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

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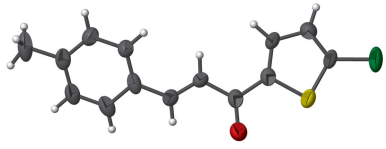
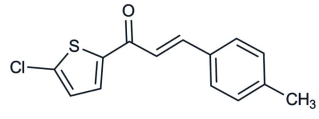
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{14}H_{11}ClOS$, 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···π 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.*, 2016*a*). Chalcones and their derivatives demonstrate a wide range of biological activities such as antidiabetic, antineoplastic, antitubercular, antiarrhythmic, hypnotic, anti-angiogenic, 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.*, 2016*b*), 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.*, 2016*b*). The *trans* conformation about the C6=C7 double bond in the

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

Cg is the centroid of the C8–C13 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14*A*···*Cg*^{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)°. 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)° and −11.7 (6)°, respectively.

In the crystal, molecules are linked by pairs of C—H···π 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-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).

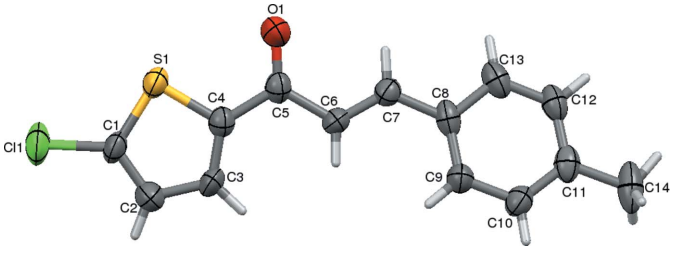


Figure 1
The molecular structure of the title compound, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

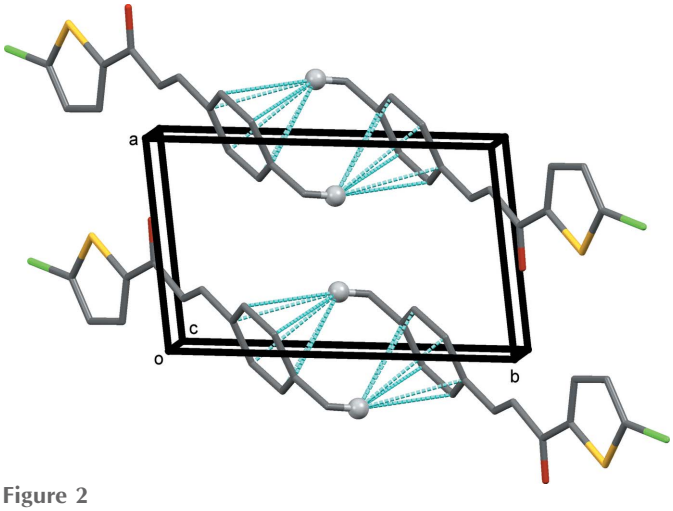


Figure 2
A view along the *c* axis of the crystal packing of the title compound. The C—H···π interactions (Table 1) are represented by dashed lines, and only H atom H14*A* (grey ball) has been included.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{14}H_{11}ClOS$
M_r	262.75
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	293
a, *b*, *c* (Å)	6.000 (2), 9.718 (4), 10.936 (4)
α, β, γ (°)	94.268 (17), 93.83 (3), 96.97 (2)
V (Å^3)	629.3 (4)
Z	2
Radiation type	Mo *K*α
μ (mm^{-1})	0.45
Crystal size (mm)	0.39 × 0.31 × 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_{int}	0.071
(sin $\theta/\lambda)_{max}$ (Å^{-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
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.51, −0.65

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

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]

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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, $P\overline{1}$
Hall symbol: -P 1
$a = 6.000$ (2) Å
$b = 9.718$ (4) Å
$c = 10.936$ (4) Å
$\alpha = 94.268$ (17)°
$\beta = 93.83$ (3)°
$\gamma = 96.97$ (2)°
$V = 629.3$ (4) Å^3
$Z = 2$
$F(000) = 272$
$D_x = 1.387$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3635 reflections
$\theta = 3.4$–$27.6°$
$\mu = 0.45$ mm^{-1}
$T = 293$ K
Block, colourless
0.39 × 0.31 × 0.27 mm

Data collection

Rigaku Saturn724+ diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 18.4 pixels mm^{-1}
profile data from ω–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 $I > 2\sigma(I)$
$R_{int} = 0.071$
$\theta_{max} = 27.6°$, $\theta_{min} = 3.4°$
$h = -7 \rightarrow 7$
$k = -11 \rightarrow 12$
$l = -13 \rightarrow 14$

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 $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.51$ e Å^{-3}
$\Delta\rho_{min} = -0.65$ 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 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 $F^2 > 2\text{sigma}(F^2)$ 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}
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 (Å²)

	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, º)

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—Cl1	−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)
Cl1—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, º)

Cg is the centroid of the C8–C13 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14*A*···*Cg*^{i}	0.96	2.77	3.576 (6)	141

Symmetry code: (i) −*x*+2, −*y*−1, −*z*+1.