IUCrData

ISSN 2414-3146

5-(5-Chloro-2-hydroxybenzoyl)-1-methyl-3-nitropyridin-2(1*H*)-one

Srinivasan Bargavi,[a] Nataraj Poomathi,[b] Narayanan Uma Maheswari[b] and Srinivasakannan Lakshmi[a]*

[a]Department of Physics, S.D.N.B. Vaishnav College for Women, Chromepet, Chennai 600 044, India, and [b]Organic Chemistry Division, CSIR Central Leather Research Institute, Chennai 600 020, India. *Correspondence e-mail: lakssdnbvc@gmail.com

Received 13 September 2017
Accepted 20 September 2017

Edited by K. Fejfarova, Institute of Biotechnology CAS, Czech Republic

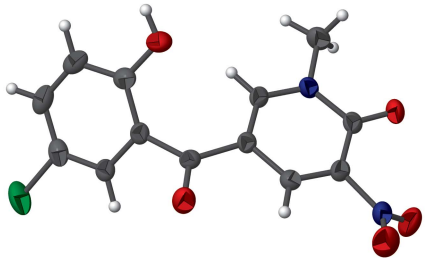
Keywords: crystal structure; nitropyridine; hydrogen bonding; framework; π–π interactions.

CCDC reference: 1575441

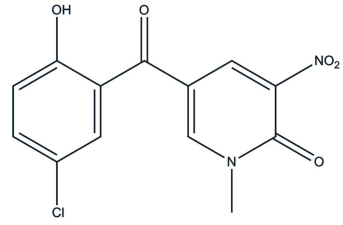
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{13}H_9ClN_2O_5$, the dihedral angle between the planes of the benzene and pyridine rings is 55.30 (13)°. The nitro group is tilted by 38.21 (10)° with respect to the mean plane of the pyridine ring. In the crystal, molecules are linked by O—H···O and C—H···O hydrogen bonds, forming a three-dimensional framework. The crystal packing is further stabilized by π–π stacking interactions [intercentroid distance = 3.5877 (17) Å].

3D view



Chemical scheme



Structure description

The pyridine moiety has profound importance in the fields of chemistry and biology (Ghosh *et al.*, 2014). Compounds that contain pyridine and its derivatives have occupied a central role in the development of coordination chemistry and biochemistry (Rajeswar *et al.*, 2014). Heterocycles are important molecular building blocks that are involved in the structural composition of crucial chemicals for humans, including pharmaceuticals, natural resources, veterinary, agricultural products, analytical reagents and dyes (Göktaş *et al.*, 2014). In drug discovery, pyridine has been used as a bioisosteric replacement of the benzene ring (Ajit Kumar *et al.*, 2011). Pyridine derivatives of different heterocyclic nuclei have shown important pharmacological properties such as anticancer (Abbas *et al.*, 2015), antimicrobial (Hussein *et al.*, 2014), antibacterial (Rani *et al.*, 2012), antimycobacterial (Banfi *et al.*, 2001) and antioxidant activities (Fadda *et al.*, 2012).

In the title compound (Fig. 1), the dihedral angle between the benzene (C1–C6) and pyridine (N1/C8–C12) rings is 55.30 (13)°. The nitro group is tilted by 38.21 (10)° with respect to the mean plane of the pyridine ring. The chlorine atom Cl1 deviates from the plane of the benzene ring by 0.009 (1) Å. The Cl1—C2—C3—C4 torsion angle of 179.7 (2)° indicates that the chlorine atom Cl1 is not quite coplanar with the phenol ring.

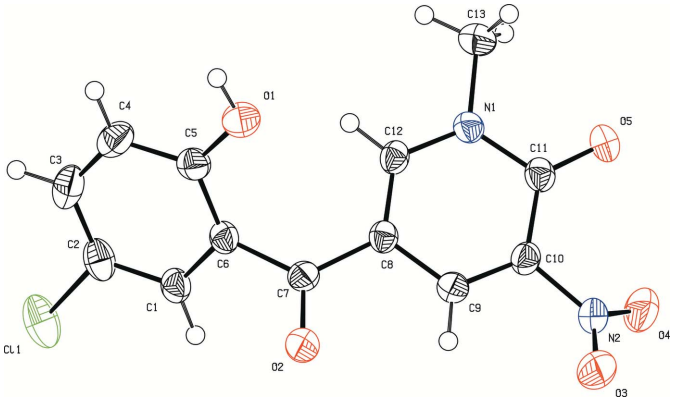


Figure 1
The molecular structure of the title compound, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

In the crystal, molecules are linked by O1—H1···O4 and O1—H1···O5 hydrogen bonds (Table 1), forming chains, which are further linked by C1—H1*A*···O4 and C12—H12···O2 hydrogen bonds, forming a two-dimensional network parallel to (100) (Fig. 2). The crystal packing also features π–π stacking interactions [$Cg1\cdots Cg2^{i}$ = 3.5877 (17) Å, interplanar distance = 3.3360 (11) Å, *Cg*1 and *Cg*2 are the centroids of rings N1/C8–C12 and C1–C6, respectively; symmetry code: (i) $2-x, -y, -\frac{1}{2}+z$].

Synthesis and crystallization

A mixture of 6-chloro-3-formylchromone (1 mmol), (*Z*)-*N*-methyl-1-(methylthio)-2-nitroethenamine (1 mmol), and indium trifluoromethanesulfonate (0.020 mmol) in ethanol (3 ml) were charged in a 25 ml round-bottomed flask and the mixture was heated at reflux. The resulting solution was stirred for 1h. The consumption of the starting material was monitored by TLC. After completion of the reaction, the product was filtered, washed with ethanol, dried under vacuum and the pure product obtained in good yield (88%). The purified compound was recrystallized from ethanol and DMSO-d_6 by using the slow evaporation method.

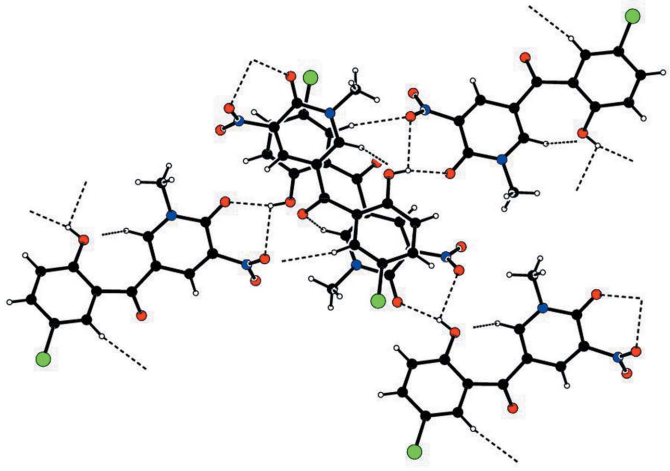


Figure 2
Hydrogen-bonding network, viewed along the *a* axis. Hydrogen bonds (see Table 1) are shown as dashed lines.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O4^{i}	0.82	2.40	2.774 (3)	109
O1—H1···O5^{i}	0.82	2.05	2.820 (3)	157
C1—H1*A*···O4^{ii}	0.93	2.49	3.412 (3)	169
C12—H12···O2^{iii}	0.93	2.56	3.111 (4)	118

Symmetry codes: (i) $-x+\frac{5}{2}, y+\frac{1}{2}, z+\frac{1}{2}$; (ii) $x-\frac{1}{2}, -y-\frac{1}{2}, z$; (iii) $-x+2, -y, z-\frac{1}{2}$.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

The authors thank the Department of Chemistry, IIT, Chennai, for the data collection.

References

Abbas, I., Gomha, S., Elaasser, M. & Bauomi, M. (2015). *Turk. J. Chem.* **39**, 334–346.
Ajit Kumar, C. & Pandeya, S. N. (2011). *Int. J. Res. Ayurveda. Pharm.* **2**, 1763–1767.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_9ClN_2O_5$
M_r	308.67
Crystal system, space group	Orthorhombic, $Pna2_1$
Temperature (K)	296
a, *b*, *c* (Å)	11.1491 (4), 15.0153 (6), 7.7045 (3)
V (Å^3)	1289.79 (9)
Z	4
Radiation type	Mo *K*α
μ (mm^{-1})	0.32
Crystal size (mm)	0.30 × 0.25 × 0.20
Data collection	
Diffractometer	Bruker Kappa APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2008)
T_{min}, T_{max}	0.908, 0.938
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	4752, 1705, 1614
R_{int}	0.015
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.594
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.026, 0.070, 1.06
No. of reflections	1705
No. of parameters	191
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.12, −0.21
Absolute structure	Flack (1983), 481 Friedel pairs
Absolute structure parameter	0.59 (3)

Computer programs: *APEX2* and *SAINT* (Bruker, 2008), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3* for Windows (Farrugia, 2012), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

Banfi, E., Mamolo, M. G., Zampieri, D., Vio, L. & Bragadin, C. M. (2001). *J. Antimicrob. Chemother.* **48**, 705–707.
Bruker (2008). *APEX2*, *SADABS* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Fadda, A. A., Sanad, M. & Abd El-Galil, I. (2012). *Int. J. Modern Org. Chem.* **1**, 136–149.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Ghosh, P. S., Manna, K., Banik, U., Das, M. & Sarkar, P. (2014). *Int. J. Pharm. Pharm. Sci.* **6**, 39–42.
Göktaş, F., Cesur, N., Şatana, D. & Uzun, M. (2014). *Turk. J. Chem.* **38**, 581–591.
Hussein, A. M., El-Adasy, A. A., Hafi, I. A., Ishak, E. A., Gawish, E. H. & El-Gaby, M. A. (2014). *J. App. Pharm.* **6**, 296–307.
Rajeswar, V. R., Dharmale, M. K. & Pingalkar, S. R. (2014). *Int. J. Curr. Res. Chem. Pharma. Sci.* **1**, 40–50.
Rani, E. S., Parameshwar, R., Babu, V. H., Ranganath, Y. S., Kumar, B. N. & Kumar, G. A. (2012). *Int. J. Pharm. Pharm. Sci.* **4**, 424–427.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

full crystallographic data

IUCrData (2017). **2**, x171345 [https://doi.org/10.1107/S2414314617013451]

5-(5-Chloro-2-hydroxybenzoyl)-1-methyl-3-nitropyridin-2(1*H*)-one

Srinivasan Bargavi, Nataraj Poomathi, Narayanan Uma Maheswari and Srinivasakannan Lakshmi

5-(5-Chloro-2-hydroxybenzoyl)-1-methyl-3-nitropyridin-2(1*H*)-one

Crystal data

$C_{13}H_9ClN_2O_5$
M_r = 308.67
Orthorhombic, $Pna2_1$
a = 11.1491 (4) Å
b = 15.0153 (6) Å
c = 7.7045 (3) Å
V = 1289.79 (9) Å^3
Z = 4
$F(000)$ = 632
D_x = 1.590 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1614 reflections
θ = 2.3–25.0°
μ = 0.32 mm^{-1}
T = 296 K
Block, colourless
0.30 × 0.25 × 0.20 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
ω and φ scans
Absorption correction: multi-scan (SADABS; Bruker, 2008)
T_{min} = 0.908, T_{max} = 0.938
4752 measured reflections
1705 independent reflections
1614 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
θ_{max} = 25.0°, θ_{min} = 2.3°
h = −13→12
k = −17→10
l = −5→9

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.070
S = 1.06
1705 reflections
191 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0449P)^2 + 0.1924P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Absolute structure: Flack (1983), 481 Friedel pairs
Absolute structure parameter: 0.59 (3)

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}
Cl1	0.63594 (6)	0.15295 (6)	0.00210 (13)	0.0547 (3)
C9	1.0658 (2)	−0.18373 (17)	0.2334 (4)	0.0292 (6)
H9	1.0034	−0.2146	0.2864	0.035*
O5	1.35111 (14)	−0.22295 (12)	0.0251 (3)	0.0389 (5)
C8	1.0640 (2)	−0.09006 (17)	0.2281 (4)	0.0262 (5)
N1	1.25635 (18)	−0.09334 (14)	0.0949 (3)	0.0290 (5)
C12	1.1613 (2)	−0.04804 (17)	0.1581 (4)	0.0278 (5)
H12	1.1621	0.0138	0.1538	0.033*
O4	1.1848 (2)	−0.36386 (13)	0.0251 (4)	0.0543 (6)
C13	1.3620 (2)	−0.04375 (19)	0.0311 (5)	0.0442 (8)
H13A	1.3441	0.0188	0.0289	0.066*
H13B	1.3816	−0.0635	−0.0839	0.066*
H13C	1.4288	−0.0543	0.1069	0.066*
O2	0.87647 (16)	−0.08953 (12)	0.3612 (3)	0.0433 (5)
C11	1.2624 (2)	−0.18667 (17)	0.0867 (3)	0.0294 (6)
O1	1.09214 (16)	0.11145 (13)	0.3731 (3)	0.0415 (5)
H1	1.1227	0.1599	0.3941	0.062*
N2	1.14859 (19)	−0.32526 (15)	0.1549 (4)	0.0371 (6)
C2	0.7722 (2)	0.1431 (2)	0.1122 (4)	0.0360 (7)
C4	0.9437 (3)	0.20821 (18)	0.2460 (4)	0.0382 (7)
H4	0.9867	0.2584	0.2792	0.046*
C6	0.9226 (2)	0.04896 (16)	0.2385 (3)	0.0277 (6)
C7	0.9520 (2)	−0.04566 (17)	0.2865 (4)	0.0278 (6)
C10	1.1577 (2)	−0.22868 (16)	0.1617 (4)	0.0288 (6)
O3	1.0998 (2)	−0.36325 (13)	0.2761 (4)	0.0542 (6)
C1	0.8133 (2)	0.05953 (17)	0.1519 (4)	0.0308 (6)
H1A	0.7685	0.0098	0.1212	0.037*
C5	0.9880 (2)	0.12375 (17)	0.2852 (4)	0.0310 (6)
C3	0.8360 (3)	0.21806 (18)	0.1579 (4)	0.0416 (7)
H3	0.8073	0.2744	0.1301	0.050*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0456 (4)	0.0718 (5)	0.0467 (5)	0.0250 (4)	−0.0078 (4)	0.0019 (5)
C9	0.0263 (11)	0.0288 (13)	0.0325 (14)	−0.0007 (11)	−0.0017 (12)	−0.0002 (13)
O5	0.0297 (10)	0.0354 (10)	0.0517 (14)	0.0080 (7)	0.0065 (10)	−0.0029 (11)
C8	0.0268 (11)	0.0255 (13)	0.0264 (13)	0.0030 (10)	−0.0027 (11)	0.0012 (12)
N1	0.0252 (10)	0.0286 (11)	0.0332 (12)	0.0016 (9)	0.0005 (9)	0.0046 (10)

C12	0.0295 (12)	0.0246 (12)	0.0294 (13)	0.0036 (10)	−0.0054 (11)	0.0014 (12)
O4	0.0604 (13)	0.0333 (11)	0.0693 (18)	0.0061 (9)	0.0146 (14)	−0.0093 (13)
C13	0.0318 (13)	0.0399 (16)	0.061 (2)	−0.0034 (11)	0.0084 (14)	0.0076 (18)
O2	0.0364 (10)	0.0330 (11)	0.0604 (14)	0.0012 (8)	0.0142 (10)	0.0032 (11)
C11	0.0273 (13)	0.0304 (13)	0.0305 (13)	0.0042 (11)	−0.0046 (11)	0.0007 (12)
O1	0.0362 (10)	0.0371 (10)	0.0513 (13)	−0.0051 (9)	−0.0084 (10)	−0.0106 (11)
N2	0.0312 (12)	0.0285 (12)	0.0515 (16)	0.0056 (10)	0.0002 (12)	0.0020 (13)
C2	0.0367 (15)	0.0445 (16)	0.0266 (15)	0.0128 (13)	0.0022 (12)	−0.0006 (12)
C4	0.0492 (16)	0.0279 (13)	0.0376 (17)	−0.0043 (12)	0.0070 (14)	−0.0041 (13)
C6	0.0274 (12)	0.0277 (13)	0.0281 (14)	0.0033 (10)	0.0037 (11)	−0.0027 (12)
C7	0.0273 (12)	0.0266 (12)	0.0294 (14)	−0.0022 (10)	−0.0009 (11)	−0.0044 (11)
C10	0.0300 (13)	0.0244 (13)	0.0320 (14)	0.0026 (10)	−0.0028 (11)	0.0026 (12)
O3	0.0534 (13)	0.0370 (11)	0.0720 (17)	0.0006 (10)	0.0165 (13)	0.0192 (12)
C1	0.0298 (13)	0.0302 (14)	0.0322 (14)	0.0040 (11)	0.0024 (12)	−0.0061 (12)
C5	0.0308 (13)	0.0328 (14)	0.0295 (14)	−0.0021 (11)	0.0056 (12)	−0.0050 (12)
C3	0.0569 (18)	0.0295 (15)	0.0383 (16)	0.0105 (13)	0.0091 (15)	0.0011 (14)

Geometric parameters (Å, º)

Cl1—C2	1.746 (3)	C11—C10	1.447 (4)
C9—C10	1.346 (4)	O1—C5	1.357 (3)
C9—C8	1.407 (4)	O1—H1	0.8200
C9—H9	0.9300	N2—O3	1.222 (4)
O5—C11	1.225 (3)	N2—C10	1.455 (3)
C8—C12	1.366 (3)	C2—C1	1.371 (4)
C8—C7	1.485 (3)	C2—C3	1.377 (4)
N1—C12	1.350 (3)	C4—C3	1.387 (4)
N1—C11	1.404 (3)	C4—C5	1.394 (4)
N1—C13	1.477 (3)	C4—H4	0.9300
C12—H12	0.9300	C6—C5	1.386 (4)
O4—N2	1.224 (3)	C6—C1	1.398 (4)
C13—H13A	0.9600	C6—C7	1.504 (3)
C13—H13B	0.9600	C1—H1A	0.9300
C13—H13C	0.9600	C3—H3	0.9300
O2—C7	1.214 (3)		
C10—C9—C8	120.0 (2)	C1—C2—C3	121.2 (3)
C10—C9—H9	120.0	C1—C2—Cl1	118.5 (2)
C8—C9—H9	120.0	C3—C2—Cl1	120.3 (2)
C12—C8—C9	117.5 (2)	C3—C4—C5	120.6 (3)
C12—C8—C7	125.4 (2)	C3—C4—H4	119.7
C9—C8—C7	116.9 (2)	C5—C4—H4	119.7
C12—N1—C11	123.8 (2)	C5—C6—C1	119.3 (2)
C12—N1—C13	119.5 (2)	C5—C6—C7	126.0 (2)
C11—N1—C13	116.7 (2)	C1—C6—C7	114.5 (2)
N1—C12—C8	122.2 (2)	O2—C7—C8	118.9 (2)
N1—C12—H12	118.9	O2—C7—C6	118.6 (2)
C8—C12—H12	118.9	C8—C7—C6	122.1 (2)

N1—C13—H13A	109.5	C9—C10—C11	124.0 (2)
N1—C13—H13B	109.5	C9—C10—N2	117.5 (2)
H13A—C13—H13B	109.5	C11—C10—N2	118.5 (2)
N1—C13—H13C	109.5	C2—C1—C6	120.1 (2)
H13A—C13—H13C	109.5	C2—C1—H1A	119.9
H13B—C13—H13C	109.5	C6—C1—H1A	119.9
O5—C11—N1	120.0 (2)	O1—C5—C6	118.0 (2)
O5—C11—C10	127.7 (2)	O1—C5—C4	122.3 (2)
N1—C11—C10	112.2 (2)	C6—C5—C4	119.6 (3)
C5—O1—H1	109.5	C2—C3—C4	119.0 (2)
O3—N2—O4	123.4 (2)	C2—C3—H3	120.5
O3—N2—C10	118.0 (3)	C4—C3—H3	120.5
O4—N2—C10	118.6 (3)		

C10—C9—C8—C12	−3.9 (4)	N1—C11—C10—C9	−0.9 (4)
C10—C9—C8—C7	171.3 (3)	O5—C11—C10—N2	−4.9 (4)
C11—N1—C12—C8	3.3 (4)	N1—C11—C10—N2	177.1 (2)
C13—N1—C12—C8	−176.1 (3)	O3—N2—C10—C9	−35.8 (4)
C9—C8—C12—N1	0.3 (4)	O4—N2—C10—C9	140.7 (3)
C7—C8—C12—N1	−174.6 (2)	O3—N2—C10—C11	146.1 (3)
C12—N1—C11—O5	178.9 (3)	O4—N2—C10—C11	−37.4 (4)
C13—N1—C11—O5	−1.6 (3)	C3—C2—C1—C6	−0.8 (4)
C12—N1—C11—C10	−2.9 (3)	Cl1—C2—C1—C6	179.4 (2)
C13—N1—C11—C10	176.6 (2)	C5—C6—C1—C2	0.7 (4)
C12—C8—C7—O2	−173.3 (3)	C7—C6—C1—C2	175.3 (2)
C9—C8—C7—O2	11.9 (4)	C1—C6—C5—O1	178.3 (2)
C12—C8—C7—C6	13.9 (4)	C7—C6—C5—O1	4.4 (4)
C9—C8—C7—C6	−160.9 (2)	C1—C6—C5—C4	0.3 (4)
C5—C6—C7—O2	124.2 (3)	C7—C6—C5—C4	−173.6 (2)
C1—C6—C7—O2	−49.9 (3)	C3—C4—C5—O1	−179.1 (3)
C5—C6—C7—C8	−62.9 (4)	C3—C4—C5—C6	−1.2 (4)
C1—C6—C7—C8	122.9 (3)	C1—C2—C3—C4	−0.1 (4)
C8—C9—C10—C11	4.3 (4)	Cl1—C2—C3—C4	179.7 (2)
C8—C9—C10—N2	−173.7 (2)	C5—C4—C3—C2	1.1 (4)
O5—C11—C10—C9	177.1 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O4^{i}	0.82	2.40	2.774 (3)	109
O1—H1···O5^{i}	0.82	2.05	2.820 (3)	157
C1—H1*A*···O4^{ii}	0.93	2.49	3.412 (3)	169
C12—H12···O2^{iii}	0.93	2.56	3.111 (4)	118

Symmetry codes: (i) −*x*+5/2, *y*+1/2, *z*+1/2; (ii) *x*−1/2, −*y*−1/2, *z*; (iii) −*x*+2, −*y*, *z*−1/2.