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N,N'-(4-Chlorobenzylidene)dipyrimidin-2-amine

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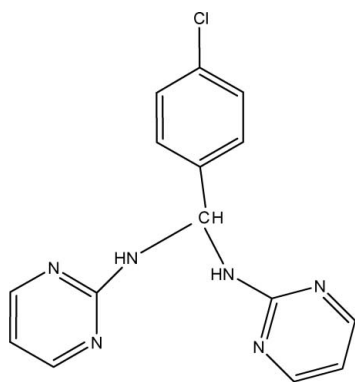
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 Key indicators: single-crystal X-ray study; $T = 120$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.061; wR factor = 0.163; data-to-parameter ratio = 19.0.

The title compound, $\text{C}_{15}\text{H}_{13}\text{ClN}_6$, contains two pyrimidine rings and one benzene ring, where the dihedral angle between the planes through the pyrimidine rings is $81.57(10)^\circ$, and those between the pyrimidine rings and the benzene ring are $84.02(8)$ and $89.46(7)^\circ$, indicating that the three rings are almost perpendicular. In the crystal, intermolecular $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds link the molecules into infinite chains along (100).

Related literature

For the biological activity of pyrimidine derivatives, see: Onal & Altral (1999); Ponticelli *et al.* (1999). For studies of the reactions of heterocyclic amines with aromatic aldehydes to prepare new ligands, see: Tabatabaee *et al.* (2006); Tabatabaee, Ghassemzadeh, Dehghan *et al.* (2007); Tabatabaee, Ghassemzadeh, Zarabi *et al.* (2007); Tabatabaee, Ghassemzadeh *et al.* (2008); Tabatabaee, Hakimi *et al.* (2008).



Experimental

Crystal data

 $\text{C}_{15}\text{H}_{13}\text{ClN}_6$
 $M_r = 312.76$

 Monoclinic, $P2_1/n$
 $a = 9.6030(14)$ Å
 $b = 10.5706(15)$ Å
 $c = 14.792(2)$ Å
 $\beta = 100.331(3)^\circ$
 $V = 1477.2(4)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.26$ mm⁻¹
 $T = 120$ K
 $0.17 \times 0.15 \times 0.14$ mm

Data collection

 Bruker SMART 1000 CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1998)
 $T_{\min} = 0.950$, $T_{\max} = 0.964$

 15899 measured reflections
 3924 independent reflections
 2429 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.063$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.061$
 $wR(F^2) = 0.163$
 $S = 1.01$
 3924 reflections
 207 parameters

 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.55$ e Å⁻³
 $\Delta\rho_{\min} = -0.28$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1N}\cdots\text{N3}^i$	0.84 (3)	2.20 (3)	3.033 (3)	174 (3)
$\text{N4}-\text{H4N}\cdots\text{N6}^{ii}$	0.83 (3)	2.24 (3)	3.057 (3)	172 (3)

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

Data collection: SMART (Bruker, 1998); cell refinement: SAINT-Plus (Bruker, 1998); data reduction: SAINT-Plus; 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.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: EZZ187).

References

- Bruker (1998). SMART and SAINT-Plus. Bruker AXS Inc., Madison, Wisconsin, USA.
- Onal, Z. & Altral, B. (1999). *Turk. J. Chem.* **23**, 401–405.
- Ponticelli, G., Spanu, A. M. T., Cocco, M. T. & Onnis, V. (1999). *Transition Met. Chem.* **24**, 370–372.
- Sheldrick, G. M. (1998). SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Tabatabaee, M., Ghassemzadeh, M., Dehghan, A. R., Khavasi, H. R. & Heravi, M. M. (2007). *Acta Cryst.* **E63**, o42–o43.
- Tabatabaee, M., Ghassemzadeh, M. & Soleimani, N. (2008). *Anal. Sci.* **24**, x173–x174.
- Tabatabaee, M., Ghassemzadeh, M., Zarabi, B., Heravi, M. M., Anary-Abbasinejad, M. & Neumüller, B. (2007). *Phosphorus Sulfur Silicon Relat. Elem.* **182**, 677–686.
- Tabatabaee, M., Ghassemzadeh, M., Zarabi, B. & Neumüller, B. (2006). *Z. Naturforsch. Teil B*, **61**, 1421–1425.
- Tabatabaee, M., Hakimi, F., Roshani, M., Mirjalili, M. & Kavasi, H. R. (2008). *Acta Cryst.* **E64**, o2112.

supporting information

Acta Cryst. (2009). E65, o2979 [doi:10.1107/S1600536809045243]

N,N'*-(4-Chlorobenzylidene)dipyrimidin-2-amine*Masoumeh Tabatabaee, Leila Masoodpour, Mitra Gassemezadeh and Fatemeh Hakimi****S1. Comment**

Pyrimidine derivatives represent a class of heterocycles of great importance. Many pyrimidines, or their derivatives, possess remarkable biological activity and have been widely used in medicinal and industrial applications (Onal & Altral, 1999; Ponticelli *et al.*, 1999). In the continuation of our recent work on the reactions of heterocyclic amines with aromatic aldehydes to prepare new ligands (Tabatabaee *et al.*, 2006; Tabatabaee, Ghassemzadeh, Dehghan *et al.*, 2007; Tabatabaee, Ghassemzadeh, Zarabi *et al.*, 2007; Tabatabaee, Ghassemzadeh *et al.*, 2008; Tabatabaee, Hakimi *et al.*, 2008) we report our results on the reaction of 2-aminopyrimidine and 4-chlorobenzaldehyde in this communication.

The crystal structure of (I) (Fig. 1) shows that one molecule of 4-chlorobenzaldehyde reacted with two molecules of 2-aminopyrimidine to form (I). Bond lengths and angles are unexceptional. The compound contains two pyrimidine (A: N2/C8/N3/C11/C10/C9 and B: N5/C12/N6/C15/C14/C13) and one benzene (C: C2/C3/C4/C5/C6/C7) rings. The dihedral angles formed by the planes through A and B is 81.57 (10)°, through A and C is 84.02 (8)° and through B and C is 89.49 (7)°, indicating that the three rings are almost perpendicular.

Intermolecular N—H···N hydrogen bonds link the molecules into infinite one-dimensional chains along (100) (Table 1 and Fig 2). An interesting feature of compound (I) is the presence of C—H··· π stacking interactions between C—H groups from one molecule and aromatic rings on adjacent molecules. The C—H··· π distance is 2.89 Å for C9—H9A···Cg3 (Cg3: C2/C3—C7), with an angle of 133.21° and 2.99 Å for C4—H4A···Cg1 (Cg1: N2/C8—C9) with an angle of 132.27° (Fig. 3).

S2. Experimental

A solution of 2-aminopyrimidine (0.951 g, 10 mmol) in EtOH (10 ml) was treated with 4-chlorobenzaldehyde (0.7 g, 5 mmol) and the resulting mixture was acidified with 37% hydrochloric acid (0.2 ml). The reaction mixture was refluxed for 12 h. The solid residue was filtered and the filtrate was kept at 293 K. Colorless crystals of the title compound were obtained after a few days (yield 92%).

S3. Refinement

The hydrogen atoms of NH groups were found in difference Fourier syntheses and refined isotropically. The H(C) atom positions were calculated and refined in isotropic approximation using a riding model with the $U_{\text{isc}}(\text{H})$ parameters equal to 1.2 $U_{\text{eq}}(\text{C}_i)$, where $U(\text{C}_i)$ are the equivalent thermal parameters of the CH and CH₂ carbon atoms to which the corresponding H atoms are bonded.

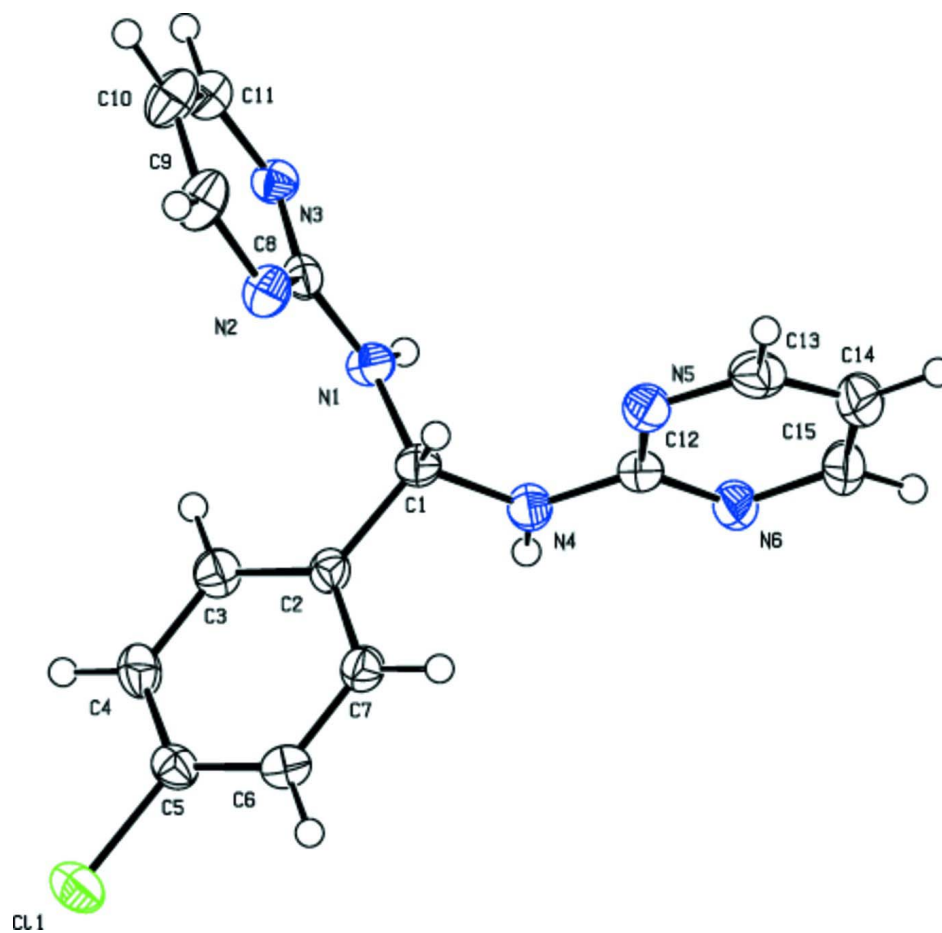


Figure 1

The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

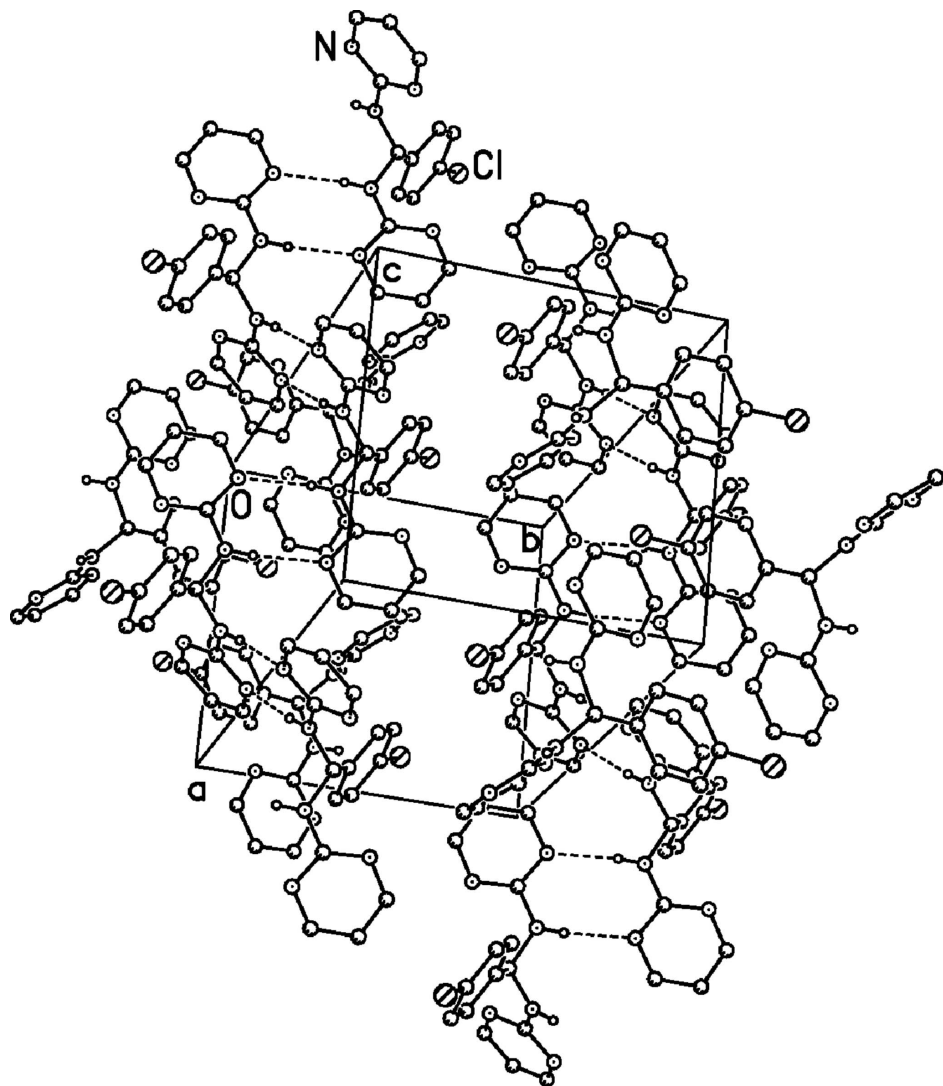


Figure 2

Packing diagram of (I), molecules are linked into infinite one dimensional chains by hydrogen-bond interactions (dashed lines).

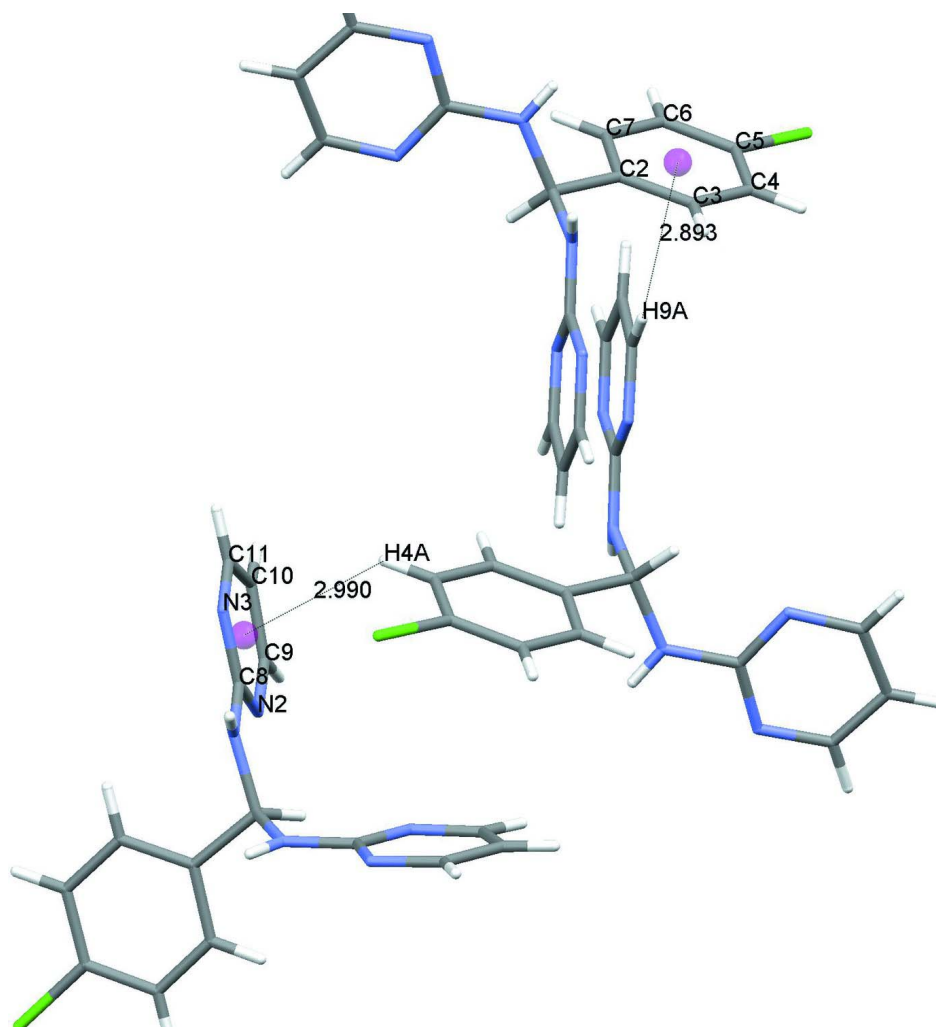


Figure 3

Intermolecular C—H \cdots π interactions (dashed lines) between aromatic rings of adjacent molecules.

N,N'-(4-Chlorobenzylidene)dipyrimidin-2-amine

Crystal data

$C_{15}H_{13}ClN_6$

$M_r = 312.76$

Monoclinic, $P2_1/n$

Hall symbol: $-P\ 2_1/n$

$a = 9.6030$ (14) Å

$b = 10.5706$ (15) Å

$c = 14.792$ (2) Å

$\beta = 100.331$ (3)°

$V = 1477.2$ (4) Å³

$Z = 4$

$F(000) = 648$

$D_x = 1.406$ Mg m⁻³

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

Cell parameters from 697 reflections

$\theta = 3\text{--}30^\circ$

$\mu = 0.26$ mm⁻¹

$T = 120$ K

Prism, colorless

$0.17 \times 0.15 \times 0.14$ mm

Data collection

Bruker SMART 1000 CCD area-detector
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
 ω scans
Absorption correction: multi-scan
(*SADABS*; Sheldrick, 1998)
 $T_{\min} = 0.950$, $T_{\max} = 0.964$

15899 measured reflections
3924 independent reflections
2429 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.063$
 $\theta_{\max} = 29.0^\circ$, $\theta_{\min} = 2.4^\circ$
 $h = -13 \rightarrow 13$
 $k = -14 \rightarrow 14$
 $l = -20 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.061$
 $wR(F^2) = 0.163$
 $S = 1.01$
3924 reflections
207 parameters
0 restraints
Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map
Hydrogen site location: mixed
H atoms treated by a mixture of independent
and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.061P)^2 + 1.85P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.55 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.28 \text{ e } \text{\AA}^{-3}$

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. 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	-0.02192 (8)	0.53784 (7)	0.18886 (5)	0.0355 (2)
N1	0.3768 (2)	0.1522 (2)	0.49990 (15)	0.0255 (5)
H1N	0.372 (3)	0.073 (3)	0.4970 (19)	0.026 (7)*
N2	0.5083 (2)	0.3334 (2)	0.53797 (15)	0.0288 (5)
N3	0.6177 (2)	0.13385 (19)	0.51466 (14)	0.0252 (5)
N4	0.1456 (2)	0.1274 (2)	0.53239 (15)	0.0271 (5)
H4N	0.100 (3)	0.085 (3)	0.490 (2)	0.026 (7)*
N5	0.1916 (2)	0.1776 (2)	0.68744 (15)	0.0297 (5)
N6	0.0426 (2)	0.0032 (2)	0.62930 (15)	0.0324 (5)
C1	0.2469 (3)	0.2170 (2)	0.51066 (17)	0.0236 (5)
H1A	0.2701	0.2771	0.5635	0.028*
C2	0.1814 (3)	0.2927 (2)	0.42613 (17)	0.0231 (5)
C3	0.2361 (3)	0.2959 (2)	0.34541 (18)	0.0281 (6)
H3A	0.3172	0.2468	0.3403	0.034*
C4	0.1729 (3)	0.3708 (3)	0.27187 (17)	0.0305 (6)
H4A	0.2106	0.3727	0.2168	0.037*

C5	0.0551 (3)	0.4423 (2)	0.27932 (17)	0.0257 (5)
C6	-0.0013 (3)	0.4405 (2)	0.35850 (18)	0.0274 (5)
H6A	-0.0824	0.4898	0.3633	0.033*
C7	0.0623 (3)	0.3653 (2)	0.43127 (17)	0.0263 (5)
H7A	0.0235	0.3635	0.4860	0.032*
C8	0.5041 (3)	0.2088 (2)	0.51871 (16)	0.0227 (5)
C9	0.6373 (3)	0.3841 (3)	0.5586 (2)	0.0346 (6)
H9A	0.6452	0.4713	0.5741	0.041*
C10	0.7596 (3)	0.3161 (3)	0.5585 (2)	0.0353 (7)
H10A	0.8505	0.3537	0.5745	0.042*
C11	0.7431 (3)	0.1905 (3)	0.53381 (18)	0.0295 (6)
H11A	0.8254	0.1421	0.5303	0.035*
C12	0.1273 (3)	0.1023 (3)	0.61980 (17)	0.0266 (5)
C13	0.1706 (3)	0.1484 (3)	0.77168 (19)	0.0346 (6)
H13A	0.2127	0.2005	0.8215	0.041*
C14	0.0906 (3)	0.0461 (3)	0.7899 (2)	0.0406 (7)
H14A	0.0802	0.0247	0.8507	0.049*
C15	0.0269 (3)	-0.0232 (3)	0.7149 (2)	0.0406 (7)
H15A	-0.0309	-0.0929	0.7247	0.049*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0406 (4)	0.0320 (4)	0.0312 (4)	0.0008 (3)	-0.0014 (3)	0.0066 (3)
N1	0.0227 (11)	0.0213 (11)	0.0316 (12)	-0.0002 (9)	0.0021 (9)	0.0004 (9)
N2	0.0303 (12)	0.0236 (11)	0.0335 (12)	-0.0039 (9)	0.0081 (9)	-0.0030 (9)
N3	0.0245 (11)	0.0236 (11)	0.0263 (11)	-0.0021 (9)	0.0016 (8)	-0.0015 (9)
N4	0.0272 (11)	0.0300 (12)	0.0229 (11)	-0.0074 (9)	0.0016 (9)	-0.0006 (9)
N5	0.0288 (12)	0.0333 (12)	0.0267 (11)	-0.0018 (10)	0.0043 (9)	-0.0002 (9)
N6	0.0310 (12)	0.0394 (13)	0.0256 (11)	-0.0081 (10)	0.0021 (9)	0.0041 (10)
C1	0.0227 (12)	0.0234 (12)	0.0238 (12)	-0.0010 (10)	0.0021 (9)	-0.0017 (10)
C2	0.0237 (12)	0.0221 (12)	0.0235 (12)	-0.0048 (10)	0.0042 (9)	0.0003 (10)
C3	0.0300 (14)	0.0268 (13)	0.0287 (13)	0.0029 (11)	0.0082 (11)	-0.0007 (11)
C4	0.0366 (15)	0.0345 (15)	0.0220 (12)	-0.0018 (12)	0.0098 (11)	-0.0007 (11)
C5	0.0288 (13)	0.0235 (12)	0.0230 (12)	-0.0030 (10)	-0.0001 (10)	-0.0001 (10)
C6	0.0238 (12)	0.0273 (13)	0.0300 (13)	0.0005 (10)	0.0023 (10)	-0.0025 (10)
C7	0.0246 (13)	0.0306 (13)	0.0245 (12)	-0.0010 (11)	0.0065 (10)	-0.0003 (10)
C8	0.0261 (13)	0.0221 (12)	0.0199 (12)	-0.0036 (10)	0.0039 (10)	0.0014 (10)
C9	0.0353 (15)	0.0254 (13)	0.0458 (16)	-0.0095 (12)	0.0147 (13)	-0.0094 (12)
C10	0.0306 (14)	0.0339 (15)	0.0437 (16)	-0.0109 (12)	0.0128 (12)	-0.0118 (13)
C11	0.0267 (13)	0.0306 (14)	0.0316 (14)	-0.0019 (11)	0.0065 (11)	-0.0009 (11)
C12	0.0225 (12)	0.0311 (14)	0.0256 (13)	0.0013 (11)	0.0028 (10)	0.0023 (11)
C13	0.0317 (15)	0.0410 (16)	0.0291 (14)	0.0026 (12)	0.0004 (11)	-0.0044 (12)
C14	0.0368 (16)	0.059 (2)	0.0258 (14)	-0.0061 (15)	0.0058 (12)	0.0057 (14)
C15	0.0346 (16)	0.0541 (19)	0.0330 (15)	-0.0126 (14)	0.0059 (12)	0.0119 (14)

Geometric parameters (Å, °)

C11—C5	1.732 (3)	C3—C4	1.394 (4)
N1—C8	1.345 (3)	C3—H3A	0.9500
N1—C1	1.456 (3)	C4—C5	1.381 (4)
N1—H1N	0.84 (3)	C4—H4A	0.9500
N2—C9	1.334 (3)	C5—C6	1.376 (4)
N2—C8	1.347 (3)	C6—C7	1.388 (4)
N3—C11	1.329 (3)	C6—H6A	0.9500
N3—C8	1.358 (3)	C7—H7A	0.9500
N4—C12	1.362 (3)	C9—C10	1.377 (4)
N4—C1	1.435 (3)	C9—H9A	0.9500
N4—H4N	0.83 (3)	C10—C11	1.378 (4)
N5—C13	1.334 (4)	C10—H10A	0.9500
N5—C12	1.339 (3)	C11—H11A	0.9500
N6—C15	1.331 (4)	C13—C14	1.381 (4)
N6—C12	1.349 (3)	C13—H13A	0.9500
C1—C2	1.523 (3)	C14—C15	1.378 (4)
C1—H1A	1.0000	C14—H14A	0.9500
C2—C3	1.389 (3)	C15—H15A	0.9500
C2—C7	1.390 (3)		
C8—N1—C1	122.2 (2)	C5—C6—H6A	120.6
C8—N1—H1N	120 (2)	C7—C6—H6A	120.6
C1—N1—H1N	116 (2)	C6—C7—C2	121.7 (2)
C9—N2—C8	115.6 (2)	C6—C7—H7A	119.2
C11—N3—C8	115.6 (2)	C2—C7—H7A	119.2
C12—N4—C1	123.3 (2)	N1—C8—N2	118.0 (2)
C12—N4—H4N	118 (2)	N1—C8—N3	116.1 (2)
C1—N4—H4N	119 (2)	N2—C8—N3	125.9 (2)
C13—N5—C12	115.7 (2)	N2—C9—C10	123.2 (3)
C15—N6—C12	115.8 (2)	N2—C9—H9A	118.4
N4—C1—N1	110.0 (2)	C10—C9—H9A	118.4
N4—C1—C2	109.5 (2)	C9—C10—C11	116.5 (3)
N1—C1—C2	113.2 (2)	C9—C10—H10A	121.8
N4—C1—H1A	108.0	C11—C10—H10A	121.8
N1—C1—H1A	108.0	N3—C11—C10	123.1 (3)
C2—C1—H1A	108.0	N3—C11—H11A	118.4
C3—C2—C7	118.4 (2)	C10—C11—H11A	118.4
C3—C2—C1	123.7 (2)	N5—C12—N6	126.1 (2)
C7—C2—C1	117.9 (2)	N5—C12—N4	118.2 (2)
C2—C3—C4	120.4 (2)	N6—C12—N4	115.7 (2)
C2—C3—H3A	119.8	N5—C13—C14	123.1 (3)
C4—C3—H3A	119.8	N5—C13—H13A	118.5
C5—C4—C3	119.8 (2)	C14—C13—H13A	118.5
C5—C4—H4A	120.1	C15—C14—C13	116.2 (3)
C3—C4—H4A	120.1	C15—C14—H14A	121.9
C6—C5—C4	120.9 (2)	C13—C14—H14A	121.9

C6—C5—C11	119.1 (2)	N6—C15—C14	123.0 (3)
C4—C5—C11	120.0 (2)	N6—C15—H15A	118.5
C5—C6—C7	118.8 (2)	C14—C15—H15A	118.5
C12—N4—C1—N1	96.1 (3)	C1—N1—C8—N3	173.6 (2)
C12—N4—C1—C2	-139.0 (2)	C9—N2—C8—N1	178.2 (2)
C8—N1—C1—N4	-150.6 (2)	C9—N2—C8—N3	-3.1 (4)
C8—N1—C1—C2	86.6 (3)	C11—N3—C8—N1	-179.7 (2)
N4—C1—C2—C3	-120.7 (3)	C11—N3—C8—N2	1.6 (4)
N1—C1—C2—C3	2.5 (3)	C8—N2—C9—C10	1.5 (4)
N4—C1—C2—C7	61.2 (3)	N2—C9—C10—C11	1.2 (4)
N1—C1—C2—C7	-175.7 (2)	C8—N3—C11—C10	1.5 (4)
C7—C2—C3—C4	0.3 (4)	C9—C10—C11—N3	-2.8 (4)
C1—C2—C3—C4	-177.9 (2)	C13—N5—C12—N6	1.4 (4)
C2—C3—C4—C5	0.0 (4)	C13—N5—C12—N4	-179.7 (2)
C3—C4—C5—C6	-0.2 (4)	C15—N6—C12—N5	-2.5 (4)
C3—C4—C5—C11	178.8 (2)	C15—N6—C12—N4	178.6 (3)
C4—C5—C6—C7	0.1 (4)	C1—N4—C12—N5	10.1 (4)
C11—C5—C6—C7	-179.0 (2)	C1—N4—C12—N6	-170.9 (2)
C5—C6—C7—C2	0.3 (4)	C12—N5—C13—C14	1.4 (4)
C3—C2—C7—C6	-0.4 (4)	N5—C13—C14—C15	-2.8 (5)
C1—C2—C7—C6	177.8 (2)	C12—N6—C15—C14	0.8 (5)
C1—N1—C8—N2	-7.6 (3)	C13—C14—C15—N6	1.6 (5)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1N...N3 ⁱ	0.84 (3)	2.20 (3)	3.033 (3)	174 (3)
N4—H4N...N6 ⁱⁱ	0.83 (3)	2.24 (3)	3.057 (3)	172 (3)

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