

3,3'-[1,4-Phenylenebis(methylene)]bis-(1-propylbenzimidazolium) dichloride dihydrate

Rosenani A. Haque,^a Muhammad Adnan Iqbal,^a Safaa A. Ahmad,^b Tze Shyang Chia^c and Hoong-Kun Fun^{c*†}

^aSchool of Chemical Sciences, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, ^bDepartment of Chemistry, College of Education Samarra, University of Tikrit, Tikrit 43001, Iraq, and ^cX-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia
Correspondence e-mail: hkfun@usm.my

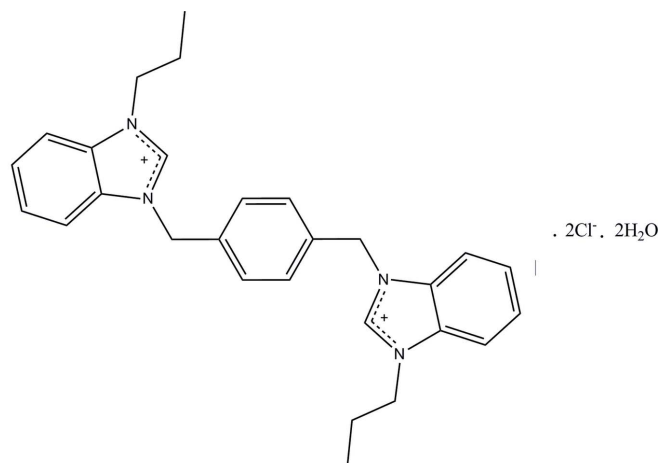
Received 18 February 2012; accepted 21 February 2012

Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.061; wR factor = 0.196; data-to-parameter ratio = 17.9.

The asymmetric unit of the title compound, $\text{C}_{28}\text{H}_{32}\text{N}_4^{2+} \cdot 2\text{Cl}^- \cdot 2\text{H}_2\text{O}$, contains half of a 3,3'-[1,4-phenylenebis(methylene)]-bis(1-propylbenzimidazolium) cation, one chloride anion and one water molecule. The complete cation is generated by a crystallographic inversion center. The central benzene ring forms a dihedral angle of $66.06(11)^\circ$ with its adjacent benzimidazolium ring system. In the crystal, the cations, anions and water molecules are linked by $\text{O}-\text{H} \cdots \text{Cl}$, $\text{C}-\text{H} \cdots \text{O}$ and $\text{C}-\text{H} \cdots \text{Cl}$ hydrogen bonds into a three-dimensional network. The crystal packing is further stabilized by $\pi-\pi$ interactions, with centroid-centroid distances of $3.5561(15)$ and $3.6708(15)$ Å.

Related literature

For details and applications of benzimidazole derivatives, see: Narasimhan *et al.* (2012). For related structures, see: Haque *et al.* (2011, 2012); Iqbal *et al.* (2012). For reference bond lengths, see: Allen *et al.* (1987). For the stability of the temperature controller used for data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

$\text{C}_{28}\text{H}_{32}\text{N}_4^{2+} \cdot 2\text{Cl}^- \cdot 2\text{H}_2\text{O}$
 $M_r = 531.51$
Monoclinic, $P2_1/c$
 $a = 8.1177(5)$ Å
 $b = 9.1042(5)$ Å
 $c = 18.3548(11)$ Å
 $\beta = 94.323(2)^\circ$

$V = 1352.66(14)$ Å³
 $Z = 2$
Mo $K\alpha$ radiation
 $\mu = 0.27$ mm⁻¹
 $T = 100$ K
 $0.47 \times 0.23 \times 0.14$ mm

Data collection

Bruker SMART APEXII CCD
area-detector diffractometer
Absorption correction: multi-scan
(SADABS; Bruker, 2009)
 $T_{\min} = 0.882$, $T_{\max} = 0.962$

11942 measured reflections
3085 independent reflections
2660 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.053$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.061$
 $wR(F^2) = 0.196$
 $S = 1.08$
3085 reflections
172 parameters

H atoms treated by a mixture of
independent and constrained
refinement
 $\Delta\rho_{\max} = 0.86$ e Å⁻³
 $\Delta\rho_{\min} = -0.48$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H} \cdots A$ | $D-\text{H}$ | $\text{H} \cdots A$ | $D \cdots A$ | $D-\text{H} \cdots A$ |
|---|--------------|---------------------|--------------|-----------------------|
| $\text{O1W}-\text{H1W1} \cdots \text{Cl1}$ | 0.93 (4) | 2.29 (4) | 3.200 (3) | 166 (4) |
| $\text{O1W}-\text{H2W1} \cdots \text{Cl1}^{\text{i}}$ | 0.84 (5) | 2.30 (4) | 3.130 (3) | 170 (4) |
| $\text{C1}-\text{H1A} \cdots \text{Cl1}^{\text{ii}}$ | 0.95 | 2.81 | 3.677 (3) | 153 |
| $\text{C4}-\text{H4B} \cdots \text{Cl1}^{\text{i}}$ | 0.99 | 2.77 | 3.741 (3) | 167 |
| $\text{C6}-\text{H6A} \cdots \text{Cl1}^{\text{ii}}$ | 0.95 | 2.76 | 3.691 (3) | 165 |
| $\text{C11}-\text{H11A} \cdots \text{O1W}$ | 0.95 | 2.14 | 3.059 (4) | 163 |
| $\text{C12}-\text{H12A} \cdots \text{Cl1}$ | 0.99 | 2.79 | 3.747 (3) | 164 |
| $\text{C12}-\text{H12B} \cdots \text{Cl1}^{\text{iii}}$ | 0.99 | 2.75 | 3.740 (3) | 175 |

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

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-3561-2009.

RAH thanks Universiti Sains Malaysia (USM) for the Research University (RU) grants Nos. 1001/PKIMIA/811157 and 1001/PKIMIA/823082. MAI is grateful to (IPS) USM for financial support [fellowship USM.IPS/JWT/1/19 (JLD 6)] and a research attachment fund [P-KM0018/10(R) – 308/AIPS/415401]. HKF and TSC thank USM for the RU grant No. 1001/PFIZIK/811160. TSC thanks the Malaysian Government and USM for the award of the post of Research Officer under the RU grant No. 1001/PSKBP/8630013.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS5076).

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
- Bruker (2009). *APEX2, SAINT and SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.
- Haque, R. A., Iqbal, M. A., Budagumpi, S., Hemamalini, M. & Fun, H.-K. (2012). *Acta Cryst.* **E68**, o573.
- Haque, R. A., Iqbal, M. A., Hemamalini, M. & Fun, H.-K. (2011). *Acta Cryst.* **E67**, o1814–o1815.
- Iqbal, M. A., Haque, R. A., Fun, H.-K. & Chia, T. S. (2012). *Acta Cryst.* **E68**, o466–o467.
- Narasimhan, B., Sharma, D. & Kumar, P. (2012). *Med. Chem. Res.* **21**, 269–283.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.

supplementary materials

Acta Cryst. (2012). E68, o845–o846 [doi:10.1107/S1600536812007738]

3,3'-[1,4-Phenylenebis(methylene)]bis(1-propylbenzimidazolium) dichloride dihydrate

Rosenani A. Haque, Muhammad Adnan Iqbal, Safaa A. Ahmad, Tze Shyang Chia and Hoong-Kun Fun

Comment

Benzimidazole constituted compounds have diverse biological and clinical applications (Narasimhan *et al.*, 2012). Previously, we have reported crystal structures of *ortho*-xylyl linked *bis*-benzimidazolium salts with heptyl (Haque *et al.*, 2011), propyl (Iqbal *et al.*, 2012), and ethyl (Haque *et al.*, 2012) substitutions. In this report, we describe the crystal structure of a *para*-xylyl linked *bis*-benzimidazolium salt with propyl substitutions.

The molecular structure of the title compound is shown in Fig. 1. The asymmetric unit of the title compound, $C_{28}H_{32}N_4^{2+} \cdot 2Cl^- \cdot 2H_2O$, consists of one half-molecule of 3,3'-[1,4-phenylenebis(methylene)]bis(1-propylbenzimidazolium) cation, one chlorine anion and one water molecule. The complete cation is generated by a crystallographic inversion center ($-x, -y + 2, -z$). The central benzene ring (C1–C3/C1A–C3A) forms a dihedral angle of 66.06 (11)° with its adjacent benzimidazolium ring (N1/N2/C5–C11) [maximum deviation = 0.031 (2) Å at atom C5]. Bond lengths (Allen *et al.*, 1987) and angles are within normal ranges and are comparable to related structures (Haque *et al.*, 2011,2012; Iqbal *et al.*, 2012).

In the crystal structure, (Fig. 2), the cations, anions and water molecules are linked by intermolecular O1W—H1W1...C11, O1W—H2W1...C11, C1—H1A...C11, C4—H4B...C11, C6—H6A...C11, C11—H11A...O1W, C12—H12A...C11, and C12—H12B...C11 hydrogen bonds (Table 1) into a three-dimensional network. The crystal packing is further stabilized by π – π interactions with $Cg1 \cdots Cg3$ ($1-x, 1-y, -z$) distance = 3.5561 (15) Å and $Cg3 \cdots Cg3$ ($1-x, 1-y, -z$) distance = 3.6708 (15) Å, where $Cg1$ and $Cg3$ are the centroids of N1/N2/C5/C10/C11 and C5–C10 rings, respectively.

Experimental

A mixture of benzimidazole (2.95 g, 25 mmol) and finely ground potassium hydroxide (2.36 g, 30 mmol) in 30 ml of DMSO was stirred at room temperature (27–28 °C) for 30 min. 1-Bromopropane (2.27 ml, 25 mmol) was added dropwise into this consistently stirred mixture with further stirring for 2 h at the same temperature. The mixture was then poured into water (400 ml) and was extracted by chloroform (5 x 20 ml). The extract was dried by magnesium sulfate and evaporated under reduced pressure to get *N*-ethylbenzimidazole (**1**) as a thick yellowish fluid. Furthermore, a mixture of **1** (1.60 g, 10 mmol) and 1,4-bis(chloromethyl)benzene (0.88 g, 5 mmol) in dioxane (30 ml) was refluxed at 100 °C for 18 h. This desired compound (**2.2Cl**) appeared as white precipitates in the light brown solution. The mixture was filtered and the precipitates were washed with fresh dioxane (3 x 5 ml), dried at room temperature for 24 h, and the soft lumps obtained were ground into a fine powder (2.15 g, 87%). Saturated solution of **2.2Cl** in methanol (0.5 ml) was exposed to diethyl ether vapours (vapour diffusion) at room temperature overnight to get single crystals suitable for X-ray diffraction study.

Refinement

Atoms H1W1 and H2W1 were located in a difference Fourier map and refined freely. The remaining H atoms were positioned geometrically [C—H = 0.95, 0.98 or 0.99 Å] and refined using a riding model with $U_{\text{iso}}(\text{H}) = 1.2$ or $1.5U_{\text{eq}}(\text{C})$. A rotating group model was applied to the methyl group.

Computing details

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT* (Bruker, 2009); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

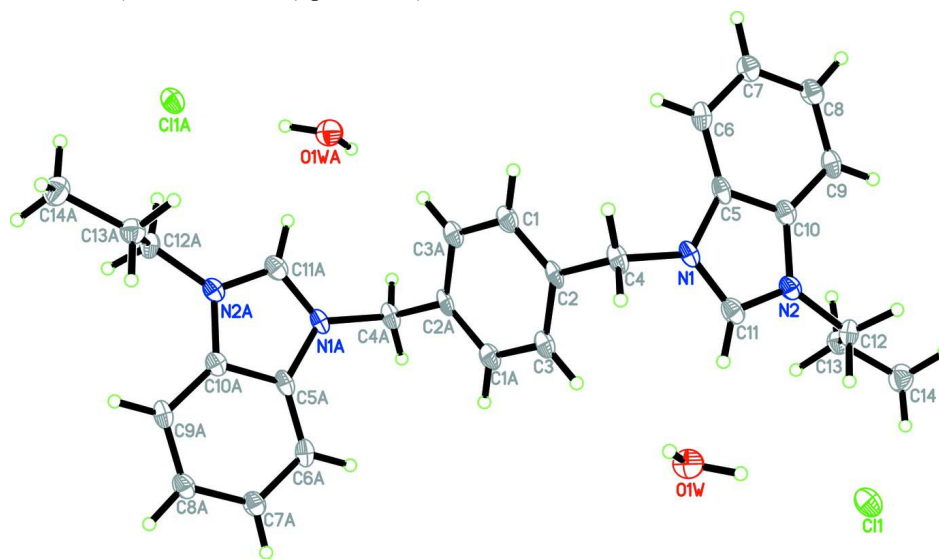


Figure 1

The molecular structure of the title compound with atom labels and 50% probability displacement ellipsoids. Atoms with suffix A were generated by symmetry code $-x, -y + 2, -z$.

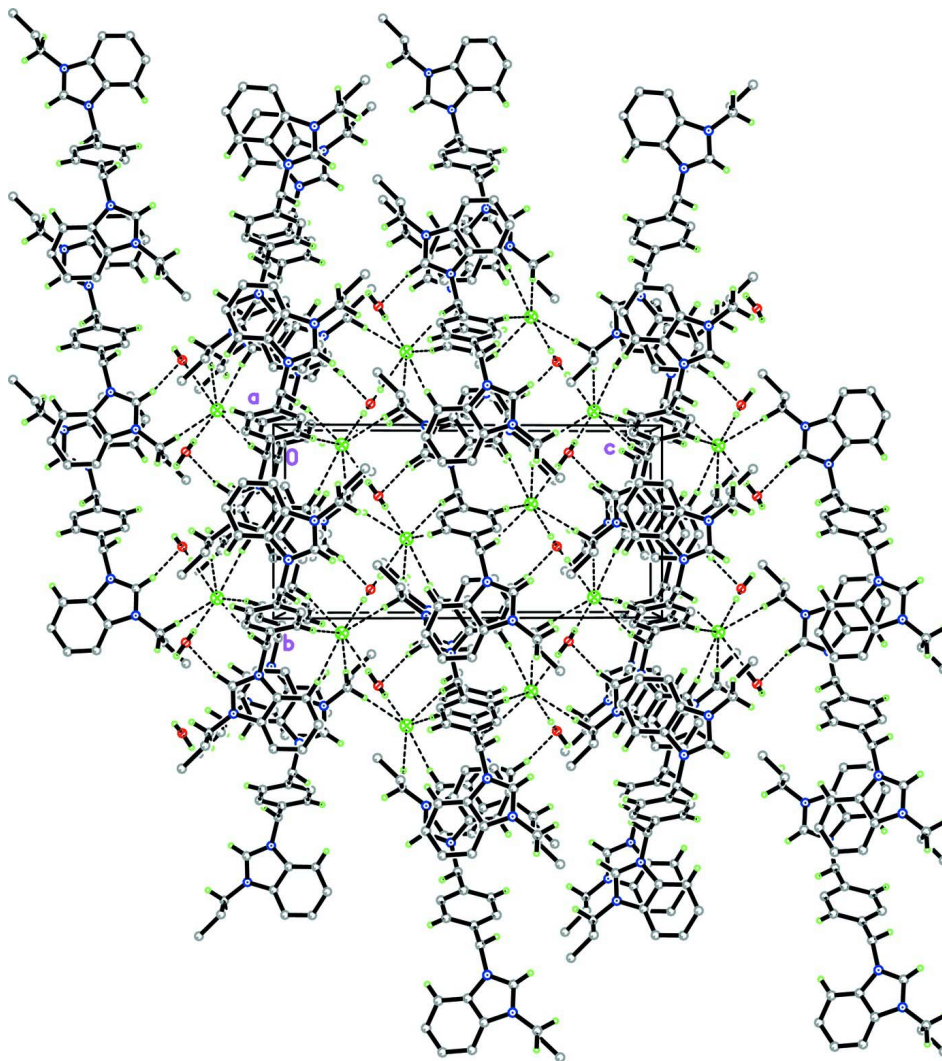


Figure 2

The crystal packing of the title compound. Intermolecular hydrogen bonds are shown as dashed lines. H atoms not involved in the hydrogen bonds have been omitted for clarity.

3,3'-[1,4-Phenylenebis(methylene)]bis(1-propylbenzimidazolium) dichloride dihydrate

Crystal data

$C_{28}H_{32}N_4^{2+} \cdot 2Cl^- \cdot 2H_2O$

$M_r = 531.51$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2ybc$

$a = 8.1177$ (5) Å

$b = 9.1042$ (5) Å

$c = 18.3548$ (11) Å

$\beta = 94.323$ (2)°

$V = 1352.66$ (14) Å³

$Z = 2$

$F(000) = 564$

$D_x = 1.305$ Mg m⁻³

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

Cell parameters from 5809 reflections

$\theta = 2.5$ – 32.5 °

$\mu = 0.27$ mm⁻¹

$T = 100$ K

Block, colourless

$0.47 \times 0.23 \times 0.14$ mm

Data collection

| | |
|--|--|
| Bruker SMART APEXII CCD area-detector diffractometer | 11942 measured reflections |
| Radiation source: fine-focus sealed tube | 3085 independent reflections |
| Graphite monochromator | 2660 reflections with $I > 2\sigma(I)$ |
| φ and ω scans | $R_{\text{int}} = 0.053$ |
| Absorption correction: multi-scan (SADABS; Bruker, 2009) | $\theta_{\text{max}} = 27.5^\circ$, $\theta_{\text{min}} = 2.2^\circ$ |
| $T_{\text{min}} = 0.882$, $T_{\text{max}} = 0.962$ | $h = -10 \rightarrow 10$ |
| | $k = -11 \rightarrow 11$ |
| | $l = -22 \rightarrow 23$ |

Refinement

| | |
|--|--|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.061$ | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2) = 0.196$ | $w = 1/[\sigma^2(F_o^2) + (0.1065P)^2 + 2.1866P]$ |
| $S = 1.08$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 3085 reflections | $(\Delta/\sigma)_{\text{max}} < 0.001$ |
| 172 parameters | $\Delta\rho_{\text{max}} = 0.86 \text{ e } \text{\AA}^{-3}$ |
| 0 restraints | $\Delta\rho_{\text{min}} = -0.48 \text{ e } \text{\AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | |

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)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|-------------|-------------|---------------|----------------------------------|
| C11 | 0.45230 (8) | 0.59270 (7) | 0.33489 (4) | 0.0240 (2) |
| O1W | 0.3206 (3) | 0.8654 (3) | 0.23712 (12) | 0.0269 (5) |
| N1 | 0.2941 (3) | 0.6933 (2) | 0.04689 (12) | 0.0175 (5) |
| N2 | 0.2605 (3) | 0.4951 (2) | 0.11124 (12) | 0.0179 (5) |
| C1 | 0.0921 (3) | 0.9600 (3) | -0.05784 (15) | 0.0201 (5) |
| H1A | 0.1549 | 0.9329 | -0.0974 | 0.024* |
| C2 | 0.1507 (3) | 0.9274 (3) | 0.01358 (15) | 0.0175 (5) |
| C3 | 0.0581 (3) | 0.9677 (3) | 0.07154 (15) | 0.0201 (5) |
| H3A | 0.0977 | 0.9458 | 0.1203 | 0.024* |
| C4 | 0.3152 (3) | 0.8500 (3) | 0.02876 (15) | 0.0206 (5) |
| H4A | 0.3796 | 0.8579 | -0.0148 | 0.025* |
| H4B | 0.3786 | 0.8994 | 0.0699 | 0.025* |
| C5 | 0.2927 (3) | 0.5759 (3) | -0.00194 (15) | 0.0170 (5) |
| C6 | 0.3152 (3) | 0.5696 (3) | -0.07626 (15) | 0.0194 (5) |

| | | | | |
|------|------------|------------|---------------|-------------|
| H6A | 0.3306 | 0.6557 | -0.1042 | 0.023* |
| C7 | 0.3140 (3) | 0.4312 (3) | -0.10732 (15) | 0.0199 (5) |
| H7A | 0.3285 | 0.4223 | -0.1580 | 0.024* |
| C8 | 0.2918 (3) | 0.3032 (3) | -0.06619 (15) | 0.0206 (5) |
| H8A | 0.2917 | 0.2104 | -0.0898 | 0.025* |
| C9 | 0.2701 (3) | 0.3095 (3) | 0.00817 (15) | 0.0199 (5) |
| H9A | 0.2553 | 0.2235 | 0.0362 | 0.024* |
| C10 | 0.2715 (3) | 0.4492 (3) | 0.03924 (14) | 0.0172 (5) |
| C11 | 0.2750 (3) | 0.6401 (3) | 0.11360 (15) | 0.0197 (5) |
| H11A | 0.2722 | 0.6979 | 0.1566 | 0.024* |
| C12 | 0.2412 (3) | 0.3977 (3) | 0.17406 (14) | 0.0197 (5) |
| H12A | 0.2731 | 0.4515 | 0.2198 | 0.024* |
| H12B | 0.3164 | 0.3125 | 0.1713 | 0.024* |
| C13 | 0.0641 (3) | 0.3427 (3) | 0.17592 (14) | 0.0208 (5) |
| H13A | 0.0267 | 0.3006 | 0.1278 | 0.025* |
| H13B | -0.0093 | 0.4262 | 0.1856 | 0.025* |
| C14 | 0.0517 (4) | 0.2262 (3) | 0.23502 (16) | 0.0276 (6) |
| H14A | -0.0607 | 0.1865 | 0.2324 | 0.041* |
| H14B | 0.0779 | 0.2705 | 0.2832 | 0.041* |
| H14C | 0.1301 | 0.1468 | 0.2274 | 0.041* |
| H1W1 | 0.377 (6) | 0.792 (5) | 0.264 (2) | 0.050 (12)* |
| H2W1 | 0.392 (6) | 0.920 (5) | 0.220 (2) | 0.046 (12)* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C11 | 0.0237 (4) | 0.0186 (4) | 0.0300 (4) | -0.0012 (2) | 0.0043 (3) | -0.0029 (2) |
| O1W | 0.0269 (11) | 0.0265 (12) | 0.0276 (11) | -0.0050 (9) | 0.0037 (8) | -0.0007 (9) |
| N1 | 0.0159 (10) | 0.0109 (10) | 0.0254 (11) | 0.0014 (8) | 0.0009 (8) | -0.0022 (8) |
| N2 | 0.0163 (10) | 0.0152 (11) | 0.0219 (11) | 0.0002 (8) | 0.0001 (8) | -0.0025 (8) |
| C1 | 0.0189 (12) | 0.0129 (12) | 0.0289 (14) | -0.0010 (9) | 0.0052 (10) | -0.0004 (10) |
| C2 | 0.0162 (11) | 0.0066 (11) | 0.0300 (14) | -0.0004 (8) | 0.0041 (9) | -0.0003 (9) |
| C3 | 0.0210 (12) | 0.0111 (12) | 0.0281 (13) | -0.0007 (9) | 0.0024 (10) | -0.0003 (10) |
| C4 | 0.0173 (12) | 0.0119 (12) | 0.0328 (14) | 0.0002 (9) | 0.0032 (10) | 0.0003 (10) |
| C5 | 0.0116 (11) | 0.0110 (12) | 0.0280 (13) | 0.0000 (8) | -0.0012 (9) | -0.0017 (9) |
| C6 | 0.0154 (12) | 0.0143 (12) | 0.0281 (14) | -0.0004 (9) | -0.0010 (9) | 0.0032 (10) |
| C7 | 0.0184 (12) | 0.0169 (13) | 0.0242 (13) | -0.0016 (9) | -0.0004 (9) | -0.0020 (10) |
| C8 | 0.0204 (12) | 0.0159 (13) | 0.0251 (13) | -0.0017 (10) | -0.0010 (9) | -0.0038 (10) |
| C9 | 0.0171 (11) | 0.0124 (12) | 0.0301 (14) | -0.0004 (9) | 0.0013 (9) | 0.0013 (10) |
| C10 | 0.0122 (11) | 0.0168 (13) | 0.0224 (13) | 0.0013 (9) | -0.0010 (9) | -0.0009 (10) |
| C11 | 0.0142 (11) | 0.0191 (13) | 0.0257 (13) | 0.0028 (9) | 0.0001 (9) | -0.0029 (10) |
| C12 | 0.0186 (12) | 0.0179 (13) | 0.0223 (13) | 0.0027 (9) | -0.0013 (9) | 0.0042 (10) |
| C13 | 0.0175 (12) | 0.0221 (14) | 0.0227 (13) | 0.0023 (10) | 0.0017 (9) | -0.0001 (10) |
| C14 | 0.0257 (14) | 0.0284 (16) | 0.0291 (15) | 0.0031 (11) | 0.0054 (11) | 0.0054 (12) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|----------|-----------|--------|-----------|
| O1W—H1W1 | 0.93 (5) | C6—C7 | 1.382 (4) |
| O1W—H2W1 | 0.84 (5) | C6—H6A | 0.9500 |
| N1—C11 | 1.337 (3) | C7—C8 | 1.407 (4) |

| | | | |
|-------------------------|-----------|---------------|-----------|
| N1—C5 | 1.395 (3) | C7—H7A | 0.9500 |
| N1—C4 | 1.478 (3) | C8—C9 | 1.390 (4) |
| N2—C11 | 1.326 (4) | C8—H8A | 0.9500 |
| N2—C10 | 1.395 (3) | C9—C10 | 1.393 (4) |
| N2—C12 | 1.472 (3) | C9—H9A | 0.9500 |
| C1—C3 ⁱ | 1.392 (4) | C11—H11A | 0.9500 |
| C1—C2 | 1.393 (4) | C12—C13 | 1.526 (4) |
| C1—H1A | 0.9500 | C12—H12A | 0.9900 |
| C2—C3 | 1.397 (4) | C12—H12B | 0.9900 |
| C2—C4 | 1.517 (3) | C13—C14 | 1.526 (4) |
| C3—C1 ⁱ | 1.392 (4) | C13—H13A | 0.9900 |
| C3—H3A | 0.9500 | C13—H13B | 0.9900 |
| C4—H4A | 0.9900 | C14—H14A | 0.9800 |
| C4—H4B | 0.9900 | C14—H14B | 0.9800 |
| C5—C6 | 1.391 (4) | C14—H14C | 0.9800 |
| C5—C10 | 1.397 (4) | | |
| H1W1—O1W—H2W1 | 107 (4) | C9—C8—C7 | 121.6 (2) |
| C11—N1—C5 | 108.4 (2) | C9—C8—H8A | 119.2 |
| C11—N1—C4 | 125.4 (2) | C7—C8—H8A | 119.2 |
| C5—N1—C4 | 126.2 (2) | C8—C9—C10 | 116.2 (2) |
| C11—N2—C10 | 108.5 (2) | C8—C9—H9A | 121.9 |
| C11—N2—C12 | 126.1 (2) | C10—C9—H9A | 121.9 |
| C10—N2—C12 | 125.4 (2) | C9—C10—N2 | 131.5 (2) |
| C3 ⁱ —C1—C2 | 120.2 (2) | C9—C10—C5 | 122.0 (2) |
| C3 ⁱ —C1—H1A | 119.9 | N2—C10—C5 | 106.5 (2) |
| C2—C1—H1A | 119.9 | N2—C11—N1 | 110.3 (2) |
| C1—C2—C3 | 119.7 (2) | N2—C11—H11A | 124.8 |
| C1—C2—C4 | 120.4 (2) | N1—C11—H11A | 124.8 |
| C3—C2—C4 | 119.9 (2) | N2—C12—C13 | 111.8 (2) |
| C1 ⁱ —C3—C2 | 120.1 (3) | N2—C12—H12A | 109.3 |
| C1 ⁱ —C3—H3A | 120.0 | C13—C12—H12A | 109.3 |
| C2—C3—H3A | 120.0 | N2—C12—H12B | 109.3 |
| N1—C4—C2 | 112.0 (2) | C13—C12—H12B | 109.3 |
| N1—C4—H4A | 109.2 | H12A—C12—H12B | 107.9 |
| C2—C4—H4A | 109.2 | C12—C13—C14 | 110.9 (2) |
| N1—C4—H4B | 109.2 | C12—C13—H13A | 109.5 |
| C2—C4—H4B | 109.2 | C14—C13—H13A | 109.5 |
| H4A—C4—H4B | 107.9 | C12—C13—H13B | 109.5 |
| C6—C5—N1 | 131.8 (2) | C14—C13—H13B | 109.5 |
| C6—C5—C10 | 121.8 (2) | H13A—C13—H13B | 108.1 |
| N1—C5—C10 | 106.3 (2) | C13—C14—H14A | 109.5 |
| C7—C6—C5 | 116.4 (2) | C13—C14—H14B | 109.5 |
| C7—C6—H6A | 121.8 | H14A—C14—H14B | 109.5 |
| C5—C6—H6A | 121.8 | C13—C14—H14C | 109.5 |
| C6—C7—C8 | 122.0 (2) | H14A—C14—H14C | 109.5 |
| C6—C7—H7A | 119.0 | H14B—C14—H14C | 109.5 |
| C8—C7—H7A | 119.0 | | |

| | | | |
|---------------------------|------------|----------------|------------|
| C3 ⁱ —C1—C2—C3 | -0.1 (4) | C8—C9—C10—N2 | -177.0 (2) |
| C3 ⁱ —C1—C2—C4 | -179.4 (2) | C8—C9—C10—C5 | 0.2 (4) |
| C1—C2—C3—C1 ⁱ | 0.1 (4) | C11—N2—C10—C9 | 177.2 (3) |
| C4—C2—C3—C1 ⁱ | 179.4 (2) | C12—N2—C10—C9 | -1.1 (4) |
| C11—N1—C4—C2 | -86.9 (3) | C11—N2—C10—C5 | -0.3 (3) |
| C5—N1—C4—C2 | 94.0 (3) | C12—N2—C10—C5 | -178.6 (2) |
| C1—C2—C4—N1 | -103.9 (3) | C6—C5—C10—C9 | -0.6 (4) |
| C3—C2—C4—N1 | 76.9 (3) | N1—C5—C10—C9 | -177.7 (2) |
| C11—N1—C5—C6 | -176.6 (3) | C6—C5—C10—N2 | 177.2 (2) |
| C4—N1—C5—C6 | 2.6 (4) | N1—C5—C10—N2 | 0.1 (3) |
| C11—N1—C5—C10 | 0.1 (3) | C10—N2—C11—N1 | 0.3 (3) |
| C4—N1—C5—C10 | 179.3 (2) | C12—N2—C11—N1 | 178.6 (2) |
| N1—C5—C6—C7 | 176.8 (2) | C5—N1—C11—N2 | -0.3 (3) |
| C10—C5—C6—C7 | 0.6 (4) | C4—N1—C11—N2 | -179.5 (2) |
| C5—C6—C7—C8 | -0.2 (4) | C11—N2—C12—C13 | 104.6 (3) |
| C6—C7—C8—C9 | -0.1 (4) | C10—N2—C12—C13 | -77.4 (3) |
| C7—C8—C9—C10 | 0.1 (4) | N2—C12—C13—C14 | 172.3 (2) |

Symmetry code: (i) $-x, -y+2, -z$.

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

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|---|-------------|---------------------|----------------------------|-------------------------------|
| O1 <i>W</i> —H1 <i>W</i> 1 \cdots C11 | 0.93 (4) | 2.29 (4) | 3.200 (3) | 166 (4) |
| O1 <i>W</i> —H2 <i>W</i> 1 \cdots C11 ⁱⁱ | 0.84 (5) | 2.30 (4) | 3.130 (3) | 170 (4) |
| C1—H1 <i>A</i> \cdots C11 ⁱⁱⁱ | 0.95 | 2.81 | 3.677 (3) | 153 |
| C4—H4 <i>B</i> \cdots C11 ⁱⁱ | 0.99 | 2.77 | 3.741 (3) | 167 |
| C6—H6 <i>A</i> \cdots C11 ⁱⁱⁱ | 0.95 | 2.76 | 3.691 (3) | 165 |
| C11—H11 <i>A</i> \cdots O1 <i>W</i> | 0.95 | 2.14 | 3.059 (4) | 163 |
| C12—H12 <i>A</i> \cdots C11 | 0.99 | 2.79 | 3.747 (3) | 164 |
| C12—H12 <i>B</i> \cdots C11 ^{iv} | 0.99 | 2.75 | 3.740 (3) | 175 |

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