

2,6-Diamino-9H-purine monohydrate and bis(2,6-diamino-9H-purin-1-ium) 2-(2-carboxylatophenyl)acetate heptahydrate: two simple structures with very complex hydrogen-bonding schemes

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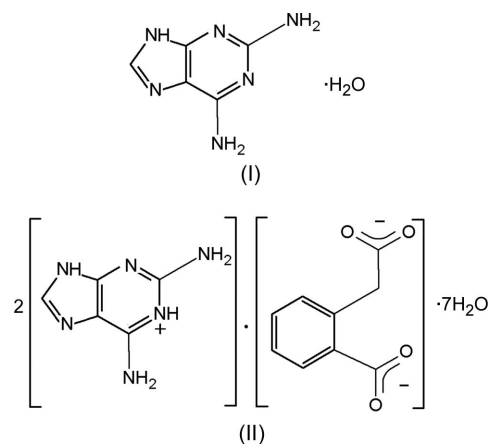
Two structures presenting an uncomplexed 2,6-diaminopurine (dap) group are reported, namely 2,6-diamino-9H-purine monohydrate, C₅H₆N₆·H₂O, (I), and bis(2,6-diamino-9H-purin-1-ium) 2-(2-carboxylatophenyl)acetate heptahydrate, 2C₅H₇N₆⁺·C₉H₆O₄²⁻·7H₂O, (II). Both structures are rather featureless from a molecular point of view, but present instead an outstanding hydrogen-bonding scheme. In compound (I), this is achieved through a rather simple independent unit content (one neutral dap and one water molecule) and takes the form of two-dimensional layers tightly connected by strong hydrogen bonds, and interlinked by much weaker hydrogen bonds and π - π interactions. In compound (II), the fundamental building blocks are more complex, consisting of two independent 2,6-diamino-9H-purin-1-ium (Hdap⁺) cations, one homophthalate [2-(2-carboxylatophenyl)acetate] dianion and seven solvent water molecules. The large number of hydrogen-bond donors and acceptors produces 26 independent interactions, leading to an extended and complicated network of hydrogen bonds in a packing organization characterized by the stacking of interleaved anionic and cationic planar arrays. These structural characteristics are compared with those of similar compounds in the literature.

Comment

Purines constitute a family of heterocyclic compounds characterized by a fused pair of pyrimidine and imidazole rings. The simplest representative is purine itself, mostly found in nature as methyl-, hydroxy- and amino-substituted derivatives.

These are essential compounds for biological systems, to the extent that many of the building blocks of DNA and RNA are purines of this kind. The relevance of these small molecules resides mainly in their highly interactive hydrogen-bonding capabilities resulting from the many active sites (both donors and acceptors) available in their structures. This condition is, of course, not restricted to life processes, and renders these compounds extremely appealing from a synthetic point of view when searching for suitable building blocks for complicated supramolecular structures. Accordingly, much structural work has been devoted to some of these purines (adenine and guanine, among others) but many have not been adequately surveyed from a structural point of view. In this latter category, 2,6-diaminopurine (C₅H₆N₆, hereinafter dap) occupies a notable position: only two occurrences of compounds containing the molecule could be found in the Cambridge Structural Database (CSD, Version of 2010; Allen, 2002). We shall return to this point in the discussion below.

Since it seemed this was a gap worth filling, we decided to explore the synthesis and crystallographic study of transition metal complexes, incorporating dap as the main ligand, through a project which is beginning to provide interesting results (in preparation). As an introductory paper to this intended series of structural reports, we report here two different structures which feature the dap molecule as an uncomplexed group. One of them, dap monohydrate, (I), presents the molecule as a neutral free base, while in the other, bis(2,6-diamino-9H-purin-1-ium) homophthalate heptahydrate or 2Hdap⁺·hpt²⁻·7H₂O [where H₂hpt is homophthalic acid or 2-(2-carboxylatophenyl)acetic acid], (II), it fulfils the role of a cation. In spite of the obvious differences displayed by these two structures, *viz.* the protonated state of the dap group, the number of solvent water molecules, *etc.*, the molecule provides a similarly complex hydrogen-bonding network, which will be discussed below.



From the molecular point of view, both structures are quite simple, since the individual components do not deviate from the expected geometries, with bond distances and angles lying within reported values for these species (CSD; Allen 2002).

Fig. 1 presents an ellipsoid plot of the asymmetric unit contents in (I), consisting of a neutral dap molecule and one solvent water molecule. As expected, the dap unit is planar

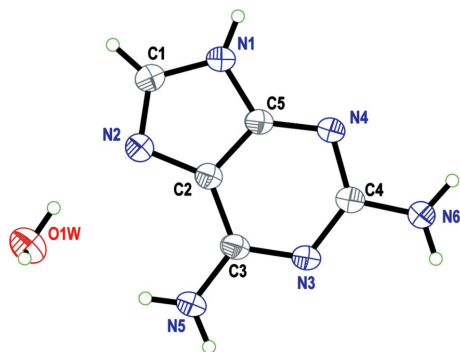


Figure 1
The molecular structure of (I), showing the atom-labelling scheme used. Displacement ellipsoids are drawn at the 50% probability level.

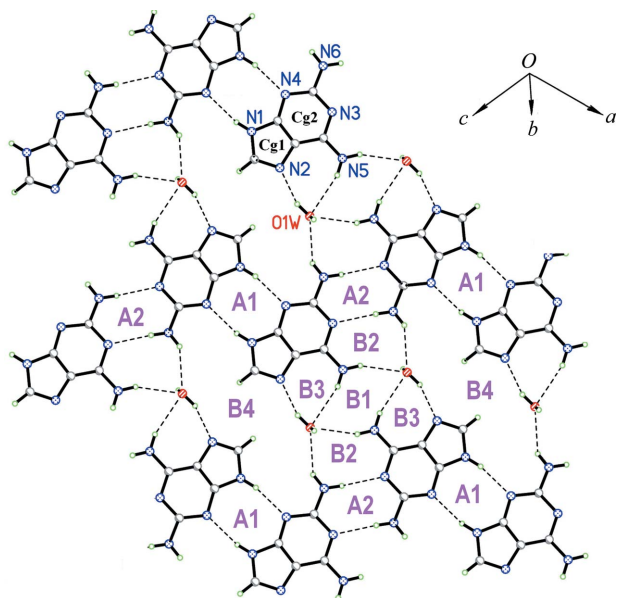


Figure 2
A packing view of (I), showing a single plane and the rings which the hydrogen bonds (dashed lines) generate. Note the definitions of the ring centroids.

[maximum deviation from the mean plane is 0.015 (1) Å for atom C1], and the mean plane defined by the molecule deviates by just 0.15 Å from the crystallographic centre of symmetry at $(0, 0, \frac{1}{2})$, so that a thin planar arrangement builds up *via* this $\bar{1}$ symmetry operation, with an overall deviation from the mean plane of 0.15 (1) Å and a maximum deviation of 0.42 (1) Å for atom N6. In the resulting two-dimensional structure parallel to $(1\bar{2}1)$, molecules of dap are interlinked by most of the existing hydrogen-bonding interactions (six out of a total of seven; Fig. 2 and Table 1) which define a number of closed structures, in particular a couple of centrosymmetric loops with graph-set descriptors $R_2^2(8)$ [For details on graph-set notation as applied to hydrogen bonding, see Bernstein *et al.* (1995).] (labelled A1 and A2 in Fig. 2) involving only dap molecules with no intervention of the solvent water molecule (first two entries in Table 1), and which define a zigzag chain along $[10\bar{1}]$. These parallel chains are in turn connected along $[111]$ by a number of loops where the water molecule plays an

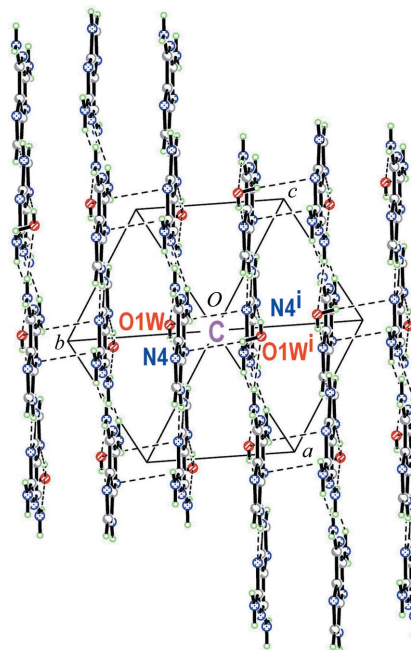


Figure 3
A packing view of (I), at right angles to that in Fig. 2, showing the way in which the planes interact. Hydrogen bonds are shown as dashed lines. [Symmetry code: (i) $-x + 1, -y + 1, -z + 1$.]

active role, being involved in four different hydrogen bonds in the plane, three of them as an acceptor (entries 3–5 in Table 1) and one as a donor (entry 6). The result is the formation of a centrosymmetric $R_4^2(8)$ ring (B1 in Fig. 2), around which another three different loops build up in pairs, with graph-set descriptors $R_2^2(8)$ (B2), $R_2^2(7)$ (B3) and a rather large $R_6^6(20)$ (B4). The planar structures stack parallel to each other at a distance of *ca* 3.5 Å (Fig. 3), and are weakly linked by a mixture of hydrogen bonds [through an $N \cdots H - O - H \cdots N$ chain involving both water H atoms (entries 6 and 7 in Table 1), which define a centrosymmetric $R_4^4(14)$ loop around the inversion centre at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$] (labelled 'C' in Fig. 3), and some π - π interactions, presumably involving the whole two-dimensional structure in a generalized fashion. Individual

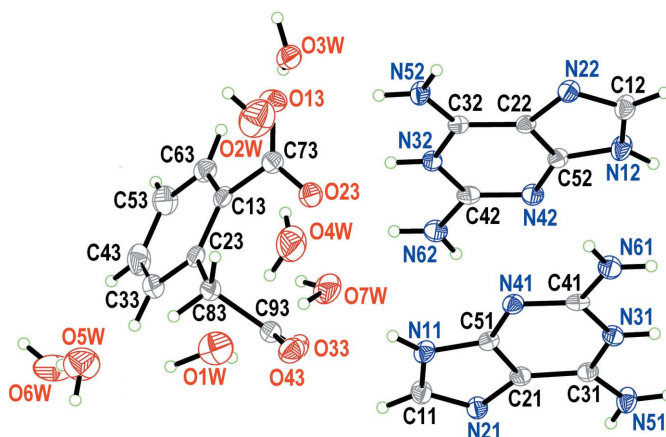


Figure 4
The molecular structure of (II), showing the atom-labelling scheme used. Displacement ellipsoids are drawn at the 30% probability level.

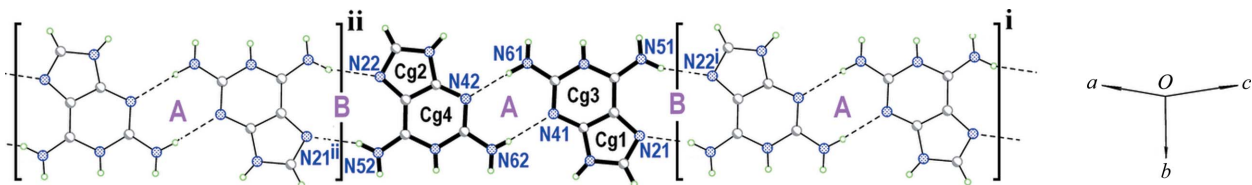


Figure 5

A partial packing view of (II), showing a single strip in the cationic structure. The reference dimeric pair of Hdpa⁺ ions is shown in bold and the symmetry-related ones by simple lines. Trailing labels 1 and 2 characterize the Hdpa⁺ ions, and 'A' and 'B' denote the rings produced by the hydrogen bonds (dashed lines). [Symmetry codes: (i) $x - 1, y, z + 1$; (ii) $x + 1, y, z - 1$.]

short contacts between parallel rings fulfilling the π - π bonding criteria are presented in Table 2.

Fig. 4 shows the molecular structure of (II). The asymmetric unit, noticeably more complex than that in structure (I), consists of two Hdpa⁺ cations (atoms identified by trailing labels 1 and 2, respectively) counterbalanced by one homophthalate hpt²⁻ anion (trailing label 3) and completed by seven solvent water molecules. It is worth mentioning, for future reference, that the two independent Hdpa⁺ cations of (II) are identical, with protonation at N taking place at the same sites, *viz.* N11 and N31, and N12 and N32.

The two independent Hdpa⁺ cations also show very small individual deviations from planarity: 0.029 (1) Å for atom N51 and 0.033 (1) Å for atom C22. In addition, they lie almost on the same plane, parallel to (101), with a slightly larger deviation from the mean plane (0.069 Å for atom N51) when both cations are considered in bulk. A peculiar result of this disposition is that these molecules and their $(x + 1, y, z - 1)$ and $(x - 1, y, z + 1)$ translation images determine almost perfect and extremely thin infinite strips running along $[\bar{1}01]$ [overall deviation from the mean plane for the whole assembly is 0.02 (1) Å, with a maximum of 0.09 (1) Å for atom N51]. This structure (to be discussed below) bears some resemblance to the planar disposition of the dap molecules in (I).

The hpt²⁻ anion of (II), in turn, presents a planar inner phenyl core [maximum deviation from the least-squares plane is 0.017 (1) Å for atom C63], the deviations from planarity residing instead in the lateral arms due to rotations in both the carboxylate and the ethyl groups. Fig. 4 gives a qualitative view of this out-of-plane geometry, while Table 3 presents a few torsion angles, quantitatively describing the situation.

In contrast with the deceptive simplicity of the structure when only the molecular aspects are considered, the assembly of the elemental units into a three-dimensional supra-molecular organization proves to be extremely complex and possesses a great richness of detail, mainly due to the very large number of hydrogen-bonding donors and acceptors present in the anions, cations and solvent molecules.

A detailed analysis of the hydrogen-bonding scheme of (II) reveals that there are 26 potentially active H atoms (14 from water and 12 from Hdap), all of which are involved, and 15 potential acceptors (seven water O—H, four hpt²⁻ O—H and four Hdap⁺ N—H), all of them active. This leads to a large ratio of 1.73 hydrogen bonds per acceptor.

As a result of the abundance of hydrogen-bonding interactions (Table 4) and π - π contacts (Table 5), the structure

naturally segregates into two well differentiated substructures, one of them cationic, made up of Hdap⁺ cations only, and the remaining one anionic and composed of hpt²⁻ anions and water molecules. Both substructures present neat well differentiated characteristics, which we shall describe below.

In the cationic substructure, the basic component is the pair of coplanar Hdap⁺ cations strongly interlinked into a dimeric hydrogen-bonded structure through head-to-tail interactions, forming $R_2^2(8)$ loops ('A' in Fig. 5) involving atoms H61A and H62A (Table 4, entries 1 and 2). These hydrogen-bonded dimers, in turn, link to their $(x + 1, y, z - 1)$ and $(x - 1, y, z + 1)$ translation images, forming $R_2^2(10)$ loops ('B' in Fig. 5) involving atoms H51B and H52B (entries 3 and 4 in Table 4), thus defining planar strips running along $[\bar{1}01]$, as shown in Fig. 5. When the *c*-glide operates on these strips, it generates a parallel image separated from the former, original, ones by a distance of roughly one-quarter of a $[101]$ translation (Fig. 6*a*), to build up broad two-dimensional structures parallel to (010) at $y \sim \frac{1}{4}, \frac{3}{4}$ (Figs. 6*a* and 6*b*). Even if there are in principle particular aromatic rings fulfilling the expected geometric conditions for π - π contacts (Table 5, first and second entries), the interaction between strips should probably be considered as a collective one, with an interplanar $d(\bar{2}02)$ distance of 3.358 (1) Å.

The anionic substructure is of a completely different nature. It is built up of hpt²⁻ anions and water molecules, generating hollow structures parallel to $[010]$ at $y \sim 0, \frac{1}{2}$ and limited by two parallel 'walls' made completely out of hydrophilic entities (water molecules and carboxylate groups), linked into a planar hydrogen-bonding network involving 13 out of the 14 available water H atoms (Table 4, entries 5 to 17, and Fig. 7*a*).

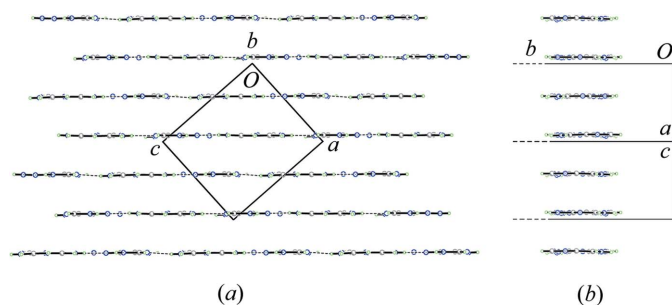


Figure 6

A partial packing view of (II), showing the relative positioning of the cationic strips shown in Fig. 5, viewed (a) along $[010]$, showing the way strips stack, and (b) at right angles to the previous view, projected along the strip direction, $[\bar{1}01]$.

Experimental

A methanol solution (20 ml) of 2,6-diaminopurine (0.5 mmol) was mixed with an aqueous solution containing homophthalic acid (0.5 mmol) and NaOH (1.0 mmol), and the resulting mixture was heated under reflux with stirring for 2 h. While trying to obtain crystals suitable for X-ray analysis by slow evaporation at room temperature, two well differentiated species appeared, *viz.* thin colourless plates of (I), as a minor component, and well developed pale-brown prisms of (II) as the major component.

Compound (I)

Crystal data

$C_5H_6N_6 \cdot H_2O$	$\gamma = 101.331 (4)^\circ$
$M_r = 168.17$	$V = 354.89 (14) \text{ \AA}^3$
Triclinic, $P\bar{1}$	$Z = 2$
$a = 6.9331 (15) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 7.1079 (16) \text{ \AA}$	$\mu = 0.12 \text{ mm}^{-1}$
$c = 7.5564 (17) \text{ \AA}$	$T = 298 \text{ K}$
$\alpha = 99.089 (4)^\circ$	$0.28 \times 0.25 \times 0.08 \text{ mm}$
$\beta = 98.029 (3)^\circ$	

Data collection

Bruker SMART CCD area-detector diffractometer	2992 measured reflections
Absorption correction: multi-scan (SADABS in SAINT-NT; Bruker, 2002)	1521 independent reflections
$T_{\min} = 0.96, T_{\max} = 0.99$	1280 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.011$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.047$	109 parameters
$wR(F^2) = 0.127$	H-atom parameters constrained
$S = 1.03$	$\Delta\rho_{\text{max}} = 0.34 \text{ e \AA}^{-3}$
1521 reflections	$\Delta\rho_{\text{min}} = -0.36 \text{ e \AA}^{-3}$

Compound (II)

Crystal data

$2C_5H_7N_6^+ \cdot C_9H_6O_4^{2-} \cdot 7H_2O$	$V = 2828.2 (12) \text{ \AA}^3$
$M_r = 606.58$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 8.928 (2) \text{ \AA}$	$\mu = 0.12 \text{ mm}^{-1}$
$b = 31.393 (8) \text{ \AA}$	$T = 298 \text{ K}$
$c = 10.091 (3) \text{ \AA}$	$0.22 \times 0.16 \times 0.12 \text{ mm}$
$\beta = 90.833 (5)^\circ$	

Data collection

Bruker SMART CCD area-detector diffractometer	23079 measured reflections
Absorption correction: multi-scan (SADABS in SAINT-NT; Bruker, 2002)	6234 independent reflections
$T_{\min} = 0.962, T_{\max} = 0.986$	3511 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.058$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.060$	379 parameters
$wR(F^2) = 0.140$	H-atom parameters constrained
$S = 1.12$	$\Delta\rho_{\text{max}} = 0.17 \text{ e \AA}^{-3}$
6234 reflections	$\Delta\rho_{\text{min}} = -0.18 \text{ e \AA}^{-3}$

All H atoms were found in a difference Fourier map; they were, however, treated differently. H atoms attached to C atoms were positioned at their expected locations and allowed to ride, with $C-H = 0.93-0.97 \text{ \AA}$ and $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(C)$. H atoms attached to N

Table 1

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N1-H1 \cdots N4^i$	0.85	2.06	2.903 (2)	170
$N6-H6A \cdots N3^{ii}$	0.85	2.21	3.059 (2)	176
$N6-H6B \cdots O1W^{iii}$	0.85	2.41	3.133 (2)	144
$N5-H5A \cdots O1W$	0.85	2.40	3.229 (2)	166
$N5-H5B \cdots O1W^{iv}$	0.85	2.34	3.145 (2)	159
$O1W-H1WA \cdots N2$	0.85	1.97	2.767 (2)	155
$O1W-H1WB \cdots N4^v$	0.85	2.54	3.387 (2)	177

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

Table 2

 $\pi-\pi$ contacts ($\text{\AA}, ^\circ$) for (I).

$Cg1$ is the centroid of the $N1/C1/N2/C2/C5$ ring and $Cg2$ is the centroid of the $N3/C3/C2/C5/N4/C4$ ring. CCD is the centre-to-centre distance (distance between ring centroids), IPD is the mean interplanar distance (distance from one plane to the neighbouring centroid) and SA is the mean slippage angle (angle subtended by the intercentroid vector to the plane normal); for details, see Janiak (2000).

Group 1/group 2	CCD (\AA)	IPD (\AA)	SA ($^\circ$)
$Cg1 \cdots Cg2^i$	3.501 (2)	3.30 (2)	19.5 (6)
$Cg2 \cdots Cg2^{ii}$	3.514 (1)	3.307 (1)	19.72 (1)

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

Table 3

Selected torsion angles ($^\circ$) for (II).

$C63-C13-C73-O23$	$-146.4 (2)$	$C33-C23-C83-C93$	$93.9 (3)$
$C23-C13-C73-O23$	$31.6 (3)$	$C13-C23-C83-C93$	$-85.3 (3)$
$C63-C13-C73-O13$	$33.1 (3)$	$C23-C83-C93-O33$	$-14.1 (3)$
$C23-C13-C73-O13$	$-148.9 (2)$	$C23-C83-C93-O43$	$166.6 (2)$

Table 4

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N61-H61A \cdots N42$	0.85	2.15	3.000 (3)	176
$N62-H62A \cdots N41$	0.85	2.17	3.016 (3)	177
$N51-H51B \cdots N22^i$	0.85	2.10	2.934 (3)	166
$N52-H52B \cdots N21^{ii}$	0.85	2.09	2.914 (3)	165
$O7W-H7WB \cdots O33$	0.85	1.92	2.722 (3)	156
$O7W-H7WA \cdots O4W^{iii}$	0.85	2.39	3.022 (4)	131
$O6W-H6WA \cdots O13^{iv}$	0.85	1.96	2.680 (3)	141
$O6W-H6WB \cdots O33^v$	0.85	2.30	2.872 (3)	125
$O5W-H5WB \cdots O6W$	0.85	1.82	2.667 (4)	173
$O5W-H5WA \cdots O1W^{vi}$	0.85	2.02	2.867 (4)	174
$O4W-H4WB \cdots O2W$	0.85	2.07	2.693 (4)	130
$O4W-H4WA \cdots O1W$	0.85	1.91	2.758 (4)	176
$O3W-H3WA \cdots O13$	0.85	1.90	2.748 (3)	178
$O3W-H3WB \cdots O43^{vii}$	0.85	2.01	2.847 (3)	170
$O2W-H2WA \cdots O3W$	0.85	1.93	2.748 (3)	163
$O2W-H2WB \cdots O5W^{viii}$	0.85	1.90	2.749 (4)	172
$O1W-H1WA \cdots O43$	0.85	1.94	2.741 (3)	156
$O1W-H1WB \cdots O5W$	0.85	2.04	2.829 (4)	154
$N62-H62B \cdots O23$	0.85	2.27	3.000 (3)	144
$N52-H52A \cdots O3W$	0.85	2.07	2.855 (3)	153
$N32-H32 \cdots O23$	0.85	1.84	2.662 (3)	162
$N12-H12 \cdots O4W^{ix}$	0.85	2.03	2.864 (3)	169
$N61-H61B \cdots O43^{ix}$	0.85	2.38	3.130 (3)	147
$N51-H51A \cdots O33^{ix}$	0.85	2.34	3.076 (3)	145
$N31-H31 \cdots O43^{ix}$	0.85	2.03	2.856 (3)	163
$N11-H11 \cdots O7W$	0.85	1.89	2.728 (3)	169

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

Table 5

π - π contacts (\AA , $^\circ$) for (II).

Cg_1 is the centroid of the N11/C11/N21/C21/C51 ring, Cg_2 of the N12/C12/N22/C22/C52 ring, Cg_3 of the N31/C31/C21/C51/N41/C41 ring, Cg_4 of the N32/C32/C22/C52/N42/C42 ring and Cg_5 of the C13/C23/C33/C43/C53/C63 ring. See Table 2 for definitions of CCD, IPD and SA.

Group 1/group 2	CCD (\AA)	IPD (\AA)	SA ($^\circ$)
$Cg_1 \cdots Cg_2^i$	3.693 (2)	3.35 (1)	24.7 (1)
$Cg_3 \cdots Cg_4^{ii}$	4.083 (2)	3.34 (1)	35.1 (3)
$Cg_5 \cdots Cg_5^{iii}$	4.184 (2)	3.74 (1)	26.52 (1)

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

and O atoms were initially refined with restrained distances to their hosts [$N-H = O-H = 0.85$ (1) \AA and water $H \cdots H = 1.35$ (1) \AA] and restricted to ride after convergence, with $U_{iso}(H) = 1.2U_{eq}(N)$ or $1.5U_{eq}(O)$.

For both compounds, data collection: *SMART-NT* (Bruker, 2001); cell refinement: *SAINT-NT* (Bruker, 2002); data reduction: *SAINT-NT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL-NT* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL-NT* and *PLATON* (Spek, 2009).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: DN3149). Services for accessing these data are described at the back of the journal.

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