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2,4-Diamino-6-methyl-1,3,5-triazine methanol solvate

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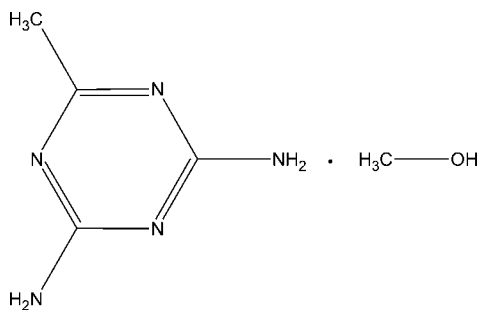
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 Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.050; wR factor = 0.146; data-to-parameter ratio = 14.2.

The crystal structure of the title compound, $\text{C}_4\text{H}_7\text{N}_5 \cdot \text{CH}_4\text{O}$, is determined by an extensive network of hydrogen bonding. A sequence of centrosymmetric dimeric associations, formed by two different $\text{N}-\text{H}(\text{amino}) \cdots \text{N}(\text{ring})$ hydrogen bonds, connects the triazine rings into a planar molecular tape. The methanol solvent molecules act as di-acceptors and mono-donors of hydrogen bonds and interlink, almost perpendicularly, the hydrogen-bonded tapes into a three-dimensional structure.

Related literature

For related literature, see: Allen (2002); Radecka-Paryzek *et al.* (2005); Šebenik *et al.* (1989); Tashiro & Oiwa (1981).



Experimental

Crystal data

 $\text{C}_4\text{H}_7\text{N}_5 \cdot \text{CH}_4\text{O}$
 $M_r = 157.19$

 Monoclinic, $C2/c$
 $a = 21.024$ (5) Å

 $b = 5.4726$ (10) Å
 $c = 14.198$ (3) Å
 $\beta = 95.66$ (2)°
 $V = 1625.6$ (6) Å³
 $Z = 8$

 Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 295$ (2) K
 $0.4 \times 0.2 \times 0.2$ mm

Data collection

 Kuma KM4 CCD diffractometer
 Absorption correction: none
 5226 measured reflections

 1737 independent reflections
 1191 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.019$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.146$
 $S = 1.07$
 1737 reflections
 122 parameters

 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.17$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.28$ e Å⁻³
Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{O1S}-\text{H1S} \cdots \text{N5}$	0.96 (4)	1.86 (4)	2.816 (2)	176 (3)
$\text{N2}-\text{H2B} \cdots \text{O1S}^{\text{i}}$	0.87 (3)	2.26 (3)	3.093 (2)	160 (2)
$\text{N2}-\text{H2A} \cdots \text{N1}^{\text{ii}}$	0.86 (2)	2.20 (2)	3.060 (2)	180 (2)
$\text{N4}-\text{H4B} \cdots \text{O1S}^{\text{iii}}$	0.89 (2)	2.27 (2)	2.956 (2)	133.2 (19)
$\text{N4}-\text{H4A} \cdots \text{N3}^{\text{iv}}$	0.88 (2)	2.15 (2)	3.024 (2)	177.3 (19)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *Stereochemical Workstation Operation Manual* (Siemens, 1989); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2183).

References

- Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.
 Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
 Oxford Diffraction (2006). *CrysAlis CCD* (Version 1.171.31.5) and *CrysAlis RED* (Version 1.171.31.5). Oxford Diffraction Poland Sp. z o.o., Wrocław, Poland.
 Radecka-Paryzek, W., Patroniak, V. & Lisowski, J. (2005). *Coord. Chem. Rev.* **249**, 2156–2175.
 Šebenik, A., Osredkar, U. & Žigon, M. (1989). *Polym. Bull.* **22**, 155–161.
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
 Siemens (1989). *Stereochemical Workstation Operation Manual*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Tashiro, T. & Oiwa, M. (1981). *J. Polym. Sci. Polym. Chem.* **19**, 645–654.

supporting information

Acta Cryst. (2008). E64, o269 [https://doi.org/10.1107/S160053680706607X]

2,4-Diamino-6-methyl-1,3,5-triazine methanol solvate**Małgorzata Kaczmarek, Wanda Radecka-Paryzek and Maciej Kubicki****S1. Comment**

Triazine compounds are used in pharmaceutical industry as coupling agents for the synthesis of peptides and as side chain of antibiotics, as well as in formulating bactericides and fungicides. 2,4-Diamino-6-methyl-1,3,5-triazine (acetoguanamine) is used as an intermediate for pharmaceuticals and as a modifier and flexibilizer of formaldehyde resins (Šebenik *et al.* 1989, Tashiro *et al.* 1981). The title compound, 2,4-diamino-6-methyl-1,3,5-triazine methanol solvate, was isolated during the efforts to prepare new lanthanide macrocyclic complexes as part of our research program involving the study of the coordination template effect in generating the supramolecular Schiff base macrocycles derived from various diamines and dicarbonyls (Radecka-Paryzek *et al.* 2005).

The bond lengths within the ring are exceptionally uniform in all the 2,4-diaminotriazine derivatives. For 38 compounds found in the CSD (Allen, 2002; search conditions: only organics, no disorder, no errors) the mean standard deviation of the C?N bond lengths is as small as 0.007 Å. The same is true for the title compound, the mean value of the C?N bond distances being 1.346 (14) Å. The triazine ring is planar (Fig. 1), with a maximum deviation from the least-squares plane of 0.013 (1) Å for N5. The amino groups are almost coplanar with the ring plane, the dihedral angles between the NH₂ groups and the ring plane are 6(3)° for N2H₂ and 2(2)° for N4H₂. Only the methyl carbon atom C61 deviates significantly from the plane by 0.055 (3) Å.

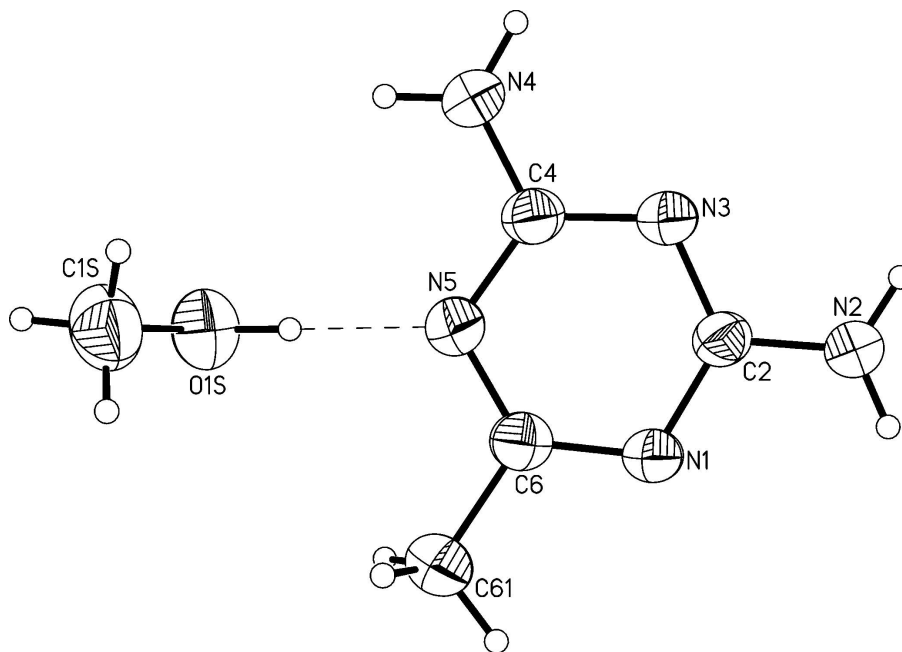
The crystal structure is determined by an extensive network of hydrogen bonds. Each NH₂ group acts as a donor in hydrogen bond with the ring nitrogen atoms of neighboring molecules, related by two different centres of symmetry. The sequence of such hydrogen-bonded dimers creates an almost planar molecular tape of molecules along the [101] direction (Fig. 2). The tapes are interlinked by hydrogen bonds with the methanol solvent molecules, which act as di-acceptors and mono-donors of hydrogen bonds (Fig. 3). As a result, a three-dimensional structure of almost perpendicular tapes is formed in the crystal.

S2. Experimental

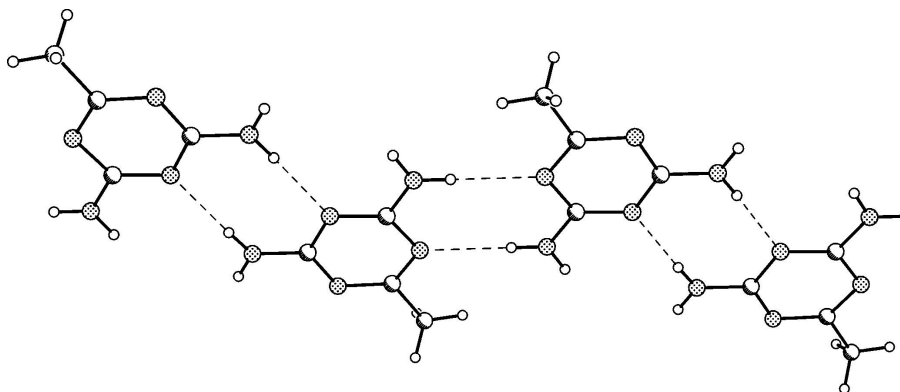
To a solution of lanthanum(III) nitrate complex of Schiff base ligand, product of [2 + 1] condensation of one molecule of 2,4-diamino-6-methyl-1,3,5-triazine with two molecules of 2,6-diacetylpyridine (0.1 mmol), in methanol (10 ml), 2,4-diamino-6-methyl-1,3,5-triazine (0.1 mmol) dissolved in hot methanol (10 ml) was added in order to receive the [2 + 2] Schiff base macrocyclic complex. After standing at room temperature for several hours, transparent crystals of the title compound were obtained. The crystals were initially transparent but after few minutes in open air they became opaque and gradually lost their crystallinity. Therefore the crystal used for data collection was sealed in a glass capillary.

S3. Refinement

The methyl hydrogen atoms were positioned geometrically (AFIX 137) and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.3 U_{\text{eq}}(\text{C})$. All other hydrogen atoms were located in difference Fourier maps and freely refined.

**Figure 1**

Anisotropic displacement ellipsoid representation (at the 50% probability level) of the molecule of the title compound, together with the numbering scheme. The hydrogen atoms are drawn as spheres with arbitrary radii. The intermolecular hydrogen bond is depicted as a dashed line.

**Figure 2**

The molecular tape along [101] direction. Hydrogen bonds are depicted as dashed lines. Symmetry codes: (i) x, y, z (ii) $-x, 2 - y, -2z$ (iii) $1/2 - x, 5/2 - y, 2 - z$ (iv) $1/2 + x, 1/2 + y, z$.

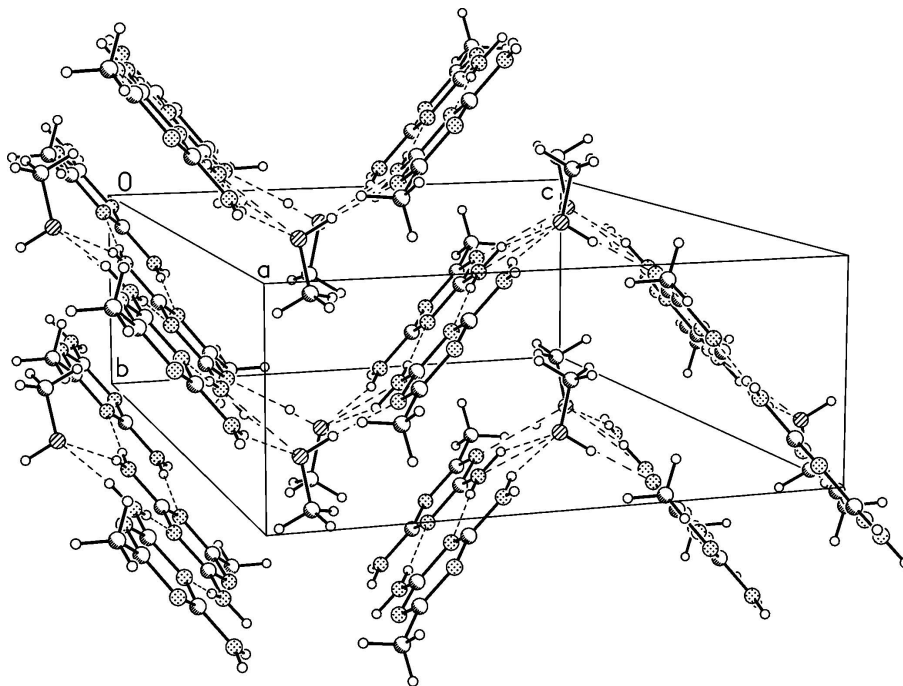


Figure 3

The packing of the molecules as seen approximately along the *a* axis. Hydrogen bonds are shown as dashed lines.

2,4-Diamino-6-methyl-1,3,5-triazine methanol solvate

Crystal data

$C_4H_7N_5 \cdot CH_4O$

$M_r = 157.19$

Monoclinic, $C2/c$

Hall symbol: $-C 2yc$

$a = 21.024 (5) \text{ \AA}$

$b = 5.4726 (10) \text{ \AA}$

$c = 14.198 (3) \text{ \AA}$

$\beta = 95.66 (2)^\circ$

$V = 1625.6 (6) \text{ \AA}^3$

$Z = 8$

$F(000) = 672$

$D_x = 1.285 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 2082 reflections

$\theta = 4\text{--}22^\circ$

$\mu = 0.10 \text{ mm}^{-1}$

$T = 295 \text{ K}$

Block, colourless

$0.4 \times 0.2 \times 0.2 \text{ mm}$

Data collection

Kuma KM-4-CCD four-circle
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: $8.1929 \text{ pixels mm}^{-1}$

ω scans

5226 measured reflections

1737 independent reflections

1191 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.019$

$\theta_{\text{max}} = 27.0^\circ$, $\theta_{\text{min}} = 2.9^\circ$

$h = -26 \rightarrow 26$

$k = -4 \rightarrow 6$

$l = -18 \rightarrow 18$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.050$

$wR(F^2) = 0.146$

$S = 1.07$

1737 reflections

122 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

$$w = 1/[\sigma^2(F_o^2) + (0.0834P)^2 + 0.3526P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.17 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.29 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.19697 (6)	1.0111 (2)	0.94292 (10)	0.0405 (4)
C2	0.14975 (8)	1.1070 (3)	0.99110 (11)	0.0375 (4)
N2	0.16650 (8)	1.2910 (3)	1.04951 (12)	0.0491 (4)
H2B	0.1372 (12)	1.360 (4)	1.0792 (17)	0.069 (7)*
H2A	0.2049 (11)	1.346 (4)	1.0518 (15)	0.056 (6)*
N3	0.08942 (6)	1.0268 (3)	0.98441 (10)	0.0410 (4)
C4	0.07625 (8)	0.8402 (3)	0.92379 (12)	0.0400 (4)
N4	0.01687 (8)	0.7548 (3)	0.91381 (14)	0.0576 (5)
H4B	0.0074 (11)	0.634 (4)	0.8728 (16)	0.065 (7)*
H4A	-0.0141 (11)	0.813 (4)	0.9441 (16)	0.055 (6)*
N5	0.11934 (6)	0.7355 (3)	0.87109 (10)	0.0412 (4)
C6	0.17841 (8)	0.8266 (3)	0.88561 (12)	0.0388 (4)
C61	0.22793 (9)	0.7086 (4)	0.83245 (15)	0.0521 (5)
H61A	0.2682	0.7898	0.8472	0.068*
H61B	0.2154	0.7205	0.7657	0.068*
H61C	0.2321	0.5397	0.8502	0.068*
O1S	0.08620 (7)	0.4874 (2)	0.69953 (10)	0.0574 (4)
H1S	0.0976 (14)	0.579 (6)	0.756 (3)	0.113 (11)*
C1S	0.08937 (12)	0.2368 (4)	0.71909 (17)	0.0707 (7)
H1S1	0.1329	0.1916	0.7380	0.092*
H1S2	0.0739	0.1469	0.6634	0.092*
H1S3	0.0635	0.2000	0.7693	0.092*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0315 (7)	0.0499 (8)	0.0406 (8)	-0.0021 (6)	0.0066 (6)	-0.0054 (6)
C2	0.0315 (8)	0.0475 (9)	0.0331 (8)	-0.0026 (7)	0.0021 (6)	-0.0012 (7)
N2	0.0350 (8)	0.0597 (10)	0.0532 (10)	-0.0065 (7)	0.0078 (7)	-0.0180 (8)
N3	0.0327 (8)	0.0499 (8)	0.0412 (8)	-0.0050 (6)	0.0078 (6)	-0.0053 (6)

C4	0.0344 (9)	0.0465 (9)	0.0397 (9)	-0.0035 (7)	0.0056 (7)	-0.0003 (7)
N4	0.0382 (9)	0.0674 (11)	0.0689 (11)	-0.0150 (8)	0.0135 (8)	-0.0252 (10)
N5	0.0387 (8)	0.0436 (8)	0.0419 (8)	-0.0041 (6)	0.0068 (6)	-0.0049 (6)
C6	0.0365 (9)	0.0442 (9)	0.0358 (9)	0.0014 (7)	0.0034 (7)	0.0019 (7)
C61	0.0441 (10)	0.0601 (11)	0.0534 (11)	0.0022 (9)	0.0113 (8)	-0.0105 (9)
O1S	0.0728 (10)	0.0551 (8)	0.0426 (8)	0.0036 (7)	-0.0031 (7)	0.0031 (6)
C1S	0.0870 (18)	0.0544 (13)	0.0731 (15)	-0.0019 (11)	0.0203 (13)	-0.0045 (11)

Geometric parameters (Å, °)

N1—C6	1.331 (2)	N5—C6	1.335 (2)
N1—C2	1.365 (2)	C6—C61	1.492 (2)
C2—N2	1.330 (2)	C61—H61A	0.9600
C2—N3	1.337 (2)	C61—H61B	0.9600
N2—H2B	0.87 (3)	C61—H61C	0.9600
N2—H2A	0.86 (2)	O1S—C1S	1.400 (2)
N3—C4	1.347 (2)	O1S—H1S	0.96 (4)
C4—N4	1.328 (2)	C1S—H1S1	0.9600
C4—N5	1.358 (2)	C1S—H1S2	0.9600
N4—H4B	0.89 (2)	C1S—H1S3	0.9600
N4—H4A	0.88 (2)		
C6—N1—C2	114.47 (14)	N1—C6—C61	117.42 (15)
N2—C2—N3	118.93 (16)	N5—C6—C61	116.44 (16)
N2—C2—N1	116.28 (15)	C6—C61—H61A	109.5
N3—C2—N1	124.78 (16)	C6—C61—H61B	109.5
C2—N2—H2B	118.5 (17)	H61A—C61—H61B	109.5
C2—N2—H2A	118.6 (14)	C6—C61—H61C	109.5
H2B—N2—H2A	123 (2)	H61A—C61—H61C	109.5
C2—N3—C4	115.34 (14)	H61B—C61—H61C	109.5
N4—C4—N3	117.85 (16)	C1S—O1S—H1S	110 (2)
N4—C4—N5	117.65 (17)	O1S—C1S—H1S1	109.5
N3—C4—N5	124.49 (15)	O1S—C1S—H1S2	109.5
C4—N4—H4B	118.5 (15)	H1S1—C1S—H1S2	109.5
C4—N4—H4A	123.7 (14)	O1S—C1S—H1S3	109.5
H4B—N4—H4A	118 (2)	H1S1—C1S—H1S3	109.5
C6—N5—C4	114.73 (15)	H1S2—C1S—H1S3	109.5
N1—C6—N5	126.14 (15)		
C6—N1—C2—N2	179.66 (15)	N4—C4—N5—C6	-178.87 (16)
C6—N1—C2—N3	0.7 (2)	N3—C4—N5—C6	2.1 (3)
N2—C2—N3—C4	179.99 (16)	C2—N1—C6—N5	1.3 (2)
N1—C2—N3—C4	-1.1 (3)	C2—N1—C6—C61	-178.50 (15)
C2—N3—C4—N4	-179.45 (16)	C4—N5—C6—N1	-2.6 (3)
C2—N3—C4—N5	-0.4 (3)	C4—N5—C6—C61	177.21 (15)

Hydrogen-bond geometry (Å, °)

<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>
O1S—H1S \cdots N5	0.96 (4)	1.86 (4)	2.816 (2)	176 (3)
N2—H2B \cdots O1S ⁱ	0.87 (3)	2.26 (3)	3.093 (2)	160 (2)
N2—H2A \cdots N1 ⁱⁱ	0.86 (2)	2.20 (2)	3.060 (2)	180 (2)
N4—H4B \cdots O1S ⁱⁱⁱ	0.89 (2)	2.27 (2)	2.956 (2)	133.2 (19)
N4—H4A \cdots N3 ^{iv}	0.88 (2)	2.15 (2)	3.024 (2)	177.3 (19)

Symmetry codes: (i) $x, -y+2, z+1/2$; (ii) $-x+1/2, -y+5/2, -z+2$; (iii) $-x, y, -z+3/2$; (iv) $-x, -y+2, -z+2$.