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

S. Naveen,[a] M. G. Prabhudeva,[b] K. Ajay Kumar,[b] N. K. Lokanath[c]* and Muneer Abdoh[d]*

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

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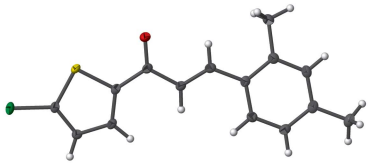
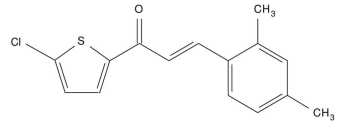
In the title compound, $C_{15}H_{13}ClOS$, the olefinic double bond adopts an *E* configuration. The molecule is nearly planar, as seen by the dihedral angle of 9.07 (8)° between the thiophene and phenyl rings. The *trans* configuration of the C═C double bond in the central enone group is confirmed by 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═C75—C8 torsion angle, 177.6 (2)°. The

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O1^{i}	0.93	2.48	3.400 (3)	169
C3—H3···S1^{i}	0.93	2.90	3.459 (2)	120

Symmetry code: (i) $x, -y+\frac{3}{2}, z+\frac{1}{2}$.

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)°] and O1—C5—C6—C7 [−4.7 (3)°] torsion angles.

In the crystal, the molecules are linked *via* weak C—H···O and C—H···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 crystals of the title compound were obtained by crystallization from methanol by the slow evaporation technique, m.p. 98–100°C.

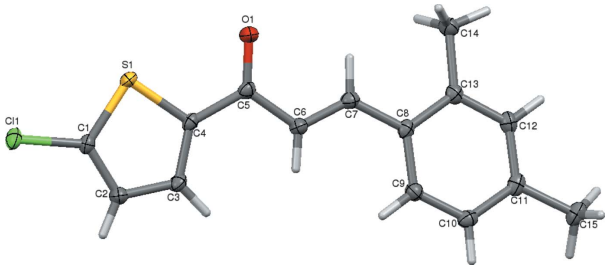


Figure 1
The molecular structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

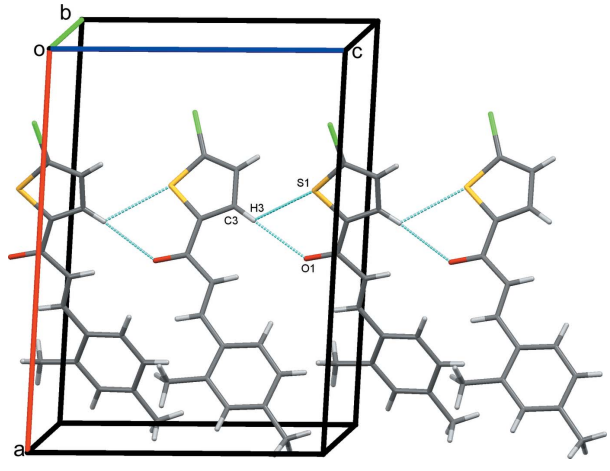


Figure 2
Packing of the molecules viewed along the *b* axis, with hydrogen bonds drawn as dashed lines.

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)
β (°)	94.293 (3)
V (Å^3)	1318.6 (3)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	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^{-1})	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^{-3})	0.50, −0.87

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

Refinement

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

Acknowledgements

The authors are grateful to the Institution of Excellence, Vijnana Bhavana, University of Mysore, India, for providing the single-crystal X-ray diffractometer facility.

References

Bruker (2013). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
Mahapatra, D. K., Asati, V. & Bharti, S. K. (2015). *Eur. J. Med. Chem.* **92**, 839–865.
Naveen, S., Dileep Kumar, A., Ajay Kumar, K., Manjunath, H. R., Lokanath, N. K. & Warad, I. (2016). *IUCrData*, **1**, x161800.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Tejkiran, P. J., Brahma Teja, M. S., Sai Siva Kumar, P., Sankar, P., Philip, R., Naveen, S., Lokanath, N. K. & Nageswara Rao, G. (2016). *J. Photochem. Photobiol. Chem.* **324**, 33–39.

full crystallographic data

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

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Crystal data

$C_{15}H_{13}ClOS$
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
β = 94.293 (3)°
V = 1318.6 (3) Å^3
Z = 4
$F(000)$ = 576
D_x = 1.394 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 2120 reflections
θ = 5.7–64.2°
μ = 3.90 mm^{-1}
T = 296 K
Rectangle, green
0.29 × 0.26 × 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^{-1}
φ and ω 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_{int} = 0.050
θ_{max} = 64.2°, θ_{min} = 5.7°
h = −18→17
k = −7→8
l = −12→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^{-3}
$\Delta\rho_{min}$ = −0.87 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	0.22467 (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 (Å²)

	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, º)

Cl1—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
Cl1—C1—S1	119.33 (13)	C3—C2—H2	124.00
Cl1—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
C4—S1—C1—Cl1	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)
Cl1—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 (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O1^{i}	0.93	2.48	3.400 (3)	169
C3—H3···S1^{i}	0.93	2.90	3.459 (2)	120

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