# organic compounds

# 2,6-Bis(1H-benzimidazol-2-yl)pyridine butyric acid monosolvate dihydrate

## Songzhu Lin,* Ruokun Jia and Aimin He

Northeast Dianli University, Jilin 132012, People's Republic of China
Correspondence e-mail: songzhulin@hotmail.com

In the title compound, $C_{19}H_{13}N_5 \cdot C_4H_8O_2 \cdot 2H_2O$, the molecular skeleton of the 2,6-bis(benzimidazol-2-yl)pyridine (bbip) molecule is essentially planar (r.m.s. deviation = 0.023 Å). An extensive three-dimensional network of intermolecular N—H⋯O, O—H⋯O and O—H⋯N hydrogen bonds consolidates the crystal packing, which also exhibits $\pi$–$\pi$ interactions between the five- and six-membered rings from neighbouring bbip molecules.

## Related literature

For background to supramolecular interactions, see: Dale *et al.* (2004); Braga *et al.* (2005); Ring *et al.* (2006). For related structures, see: Freire *et al.* (2003); Xiao *et al.* (2010).
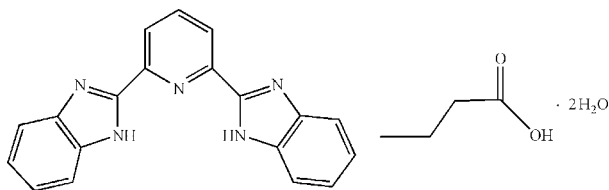


## Experimental

### Crystal data

| | |
|---|---|
| $C_{19}H_{13}N_5 \cdot C_4H_8O_2 \cdot 2H_2O$ | $\gamma = 104.76\ (3)°$ |
| $M_r = 435.48$ | $V = 1131.5\ (4)$ Å$^3$ |
| Triclinic, $P\bar{1}$ | $Z = 2$ |
| $a = 9.3950\ (19)$ Å | Mo $K\alpha$ radiation |
| $b = 9.5611\ (19)$ Å | $\mu = 0.09$ mm$^{-1}$ |
| $c = 13.805\ (3)$ Å | $T = 295$ K |
| $\alpha = 103.27\ (2)°$ | $0.20 \times 0.18 \times 0.15$ mm |
| $\beta = 99.91\ (3)°$ | |

### Data collection

| | |
|---|---|
| Enraf–Nonius CAD-4 diffractometer | 3981 independent reflections |
| Absorption correction: $\psi$ scan (North *et al.*, 1968) | 3195 reflections with $I > 2\sigma(I)$ |
| | $R_{int} = 0.019$ |
| $T_{min} = 0.982,\ T_{max} = 0.987$ | 3 standard reflections every 100 reflections |
| 8683 measured reflections | intensity decay: none |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)] = 0.045$ | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2) = 0.144$ | |
| $S = 1.10$ | |
| 3981 reflections | $\Delta\rho_{max} = 0.22$ e Å$^{-3}$ |
| 318 parameters | $\Delta\rho_{min} = -0.25$ e Å$^{-3}$ |
| 6 restraints | |

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

| $D$—H⋯$A$ | $D$—H | H⋯$A$ | $D$⋯$A$ | $D$—H⋯$A$ |
|---|---|---|---|---|
| N1—H2⋯O2W | 0.90 (2) | 2.12 (1) | 3.006 (2) | 166 (2) |
| N5—H1⋯O2W | 0.86 (2) | 2.234 (19) | 3.083 (2) | 169.1 (17) |
| O1W—H2W1⋯N4 | 0.83 (2) | 1.96 (1) | 2.7901 (19) | 178 (3) |
| O2W—H2W2⋯O1W$^i$ | 0.82 (2) | 2.04 (1) | 2.852 (2) | 168 (3) |
| O2W—H1W2⋯O1W$^{ii}$ | 0.81 (2) | 2.06 (1) | 2.856 (2) | 168 (3) |
| O1W—H1W1⋯O1$^{iii}$ | 0.82 (2) | 2.00 (1) | 2.795 (2) | 166 (3) |
| O2—H3⋯N2 | 0.85 (2) | 1.89 (1) | 2.712 (2) | 164 (2) |

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5296).

## References

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# supporting information

## 2,6-Bis(1*H*-benzimidazol-2-yl)pyridine butyric acid monosolvate dihydrate

**Songzhu Lin, Ruokun Jia and Aimin He**

### S1. Comment

Crystal engineering of extended frameworks can readily be achieved using a variety of supramolecular interactions (Braga *et al.*, 2005; Ring *et al.*, 2006). However, the prediction of the solid state packing of simple organic cocrystals still remains an ongoing challenge (Dale *et al.*, 2004). The supramolecular interactions, such as hydrogen bonding and $\pi$-$\pi$ stacking interactions, can create network materials with infinite 1-D, 2-D or 3-D structural motifs. The supramolecular structures with 2,6-bis(benzimidazol-2-yl)pyridine have been reported in recent year (Freire *et al.*, 2003; Xiao *et al.*, 2010). As a continuation of those works, we report the crystal structure of the title compound (I).

In (I) (Fig. 1), all bond lengths and angles are normal and correspond to those observed in the related structures (Freire *et al.*, 2003; Xiao *et al.*, 2010). The aromatic C—C and C—N bond lengths in both the benzimidazole and pyridine rings are within the usual range. All C and N atoms in the 2,6-bis(benzimidazol-2-yl)pyridine molecule are almost coplanr with the largest deviation of 0.060 Å for C4.

In the crystal, the N—H···O, N—H···O and O—H···O hydrogen bonds (Table 1) consolidate the packing, which exhibits $\pi$–$\pi$ interactions with the following center-to-center distances - $Cg1\cdots Cg1$=3.625 (2) Å and $Cg2\cdots Cg3$=3.775 (2) Å, where $Cg1$, $Cg2$ and $Cg3$ are centroids of N4/C13/N5/C19/C14, C14—C19 and N3/C8—C12, respectively.

### S2. Experimental

2,6-Bis(benzimidazol-2-yl)pyridine (0.062 g, 0.20 mmol) and butyric acid (0.018 g, 0.20 mmol) were dissolved in 30 ml solution mixed with ethanol and water by 2:1(V/V), then heated to refluxed for 6 h and cooled to the room temperature. Single crystals suitable for X-ray measurements were obtained by recrystallization at room temperature.

### S3. Refinement

N- and O-bound H atoms were found in a difference Fourier map, and isotropically refined with the restraints (O—H = 0.82 (2), 0.85 (2) Å; N—H = 0.88 (2) Å). C-bound H atoms were fixed geometrically (C—H = 0.93–0.97 Å), and refined as riding, with $U_{iso}$(H) =1.2–1.5 $U_{eq}$(C).
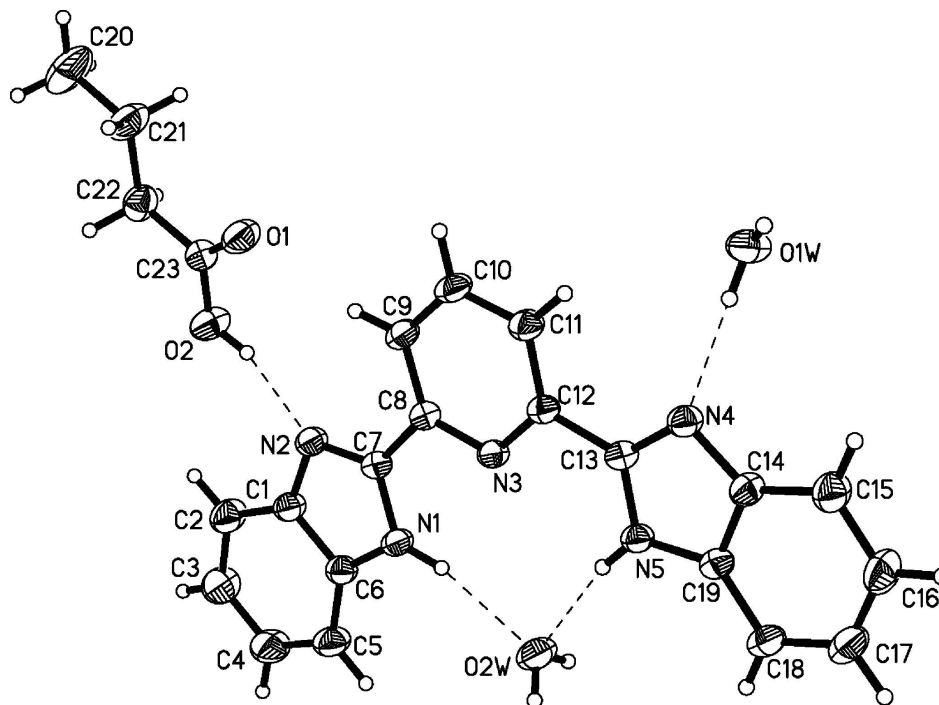
**Figure 1**

View of (I) showing 30% probability displacement ellipsoids and the atom-numbering scheme. Dashed lines indicate hydrogen bonds.

## 2,6-Bis(1*H*-benzimidazol-2-yl)pyridine butyric acid monosolvate dihydrate

*Crystal data*

| | |
|---|---|
| $C_{19}H_{13}N_5 \cdot C_4H_8O_2 \cdot 2H_2O$ | $Z = 2$ |
| $M_r = 435.48$ | $F(000) = 460$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.278$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $a = 9.3950$ (19) Å | Cell parameters from 25 reflections |
| $b = 9.5611$ (19) Å | $\theta = 4$–14° |
| $c = 13.805$ (3) Å | $\mu = 0.09$ mm$^{-1}$ |
| $\alpha = 103.27$ (2)° | $T = 295$ K |
| $\beta = 99.91$ (3)° | Block, colourless |
| $\gamma = 104.76$ (3)° | $0.20 \times 0.18 \times 0.15$ mm |
| $V = 1131.5$ (4) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Enraf–Nonius CAD-4 diffractometer | 3981 independent reflections |
| Radiation source: fine-focus sealed tube | 3195 reflections with $I > 2\sigma(I)$ |
| Graphite monochromator | $R_{int} = 0.019$ |
| $\omega$ scans | $\theta_{max} = 25.0°$, $\theta_{min} = 3.1°$ |
| Absorption correction: $\psi$ scan (North *et al.*, 1968) | $h = -11 \rightarrow 11$ |
| | $k = -11 \rightarrow 10$ |
| $T_{min} = 0.982$, $T_{max} = 0.987$ | $l = -16 \rightarrow 16$ |
| 8683 measured reflections | 3 standard reflections every 100 reflections |
| | intensity decay: none |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: inferred from |
| Least-squares matrix: full | neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.045$ | H atoms treated by a mixture of independent |
| $wR(F^2) = 0.144$ | and constrained refinement |
| $S = 1.10$ | $w = 1/[\sigma^2(F_o^2) + (0.0925P)^2 + 0.059P]$ |
| 3981 reflections | where $P = (F_o^2 + 2F_c^2)/3$ |
| 318 parameters | $(\Delta/\sigma)_{max} < 0.001$ |
| 6 restraints | $\Delta\rho_{max} = 0.22$ e Å$^{-3}$ |
| Primary atom site location: structure-invariant | $\Delta\rho_{min} = -0.25$ e Å$^{-3}$ |
| direct methods | Extinction correction: *SHELXL97* (Sheldrick, |
| Secondary atom site location: difference Fourier | 2008), Fc$^*$=kFc[1+0.001xFc$^2\lambda^3$/sin(2$\theta$)]$^{-1/4}$ |
| map | Extinction coefficient: 0.007 (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 (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| N1 | 0.12979 (14) | 0.51453 (15) | −0.18153 (10) | 0.0466 (3) |
| N2 | −0.05854 (14) | 0.30191 (14) | −0.26517 (10) | 0.0482 (3) |
| N3 | −0.04327 (13) | 0.62903 (14) | −0.06414 (9) | 0.0407 (3) |
| N4 | −0.10826 (15) | 0.91520 (15) | 0.12949 (10) | 0.0473 (3) |
| N5 | 0.10055 (14) | 0.91107 (14) | 0.07405 (10) | 0.0423 (3) |
| C1 | 0.06996 (18) | 0.29713 (18) | −0.30066 (12) | 0.0491 (4) |
| C2 | 0.0931 (2) | 0.1845 (2) | −0.37465 (15) | 0.0685 (5) |
| H2B | 0.0151 | 0.0957 | −0.4100 | 0.082* |
| C3 | 0.2354 (2) | 0.2091 (2) | −0.39367 (18) | 0.0796 (6) |
| H3B | 0.2528 | 0.1363 | −0.4437 | 0.095* |
| C4 | 0.3535 (2) | 0.3401 (3) | −0.33982 (19) | 0.0801 (7) |
| H4B | 0.4485 | 0.3517 | −0.3538 | 0.096* |
| C5 | 0.3335 (2) | 0.4530 (2) | −0.26625 (15) | 0.0652 (5) |
| H5B | 0.4126 | 0.5406 | −0.2303 | 0.078* |
| C6 | 0.18948 (18) | 0.42972 (18) | −0.24846 (12) | 0.0479 (4) |
| C7 | −0.01687 (16) | 0.43359 (16) | −0.19367 (11) | 0.0424 (4) |
| C8 | −0.11129 (17) | 0.49146 (16) | −0.13058 (11) | 0.0422 (4) |
| C9 | −0.25971 (18) | 0.40738 (18) | −0.13928 (13) | 0.0526 (4) |
| H9A | −0.3028 | 0.3116 | −0.1857 | 0.063* |
| C10 | −0.34091 (19) | 0.46967 (19) | −0.07748 (14) | 0.0575 (5) |
| H10A | −0.4405 | 0.4162 | −0.0816 | 0.069* |
| C11 | −0.27421 (18) | 0.61135 (18) | −0.00955 (13) | 0.0511 (4) |
| H11A | −0.3277 | 0.6555 | 0.0326 | 0.061* |

| | | | |
|---|---|---|---|
| C12 | −0.12534 (16) | 0.68670 (16) | −0.00542 (11) | 0.0408 (4) |
| C13 | −0.04638 (16) | 0.83768 (16) | 0.06640 (11) | 0.0409 (3) |
| C14 | 0.00703 (18) | 1.04751 (17) | 0.18249 (11) | 0.0464 (4) |
| C15 | 0.0069 (2) | 1.1702 (2) | 0.25982 (14) | 0.0621 (5) |
| H15A | −0.0794 | 1.1716 | 0.2839 | 0.075* |
| C16 | 0.1385 (2) | 1.2887 (2) | 0.29928 (15) | 0.0689 (5) |
| H16A | 0.1412 | 1.3716 | 0.3511 | 0.083* |
| C17 | 0.2687 (2) | 1.2877 (2) | 0.26338 (14) | 0.0633 (5) |
| H17A | 0.3556 | 1.3705 | 0.2918 | 0.076* |
| C18 | 0.27226 (19) | 1.16824 (18) | 0.18751 (13) | 0.0536 (4) |
| H18A | 0.3589 | 1.1682 | 0.1636 | 0.064* |
| C19 | 0.13926 (17) | 1.04728 (17) | 0.14830 (11) | 0.0438 (4) |
| O1 | −0.44196 (15) | 0.06635 (14) | −0.27630 (11) | 0.0743 (4) |
| O2 | −0.28356 (15) | 0.04486 (15) | −0.37563 (10) | 0.0657 (4) |
| C20 | −0.7674 (3) | −0.3547 (3) | −0.4697 (2) | 0.1110 (10) |
| H20A | −0.8595 | −0.3850 | −0.4481 | 0.167* |
| H20B | −0.7183 | −0.4317 | −0.4725 | 0.167* |
| H20C | −0.7909 | −0.3404 | −0.5366 | 0.167* |
| C21 | −0.6624 (2) | −0.2079 (2) | −0.39388 (18) | 0.0824 (7) |
| H21A | −0.7139 | −0.1315 | −0.3902 | 0.099* |
| H21B | −0.6411 | −0.2228 | −0.3263 | 0.099* |
| C22 | −0.5148 (2) | −0.1512 (2) | −0.42169 (14) | 0.0611 (5) |
| H22A | −0.4618 | −0.2261 | −0.4227 | 0.073* |
| H22B | −0.5366 | −0.1408 | −0.4905 | 0.073* |
| C23 | −0.41213 (18) | −0.00337 (18) | −0.35032 (13) | 0.0500 (4) |
| O1W | −0.40511 (15) | 0.92334 (16) | 0.11934 (11) | 0.0647 (4) |
| O2W | 0.32779 (16) | 0.81969 (16) | −0.04472 (12) | 0.0690 (4) |
| H2 | 0.1745 (19) | 0.6070 (13) | −0.1365 (11) | 0.057 (5)* |
| H1 | 0.156 (2) | 0.873 (2) | 0.0390 (14) | 0.056 (5)* |
| H2W1 | −0.3168 (14) | 0.920 (3) | 0.1236 (19) | 0.100 (8)* |
| H2W2 | 0.350 (3) | 0.885 (2) | −0.0747 (19) | 0.116 (10)* |
| H1W2 | 0.407 (2) | 0.839 (3) | −0.0027 (17) | 0.129 (12)* |
| H1W1 | −0.435 (3) | 0.928 (3) | 0.1717 (13) | 0.106 (9)* |
| H3 | −0.228 (2) | 0.1309 (16) | −0.3376 (16) | 0.096 (8)* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0350 (7) | 0.0439 (7) | 0.0510 (7) | 0.0049 (5) | 0.0084 (6) | 0.0045 (6) |
| N2 | 0.0399 (7) | 0.0449 (7) | 0.0514 (7) | 0.0072 (6) | 0.0116 (6) | 0.0037 (6) |
| N3 | 0.0329 (6) | 0.0430 (7) | 0.0420 (6) | 0.0082 (5) | 0.0075 (5) | 0.0094 (5) |
| N4 | 0.0398 (7) | 0.0497 (7) | 0.0466 (7) | 0.0087 (6) | 0.0125 (6) | 0.0070 (6) |
| N5 | 0.0336 (7) | 0.0437 (7) | 0.0456 (7) | 0.0076 (5) | 0.0109 (5) | 0.0089 (6) |
| C1 | 0.0409 (9) | 0.0491 (9) | 0.0513 (9) | 0.0088 (7) | 0.0121 (7) | 0.0078 (7) |
| C2 | 0.0587 (11) | 0.0579 (11) | 0.0726 (12) | 0.0080 (9) | 0.0231 (9) | −0.0063 (9) |
| C3 | 0.0684 (13) | 0.0722 (13) | 0.0897 (14) | 0.0193 (10) | 0.0379 (11) | −0.0039 (11) |
| C4 | 0.0530 (12) | 0.0789 (13) | 0.1034 (16) | 0.0164 (10) | 0.0377 (11) | 0.0069 (12) |
| C5 | 0.0414 (9) | 0.0656 (11) | 0.0781 (12) | 0.0069 (8) | 0.0204 (9) | 0.0071 (10) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C6 | 0.0401 (8) | 0.0490 (8) | 0.0510 (9) | 0.0116 (7) | 0.0120 (7) | 0.0094 (7) |
| C7 | 0.0351 (8) | 0.0431 (8) | 0.0445 (8) | 0.0082 (6) | 0.0065 (6) | 0.0106 (6) |
| C8 | 0.0364 (8) | 0.0405 (7) | 0.0456 (8) | 0.0082 (6) | 0.0073 (6) | 0.0104 (6) |
| C9 | 0.0399 (9) | 0.0417 (8) | 0.0638 (10) | 0.0011 (7) | 0.0123 (8) | 0.0048 (7) |
| C10 | 0.0368 (8) | 0.0498 (9) | 0.0759 (12) | −0.0013 (7) | 0.0209 (8) | 0.0102 (8) |
| C11 | 0.0403 (9) | 0.0485 (9) | 0.0615 (10) | 0.0072 (7) | 0.0215 (7) | 0.0104 (7) |
| C12 | 0.0337 (8) | 0.0426 (8) | 0.0441 (8) | 0.0075 (6) | 0.0092 (6) | 0.0138 (6) |
| C13 | 0.0340 (8) | 0.0431 (8) | 0.0415 (8) | 0.0064 (6) | 0.0079 (6) | 0.0114 (6) |
| C14 | 0.0438 (9) | 0.0447 (8) | 0.0432 (8) | 0.0073 (7) | 0.0080 (7) | 0.0076 (7) |
| C15 | 0.0603 (11) | 0.0585 (10) | 0.0579 (10) | 0.0134 (9) | 0.0179 (8) | 0.0011 (8) |
| C16 | 0.0769 (14) | 0.0522 (10) | 0.0569 (10) | 0.0108 (9) | 0.0056 (9) | −0.0058 (8) |
| C17 | 0.0569 (11) | 0.0499 (9) | 0.0618 (11) | −0.0010 (8) | −0.0030 (9) | 0.0077 (8) |
| C18 | 0.0413 (9) | 0.0513 (9) | 0.0569 (10) | 0.0023 (7) | 0.0027 (7) | 0.0138 (8) |
| C19 | 0.0387 (8) | 0.0453 (8) | 0.0416 (8) | 0.0073 (6) | 0.0041 (6) | 0.0118 (7) |
| O1 | 0.0559 (8) | 0.0539 (7) | 0.0927 (10) | 0.0011 (6) | 0.0302 (7) | −0.0089 (7) |
| O2 | 0.0543 (8) | 0.0667 (8) | 0.0587 (7) | 0.0007 (6) | 0.0171 (6) | 0.0021 (6) |
| C20 | 0.103 (2) | 0.0735 (15) | 0.1010 (18) | −0.0274 (14) | −0.0127 (15) | 0.0078 (13) |
| C21 | 0.0645 (13) | 0.0620 (12) | 0.0910 (15) | −0.0090 (10) | 0.0110 (11) | 0.0033 (11) |
| C22 | 0.0641 (12) | 0.0533 (9) | 0.0529 (9) | 0.0096 (9) | 0.0011 (8) | 0.0096 (8) |
| C23 | 0.0453 (9) | 0.0477 (9) | 0.0533 (9) | 0.0119 (7) | 0.0087 (7) | 0.0123 (7) |
| O1W | 0.0420 (7) | 0.0799 (9) | 0.0668 (8) | 0.0146 (6) | 0.0135 (6) | 0.0150 (7) |
| O2W | 0.0487 (8) | 0.0607 (8) | 0.0793 (9) | −0.0024 (6) | 0.0019 (7) | 0.0172 (7) |

## Geometric parameters (Å, º)

| | | | |
|---|---|---|---|
| N1—C7 | 1.358 (2) | C11—H11A | 0.9300 |
| N1—C6 | 1.375 (2) | C12—C13 | 1.470 (2) |
| N1—H2 | 0.90 (2) | C14—C15 | 1.393 (2) |
| N2—C7 | 1.3259 (19) | C14—C19 | 1.403 (2) |
| N2—C1 | 1.386 (2) | C15—C16 | 1.371 (3) |
| N3—C12 | 1.3350 (19) | C15—H15A | 0.9300 |
| N3—C8 | 1.3401 (19) | C16—C17 | 1.398 (3) |
| N4—C13 | 1.3203 (19) | C16—H16A | 0.9300 |
| N4—C14 | 1.383 (2) | C17—C18 | 1.373 (3) |
| N5—C13 | 1.3533 (19) | C17—H17A | 0.9300 |
| N5—C19 | 1.377 (2) | C18—C19 | 1.391 (2) |
| N5—H1 | 0.86 (2) | C18—H18A | 0.9300 |
| C1—C2 | 1.393 (2) | O1—C23 | 1.203 (2) |
| C1—C6 | 1.401 (2) | O2—C23 | 1.315 (2) |
| C2—C3 | 1.379 (3) | O2—H3 | 0.85 (2) |
| C2—H2B | 0.9300 | C20—C21 | 1.518 (3) |
| C3—C4 | 1.391 (3) | C20—H20A | 0.9600 |
| C3—H3B | 0.9300 | C20—H20B | 0.9600 |
| C4—C5 | 1.378 (3) | C20—H20C | 0.9600 |
| C4—H4B | 0.9300 | C21—C22 | 1.504 (3) |
| C5—C6 | 1.388 (2) | C21—H21A | 0.9700 |
| C5—H5B | 0.9300 | C21—H21B | 0.9700 |
| C7—C8 | 1.465 (2) | C22—C23 | 1.496 (2) |

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|---|---|---|---|
| C8—C9 | 1.390 (2) | C22—H22A | 0.9700 |
| C9—C10 | 1.374 (2) | C22—H22B | 0.9700 |
| C9—H9A | 0.9300 | O1W—H2W1 | 0.83 (2) |
| C10—C11 | 1.374 (2) | O1W—H1W1 | 0.82 (2) |
| C10—H10A | 0.9300 | O2W—H2W2 | 0.82 (2) |
| C11—C12 | 1.385 (2) | O2W—H1W2 | 0.81 (2) |
| | | | |
| C7—N1—C6 | 107.54 (13) | N4—C13—N5 | 113.13 (13) |
| C7—N1—H2 | 123.1 (12) | N4—C13—C12 | 124.76 (13) |
| C6—N1—H2 | 129.3 (12) | N5—C13—C12 | 122.12 (13) |
| C7—N2—C1 | 104.86 (13) | N4—C14—C15 | 129.84 (16) |
| C12—N3—C8 | 117.15 (13) | N4—C14—C19 | 110.09 (13) |
| C13—N4—C14 | 104.64 (13) | C15—C14—C19 | 120.07 (16) |
| C13—N5—C19 | 107.30 (13) | C16—C15—C14 | 117.73 (18) |
| C13—N5—H1 | 123.4 (13) | C16—C15—H15A | 121.1 |
| C19—N5—H1 | 129.3 (13) | C14—C15—H15A | 121.1 |
| N2—C1—C2 | 130.11 (16) | C15—C16—C17 | 121.52 (17) |
| N2—C1—C6 | 109.96 (14) | C15—C16—H16A | 119.2 |
| C2—C1—C6 | 119.92 (16) | C17—C16—H16A | 119.2 |
| C3—C2—C1 | 117.73 (18) | C18—C17—C16 | 122.03 (17) |
| C3—C2—H2B | 121.1 | C18—C17—H17A | 119.0 |
| C1—C2—H2B | 121.1 | C16—C17—H17A | 119.0 |
| C2—C3—C4 | 121.61 (18) | C17—C18—C19 | 116.43 (17) |
| C2—C3—H3B | 119.2 | C17—C18—H18A | 121.8 |
| C4—C3—H3B | 119.2 | C19—C18—H18A | 121.8 |
| C5—C4—C3 | 121.71 (18) | N5—C19—C18 | 132.95 (15) |
| C5—C4—H4B | 119.1 | N5—C19—C14 | 104.84 (14) |
| C3—C4—H4B | 119.1 | C18—C19—C14 | 122.20 (15) |
| C4—C5—C6 | 116.72 (18) | C23—O2—H3 | 113.9 (17) |
| C4—C5—H5B | 121.6 | C21—C20—H20A | 109.5 |
| C6—C5—H5B | 121.6 | C21—C20—H20B | 109.5 |
| N1—C6—C5 | 132.63 (16) | H20A—C20—H20B | 109.5 |
| N1—C6—C1 | 105.07 (14) | C21—C20—H20C | 109.5 |
| C5—C6—C1 | 122.27 (16) | H20A—C20—H20C | 109.5 |
| N2—C7—N1 | 112.57 (13) | H20B—C20—H20C | 109.5 |
| N2—C7—C8 | 126.42 (14) | C22—C21—C20 | 113.3 (2) |
| N1—C7—C8 | 121.00 (13) | C22—C21—H21A | 108.9 |
| N3—C8—C9 | 123.26 (14) | C20—C21—H21A | 108.9 |
| N3—C8—C7 | 115.06 (13) | C22—C21—H21B | 108.9 |
| C9—C8—C7 | 121.67 (14) | C20—C21—H21B | 108.9 |
| C10—C9—C8 | 118.15 (15) | H21A—C21—H21B | 107.7 |
| C10—C9—H9A | 120.9 | C23—C22—C21 | 114.24 (16) |
| C8—C9—H9A | 120.9 | C23—C22—H22A | 108.7 |
| C9—C10—C11 | 119.67 (15) | C21—C22—H22A | 108.7 |
| C9—C10—H10A | 120.2 | C23—C22—H22B | 108.7 |
| C11—C10—H10A | 120.2 | C21—C22—H22B | 108.7 |
| C10—C11—C12 | 118.33 (15) | H22A—C22—H22B | 107.6 |
| C10—C11—H11A | 120.8 | O1—C23—O2 | 122.25 (16) |

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|---|---|---|---|
| C12—C11—H11A | 120.8 | O1—C23—C22 | 124.19 (16) |
| N3—C12—C11 | 123.43 (14) | O2—C23—C22 | 113.56 (15) |
| N3—C12—C13 | 115.37 (13) | H2W1—O1W—H1W1 | 117 (3) |
| C11—C12—C13 | 121.19 (14) | H2W2—O2W—H1W2 | 102 (3) |
| | | | |
| C7—N2—C1—C2 | 178.8 (2) | C8—N3—C12—C13 | 179.95 (13) |
| C7—N2—C1—C6 | −0.07 (19) | C10—C11—C12—N3 | 0.1 (3) |
| N2—C1—C2—C3 | −178.9 (2) | C10—C11—C12—C13 | −179.18 (15) |
| C6—C1—C2—C3 | −0.1 (3) | C14—N4—C13—N5 | −0.62 (17) |
| C1—C2—C3—C4 | 1.3 (4) | C14—N4—C13—C12 | 179.13 (14) |
| C2—C3—C4—C5 | −1.3 (4) | C19—N5—C13—N4 | 0.37 (18) |
| C3—C4—C5—C6 | 0.0 (4) | C19—N5—C13—C12 | −179.38 (13) |
| C7—N1—C6—C5 | −177.18 (19) | N3—C12—C13—N4 | 178.66 (14) |
| C7—N1—C6—C1 | 0.76 (18) | C11—C12—C13—N4 | −2.0 (2) |
| C4—C5—C6—N1 | 178.9 (2) | N3—C12—C13—N5 | −1.6 (2) |
| C4—C5—C6—C1 | 1.2 (3) | C11—C12—C13—N5 | 177.75 (14) |
| N2—C1—C6—N1 | −0.43 (19) | C13—N4—C14—C15 | −178.98 (18) |
| C2—C1—C6—N1 | −179.43 (17) | C13—N4—C14—C19 | 0.63 (17) |
| N2—C1—C6—C5 | 177.78 (16) | N4—C14—C15—C16 | −179.71 (17) |
| C2—C1—C6—C5 | −1.2 (3) | C19—C14—C15—C16 | 0.7 (3) |
| C1—N2—C7—N1 | 0.58 (18) | C14—C15—C16—C17 | 0.2 (3) |
| C1—N2—C7—C8 | −178.33 (15) | C15—C16—C17—C18 | −0.4 (3) |
| C6—N1—C7—N2 | −0.87 (19) | C16—C17—C18—C19 | −0.3 (3) |
| C6—N1—C7—C8 | 178.11 (14) | C13—N5—C19—C18 | −179.13 (17) |
| C12—N3—C8—C9 | −1.1 (2) | C13—N5—C19—C14 | 0.05 (17) |
| C12—N3—C8—C7 | 179.62 (13) | C17—C18—C19—N5 | −179.70 (17) |
| N2—C7—C8—N3 | −179.30 (14) | C17—C18—C19—C14 | 1.2 (2) |
| N1—C7—C8—N3 | 1.9 (2) | N4—C14—C19—N5 | −0.42 (17) |
| N2—C7—C8—C9 | 1.4 (3) | C15—C14—C19—N5 | 179.24 (15) |
| N1—C7—C8—C9 | −177.42 (15) | N4—C14—C19—C18 | 178.87 (14) |
| N3—C8—C9—C10 | 0.9 (3) | C15—C14—C19—C18 | −1.5 (3) |
| C7—C8—C9—C10 | −179.91 (15) | C20—C21—C22—C23 | 177.6 (2) |
| C8—C9—C10—C11 | −0.1 (3) | C21—C22—C23—O1 | 0.2 (3) |
| C9—C10—C11—C12 | −0.4 (3) | C21—C22—C23—O2 | −179.94 (17) |
| C8—N3—C12—C11 | 0.6 (2) | | |

*Hydrogen-bond geometry (Å, °)*

| D—H···A | D—H | H···A | D···A | D—H···A |
|---|---|---|---|---|
| N1—H2···O2W | 0.90 (2) | 2.12 (1) | 3.006 (2) | 166 (2) |
| N5—H1···O2W | 0.86 (2) | 2.234 (19) | 3.083 (2) | 169.1 (17) |
| O1W—H2W1···N4 | 0.83 (2) | 1.96 (1) | 2.7901 (19) | 178 (3) |
| O2W—H2W2···O1W[i] | 0.82 (2) | 2.04 (1) | 2.852 (2) | 168 (3) |
| O2W—H1W2···O1W[ii] | 0.81 (2) | 2.06 (1) | 2.856 (2) | 168 (3) |
| O1W—H1W1···O1[iii] | 0.82 (2) | 2.00 (1) | 2.795 (2) | 166 (3) |
| O2—H3···N2 | 0.85 (2) | 1.89 (1) | 2.712 (2) | 164 (2) |

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