

# Melaminium nitrate–melamine–water (1/1/1)

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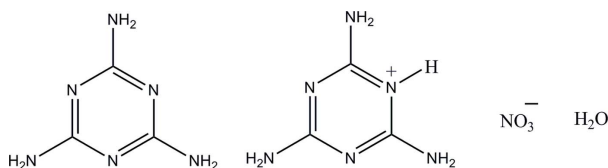
Received 22 October 2010; accepted 27 October 2010

Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{N}-\text{C}) = 0.003$  Å; disorder in main residue;  $R$  factor = 0.047;  $wR$  factor = 0.123; data-to-parameter ratio = 9.5.

In the crystal structure of the title salt,  $\text{C}_3\text{H}_7\text{N}_6^{+}\cdot\text{NO}_3^{-}\cdot\text{C}_3\text{H}_6\text{N}_6\cdot\text{H}_2\text{O}$ , the asymmetric unit consists of two neutral melamine (1,3,5-triazine-2,4,6-triamine) molecules, two melaminium cations, two nitrate anions and two solvent water molecules. One of the nitrate anions is disordered over two sets of positions, with a refined occupancy ratio of 0.909 (3):0.091 (3). The cations and neutral molecules are approximately planar, with maximum deviations of 0.018 (2), 0.024 (2), 0.019 (2) and 0.007 (2) Å for each, respectively. In the crystal structure, melaminium cations and neutral melamine molecules self-assemble *via*  $\text{N}-\text{H}\cdots\text{N}$  hydrogen bonds to form a supramolecular hexagonal-shaped motif. In addition, the nitrate anions and water molecules are connected by  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds to form a three-dimensional network.

## Related literature

For applications of melamine, see: Rima *et al.* (2008); Cook *et al.* (2005); Ramos Silva *et al.* (2008). For related structures, see: Debrus *et al.* (2007); Zhao & Shi (2010); Marchewka & Pietraszko (2003); Marchewka (2002). For applications of hydrogen bonding, see: Aghabozorg *et al.* (2008).



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<sup>§</sup> Thomson Reuters ResearcherID: A-3561-2009.

## Experimental

### Crystal data

$\text{C}_3\text{H}_7\text{N}_6^{+}\cdot\text{NO}_3^{-}\cdot\text{C}_3\text{H}_6\text{N}_6\cdot\text{H}_2\text{O}$   
 $M_r = 333.31$   
Triclinic,  $P\bar{1}$   
 $a = 7.7759$  (1) Å  
 $b = 9.0035$  (1) Å  
 $c = 19.4573$  (3) Å  
 $\alpha = 96.182$  (1)°  
 $\beta = 90.854$  (1)°

$\gamma = 99.828$  (1)°  
 $V = 1333.64$  (3) Å<sup>3</sup>  
 $Z = 4$   
Mo  $K\alpha$  radiation  
 $\mu = 0.14$  mm<sup>-1</sup>  
 $T = 296$  K  
 $0.21 \times 0.14 \times 0.09$  mm

### Data collection

Bruker SMART APEXII CCD  
area-detector diffractometer  
Absorption correction: multi-scan  
(*SADABS*; Bruker, 2009)  
 $T_{\min} = 0.972$ ,  $T_{\max} = 0.987$

22196 measured reflections  
5160 independent reflections  
3689 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.042$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.047$   
 $wR(F^2) = 0.123$   
 $S = 1.03$   
5160 reflections

543 parameters  
All H-atom parameters refined  
 $\Delta\rho_{\text{max}} = 0.21$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.45$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$                                  | $D-H$    | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--|----------|-------------|-------------|---------------|
| $\text{N4A}-\text{H1A}\cdots\text{O2W}^i$      | 0.92 (2) | 2.05 (2)    | 2.965 (2)   | 174.2 (17)    |
| $\text{N4B}-\text{H1B}\cdots\text{N1D}$        | 0.85 (2) | 2.18 (2)    | 3.025 (2)   | 174.1 (18)    |
| $\text{N4D}-\text{H1D}\cdots\text{O3AA}$       | 0.88 (2) | 2.27 (2)    | 3.077 (2)   | 153 (2)       |
| $\text{N1A}-\text{H1N}\cdots\text{O1W}^{ii}$   | 0.91 (2) | 1.89 (2)    | 2.771 (2)   | 165 (2)       |
| $\text{N4A}-\text{H2A}\cdots\text{O1B}^i$      | 0.96 (3) | 2.03 (3)    | 2.838 (2)   | 141 (2)       |
| $\text{N4B}-\text{H2B}\cdots\text{O3AA}$       | 0.93 (3) | 2.05 (2)    | 2.810 (2)   | 138.8 (18)    |
| $\text{N4C}-\text{H2C}\cdots\text{N2D}^{iii}$  | 0.88 (3) | 2.07 (3)    | 2.945 (2)   | 176 (2)       |
| $\text{N4D}-\text{H2D}\cdots\text{N2C}^{iii}$  | 0.84 (2) | 2.23 (2)    | 3.069 (2)   | 172 (2)       |
| $\text{N1C}-\text{H2N}\cdots\text{O3B}$        | 0.93 (2) | 1.91 (2)    | 2.836 (2)   | 177 (2)       |
| $\text{O2W}-\text{H1W2}\cdots\text{N1D}$       | 0.86 (3) | 2.04 (3)    | 2.899 (2)   | 174 (3)       |
| $\text{N5A}-\text{H3A}\cdots\text{N2B}$        | 0.88 (3) | 2.20 (3)    | 3.062 (2)   | 165 (2)       |
| $\text{N5B}-\text{H3B}\cdots\text{N2A}$        | 0.82 (3) | 2.30 (3)    | 3.119 (2)   | 175 (2)       |
| $\text{N5C}-\text{H3C}\cdots\text{O2AA}^{iii}$ | 0.89 (2) | 2.06 (2)    | 2.799 (2)   | 140 (2)       |
| $\text{N5D}-\text{H3D}\cdots\text{O3AA}^{iv}$  | 0.86 (2) | 2.53 (2)    | 3.232 (3)   | 140 (2)       |
| $\text{N5A}-\text{H4A}\cdots\text{O1AA}$       | 0.86 (2) | 2.11 (2)    | 2.963 (2)   | 171 (2)       |
| $\text{N5B}-\text{H4B}\cdots\text{O2B}^i$      | 0.86 (2) | 2.20 (2)    | 3.045 (2)   | 167 (2)       |
| $\text{N5C}-\text{H4C}\cdots\text{N3D}^v$      | 0.89 (3) | 2.12 (3)    | 2.996 (2)   | 173 (2)       |
| $\text{N5D}-\text{H4D}\cdots\text{N3C}^v$      | 0.81 (3) | 2.30 (3)    | 3.105 (3)   | 173 (2)       |
| $\text{N6A}-\text{H5A}\cdots\text{N3B}^{vi}$   | 0.80 (2) | 2.13 (2)    | 2.926 (2)   | 175.9 (19)    |
| $\text{N6B}-\text{H5B}\cdots\text{N3A}^{vii}$  | 0.84 (2) | 2.18 (2)    | 3.020 (2)   | 177 (2)       |
| $\text{N6C}-\text{H5C}\cdots\text{O2W}$        | 0.93 (3) | 2.00 (3)    | 2.849 (2)   | 151 (2)       |
| $\text{N6D}-\text{H5D}\cdots\text{O2AA}^{vii}$ | 0.93 (3) | 2.22 (3)    | 3.126 (2)   | 167 (2)       |
| $\text{N6A}-\text{H6A}\cdots\text{O1W}^{ii}$   | 0.90 (2) | 2.55 (2)    | 3.262 (2)   | 137.2 (15)    |
| $\text{N6A}-\text{H6A}\cdots\text{O2B}^{viii}$ | 0.90 (2) | 2.14 (2)    | 2.841 (2)   | 135.2 (17)    |
| $\text{N6B}-\text{H6B}\cdots\text{O1AA}^{vii}$ | 0.89 (2) | 2.17 (3)    | 2.806 (2)   | 128 (2)       |
| $\text{N6C}-\text{H6C}\cdots\text{O1B}$        | 0.90 (2) | 1.97 (2)    | 2.868 (2)   | 174.6 (18)    |
| $\text{N6D}-\text{H6D}\cdots\text{N1B}$        | 0.91 (2) | 2.18 (2)    | 3.088 (2)   | 172 (2)       |

Symmetry codes: (i)  $-x+1, -y+1, -z$ ; (ii)  $x+1, y, z-1$ ; (iii)  $-x+1, -y+2, -z+1$ ; (iv)  $-x+1, -y+1, -z+1$ ; (v)  $-x, -y+1, -z+1$ ; (vi)  $x+1, y+1, z$ ; (vii)  $x-1, y-1, z$ ; (viii)  $-x+2, -y+2, -z$ .

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* and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

The authors thank the Malaysian Government for a USM-RU-PRGS grant (No. 1001/PKIMIA/842020) and an RU grant

(No. 10001/PKIMIA/814019) which partly supported this work. HKF and MH thank the Malaysian Government and Universiti Sains Malaysia for a Research University grant (No. 1001/PFIZIK/811160). MH also thanks Universiti Sains Malaysia for a postdoctoral research fellowship.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5157).

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**supplementary materials**

*Acta Cryst.* (2010). E66, o3033–o3034 [ doi:10.1107/S1600536810043941 ]

## Melaminium nitrate-melamine-water (1/1/1)

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### Comment

1,3,5-triazine-2,4,6-triamine is an organic base also known as melamine. Melamine is very widely used in several industries, such as the production of melamine foam in polymeric cleaning (Rima *et al.*, 2008) and also as a chemical intermediate in amino resin and plastics manufacturing (Cook *et al.*, 2005). Melamine can be a proton acceptor and will form 2,4,6-triamino-1,3,5-triazine-1-ium (Ramos Silva *et al.*, 2008). Recently many melaminium complexes in crystalline form has been reported, such as melaminium-bis(trichloroacetate) monohydrate (Debrus *et al.*, 2007), melaminium iodide monohydrate (Zhao & Shi, 2010) and melaminium citrate (Marchewka & Pietraszko, 2003). Melaminium salt crystals have shown interesting properties like nonlinear optical behaviour (Marchewka, 2002). In the formation of melaminium salt crystals, molecules are bound to each other via hydrogen bonds. Hydrogen bonding plays an important role in the catalytic, biochemical activities and also in supramolecular chemistry and crystal engineering (Aghabozorg *et al.*, 2008). Here, we report the crystal structure of a melaminium salt. This crystal was obtained as a by-product during our attempt to form crown complexes with melamine.

The asymmetric unit of the title compound consists of two crystallographically independent protonated melaminium cations (A & C), two nitrate anions (A & B), two neutral melamine molecules (B & D) and two water molecules (Fig. 1). One of the nitrate anion is disordered over two sets of position, with refined occupancy ratios of 0.909 (3):0.091 (3). The protonated and neutral melamine molecules are essentially planar, with a maximum deviation of 0.018 (2) Å for atom C2A (molecule A), 0.024 (2) Å for atom C2C (molecule C), 0.019 (2) Å for atom C2B (molecule B) and 0.007 (2) Å for atom C2D (molecule D).

In the crystal structure (Fig.2), the protonated melaminium cations and the neutral melamine molecules self-assemble via N—H $\cdots$ N hydrogen bonds to form a supramolecular hexagonal motif. Furthermore, the nitrate anions and water molecules are connected by N—H $\cdots$ O (Table 1) hydrogen bonds to form a three-dimensional network.

### Experimental

0.36 g (2.856 mmol) of melamine, 0.50 g (2.856 mmol) of 1,4-bis(chloromethyl)benzene and 1.0 ml triethylamine were added into 40 mL acetonitrile and refluxed for 72 hours at 348 K. The white precipitate was collected by simple filtration and dried at 373 K for 24 hours. About 0.5 g of the white precipitate was dissolved in 10 mL distilled water followed by 0.5 g (1.718 mmol) of cobalt(II) nitrate. The pH was adjusted to 7.0 by a few drops of 1.0 M sodium hydroxide. The mixture was stirred for 2 h and then filtered off. The resulting mixture was kept at room temperature for recrystallization. Recrystallization was carried out twice by using distilled water to get the pure crystal.

### Refinement

All the H atoms were located in a difference Fourier map and allowed to refine freely [N—H = 0.80 (2)–0.96 (2) Å and O—H = 0.85 (4)–0.96 (3) Å]. One of the nitrate anion is disordered over two sets of positions, with refined occupancy ratios of 0.909 (3):0.091 (3).

## Figures

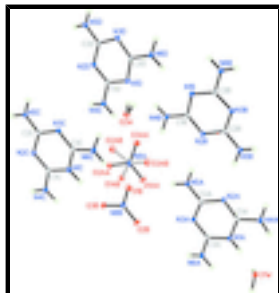


Fig. 1. The asymmetric unit of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme. Open bonds represents disorder components.

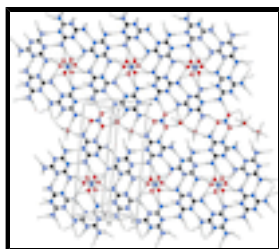


Fig. 2. The crystal packing of the title compound, showing the hydrogen-bonded (dashed lines) network.

## 2,4,6-triamino-1,3,5-triazin-1-ium nitrate-1,3,5-triazine-2,4,6-triamine- water (1/1/1)

### Crystal data

$C_3H_7N_6^+ \cdot NO_3^- \cdot C_3H_6N_6 \cdot H_2O$

$M_r = 333.31$

Triclinic,  $P\bar{1}$

Hall symbol: -P 1

$a = 7.7759$  (1) Å

$b = 9.0035$  (1) Å

$c = 19.4573$  (3) Å

$\alpha = 96.182$  (1)°

$\beta = 90.854$  (1)°

$\gamma = 99.828$  (1)°

$V = 1333.64$  (3) Å<sup>3</sup>

$Z = 4$

$F(000) = 696$

$D_x = 1.660$  Mg m<sup>-3</sup>

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

Cell parameters from 3833 reflections

$\theta = 2.3$ – $29.8$ °

$\mu = 0.14$  mm<sup>-1</sup>

$T = 296$  K

Block, purple

$0.21 \times 0.14 \times 0.09$  mm

### Data collection

Bruker SMART APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube graphite

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan (SADABS; Bruker, 2009)

$T_{\min} = 0.972$ ,  $T_{\max} = 0.987$

22196 measured reflections

5160 independent reflections

3689 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.042$

$\theta_{\max} = 26.0$ °,  $\theta_{\min} = 2.1$ °

$h = -8 \rightarrow 9$

$k = -11 \rightarrow 11$

$l = -23 \rightarrow 23$

*Refinement*

|                                 |  |
|---------------------------------|--|
| Refinement on $F^2$             | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full      | Secondary atom site location: difference Fourier map           |
| $R[F^2 > 2\sigma(F^2)] = 0.047$ | Hydrogen site location: inferred from neighbouring sites       |
| $wR(F^2) = 0.123$               | All H-atom parameters refined                                  |
| $S = 1.03$                      | $w = 1/[\sigma^2(F_o^2) + (0.0625P)^2 + 0.2158P]$              |
| 5160 reflections                | where $P = (F_o^2 + 2F_c^2)/3$                                 |
| 543 parameters                  | $(\Delta/\sigma)_{\max} < 0.001$                               |
| 0 restraints                    | $\Delta\rho_{\max} = 0.21 \text{ e } \text{\AA}^{-3}$          |
|                                 | $\Delta\rho_{\min} = -0.45 \text{ e } \text{\AA}^{-3}$         |

*Special details*

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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  $F^2 > 2\sigma(F^2)$  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 ( $\text{\AA}^2$ )*

|     | <i>x</i>   | <i>y</i>     | <i>z</i>      | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|-----|------------|--------------|---------------|----------------------------------|-----------|
| N1A | 0.9356 (2) | 0.62933 (18) | -0.08016 (9)  | 0.0142 (4)                       |           |
| N2A | 0.7288 (2) | 0.48794 (18) | -0.01488 (8)  | 0.0149 (4)                       |           |
| N3A | 0.9137 (2) | 0.71794 (18) | 0.03718 (8)   | 0.0139 (4)                       |           |
| N4A | 0.7628 (2) | 0.4097 (2)   | -0.12989 (9)  | 0.0176 (4)                       |           |
| N5A | 0.7043 (2) | 0.5825 (2)   | 0.09767 (9)   | 0.0172 (4)                       |           |
| N6A | 1.1157 (2) | 0.8451 (2)   | -0.03080 (10) | 0.0165 (4)                       |           |
| C1A | 0.8069 (2) | 0.5077 (2)   | -0.07424 (10) | 0.0139 (4)                       |           |
| C2A | 0.7836 (2) | 0.5972 (2)   | 0.03848 (10)  | 0.0145 (4)                       |           |
| C3A | 0.9888 (2) | 0.7321 (2)   | -0.02329 (10) | 0.0136 (4)                       |           |
| N1B | 0.2450 (2) | 0.17275 (18) | 0.20073 (8)   | 0.0157 (4)                       |           |
| N2B | 0.4686 (2) | 0.29201 (18) | 0.13081 (8)   | 0.0149 (4)                       |           |
| N3B | 0.2675 (2) | 0.07228 (18) | 0.08247 (8)   | 0.0152 (4)                       |           |
| N4B | 0.4452 (2) | 0.3774 (2)   | 0.24538 (9)   | 0.0183 (4)                       |           |
| N5B | 0.4750 (2) | 0.1976 (2)   | 0.01677 (9)   | 0.0160 (4)                       |           |
| N6B | 0.0627 (2) | -0.0409 (2)  | 0.15269 (10)  | 0.0196 (4)                       |           |
| C1B | 0.3839 (2) | 0.2789 (2)   | 0.19097 (10)  | 0.0148 (4)                       |           |
| C2B | 0.4012 (2) | 0.1864 (2)   | 0.07766 (10)  | 0.0129 (4)                       |           |
| C3B | 0.1951 (2) | 0.0700 (2)   | 0.14507 (10)  | 0.0148 (4)                       |           |

## supplementary materials

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|      |              |              |              |             |           |
|------|--------------|--------------|--------------|-------------|-----------|
| N1C  | 0.4424 (2)   | 1.05878 (19) | 0.34890 (9)  | 0.0170 (4)  |           |
| N2C  | 0.4197 (2)   | 1.15377 (18) | 0.46499 (8)  | 0.0143 (4)  |           |
| N3C  | 0.2251 (2)   | 0.92779 (18) | 0.41389 (8)  | 0.0158 (4)  |           |
| N4C  | 0.6243 (2)   | 1.2761 (2)   | 0.39577 (10) | 0.0182 (4)  |           |
| N5C  | 0.2192 (2)   | 1.0143 (2)   | 0.52802 (9)  | 0.0155 (4)  |           |
| N6C  | 0.2541 (3)   | 0.8500 (2)   | 0.29859 (9)  | 0.0221 (4)  |           |
| C1C  | 0.4950 (2)   | 1.1634 (2)   | 0.40435 (10) | 0.0149 (4)  |           |
| C2C  | 0.2901 (2)   | 1.0327 (2)   | 0.46781 (10) | 0.0140 (4)  |           |
| C3C  | 0.3043 (3)   | 0.9436 (2)   | 0.35478 (10) | 0.0162 (4)  |           |
| N1D  | 0.2208 (2)   | 0.40060 (18) | 0.37041 (8)  | 0.0155 (4)  |           |
| N2D  | 0.2450 (2)   | 0.48189 (18) | 0.49238 (8)  | 0.0149 (4)  |           |
| N3D  | 0.0365 (2)   | 0.26250 (18) | 0.44780 (8)  | 0.0145 (4)  |           |
| N4D  | 0.4156 (2)   | 0.6117 (2)   | 0.41573 (10) | 0.0180 (4)  |           |
| N5D  | 0.0681 (3)   | 0.3419 (2)   | 0.56411 (9)  | 0.0174 (4)  |           |
| N6D  | 0.0204 (2)   | 0.1911 (2)   | 0.32993 (9)  | 0.0180 (4)  |           |
| C1D  | 0.2918 (2)   | 0.4954 (2)   | 0.42667 (10) | 0.0139 (4)  |           |
| C2D  | 0.1182 (2)   | 0.3630 (2)   | 0.49989 (10) | 0.0142 (4)  |           |
| C3D  | 0.0951 (3)   | 0.2865 (2)   | 0.38480 (10) | 0.0137 (4)  |           |
| N8A  | 0.7624 (2)   | 0.76433 (19) | 0.28672 (9)  | 0.0178 (4)* |           |
| O1AA | 0.8234 (2)   | 0.77664 (18) | 0.22811 (8)  | 0.0211 (4)  | 0.909 (3) |
| O2AA | 0.7867 (2)   | 0.87285 (18) | 0.33240 (8)  | 0.0293 (5)  | 0.909 (3) |
| O3AA | 0.6809 (2)   | 0.63698 (18) | 0.30001 (8)  | 0.0305 (5)  | 0.909 (3) |
| O1AB | 0.887 (2)    | 0.8747 (18)  | 0.2933 (8)   | 0.021 (4)*  | 0.091 (3) |
| O2AB | 0.649 (2)    | 0.7781 (19)  | 0.3371 (8)   | 0.026 (5)*  | 0.091 (3) |
| O3AB | 0.721 (2)    | 0.673 (2)    | 0.2387 (9)   | 0.029 (5)*  | 0.091 (3) |
| N8B  | 0.5925 (2)   | 0.96755 (18) | 0.17530 (8)  | 0.0163 (4)  |           |
| O1B  | 0.46913 (19) | 0.86023 (16) | 0.18015 (7)  | 0.0240 (4)  |           |
| O2B  | 0.66849 (18) | 0.98031 (16) | 0.11995 (7)  | 0.0200 (3)  |           |
| O3B  | 0.64020 (18) | 1.06308 (15) | 0.22762 (7)  | 0.0191 (3)  |           |
| O1W  | 0.1736 (2)   | 0.67667 (17) | 0.81644 (8)  | 0.0188 (3)  |           |
| O2W  | 0.0718 (2)   | 0.54790 (17) | 0.26433 (8)  | 0.0217 (4)  |           |
| H1N  | 0.997 (3)    | 0.639 (3)    | -0.1192 (12) | 0.028 (6)*  |           |
| H2N  | 0.504 (3)    | 1.061 (3)    | 0.3083 (12)  | 0.032 (7)*  |           |
| H1A  | 0.816 (3)    | 0.430 (2)    | -0.1706 (12) | 0.023 (6)*  |           |
| H2A  | 0.668 (3)    | 0.328 (3)    | -0.1251 (13) | 0.044 (8)*  |           |
| H3A  | 0.622 (3)    | 0.504 (3)    | 0.1019 (11)  | 0.022 (6)*  |           |
| H4A  | 0.739 (3)    | 0.648 (3)    | 0.1329 (11)  | 0.019 (6)*  |           |
| H5A  | 1.154 (3)    | 0.905 (3)    | 0.0016 (12)  | 0.020 (6)*  |           |
| H6A  | 1.157 (3)    | 0.853 (2)    | -0.0732 (12) | 0.020 (6)*  |           |
| H1B  | 0.382 (3)    | 0.377 (2)    | 0.2808 (11)  | 0.018 (6)*  |           |
| H2B  | 0.531 (3)    | 0.460 (3)    | 0.2400 (11)  | 0.030 (7)*  |           |
| H3B  | 0.543 (3)    | 0.276 (3)    | 0.0113 (11)  | 0.022 (6)*  |           |
| H4B  | 0.426 (3)    | 0.137 (3)    | -0.0177 (12) | 0.025 (6)*  |           |
| H5B  | 0.021 (3)    | -0.106 (3)   | 0.1195 (12)  | 0.023 (6)*  |           |
| H6B  | 0.014 (3)    | -0.044 (3)   | 0.1938 (12)  | 0.027 (7)*  |           |
| H1C  | 0.676 (3)    | 1.277 (3)    | 0.3554 (13)  | 0.030 (7)*  |           |
| H2C  | 0.668 (3)    | 1.346 (3)    | 0.4295 (13)  | 0.033 (7)*  |           |
| H3C  | 0.259 (3)    | 1.082 (3)    | 0.5641 (12)  | 0.025 (6)*  |           |
| H4C  | 0.138 (3)    | 0.934 (3)    | 0.5318 (12)  | 0.032 (7)*  |           |

|      |            |           |             |             |
|------|------------|-----------|-------------|-------------|
| H5C  | 0.172 (4)  | 0.762 (3) | 0.3000 (13) | 0.047 (8)*  |
| H6C  | 0.318 (3)  | 0.858 (2) | 0.2606 (11) | 0.016 (6)*  |
| H1D  | 0.458 (3)  | 0.616 (2) | 0.3741 (11) | 0.014 (6)*  |
| H2D  | 0.459 (3)  | 0.669 (3) | 0.4510 (12) | 0.024 (6)*  |
| H3D  | 0.129 (3)  | 0.396 (3) | 0.5980 (12) | 0.030 (7)*  |
| H4D  | -0.005 (3) | 0.268 (3) | 0.5669 (11) | 0.026 (7)*  |
| H5D  | -0.051 (3) | 0.102 (3) | 0.3382 (12) | 0.034 (7)*  |
| H6D  | 0.082 (3)  | 0.194 (3) | 0.2905 (12) | 0.027 (6)*  |
| H1W1 | 0.203 (4)  | 0.765 (3) | 0.7943 (14) | 0.054 (9)*  |
| H2W1 | 0.270 (4)  | 0.673 (4) | 0.8371 (17) | 0.084 (12)* |
| H1W2 | 0.116 (4)  | 0.499 (3) | 0.2936 (15) | 0.055 (9)*  |
| H2W2 | -0.034 (4) | 0.480 (3) | 0.2477 (13) | 0.042 (7)*  |

*Atomic displacement parameters ( $\text{\AA}^2$ )*

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$    | $U^{13}$    | $U^{23}$    |
|-----|-------------|-------------|-------------|-------------|-------------|-------------|
| N1A | 0.0151 (9)  | 0.0154 (9)  | 0.0106 (9)  | -0.0008 (7) | 0.0026 (7)  | 0.0005 (7)  |
| N2A | 0.0170 (9)  | 0.0132 (9)  | 0.0134 (9)  | 0.0003 (7)  | 0.0005 (7)  | 0.0004 (7)  |
| N3A | 0.0148 (9)  | 0.0140 (9)  | 0.0118 (9)  | -0.0006 (7) | 0.0013 (7)  | 0.0015 (7)  |
| N4A | 0.0202 (10) | 0.0178 (10) | 0.0128 (9)  | -0.0014 (8) | 0.0022 (8)  | -0.0009 (7) |
| N5A | 0.0201 (10) | 0.0155 (10) | 0.0130 (9)  | -0.0047 (8) | 0.0032 (8)  | -0.0005 (8) |
| N6A | 0.0190 (10) | 0.0168 (9)  | 0.0104 (10) | -0.0043 (7) | 0.0027 (8)  | -0.0021 (8) |
| C1A | 0.0125 (10) | 0.0135 (10) | 0.0155 (11) | 0.0017 (8)  | -0.0004 (8) | 0.0017 (8)  |
| C2A | 0.0137 (10) | 0.0131 (10) | 0.0171 (11) | 0.0023 (8)  | -0.0003 (8) | 0.0038 (8)  |
| C3A | 0.0133 (10) | 0.0128 (10) | 0.0153 (10) | 0.0038 (8)  | -0.0014 (8) | 0.0016 (8)  |
| N1B | 0.0150 (9)  | 0.0172 (9)  | 0.0137 (9)  | -0.0002 (7) | 0.0011 (7)  | 0.0006 (7)  |
| N2B | 0.0151 (9)  | 0.0156 (9)  | 0.0130 (9)  | 0.0005 (7)  | 0.0013 (7)  | 0.0007 (7)  |
| N3B | 0.0151 (9)  | 0.0157 (9)  | 0.0134 (9)  | -0.0007 (7) | -0.0004 (7) | 0.0010 (7)  |
| N4B | 0.0180 (9)  | 0.0215 (10) | 0.0116 (9)  | -0.0046 (8) | 0.0049 (8)  | -0.0027 (8) |
| N5B | 0.0172 (9)  | 0.0156 (10) | 0.0122 (9)  | -0.0041 (8) | 0.0016 (7)  | -0.0003 (8) |
| N6B | 0.0201 (10) | 0.0228 (10) | 0.0118 (10) | -0.0067 (8) | 0.0027 (8)  | -0.0011 (8) |
| C1B | 0.0126 (10) | 0.0156 (10) | 0.0157 (11) | 0.0005 (8)  | 0.0004 (8)  | 0.0030 (8)  |
| C2B | 0.0124 (10) | 0.0135 (10) | 0.0133 (10) | 0.0031 (8)  | -0.0012 (8) | 0.0024 (8)  |
| C3B | 0.0131 (10) | 0.0173 (11) | 0.0139 (10) | 0.0011 (8)  | 0.0001 (8)  | 0.0029 (8)  |
| N1C | 0.0196 (9)  | 0.0160 (9)  | 0.0131 (9)  | -0.0026 (7) | 0.0029 (7)  | -0.0004 (7) |
| N2C | 0.0149 (9)  | 0.0149 (9)  | 0.0127 (9)  | 0.0015 (7)  | 0.0021 (7)  | 0.0009 (7)  |
| N3C | 0.0189 (9)  | 0.0136 (9)  | 0.0134 (9)  | -0.0011 (7) | 0.0013 (7)  | 0.0012 (7)  |
| N4C | 0.0205 (10) | 0.0179 (10) | 0.0131 (10) | -0.0037 (8) | 0.0036 (8)  | -0.0008 (8) |
| N5C | 0.0179 (9)  | 0.0137 (9)  | 0.0129 (9)  | -0.0021 (8) | 0.0019 (7)  | -0.0008 (7) |
| N6C | 0.0271 (11) | 0.0206 (10) | 0.0142 (10) | -0.0064 (8) | 0.0055 (8)  | -0.0020 (8) |
| C1C | 0.0142 (10) | 0.0145 (10) | 0.0163 (11) | 0.0037 (8)  | -0.0006 (8) | 0.0014 (8)  |
| C2C | 0.0144 (10) | 0.0127 (10) | 0.0152 (10) | 0.0028 (8)  | 0.0001 (8)  | 0.0021 (8)  |
| C3C | 0.0169 (10) | 0.0137 (10) | 0.0172 (11) | 0.0010 (8)  | 0.0011 (8)  | 0.0016 (8)  |
| N1D | 0.0168 (9)  | 0.0140 (9)  | 0.0139 (9)  | -0.0012 (7) | 0.0007 (7)  | 0.0001 (7)  |
| N2D | 0.0164 (9)  | 0.0150 (9)  | 0.0130 (9)  | 0.0019 (7)  | 0.0010 (7)  | 0.0015 (7)  |
| N3D | 0.0162 (9)  | 0.0142 (9)  | 0.0119 (9)  | 0.0000 (7)  | 0.0005 (7)  | 0.0004 (7)  |
| N4D | 0.0228 (10) | 0.0174 (10) | 0.0106 (10) | -0.0044 (8) | 0.0022 (8)  | -0.0007 (8) |
| N5D | 0.0221 (10) | 0.0152 (10) | 0.0118 (9)  | -0.0045 (8) | 0.0014 (8)  | -0.0005 (8) |



## supplementary materials

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|      |             |             |             |             |             |             |
|------|-------------|-------------|-------------|-------------|-------------|-------------|
| N6D  | 0.0211 (10) | 0.0182 (10) | 0.0118 (9)  | -0.0040 (8) | 0.0023 (8)  | -0.0003 (7) |
| C1D  | 0.0145 (10) | 0.0132 (10) | 0.0151 (10) | 0.0059 (8)  | 0.0003 (8)  | 0.0009 (8)  |
| C2D  | 0.0151 (10) | 0.0147 (10) | 0.0137 (10) | 0.0037 (8)  | 0.0015 (8)  | 0.0037 (8)  |
| C3D  | 0.0152 (10) | 0.0143 (10) | 0.0123 (10) | 0.0040 (8)  | 0.0005 (8)  | 0.0029 (8)  |
| O1AA | 0.0246 (9)  | 0.0249 (10) | 0.0128 (8)  | 0.0000 (7)  | 0.0057 (7)  | 0.0038 (7)  |
| O2AA | 0.0517 (13) | 0.0175 (9)  | 0.0141 (9)  | -0.0037 (8) | 0.0020 (8)  | -0.0033 (7) |
| O3AA | 0.0362 (11) | 0.0232 (10) | 0.0240 (10) | -0.0163 (8) | 0.0075 (8)  | 0.0003 (7)  |
| N8B  | 0.0170 (9)  | 0.0162 (9)  | 0.0150 (9)  | 0.0011 (7)  | 0.0005 (7)  | 0.0020 (7)  |
| O1B  | 0.0221 (8)  | 0.0201 (8)  | 0.0247 (8)  | -0.0096 (6) | 0.0060 (7)  | -0.0002 (6) |
| O2B  | 0.0226 (8)  | 0.0227 (8)  | 0.0130 (7)  | -0.0007 (6) | 0.0058 (6)  | 0.0013 (6)  |
| O3B  | 0.0232 (8)  | 0.0179 (8)  | 0.0132 (7)  | -0.0020 (6) | 0.0019 (6)  | -0.0028 (6) |
| O1W  | 0.0165 (8)  | 0.0196 (8)  | 0.0191 (8)  | -0.0011 (6) | 0.0027 (7)  | 0.0031 (6)  |
| O2W  | 0.0239 (9)  | 0.0194 (8)  | 0.0204 (8)  | -0.0015 (7) | -0.0047 (7) | 0.0048 (7)  |

### *Geometric parameters (Å, °)*

|         |           |          |            |
|---------|-----------|----------|------------|
| N1A—C1A | 1.368 (2) | N4C—C1C  | 1.327 (3)  |
| N1A—C3A | 1.374 (2) | N4C—H1C  | 0.89 (2)   |
| N1A—H1N | 0.91 (2)  | N4C—H2C  | 0.88 (2)   |
| N2A—C1A | 1.328 (2) | N5C—C2C  | 1.317 (2)  |
| N2A—C2A | 1.361 (2) | N5C—H3C  | 0.90 (2)   |
| N3A—C3A | 1.330 (2) | N5C—H4C  | 0.89 (3)   |
| N3A—C2A | 1.355 (2) | N6C—C3C  | 1.317 (3)  |
| N4A—C1A | 1.322 (2) | N6C—H5C  | 0.93 (3)   |
| N4A—H1A | 0.92 (2)  | N6C—H6C  | 0.90 (2)   |
| N4A—H2A | 0.96 (3)  | N1D—C3D  | 1.348 (2)  |
| N5A—C2A | 1.323 (3) | N1D—C1D  | 1.361 (2)  |
| N5A—H3A | 0.88 (2)  | N2D—C1D  | 1.347 (2)  |
| N5A—H4A | 0.86 (2)  | N2D—C2D  | 1.348 (2)  |
| N6A—C3A | 1.313 (2) | N3D—C3D  | 1.341 (2)  |
| N6A—H5A | 0.80 (2)  | N3D—C2D  | 1.356 (2)  |
| N6A—H6A | 0.89 (2)  | N4D—C1D  | 1.333 (3)  |
| N1B—C1B | 1.345 (2) | N4D—H1D  | 0.88 (2)   |
| N1B—C3B | 1.352 (2) | N4D—H2D  | 0.84 (2)   |
| N2B—C1B | 1.358 (2) | N5D—C2D  | 1.338 (3)  |
| N2B—C2B | 1.360 (2) | N5D—H3D  | 0.86 (2)   |
| N3B—C2B | 1.343 (2) | N5D—H4D  | 0.81 (2)   |
| N3B—C3B | 1.350 (2) | N6D—C3D  | 1.352 (2)  |
| N4B—C1B | 1.332 (2) | N6D—H5D  | 0.93 (2)   |
| N4B—H1B | 0.85 (2)  | N6D—H6D  | 0.91 (2)   |
| N4B—H2B | 0.93 (2)  | N8A—O3AB | 1.180 (17) |
| N5B—C2B | 1.330 (2) | N8A—O2AA | 1.235 (2)  |
| N5B—H3B | 0.82 (2)  | N8A—O1AA | 1.250 (2)  |
| N5B—H4B | 0.86 (2)  | N8A—O1AB | 1.257 (16) |
| N6B—C3B | 1.328 (3) | N8A—O3AA | 1.266 (2)  |
| N6B—H5B | 0.85 (2)  | N8A—O2AB | 1.340 (16) |
| N6B—H6B | 0.89 (2)  | N8B—O2B  | 1.243 (2)  |
| N1C—C1C | 1.362 (2) | N8B—O1B  | 1.253 (2)  |
| N1C—C3C | 1.375 (3) | N8B—O3B  | 1.267 (2)  |

|             |             |               |             |
|-------------|-------------|---------------|-------------|
| N1C—H2N     | 0.93 (2)    | O1W—H1W1      | 0.94 (3)    |
| N2C—C1C     | 1.330 (2)   | O1W—H2W1      | 0.85 (4)    |
| N2C—C2C     | 1.359 (2)   | O2W—H1W2      | 0.86 (3)    |
| N3C—C3C     | 1.322 (2)   | O2W—H2W2      | 0.96 (3)    |
| N3C—C2C     | 1.362 (2)   |               |             |
| C1A—N1A—C3A | 119.68 (17) | H3C—N5C—H4C   | 123 (2)     |
| C1A—N1A—H1N | 122.0 (14)  | C3C—N6C—H5C   | 121.6 (16)  |
| C3A—N1A—H1N | 117.9 (14)  | C3C—N6C—H6C   | 119.1 (13)  |
| C1A—N2A—C2A | 115.58 (16) | H5C—N6C—H6C   | 118 (2)     |
| C3A—N3A—C2A | 115.82 (16) | N4C—C1C—N2C   | 120.93 (18) |
| C1A—N4A—H1A | 118.4 (13)  | N4C—C1C—N1C   | 117.64 (18) |
| C1A—N4A—H2A | 116.2 (15)  | N2C—C1C—N1C   | 121.43 (18) |
| H1A—N4A—H2A | 125 (2)     | N5C—C2C—N2C   | 117.48 (17) |
| C2A—N5A—H3A | 120.6 (14)  | N5C—C2C—N3C   | 116.36 (18) |
| C2A—N5A—H4A | 118.9 (14)  | N2C—C2C—N3C   | 126.15 (17) |
| H3A—N5A—H4A | 120 (2)     | N6C—C3C—N3C   | 121.51 (18) |
| C3A—N6A—H5A | 120.8 (16)  | N6C—C3C—N1C   | 116.79 (18) |
| C3A—N6A—H6A | 117.5 (14)  | N3C—C3C—N1C   | 121.70 (17) |
| H5A—N6A—H6A | 122 (2)     | C3D—N1D—C1D   | 114.55 (16) |
| N4A—C1A—N2A | 120.93 (18) | C1D—N2D—C2D   | 114.67 (16) |
| N4A—C1A—N1A | 117.61 (18) | C3D—N3D—C2D   | 114.38 (16) |
| N2A—C1A—N1A | 121.47 (17) | C1D—N4D—H1D   | 119.3 (13)  |
| N5A—C2A—N3A | 116.94 (17) | C1D—N4D—H2D   | 116.6 (15)  |
| N5A—C2A—N2A | 116.77 (18) | H1D—N4D—H2D   | 123 (2)     |
| N3A—C2A—N2A | 126.28 (18) | C2D—N5D—H3D   | 117.8 (15)  |
| N6A—C3A—N3A | 121.21 (18) | C2D—N5D—H4D   | 115.0 (16)  |
| N6A—C3A—N1A | 117.70 (18) | H3D—N5D—H4D   | 126 (2)     |
| N3A—C3A—N1A | 121.08 (17) | C3D—N6D—H5D   | 118.3 (14)  |
| C1B—N1B—C3B | 114.54 (16) | C3D—N6D—H6D   | 115.0 (14)  |
| C1B—N2B—C2B | 114.46 (16) | H5D—N6D—H6D   | 120 (2)     |
| C2B—N3B—C3B | 115.23 (16) | N4D—C1D—N2D   | 117.56 (18) |
| C1B—N4B—H1B | 116.4 (14)  | N4D—C1D—N1D   | 117.41 (18) |
| C1B—N4B—H2B | 120.1 (14)  | N2D—C1D—N1D   | 125.02 (18) |
| H1B—N4B—H2B | 121 (2)     | N5D—C2D—N2D   | 117.53 (18) |
| C2B—N5B—H3B | 118.4 (15)  | N5D—C2D—N3D   | 116.89 (18) |
| C2B—N5B—H4B | 117.1 (15)  | N2D—C2D—N3D   | 125.58 (17) |
| H3B—N5B—H4B | 122 (2)     | N3D—C3D—N1D   | 125.78 (17) |
| C3B—N6B—H5B | 122.0 (15)  | N3D—C3D—N6D   | 118.13 (18) |
| C3B—N6B—H6B | 118.3 (14)  | N1D—C3D—N6D   | 116.07 (17) |
| H5B—N6B—H6B | 120 (2)     | O3AB—N8A—O2AA | 169.5 (9)   |
| N4B—C1B—N1B | 117.02 (18) | O3AB—N8A—O1AA | 57.0 (9)    |
| N4B—C1B—N2B | 117.46 (17) | O2AA—N8A—O1AA | 120.86 (17) |
| N1B—C1B—N2B | 125.51 (17) | O3AB—N8A—O1AB | 129.1 (12)  |
| N5B—C2B—N3B | 118.21 (17) | O2AA—N8A—O1AB | 52.2 (7)    |
| N5B—C2B—N2B | 116.93 (17) | O1AA—N8A—O1AB | 73.0 (8)    |
| N3B—C2B—N2B | 124.86 (17) | O3AB—N8A—O3AA | 63.9 (9)    |
| N6B—C3B—N3B | 117.65 (18) | O2AA—N8A—O3AA | 119.80 (17) |
| N6B—C3B—N1B | 117.06 (18) | O1AA—N8A—O3AA | 119.28 (16) |
| N3B—C3B—N1B | 125.28 (17) | O1AB—N8A—O3AA | 154.5 (8)   |

## supplementary materials

|                 |              |                 |              |
|-----------------|--------------|-----------------|--------------|
| C1C—N1C—C3C     | 119.51 (17)  | O3AB—N8A—O2AB   | 118.6 (12)   |
| C1C—N1C—H2N     | 120.2 (15)   | O2AA—N8A—O2AB   | 58.6 (7)     |
| C3C—N1C—H2N     | 120.2 (14)   | O1AA—N8A—O2AB   | 156.4 (7)    |
| C1C—N2C—C2C     | 115.68 (16)  | O1AB—N8A—O2AB   | 110.8 (11)   |
| C3C—N3C—C2C     | 115.38 (17)  | O3AA—N8A—O2AB   | 67.8 (7)     |
| C1C—N4C—H1C     | 118.9 (15)   | O2B—N8B—O1B     | 120.62 (16)  |
| C1C—N4C—H2C     | 122.7 (15)   | O2B—N8B—O3B     | 120.05 (16)  |
| H1C—N4C—H2C     | 118 (2)      | O1B—N8B—O3B     | 119.34 (16)  |
| C2C—N5C—H3C     | 117.8 (14)   | H1W1—O1W—H2W1   | 102 (3)      |
| C2C—N5C—H4C     | 119.4 (15)   | H1W2—O2W—H2W2   | 104 (2)      |
| C2A—N2A—C1A—N4A | -179.15 (18) | C2C—N2C—C1C—N4C | -179.32 (18) |
| C2A—N2A—C1A—N1A | 0.9 (3)      | C2C—N2C—C1C—N1C | 0.9 (3)      |
| C3A—N1A—C1A—N4A | -178.41 (18) | C3C—N1C—C1C—N4C | -177.41 (18) |
| C3A—N1A—C1A—N2A | 1.5 (3)      | C3C—N1C—C1C—N2C | 2.4 (3)      |
| C3A—N3A—C2A—N5A | -178.58 (18) | C1C—N2C—C2C—N5C | 176.16 (18)  |
| C3A—N3A—C2A—N2A | 2.8 (3)      | C1C—N2C—C2C—N3C | -4.2 (3)     |
| C1A—N2A—C2A—N5A | 178.11 (18)  | C3C—N3C—C2C—N5C | -176.55 (18) |
| C1A—N2A—C2A—N3A | -3.2 (3)     | C3C—N3C—C2C—N2C | 3.8 (3)      |
| C2A—N3A—C3A—N6A | 179.66 (19)  | C2C—N3C—C3C—N6C | -179.85 (19) |
| C2A—N3A—C3A—N1A | 0.0 (3)      | C2C—N3C—C3C—N1C | -0.1 (3)     |
| C1A—N1A—C3A—N6A | 178.31 (18)  | C1C—N1C—C3C—N6C | 176.93 (18)  |
| C1A—N1A—C3A—N3A | -2.0 (3)     | C1C—N1C—C3C—N3C | -2.8 (3)     |
| C3B—N1B—C1B—N4B | -176.03 (18) | C2D—N2D—C1D—N4D | 179.39 (17)  |
| C3B—N1B—C1B—N2B | 2.6 (3)      | C2D—N2D—C1D—N1D | 0.3 (3)      |
| C2B—N2B—C1B—N4B | 178.92 (18)  | C3D—N1D—C1D—N4D | -179.25 (17) |
| C2B—N2B—C1B—N1B | 0.3 (3)      | C3D—N1D—C1D—N2D | -0.2 (3)     |
| C3B—N3B—C2B—N5B | -178.07 (17) | C1D—N2D—C2D—N5D | 178.84 (17)  |
| C3B—N3B—C2B—N2B | 2.4 (3)      | C1D—N2D—C2D—N3D | -1.2 (3)     |
| C1B—N2B—C2B—N5B | 177.45 (17)  | C3D—N3D—C2D—N5D | -178.23 (17) |
| C1B—N2B—C2B—N3B | -3.0 (3)     | C3D—N3D—C2D—N2D | 1.8 (3)      |
| C2B—N3B—C3B—N6B | -179.74 (18) | C2D—N3D—C3D—N1D | -1.7 (3)     |
| C2B—N3B—C3B—N1B | 1.0 (3)      | C2D—N3D—C3D—N6D | 179.96 (17)  |
| C1B—N1B—C3B—N6B | 177.44 (18)  | C1D—N1D—C3D—N3D | 0.9 (3)      |
| C1B—N1B—C3B—N3B | -3.3 (3)     | C1D—N1D—C3D—N6D | 179.31 (17)  |

### 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> |
|------------------------------|-------------|---------------|-----------------------|-------------------------|
| N4A—H1A...O2W <sup>i</sup>   | 0.92 (2)    | 2.05 (2)      | 2.965 (2)             | 174.2 (17)              |
| N4B—H1B...N1D                | 0.85 (2)    | 2.18 (2)      | 3.025 (2)             | 174.1 (18)              |
| N4D—H1D...O3AA               | 0.88 (2)    | 2.27 (2)      | 3.077 (2)             | 153 (2)                 |
| N1A—H1N...O1W <sup>ii</sup>  | 0.91 (2)    | 1.89 (2)      | 2.771 (2)             | 165 (2)                 |
| N4A—H2A...O1B <sup>i</sup>   | 0.96 (3)    | 2.03 (3)      | 2.838 (2)             | 141 (2)                 |
| N4B—H2B...O3AA               | 0.93 (3)    | 2.05 (2)      | 2.810 (2)             | 138.8 (18)              |
| N4C—H2C...N2D <sup>iii</sup> | 0.88 (3)    | 2.07 (3)      | 2.945 (2)             | 176 (2)                 |
| N4D—H2D...N2C <sup>iii</sup> | 0.84 (2)    | 2.23 (2)      | 3.069 (2)             | 172 (2)                 |
| N1C—H2N...O3B                | 0.93 (2)    | 1.91 (2)      | 2.836 (2)             | 177 (2)                 |
| O2W—H1W2...N1D               | 0.86 (3)    | 2.04 (3)      | 2.899 (2)             | 174 (3)                 |

|                             |          |          |           |            |
|-----------------------------|----------|----------|-----------|------------|
| N5A—H3A…N2B                 | 0.88 (3) | 2.20 (3) | 3.062 (2) | 165 (2)    |
| N5B—H3B…N2A                 | 0.82 (3) | 2.30 (3) | 3.119 (2) | 175 (2)    |
| N5C—H3C…O2AA <sup>iii</sup> | 0.89 (2) | 2.06 (2) | 2.799 (2) | 140 (2)    |
| N5D—H3D…O3AA <sup>iv</sup>  | 0.86 (2) | 2.53 (2) | 3.232 (3) | 140 (2)    |
| N5A—H4A…O1AA                | 0.86 (2) | 2.11 (2) | 2.963 (2) | 171 (2)    |
| N5B—H4B…O2B <sup>i</sup>    | 0.86 (2) | 2.20 (2) | 3.045 (2) | 167 (2)    |
| N5C—H4C…N3D <sup>v</sup>    | 0.89 (3) | 2.12 (3) | 2.996 (2) | 173 (2)    |
| N5D—H4D…N3C <sup>v</sup>    | 0.81 (3) | 2.30 (3) | 3.105 (3) | 173 (2)    |
| N6A—H5A…N3B <sup>vi</sup>   | 0.80 (2) | 2.13 (2) | 2.926 (2) | 175.9 (19) |
| N6B—H5B…N3A <sup>vii</sup>  | 0.84 (2) | 2.18 (2) | 3.020 (2) | 177 (2)    |
| N6C—H5C…O2W                 | 0.93 (3) | 2.00 (3) | 2.849 (2) | 151 (2)    |
| N6D—H5D…O2AA <sup>vii</sup> | 0.93 (3) | 2.22 (3) | 3.126 (2) | 167 (2)    |
| N6A—H6A…O1W <sup>ii</sup>   | 0.90 (2) | 2.55 (2) | 3.262 (2) | 137.2 (15) |
| N6A—H6A…O2B <sup>viii</sup> | 0.90 (2) | 2.14 (2) | 2.841 (2) | 135.2 (17) |
| N6B—H6B…O1AA <sup>vii</sup> | 0.89 (2) | 2.17 (3) | 2.806 (2) | 128 (2)    |
| N6C—H6C…O1B                 | 0.90 (2) | 1.97 (2) | 2.868 (2) | 174.6 (18) |
| N6D—H6D…N1B                 | 0.91 (2) | 2.18 (2) | 3.088 (2) | 172 (2)    |

Symmetry codes: (i)  $-x+1, -y+1, -z$ ; (ii)  $x+1, y, z-1$ ; (iii)  $-x+1, -y+2, -z+1$ ; (iv)  $-x+1, -y+1, -z+1$ ; (v)  $-x, -y+1, -z+1$ ; (vi)  $x+1, y+1, z$ ; (vii)  $x-1, y-1, z$ ; (viii)  $-x+2, -y+2, -z$ .

Fig. 1

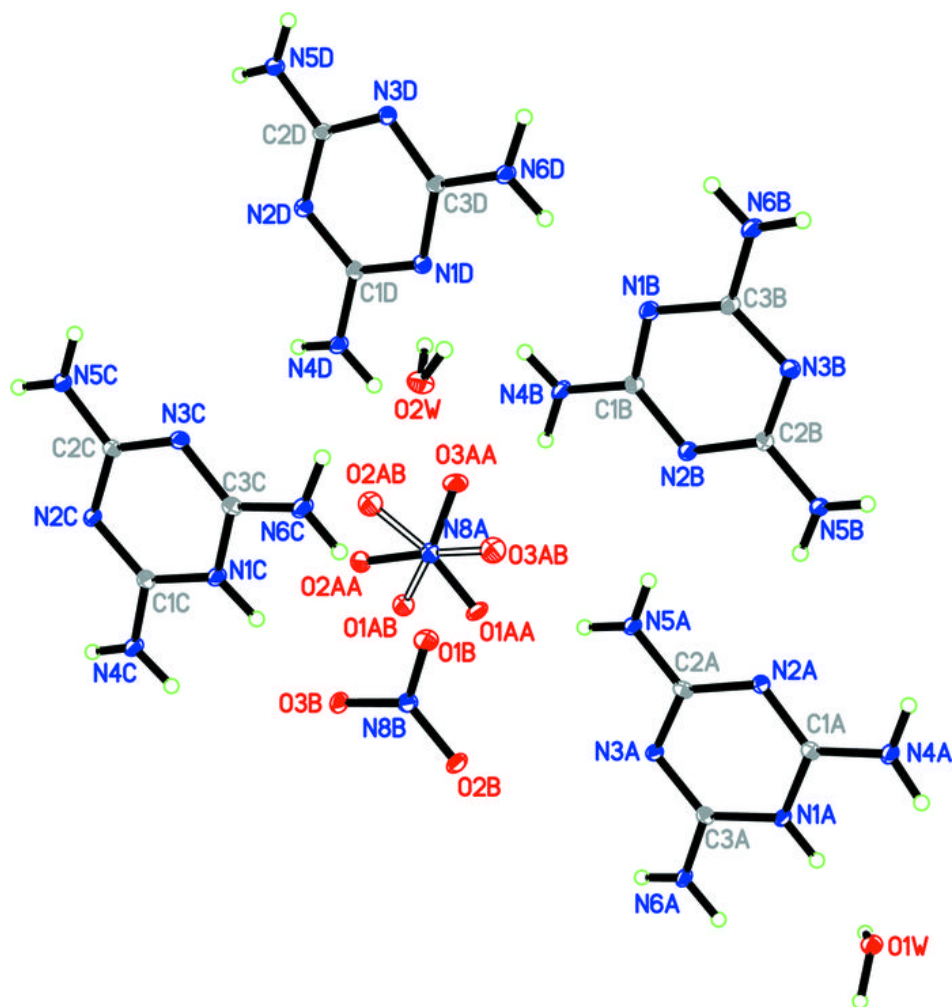


Fig. 2

