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Crystal structure and Hirshfeld surface analysis of (2*E*)-3-(2,4-dichlorophenyl)-1-(2,5-dichlorothiophen-3-yl)prop-2-en-1-one

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The molecular structure of the title compound, $C_{13}H_6Cl_4OS$, consists of a 2,5-dichlorothiophene ring and a 2,4-dichlorophenyl ring linked *via* a prop-2-en-1-one spacer. The dihedral angle between the 2,5-dichlorothiophene ring and the 2,4-dichlorophenyl ring is 12.24 (15)°. The molecule has an *E* configuration about the C=C bond and the carbonyl group is *syn* with respect to the C=C bond. The molecular conformation is stabilized by intramolecular C—H···Cl contacts, producing *S*(6) and *S*(5) ring motifs. In the crystal, the molecules are linked along the *a*-axis direction through face-to-face π-stacking between the thiophene rings and the benzene rings of the molecules in zigzag sheets lying parallel to the *bc* plane along the *c* axis. The intermolecular interactions in the crystal packing were further analysed using Hirshfeld surface analysis, which indicates that the most significant contacts are Cl···H/H···Cl (20.8%), followed by Cl···Cl (18.7%), C···C (11.9%), Cl···S/S···Cl (10.9%), H···H (10.1%), C···H/H···C (9.3%) and O···H/H···O (7.6%).

1. Chemical context

Compounds bearing the 1,3-diphenyl-2-propen-1-one framework and belong to the flavonoid family are commonly called by its generic name 'chalcone'. These are abundant in nature, ranging from ferns to higher plants, and are considered to be the precursors of flavonoids and isoflavonoids, in which the two aromatic rings are joined by a three carbon α,β-unsaturated carbonyl system. In plants, chalcones are converted to the corresponding (2*S*)-flavanones in a stereospecific reaction catalysed by the enzyme chalcone isomerase. The chemistry of chalcones remains a fascination among researchers because of the large number of replaceable hydrogen atoms that allows a number of derivatives with a variety of promising biological activities. They are found in fruits and vegetables, which attracted attention because of their pharmacological activities such as anti-inflamatory (Yadav *et al.*, 2011), antifungal (Mahapatra *et al.*, 2015), antiviral (Nowakowska, 2007; Chimenti *et al.*, 2010; Elarfi &Al-Difar, 2012), antioxidant (Ferreira *et al.*, 2006) and anticancer (Stiborova *et al.*, 2011 activities). The synthesis and antimicrobial evaluation of new chalcones containing a 2,5-dichlorothiophene moiety has been

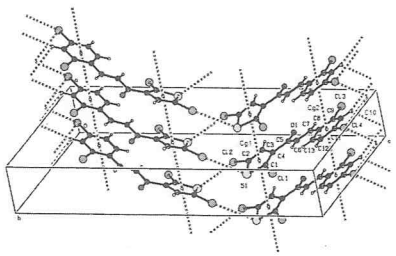

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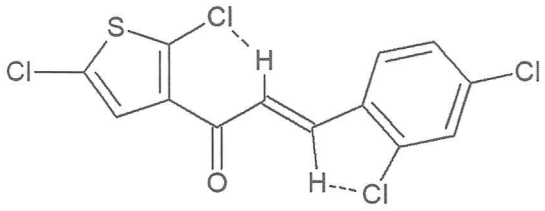
Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6A···Cl1	0.93	2.48	3.220 (3)	136
C7—H7A···Cl3	0.93	2.65	3.075 (3)	108

reported (Tomar *et al.*, 2007). In recent years, chalcones have been used in the field of materials science as non-linear optical devices (Raghavendra *et al.*, 2017; Chandra Shekhara Shetty *et al.*, 2016). In view of all the above and as part of our ongoing work (Harrison *et al.*, 2010; Jasinski *et al.*, 2010; Dutkiewicz *et al.*, 2010) herewith we report the crystal and molecular structure of the title compound.



2. Structural commentary

The title compound, Fig. 1, is constructed from two aromatic rings (2,5-dichlorothiophene and terminal 2,4-dichlorophenyl rings), which are linked by a C=C—C(=O)—C enone bridge. Probably as a result of the steric repulsion between the chlorine atoms of the adjacent molecules, the C3—C4—C5—O1 and O1—C5—C6—C7 torsion angles about the enone bridge are −11.8 (5) and 0.4 (6)°, respectively. Hence, the dihedral angle between the 2,5-dichlorothiophene ring and the 2,4-dichlorophenyl ring increases to 12.24 (15)°. The bond lengths and angles in the title compound are comparable with those of the related compounds (*E*)-3-(3,4-dimethoxyphenyl)-1-(1-hydroxynaphthalen-2yl)prop-2-en-1-one (Ezhilarasi *et al.*, 2015), (*E*)-1-(3-bromophenyl)-3-(3,4-dimethoxyphenyl)-prop-2-en-1-one (Escobar *et al.*, 2012) and (*E*)-3-(2-bromophenyl)-1-(3,4-dimethoxyphenyl)prop-2-en-1-one (Li *et al.*, 2012). The molecular conformation of the title compound is stabilized by intramolecular C—H···Cl contacts (Table 1), producing *S*(6) and *S*(5) ring motifs.

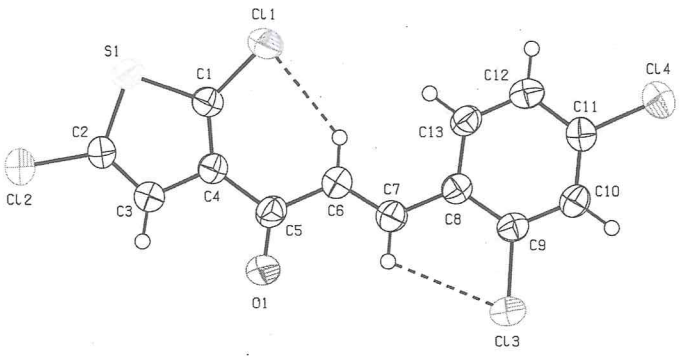


Figure 1
The molecular structure of the title compound, showing the atom labelling and displacement ellipsoids drawn at the 50% probability level. The two intramolecular C—H···Cl contacts (see Table1) are shown as dashed lines.

Table 2
Summary of short interatomic contacts (Å) in the title compound.

Contact	Distance	Symmetry operation
Cl2···S1	3.660 (1)	$\frac{1}{2}+x, \frac{3}{2}-y, 2-z$
H10A···Cl4	3.03	$-\frac{1}{2}+x, \frac{3}{2}-y, 1-z$
C8···C9	3.573 (4)	$1+x, y, z$

3. Supramolecular features and Hirshfeld surface analysis

In the crystal, conventional hydrogen bonds are not observed. π-stacking is observed between the thiophene rings (S1/C1–C4, centroid *Cg*1) of adjacent molecules in the alternating sheets along the [100] direction [*Cg*1···*Cg*1^{i,ii}: centroid–centroid distance = 3.987 (2) Å, shortest perpendicular distance for the centroid of one ring to the plane of the other = 3.6143 (12) Å, ring-centroid offset = 1.683 Å; symmetry codes: (i) $-1+x, y, z$; (ii) $1+x, y, z$] and between the benzene rings (C8–C13, centroid *Cg*2) of the same molecules [*Cg*2···*Cg*2^{i,ii}: centroid–centroid distance = 3.987 (2) Å, shortest perpendicular distance = 3.5213 (13) Å, offset = 1.869 Å]. As shown Fig. 2, the molecules are packed to form zigzag sheets lying parallel to (011) along the *c*-axis direction through face-to-face π-stacking between the thiophene and benzene rings of pairs of adjacent molecules along the [100] direction (Cl···S and Cl···H interactions; Table 2 and Fig. 2). The Cl···S contact, at 3.660 (1) Å, is equal to the sum of the van der Waals radii of S and Cl atoms (3.65 Å; Pauling, 1960).

Hirshfeld surfaces and fingerprint plots were generated for the title compound using *CrystalExplorer* (McKinnon *et al.*, 2007). Hirshfeld surfaces enable the visualization of intermolecular interactions by different colours and colour intensity, representing short or long contacts and indicating the

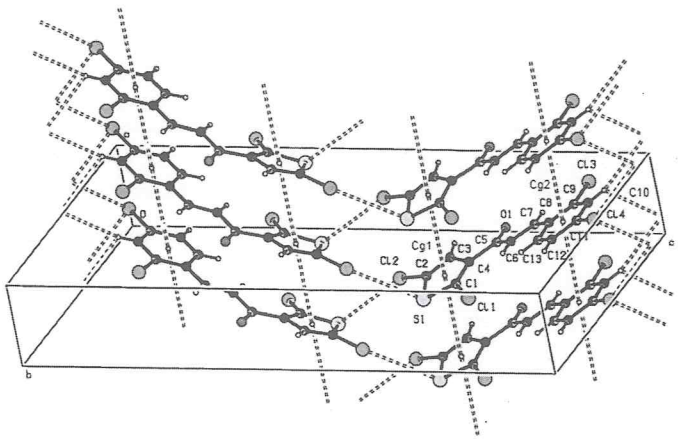


Figure 2
A view of the offset face-to-face π-stacking in the title compound, with the thick dashed lines indicating centroid-to-centroid interactions. The Cl···H and Cl···S interactions are also shown as dashed lines.

relative strength of the interactions. The overall two-dimensional fingerprint plot for the title compound and those delineated into Cl···H/ H···Cl, Cl···Cl, C···C, Cl···S/S···Cl, H···H, C···H/H···C and O···H/H···O contacts are illustrated in Fig. 3; the percentage contributions from the different interatomic contacts to the Hirshfeld surfaces are as follows: Cl···H/ H···Cl (20.8%), Cl···Cl (18.7%), C···C (11.9%), Cl···S/S···Cl (10.9%), H···H (10.1%), C···H/H···C (9.3%) and O···H/H···O (7.6%). The contributions of the other weak intermolecular contacts to the Hirshfeld surfaces are Cl···C/C···Cl (3.6%), S···C/C···S (2.8%), Cl···O/O···Cl (2.3%), S···S (0.9%), O···O (0.6%) and C···O/O···C (0.6%).

The C—H···Cl interactions appear as two distinct spikes in the fingerprint plot (Fig. 3*b*) of the title compound, where the sum of Cl···H/H···Cl interactions comprises 20.8% of the total Hirshfeld surface area of the molecule. The Cl···H/H···Cl interactions represented by the spikes in the bottom right and left region ($d_e + d_i \simeq 2.83$ Å) indicate that the hydrogen atoms are in contact with the Cl atoms to build the two-dimensional supramolecular framework [d_e and d_i represent the distances from a point on the Hirshfeld surface to the nearest atoms outside (external) and inside (internal) the surface, respectively]. Cl···Cl contacts (Fig. 3*c*; 18.7%) are disfavoured when the number of H atoms on the molecular surface is large because of competition with the more attractive H···Cl contacts. Cl···Cl contacts from a parallel alignment of C—Cl bonds (C10—H10*A*···Cl4^{iii}; (iii) $-\frac{1}{2}+x, \frac{3}{2}-y, 1-z$] may be indicated. They are known in the literature as type-I halogen–halogen interactions (Bui *et al.*, 2009), with both C—Cl···Cl angles equal to one another. In the present case, these angles are close to 165°. The C···C contacts (Fig. 3*d*); 11.9%) reflect π–π interactions between the above-mentioned aromatic rings. The S···Cl contacts (Fig. 3*e*; 10.9%) contracted to a much lesser degree. The C···H/H···C interactions (Fig. 3*g*) account for 9.3% of the total Hirshfeld surface of the molecules. The scattered points in the breakdown of the fingerprint plot show the π–π stacking interactions. In the fingerprint plot delineated into H···O/O···H contacts (Fig. 3*h*), the 7.6% contribution to the Hirshfeld surface arises from intermolecular C=O···H hydrogen bonding and is viewed as pair of spikes with the tip at $de + di \sim 2.9$ Å.

The large number of Cl···H/ H···Cl, Cl···Cl, C···C, Cl···S/S···Cl, H···H, C···H/H···C and O···H/H···O interactions suggest that van der Waals interactions and hydrogen bonding play the major roles in the crystal packing (Hathwar *et al.*, 2015).

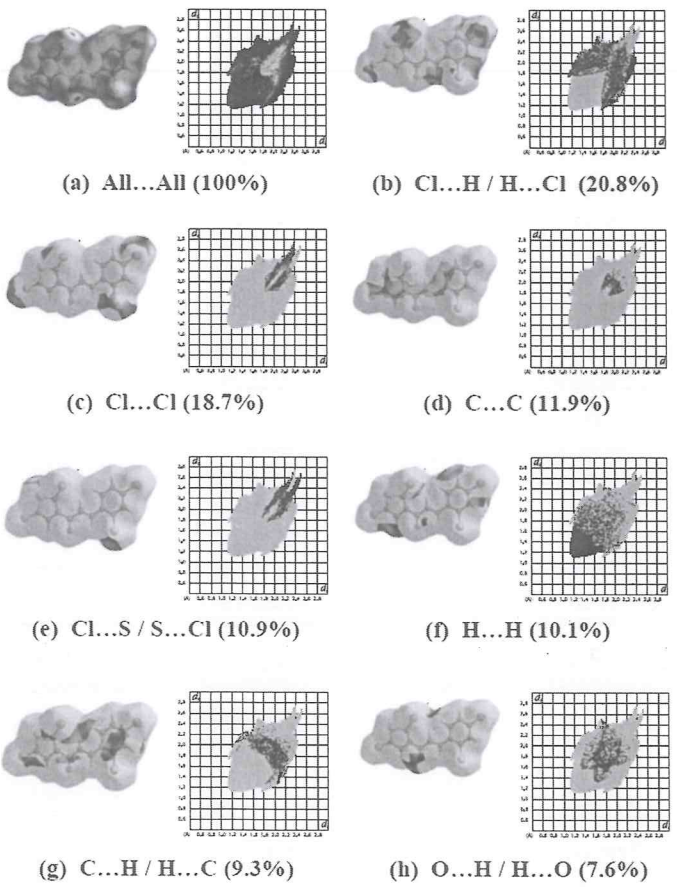


Figure 3
The two-dimensional fingerprint plots of the title compound, showing (*a*) all interactions, and delineated into (*b*) Cl···H/ H···Cl, (*c*) Cl···Cl, (*d*) C···C, (*e*) Cl···S/S···Cl, (*f*) H···H, (*g*) C···H/H···C and (*h*) O···H/ H···O interactions.

4. Database survey

The closest related compounds with the same skeleton and containing a similar bis-chalcone moiety to the title compound but with different substituents on the aromatic rings are: (2*E*)-1-(5-chlorothiophen-2-yl)-3-(4-ethylphenyl)prop-2-en-1-one [(I); Naik *et al.*, 2015], (2*E*)-1-(5-bromothiophen-2-yl)-3-(4-ethylphenyl)prop- 2-en-1-one [(II); Naik *et al.*, 2015], (2*E*)-1-(5-chlorothiophen-2-yl)-3-(4-ethoxyphenyl)prop-2-en-1-one [(III); Naik *et al.*, 2015], (2*E*)-1-(5-bromothiophen-2-yl)-3-(4-ethoxyphenyl)prop-2-en-1-one [(IV); Naik *et al.*, 2015], (2*E*)-3-(4-bromophenyl)-1-(5-chlorothiophen-2-yl)prop-2-en-1-one [(V); Naik *et al.*, 2015], (2*E*)-1-(5-bromothiophen-2-yl)-3-(3-methoxyphenyl)prop-2-en-1-one [(VI); Naik *et al.*, 2015], (*E*)-1-(5-chlorothiophen-2-yl)-3-(p-tolyl)prop-2-en-1-one [(VII); Kumara *et al.*, 2017], (*E*)-1-(5-chlorothiophen-2-yl)-3-(2,4-dimethylphenyl) prop-2-en-1-one [(VIII); Naveen *et al.*, 2016], (2*E*)-1-(5-bromothiophen- 2-yl)-3-(2-chlorophenyl)prop-2-en-1-one [(IX); Anitha *et al.*, 2015], (2*E*)-1-[4-hydroxy-3-(morpholin-4-ylmethyl)phenyl]-3-(thiophen-2-yl)prop-2-en-1-one [(X); Yesilyurt *et al.*, 2018] and (*E*)-1-(2-aminophenyl)-3-(thiophen-2-yl)prop-2-en-1-one [(XI); Chantrapromma *et al.*, 2013].

In (I) and (II), the structures are isostructural in space group $P1$, while (III) and (IV) are isostructural in space group $P2_1/c$. There are no hydrogen bonds of any kind in the structures of compounds (I) and (II), but in the structures of compounds (III) and (IV), the molecules are linked into $C(7)$ chains by means of C—H···O hydrogen bonds. In (V), there are again no hydrogen bonds nor π–π stacking interactions

Table 3
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_6Cl_4OS$
M_r	352.04
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	294
a, b, c (Å)	3.9867 (3), 13.4564 (11), 25.573 (2)
V (Å^3)	1371.91 (19)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	1.00
Crystal size (mm)	0.63 × 0.23 × 0.11
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2007)
T_{min}, T_{max}	0.757, 0.894
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	11402, 4226, 3425
R_{int}	0.026
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.720
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.038, 0.102, 1.03
No. of reflections	4226
No. of parameters	172
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.25, −0.20
Absolute structure	Flack x determined using 1124 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.04 (5)

Computer programs: *APEX2* and *SAINT* (Bruker, 2007), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2009).

but in (VI), the molecules are linked into $C(5)$ chains by C—H···O hydrogen bonds. In each of compounds (I)–(VI), the molecular skeletons are close to planarity, and there are short halogen–halogen contacts in the structures of compounds (II) and (V) and a short Br···O contact in the structure of compound (VI).

In (VII), the molecule is non-planar, with a dihedral angle of 22.6 (2)° between the aromatic rings. The molecules are linked by pairs of C—H···π interactions, forming inversion dimers. There are no other significant intermolecular interactions present. In (VIII), the molecule is nearly planar, the dihedral angle between the thiophene and phenyl rings being 9.07 (8)°. The molecules are linked *via* weak C—H···O and C—H···S hydrogen bonds, forming chains propagating along the *c*-axis direction. In (IX), the thienyl ring is not coplanar with the benzene ring, their planes forming a dihedral angle of 13.2 (4)°. In the crystal, molecules stack along the *a*-axis direction, with the interplanar separation between the thienyl rings and between the benzene rings being 3.925 (6) Å. In (X), the thiophene ring forms a dihedral angle of 26.04 (9)° with the benzene ring. The molecular conformation is stabilized by an O—H···N hydrogen bond. The molecules are connected through C—H···O hydrogen bonds, forming wave-like layers parallel to the *ab* plane, which are further linked into a three-dimensional network by C—H···π interactions. In (XI), the molecule is almost planar with a dihedral angle of 3.73 (8)° between the phenyl and thiophene rings. An intramolecular N—H···O hydrogen bond generates an $S(6)$ ring motif. Adjacent molecules are linked into dimers in an anti-parallel face-to-face manner by pairs of C—H···O interactions. Neighboring dimers are further linked into chains along the *c*-axis direction by N—H···N hydrogen bonds.

5. Synthesis and crystallization

The title compound was synthesized as per the procedure reported earlier (Kumar *et al.*, 2013*a*,*b*; Chidan Kumar *et al.*, 2014). 1-(2,5-Dichlorothiophen-3-yl)ethanone (0.01 mol) (Harrison *et al.*, 2010) and 2,4-dichlorobenzaldehyde (0.01 mol) was dissolved in 20 ml methanol. A catalytic amount of NaOH was added to the solution dropwise with vigorous stirring. The reaction mixture was stirred for about 2 h at room temperature. The formed crude products were filtered, washed successively with distilled water and recrystallized from methanol to get the title chalcone. The melting point (381–383 K) was determined by Stuart Scientific (UK) apparatus.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. C-bound H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for C—H. Owing to poor agreement between observed and calculated intensities, twelve outliers (2 7 2, 2 8 0, 2 8 1, 0 1 28, 2 8 23, 0 14 8, 0 0 6, 3 0 29, 1 0 8, 0 17 4, 1 3 27, 2 12 19) were omitted in the final cycles of refinement.

Acknowledgements

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supporting information

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Crystal structure and Hirshfeld surface analysis of (2*E*)-3-(2,4-dichlorophenyl)-1-(2,5-dichlorothiophen-3-yl)prop-2-en-1-one

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Computing details

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT* (Bruker, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2009).

(2*E*)-3-(2,4-Dichlorophenyl)-1-(2,5-dichlorothiophen-3-yl)prop-2-en-1-one

Crystal data

$C_{13}H_6Cl_4OS$
$M_r = 352.04$
Orthorhombic, $P2_12_12_1$
$a = 3.9867$ (3) Å
$b = 13.4564$ (11) Å
$c = 25.573$ (2) Å
$V = 1371.91$ (19) Å^3
$Z = 4$
$F(000) = 704$
$D_x = 1.704$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4362 reflections
$\theta = 2.2$–$28.5°$
$\mu = 1.00$ mm^{-1}
$T = 294$ K
Block, yellow
0.63 × 0.23 × 0.11 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2007)
$T_{min} = 0.757$, $T_{max} = 0.894$
11402 measured reflections
4226 independent reflections
3425 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 30.8°$, $\theta_{min} = 1.6°$
$h = -5 \rightarrow 2$
$k = -19 \rightarrow 19$
$l = -36 \rightarrow 36$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.102$
$S = 1.03$
4226 reflections
172 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0581P)^2 + 0.011P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}
Absolute structure: Flack x determined using 1124 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.04 (5)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	1.1673 (8)	0.77797 (19)	0.84012 (9)	0.0391 (6)
C2	1.2553 (8)	0.6794 (2)	0.91880 (10)	0.0419 (6)
C3	1.1115 (8)	0.6258 (2)	0.88062 (10)	0.0410 (6)
H3A	1.052432	0.559246	0.884223	0.049*
C4	1.0587 (8)	0.6820 (2)	0.83366 (10)	0.0382 (6)
C5	0.9016 (9)	0.6327 (2)	0.78763 (10)	0.0444 (7)
C6	0.7779 (10)	0.6938 (2)	0.74420 (11)	0.0493 (7)
H6A	0.809837	0.762158	0.746266	0.059*
C7	0.6253 (9)	0.6588 (2)	0.70264 (10)	0.0462 (7)
H7A	0.596005	0.590316	0.700760	0.055*
C8	0.4975 (8)	0.7177 (2)	0.65917 (10)	0.0386 (6)
C9	0.3384 (8)	0.67552 (19)	0.61621 (10)	0.0403 (6)
C10	0.2191 (8)	0.7316 (2)	0.57472 (10)	0.0431 (6)
H10A	0.112503	0.701315	0.546561	0.052*
C11	0.2620 (8)	0.8330 (2)	0.57612 (10)	0.0425 (7)
C12	0.4192 (9)	0.8788 (2)	0.61805 (11)	0.0465 (7)
H12A	0.447771	0.947373	0.618483	0.056*
C13	0.5316 (9)	0.8219 (2)	0.65879 (11)	0.0438 (7)
H13A	0.633719	0.852975	0.687101	0.053*
O1	0.8718 (9)	0.54311 (16)	0.78790 (9)	0.0721 (9)
S1	1.3313 (2)	0.80047 (5)	0.90119 (3)	0.04511 (19)
Cl1	1.1738 (3)	0.87633 (5)	0.79734 (3)	0.0556 (2)
Cl2	1.3606 (3)	0.63887 (6)	0.98017 (3)	0.0593 (2)
Cl3	0.2772 (3)	0.54840 (5)	0.61241 (3)	0.0639 (3)
Cl4	0.1204 (3)	0.90504 (6)	0.52453 (3)	0.0605 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0422 (17)	0.0375 (11)	0.0377 (11)	0.0021 (13)	0.0062 (12)	−0.0015 (9)
C2	0.0429 (18)	0.0443 (13)	0.0384 (12)	0.0015 (13)	−0.0020 (11)	0.0019 (10)
C3	0.0430 (18)	0.0392 (12)	0.0408 (12)	−0.0001 (13)	−0.0012 (12)	0.0003 (10)
C4	0.0376 (16)	0.0406 (12)	0.0364 (11)	0.0014 (12)	0.0021 (11)	−0.0021 (10)
C5	0.051 (2)	0.0462 (14)	0.0362 (12)	−0.0046 (14)	0.0005 (12)	−0.0046 (10)
C6	0.059 (2)	0.0451 (13)	0.0437 (13)	−0.0026 (15)	−0.0080 (14)	−0.0013 (11)
C7	0.058 (2)	0.0429 (13)	0.0382 (12)	−0.0006 (15)	0.0010 (14)	−0.0022 (10)
C8	0.0385 (16)	0.0415 (13)	0.0358 (11)	0.0001 (12)	0.0045 (11)	−0.0038 (10)
C9	0.0416 (16)	0.0380 (11)	0.0412 (12)	−0.0018 (13)	0.0027 (13)	−0.0046 (9)
C10	0.0433 (18)	0.0481 (13)	0.0378 (12)	0.0008 (13)	0.0001 (12)	−0.0066 (10)

C11	0.0387 (18)	0.0488 (14)	0.0401 (12)	0.0061 (13)	0.0018 (11)	0.0002 (10)
C12	0.048 (2)	0.0396 (13)	0.0522 (15)	−0.0007 (13)	0.0013 (14)	−0.0061 (11)
C13	0.0468 (19)	0.0422 (13)	0.0426 (13)	−0.0004 (13)	−0.0034 (13)	−0.0080 (11)
O1	0.123 (3)	0.0414 (11)	0.0517 (12)	−0.0110 (15)	−0.0209 (16)	−0.0012 (9)
S1	0.0504 (5)	0.0422 (3)	0.0427 (3)	−0.0035 (3)	−0.0008 (3)	−0.0055 (3)
Cl1	0.0766 (6)	0.0403 (3)	0.0498 (4)	−0.0043 (4)	−0.0001 (4)	0.0048 (3)
Cl2	0.0740 (6)	0.0589 (4)	0.0450 (3)	−0.0016 (4)	−0.0159 (4)	0.0056 (3)
Cl3	0.0883 (8)	0.0410 (3)	0.0625 (4)	−0.0127 (4)	−0.0148 (5)	−0.0023 (3)
Cl4	0.0684 (6)	0.0556 (4)	0.0576 (4)	0.0076 (4)	−0.0106 (4)	0.0080 (3)

Geometric parameters (Å, º)

C1—C4	1.372 (4)	C7—C8	1.458 (4)
C1—Cl1	1.717 (3)	C7—H7A	0.9300
C1—S1	1.720 (3)	C8—C9	1.390 (4)
C2—C3	1.343 (4)	C8—C13	1.408 (4)
C2—Cl2	1.714 (3)	C9—C10	1.386 (4)
C2—S1	1.717 (3)	C9—Cl3	1.731 (3)
C3—C4	1.435 (4)	C10—C11	1.375 (4)
C3—H3A	0.9300	C10—H10A	0.9300
C4—C5	1.489 (4)	C11—C12	1.387 (4)
C5—O1	1.212 (4)	C11—Cl4	1.732 (3)
C5—C6	1.467 (4)	C12—C13	1.368 (4)
C6—C7	1.312 (4)	C12—H12A	0.9300
C6—H6A	0.9300	C13—H13A	0.9300
C4—C1—Cl1	130.8 (2)	C8—C7—H7A	117.1
C4—C1—S1	113.3 (2)	C9—C8—C13	116.5 (3)
Cl1—C1—S1	115.92 (16)	C9—C8—C7	122.7 (3)
C3—C2—Cl2	126.8 (2)	C13—C8—C7	120.9 (3)
C3—C2—S1	113.3 (2)	C10—C9—C8	122.6 (3)
Cl2—C2—S1	119.95 (17)	C10—C9—Cl3	116.5 (2)
C2—C3—C4	112.8 (3)	C8—C9—Cl3	120.8 (2)
C2—C3—H3A	123.6	C11—C10—C9	118.5 (3)
C4—C3—H3A	123.6	C11—C10—H10A	120.7
C1—C4—C3	110.5 (2)	C9—C10—H10A	120.7
C1—C4—C5	130.3 (2)	C10—C11—C12	121.2 (3)
C3—C4—C5	119.2 (3)	C10—C11—Cl4	119.7 (2)
O1—C5—C6	121.9 (3)	C12—C11—Cl4	119.2 (2)
O1—C5—C4	118.7 (3)	C13—C12—C11	119.2 (3)
C6—C5—C4	119.3 (3)	C13—C12—H12A	120.4
C7—C6—C5	124.6 (3)	C11—C12—H12A	120.4
C7—C6—H6A	117.7	C12—C13—C8	122.0 (3)
C5—C6—H6A	117.7	C12—C13—H13A	119.0
C6—C7—C8	125.7 (3)	C8—C13—H13A	119.0
C6—C7—H7A	117.1	C2—S1—C1	90.24 (13)
Cl2—C2—C3—C4	−179.6 (2)	C13—C8—C9—C10	0.3 (5)

S1—C2—C3—C4	0.7 (4)	C7—C8—C9—C10	−179.5 (3)
Cl1—C1—C4—C3	178.6 (3)	C13—C8—C9—Cl3	−179.3 (3)
S1—C1—C4—C3	0.2 (4)	C7—C8—C9—Cl3	0.9 (4)
Cl1—C1—C4—C5	−2.0 (6)	C8—C9—C10—C11	0.4 (5)
S1—C1—C4—C5	179.6 (3)	Cl3—C9—C10—C11	179.9 (3)
C2—C3—C4—C1	−0.6 (4)	C9—C10—C11—C12	−0.3 (5)
C2—C3—C4—C5	179.9 (3)	C9—C10—C11—Cl4	179.2 (2)
C1—C4—C5—O1	168.9 (4)	C10—C11—C12—C13	−0.3 (5)
C3—C4—C5—O1	−11.8 (5)	Cl4—C11—C12—C13	−179.9 (3)
C1—C4—C5—C6	−13.1 (5)	C11—C12—C13—C8	1.0 (5)
C3—C4—C5—C6	166.3 (3)	C9—C8—C13—C12	−1.0 (5)
O1—C5—C6—C7	0.4 (6)	C7—C8—C13—C12	178.8 (3)
C4—C5—C6—C7	−177.6 (3)	C3—C2—S1—C1	−0.5 (3)
C5—C6—C7—C8	179.5 (3)	Cl2—C2—S1—C1	179.8 (2)
C6—C7—C8—C9	179.9 (4)	C4—C1—S1—C2	0.1 (3)
C6—C7—C8—C13	0.1 (5)	Cl1—C1—S1—C2	−178.5 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6*A*···Cl1	0.93	2.48	3.220 (3)	136
C7—H7*A*···Cl3	0.93	2.65	3.075 (3)	108

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