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(2*E*)-1-(2-Thienyl)-3-(2,3,5-trichlorophenyl)prop-2-en-1-one

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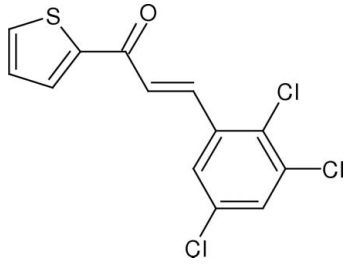
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.032; wR factor = 0.088; data-to-parameter ratio = 23.0.

The geometric parameters of the title compound, $C_{13}H_7Cl_3OS$, are in the usual ranges. The molecule is almost planar (r.m.s. deviation of all non-H atoms 0.193 Å). The dihedral angle between the two aromatic rings is 14.41 (7)°. The C=C double bond is *trans* configured. The bond between the carbonyl bond and the thienyl ring is in an eclipsed conformation. The crystal packing is stabilized by a weak C—H···O hydrogen bond.

Related literature

For related literature, see: Achanta *et al.* (2006); Anuradha *et al.* (2006); Binder *et al.* (1985); Daikonya *et al.* (2004); Gao & Ng (2006); Han *et al.* (2006); Hsu *et al.* (2006); Khatib *et al.* (2005); Kiat *et al.* (2006); Kim, Choi *et al.* (2006); Kim, Kim *et al.* (2006); Sarojini *et al.* (2007); Wirasathien *et al.* (2006); Yathirajan *et al.* (2007); Yathirajan, Ashalatha *et al.* (2006); Yathirajan, Narayana *et al.* (2006).



Experimental

Crystal data

$C_{13}H_7Cl_3OS$
M_r = 317.60
Monoclinic, $P2_1/c$
a = 16.7102 (11) Å
b = 7.5130 (4) Å
c = 10.8805 (8) Å
β = 104.395 (5)°
V = 1323.09 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.83 mm^{-1}
T = 173 (2) K
0.31 × 0.25 × 0.19 mm

Data collection

Stoe IPDSII two-circle diffractometer
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.783, T_{max} = 0.858
20538 measured reflections
3764 independent reflections
3276 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.088
S = 1.03
3764 reflections
164 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, °).

D—H···A	D—H	H···A	D···A	D—H···A
C16—H16···O1^{i}	0.95	2.45	3.3948 (17)	174

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

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

BN thanks Mangalore University for research facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2441).

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supplementary materials

Acta Cryst. (2007). E**63**, o4422-o4423 [**doi:10.1107/S1600536807051768**]

(2*E*)-1-(2-Thienyl)-3-(2,3,5-trichlorophenyl)prop-2-en-1-one

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Comment

Chalcones, 1,3-diphenyl-1-propan-3-one derivatives, have a wide range of biological properties, including anticancer (Achanta *et al.*, 2006; Kim, Choi *et al.*, 2006), antiproliferative (Hsu *et al.*, 2006), anti malarial (Wirasathien *et al.*, 2006), anti inflammatory (Anuradha *et al.*, 2006), anti-allergic (Daikonya *et al.*, 2004) and antagonist (Kim, Kim *et al.*, 2006). Chalcones exhibit inhibitory activity against nitric oxide production (Han *et al.*, 2006), dengue 2 virus NS3 protease (Ki-at *et al.*, 2006) and tyrosinase (Khatib *et al.*, 2005). Some chalcone derivatives exhibit nonlinear optical properties (Gao & Ng, 2006). Thiophene analogues of antiviral chalcones have been studied (Binder *et al.*, 1985). The crystal structures of (2E)-1-(3-bromo-2-thienyl)-3-(4-chlorophenyl)prop-2-en-1-one (Yathirajan, Ashalatha *et al.*, 2006), (2E)-1-(3-bromo-2-thienyl)-3-(4-methoxy-2,3,6-trimethylphenyl)prop-2-en-1-one (Yathirajan, Narayana *et al.*, 2006), (2E)-1-(3-methyl-2-thienyl)-3-(3-nitrophenyl)prop-2-en-1-one (Sarojini *et al.*, 2007) and (2E)-1-(3-methyl-2-thienyl)-3-(3-nitrophenyl)prop-2-en-1-one(Yathirajan *et al.*, 2007), have been reported. The structure determination of the title compound was undertaken as a part of our study on chalcones and related derivatives.

Geometric parameters of the title compound are in the usual ranges. The molecule is almost planar (r.m.s. deviation of all non-H atoms 0.193 Å). The C–C double bond is *trans* configured. The bond between the carbonyl bond and the thienyl ring is in an eclipsed conformation. The crystal packing is stabilized by a weak C—H···O hydrogen bond.

Experimental

The title compound was prepared by the Claisen-Schmidt condensation of equimolar quantities of 1-(2-thienyl)ethanone (1.26 g, 0.01 mol) and 2,3,5-trichlorobenzaldehyde (2.09 g, 0.01 mol) in ethanol (25 ml), in the presence of sodium hydroxide (8 ml, 10%) (see reaction scheme). The product was recrystallized from ethyl acetate by slow evaporation [m.p.: 378 K]. Analysis for $C_{13}H_7Cl_3OS$: Found (Calculated) C 49.10 (49.16), H 2.19 (2.22), S 10.02% (10.10%).

Refinement

All H atoms were found in a difference map, but geometrically positioned and refined with fixed individual displacement parameters [U(H) = 1.2 U_{eq}(C)] using a riding model with C—H = 0.95 Å.

Figures

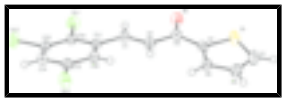

Fig. 1. Perspective view of the title compound with the atom numbering; displacement ellipsoids are at the 50% probability level.

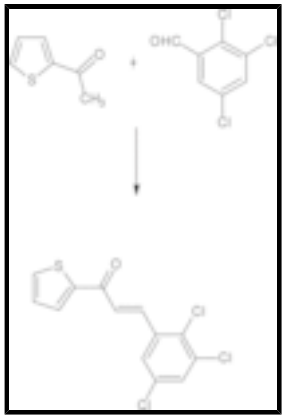

Fig. 2. The formation of the title compound.

(2E)-1-(2-Thienyl)-3-(2,3,5-trichlorophenyl)prop-2-en-1-one

Crystal data

$C_{13}H_7Cl_3OS$	F_{000} = 640
M_r = 317.60	D_x = 1.594 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 21250 reflections
a = 16.7102 (11) Å	θ = 3.8–29.8º
b = 7.5130 (4) Å	μ = 0.83 mm^{-1}
c = 10.8805 (8) Å	T = 173 (2) K
β = 104.395 (5)º	Block, colourless
V = 1323.09 (15) Å^3	0.31 × 0.25 × 0.19 mm
Z = 4	

Data collection

Stoe IPDSII two-circle diffractometer	3764 independent reflections
Radiation source: fine-focus sealed tube	3276 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.037
T = 173(2) K	θ_{max} = 29.8º
ω scans	θ_{min} = 3.7º
Absorption correction: multi-scan (MULABS; Spek, 2003; Blessing, 1995)	h = −23→23
T_{min} = 0.783, T_{max} = 0.858	k = −10→10
20538 measured reflections	l = −15→15

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.032	$w = 1/[\sigma^2(F_o^2) + (0.0505P)^2 + 0.3998P]$

	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.088$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.04$	$\Delta\rho_{max} = 0.37$ e Å^{-3}
3764 reflections	$\Delta\rho_{min} = -0.40$ e Å^{-3}
164 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0137 (17)
Secondary atom site location: difference Fourier map	

Special details

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.87602 (2)	0.58544 (6)	0.89280 (3)	0.03789 (11)
Cl2	1.01174 (2)	0.38496 (6)	0.79526 (4)	0.04403 (12)
Cl3	0.81790 (3)	0.39170 (6)	0.31918 (4)	0.04020 (12)
S1	0.42317 (2)	0.91675 (6)	0.66226 (4)	0.03423 (11)
O1	0.60078 (7)	0.86595 (15)	0.78673 (9)	0.0314 (2)
C1	0.71879 (8)	0.67660 (18)	0.69127 (12)	0.0242 (2)
H1	0.7324	0.7170	0.7767	0.029*
C2	0.64235 (8)	0.71009 (19)	0.62167 (12)	0.0251 (2)
H2	0.6267	0.6743	0.5353	0.030*
C3	0.58164 (8)	0.80322 (17)	0.67877 (12)	0.0231 (2)
C4	0.49649 (8)	0.81260 (17)	0.60132 (12)	0.0223 (2)
C5	0.46220 (8)	0.74799 (18)	0.47791 (12)	0.0242 (2)
H5	0.4923	0.6856	0.4280	0.029*
C6	0.37602 (9)	0.7891 (2)	0.43787 (15)	0.0338 (3)
H6	0.3421	0.7577	0.3570	0.041*
C7	0.34764 (10)	0.8780 (2)	0.52797 (16)	0.0359 (3)
H7	0.2918	0.9143	0.5169	0.043*
C11	0.78359 (8)	0.58274 (17)	0.64720 (12)	0.0226 (2)
C12	0.85862 (8)	0.53493 (18)	0.73275 (12)	0.0253 (2)
C13	0.91959 (8)	0.44393 (19)	0.69000 (14)	0.0283 (3)
C14	0.90810 (9)	0.39832 (19)	0.56313 (14)	0.0294 (3)
H14	0.9496	0.3364	0.5344	0.035*
C15	0.83386 (8)	0.44621 (19)	0.47927 (13)	0.0264 (3)

C16	0.77229 (8)	0.53606 (18)	0.51859 (12)	0.0243 (2)
H16	0.7223	0.5663	0.4588	0.029*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.03548 (19)	0.0486 (2)	0.02455 (17)	0.00456 (15)	−0.00205 (13)	−0.00448 (14)
Cl2	0.02560 (18)	0.0533 (3)	0.0459 (2)	0.01070 (15)	−0.00474 (15)	−0.00116 (18)
Cl3	0.0422 (2)	0.0515 (2)	0.02833 (18)	0.01138 (16)	0.01151 (14)	−0.00645 (15)
S1	0.03290 (19)	0.0422 (2)	0.03047 (18)	0.01007 (14)	0.01326 (14)	−0.00051 (14)
O1	0.0322 (5)	0.0395 (6)	0.0224 (4)	0.0022 (4)	0.0068 (4)	−0.0056 (4)
C1	0.0251 (6)	0.0262 (6)	0.0214 (5)	0.0010 (4)	0.0064 (4)	−0.0001 (4)
C2	0.0248 (6)	0.0302 (6)	0.0209 (5)	0.0021 (5)	0.0067 (4)	−0.0010 (5)
C3	0.0252 (6)	0.0243 (6)	0.0207 (5)	0.0012 (4)	0.0075 (4)	0.0020 (4)
C4	0.0243 (6)	0.0223 (6)	0.0223 (5)	0.0029 (4)	0.0093 (4)	0.0019 (4)
C5	0.0206 (5)	0.0308 (6)	0.0208 (5)	0.0025 (4)	0.0045 (4)	−0.0005 (5)
C6	0.0271 (6)	0.0386 (8)	0.0326 (7)	0.0003 (6)	0.0018 (5)	−0.0004 (6)
C7	0.0255 (6)	0.0404 (8)	0.0427 (8)	0.0060 (6)	0.0102 (6)	0.0078 (6)
C11	0.0212 (5)	0.0231 (6)	0.0231 (5)	−0.0008 (4)	0.0049 (4)	0.0018 (4)
C12	0.0233 (6)	0.0264 (6)	0.0242 (6)	−0.0013 (4)	0.0024 (5)	0.0007 (5)
C13	0.0205 (6)	0.0282 (6)	0.0337 (7)	0.0011 (5)	0.0018 (5)	0.0018 (5)
C14	0.0237 (6)	0.0292 (6)	0.0358 (7)	0.0026 (5)	0.0085 (5)	−0.0009 (5)
C15	0.0265 (6)	0.0277 (6)	0.0258 (6)	0.0008 (5)	0.0079 (5)	−0.0011 (5)
C16	0.0226 (5)	0.0268 (6)	0.0232 (5)	0.0017 (4)	0.0054 (4)	0.0015 (5)

Geometric parameters (Å, °)

Cl1—C12	1.7348 (14)	C5—C6	1.4308 (19)
Cl2—C13	1.7327 (14)	C5—H5	0.9500
Cl3—C15	1.7441 (14)	C6—C7	1.365 (2)
S1—C7	1.7016 (18)	C6—H6	0.9500
S1—C4	1.7196 (13)	C7—H7	0.9500
O1—C3	1.2318 (16)	C11—C16	1.4090 (18)
C1—C2	1.3370 (18)	C11—C12	1.4096 (17)
C1—C11	1.4691 (18)	C12—C13	1.3990 (19)
C1—H1	0.9500	C13—C14	1.389 (2)
C2—C3	1.4893 (18)	C14—C15	1.3921 (19)
C2—H2	0.9500	C14—H14	0.9500
C3—C4	1.4641 (18)	C15—C16	1.3844 (18)
C4—C5	1.4087 (17)	C16—H16	0.9500
C7—S1—C4	92.21 (7)	C6—C7—H7	123.7
C2—C1—C11	125.69 (12)	S1—C7—H7	123.7
C2—C1—H1	117.2	C16—C11—C12	118.04 (12)
C11—C1—H1	117.2	C16—C11—C1	121.06 (11)
C1—C2—C3	120.40 (12)	C12—C11—C1	120.90 (12)
C1—C2—H2	119.8	C13—C12—C11	120.44 (12)
C3—C2—H2	119.8	C13—C12—Cl1	119.11 (10)
O1—C3—C4	121.01 (12)	C11—C12—Cl1	120.44 (10)

O1—C3—C2	122.36 (12)	C14—C13—C12	121.27 (12)
C4—C3—C2	116.60 (11)	C14—C13—Cl2	118.33 (11)
C5—C4—C3	129.70 (11)	C12—C13—Cl2	120.40 (11)
C5—C4—S1	111.37 (9)	C13—C14—C15	117.92 (13)
C3—C4—S1	118.92 (9)	C13—C14—H14	121.0
C4—C5—C6	110.85 (12)	C15—C14—H14	121.0
C4—C5—H5	124.6	C16—C15—C14	122.22 (13)
C6—C5—H5	124.6	C16—C15—Cl3	118.83 (10)
C7—C6—C5	112.95 (13)	C14—C15—Cl3	118.95 (11)
C7—C6—H6	123.5	C15—C16—C11	120.12 (12)
C5—C6—H6	123.5	C15—C16—H16	119.9
C6—C7—S1	112.61 (11)	C11—C16—H16	119.9
C11—C1—C2—C3	178.59 (12)	C16—C11—C12—C13	0.05 (19)
C1—C2—C3—O1	6.4 (2)	C1—C11—C12—C13	179.34 (12)
C1—C2—C3—C4	−171.87 (12)	C16—C11—C12—Cl1	−179.07 (10)
O1—C3—C4—C5	−179.18 (14)	C1—C11—C12—Cl1	0.22 (18)
C2—C3—C4—C5	−0.9 (2)	C11—C12—C13—C14	0.0 (2)
O1—C3—C4—S1	1.18 (18)	Cl1—C12—C13—C14	179.10 (11)
C2—C3—C4—S1	179.47 (9)	C11—C12—C13—Cl2	−179.92 (10)
C7—S1—C4—C5	−0.21 (11)	Cl1—C12—C13—Cl2	−0.79 (17)
C7—S1—C4—C3	179.49 (11)	C12—C13—C14—C15	0.1 (2)
C3—C4—C5—C6	−179.12 (13)	Cl2—C13—C14—C15	179.98 (11)
S1—C4—C5—C6	0.54 (15)	C13—C14—C15—C16	−0.2 (2)
C4—C5—C6—C7	−0.70 (19)	C13—C14—C15—Cl3	179.67 (11)
C5—C6—C7—S1	0.55 (19)	C14—C15—C16—C11	0.2 (2)
C4—S1—C7—C6	−0.19 (13)	Cl3—C15—C16—C11	−179.66 (10)
C2—C1—C11—C16	7.3 (2)	C12—C11—C16—C15	−0.13 (19)
C2—C1—C11—C12	−171.96 (13)	C1—C11—C16—C15	−179.42 (12)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C16—H16···O1^{i}	0.95	2.45	3.3948 (17)	174

Symmetry codes: (i) *x*, −*y*+3/2, *z*−1/2.

Fig. 1

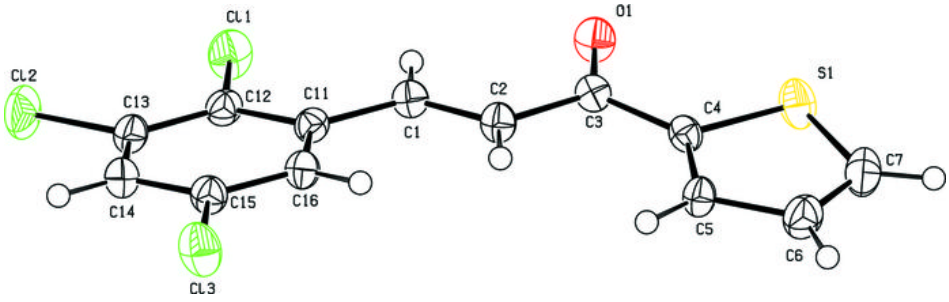

Fig. 2