

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

## 3-Benzoyl-5-chlorouracil

Graeme J. Gainsford\* and Keith Clinch

Industrial Research Limited, PO Box 31-310, Lower Hutt, New Zealand

Correspondence e-mail: g.gainsford@irl.cri.nz

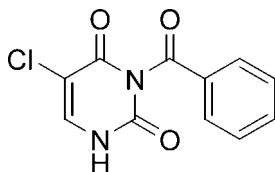
Received 4 January 2009; accepted 11 January 2009

Key indicators: single-crystal X-ray study;  $T = 133$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.041;  $wR$  factor = 0.110; data-to-parameter ratio = 17.9.

The dihedral angle between the planes of two aromatic rings in the title compound [systematic name: 3-benzoyl-5-chloropyrimidine-2,4(1*H*,3*H*)-dione],  $\text{C}_{11}\text{H}_7\text{ClN}_2\text{O}_3$ , is  $86.79$  (6)°. Centrosymmetric dimers formed by  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds are linked through  $\text{C}-\text{H}\cdots\text{O}$  interactions, forming a two-dimensional network parallel to (10 $\bar{1}$ ).

## Related literature

For a related structure, see: Parvez *et al.* (2007). For graph-set notation, see: Bernstein *et al.* (1995). For the synthesis, see: Birck *et al.* (2009).



## Experimental

## Crystal data

 $\text{C}_{11}\text{H}_7\text{ClN}_2\text{O}_3$  $M_r = 250.64$ Monoclinic,  $C2/c$  $a = 21.9357$  (9) Å $b = 5.4020$  (2) Å $c = 19.9642$  (9) Å $\beta = 113.169$  (2)° $V = 2174.89$  (16) Å<sup>3</sup> $Z = 8$ Mo  $K\alpha$  radiation $\mu = 0.35$  mm<sup>-1</sup> $T = 133$  (2) K $0.34 \times 0.21 \times 0.03$  mm

## Data collection

Bruker APEXII CCD area-detector diffractometer

Absorption correction: multi-scan

(SADABS; Blessing, 1995;

Bruker, 2006)

 $T_{\min} = 0.810$ ,  $T_{\max} = 0.990$ 

24616 measured reflections

2899 independent reflections

2189 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.047$ 

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.041$  $wR(F^2) = 0.110$  $S = 1.07$ 

2899 reflections

162 parameters

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.37$  e Å<sup>-3</sup> $\Delta\rho_{\text{min}} = -0.42$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O14}^i$	0.86 (3)	1.91 (3)	2.770 (2)	173 (3)
$\text{C9}-\text{H9}\cdots\text{O15}^{ii}$	0.95	2.46	3.182 (3)	133
$\text{C10}-\text{H10}\cdots\text{O15}^{iii}$	0.95	2.57	3.447 (3)	153

Symmetry codes: (i)  $-x + \frac{3}{2}, -y + \frac{5}{2}, -z + 1$ ; (ii)  $x, y + 1, z$ ; (iii)  $-x + 1, y + 1, -z + \frac{1}{2}$ .

Data collection: APEX2 (Bruker, 2006); cell refinement: SAINT (Bruker, 2006); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP in WinGX (Farrugia, 1999) and Mercury (Bruno *et al.*, 2002); software used to prepare material for publication: SHELXL97 and PLATON (Spek, 2003).

We thank Dr J. Wikaira and Dr C. Fitchett of the University of Canterbury, New Zealand, for their assistance with the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2757).

## References

- Bernstein, J., Davis, R. E., Shimon, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
- Birck, M. T., Clinch, K., Gainsford, G. J., Schramm, V. L. & Tyler, P. C. (2009). *Helv. Chim. Acta*. Submitted.
- Blessing, R. H. (1995). *Acta Cryst.* **A51**, 33–38.
- Bruker (2006). APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruno, I. J., Cole, J. C., Edgington, P. R., Kessler, M., Macrae, C. F., McCabe, P., Pearson, J. & Taylor, R. (2002). *Acta Cryst.* **B58**, 389–397.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Parvez, M., Phillips, S. E. & Sutherland, T. C. (2007). *Acta Cryst.* **E63**, o733–o734.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

## supporting information

*Acta Cryst.* (2009). E65, o342 [doi:10.1107/S1600536809001287]

## 3-Benzoyl-5-chlorouracil

Graeme J. Gainsford and Keith Clinch

### S1. Comment

The title compound, (I), was prepared for incorporation into potential thymidine phosphorylase inhibitors (Birck *et al.*, 2009). Its molecular structure is shown in Fig.1, labelled in the same way as the closely related 5-methyl adduct, 3-benzoylthymine (II) (Parvez *et al.*, 2007). The dihedral angle between the aromatic rings in (I) is 86.79 (10)° compared with 83.82 (6)° in (II). The N3–C7–C8–C9 torsion angle of the ring-linkage is -6.9 (2)° in (I) and -11.8 (2)° in (II). Bond distances are normal.

The crystal packing is dominated by centrosymmetric N1—H1N···O14 hydrogen bonded dimers (common graph-set  $R_2^2(8)$ , Bernstein *et al.*, 1995) linked by weaker C—H···O interactions (Table 1). These two types of packing interactions are also found in (II), though not reported, as is illustrated in the comparison Fig 2. The replacement of the methyl group in (II) by chlorine in (I) has not enhanced the packing interactions: neither group/atom play a significant role.

### S2. Experimental

Synthetic details are given in Birck *et al.* (2009).

### S3. Refinement

Atoms H1N and H6 were located in a difference map and refined freely. All other H atoms were restrained using riding models (C—H = 0.95 Å), with  $U_{iso}$  values constrained to 1.2 times that of the  $U_{eq}$  of their parent atom.

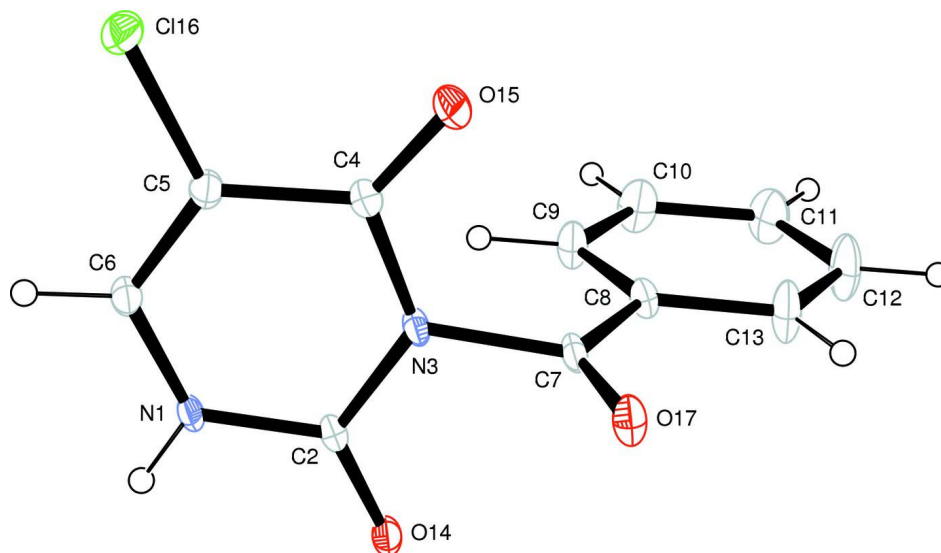


Figure 1

Molecular structure of the title compound. Displacement ellipsoids are shown at the 30% probability level.

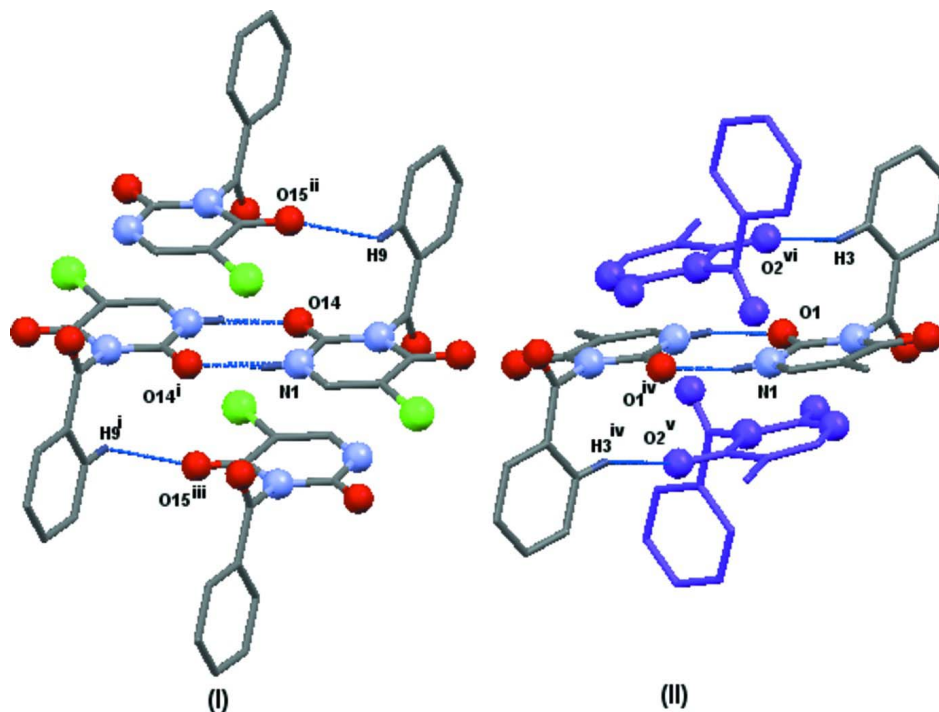


Figure 2

Comparison of similar hydrogen bond interactions (dotted lines) in (I) & (II) (MERCURY; Bruno *et al.*, 2002). Only the hydrogen atoms involved are included. Nitrogen and Oxygen atoms are shown as balls. Colours: Nitrogen, blue (light gray); Oxygen, red (black); Carbon, (gray); Hydrogen, dark blue. Coordinates of (II) are labelled as in deposited data (SEVQUG) and additional molecules shown in purple for clarity. Symmetry Codes: (i)  $3/2 - x, 5/2 - y, 1 - z$  (ii)  $x, y + 1, z$  (iii)  $3/2 - x, 3/2 - y, 1 - z$  (iv)  $2 - x, 1 - y, -z$  (v)  $1/2 + x, 1/2 - y, -z$  (vi)  $3/2 - x, 1/2 + y, z$

### 3-Benzoyl-5-chloro-pyrimidine-2,4(1H,3H)-dione

#### Crystal data

$C_{11}H_7ClN_2O_3$   
 $M_r = 250.64$   
 Monoclinic,  $C2/c$   
 Hall symbol:  $-C 2yc$   
 $a = 21.9357 (9) \text{ \AA}$   
 $b = 5.4020 (2) \text{ \AA}$   
 $c = 19.9642 (9) \text{ \AA}$   
 $\beta = 113.169 (2)^\circ$   
 $V = 2174.89 (16) \text{ \AA}^3$   
 $Z = 8$

$F(000) = 1024$   
 $D_x = 1.531 \text{ Mg m}^{-3}$   
 Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$   
 Cell parameters from 6298 reflections  
 $\theta = 2.2\text{--}28.7^\circ$   
 $\mu = 0.35 \text{ mm}^{-1}$   
 $T = 133 \text{ K}$   
 Plate, colourless  
 $0.34 \times 0.21 \times 0.03 \text{ mm}$

#### Data collection

Bruker–Nonius APEXII CCD area-detector  
 diffractometer  
 Radiation source: fine-focus sealed tube  
 Graphite monochromator  
 Detector resolution:  $8.333 \text{ pixels mm}^{-1}$

$\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan  
 (SADABS; Blessing, 1995; Bruker, 2006)  
 $T_{\min} = 0.810, T_{\max} = 0.990$   
 24616 measured reflections

2899 independent reflections  
 2189 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.047$   
 $\theta_{\text{max}} = 29.0^\circ$ ,  $\theta_{\text{min}} = 3.5^\circ$

$h = -29 \rightarrow 29$   
 $k = -7 \rightarrow 7$   
 $l = -27 \rightarrow 27$

*Refinement*

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.041$   
 $wR(F^2) = 0.110$   
 $S = 1.07$   
 2899 reflections  
 162 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 atoms treated by a mixture of independent  
 and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.0468P)^2 + 2.9413P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.37 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.42 \text{ e } \text{\AA}^{-3}$

*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.** An extinction parameter was refined. 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 ( $\text{\AA}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl16	0.72366 (2)	0.47956 (9)	0.28923 (3)	0.03151 (14)
O14	0.68050 (6)	1.0941 (2)	0.50072 (7)	0.0230 (3)
O15	0.61273 (7)	0.4522 (3)	0.34170 (8)	0.0316 (3)
O17	0.60738 (6)	0.5865 (3)	0.49746 (7)	0.0259 (3)
N1	0.73929 (7)	1.0080 (3)	0.43155 (8)	0.0196 (3)
H1N	0.7670 (11)	1.123 (5)	0.4545 (12)	0.034 (6)*
N3	0.64590 (6)	0.7803 (3)	0.41885 (8)	0.0185 (3)
C2	0.68836 (8)	0.9710 (3)	0.45337 (9)	0.0178 (3)
C4	0.65184 (8)	0.6191 (3)	0.36658 (10)	0.0212 (4)
C5	0.70867 (8)	0.6748 (3)	0.34898 (10)	0.0206 (3)
C6	0.74965 (8)	0.8616 (3)	0.38121 (10)	0.0204 (3)
H6	0.7865 (11)	0.905 (4)	0.3704 (11)	0.025 (5)*
C7	0.59307 (8)	0.7215 (3)	0.44632 (10)	0.0189 (3)
C8	0.52873 (8)	0.8413 (3)	0.40703 (10)	0.0204 (4)
C9	0.51969 (9)	1.0174 (4)	0.35368 (11)	0.0280 (4)
H9	0.5554	1.0604	0.3403	0.034*
C10	0.45871 (10)	1.1310 (4)	0.31977 (12)	0.0367 (5)
H10	0.4524	1.2524	0.2832	0.044*
C11	0.40709 (10)	1.0666 (5)	0.33943 (13)	0.0398 (5)

H11	0.3650	1.1426	0.3156	0.048*
C12	0.41587 (10)	0.8944 (5)	0.39288 (14)	0.0458 (6)
H12	0.3800	0.8527	0.4061	0.055*
C13	0.47664 (10)	0.7816 (4)	0.42752 (13)	0.0375 (5)
H13	0.4830	0.6640	0.4651	0.045*

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C116	0.0351 (3)	0.0321 (3)	0.0358 (3)	−0.0100 (2)	0.0230 (2)	−0.0132 (2)
O14	0.0189 (6)	0.0250 (6)	0.0294 (7)	−0.0055 (5)	0.0141 (5)	−0.0060 (5)
O15	0.0311 (7)	0.0315 (8)	0.0372 (8)	−0.0164 (6)	0.0186 (6)	−0.0120 (6)
O17	0.0181 (6)	0.0285 (7)	0.0323 (7)	0.0009 (5)	0.0112 (5)	0.0095 (6)
N1	0.0141 (6)	0.0217 (7)	0.0250 (8)	−0.0068 (6)	0.0098 (6)	−0.0051 (6)
N3	0.0131 (6)	0.0190 (7)	0.0249 (8)	−0.0042 (5)	0.0091 (6)	−0.0012 (6)
C2	0.0122 (7)	0.0185 (8)	0.0225 (8)	−0.0023 (6)	0.0065 (6)	0.0010 (6)
C4	0.0189 (8)	0.0226 (8)	0.0230 (9)	−0.0047 (7)	0.0091 (7)	−0.0011 (7)
C5	0.0193 (8)	0.0212 (8)	0.0239 (9)	−0.0016 (6)	0.0112 (7)	−0.0022 (7)
C6	0.0157 (7)	0.0232 (8)	0.0236 (9)	−0.0014 (7)	0.0092 (7)	−0.0001 (7)
C7	0.0138 (7)	0.0194 (8)	0.0257 (9)	−0.0047 (6)	0.0099 (7)	−0.0015 (7)
C8	0.0143 (7)	0.0212 (8)	0.0259 (9)	−0.0010 (6)	0.0079 (7)	0.0027 (7)
C9	0.0210 (8)	0.0324 (10)	0.0341 (10)	0.0011 (8)	0.0145 (8)	0.0095 (8)
C10	0.0299 (10)	0.0415 (12)	0.0403 (13)	0.0105 (9)	0.0156 (9)	0.0181 (10)
C11	0.0218 (9)	0.0514 (14)	0.0462 (13)	0.0136 (9)	0.0136 (9)	0.0153 (11)
C12	0.0188 (9)	0.0631 (16)	0.0621 (16)	0.0099 (10)	0.0229 (10)	0.0265 (13)
C13	0.0210 (9)	0.0467 (13)	0.0504 (13)	0.0052 (9)	0.0202 (9)	0.0245 (10)

*Geometric parameters (Å, °)*

C116—C5	1.7179 (18)	C7—C8	1.468 (2)
O14—C2	1.222 (2)	C8—C9	1.383 (3)
O15—C4	1.209 (2)	C8—C13	1.394 (2)
O17—C7	1.192 (2)	C9—C10	1.382 (3)
N1—C2	1.364 (2)	C9—H9	0.95
N1—C6	1.366 (2)	C10—C11	1.381 (3)
N1—H1N	0.87 (3)	C10—H10	0.95
N3—C2	1.378 (2)	C11—C12	1.371 (3)
N3—C4	1.404 (2)	C11—H11	0.95
N3—C7	1.498 (2)	C12—C13	1.379 (3)
C4—C5	1.453 (2)	C12—H12	0.95
C5—C6	1.336 (2)	C13—H13	0.95
C6—H6	0.94 (2)		
C2—N1—C6	122.97 (15)	C8—C7—N3	115.49 (14)
C2—N1—H1N	115.5 (15)	C9—C8—C13	119.96 (17)
C6—N1—H1N	121.3 (15)	C9—C8—C7	122.09 (15)
C2—N3—C4	126.39 (14)	C13—C8—C7	117.87 (16)
C2—N3—C7	116.31 (14)	C10—C9—C8	120.02 (17)

C4—N3—C7	116.87 (13)	C10—C9—H9	120.0
O14—C2—N1	123.34 (15)	C8—C9—H9	120.0
O14—C2—N3	121.36 (14)	C11—C10—C9	119.52 (19)
N1—C2—N3	115.29 (15)	C11—C10—H10	120.2
O15—C4—N3	120.65 (15)	C9—C10—H10	120.2
O15—C4—C5	126.35 (17)	C12—C11—C10	120.82 (19)
N3—C4—C5	113.00 (15)	C12—C11—H11	119.6
C6—C5—C4	121.21 (16)	C10—C11—H11	119.6
C6—C5—Cl16	121.45 (13)	C11—C12—C13	120.15 (19)
C4—C5—Cl16	117.23 (13)	C11—C12—H12	119.9
C5—C6—N1	121.09 (16)	C13—C12—H12	119.9
C5—C6—H6	123.4 (13)	C12—C13—C8	119.51 (19)
N1—C6—H6	115.4 (13)	C12—C13—H13	120.2
O17—C7—C8	126.97 (15)	C8—C13—H13	120.2
O17—C7—N3	117.53 (15)		
C6—N1—C2—O14	176.60 (17)	C2—N3—C7—O17	-84.6 (2)
C6—N1—C2—N3	-2.5 (2)	C4—N3—C7—O17	88.4 (2)
C4—N3—C2—O14	-176.59 (17)	C2—N3—C7—C8	94.63 (18)
C7—N3—C2—O14	-4.3 (2)	C4—N3—C7—C8	-92.32 (19)
C4—N3—C2—N1	2.6 (2)	O17—C7—C8—C9	172.27 (19)
C7—N3—C2—N1	174.86 (14)	N3—C7—C8—C9	-6.9 (3)
C2—N3—C4—O15	177.38 (17)	O17—C7—C8—C13	-4.6 (3)
C7—N3—C4—O15	5.1 (3)	N3—C7—C8—C13	176.25 (18)
C2—N3—C4—C5	-1.5 (2)	C13—C8—C9—C10	-1.2 (3)
C7—N3—C4—C5	-173.75 (15)	C7—C8—C9—C10	-177.94 (19)
O15—C4—C5—C6	-178.47 (19)	C8—C9—C10—C11	-0.2 (4)
N3—C4—C5—C6	0.3 (3)	C9—C10—C11—C12	1.0 (4)
O15—C4—C5—Cl16	-2.2 (3)	C10—C11—C12—C13	-0.4 (4)
N3—C4—C5—Cl16	176.58 (12)	C11—C12—C13—C8	-0.9 (4)
C4—C5—C6—N1	-0.4 (3)	C9—C8—C13—C12	1.7 (4)
Cl16—C5—C6—N1	-176.52 (14)	C7—C8—C13—C12	178.6 (2)
C2—N1—C6—C5	1.6 (3)		

Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1N $\cdots$ O14 <sup>i</sup>	0.86 (3)	1.91 (3)	2.770 (2)	173 (3)
C9—H9 $\cdots$ O15 <sup>ii</sup>	0.95	2.46	3.182 (3)	133
C10—H10 $\cdots$ O15 <sup>iii</sup>	0.95	2.57	3.447 (3)	153

Symmetry codes: (i)  $-x+3/2, -y+5/2, -z+1$ ; (ii)  $x, y+1, z$ ; (iii)  $-x+1, y+1, -z+1/2$ .