C7—H7	0.9300
C4—C5	1.469 (6)	C9—H9	0.9300
C5—C6	1.484 (6)	C10—H10	0.9300
C6—C7	1.326 (6)	C12—H12	0.9300
C7—C8	1.466 (6)	C13—H13	0.9300
C8—C9	1.383 (7)	C14—H14A	0.9600
C8—C13	1.414 (6)	C14—H14B	0.9600
C9—C10	1.397 (7)	C14—H14C	0.9600
C10—C11	1.391 (6)		
C1—S1—C4	91.1 (2)	C8—C13—C12	119.1 (5)
Cl1—C1—S1	119.3 (3)	C1—C2—H2	124.00
Cl1—C1—C2	127.7 (4)	C3—C2—H2	124.00
S1—C1—C2	113.0 (4)	C2—C3—H3	124.00
C1—C2—C3	111.9 (5)	C4—C3—H3	124.00
C2—C3—C4	112.7 (4)	C5—C6—H6	120.00
S1—C4—C3	111.2 (3)	C7—C6—H6	120.00
S1—C4—C5	117.9 (3)	C6—C7—H7	116.00
C3—C4—C5	130.9 (4)	C8—C7—H7	116.00
O1—C5—C4	118.7 (4)	C8—C9—H9	119.00
O1—C5—C6	123.1 (4)	C10—C9—H9	119.00
C4—C5—C6	118.2 (4)	C9—C10—H10	120.00
C5—C6—C7	120.9 (4)	C11—C10—H10	120.00
C6—C7—C8	127.4 (4)	C11—C12—H12	119.00
C7—C8—C9	123.2 (4)	C13—C12—H12	119.00
C7—C8—C13	117.8 (5)	C8—C13—H13	120.00
C9—C8—C13	119.0 (4)	C12—C13—H13	120.00
C8—C9—C10	121.3 (4)	C11—C14—H14A	109.00
C9—C10—C11	120.8 (4)	C11—C14—H14B	110.00

C10—C11—C12	117.4 (4)	C11—C14—H14C	109.00
C10—C11—C14	121.3 (5)	H14A—C14—H14B	109.00
C12—C11—C14	121.3 (4)	H14A—C14—H14C	109.00
C11—C12—C13	122.5 (4)	H14B—C14—H14C	110.00
C4—S1—C1—C11	−179.2 (3)	C4—C5—C6—C7	167.3 (4)
C4—S1—C1—C2	−0.4 (4)	C5—C6—C7—C8	178.3 (4)
C1—S1—C4—C3	0.1 (4)	C6—C7—C8—C9	−8.4 (7)
C1—S1—C4—C5	177.8 (3)	C6—C7—C8—C13	173.4 (4)
C11—C1—C2—C3	179.3 (4)	C7—C8—C9—C10	−177.1 (4)
S1—C1—C2—C3	0.7 (6)	C13—C8—C9—C10	1.2 (7)
C1—C2—C3—C4	−0.6 (6)	C7—C8—C13—C12	177.9 (4)
C2—C3—C4—S1	0.3 (5)	C9—C8—C13—C12	−0.5 (7)
C2—C3—C4—C5	−177.0 (4)	C8—C9—C10—C11	−1.0 (7)
S1—C4—C5—O1	0.7 (5)	C9—C10—C11—C12	0.1 (7)
S1—C4—C5—C6	−178.3 (3)	C9—C10—C11—C14	179.8 (4)
C3—C4—C5—O1	177.9 (5)	C10—C11—C12—C13	0.6 (7)
C3—C4—C5—C6	−1.1 (7)	C14—C11—C12—C13	−179.1 (5)
O1—C5—C6—C7	−11.7 (6)	C11—C12—C13—C8	−0.4 (7)

*Hydrogen-bond geometry (Å, °)*

Cg is the centroid of the C8–C13 ring.

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
C14—H14A $\cdots$ Cg <sup>i</sup>	0.96	2.77	3.576 (6)	141

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