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1-[(6-Chloropyridin-3-yl)methyl]-3-phenylimidazolidine-2,4-dione

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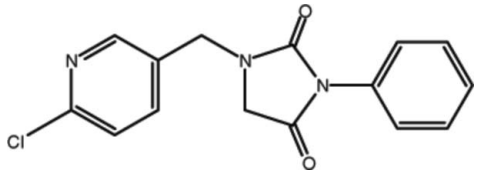
Key indicators: single-crystal X-ray study; T = 297 K; mean σ(C–C) = 0.003 Å; R factor = 0.056; wR factor = 0.154; data-to-parameter ratio = 15.2.

In the title compound, $C_{15}H_{12}ClN_3O_2$, the imidazolidine ring system is almost planar; the dihedral angles between it and the phenyl and pyridine rings are 74.44 (12) and 83.75 (12)°, respectively. The structure is stabilized by weak C—H···O hydrogen bonds, as well as C—H···π interactions.

Related literature

Many derivatives of imidazolidine have been prepared; biological and pharmaceutical activities have been studied by Lee *et al.* (1997) and Hirai *et al.* (1999).

For related literature, see: Sasada (1984); Wang *et al.* (1998).



Experimental

Crystal data

$C_{15}H_{12}ClN_3O_2$
M_r = 301.73
Monoclinic, $P2_1/n$
a = 7.7106 (10) Å
b = 6.3329 (9) Å
c = 28.673 (4) Å
β = 91.552 (2)°
V = 1399.6 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.28 mm^{-1}
T = 297 (2) K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.921, T_{max} = 0.946
7874 measured reflections
2882 independent reflections
2353 reflections with $I > 2\sigma(I)$
R_{int} = 0.087

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.154
S = 1.06
2882 reflections
190 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

*Cg*1 is the centroid of the C10–C13/C14/C15 ring.

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11···O2^{i}	0.93	2.53	3.205 (3)	129
C14—H14···O1^{ii}	0.93	2.44	3.221 (3)	142
C6—H6*A*···*Cg*1^{iii}	0.97	2.80	3.751 (3)	168
C15—H15···*Cg*1^{ii}	0.97	2.90	3.637 (3)	137

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); 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: AT2329).

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supplementary materials

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1-[(6-Chloropyridin-3-yl)methyl]-3-phenylimidazolidine-2,4-dione

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Comment

Imidazolidine-2,4-diones became of recent importance in the research and development of agrochemicals due to their wide-spread biological activites. Some of them have been used as herbicides, fungicides, *etc*. (Lee *et al.*, 1997; Hirai *et al.*, 1999). Neonicotinoid insecticides as nicotinic acetylcholine receptor inhibitors have attracted increasing attention because of their safety, low toxicity, wide range of activities and high potency. It has been found that most biologically active nicotinic compounds contain the (aminomethyl)-pyridine moiety. As a continuation of our search for new biologically active heterocyclic compounds, we report here the crystal structure of the title compound, (I) (Fig.1).

Selected bonds in (I) are listed in Table 1. In the crystal structure, the C7—N2, C8—N3, C9—N2 and C9—N3 bonds are significantly shorter than a normal single C—N bond (1.47 Å; Sasada, 1984) and close to the value for a C═N bond (1.28 Å; Wang *et al.*, 1998), which is indicative of signficant double-bond character. Weak C—H···O hydrogen bond (Table 2) and C—H···π interactions contribute to the stability of the crystal structure.

Experimental

The mixture of 3-phenylimidazolidine-2,4-dione (0.53 g, 3 mmol) and potassium carbonate (0.41 g, 3 mmol) dissolved in 10 ml of acetonitrile was stirred magnetically for 10–15 min. Then the solution of 2-chloro-5-(chloromethyl)pyridine (3 mmol, 0.49 g) in 10 ml of acetonitrile was added dropwise at 273 K for 30 min. The mixture was stirred for 1 h at room temperature, then refluxed for 3 h. The workup involved stripping of the solvent followed by an addition of water and extraction of the product mixture into dichloromethane, after phase separation, drying over anhydrous sodium sulfate, filtration and evaporation, the crude product was recrystallized from ethyl acetate and petroleum etherto give the product as colourless crystals; yield 57%; m.p. 377–379 K.

Refinement

H atoms bonded to C were placed at calculated positions, with C—H distances of 0.97 and 0.93 Å for H atoms bonded to sp^3 and sp^2 C atoms, respectively. They were refined using a riding model, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

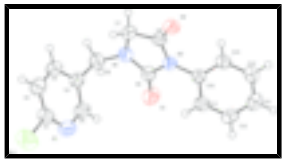


Fig. 1. View of the molecular structure of (I), showing the atom labelling scheme and with displacement ellipsoids drawn at the 50% probability level.

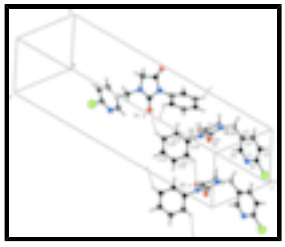


Fig. 2. A partial view of the crystal packing of (I), showing the formation of C—H···O hydrogen-bonded and C—H···π packing interactions, showing as dashed lines.

1-[(6-Chloropyridin-3-yl)methyl]-3-phenylimidazolidine-2,4-dione

Crystal data

$C_{15}H_{12}ClN_3O_2$	F_{000} = 624
M_r = 301.73	D_x = 1.432 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 3094 reflections
a = 7.7106 (10) Å	θ = 2.7–27.0º
b = 6.3329 (9) Å	μ = 0.28 mm^{-1}
c = 28.673 (4) Å	T = 297 (2) K
β = 91.552 (2)º	Block, colourless
V = 1399.6 (3) Å^3	0.30 × 0.20 × 0.20 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	2882 independent reflections
Radiation source: fine-focus sealed tube	2353 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.087
T = 297(2) K	θ_{max} = 26.5º
φ and ω scans	θ_{min} = 2.7º
Absorption correction: multi-scan ?	h = −9→9
T_{min} = 0.921, T_{max} = 0.946	k = −7→7
7874 measured reflections	l = −33→35

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.056	H-atom parameters constrained
$wR(F^2)$ = 0.154	$w = 1/[\sigma^2(F_o^2) + (0.0605P)^2 + 0.5771P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.06	$(\Delta/\sigma)_{max} < 0.001$
2882 reflections	$\Delta\rho_{max}$ = 0.27 e Å^{-3}

190 parameters	$\Delta\rho_{min} = -0.23$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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	0.2372 (3)	0.5225 (4)	0.99690 (8)	0.0533 (6)
C2	0.2084 (3)	0.7347 (4)	1.00312 (9)	0.0587 (7)
H2	0.1893	0.7905	1.0325	0.070*
C3	0.2090 (3)	0.8609 (4)	0.96422 (9)	0.0537 (6)
H3	0.1930	1.0059	0.9670	0.064*
C4	0.2335 (3)	0.7713 (4)	0.92095 (8)	0.0428 (5)
C5	0.2527 (3)	0.5546 (4)	0.91929 (8)	0.0541 (6)
H5	0.2632	0.4920	0.8902	0.065*
C6	0.2454 (3)	0.9009 (4)	0.87684 (8)	0.0479 (5)
H6A	0.1835	0.8290	0.8516	0.057*
H6B	0.1900	1.0366	0.8814	0.057*
C7	0.5401 (3)	1.0844 (4)	0.88673 (9)	0.0479 (5)
H7A	0.5344	1.0733	0.9204	0.057*
H7B	0.5117	1.2278	0.8774	0.057*
C8	0.7167 (3)	1.0213 (3)	0.87015 (7)	0.0406 (5)
C9	0.5137 (3)	0.7895 (4)	0.83985 (8)	0.0428 (5)
C10	0.8204 (3)	0.7462 (3)	0.81482 (7)	0.0367 (5)
C11	0.8776 (3)	0.5483 (3)	0.82859 (8)	0.0452 (5)
H11	0.8337	0.4837	0.8549	0.054*
C12	1.0016 (3)	0.4480 (4)	0.80236 (9)	0.0510 (6)
H12	1.0413	0.3148	0.8112	0.061*
C13	1.0665 (3)	0.5432 (4)	0.76354 (8)	0.0514 (6)
H13	1.1500	0.4745	0.7463	0.062*
C14	1.0083 (3)	0.7397 (4)	0.75001 (8)	0.0494 (6)
H14	1.0524	0.8038	0.7237	0.059*
C15	0.8838 (3)	0.8422 (4)	0.77574 (8)	0.0431 (5)
H15	0.8434	0.9747	0.7666	0.052*
Cl1	0.24657 (13)	0.35783 (14)	1.04586 (3)	0.0857 (3)
N1	0.2570 (3)	0.4292 (3)	0.95662 (7)	0.0618 (6)
N2	0.4254 (2)	0.9345 (3)	0.86390 (6)	0.0447 (5)

N3	0.6895 (2)	0.8510 (3)	0.84105 (6)	0.0392 (4)
O1	0.4589 (2)	0.6335 (3)	0.82032 (7)	0.0632 (5)
O2	0.8541 (2)	1.1011 (3)	0.88035 (6)	0.0530 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0629 (15)	0.0527 (14)	0.0441 (13)	−0.0047 (11)	−0.0008 (11)	0.0072 (11)
C2	0.0751 (18)	0.0575 (16)	0.0437 (13)	−0.0037 (13)	0.0053 (12)	−0.0116 (11)
C3	0.0658 (15)	0.0410 (13)	0.0545 (14)	−0.0010 (11)	0.0071 (12)	−0.0083 (11)
C4	0.0385 (11)	0.0436 (12)	0.0464 (12)	0.0019 (9)	0.0023 (9)	−0.0044 (9)
C5	0.0758 (17)	0.0441 (13)	0.0424 (12)	0.0036 (12)	0.0053 (11)	−0.0072 (10)
C6	0.0429 (12)	0.0516 (13)	0.0492 (13)	0.0053 (10)	0.0011 (10)	0.0053 (10)
C7	0.0519 (13)	0.0377 (11)	0.0543 (13)	0.0007 (10)	0.0053 (10)	−0.0050 (10)
C8	0.0499 (12)	0.0316 (10)	0.0404 (11)	−0.0004 (9)	0.0015 (9)	0.0035 (9)
C9	0.0422 (11)	0.0474 (12)	0.0388 (11)	−0.0012 (9)	0.0006 (9)	−0.0013 (10)
C10	0.0360 (10)	0.0366 (11)	0.0373 (10)	−0.0022 (8)	−0.0017 (8)	−0.0030 (8)
C11	0.0552 (13)	0.0395 (12)	0.0408 (12)	−0.0004 (10)	0.0013 (10)	0.0034 (9)
C12	0.0567 (14)	0.0399 (12)	0.0560 (14)	0.0107 (10)	−0.0047 (11)	−0.0009 (10)
C13	0.0485 (13)	0.0599 (15)	0.0456 (13)	0.0069 (11)	0.0007 (10)	−0.0129 (11)
C14	0.0457 (12)	0.0615 (15)	0.0413 (12)	−0.0044 (11)	0.0049 (9)	0.0039 (11)
C15	0.0479 (12)	0.0392 (12)	0.0419 (12)	0.0000 (9)	−0.0012 (9)	0.0052 (9)
Cl1	0.1187 (7)	0.0829 (6)	0.0553 (5)	−0.0030 (5)	−0.0017 (4)	0.0236 (4)
N1	0.0920 (17)	0.0422 (11)	0.0513 (12)	0.0049 (11)	0.0054 (11)	0.0003 (9)
N2	0.0439 (10)	0.0453 (10)	0.0452 (10)	−0.0003 (8)	0.0036 (8)	−0.0028 (8)
N3	0.0399 (9)	0.0372 (9)	0.0406 (9)	−0.0015 (7)	0.0012 (7)	−0.0038 (7)
O1	0.0538 (10)	0.0663 (12)	0.0697 (12)	−0.0135 (8)	0.0043 (9)	−0.0294 (10)
O2	0.0480 (9)	0.0454 (9)	0.0657 (11)	−0.0098 (7)	0.0012 (8)	−0.0099 (8)

Geometric parameters (Å, °)

C1—N1	1.310 (3)	C8—O2	1.203 (3)
C1—C2	1.374 (4)	C8—N3	1.376 (3)
C1—Cl1	1.749 (2)	C9—O1	1.206 (3)
C2—C3	1.372 (4)	C9—N2	1.345 (3)
C2—H2	0.9300	C9—N3	1.410 (3)
C3—C4	1.382 (3)	C10—C15	1.376 (3)
C3—H3	0.9300	C10—C11	1.382 (3)
C4—C5	1.382 (3)	C10—N3	1.438 (3)
C4—C6	1.512 (3)	C11—C12	1.387 (3)
C5—N1	1.332 (3)	C11—H11	0.9300
C5—H5	0.9300	C12—C13	1.372 (3)
C6—N2	1.462 (3)	C12—H12	0.9300
C6—H6A	0.9700	C13—C14	1.375 (4)
C6—H6B	0.9700	C13—H13	0.9300
C7—N2	1.443 (3)	C14—C15	1.388 (3)
C7—C8	1.508 (3)	C14—H14	0.9300
C7—H7A	0.9700	C15—H15	0.9300
C7—H7B	0.9700		

N1—C1—C2	125.3 (2)	N3—C8—C7	105.94 (18)
N1—C1—Cl1	115.8 (2)	O1—C9—N2	128.4 (2)
C2—C1—Cl1	118.8 (2)	O1—C9—N3	124.2 (2)
C3—C2—C1	117.4 (2)	N2—C9—N3	107.36 (18)
C3—C2—H2	121.3	C15—C10—C11	121.0 (2)
C1—C2—H2	121.3	C15—C10—N3	119.45 (19)
C2—C3—C4	119.6 (2)	C11—C10—N3	119.49 (19)
C2—C3—H3	120.2	C10—C11—C12	118.7 (2)
C4—C3—H3	120.2	C10—C11—H11	120.7
C5—C4—C3	117.1 (2)	C12—C11—H11	120.7
C5—C4—C6	120.1 (2)	C13—C12—C11	120.7 (2)
C3—C4—C6	122.8 (2)	C13—C12—H12	119.6
N1—C5—C4	124.4 (2)	C11—C12—H12	119.6
N1—C5—H5	117.8	C12—C13—C14	120.2 (2)
C4—C5—H5	117.8	C12—C13—H13	119.9
N2—C6—C4	111.77 (18)	C14—C13—H13	119.9
N2—C6—H6A	109.3	C13—C14—C15	119.9 (2)
C4—C6—H6A	109.3	C13—C14—H14	120.0
N2—C6—H6B	109.3	C15—C14—H14	120.0
C4—C6—H6B	109.3	C10—C15—C14	119.5 (2)
H6A—C6—H6B	107.9	C10—C15—H15	120.3
N2—C7—C8	103.32 (18)	C14—C15—H15	120.3
N2—C7—H7A	111.1	C1—N1—C5	116.0 (2)
C8—C7—H7A	111.1	C9—N2—C7	111.67 (18)
N2—C7—H7B	111.1	C9—N2—C6	121.88 (19)
C8—C7—H7B	111.1	C7—N2—C6	123.74 (19)
H7A—C7—H7B	109.1	C8—N3—C9	111.24 (17)
O2—C8—N3	126.5 (2)	C8—N3—C10	125.55 (17)
O2—C8—C7	127.5 (2)	C9—N3—C10	123.20 (17)
N1—C1—C2—C3	−3.4 (4)	C4—C5—N1—C1	1.7 (4)
Cl1—C1—C2—C3	177.4 (2)	O1—C9—N2—C7	173.2 (2)
C1—C2—C3—C4	1.6 (4)	N3—C9—N2—C7	−6.8 (3)
C2—C3—C4—C5	1.4 (4)	O1—C9—N2—C6	11.2 (4)
C2—C3—C4—C6	−176.6 (2)	N3—C9—N2—C6	−168.84 (19)
C3—C4—C5—N1	−3.2 (4)	C8—C7—N2—C9	4.3 (3)
C6—C4—C5—N1	174.8 (2)	C8—C7—N2—C6	165.91 (19)
C5—C4—C6—N2	−79.0 (3)	C4—C6—N2—C9	83.1 (3)
C3—C4—C6—N2	98.9 (3)	C4—C6—N2—C7	−76.7 (3)
N2—C7—C8—O2	−179.4 (2)	O2—C8—N3—C9	175.4 (2)
N2—C7—C8—N3	0.0 (2)	C7—C8—N3—C9	−4.1 (2)
C15—C10—C11—C12	0.5 (3)	O2—C8—N3—C10	−4.7 (3)
N3—C10—C11—C12	178.9 (2)	C7—C8—N3—C10	175.93 (19)
C10—C11—C12—C13	0.0 (3)	O1—C9—N3—C8	−173.1 (2)
C11—C12—C13—C14	−0.3 (4)	N2—C9—N3—C8	6.8 (2)
C12—C13—C14—C15	0.1 (4)	O1—C9—N3—C10	6.9 (3)
C11—C10—C15—C14	−0.7 (3)	N2—C9—N3—C10	−173.14 (18)
N3—C10—C15—C14	−179.06 (19)	C15—C10—N3—C8	−74.4 (3)
C13—C14—C15—C10	0.4 (3)	C11—C10—N3—C8	107.2 (2)

C2—C1—N1—C5	1.8 (4)	C15—C10—N3—C9	105.5 (2)
Cl1—C1—N1—C5	−179.0 (2)	C11—C10—N3—C9	−72.9 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11···O2^{i}	0.93	2.53	3.205 (3)	129
C14—H14···O1^{ii}	0.93	2.44	3.221 (3)	142
C6—H6A···Cg1^{iii}	0.97	2.80	3.751 (3)	168
C15—H15···Cg1^{ii}	0.97	2.90	3.637 (3)	137

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

Fig. 1

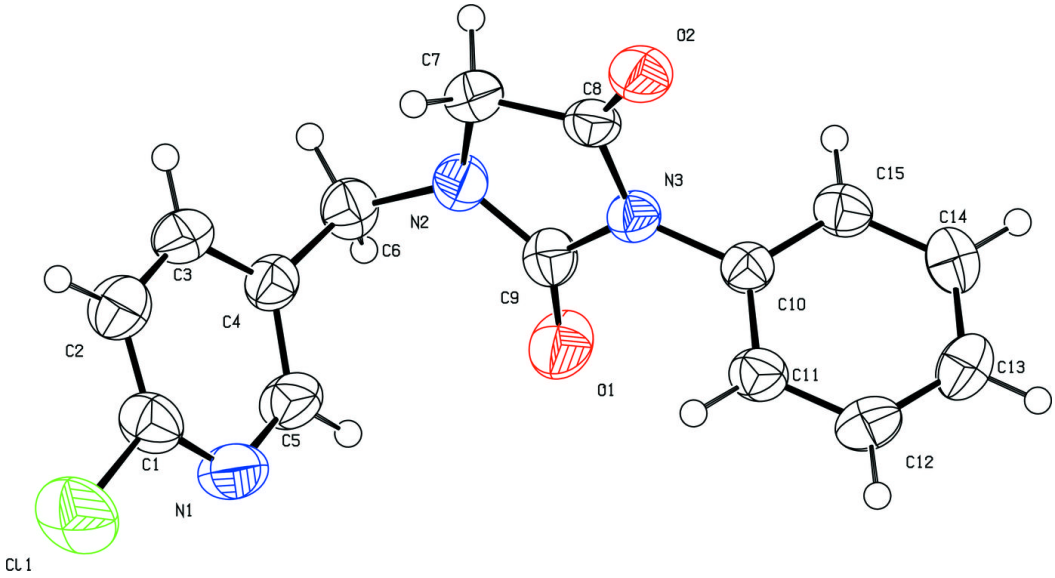
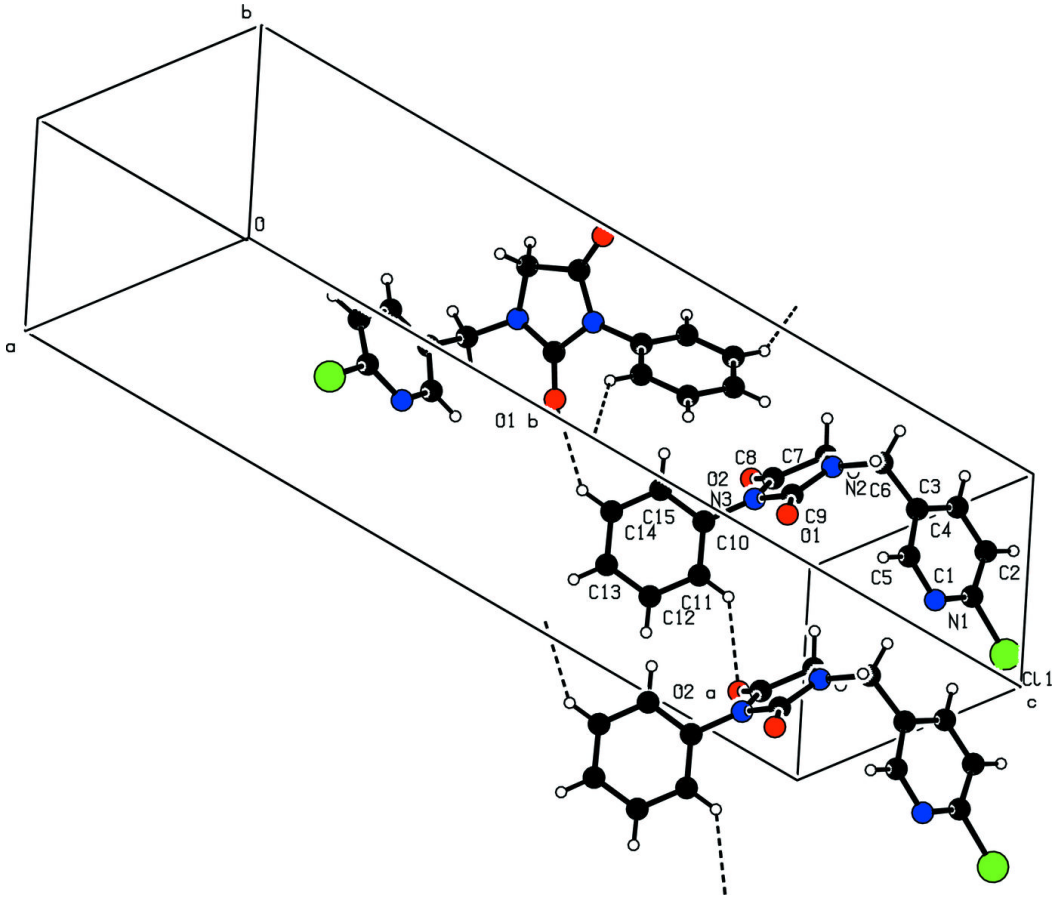


Fig. 2


b
0
a
01 b
C8
C7
02
N3
N2
C6
C3
C9
01
C4
C15
C14
C10
C5
C1
C2
C13
C11
C12
N1
02 a
Cl 1
c