

Supramolecular structures constructed by 3-(2-amino-6-chloropyrimidin-4-yl)-1,1-dimethylprop-2-yn-1-ol monohydrate and 3-[2-amino-6-(3-hydroxy-3,3-dimethylprop-1-yn-1-yl)pyrimidin-4-yl]-1,1-dimethylprop-2-yn-1-ol

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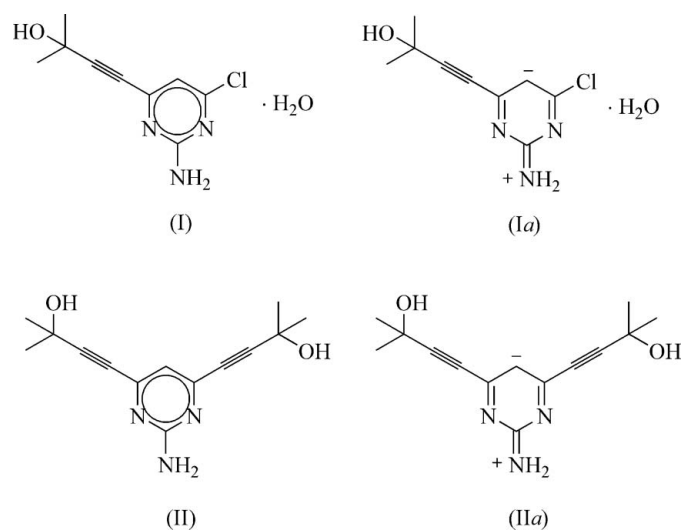
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The molecule of 3-(2-amino-6-chloropyrimidin-4-yl)-1,1-dimethylprop-2-yn-1-ol monohydrate, $C_9H_{10}ClN_3O \cdot H_2O$, (I), shows a very polarized molecular–electronic structure, while the polarization is slight for 3-[2-amino-6-(3-hydroxy-3,3-dimethylprop-1-yn-1-yl)pyrimidin-4-yl]-1,1-dimethylprop-2-yn-1-ol, $C_{14}H_{17}N_3O_2$, (II). In the supramolecular structure of (I), a combination of hard $N-H \cdots N$ hydrogen bonds and soft $C-H \cdots N$ hydrogen bonds creates a molecular column. Aromatic $\pi-\pi$ stackings between the pyrimidine rings stabilize the column with perpendicular and centroid–centroid distances of 3.283 (3) and 3.588 (1) Å, respectively. Short $Cl \cdots Cl$ contacts further link neighbouring molecular columns, creating a hydrophilic tube in which water molecules are fixed by various hydrogen bonds. In the packing of (II), a one-dimensional molecular chain is formed through several contacts involving hard $N-H \cdots O(N)$ and $O-H \cdots O(N)$ and soft $C-H \cdots O$ hydrogen bonds. Interchain $O-H \cdots O$ hydrogen bonds link the chains giving a two-dimensional stepped network. It is anticipated that study of the influence of hydrogen bonding on the patterns of base pairing and molecular packing in aminopyrimidine structures will shed significant light on nucleic acid structures as well as their functions.

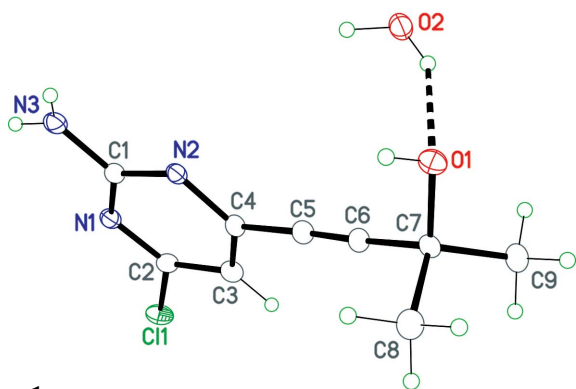
Comment

Aminopyrimidines have attracted considerable attention owing to their biological activities and molecular structures. In this group of compounds, 2-aminopyrimidines are of particular interest as adduct creators because of their potential ability to form stable hydrogen-bonded chains *via* their stereochemically associated amino groups and annular N atoms

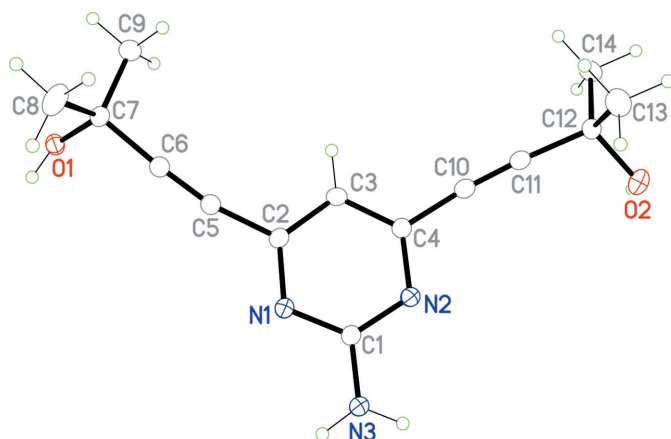
(Lynch *et al.*, 2000; Lynch & Jones, 2004). In the crystal structures of 2-aminopyrimidine derivatives that have been reported, the majority are modified by functional groups such as alkyl (Muthiah *et al.*, 2006), aryl (Fun *et al.*, 2006; Gallagher *et al.*, 2004), alkylamino (Lynch *et al.*, 2004; Quesada, Marchal *et al.*, 2002; Quesada *et al.*, 2004) and alkoxy (Glidewell *et al.*, 2002; Quesada, Low *et al.*, 2002). However, the acetylenyl moiety may be a good choice as the linker between 2-aminopyrimidine and some other hydrogen-bonding blocks since the low rotational barrier about the $sp-sp^2$ bond permits accessibility to favourable binding geometries. The crystal structures of the acetylenyl-group-bridged pyridine–aminopyrimidine compounds have been described using the pyridine moiety as an additional hydrogen-bond acceptor (Aakeröy *et al.*, 2005, 2007, 2009). Our goal is to design and synthesize acetylenyl-linker-bridged 2-aminopyrimidine derivatives with the OH group potentially acting as both hydrogen-bond acceptor and donor. Herein we report the crystal structures of two such 2-aminopyrimidine compounds, namely, 3-(2-amino-6-chloropyrimidin-4-yl)-1,1-dimethylprop-2-yn-1-ol monohydrate, (I), and 3-[2-amino-6-(3-hydroxy-3,3-dimethylprop-1-yn-1-yl)pyrimidin-4-yl]-1,1-dimethylprop-2-yn-1-ol, (II).



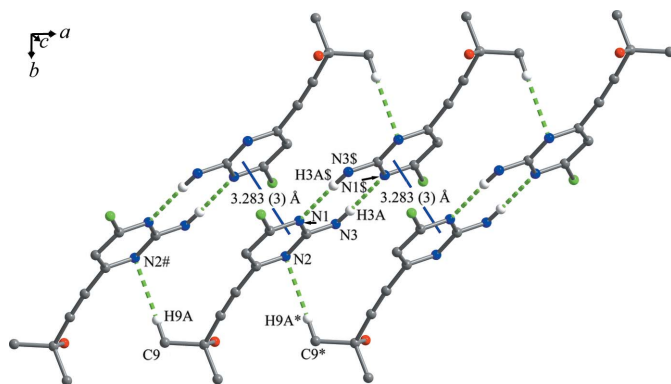
Compound (I) crystallizes in the space group $P2_1/n$ with one monoalkynyl-substituted aminopyrimidine molecule and one water molecule in the asymmetric unit (Fig. 1). The main molecule adopts a conformation in which the amino group, pyrimidine unit, Cl atom and triple bond are almost coplanar. The OH group is slightly twisted out of this plane, with the $O1-C7-C4-N2$ torsion angle being $-24.9(2)^\circ$. The H atom of the OH group in the main molecule and one H atom of the water solvent are each disordered over two positions with refined site-occupancy factors of 0.5. In one component, the OH group acts as a hydrogen-bond donor to the O atom of the water molecule through the intermolecular $O1-H1'A \cdots O2$ hydrogen bond (Table 1), while in the other component, the direction of the interaction is reversed so that the water molecule acts as the donor to give an $O2-H2A \cdots O1$ hydrogen bond. We attribute the disorder in these hydrogen-

**Figure 1**

The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level for non-H atoms and H atoms are shown as small spheres of arbitrary radii. For clarity, only one component of each of the disordered H atoms on O1 and O2 is shown. The dashed line represents a hydrogen bond between the main molecule and water.

**Figure 2**

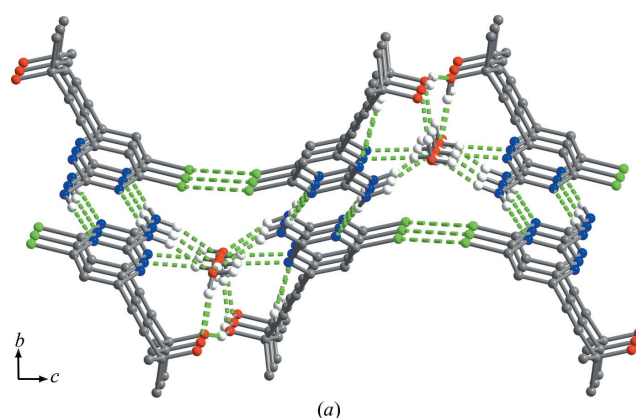
The molecular structure of (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level for non-H atoms and H atoms are shown as small spheres of arbitrary radii.

**Figure 3**

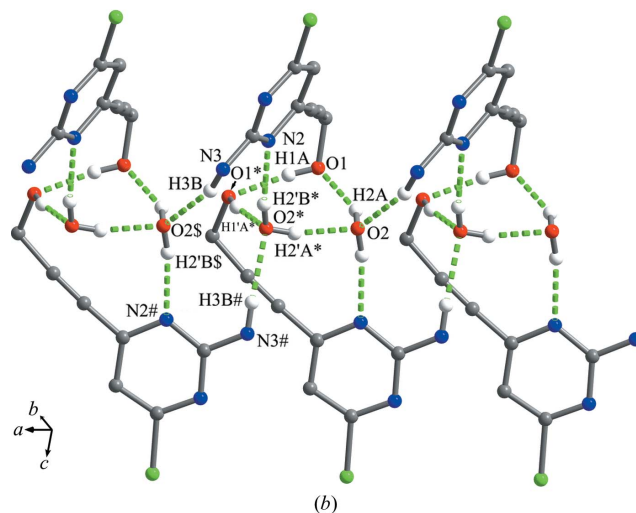
The molecular column of (I), consisting of two hydrogen-bonded one-dimensional chains in the reverse orientation, showing the perpendicular distances between inversion-related pyrimidine rings. H atoms not involved in the motifs and the water molecules have been omitted for clarity. Atoms marked with asterisks (*), hashes (#) and dollar signs (\$) are at the symmetry positions $(x + 1, y, z)$, $(x - 1, y, z)$ and $(-x + 3, -y, -z + 1)$, respectively.

bonding motifs to a frustration in the arrangement of H atoms within the extended structure, one result of which is that no site occupancy greater than 0.5 is possible without introducing H atoms at mutually incompatible positions.

Compound (II), a dialkynyl-substituted derivative, crystallizes in the space group $P\bar{1}$ with only one molecule in the asymmetric unit (Fig. 2). Similarly to (I), both OH groups are twisted to the same side of the approximate plane defined by the amino group and pyrimidine ring, with O1—C7—C2—N1 and O2—C12—C4—N2 torsion angles of $-57.66(16)$ and $24.00(14)^\circ$, respectively. Remarkably, the two triple bonds deviate from the approximate plane, with C2—C5—C6 and C4—C10—C11 angles of $169.57(16)$ and $171.94(17)^\circ$, respectively, which are smaller than those in (I) and in similar structures (Singelenberg & van Eijck, 1987; Pollagi *et al.*, 1994; Aakeröy *et al.*, 2005, 2007, 2009). This bend meets the stereochemical demand for dimer formation in the supramolecular structure.



(a)



(b)

Figure 4

(a) The hydrophilic tube of (I), viewed along the a axis. H atoms not involved in hydrogen bonding have been omitted for clarity. (b) Part of the hydrophilic tube, showing the $R_2^4(8)$ and $R_2^4(12)$ motifs; methyl groups and H atoms not involved in the hydrogen bonding have been omitted for clarity. Atoms marked with asterisks (*), hashes (#) and dollar signs (\$) are at the symmetry positions $(-x + \frac{3}{2}, y, -z + \frac{3}{2})$, $(-x + \frac{5}{2}, y, -z + \frac{3}{2})$ and $(x + 1, y, z)$, respectively. Dashed lines represent hydrogen bonds and Cl...Cl contacts.

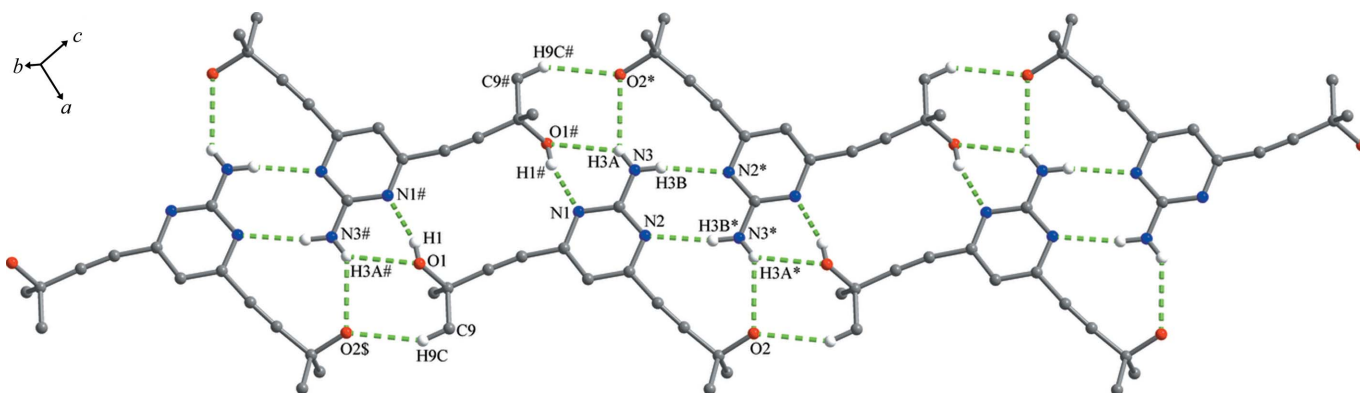


Figure 5

The hydrogen-bonded one-dimensional chain of (II), showing the supramolecular motifs; H atoms not involved have been omitted for clarity. Atoms marked with asterisks (*), hashes (#) and dollar signs (\$) are at the symmetry positions $(-x + 1, -y + 1, -z + 1)$, $(-x, -y + 2, -z)$ and $(x - 1, y + 1, z - 1)$, respectively.

Some intramolecular bond distances in (I) and (II) are found to be unusual when compared with the typical values (Allen *et al.*, 1987) for similar bond types. For (I), there is a clear distinction between the longer and the shorter C–N bonds of the pyrimidine ring (Table 2). The N1–C2, N3–C1 and C4–N2 bond distances are all short for their types. The extremely short N1–C2 bond [1.306 (3) Å] and the N3–C1 bond [1.324 (3) Å] illustrate the consequence of the electronic delocalization influenced by the Cl atom. These observations confirm that the charge-separated form (Ia) (see Scheme) is a dominant contributor to the overall molecular–electronic structure, as generally found for substituted 2-amino-5-nitrosopyrimidines (Quesada, Low *et al.*, 2002; Quesada, Marchal *et al.*, 2002; Quesada *et al.*, 2004). By contrast, when the Cl substituent is not present, the distance distinction among the C–N bonds in (II) is considerably less pronounced. This suggests that the polarized form (IIa) is only a minor contributor to the overall electronic structure. The triple bond lengths in (I) and (II) range from 1.193 (2) to 1.196 (4) Å and are in agreement with the values reported for similar structures (Singelenberg & van Eijck, 1987; Pollagi *et al.*, 1994; Aakeröy *et al.*, 2005, 2007, 2009).

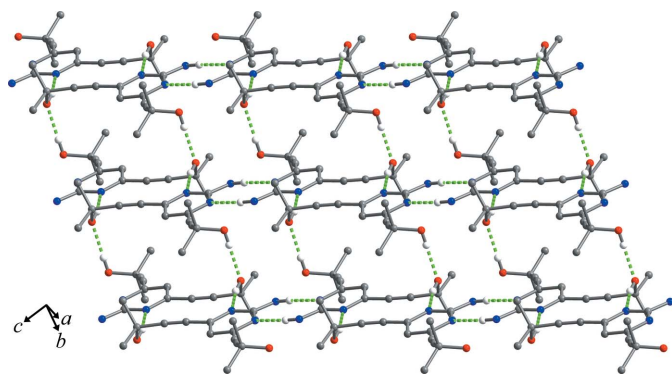


Figure 6

The two-dimensional stepped network of (II), formed via O–H...O hydrogen bonds, with H atoms not involved omitted for clarity.

In the supramolecular structure of (I), a hydrogen-bonded infinite chain is firstly formed by a combination of intermolecular C9–H9A...N2($x - 1, y, z$) hydrogen bonds (Table 1), which locally creates a $C(7)$ motif (Bernstein *et al.*, 1995) at each link in the chain (Fig. 3). Two adjacent such chains run in opposing directions and then generate a molecular column through a pair of centrosymmetrically related N–H...N hydrogen bonds between the amino groups as donors (N3–H3A) and the pyrimidine N atoms (N1) of neighbouring molecules, producing an eight-membered $R_2^2(8)$ ring motif. Additionally, π – π contacts (Tsuzuki *et al.*, 2002) occur between the parallel C1/N1/C2/C3/C4/N2 pyrimidine rings at (x, y, z) and $(-x + 2, -y, -z + 1)$. The perpendicular distance from the centroid of one ring plane to the other plane and the centroid–centroid distance are 3.283 (3) and 3.588 (1) Å, respectively. The aromatic π -stacking force is an important factor in the stabilization of the one-dimensional molecular column. Finally, a triangular tube is formed by short intercolumn Cl...Cl contacts and O–H1A...O1($-x + \frac{3}{2}, y, -z + \frac{3}{2}$) hydrogen bonds (Table 1 and Fig. 4a). The Cl1...Cl1($-x + \frac{5}{2}, y, -z + \frac{1}{2}$) distance [3.462 (1) Å] is some 0.04 Å shorter than the van der Waals separation based on a radius of 1.75 Å (Bondi, 1964), within the ranges discussed by Price *et al.* (1994) and Lommerse *et al.* (1996), and both C–Cl...Cl angles are 142.62 (9)°. This kind of arrangement results in the inner surface of the tube being more hydrophilic with the hydrophilic NH₂ groups, OH groups, N atoms of pyrimidine rings and Cl atoms all pointing inward. As a result, water molecules are sequentially fixed in this hydrophilic cavity by a combination of O–H...O, N–H...O and O–H...N hydrogen bonds (Table 1) between the water molecules and these hydrophilic moieties, alternately creating $R_4^4(8)$ and $R_4^4(12)$ ring motifs which cross at O atoms of the filled water molecules (Fig. 4b).

In the packing of (II), a number of hard N–H...O(N) and O–H...O(N) and soft C–H...O hydrogen bonds are also observed (Table 3). Firstly, a centrosymmetric dimer is formed by co-operative N–H...O and O–H...O hydrogen bonds between two neighbouring coplanar molecules (Fig. 5). In this dimer, the amino group (N3) acts as a double hydrogen-bond

donor, where the acceptors are the annular N atom (N2) and the hydroxy O atom (O2) in the molecule at $(-x + 1, -y + 1, -z + 1)$. The former interaction creates a centrosymmetric $R_2^2(8)$ motif (Bernstein *et al.*, 1995), while the latter interaction creates two inversion-related $R_2^2(9)$ motifs. These three rings are fused, thus producing a $R_2^2(18)$ motif around the circumference. Next, each dimer is linked to its neighbours to produce a one-dimensional molecular chain by a combination of interdimer $N3-H3A \cdots O1(-x, -y + 2, -z)$, $O1-H1 \cdots N1(-x, -y + 2, -z)$ and $C9-H9C \cdots O2(x - 1, y + 1, z - 1)$ hydrogen bonds, locally generating one $R_2^2(14)$ and two distinct $R_2^2(6)$ motifs. One $R_2^2(6)$ motif involves two molecules, while the other involves three molecules. Finally, the inter-chain $O2-H2 \cdots O1(-x + 1, -y + 1, -z)$ hydrogen bonds associate these chains into a two-dimensional stepped network that extends parallel to the (110) plane (Fig. 6).

Experimental

2-Methylbut-3-yn-2-ol (0.293 ml, 3.00 mmol), Et_3N (1 ml) and CH_3CN (5 ml) were added to a nitrogen-purged flask containing 2-amino-4,6-dichloropyrimidine (0.164 g, 1.00 mmol), CuI (0.019 g, 0.10 mmol), 5% Pd/C (0.106 g, 0.05 mmol), PPh_3 (0.026 g, 0.10 mmol) and the mixture was stirred at 313 K for 6 h. The resulting mixture was cooled to room temperature and filtered. After removal of the solvent under reduced pressure, the residue was dissolved in $EtOAc$ and washed with brine. The organic layer was dried over anhydrous $MgSO_4$. The solvent was evaporated *in vacuo* and the crude products were chromatographed on a silica gel column ($EtOAc$ /petroleum ether = 3:2) to give (I) (water-free) as a colourless solid (yield 35%, $R_F = 0.6$, m.p. 431 K) and (II) as a colourless solid (yield 42%, $R_F = 0.3$, m.p. 460 K). 1H NMR (300 MHz, $DMSO-d_6$) for (I) (water-free): δ 7.24 (s, 2H), 6.67 (s, 1H), 5.64 (s, 1H), 1.43 (s, 6H); for (II): δ 6.87 (s, 2H), 6.55 (s, 1H), 5.60 (s, 2H), 1.43 (s, 12H). Single crystals of (I) and (II) suitable for X-ray diffraction analysis were obtained by slow vapour diffusion of pentane into a solution of (I) (no water) in $EtOAc$ at 298 K, and slow evaporation of a solution of (II) in $EtOAc$ at 298 K.

Compound (I)

Crystal data

$C_9H_{10}ClN_3O \cdot H_2O$	$V = 1119.0$ (5) \AA^3
$M_r = 229.67$	$Z = 4$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 6.0021$ (15) \AA	$\mu = 0.33$ mm^{-1}
$b = 11.003$ (3) \AA	$T = 173$ K
$c = 16.960$ (4) \AA	$0.40 \times 0.16 \times 0.12$ mm
$\beta = 92.566$ (4) $^\circ$	

Data collection

Bruker SMART CCD area-detector diffractometer	5489 measured reflections
Absorption correction: multi-scan (<i>SADABS</i> ; Bruker, 1999)	1988 independent reflections
$T_{\min} = 0.881$, $T_{\max} = 0.962$	1767 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.030$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.052$	139 parameters
$wR(F^2) = 0.115$	H-atom parameters constrained
$S = 1.21$	$\Delta\rho_{\text{max}} = 0.27$ e \AA^{-3}
1988 reflections	$\Delta\rho_{\text{min}} = -0.28$ e \AA^{-3}

Table 1

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$O1-H1A \cdots O1^i$	0.85	1.88	2.708 (4)	164
$O1-H1'A \cdots O2$	0.85	1.95	2.791 (3)	171
$O2-H2A \cdots O1$	0.85	2.02	2.791 (3)	152
$O2-H2'A \cdots O2^i$	0.85	1.97	2.803 (4)	165
$O2-H2'B \cdots N2^j$	0.85	2.09	2.933 (3)	171
$C9-H9A \cdots N2^{ii}$	0.98	2.55	3.462 (3)	156
$N3-H3A \cdots N1^{iii}$	0.88	2.19	3.069 (3)	177
$N3-H3B \cdots O2^{iv}$	0.88	2.14	2.986 (3)	163

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

Table 2

Selected bond lengths (\AA) for (I) and (II).

	(I)	(II)
$N1-C2$	1.306 (3)	1.3410 (19)
$N2-C1$	1.352 (3)	1.3487 (19)
$N3-C1$	1.325 (3)	1.333 (2)
$C1-N1$	1.358 (3)	1.3573 (19)
$C2-C3$	1.378 (4)	1.383 (2)
$C3-C4$	1.381 (3)	1.385 (2)
$C4-N2$	1.342 (3)	1.3346 (19)

Table 3

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N3-H3B \cdots N2^i$	0.87	2.26	3.1207 (18)	170
$N3-H3A \cdots O2^j$	0.86	2.61	3.3390 (19)	143
$N3-H3A \cdots O1^{ii}$	0.86	2.58	3.2705 (17)	137
$O1-H1 \cdots N1^{iii}$	0.82	1.99	2.7808 (16)	163
$C9-H9C \cdots O2^{iii}$	0.96	2.56	3.4167 (18)	149
$O2-H2 \cdots O1^{iv}$	0.82	1.95	2.7710 (16)	176

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

Compound (II)

Crystal data

$C_{14}H_{17}N_3O_2$	$\gamma = 73.104$ (3) $^\circ$
$M_r = 259.31$	$V = 719.0$ (3) \AA^3
Triclinic, $P\bar{1}$	$Z = 2$
$a = 7.9966$ (17) \AA	Mo $K\alpha$ radiation
$b = 9.737$ (2) \AA	$\mu = 0.08$ mm^{-1}
$c = 9.833$ (2) \AA	$T = 173$ K
$\alpha = 78.976$ (3) $^\circ$	$0.38 \times 0.32 \times 0.19$ mm
$\beta = 86.217$ (3) $^\circ$	

Data collection

Bruker SMART CCD area-detector diffractometer	3832 measured reflections
Absorption correction: multi-scan (<i>SADABS</i> ; Bruker, 1999)	2644 independent reflections
$T_{\min} = 0.969$, $T_{\max} = 0.985$	2316 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.016$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$	178 parameters
$wR(F^2) = 0.112$	H-atom parameters constrained
$S = 1.02$	$\Delta\rho_{\text{max}} = 0.16$ e \AA^{-3}
2644 reflections	$\Delta\rho_{\text{min}} = -0.23$ e \AA^{-3}

In the absence of significant anomalous scattering effects, Friedel pairs were averaged. For (I), H atoms attached to C atoms were

included in calculated positions and refined as riding [C–H = 0.98 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ (methyl); C–H = 0.95 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ (aromatic)]. H atoms attached to N atoms were initially refined with restrained distances to their hosts [N–H = 0.88 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$]. The H atoms of the water molecule and the OH group were located in a difference map and subsequently refined with restraints of O–H = 0.85 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{O})$. The water molecule is rotationally disordered, such that one of the H atoms has full occupancy, while the other is disordered over two sites, each of half occupancy. This model was developed by including symmetry considerations which limit the site occupancy of each disordered H-atom component to 0.5. The H atom of the OH group is also disordered over two positions of equal occupancy. For (II), H atoms attached to C atoms were included in calculated positions and refined as riding [C–H = 0.96 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ (methyl); C–H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ (aromatic)]. H atoms attached to N and O atoms were initially refined with restrained distances to their hosts [N–H = 0.86 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$; O–H = 0.82 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$].

For both compounds, data collection: *SMART* (Bruker, 1999); cell refinement: *SMART*; data reduction: *SAINTE* (Bruker, 1999); 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: *SHELXTL*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: YP3004). Services for accessing these data are described at the back of the journal.

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