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(3-Chlorophenyl)[(*E*)-2-(1,3-dithiolan-2-ylidene)hydrazinylidene]methyl 3-chlorobenzoate

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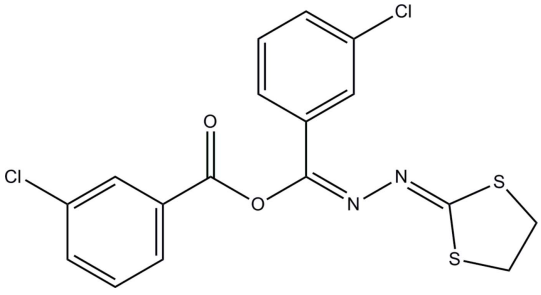
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.003 Å; R factor = 0.030; wR factor = 0.074; data-to-parameter ratio = 18.1.

In the title compound, $C_{17}H_{12}Cl_2N_2O_2S_2$, the dithiacyclopentane ring has an envelope conformation with one of the methylene C atoms as the flap. The chlorophenyl rings make a dihedral angle of 82.63 (7)°. In the crystal, π–π interactions between the benzene rings of neighbouring molecules [centroid–centroid distance = 3.547 (2) Å] link the molecules into inversion dimers. Weak non-classical C—H$\cdots X$ (X = O, N, Cl) interactions further consolidate the packing, forming a layer structure parallel to (110).

Related literature

For applications of heterocyclic dithiolane compounds, see: Tanaka *et al.* (1976); Wang *et al.* (1994). For the crystal structure of (*E*)-[2-(1,3-dithiolan-2-ylidene)hydrazinylidene]-(3-fluorophenyl)methyl 3-fluorobenzoate, see: Yin (2013).



Experimental

Crystal data

$C_{17}H_{12}Cl_2N_2O_2S_2$
M_r = 411.31
Triclinic, $P\overline{1}$
a = 8.960 (5) Å
b = 9.944 (6) Å
c = 11.128 (6) Å
α = 104.174 (8)°
β = 111.041 (7)°
γ = 99.410 (2)°
V = 861.9 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.63 mm^{-1}
T = 113 K
0.34 × 0.25 × 0.20 mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Absorption correction: multi-scan (*CrystalClear-SM Expert*; Rigaku/MSC, 2009)
T_{min} = 0.884, T_{max} = 0.884
11088 measured reflections
4080 independent reflections
3115 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.074
S = 0.95
4080 reflections
226 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C10—H10$A\cdots$N1^{i}	0.95	2.59	3.534 (2)	173
C14—H14$A\cdots$Cl1^{ii}	0.95	2.80	3.727 (2)	165
C16—H16$B\cdots$O2^{iii}	0.99	2.48	3.274 (3)	137

Symmetry codes: (i) $-x, -y+1, -z+1$; (ii) $x, y+1, z$; (iii) $x+1, y, z$.

Data collection: *CrystalClear-SM Expert* (Rigaku/MSC, 2009); cell refinement: *CrystalClear-SM Expert*; data reduction: *CrystalClear-SM Expert*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5400).

References

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Yin, L. (2013). *Acta Cryst.* E**69**, o571.

supporting information

Acta Cryst. (2013). E**69**, o714 [https://doi.org/10.1107/S1600536813009239]

(3-Chlorophenyl)[(*E*)-2-(1,3-dithiolan-2-ylidene)hydrazinylidene]methyl 3-chlorobenzoate

Ling Yin

S1. Comment

Many dithiolan heterocyclic compounds have been widely used as potent and broad-spectrum fungicides (Tanaka *et al.*, 1976; Wang *et al.*, 1994). In order to search for new heterocylic compounds with higher biological activities, we synthesized the (*E*)-((1,3-dithiolan-2-yl)diazenyl)(3-chlorophenyl)methyl 3-chlorobenzoate (I) and described its structure here.

In (I) (Fig. 1), the dithiacyclopentane ring has an envelope conformation with C16 atom as a flap. Two chlorophenyl rings (C1—C6 and C9—C14) in the molecule form a dihedral angle of 82.63 (7)°. All bond lengths and angles are normal and in a good agreement with those reported previously for related compounds (Yin, 2013)

In the crystal, π-π interactions between the benzene rings from two neighbouring molecules [centroid-centroid distance of 3.547 (2) Å] link the latters into centrosymmetric dimer, and weak non-classical C—H···*X* (*X*=O, N, Cl) interactions (Table 1) consolidate further the packing.

S2. Experimental

1.34 g (10 mmol) of (1,3-dithiolan-2-ylidene)hydrazine and 20 mmol triethylamine was dissolved in 15 ml of dichloromethane and stirred at room temperature, 3.50 g (20 mmol) 3-chlorobenzoyl chloride was added dropwise to the mixture. The reaction mixture was stirred vigorously at 0 centigrade for 4 h. The reaction mixture was poured into 200 ml of water and extracted with three 50-ml portions of dichloromethane. The combined extracts were washed with saturated brine, dried over anhydrous sodium sulfate and evaporated on a rotary evaporator to afford the crude product, which was purified by column chromatography to yield the pure product as colorless crystals. Single crystals suitable for X-ray diffraction were obtained through slow evaporation of a solution of the pure title compound in ethanol.

S3. Refinement

All H atoms bonded on carbon were found on difference maps, with C–H = 0.93 or 0.97 Å, and included in the final cycles of refinement using a riding model, with U_{iso}(H) = 1.2U_{eq}(C).

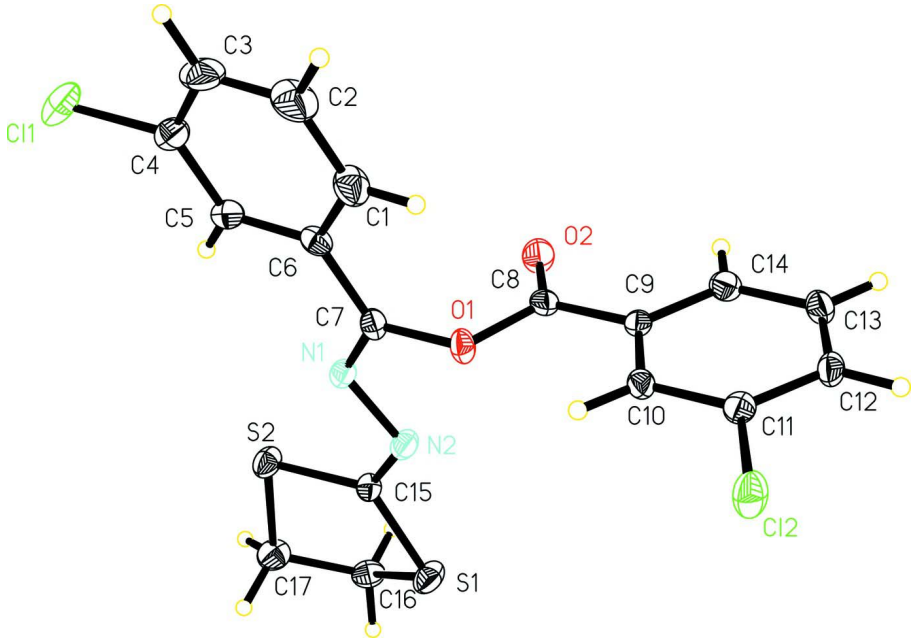

Figure 1

View of the title compound showing the atomic numbering and 50% probability displacement ellipsoids.

(3-Chlorophenyl)[(*E*)-2-(1,3-dithiolan-2-ylidene)hydrazinylidene]methyl 3-chlorobenzoate

Crystal data

$C_{17}H_{12}Cl_2N_2O_2S_2$
$M_r = 411.31$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 8.960$ (5) Å
$b = 9.944$ (6) Å
$c = 11.128$ (6) Å
$\alpha = 104.174$ (8)°
$\beta = 111.041$ (7)°
$\gamma = 99.410$ (2)°
$V = 861.9$ (8) Å^3
$Z = 2$
$F(000) = 420$
$D_x = 1.585$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3010 reflections
$\theta = 2.1$–27.9°
$\mu = 0.63$ mm^{-1}
$T = 113$ K
Block, colourless
0.34 × 0.25 × 0.20 mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Radiation source: rotating anode
Multilayer monochromator
Detector resolution: 14.63 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (*CrystalClear-SM Expert*; Rigaku/MSC, 2009)
$T_{min} = 0.884$, $T_{max} = 0.884$
11088 measured reflections
4080 independent reflections
3115 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 27.9°$, $\theta_{min} = 2.1°$
$h = -11 \rightarrow 11$
$k = -13 \rightarrow 13$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.074$
$S = 0.95$
4080 reflections
226 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
$w = 1/[\sigma^2(F_o^2) + (0.0343P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.46$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.30877 (5)	0.64714 (4)	0.23284 (4)	0.02513 (11)
S2	0.09584 (5)	0.34521 (4)	0.09623 (4)	0.02211 (11)
Cl1	−0.52910 (5)	−0.06840 (4)	0.12078 (4)	0.03188 (12)
Cl2	0.10357 (5)	1.00841 (4)	0.89309 (4)	0.02569 (11)
O1	−0.11886 (13)	0.59701 (10)	0.42806 (10)	0.0180 (2)
O2	−0.27715 (13)	0.66856 (11)	0.26079 (10)	0.0234 (3)
N1	−0.07149 (15)	0.43373 (13)	0.26298 (12)	0.0168 (3)
N2	0.05526 (15)	0.55241 (13)	0.28369 (13)	0.0203 (3)
C1	−0.3769 (2)	0.38581 (18)	0.40729 (17)	0.0269 (4)
H1B	−0.3438	0.4807	0.4699	0.032*
C2	−0.5067 (2)	0.2817 (2)	0.39886 (19)	0.0356 (4)
H2B	−0.5629	0.3060	0.4555	0.043*
C3	−0.5555 (2)	0.1434 (2)	0.30938 (18)	0.0320 (4)
H3A	−0.6454	0.0725	0.3034	0.038*
C4	−0.47179 (19)	0.10906 (17)	0.22822 (15)	0.0222 (4)
C5	−0.34232 (18)	0.21123 (16)	0.23301 (14)	0.0180 (3)
H5A	−0.2872	0.1863	0.1756	0.022*
C6	−0.29455 (18)	0.35118 (16)	0.32354 (15)	0.0173 (3)
C7	−0.15643 (18)	0.46156 (15)	0.33172 (14)	0.0163 (3)
C8	−0.19326 (17)	0.69437 (16)	0.37966 (15)	0.0167 (3)
C9	−0.15680 (17)	0.82978 (15)	0.49075 (14)	0.0156 (3)
C10	−0.05798 (17)	0.84909 (15)	0.62690 (15)	0.0162 (3)
H10A	−0.0168	0.7734	0.6523	0.019*
C11	−0.02184 (18)	0.98142 (16)	0.72356 (14)	0.0172 (3)
C12	−0.08166 (18)	1.09324 (16)	0.68971 (15)	0.0193 (3)

H12A	−0.0540	1.1835	0.7580	0.023*
C13	−0.18269 (19)	1.07119 (16)	0.55451 (16)	0.0213 (3)
H13A	−0.2265	1.1462	0.5300	0.026*
C14	−0.21989 (18)	0.94018 (16)	0.45513 (16)	0.0195 (3)
H14A	−0.2885	0.9257	0.3626	0.023*
C15	0.13750 (18)	0.51590 (16)	0.21249 (15)	0.0172 (3)
C16	0.31996 (19)	0.54981 (17)	0.07793 (15)	0.0225 (4)
H16A	0.2393	0.5664	−0.0013	0.027*
H16B	0.4331	0.5832	0.0830	0.027*
C17	0.27909 (19)	0.39074 (17)	0.06257 (16)	0.0246 (4)
H17A	0.3744	0.3703	0.1278	0.030*
H17B	0.2568	0.3319	−0.0310	0.030*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0235 (2)	0.0184 (2)	0.0313 (2)	−0.00078 (17)	0.01585 (19)	0.00201 (18)
S2	0.0228 (2)	0.0166 (2)	0.0255 (2)	0.00214 (17)	0.01373 (18)	0.00125 (17)
Cl1	0.0338 (2)	0.0226 (2)	0.0255 (2)	−0.00746 (19)	0.00331 (19)	0.00860 (18)
Cl2	0.0353 (2)	0.0202 (2)	0.0180 (2)	0.00879 (18)	0.00931 (17)	0.00234 (16)
O1	0.0231 (6)	0.0137 (5)	0.0157 (5)	0.0080 (5)	0.0067 (4)	0.0024 (4)
O2	0.0237 (6)	0.0210 (6)	0.0187 (6)	0.0067 (5)	0.0025 (5)	0.0042 (5)
N1	0.0171 (6)	0.0139 (6)	0.0194 (6)	0.0033 (5)	0.0083 (5)	0.0053 (5)
N2	0.0192 (7)	0.0143 (7)	0.0258 (7)	0.0013 (5)	0.0111 (6)	0.0037 (6)
C1	0.0310 (10)	0.0262 (9)	0.0335 (9)	0.0128 (8)	0.0210 (8)	0.0116 (8)
C2	0.0345 (10)	0.0436 (11)	0.0494 (12)	0.0182 (9)	0.0324 (10)	0.0225 (10)
C3	0.0201 (9)	0.0381 (11)	0.0453 (11)	0.0056 (8)	0.0169 (8)	0.0231 (9)
C4	0.0191 (8)	0.0230 (9)	0.0210 (8)	0.0025 (7)	0.0033 (7)	0.0111 (7)
C5	0.0162 (8)	0.0217 (8)	0.0170 (7)	0.0044 (7)	0.0066 (6)	0.0087 (7)
C6	0.0166 (7)	0.0220 (8)	0.0177 (8)	0.0089 (7)	0.0080 (6)	0.0100 (6)
C7	0.0192 (8)	0.0147 (8)	0.0138 (7)	0.0068 (6)	0.0050 (6)	0.0042 (6)
C8	0.0135 (7)	0.0167 (8)	0.0221 (8)	0.0047 (6)	0.0093 (6)	0.0071 (6)
C9	0.0130 (7)	0.0135 (7)	0.0205 (8)	0.0028 (6)	0.0086 (6)	0.0039 (6)
C10	0.0160 (7)	0.0138 (7)	0.0218 (8)	0.0050 (6)	0.0106 (6)	0.0061 (6)
C11	0.0170 (8)	0.0174 (8)	0.0190 (8)	0.0036 (6)	0.0105 (6)	0.0051 (6)
C12	0.0208 (8)	0.0132 (8)	0.0260 (8)	0.0044 (6)	0.0143 (7)	0.0030 (6)
C13	0.0214 (8)	0.0162 (8)	0.0305 (9)	0.0086 (7)	0.0126 (7)	0.0098 (7)
C14	0.0172 (8)	0.0205 (8)	0.0208 (8)	0.0060 (7)	0.0072 (6)	0.0074 (7)
C15	0.0177 (8)	0.0140 (7)	0.0192 (8)	0.0045 (6)	0.0067 (6)	0.0060 (6)
C16	0.0186 (8)	0.0263 (9)	0.0236 (8)	0.0035 (7)	0.0109 (7)	0.0084 (7)
C17	0.0228 (9)	0.0259 (9)	0.0257 (9)	0.0050 (7)	0.0139 (7)	0.0047 (7)

Geometric parameters (Å, º)

S1—C15	1.7493 (17)	C5—C6	1.394 (2)
S1—C16	1.8048 (18)	C5—H5A	0.9500
S2—C15	1.7507 (17)	C6—C7	1.473 (2)
S2—C17	1.8228 (18)	C8—C9	1.483 (2)

Cl1—C4	1.7427 (18)	C9—C14	1.392 (2)
Cl2—C11	1.7393 (17)	C9—C10	1.398 (2)
O1—C8	1.3713 (17)	C10—C11	1.382 (2)
O1—C7	1.4008 (17)	C10—H10A	0.9500
O2—C8	1.1989 (18)	C11—C12	1.386 (2)
N1—C7	1.2744 (19)	C12—C13	1.388 (2)
N1—N2	1.4058 (18)	C12—H12A	0.9500
N2—C15	1.2917 (19)	C13—C14	1.385 (2)
C1—C2	1.383 (2)	C13—H13A	0.9500
C1—C6	1.397 (2)	C14—H14A	0.9500
C1—H1B	0.9500	C16—C17	1.516 (2)
C2—C3	1.376 (3)	C16—H16A	0.9900
C2—H2B	0.9500	C16—H16B	0.9900
C3—C4	1.384 (2)	C17—H17A	0.9900
C3—H3A	0.9500	C17—H17B	0.9900
C4—C5	1.387 (2)		

C15—S1—C16	94.86 (8)	C10—C9—C8	121.62 (13)
C15—S2—C17	95.15 (7)	C11—C10—C9	118.14 (13)
C8—O1—C7	115.97 (11)	C11—C10—H10A	120.9
C7—N1—N2	114.80 (13)	C9—C10—H10A	120.9
C15—N2—N1	111.40 (13)	C10—C11—C12	122.18 (14)
C2—C1—C6	119.91 (16)	C10—C11—Cl2	118.80 (11)
C2—C1—H1B	120.0	C12—C11—Cl2	119.01 (12)
C6—C1—H1B	120.0	C11—C12—C13	118.94 (14)
C3—C2—C1	120.83 (16)	C11—C12—H12A	120.5
C3—C2—H2B	119.6	C13—C12—H12A	120.5
C1—C2—H2B	119.6	C14—C13—C12	120.22 (14)
C2—C3—C4	119.02 (16)	C14—C13—H13A	119.9
C2—C3—H3A	120.5	C12—C13—H13A	119.9
C4—C3—H3A	120.5	C13—C14—C9	120.04 (14)
C3—C4—C5	121.63 (16)	C13—C14—H14A	120.0
C3—C4—Cl1	118.48 (13)	C9—C14—H14A	120.0
C5—C4—Cl1	119.87 (13)	N2—C15—S1	118.01 (12)
C4—C5—C6	118.83 (15)	N2—C15—S2	126.35 (12)
C4—C5—H5A	120.6	S1—C15—S2	115.64 (9)
C6—C5—H5A	120.6	C17—C16—S1	107.62 (11)
C5—C6—C1	119.77 (14)	C17—C16—H16A	110.2
C5—C6—C7	120.01 (13)	S1—C16—H16A	110.2
C1—C6—C7	120.21 (14)	C17—C16—H16B	110.2
N1—C7—O1	122.71 (13)	S1—C16—H16B	110.2
N1—C7—C6	122.70 (14)	H16A—C16—H16B	108.5
O1—C7—C6	114.45 (13)	C16—C17—S2	108.49 (11)
O2—C8—O1	122.10 (14)	C16—C17—H17A	110.0
O2—C8—C9	125.99 (14)	S2—C17—H17A	110.0
O1—C8—C9	111.91 (12)	C16—C17—H17B	110.0
C14—C9—C10	120.46 (13)	S2—C17—H17B	110.0
C14—C9—C8	117.88 (13)	H17A—C17—H17B	108.4

C7—N1—N2—C15	179.07 (13)	O1—C8—C9—C14	177.72 (12)
C6—C1—C2—C3	0.5 (3)	O2—C8—C9—C10	179.57 (14)
C1—C2—C3—C4	0.5 (3)	O1—C8—C9—C10	−0.29 (19)
C2—C3—C4—C5	−1.3 (2)	C14—C9—C10—C11	−1.6 (2)
C2—C3—C4—Cl1	177.27 (12)	C8—C9—C10—C11	176.34 (13)
C3—C4—C5—C6	0.9 (2)	C9—C10—C11—C12	0.7 (2)
Cl1—C4—C5—C6	−177.58 (10)	C9—C10—C11—Cl2	−179.01 (11)
C4—C5—C6—C1	0.1 (2)	C10—C11—C12—C13	0.7 (2)
C4—C5—C6—C7	179.44 (13)	Cl2—C11—C12—C13	−179.55 (11)
C2—C1—C6—C5	−0.8 (2)	C11—C12—C13—C14	−1.3 (2)
C2—C1—C6—C7	179.86 (14)	C12—C13—C14—C9	0.4 (2)
N2—N1—C7—O1	−4.57 (19)	C10—C9—C14—C13	1.1 (2)
N2—N1—C7—C6	179.94 (12)	C8—C9—C14—C13	−176.96 (13)
C8—O1—C7—N1	89.63 (17)	N1—N2—C15—S1	−176.17 (9)
C8—O1—C7—C6	−94.53 (15)	N1—N2—C15—S2	3.35 (19)
C5—C6—C7—N1	−3.2 (2)	C16—S1—C15—N2	−163.32 (12)
C1—C6—C7—N1	176.16 (14)	C16—S1—C15—S2	17.11 (9)
C5—C6—C7—O1	−179.00 (11)	C17—S2—C15—N2	−174.68 (14)
C1—C6—C7—O1	0.33 (19)	C17—S2—C15—S1	4.84 (9)
C7—O1—C8—O2	−4.5 (2)	C15—S1—C16—C17	−37.74 (12)
C7—O1—C8—C9	175.41 (12)	S1—C16—C17—S2	45.83 (13)
O2—C8—C9—C14	−2.4 (2)	C15—S2—C17—C16	−30.73 (12)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10*A*···N1^{i}	0.95	2.59	3.534 (2)	173
C14—H14*A*···Cl1^{ii}	0.95	2.80	3.727 (2)	165
C16—H16*B*···O2^{iii}	0.99	2.48	3.274 (3)	137

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