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(*E*)-1-(2,5-Dichloro-3-thienyl)-3-(3,4-dimethoxyphenyl)prop-2-en-1-one

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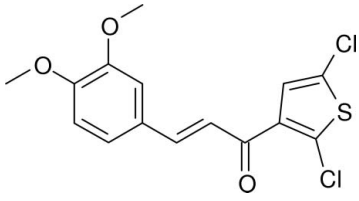
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.004 Å; R factor = 0.048; wR factor = 0.113; data-to-parameter ratio = 17.7.

In the title compound, $C_{15}H_{12}Cl_2O_3S$, the prop-2-en-1-one (enone) fragment is almost planar [C—C—C—O = 2.2 (4)°] and it subtends dihedral angles of 11.9 (2) and 11.0 (2)° with the thiophene and benzene rings, respectively. The dihedral angle between the aromatic rings is 3.47 (16)°. In the crystal, weak C—H···O and C—H···Cl interactions link the molecules, leading to $R_2^2(14)$, $R_2^2(24)$ and $C(11)$ supramolecular motifs occurring within the three-dimensional network. Weak aromatic π–π stacking [centroid–centroid separations = 3.6823 (15) and 3.8722 (15) Å] may also help to consolidate the packing.

Related literature

For a related structure and background references, see: Jasinski *et al.* (2010). For reference structural data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{15}H_{12}Cl_2O_3S$ M_r = 343.21
Monoclinic, $P2_1/n$
a = 8.9331 (2) Å
b = 8.9997 (2) Å
c = 18.8210 (5) Å
β = 100.181 (1)°
V = 1489.29 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.58 mm^{-1}
T = 120 K
0.24 × 0.12 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan [*SADABS* (Bruker, 2003) and Blessing (1995)]
T_{min} = 0.873, T_{max} = 0.944
22032 measured reflections
3424 independent reflections
2834 reflections with $I > 2\sigma(I)$
R_{int} = 0.056

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.113
S = 1.10
3424 reflections
193 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.72 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O3^{i}	0.95	2.53	3.227 (3)	130
C12—H12···O1^{ii}	0.95	2.55	3.441 (3)	157
C14—H14A···O3^{iii}	0.98	2.53	3.474 (3)	161
C15—H15B···Cl1^{iv}	0.98	2.82	3.647 (3)	142

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$; (iii) $x+\frac{1}{2}, -y+\frac{1}{2}, z+\frac{1}{2}$; (iv) $-x, -y+1, -z+1$.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *COLLECT*; data reduction: *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG5014).

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supporting information

Acta Cryst. (2010). E**66**, o2479 [doi:10.1107/S1600536810035142]

(*E*)-1-(2,5-Dichloro-3-thienyl)-3-(3,4-dimethoxyphenyl)prop-2-en-1-one

William T. A. Harrison, C. S. Chidan Kumar, H. S. Yathirajan, A. N. Mayekar and B. Narayana

S1. Comment

The title compound, (I), (Fig. 1), was prepared as part of our ongoing structural studies (Jasinski *et al.*, 2010) of chalcone-like compounds, in which substituted aromatic ring systems are linked by a prop-2-en-1-one bridge.

The prop-2-en-1-one fragment in (I) is almost planar [C7—C8—C9—O3 = 2.2 (4)°] and it subtends dihedral angles of 11.9 (2) and 11.0 (2)° with the thiophene and benzene rings, respectively. The dihedral angle between the aromatic rings is 3.47 (16)°. The carbon atoms of the methoxy groups are close to co-planar with their attached benzene ring [displacements of 0.033 (5) and 0.100 (5)Å for C14 and C15, respectively]. Otherwise, the bond lengths for (I) fall within their expected ranges (Allen *et al.*, 1987) and are similar to those in a related structure (Jasinski *et al.*, 2010).

In the crystal, three weak C—H···O and one C—H···Cl interactions (Table 1) link the molecules. Considered individually, they generate the following motifs: the C3—H3 bond generates inversion dimers containing $R_2^2(14)$ rings, whereas the C12—H12 bond leads to C(11) chains propagating in [010]. The methyl-H bonds lead to inversion-generated $R_2^2(24)$ loops (for C15—H15B) and C(11) chains (for C14—H14A). Taken together, these four interactions generate a three-dimensional network. Weak aromatic π-π stacking [centroid-centroid separations = 3.6823 (15) and 3.8722 (15) Å] may also help to consolidate the packing.

S2. Experimental

2,5-Dichloro-3-acetylthiophene was obtained as a gift sample from SeQuent Scientific ltd., New Mangalore, India. 1-(2,5-Dichlorothiophen-3-yl)ethanone (1.95 g, 0.01 mol) was mixed with 3,4-dimethoxybenzaldehyde (1.66 g, 0.01 mol) and dissolved in ethanol (30 ml). To this, 3 ml of 50% KOH was added. The reaction mixture was stirred for 6 h. The resulting crude solid was filtered, washed successively with distilled water and finally recrystallized from ethanol (95%) to give the pure chalcone. Irregular yellow crystals of (I) were obtained by the slow evaporation of DMF solution (m.p.: 389–391 K).

S3. Refinement

The hydrogen atoms were geometrically placed (C—H = 0.95–0.98 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). A rotating group model was applied to the methyl group.

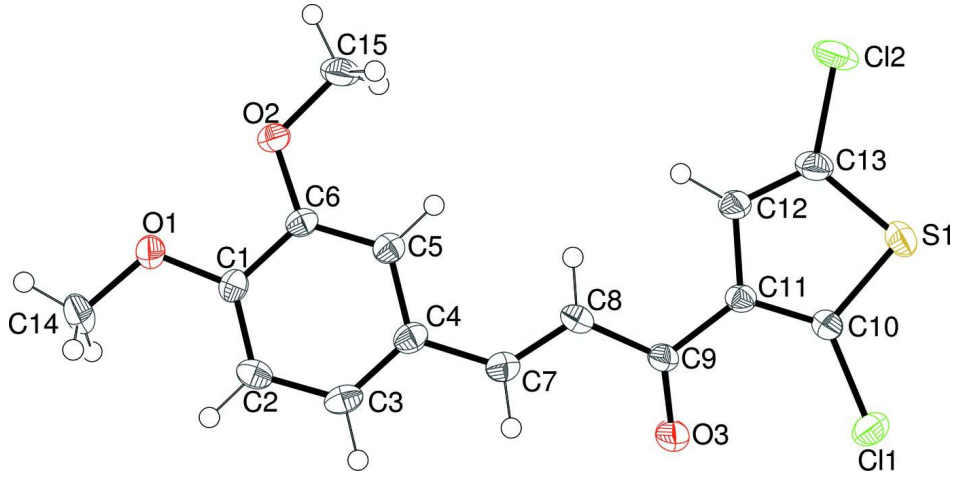

Figure 1

View of the molecular structure of (I) showing 50% displacement ellipsoids (arbitrary spheres for the H atoms).

(*E*)-1-(2,5-Dichloro-3-thienyl)-3-(3,4-dimethoxyphenyl)prop-2-en-1-one

Crystal data

$C_{15}H_{12}Cl_2O_3S$
$M_r = 343.21$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 8.9331\ (2)$ Å
$b = 8.9997\ (2)$ Å
$c = 18.8210\ (5)$ Å
$\beta = 100.181\ (1)°$
$V = 1489.29\ (6)$ Å^3
$Z = 4$
$F(000) = 704$
$D_x = 1.531$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 20728 reflections
$\theta = 2.9–27.5°$
$\mu = 0.58$ mm^{-1}
$T = 120$ K
Fragment, yellow
0.24 × 0.12 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan [*SADABS* (Bruker, 2003) and Blessing (1995)]
$T_{min} = 0.873$, $T_{max} = 0.944$
22032 measured reflections
3424 independent reflections
2834 reflections with $I > 2\sigma(I)$
$R_{int} = 0.056$
$\theta_{max} = 27.6°$, $\theta_{min} = 3.2°$
$h = -11\rightarrow11$
$k = -11\rightarrow11$
$l = -24\rightarrow24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.113$
$S = 1.10$
3424 reflections
193 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
$w = 1/[\sigma^2(F_o^2) + (0.034P)^2 + 1.9239P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.72$ e Å^{-3}
$\Delta\rho_{min} = -0.41$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.011 (2)

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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.4943 (3)	0.2226 (3)	0.72975 (12)	0.0218 (5)
C2	0.5681 (3)	0.2506 (3)	0.67246 (14)	0.0253 (5)
H2	0.6632	0.2050	0.6705	0.030*
C3	0.5013 (3)	0.3471 (3)	0.61723 (14)	0.0273 (5)
H3	0.5512	0.3644	0.5774	0.033*
C4	0.3644 (3)	0.4175 (3)	0.61939 (13)	0.0248 (5)
C5	0.2908 (3)	0.3901 (3)	0.67817 (13)	0.0227 (5)
H5	0.1977	0.4388	0.6809	0.027*
C6	0.3533 (3)	0.2927 (3)	0.73192 (12)	0.0207 (5)
C7	0.2948 (3)	0.5123 (3)	0.55890 (14)	0.0269 (5)
H7	0.3459	0.5142	0.5187	0.032*
C8	0.1686 (3)	0.5971 (3)	0.55235 (13)	0.0232 (5)
H8	0.1154	0.6027	0.5918	0.028*
C9	0.1115 (3)	0.6799 (3)	0.48719 (13)	0.0230 (5)
C10	−0.0865 (3)	0.8706 (3)	0.43372 (13)	0.0217 (5)
C11	−0.0291 (3)	0.7698 (3)	0.48589 (12)	0.0213 (5)
C12	−0.1241 (3)	0.7591 (3)	0.53941 (13)	0.0265 (5)
H12	−0.1035	0.6955	0.5803	0.032*
C13	−0.2457 (3)	0.8490 (3)	0.52538 (14)	0.0298 (6)
C14	0.6852 (3)	0.0533 (3)	0.78546 (16)	0.0382 (7)
H14A	0.7073	−0.0128	0.8274	0.057*
H14B	0.7672	0.1264	0.7875	0.057*
H14C	0.6776	−0.0053	0.7411	0.057*
C15	0.1441 (3)	0.3186 (3)	0.79360 (15)	0.0317 (6)
H15A	0.1087	0.2819	0.8368	0.047*
H15B	0.0709	0.2905	0.7505	0.047*
H15C	0.1532	0.4270	0.7960	0.047*
O1	0.5440 (2)	0.1289 (2)	0.78610 (9)	0.0284 (4)
O2	0.28886 (19)	0.25494 (19)	0.78990 (9)	0.0265 (4)
O3	0.1727 (2)	0.6738 (2)	0.43354 (10)	0.0417 (5)
S1	−0.25196 (7)	0.95280 (7)	0.44816 (4)	0.02811 (19)
Cl1	−0.01562 (8)	0.92675 (7)	0.35926 (3)	0.03212 (19)
Cl2	−0.38786 (8)	0.86816 (11)	0.57543 (4)	0.0513 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0237 (11)	0.0212 (12)	0.0189 (11)	−0.0020 (9)	−0.0004 (9)	−0.0012 (9)
C2	0.0203 (11)	0.0268 (13)	0.0298 (13)	−0.0032 (10)	0.0076 (10)	−0.0045 (10)
C3	0.0309 (13)	0.0281 (13)	0.0250 (13)	−0.0083 (11)	0.0105 (10)	0.0000 (10)
C4	0.0299 (13)	0.0216 (12)	0.0238 (12)	−0.0040 (10)	0.0073 (10)	0.0007 (10)
C5	0.0266 (12)	0.0204 (12)	0.0213 (12)	−0.0007 (9)	0.0050 (10)	−0.0023 (9)
C6	0.0256 (11)	0.0190 (11)	0.0182 (11)	−0.0033 (9)	0.0056 (9)	−0.0021 (9)
C7	0.0264 (12)	0.0298 (14)	0.0260 (13)	−0.0011 (10)	0.0084 (10)	0.0025 (10)
C8	0.0204 (11)	0.0237 (12)	0.0267 (12)	0.0006 (9)	0.0074 (10)	−0.0039 (10)
C9	0.0220 (11)	0.0271 (13)	0.0208 (12)	0.0034 (10)	0.0062 (9)	−0.0009 (10)
C10	0.0195 (11)	0.0242 (12)	0.0211 (12)	0.0005 (9)	0.0025 (9)	−0.0032 (9)
C11	0.0213 (11)	0.0247 (12)	0.0180 (11)	0.0003 (9)	0.0040 (9)	−0.0030 (9)
C12	0.0261 (12)	0.0357 (14)	0.0181 (12)	0.0048 (11)	0.0051 (10)	−0.0003 (10)
C13	0.0265 (12)	0.0410 (15)	0.0232 (13)	0.0034 (11)	0.0077 (10)	−0.0050 (11)
C14	0.0296 (14)	0.0439 (17)	0.0400 (16)	0.0158 (12)	0.0033 (12)	0.0039 (13)
C15	0.0320 (13)	0.0358 (15)	0.0307 (14)	0.0038 (11)	0.0152 (11)	−0.0037 (11)
O1	0.0297 (9)	0.0313 (10)	0.0242 (9)	0.0095 (8)	0.0045 (7)	0.0036 (7)
O2	0.0308 (9)	0.0277 (9)	0.0230 (9)	0.0043 (7)	0.0106 (7)	0.0036 (7)
O3	0.0426 (11)	0.0582 (14)	0.0277 (10)	0.0238 (10)	0.0159 (9)	0.0103 (9)
S1	0.0233 (3)	0.0303 (4)	0.0296 (3)	0.0064 (3)	0.0016 (2)	−0.0019 (3)
Cl1	0.0378 (4)	0.0324 (4)	0.0278 (3)	−0.0019 (3)	0.0102 (3)	0.0059 (3)
Cl2	0.0363 (4)	0.0820 (6)	0.0411 (4)	0.0219 (4)	0.0215 (3)	0.0036 (4)

Geometric parameters (Å, º)

C1—O1	1.367 (3)	C9—C11	1.491 (3)
C1—C2	1.383 (3)	C10—C11	1.368 (3)
C1—C6	1.415 (3)	C10—Cl1	1.713 (2)
C2—C3	1.404 (4)	C10—S1	1.717 (2)
C2—H2	0.9500	C11—C12	1.430 (3)
C3—C4	1.385 (4)	C12—C13	1.343 (4)
C3—H3	0.9500	C12—H12	0.9500
C4—C5	1.405 (3)	C13—Cl2	1.718 (3)
C4—C7	1.470 (3)	C13—S1	1.720 (3)
C5—C6	1.380 (3)	C14—O1	1.435 (3)
C5—H5	0.9500	C14—H14A	0.9800
C6—O2	1.364 (3)	C14—H14B	0.9800
C7—C8	1.348 (3)	C14—H14C	0.9800
C7—H7	0.9500	C15—O2	1.427 (3)
C8—C9	1.448 (3)	C15—H15A	0.9800
C8—H8	0.9500	C15—H15B	0.9800
C9—O3	1.231 (3)	C15—H15C	0.9800
O1—C1—C2	125.7 (2)	C11—C10—Cl1	129.58 (18)
O1—C1—C6	114.9 (2)	C11—C10—S1	113.31 (18)
C2—C1—C6	119.4 (2)	Cl1—C10—S1	117.10 (14)

C1—C2—C3	119.4 (2)	C10—C11—C12	110.9 (2)
C1—C2—H2	120.3	C10—C11—C9	125.4 (2)
C3—C2—H2	120.3	C12—C11—C9	123.7 (2)
C4—C3—C2	121.5 (2)	C13—C12—C11	112.4 (2)
C4—C3—H3	119.2	C13—C12—H12	123.8
C2—C3—H3	119.2	C11—C12—H12	123.8
C3—C4—C5	118.8 (2)	C12—C13—Cl2	127.1 (2)
C3—C4—C7	119.9 (2)	C12—C13—S1	113.43 (19)
C5—C4—C7	121.3 (2)	Cl2—C13—S1	119.47 (16)
C6—C5—C4	120.3 (2)	O1—C14—H14A	109.5
C6—C5—H5	119.9	O1—C14—H14B	109.5
C4—C5—H5	119.9	H14A—C14—H14B	109.5
O2—C6—C5	124.9 (2)	O1—C14—H14C	109.5
O2—C6—C1	114.6 (2)	H14A—C14—H14C	109.5
C5—C6—C1	120.6 (2)	H14B—C14—H14C	109.5
C8—C7—C4	129.2 (2)	O2—C15—H15A	109.5
C8—C7—H7	115.4	O2—C15—H15B	109.5
C4—C7—H7	115.4	H15A—C15—H15B	109.5
C7—C8—C9	122.1 (2)	O2—C15—H15C	109.5
C7—C8—H8	118.9	H15A—C15—H15C	109.5
C9—C8—H8	118.9	H15B—C15—H15C	109.5
O3—C9—C8	122.1 (2)	C1—O1—C14	116.9 (2)
O3—C9—C11	120.4 (2)	C6—O2—C15	116.97 (19)
C8—C9—C11	117.5 (2)	C10—S1—C13	89.97 (12)

O1—C1—C2—C3	178.3 (2)	S1—C10—C11—C12	0.2 (3)
C6—C1—C2—C3	−0.5 (4)	Cl1—C10—C11—C9	−2.3 (4)
C1—C2—C3—C4	1.3 (4)	S1—C10—C11—C9	179.10 (19)
C2—C3—C4—C5	−0.5 (4)	O3—C9—C11—C10	−12.1 (4)
C2—C3—C4—C7	−177.2 (2)	C8—C9—C11—C10	170.1 (2)
C3—C4—C5—C6	−1.1 (4)	O3—C9—C11—C12	166.6 (3)
C7—C4—C5—C6	175.6 (2)	C8—C9—C11—C12	−11.1 (4)
C4—C5—C6—O2	−177.8 (2)	C10—C11—C12—C13	0.4 (3)
C4—C5—C6—C1	1.9 (4)	C9—C11—C12—C13	−178.6 (2)
O1—C1—C6—O2	−0.3 (3)	C11—C12—C13—Cl2	179.3 (2)
C2—C1—C6—O2	178.7 (2)	C11—C12—C13—S1	−0.8 (3)
O1—C1—C6—C5	179.9 (2)	C2—C1—O1—C14	0.1 (4)
C2—C1—C6—C5	−1.1 (3)	C6—C1—O1—C14	178.9 (2)
C3—C4—C7—C8	−175.4 (3)	C5—C6—O2—C15	1.8 (3)
C5—C4—C7—C8	8.0 (4)	C1—C6—O2—C15	−178.0 (2)
C4—C7—C8—C9	−177.4 (2)	C11—C10—S1—C13	−0.5 (2)
C7—C8—C9—O3	2.2 (4)	Cl1—C10—S1—C13	−179.29 (16)
C7—C8—C9—C11	179.9 (2)	C12—C13—S1—C10	0.7 (2)
Cl1—C10—C11—C12	178.78 (19)	Cl2—C13—S1—C10	−179.34 (18)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O3^{i}	0.95	2.53	3.227 (3)	130
C12—H12···O1^{ii}	0.95	2.55	3.441 (3)	157
C14—H14*A*···O3^{iii}	0.98	2.53	3.474 (3)	161
C15—H15*B*···Cl1^{iv}	0.98	2.82	3.647 (3)	142

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