

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

2,6-Diamino-4-(4-chlorophenyl)-1-methyl-1,4-dihydropyridine-3,5-dicarbonitrile

Michael Purushothaman,^{a*} Kaliyaperumal Thanigaimani,^b Suhana Arshad,^b Sekar Silambarasan,^a Ibrahim Abdul Razak^{b‡} and Kather Mohideen Sithick Ali^a

^aDepartment of Chemistry, Jamal Mohamed College (Autonomous), Tiruchirappalli 620 020, Tamil Nadu, India, and ^bSchool of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia

Correspondence e-mail: purush.alpha@gmail.com

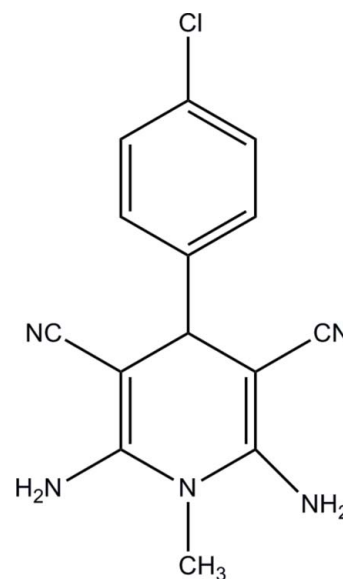
Received 10 June 2014; accepted 18 June 2014

Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.072; wR factor = 0.207; data-to-parameter ratio = 15.4.

In the title compound, $\text{C}_{14}\text{H}_{12}\text{ClN}_5$, the dihydropyridine ring adopts a shallow boat conformation. The dihedral angle between the plane of this ring and that of the chlorobenzene ring is 69.15 (15)°. In the crystal, molecules are linked by $\text{N}-\text{H}\cdots\text{N}$ and $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bonds, generating (001) sheets.

Related literature

For background to malononitrile, see: Fatiadi (1978); Raghukumar *et al.* (2003). For the stability of the temperature controller used for the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

$\text{C}_{14}\text{H}_{12}\text{ClN}_5$
 $M_r = 285.74$
 Triclinic, $P\bar{1}$
 $a = 8.3893$ (4) Å
 $b = 8.4679$ (5) Å
 $c = 10.2571$ (6) Å
 $\alpha = 93.148$ (4)°
 $\beta = 112.478$ (3)°
 $\gamma = 93.929$ (3)°
 $V = 669.11$ (6) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.28$ mm⁻¹
 $T = 100$ K
 $0.37 \times 0.28 \times 0.16$ mm

Data collection

Bruker SMART APEXII CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2009)
 $T_{\min} = 0.904$, $T_{\max} = 0.956$
 9775 measured reflections
 3049 independent reflections
 2435 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.053$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.072$
 $wR(F^2) = 0.207$
 $S = 1.05$
 3049 reflections
 198 parameters
 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 1.17$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.48$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|---|--------------|--------------------|-------------|----------------------|
| $\text{N2}-\text{H1N2}\cdots\text{N5}^{\text{i}}$ | 0.86 (5) | 2.21 (5) | 3.043 (4) | 163 (5) |
| $\text{N2}-\text{H2N2}\cdots\text{Cl1}^{\text{ii}}$ | 0.89 (4) | 2.75 (4) | 3.588 (3) | 158 (3) |
| $\text{N3}-\text{H2N3}\cdots\text{N4}^{\text{iii}}$ | 0.83 (4) | 2.27 (4) | 3.071 (4) | 161 (4) |

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

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); 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).

‡ Thomson Reuters ResearcherID: A-5599-2009.

KT and IAR thank the Malaysian Government and Universiti Sains Malaysia (USM) for the research facilities and USM Short Term Grant, No. 304/PFIZIK/6312078. MP, SS and KMSA thank the Management and the Principal, Jamal Mohamed College (Autonomous), for providing research facilities. MP thanks the UGC–SERO, Hyderabad, for a minor research project. KT thanks The Academy of Sciences for the Developing World and USM for the TWAS–USM fellowship.

Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7240).

References

- Bruker (2009). *SADABS*, *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.
- Fatiadi, A. J. (1978). *Synthesis*, **3**, 165–204.
- Raghukumar, V., Thiirumalai, D., Ramakrishnan, V., Karunakara, V. & Ramamurthy, P. (2003). *Tetrahedron*, **59**, 3761–3768.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.

supporting information

Acta Cryst. (2014). E70, o812–o813 [https://doi.org/10.1107/S1600536814014354]

2,6-Diamino-4-(4-chlorophenyl)-1-methyl-1,4-dihydropyridine-3,5-dicarbonitrile

Michael Purushothaman, Kaliyaperumal Thanigaimani, Suhana Arshad, Sekar Silambarasan, Ibrahim Abdul Razak and Kather Mohideen Sithick Ali

S1. Comment

Malononitrile is a simple and versatile reagent for the synthesis of heterocyclic compounds and precursors of novel compounds. It exhibits a unique reactivity due to the strong electron withdrawing cyano groups to activate the methylene group and the polar multiple bond suitable for nucleophilic addition (Fatiadi, 1978). Malononitrile is used as reactant or reaction intermediate in various multicomponent reactions to prepare heterocyclic compounds. The three component reactions of malononitrile, aldehyde and amine show very chemical diversity, from which several kinds of products were separated (Raghukumar *et al.*, 2003). The crystal structure of the title compound (I) is presented here.

The molecular structure of the title compound is shown in Fig. 1. The pyridine ring (N1/C7—C11) adopts a boat conformation with puckering parameters $Q = 0.402(3) \text{ \AA}$, $\Theta = 79.6(4)^\circ$ and $\Phi = 177.2(4)^\circ$. The dihedral angle between the pyridine (N1/C7—C11) and benzene (C1—C6) rings is $69.15(15)^\circ$.

The crystal structure shown in Fig. 2 features $N2—H1N2 \cdots N5^i$ and $N3—H2N3 \cdots N4^{iii}$ hydrogen bonds (symmetry code in Table 1) to result in tetrameric association of molecules, generated by inversion. These tetramers are then connected *via* $N2—H2N2 \cdots C11^{ii}$ hydrogen bond (symmetry code in Table 1), forming a layer parallel to the *ab* plane.

S2. Experimental

Compound (I) was prepared by the reaction of *p*-chlorobenzaldehyde (1 mmol), malononitrile (1 mmol) and methylamine (1 mmol) in a mixed solvent of methanol and water (5:1) was stirred at room temperature about an hour. The resulting precipitate was collected by filtration and washed with methanol to afford pure product, *m.p.*: 290 °C. The product was crystallized from methanol solution as colourless plates.

S3. Refinement

N-bound H atoms were located in a difference Fourier maps and allowed to be refined freely [refined distance: N–H = $0.83(4)–0.89(4) \text{ \AA}$]. The remaining hydrogen atoms were positioned geometrically [C–H = 0.95 or 0.98 \AA] and were refined using a riding model, with $U_{iso}(H) = 1.2 U_{eq}(C)$ or $1.5 U_{eq}(\text{methyl C})$. A rotating-group model was used for the methyl group.

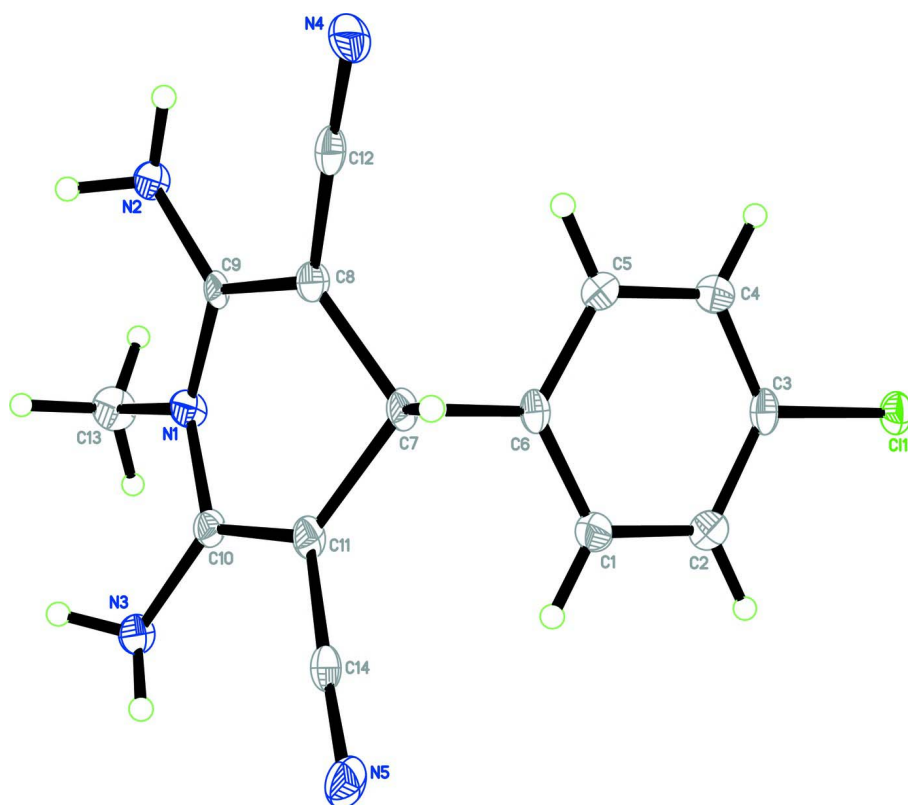


Figure 1

The molecular structure of the title compound with 50% probability displacement ellipsoids.

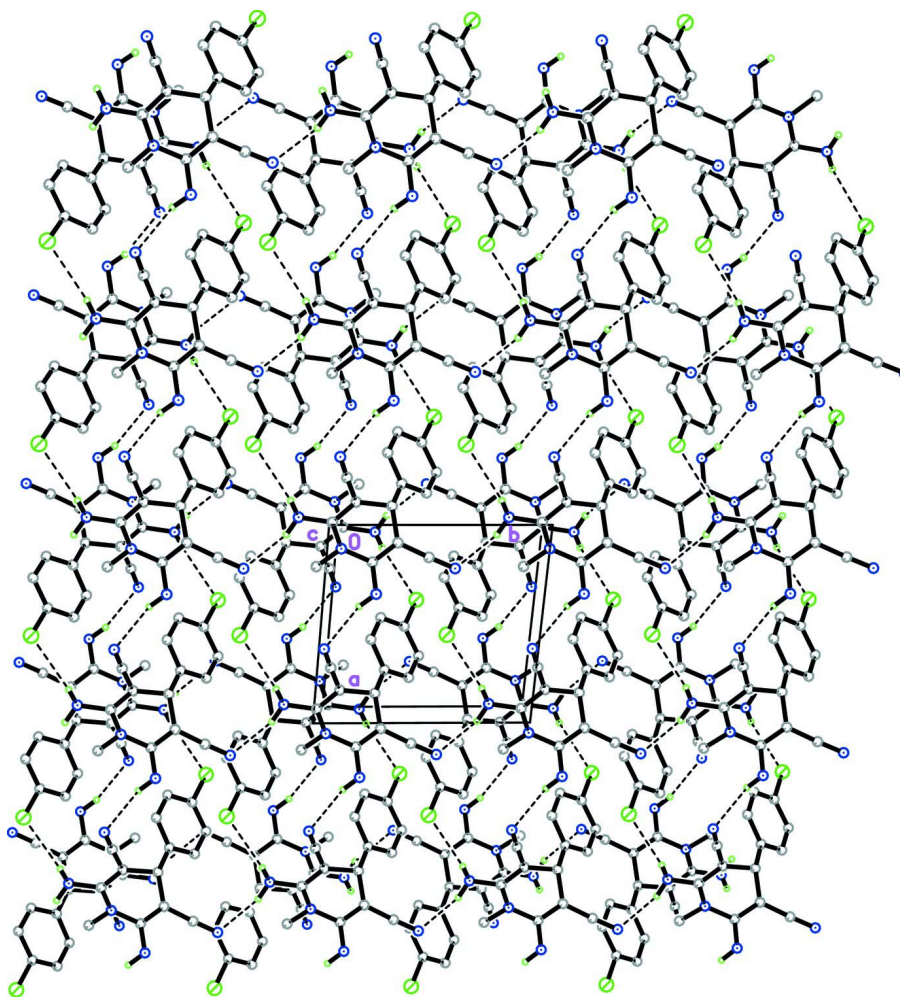


Figure 2

The crystal packing of the title compound. The H atoms not involved in the intermolecular interactions (dashed lines) have been omitted for clarity.

2,6-Diamino-4-(4-chlorophenyl)-1-methyl-1,4-dihydropyridine-3,5-dicarbonitrile

Crystal data

$C_{14}H_{12}ClN_5$

$M_r = 285.74$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 8.3893$ (4) Å

$b = 8.4679$ (5) Å

$c = 10.2571$ (6) Å

$\alpha = 93.148$ (4)°

$\beta = 112.478$ (3)°

$\gamma = 93.929$ (3)°

$V = 669.11$ (6) Å³

$Z = 2$

$F(000) = 296$

$D_x = 1.418$ Mg m⁻³

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

Cell parameters from 4444 reflections

$\theta = 2.6$ – 32.3 °

$\mu = 0.28$ mm⁻¹

$T = 100$ K

Plate, colourless

$0.37 \times 0.28 \times 0.16$ mm

Data collection

Bruker SMART APEXII CCD
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
 φ and ω scans
Absorption correction: multi-scan
(*SADABS*; Bruker, 2009)
 $T_{\min} = 0.904$, $T_{\max} = 0.956$

9775 measured reflections
3049 independent reflections
2435 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.053$
 $\theta_{\max} = 27.5^\circ$, $\theta_{\min} = 2.2^\circ$
 $h = -10 \rightarrow 10$
 $k = -10 \rightarrow 9$
 $l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.072$
 $wR(F^2) = 0.207$
 $S = 1.05$
3049 reflections
198 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
 $w = 1/[\sigma^2(F_o^2) + (0.1391P)^2 + 0.3591P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 1.17 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.48 \text{ e } \text{\AA}^{-3}$

Special details

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

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)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|-------------|-------------|--------------|----------------------------------|
| C11 | 0.41603 (9) | 0.41701 (9) | -0.28944 (7) | 0.0186 (3) |
| N1 | 1.1252 (3) | 0.0360 (3) | 0.2661 (3) | 0.0153 (5) |
| N2 | 0.9603 (3) | -0.1777 (3) | 0.3020 (3) | 0.0164 (5) |
| N3 | 1.3641 (3) | 0.2241 (3) | 0.3245 (3) | 0.0171 (6) |
| N4 | 0.6529 (3) | 0.0004 (3) | 0.4178 (3) | 0.0184 (6) |
| N5 | 1.2267 (3) | 0.5888 (3) | 0.4289 (3) | 0.0198 (6) |
| C1 | 0.8304 (4) | 0.4173 (4) | 0.0726 (3) | 0.0181 (6) |
| H1A | 0.9409 | 0.4758 | 0.1176 | 0.022* |
| C2 | 0.7184 (4) | 0.4557 (4) | -0.0597 (3) | 0.0189 (6) |
| H2A | 0.7516 | 0.5389 | -0.1053 | 0.023* |
| C3 | 0.5572 (4) | 0.3693 (4) | -0.1231 (3) | 0.0157 (6) |
| C4 | 0.5057 (4) | 0.2498 (4) | -0.0585 (3) | 0.0187 (6) |
| H4A | 0.3943 | 0.1930 | -0.1031 | 0.022* |
| C5 | 0.6194 (4) | 0.2132 (4) | 0.0734 (3) | 0.0182 (6) |

| | | | | |
|------|------------|-------------|------------|-------------|
| H5A | 0.5844 | 0.1308 | 0.1188 | 0.022* |
| C6 | 0.7834 (3) | 0.2950 (3) | 0.1401 (3) | 0.0136 (6) |
| C7 | 0.9013 (3) | 0.2604 (3) | 0.2911 (3) | 0.0133 (6) |
| H7A | 0.8677 | 0.3263 | 0.3587 | 0.016* |
| C8 | 0.8816 (4) | 0.0898 (3) | 0.3211 (3) | 0.0137 (6) |
| C9 | 0.9872 (3) | -0.0158 (3) | 0.2979 (3) | 0.0110 (6) |
| C10 | 1.1955 (4) | 0.1912 (3) | 0.3081 (3) | 0.0135 (6) |
| C11 | 1.0935 (3) | 0.3048 (3) | 0.3278 (3) | 0.0141 (6) |
| C12 | 0.7568 (4) | 0.0383 (3) | 0.3743 (3) | 0.0153 (6) |
| C13 | 1.2207 (4) | -0.0783 (4) | 0.2184 (3) | 0.0200 (7) |
| H13A | 1.1391 | -0.1654 | 0.1568 | 0.030* |
| H13B | 1.3055 | -0.1210 | 0.3008 | 0.030* |
| H13C | 1.2811 | -0.0245 | 0.1657 | 0.030* |
| C14 | 1.1690 (4) | 0.4592 (4) | 0.3845 (3) | 0.0145 (6) |
| H1N2 | 1.050 (6) | -0.226 (6) | 0.345 (5) | 0.043 (12)* |
| H2N2 | 0.873 (5) | -0.220 (5) | 0.322 (4) | 0.022 (9)* |
| H1N3 | 1.414 (5) | 0.315 (6) | 0.355 (4) | 0.030 (11)* |
| H2N3 | 1.425 (5) | 0.150 (5) | 0.355 (4) | 0.029 (11)* |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|-------------|-------------|--------------|
| C11 | 0.0168 (4) | 0.0188 (4) | 0.0178 (4) | 0.0060 (3) | 0.0030 (3) | 0.0034 (3) |
| N1 | 0.0126 (11) | 0.0132 (12) | 0.0215 (12) | 0.0042 (9) | 0.0080 (10) | -0.0010 (10) |
| N2 | 0.0137 (12) | 0.0082 (12) | 0.0282 (14) | 0.0055 (10) | 0.0084 (11) | 0.0015 (10) |
| N3 | 0.0126 (12) | 0.0132 (13) | 0.0292 (14) | 0.0081 (11) | 0.0107 (11) | 0.0041 (11) |
| N4 | 0.0145 (12) | 0.0168 (13) | 0.0261 (14) | 0.0081 (10) | 0.0090 (11) | 0.0030 (11) |
| N5 | 0.0210 (13) | 0.0136 (13) | 0.0244 (13) | 0.0080 (10) | 0.0074 (11) | 0.0020 (10) |
| C1 | 0.0150 (13) | 0.0141 (14) | 0.0235 (15) | 0.0024 (11) | 0.0056 (12) | -0.0009 (12) |
| C2 | 0.0189 (14) | 0.0164 (15) | 0.0226 (15) | 0.0036 (12) | 0.0088 (12) | 0.0034 (12) |
| C3 | 0.0175 (13) | 0.0154 (14) | 0.0153 (13) | 0.0113 (11) | 0.0058 (11) | 0.0010 (11) |
| C4 | 0.0156 (13) | 0.0162 (15) | 0.0232 (15) | 0.0019 (11) | 0.0062 (12) | 0.0023 (12) |
| C5 | 0.0178 (14) | 0.0135 (14) | 0.0232 (15) | 0.0020 (11) | 0.0074 (12) | 0.0048 (12) |
| C6 | 0.0139 (13) | 0.0130 (14) | 0.0168 (13) | 0.0094 (11) | 0.0081 (11) | 0.0002 (11) |
| C7 | 0.0117 (12) | 0.0118 (14) | 0.0173 (14) | 0.0068 (10) | 0.0057 (11) | 0.0005 (11) |
| C8 | 0.0131 (12) | 0.0112 (13) | 0.0189 (14) | 0.0056 (11) | 0.0080 (11) | 0.0016 (11) |
| C9 | 0.0092 (12) | 0.0084 (13) | 0.0146 (13) | 0.0072 (10) | 0.0028 (10) | -0.0003 (10) |
| C10 | 0.0124 (13) | 0.0136 (14) | 0.0160 (13) | 0.0054 (11) | 0.0064 (11) | 0.0022 (11) |
| C11 | 0.0129 (13) | 0.0131 (14) | 0.0174 (14) | 0.0064 (11) | 0.0065 (11) | 0.0003 (11) |
| C12 | 0.0151 (13) | 0.0130 (14) | 0.0167 (14) | 0.0091 (11) | 0.0036 (11) | 0.0022 (11) |
| C13 | 0.0195 (14) | 0.0142 (15) | 0.0297 (17) | 0.0075 (12) | 0.0130 (13) | -0.0041 (12) |
| C14 | 0.0114 (12) | 0.0164 (15) | 0.0160 (13) | 0.0086 (11) | 0.0041 (11) | 0.0032 (11) |

Geometric parameters (Å, °)

| | | | |
|--------|-----------|--------|-----------|
| C11—C3 | 1.753 (3) | C3—C4 | 1.372 (4) |
| N1—C9 | 1.369 (4) | C4—C5 | 1.391 (4) |
| N1—C10 | 1.378 (4) | C4—H4A | 0.9500 |

| | | | |
|--------------|------------|---------------|------------|
| N1—C13 | 1.474 (4) | C5—C6 | 1.392 (4) |
| N2—C9 | 1.379 (4) | C5—H5A | 0.9500 |
| N2—H1N2 | 0.86 (5) | C6—C7 | 1.543 (4) |
| N2—H2N2 | 0.89 (4) | C7—C8 | 1.505 (4) |
| N3—C10 | 1.366 (4) | C7—C11 | 1.523 (4) |
| N3—H1N3 | 0.83 (5) | C7—H7A | 1.0000 |
| N3—H2N3 | 0.83 (4) | C8—C9 | 1.373 (4) |
| N4—C12 | 1.155 (4) | C8—C12 | 1.410 (4) |
| N5—C14 | 1.162 (4) | C10—C11 | 1.386 (4) |
| C1—C6 | 1.393 (4) | C11—C14 | 1.403 (4) |
| C1—C2 | 1.395 (4) | C13—H13A | 0.9800 |
| C1—H1A | 0.9500 | C13—H13B | 0.9800 |
| C2—C3 | 1.388 (4) | C13—H13C | 0.9800 |
| C2—H2A | 0.9500 | | |
| | | | |
| C9—N1—C10 | 118.3 (2) | C8—C7—C11 | 107.0 (2) |
| C9—N1—C13 | 120.6 (2) | C8—C7—C6 | 113.9 (2) |
| C10—N1—C13 | 119.7 (2) | C11—C7—C6 | 113.9 (2) |
| C9—N2—H1N2 | 117 (3) | C8—C7—H7A | 107.2 |
| C9—N2—H2N2 | 121 (3) | C11—C7—H7A | 107.2 |
| H1N2—N2—H2N2 | 108 (4) | C6—C7—H7A | 107.2 |
| C10—N3—H1N3 | 120 (3) | C9—C8—C12 | 119.9 (3) |
| C10—N3—H2N3 | 113 (3) | C9—C8—C7 | 119.8 (3) |
| H1N3—N3—H2N3 | 115 (4) | C12—C8—C7 | 120.3 (2) |
| C6—C1—C2 | 121.3 (3) | C8—C9—N1 | 120.7 (3) |
| C6—C1—H1A | 119.4 | C8—C9—N2 | 123.1 (3) |
| C2—C1—H1A | 119.4 | N1—C9—N2 | 116.2 (2) |
| C3—C2—C1 | 118.4 (3) | N3—C10—N1 | 116.7 (3) |
| C3—C2—H2A | 120.8 | N3—C10—C11 | 123.7 (3) |
| C1—C2—H2A | 120.8 | N1—C10—C11 | 119.6 (3) |
| C4—C3—C2 | 121.8 (3) | C10—C11—C14 | 119.7 (3) |
| C4—C3—C11 | 119.6 (2) | C10—C11—C7 | 119.9 (3) |
| C2—C3—C11 | 118.5 (2) | C14—C11—C7 | 120.4 (2) |
| C3—C4—C5 | 118.9 (3) | N4—C12—C8 | 178.0 (3) |
| C3—C4—H4A | 120.5 | N1—C13—H13A | 109.5 |
| C5—C4—H4A | 120.5 | N1—C13—H13B | 109.5 |
| C4—C5—C6 | 121.3 (3) | H13A—C13—H13B | 109.5 |
| C4—C5—H5A | 119.3 | N1—C13—H13C | 109.5 |
| C6—C5—H5A | 119.3 | H13A—C13—H13C | 109.5 |
| C1—C6—C5 | 118.2 (3) | H13B—C13—H13C | 109.5 |
| C1—C6—C7 | 121.4 (3) | N5—C14—C11 | 177.8 (3) |
| C5—C6—C7 | 120.1 (3) | | |
| | | | |
| C6—C1—C2—C3 | 0.3 (5) | C7—C8—C9—N1 | 8.9 (4) |
| C1—C2—C3—C4 | 0.7 (5) | C12—C8—C9—N2 | 10.8 (4) |
| C1—C2—C3—C11 | -180.0 (2) | C7—C8—C9—N2 | -169.8 (3) |
| C2—C3—C4—C5 | -0.8 (5) | C10—N1—C9—C8 | 23.8 (4) |
| C11—C3—C4—C5 | 179.9 (2) | C13—N1—C9—C8 | -169.9 (3) |

| | | | |
|---------------|------------|----------------|------------|
| C3—C4—C5—C6 | -0.2 (5) | C10—N1—C9—N2 | -157.4 (3) |
| C2—C1—C6—C5 | -1.3 (4) | C13—N1—C9—N2 | 8.9 (4) |
| C2—C1—C6—C7 | -175.4 (3) | C9—N1—C10—N3 | 156.2 (3) |
| C4—C5—C6—C1 | 1.2 (4) | C13—N1—C10—N3 | -10.2 (4) |
| C4—C5—C6—C7 | 175.4 (3) | C9—N1—C10—C11 | -25.5 (4) |
| C1—C6—C7—C8 | -152.0 (3) | C13—N1—C10—C11 | 168.1 (3) |
| C5—C6—C7—C8 | 34.0 (3) | N3—C10—C11—C14 | -8.3 (4) |
| C1—C6—C7—C11 | -28.9 (4) | N1—C10—C11—C14 | 173.5 (3) |
| C5—C6—C7—C11 | 157.1 (3) | N3—C10—C11—C7 | 173.1 (3) |
| C11—C7—C8—C9 | -34.8 (3) | N1—C10—C11—C7 | -5.1 (4) |
| C6—C7—C8—C9 | 92.0 (3) | C8—C7—C11—C10 | 32.9 (4) |
| C11—C7—C8—C12 | 144.5 (3) | C6—C7—C11—C10 | -93.9 (3) |
| C6—C7—C8—C12 | -88.7 (3) | C8—C7—C11—C14 | -145.7 (3) |
| C12—C8—C9—N1 | -170.4 (3) | C6—C7—C11—C14 | 87.6 (3) |

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> |
|-----------------------------|-------------|---------------|-----------------------|-------------------------|
| N2—H1N2...N5 ⁱ | 0.86 (5) | 2.21 (5) | 3.043 (4) | 163 (5) |
| N2—H2N2...C11 ⁱⁱ | 0.89 (4) | 2.75 (4) | 3.588 (3) | 158 (3) |
| N3—H2N3...N4 ⁱⁱⁱ | 0.83 (4) | 2.27 (4) | 3.071 (4) | 161 (4) |

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