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1-(2,5-Dichloro-3-thienyl)ethanone: infinite sheets mediated by O···Cl halogen bonds

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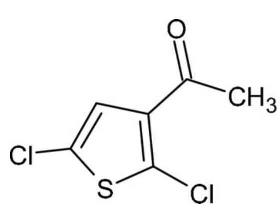
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.002 Å; R factor = 0.031; wR factor = 0.078; data-to-parameter ratio = 19.1.

In the title compound, $C_6H_4Cl_2OS$, the acetyl group is almost coplanar with the thiophene ring [dihedral angle = 4.01 (2)°]. In the crystal, short intermolecular O···Cl contacts [2.9494 (14) and 3.1191 (14) Å] link the molecules into infinite (100) sheets and aromatic π–π stacking [centroid–centroid separation = 3.5422 (10) Å] consolidates the packing.

Related literature

For a related structure and background references, see: Jasinski *et al.* (2010). For a related structure, see: Wen & Rasmussen (2007). For reference structural data, see: Allen *et al.* (1987). For a discussion of halogen bonding, see: Metrangalo & Resnati (2001).



Experimental

Crystal data

$C_6H_4Cl_2OS$	V = 1535.42 (5) Å^3
M_r = 195.05	Z = 8
Orthorhombic, *Pbca*	Mo $K\alpha$ radiation
a = 13.0980 (3) Å	μ = 1.04 mm^{-1}
b = 7.1790 (1) Å	T = 120 K
c = 16.3290 (3) Å	0.22 × 0.14 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer	12928 measured reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)	1758 independent reflections
T_{min} = 0.804, T_{max} = 0.922	1538 reflections with $I > 2\sigma(I)$
	R_{int} = 0.050

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031	92 parameters
$wR(F^2)$ = 0.078	H-atom parameters constrained
S = 1.07	$\Delta\rho_{max}$ = 0.34 e Å^{-3}
1758 reflections	$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor 1997), *SCALEPACK* and *SORTAV* (Blessing, 1995); 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: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JJ2050).

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supplementary materials

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1-(2,5-Dichloro-3-thienyl)ethanone: infinite sheets mediated by O...Cl halogen bonds

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Comment

The structure of the title compound, (I), (Fig. 1), was determined as part of our ongoing studies (Jasinski *et al.*, 2010) of thiophene derivatives as possible candidates for non-linear optical materials.

The five-membered ring in (I) is almost planar (r.m.s. deviation = 0.002 Å) and the pendant atoms deviate from the ring plane by 0.019 (1)Å (Cl1), 0.011 (1)Å (Cl2), 0.110 (1)Å (O1), 0.026 (2)Å (C5) and -0.045(20Å (C6). The dihedral angle between C1/C2/C3/C4/S1 and C5/C6/O1 is 4.01 (2)°. Otherwise, the bond lengths for (I) fall within their expected ranges (Allen *et al.*, 1987) and are similar to those in related structures (Wen & Rasmussen, 2007).

In the crystal of (I), short O···Cl contacts of 2.9494 (14)Å and 3.1191 (14)Å are evident, compared to an expected van der Waals' separation of about 3.27Å for these atoms. If theses contacts are considered to be bonding interactions (Metrangalo & Resnati, 2001), then infinite (100) sheets result (Fig. 2). Seventeen-membered rings containining four O···Cl bonds are apparent within the sheet. The packing is consolidated by aromatic π-π stacking interactions, with a centroid–centroid separation of 3.5422 (10)Å between inversion-related thiophene rings.

Experimental

2,5-Dichloro-3-acetylthiophene was obtained as a gift sample from SeQuent Scientific Ltd., New Mangalore, India. Colourless blocks of (I) were grown by the slow evaporation of a methanol solution (M.P.: 314–316 K).

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 rigid-group model was applied to the methyl group.

Figures

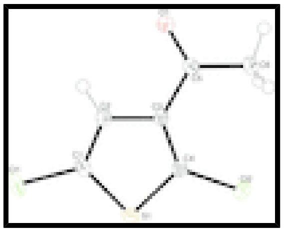

Fig. 1. View of the molecular structure of (I) showing 50% displacement ellipsoids (arbitrary spheres for the H atoms).

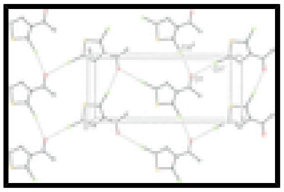

Fig. 2. Partial packing diagram for (I) showing part of a (100) sheet arising from short O···Cl contacts (H atoms omitted for clarity). Symmetry codes: (i) *x*, 3/2–y, 1/2 + *z*; (ii) *x*, 1 + *y*, *z*.

1-(2,5-Dichloro-3-thienyl)ethanone

Crystal data

$C_6H_4Cl_2OS$	$F(000) = 784$
$M_r = 195.05$	$D_x = 1.688$ Mg m^{-3}
Orthorhombic, *Pbca*	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ac 2ab	Cell parameters from 12696 reflections
$a = 13.0980$ (3) Å	$\theta = 2.9$–$27.5°$
$b = 7.1790$ (1) Å	$\mu = 1.04$ mm^{-1}
$c = 16.3290$ (3) Å	$T = 120$ K
$V = 1535.42$ (5) Å^3	Cut block, colourless
$Z = 8$	0.22 × 0.14 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer	1758 independent reflections
Radiation source: fine-focus sealed tube	1538 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.050$
ω and φ scans	$\theta_{max} = 27.5°$, $\theta_{min} = 2.9°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)	$h = -17 \rightarrow 17$
$T_{min} = 0.804$, $T_{max} = 0.922$	$k = -9 \rightarrow 9$
12928 measured reflections	$l = -21 \rightarrow 19$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.031$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.078$	H-atom parameters constrained
$S = 1.07$	$w = 1/[\sigma^2(F_o^2) + (0.0196P)^2 + 1.1699P]$ where $P = (F_o^2 + 2F_c^2)/3$
1758 reflections	$(\Delta/\sigma)_{max} = 0.002$
92 parameters	$\Delta\rho_{max} = 0.34$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.28$ e Å^{-3}

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.36993 (13)	0.4974 (3)	0.44792 (10)	0.0181 (4)
C2	0.36763 (13)	0.5824 (2)	0.52170 (11)	0.0178 (4)
H2	0.3618	0.7134	0.5285	0.021*
C3	0.37494 (12)	0.4537 (2)	0.58881 (10)	0.0153 (4)
C4	0.38165 (13)	0.2736 (2)	0.56061 (11)	0.0172 (4)
C5	0.37563 (13)	0.5245 (3)	0.67461 (11)	0.0181 (4)
C6	0.37733 (14)	0.3927 (3)	0.74569 (11)	0.0227 (4)
H6A	0.3730	0.4634	0.7969	0.034*
H6B	0.4410	0.3210	0.7448	0.034*
H6C	0.3191	0.3074	0.7418	0.034*
O1	0.37515 (11)	0.69263 (19)	0.68546 (8)	0.0299 (3)
S1	0.38011 (3)	0.25777 (6)	0.45563 (3)	0.02062 (14)
Cl1	0.36483 (4)	0.60020 (7)	0.35316 (3)	0.02524 (14)
Cl2	0.39243 (4)	0.06821 (6)	0.61358 (3)	0.02400 (14)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0175 (9)	0.0234 (10)	0.0134 (9)	−0.0004 (7)	−0.0011 (6)	0.0017 (7)
C2	0.0191 (9)	0.0176 (8)	0.0168 (9)	0.0001 (7)	0.0000 (7)	0.0017 (7)
C3	0.0154 (8)	0.0162 (8)	0.0144 (9)	0.0004 (6)	0.0010 (6)	0.0010 (7)
C4	0.0174 (9)	0.0179 (8)	0.0163 (8)	−0.0007 (7)	0.0008 (7)	0.0006 (7)
C5	0.0190 (9)	0.0198 (8)	0.0154 (9)	0.0003 (7)	0.0005 (6)	−0.0007 (7)
C6	0.0310 (11)	0.0227 (10)	0.0145 (10)	0.0001 (7)	0.0021 (7)	0.0022 (7)
O1	0.0548 (10)	0.0174 (7)	0.0175 (7)	0.0006 (6)	−0.0007 (6)	−0.0024 (6)
S1	0.0244 (3)	0.0215 (3)	0.0160 (3)	−0.00193 (17)	0.00004 (17)	−0.00486 (17)
Cl1	0.0251 (3)	0.0372 (3)	0.0134 (2)	0.00171 (19)	−0.00100 (16)	0.00583 (18)
Cl2	0.0332 (3)	0.0138 (2)	0.0250 (3)	0.00051 (17)	−0.00035 (19)	0.00208 (17)

Geometric parameters (Å, °)

C1—C2	1.351 (2)	C4—Cl2	1.7151 (18)
C1—Cl1	1.7156 (18)	C4—S1	1.7181 (19)

C1—S1	1.7303 (19)	C5—O1	1.220 (2)
C2—C3	1.437 (2)	C5—C6	1.498 (2)
C2—H2	0.9500	C6—H6A	0.9800
C3—C4	1.376 (2)	C6—H6B	0.9800
C3—C5	1.490 (2)	C6—H6C	0.9800

C2—C1—Cl1	127.54 (15)	Cl2—C4—S1	116.58 (10)
C2—C1—S1	112.71 (13)	O1—C5—C3	118.28 (16)
Cl1—C1—S1	119.75 (10)	O1—C5—C6	120.83 (16)
C1—C2—C3	112.84 (16)	C3—C5—C6	120.88 (16)
C1—C2—H2	123.6	C5—C6—H6A	109.5
C3—C2—H2	123.6	C5—C6—H6B	109.5
C4—C3—C2	110.71 (15)	H6A—C6—H6B	109.5
C4—C3—C5	129.40 (16)	C5—C6—H6C	109.5
C2—C3—C5	119.88 (15)	H6A—C6—H6C	109.5
C3—C4—Cl2	130.13 (14)	H6B—C6—H6C	109.5
C3—C4—S1	113.28 (13)	C4—S1—C1	90.45 (8)

Cl1—C1—C2—C3	−179.10 (13)	C4—C3—C5—O1	175.49 (17)
S1—C1—C2—C3	0.4 (2)	C2—C3—C5—O1	−3.7 (3)
C1—C2—C3—C4	−0.5 (2)	C4—C3—C5—C6	−4.2 (3)
C1—C2—C3—C5	178.80 (15)	C2—C3—C5—C6	176.57 (15)
C2—C3—C4—Cl2	179.62 (14)	C3—C4—S1—C1	−0.14 (14)
C5—C3—C4—Cl2	0.4 (3)	Cl2—C4—S1—C1	−179.47 (11)
C2—C3—C4—S1	0.40 (19)	C2—C1—S1—C4	−0.18 (14)
C5—C3—C4—S1	−178.85 (14)	Cl1—C1—S1—C4	179.40 (11)

Fig. 1

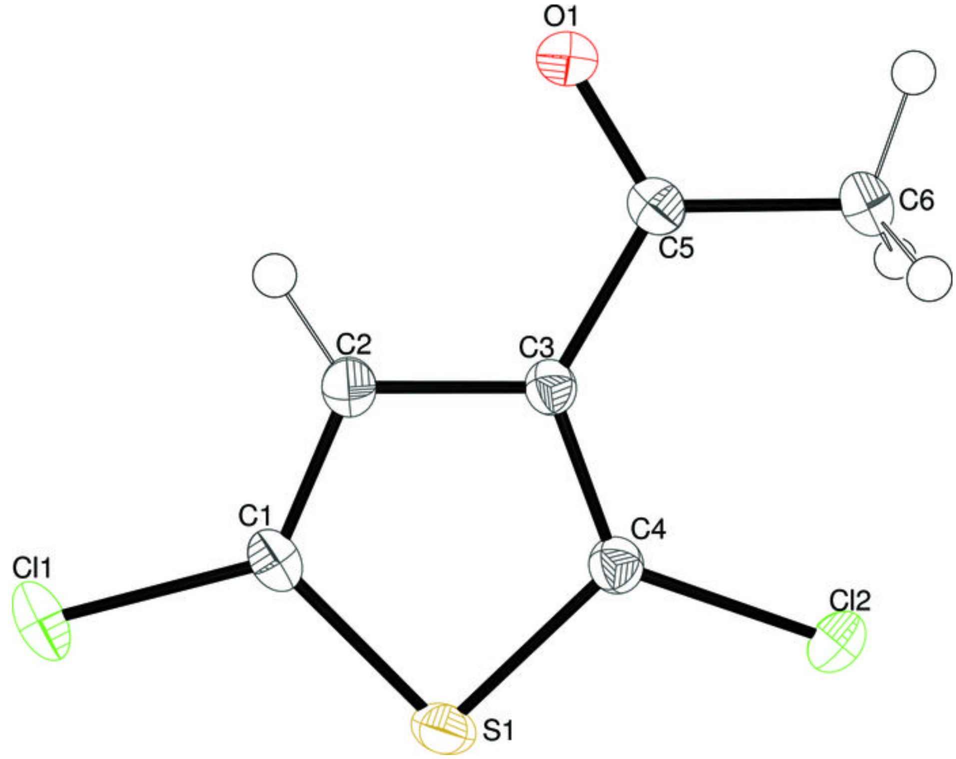

Fig. 2