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3-(2,4-Dichlorophenyl)-5-phenyl-1,2,4-oxadiazole

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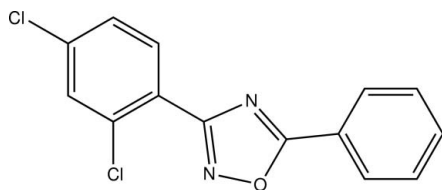
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 Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.035; wR factor = 0.121; data-to-parameter ratio = 13.2.

In the title compound, $\text{C}_{14}\text{H}_8\text{Cl}_2\text{N}_2\text{O}$, the dihedral angles between the plane of the oxadiazole ring and those of the benzene rings are 2.3 (1) and 9.5 (1)°. In the crystal, molecules are linked into chains along the c axis by $\text{C}-\text{H}\cdots\text{Cl}$ interactions. These chains are stacked along the a axis.

Related literature

For the biological properties of heterocyclic compounds including oxadiazoles, see: Andersen *et al.* (1994); Showell *et al.* (1991); Watjen *et al.* (1989); Swain *et al.* (1991); Clitherow *et al.* (1996). For their pharmacological and medicinal activity, see: Isloor *et al.* (2010); Chandrakantha *et al.* (2010). For a related structure, see: Wang *et al.* (2006). For the stability of the temperature controller used for the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

$\text{C}_{14}\text{H}_8\text{Cl}_2\text{N}_2\text{O}$	$c = 14.6949$ (9) Å
$M_r = 291.12$	$\alpha = 99.044$ (2)°
Triclinic, $P\bar{1}$	$\beta = 91.158$ (2)°
$a = 3.8035$ (2) Å	$\gamma = 98.891$ (2)°
$b = 10.9666$ (7) Å	$V = 597.43$ (6) Å ³

* Thomson Reuters ResearcherID: A-3561-2009.

$Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.53$ mm⁻¹

$T = 100$ K
 $0.41 \times 0.13 \times 0.09$ mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	12084 measured reflections 2692 independent reflections
Absorption correction: multi-scan (SADABS; Bruker, 2005)	2355 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.035$
$T_{\text{min}} = 0.809$, $T_{\text{max}} = 0.953$	

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.035$	204 parameters
$wR(F^2) = 0.121$	All H-atom parameters refined
$S = 1.14$	$\Delta\rho_{\text{max}} = 0.55$ e Å ⁻³
2692 reflections	$\Delta\rho_{\text{min}} = -0.39$ e Å ⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C3}-\text{H3A}\cdots\text{Cl1}^i$	0.95 (3)	2.81 (3)	3.577 (2)	138.6 (19)

 Symmetry code: (i) $x + 1, y, z - 1$.

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL and PLATON (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2743).

References

- Andersen, K. E., Jørgensen, A. S. & Braestrup, C. (1994). *Eur. J. Med. Chem.* **29**, 393-399.
- Bruker (2005). APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
- Chandrakantha, B., Shetty, P., Nambiyar, V., Isloor, N. & Isloor, A. M. (2010). *Eur. J. Med. Chem.* **45**, 1206-1210.
- Clitherow, J. W., Beswick, P., Irving, W. J., Scopes, D. I. C., Barnes, J. C., Clapham, J., Brown, J. D., Evans, D. J. & Hayes, A. G. (1996). *Bioorg. Med. Chem. Lett.* **6**, 833-838.
- Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105-107.
- Isloor, A. M., Kalluraya, B. & Pai, K. S. (2010). *Eur. J. Med. Chem.* **45**, 825-830.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112-122.
- Showell, G. A., Gibbons, T. L., Kneen, C. O., MacLeod, A. M., Merchant, K., Saunders, J., Freedman, S. B., Patel, S. & Baker, R. (1991). *J. Med. Chem.* **34**, 1086-1094.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148-155.
- Swain, C. J., Baker, R., Kneen, C., Moseley, J., Saunders, J., Seward, E. M., Stevenson, G., Beer, M., Stanton, J. & Watling, K. (1991). *J. Med. Chem.* **34**, 140-151.
- Wang, H.-B., Liu, Z.-Q., Wang, H.-B. & Yan, X.-C. (2006). *Acta Cryst.* **E62**, o4715-o4716.
- Watjen, F., Baker, R., Engelstoff, M., Herbert, R., MacLeod, A., Knight, A., Merchant, K., Moseley, J., Saunders, J., Swain, C. J., Wang, E. & Springer, J. P. (1989). *J. Med. Chem.* **32**, 282-2291.

supporting information

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3-(2,4-Dichlorophenyl)-5-phenyl-1,2,4-oxadiazole

Hoong-Kun Fun, Mohd Mustaqim Rosli, Sankappa Rai, Arun M. Isloor and Prakash Shetty

S1. Comment

Heterocyclic compounds are becoming increasingly important in recent years due to their pharmacological activities (Isloor *et al.* 2010). Nitrogen- and oxygen-containing five/six membered heterocyclic compounds are of enormous significance in the field of medicinal chemistry (Chandrakantha *et al.*, 2010). Oxadiazoles play a very vital role in the preparation of various biologically active drugs with anti-inflammatory (Andersen *et al.*, 1994), anti-cancer (Showell *et al.*, 1991), anti-HIV (Watjen *et al.*, 1989), anti-diabetic and anti-microbial (Swain *et al.*, 1991) activities. The results of biological studies showed that oxadiazole derivatives also possess maximum anti-inflammatory, analgesic and minimum ulcerogenic and lipid per-oxidation (Clitherow *et al.*, 1996) properties.

The geometrical parameters of (I) are within the normal range and comparable with those for a related structure (Wang *et al.*, 2006). The mean plane of of the oxadiazole ring (C7/C8/N1/N2/O1) is almost coplanar with the C9-C14 benzene ring [dihedral angle = 2.3 (1)°] but slightly twisted with the C1-C6 benzene ring [dihedral angle = 9.5 (1)°].

The C-H...Cl (Table 1) interactions link the molecules into infinite chains along the c-axis and these chains are stacked along the a-axis.

S2. Experimental

The title compound was prepared by heating a solution of 2,4-dichloro- N'-hydroxy-benzamidine (1 g,0.0042 mol) and benzoylchloride (0.65 g,0.004 mol) in pyridine (30 ml). The reaction mixture was heated at 114 °C for 1.5 hour and concentrated under vacuum. Further purification was done by column chromatography. The solid obtained was recrystallised using dichloromethane. Yield: 1 g (76%), Melting point 413-415 K.

S3. Refinement

All H atoms were located in a difference Fourier map and refined freely.

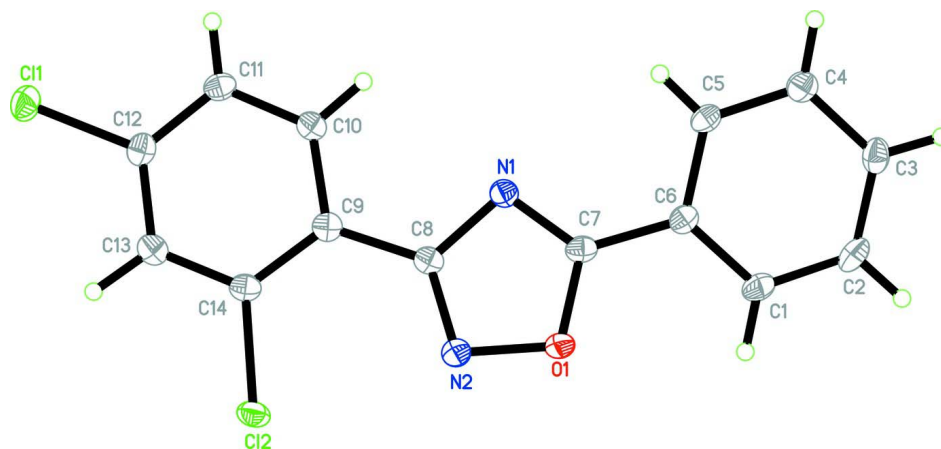


Figure 1

The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

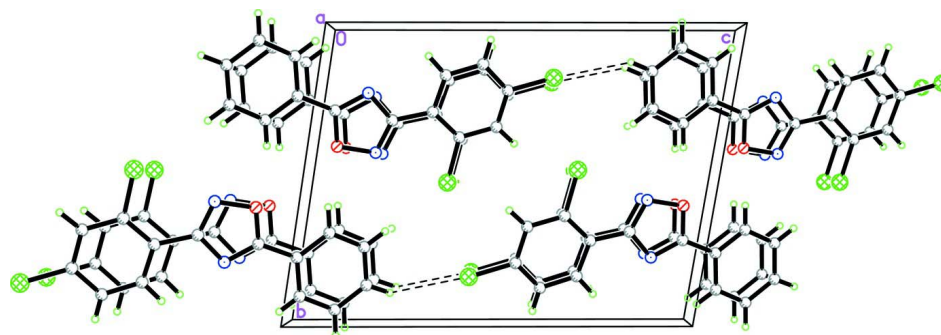


Figure 2

The crystal structure of (I), showing infinite chains along the c-axis.

3-(2,4-Dichlorophenyl)-5-phenyl-1,2,4-oxadiazole

Crystal data

$C_{14}H_8Cl_2N_2O$

$M_r = 291.12$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 3.8035$ (2) Å

$b = 10.9666$ (7) Å

$c = 14.6949$ (9) Å

$\alpha = 99.044$ (2)°

$\beta = 91.158$ (2)°

$\gamma = 98.891$ (2)°

$V = 597.43$ (6) Å³

$Z = 2$

$F(000) = 296$

$D_x = 1.618$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 7625 reflections

$\theta = 2.5$ – 36.0 °

$\mu = 0.53$ mm⁻¹

$T = 100$ K

Block, colourless

$0.41 \times 0.13 \times 0.09$ mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2005)

$T_{\min} = 0.809$, $T_{\max} = 0.953$

12084 measured reflections

2692 independent reflections

2355 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.035$
 $\theta_{\text{max}} = 27.5^\circ$, $\theta_{\text{min}} = 1.4^\circ$
 $h = -4 \rightarrow 4$

$k = -14 \rightarrow 14$
 $l = -19 \rightarrow 19$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.121$
 $S = 1.14$
 2692 reflections
 204 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
 All H-atom parameters refined
 $w = 1/[\sigma^2(F_o^2) + (0.0727P)^2 + 0.3227P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.55 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.39 \text{ e } \text{\AA}^{-3}$

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cyrosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

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.

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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.67894 (14)	0.17290 (4)	0.56305 (3)	0.02303 (17)
C12	0.74585 (13)	0.52050 (4)	0.34575 (3)	0.01905 (16)
O1	1.0808 (4)	0.40239 (12)	0.07273 (9)	0.0188 (3)
N1	1.1293 (4)	0.22993 (14)	0.12866 (10)	0.0152 (3)
N2	0.9704 (5)	0.42193 (15)	0.16440 (11)	0.0186 (3)
C1	1.3840 (5)	0.31992 (18)	-0.09742 (13)	0.0182 (4)
C2	1.5167 (6)	0.27272 (19)	-0.18064 (13)	0.0207 (4)
C3	1.5623 (5)	0.14861 (19)	-0.19917 (13)	0.0197 (4)
C4	1.4763 (5)	0.06975 (18)	-0.13435 (12)	0.0180 (4)
C5	1.3476 (5)	0.11647 (17)	-0.05099 (12)	0.0157 (4)
C6	1.3013 (5)	0.24188 (17)	-0.03204 (12)	0.0146 (4)
C7	1.1707 (5)	0.28734 (17)	0.05746 (12)	0.0152 (4)
C8	1.0052 (5)	0.31698 (17)	0.19311 (12)	0.0141 (4)
C9	0.9255 (5)	0.28872 (16)	0.28577 (12)	0.0146 (4)
C10	0.9670 (5)	0.16979 (17)	0.30360 (12)	0.0155 (4)
C11	0.8962 (5)	0.13252 (18)	0.38802 (13)	0.0172 (4)
C12	0.7795 (5)	0.21663 (18)	0.45703 (12)	0.0171 (4)
C13	0.7382 (5)	0.33600 (18)	0.44337 (12)	0.0172 (4)
C14	0.8094 (5)	0.37087 (16)	0.35794 (12)	0.0155 (4)
H1A	1.360 (7)	0.402 (3)	-0.0859 (17)	0.026 (6)*

H2A	1.572 (7)	0.323 (2)	-0.2233 (18)	0.024 (6)*
H3A	1.664 (7)	0.121 (2)	-0.2558 (18)	0.029 (7)*
H4A	1.483 (6)	-0.015 (2)	-0.1458 (15)	0.015 (5)*
H5A	1.325 (7)	0.068 (2)	-0.0091 (17)	0.020 (6)*
H10A	1.028 (7)	0.109 (2)	0.2564 (16)	0.019 (6)*
H11A	0.922 (6)	0.054 (2)	0.3967 (15)	0.014 (5)*
H13A	0.635 (7)	0.390 (2)	0.4924 (17)	0.022 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0276 (3)	0.0272 (3)	0.0163 (2)	0.0063 (2)	0.00286 (18)	0.00754 (18)
Cl2	0.0228 (3)	0.0169 (2)	0.0191 (2)	0.00957 (18)	-0.00111 (17)	0.00205 (17)
O1	0.0261 (8)	0.0160 (6)	0.0165 (6)	0.0086 (5)	0.0003 (5)	0.0045 (5)
N1	0.0149 (8)	0.0165 (7)	0.0148 (7)	0.0051 (6)	-0.0008 (6)	0.0025 (6)
N2	0.0233 (9)	0.0185 (8)	0.0160 (7)	0.0077 (6)	0.0014 (6)	0.0043 (6)
C1	0.0180 (10)	0.0182 (9)	0.0199 (9)	0.0044 (7)	-0.0031 (7)	0.0068 (7)
C2	0.0208 (10)	0.0256 (10)	0.0180 (9)	0.0034 (8)	-0.0013 (7)	0.0112 (7)
C3	0.0185 (10)	0.0277 (10)	0.0139 (8)	0.0062 (8)	-0.0005 (7)	0.0039 (7)
C4	0.0174 (10)	0.0194 (9)	0.0178 (8)	0.0050 (7)	-0.0031 (7)	0.0035 (7)
C5	0.0138 (9)	0.0173 (8)	0.0171 (8)	0.0031 (7)	-0.0019 (6)	0.0063 (7)
C6	0.0121 (9)	0.0171 (8)	0.0148 (8)	0.0026 (7)	-0.0031 (6)	0.0037 (6)
C7	0.0128 (9)	0.0153 (8)	0.0181 (8)	0.0037 (7)	-0.0037 (6)	0.0036 (6)
C8	0.0106 (9)	0.0157 (8)	0.0160 (8)	0.0035 (6)	-0.0028 (6)	0.0016 (6)
C9	0.0103 (9)	0.0176 (9)	0.0157 (8)	0.0023 (7)	-0.0034 (6)	0.0026 (6)
C10	0.0137 (9)	0.0166 (8)	0.0163 (8)	0.0040 (7)	-0.0016 (6)	0.0013 (6)
C11	0.0170 (10)	0.0163 (9)	0.0191 (8)	0.0032 (7)	-0.0021 (7)	0.0050 (7)
C12	0.0146 (9)	0.0222 (9)	0.0150 (8)	0.0027 (7)	-0.0016 (6)	0.0049 (7)
C13	0.0150 (9)	0.0203 (9)	0.0164 (8)	0.0050 (7)	-0.0024 (7)	0.0009 (7)
C14	0.0128 (9)	0.0157 (8)	0.0183 (8)	0.0036 (7)	-0.0029 (6)	0.0026 (7)

Geometric parameters (Å, °)

Cl1—C12	1.7338 (18)	C4—H4A	0.92 (2)
Cl2—C14	1.7310 (18)	C5—C6	1.399 (3)
O1—C7	1.344 (2)	C5—H5A	0.87 (2)
O1—N2	1.413 (2)	C6—C7	1.457 (2)
N1—C7	1.303 (2)	C8—C9	1.470 (2)
N1—C8	1.380 (2)	C9—C10	1.401 (3)
N2—C8	1.311 (2)	C9—C14	1.405 (3)
C1—C2	1.387 (3)	C10—C11	1.385 (3)
C1—C6	1.394 (2)	C10—H10A	0.94 (2)
C1—H1A	0.91 (3)	C11—C12	1.388 (3)
C2—C3	1.384 (3)	C11—H11A	0.91 (2)
C2—H2A	0.91 (3)	C12—C13	1.387 (3)
C3—C4	1.395 (3)	C13—C14	1.389 (3)
C3—H3A	0.95 (3)	C13—H13A	0.99 (3)
C4—C5	1.381 (3)		

C7—O1—N2	106.71 (14)	O1—C7—C6	119.03 (16)
C7—N1—C8	102.57 (15)	N2—C8—N1	114.69 (16)
C8—N2—O1	103.00 (15)	N2—C8—C9	125.14 (17)
C2—C1—C6	119.53 (18)	N1—C8—C9	120.17 (15)
C2—C1—H1A	119.2 (16)	C10—C9—C14	117.16 (16)
C6—C1—H1A	121.2 (16)	C10—C9—C8	117.41 (16)
C3—C2—C1	120.30 (17)	C14—C9—C8	125.43 (16)
C3—C2—H2A	119.8 (16)	C11—C10—C9	122.41 (17)
C1—C2—H2A	119.9 (16)	C11—C10—H10A	116.9 (14)
C2—C3—C4	120.40 (18)	C9—C10—H10A	120.5 (14)
C2—C3—H3A	117.6 (16)	C10—C11—C12	118.35 (17)
C4—C3—H3A	122.0 (16)	C10—C11—H11A	120.0 (14)
C5—C4—C3	119.60 (18)	C12—C11—H11A	121.6 (14)
C5—C4—H4A	116.9 (14)	C13—C12—C11	121.57 (17)
C3—C4—H4A	123.3 (14)	C13—C12—C11	118.49 (15)
C4—C5—C6	120.14 (17)	C11—C12—C11	119.94 (15)
C4—C5—H5A	117.2 (16)	C12—C13—C14	118.95 (17)
C6—C5—H5A	122.2 (16)	C12—C13—H13A	118.8 (14)
C1—C6—C5	120.04 (17)	C14—C13—H13A	121.9 (14)
C1—C6—C7	121.86 (17)	C13—C14—C9	121.56 (17)
C5—C6—C7	118.10 (16)	C13—C14—C12	116.22 (14)
N1—C7—O1	113.04 (16)	C9—C14—C12	122.21 (14)
N1—C7—C6	127.93 (16)		
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C7—O1—N2—C8	-0.24 (19)	C7—N1—C8—N2	0.1 (2)
C6—C1—C2—C3	0.8 (3)	C7—N1—C8—C9	-179.53 (16)
C1—C2—C3—C4	-0.2 (3)	N2—C8—C9—C10	177.93 (18)
C2—C3—C4—C5	-0.5 (3)	N1—C8—C9—C10	-2.5 (3)
C3—C4—C5—C6	0.5 (3)	N2—C8—C9—C14	-1.8 (3)
C2—C1—C6—C5	-0.9 (3)	N1—C8—C9—C14	177.79 (17)
C2—C1—C6—C7	178.25 (17)	C14—C9—C10—C11	0.4 (3)
C4—C5—C6—C1	0.2 (3)	C8—C9—C10—C11	-179.33 (17)
C4—C5—C6—C7	-178.94 (17)	C9—C10—C11—C12	0.1 (3)
C8—N1—C7—O1	-0.2 (2)	C10—C11—C12—C13	-0.9 (3)
C8—N1—C7—C6	179.12 (18)	C10—C11—C12—C11	178.69 (14)
N2—O1—C7—N1	0.3 (2)	C11—C12—C13—C14	1.2 (3)
N2—O1—C7—C6	-179.11 (15)	C11—C12—C13—C14	-178.38 (14)
C1—C6—C7—N1	-169.98 (19)	C12—C13—C14—C9	-0.7 (3)
C5—C6—C7—N1	9.1 (3)	C12—C13—C14—C12	179.14 (14)
C1—C6—C7—O1	9.4 (3)	C10—C9—C14—C13	-0.1 (3)
C5—C6—C7—O1	-171.53 (16)	C8—C9—C14—C13	179.63 (17)
O1—N2—C8—N1	0.1 (2)	C10—C9—C14—C12	-179.90 (14)
O1—N2—C8—C9	179.68 (16)	C8—C9—C14—C12	-0.2 (3)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C3—H3A \cdots Cl1 ⁱ	0.95 (3)	2.81 (3)	3.577 (2)	138.6 (19)

Symmetry code: (i) $x+1, y, z-1$.