

## Hydrate frameworks involving the pyridazino[4,5-*d*]pyridazine unit as a multiple hydrogen-bond acceptor

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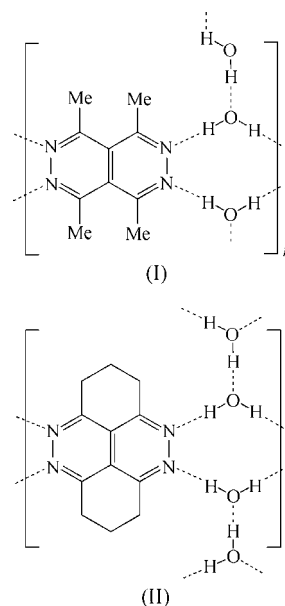
1,4,5,8-Tetramethylpyridazino[4,5-*d*]pyridazine trihydrate, C<sub>10</sub>H<sub>12</sub>N<sub>4</sub>·3H<sub>2</sub>O, (I), and 1,2,3,6,7,8-hexahydrocinnolino[5,4,3-*cde*]cinnoline tetrahydrate, C<sub>12</sub>H<sub>12</sub>N<sub>4</sub>·4H<sub>2</sub>O, (II), exhibit exceptional functionality of the condensed N<sub>4</sub>-heteroaromatic frame as a symmetric acceptor of four hydrogen bonds [N···O = 2.843 (2)–2.8716 (10) Å]. Thus, all the N atoms of the electron-deficient and highly  $\pi$ -acidic polynitrogen heterocycles function as lone-pair donors. In (I), all the molecular components lie on or across special positions; the site symmetry is  $2/m$  for the organic and  $m2m$  and  $m$  for the two water molecules. In (II), the organic polycycle lies across a crystallographic inversion center. Both structures involve a hydrogen-bonded centrosymmetric water–pyridazine dimer as the basic supramolecular unit, which is integrated into two-dimensional [in (I)] and three-dimensional [in (II)] hydrate frameworks by hydrogen bonding with the additional water molecules [O···O = 2.744 (2)–2.8827 (19) Å]. The hydrate connectivity exists in the form of an (H<sub>2</sub>O)<sub>3</sub> trimer in (I) and as a one-dimensional zigzag (H<sub>2</sub>O)<sub>*n*</sub> chain in (II).

### Comment

Heteroaromatic systems of diazines, triazines and 1,2,4,5-tetrazine provide multiple N-atom sites for sustaining coordination to metal centers and interacting with suitable hydrogen-bond donating groups. Both these types of interactions are applicable to the design of complex frameworks that could incorporate the azine module as an origin of the net connectivity. However, unlike the prototypal single N-atom donor pyridine (which is efficient either as a ligand in coordination chemistry or as an acceptor of hydrogen bonding), the nitrogen-rich electron-deficient azines are very weak lone-pair donors and their potential for crystal design has scarcely been explored. In particular, the multiple hydrogen-bond

acceptor function is uncommon even for pyridazine, a relatively basic diazine, and has been observed only in the dihydrate of pyridazine-3,6-dicarboxylic acid (Sueur *et al.*, 1987), in pyridazine-3,6-dimethanol (Abraham *et al.*, 1988) and in the adduct with substituted silanol (Ruud *et al.*, 1991), while the only precedent for multiple hydrogen bonding of tetrazine is self-complementary interactions in 3,6-diamino-1,2,4,5-tetrazine, with very long N–H···N separations of 3.09 Å (Krieger *et al.*, 1987).

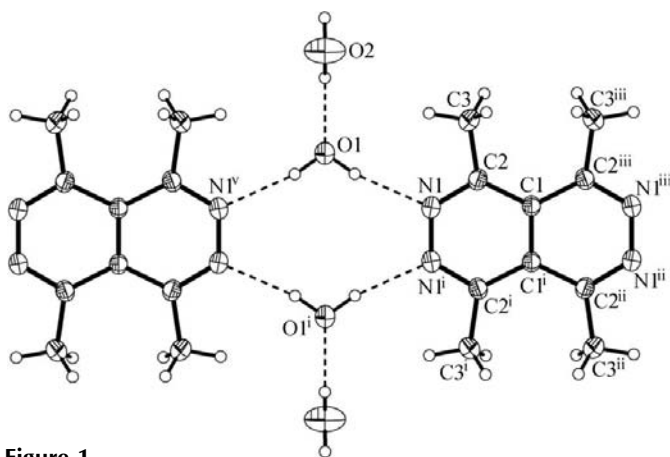
A more efficient lone-pair donating ability may be anticipated for polycyclic species combining several azine functions, as provided by pyridazino[4,5-*d*]pyridazines, a system of two *d*-edge-sharing pyridazine cycles. Although annelation of an N-heteroaromatic ring decreases the energy of the lowest unoccupied molecular orbital (LUMO) by a similar degree to the introduction of an electron-withdrawing substituent (Haider, 1991), the condensed pyridazines retain relatively efficient donor properties and are well suited for the preparation of coordination polymers (Gural'skiy *et al.*, 2006; Solntsev *et al.*, 2004). Even more rich and versatile applications for such a paradigmatic molecular building block may be found in the realm of hydrogen-bonded solids, and it is especially interesting to explore whether pyridazino[4,5-*d*]pyridazines are able to sustain multiple interactions for generation of hydrogen-bonded architecture. In this context, we have examined two closely related pyridazino[4,5-*d*]pyridazine representatives, which readily form hydrates, namely 1,4,5,8-tetramethylpyridazino[4,5-*d*]pyridazine trihydrate, (I), and 1,2,3,6,7,8-hexahydrocinnolino[5,4,3-*cde*]cinnoline tetrahydrate, (II), and we report their structures here.



The composition of (I) was previously reported as  $L \cdot 2H_2O$  on the basis of microanalysis data (Adembri *et al.*, 1970). Each of the crystallographically independent molecules, namely one molecule of the condensed heterocycle and two water molecules, lie on or across special positions. The organic molecules lie across sites of  $2/m$  symmetry, which implies that two atoms of the shared edge [C1–C1<sup>1</sup>; symmetry code: (i)  $x, -y+1, -z$ ]

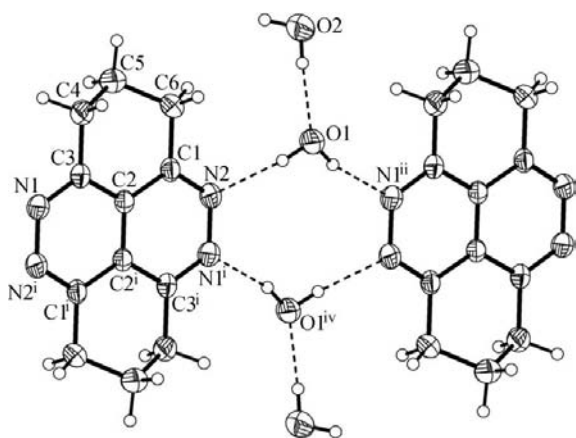
are related by inversion and lie in the crystallographic mirror plane. The site symmetry of O2 is  $m2m$ , with the O and H atoms lying in a mirror plane and the pair of H atoms related by a mirror plane perpendicular to the first mirror plane. The second water molecule (O1) lies across a crystallographic mirror plane at  $x = 0$ . The asymmetric unit of (II) comprises two water molecules and a half-molecule of the organic component lying across a center of inversion.

The basic pattern of pyridazine–water interactions is similar for both structures; the components complement each other as the double donors and acceptors of hydrogen bonding and afford centrosymmetric aqua–pyridazine dimers (Figs. 1 and 2). The corresponding N···O separations are similar [2.8716 (10) Å for (I), and 2.843 (2) and 2.870 (2) Å for (II); Tables 2 and 4], and are characteristic for O–H···N hydrogen bonding and only slightly longer than the values for pyridazine interacting with the much more acidic 1,3-dihydroxy-1,1,3,3-tetraphenyldisiloxane [N···O = 2.751 (3) Å; Ruud *et al.*, 1991].



**Figure 1**

The structure of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. Dashed lines indicate hydrogen bonds. [Symmetry codes: (i)  $x, -y + 1, -z$ ; (ii)  $-x + 1, -y + 1, -z$ ; (iii)  $-x + 1, y, z$ ; (v)  $-x, y, z$ .]

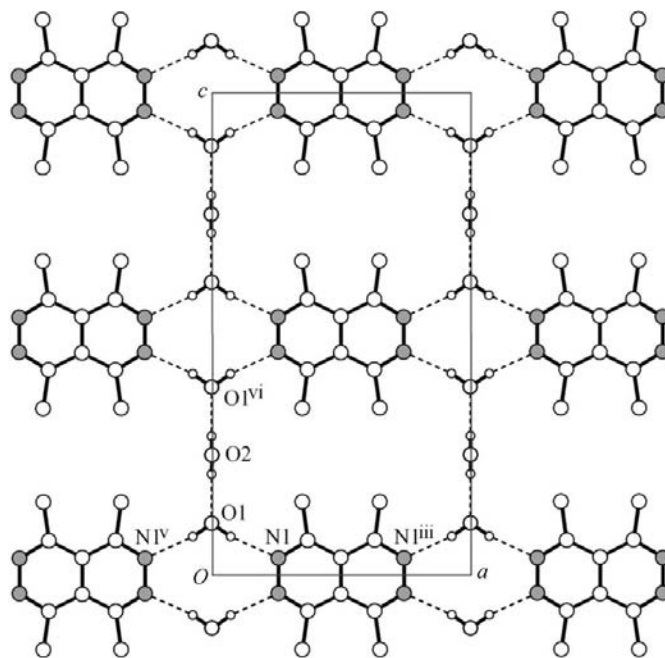


**Figure 2**

The structure of (II), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 40% probability level and H atoms are shown as small spheres of arbitrary radii. Dashed lines indicate hydrogen bonds. [Symmetry codes: (i)  $-x + 1, -y + 1, -z + 2$ ; (ii)  $x + 1, y, z - 1$ ; (iv)  $-x + 2, -y + 1, -z + 1$ .]

Pyridazine-3,6-dicarboxylic acid dihydrate (Sueur *et al.*, 1987), one of the few precedents for the double hydrogen bonding of water molecules and pyridazine, has comparable N···O separations [2.896 (3) Å]. In (I) and (II), each of the N atoms of the bicyclic frame accepts hydrogen bonding; such tetrafunctional behavior of the pyridazino[4,5-*d*]pyridazine unit is an unprecedented feature, suggesting a significant  $\sigma$ -donor ability towards multiple electrophilic centers. The latter is worth noting in view of the similarity of pyridazino[4,5-*d*]pyridazines and 1,2,4,5-tetrazines. Both are very electron deficient (Haider, 1991) and commonly function rather as strong  $\pi$ -acids, sustaining very short interactions between the  $\pi$  clouds and negatively polarized atoms (Gural'skiy *et al.*, 2006).

Hydrogen-bonding interactions in (I) and (II) result in the same principal motif in the form of aqua–organic  $\{L \cdot 2H_2O\}_n$  ribbons, while the mode of further interconnection is sensitive to the molecular shape. In (I), neighboring ribbons (symmetry code:  $-x, -y + 1, z + \frac{1}{2}$ ) are linked by the remaining water molecules, forming pairs of equivalent hydrogen bonds [O2–H···O1 = 2.7881 (15) Å], into corrugated layers parallel to the *ac* plane (Fig. 3). Thus, the water–water linkage itself is very simple – it exists as an  $(H_2O)_3$  trimer. The entire hydrogen-bonded topology may be regarded as a planar (4,4)-net involving the aqua–pyridazine dimers as the four-connected net nodes and the organic frameworks and water molecules (O2) as the links. The layers are stacked in such a way that atom O2 forms contacts with four methyl groups from an adjacent layer. These C3–H3C···O2<sup>iv</sup> [symmetry code: (iv)  $x + \frac{1}{2}, y + \frac{1}{2}, z$ ] interactions reflect very weak, but directional,

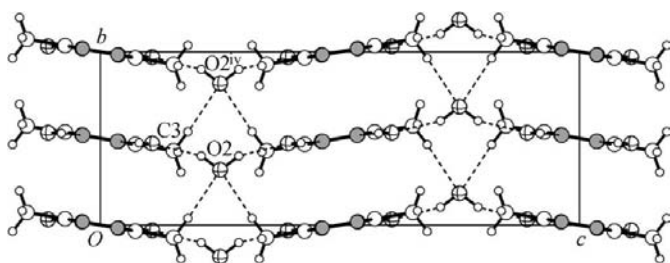


**Figure 3**

A projection of the structure of (I) on to the *ac* plane, showing the principal hydrogen-bonding interactions as dashed lines, and showing the trimeric  $(H_2O)_3$  aqua ensembles and diaqua–organic ribbons running along the *a* direction. N atoms are shaded gray. [Symmetry codes: (iii)  $-x + 1, y, z$ ; (vi)  $-x, y, -z + \frac{1}{2}$ .]

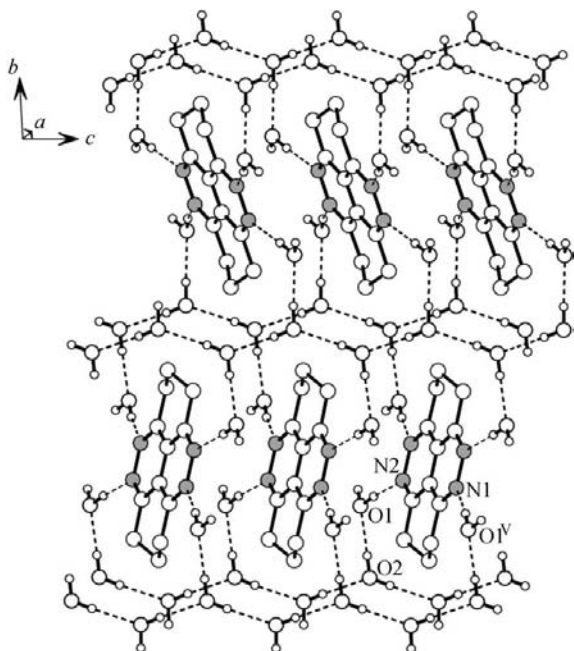
hydrogen bonding (Desiraju & Steiner, 1999), with a C—H···O angle of 175 (1)° (Fig. 4).

Considering the shape-complementary alignment of the components in (I) (Fig. 3), any further substitution into the organic framework will result in interference, and therefore substitution may be viewed as a tool for the modification of the pattern. This logic can be applied to the related structure of (II), which adopts a more complex three-dimensional framework (Figs. 5 and 6). The diaqua-organic ribbons are packed on top of one another (as depicted in Fig. 6 by molecules represented with filled and unfilled bonds), and these stacks are crosslinked by extended aqua chains running along the *c* direction. Thus, the typical hydrogen bonds O2—H4W···O2<sup>iii</sup> [2.8827 (19) Å; symmetry code: (iii)  $x, -y + \frac{1}{2}, z + \frac{1}{2}$ ] arrange the molecules into zigzag chains, similar to the



**Figure 4**

A projection of the structure of (I) on to the *bc* plane, showing the packing of hydrogen-bonded corrugated layers and directional C—H···O interactions that occur between successive layers. Dashed lines indicate hydrogen bonds, N atoms are shaded gray and O atoms are shown as crossed spheres. [Symmetry code: (iv)  $x + \frac{1}{2}, y + \frac{1}{2}, z$ .]

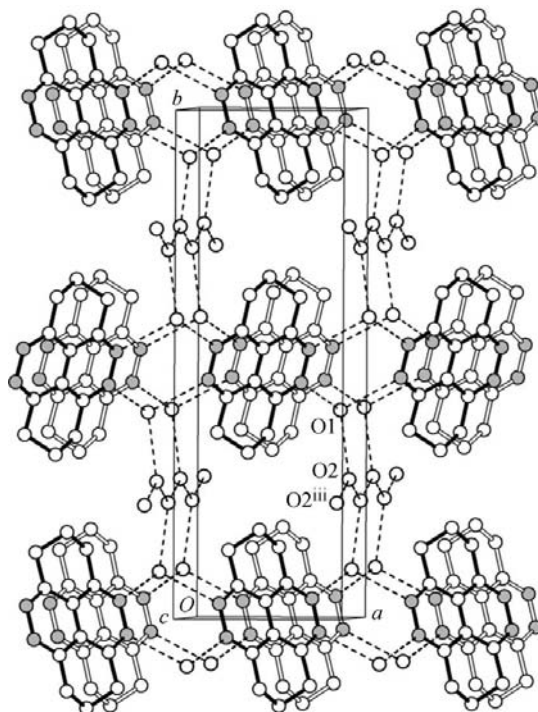


**Figure 5**

A fragment of the structure of (II), demonstrating the function of the organic molecules as symmetric acceptors of four hydrogen bonds (indicated as dashed lines) and the mode of their incorporation with the hydrate linkage. N atoms are shaded gray. [Symmetry code: (v)  $x - 1, y, z + 1$ .]

one-dimensional hydrogen-bonded —OH···(OH)<sub>*n*</sub>— motif in pentafluorophenol (Das *et al.*, 2006), while a second O2H function provides bonding to atom O1 in the aqua-pyridazine dimer [O2—H3W···O1 = 2.744 (2) Å]. Therefore, the entire topology is best described as a uniform 3,4-heterocoordinated net (three-letter notation 'f<sub>3</sub>c'; Bonneau *et al.*, 2004), which includes O2 water molecules as three- and pyridazine-aqua dimers as four-connected nodes (in a 2:1 proportion).

For both compounds, the geometry of the organic molecules is consistent with the structure of unsubstituted pyridazino-[4,5-*d*]pyridazine (Sabelli *et al.*, 1969), and it is strongly suggestive of a significant contribution of the bis-azine (—C=N—N=C—) resonance structure with relatively long pyridazine *c* and *e* bonds [C—C = 1.418 (3)–1.4413 (10) Å] and short C—N bonds [1.3137 (12)–1.315 (2) Å; Tables 1 and 3]. The latter are shorter than the C—N bonds [1.334 (7) Å] in the highly conjugated 1,2,4,5-tetrazine (Bertinotti *et al.*, 1956). The distortion of the molecular framework in the structure of (I) and a certain elongation of the pyridazine *d* and *c* bonds [1.409 (2) and 1.4413 (10) Å] are influenced by an evident steric repulsion between methyl groups in *peri* positions, as occurs for 1,4,5,8-tetramethylnaphthalene (Shiner *et al.*, 1984). This steric interaction is reflected in the angles adopted by the methyl groups [N1—C2—C3 = 113.72 (8)° and C1—C2—C3 = 125.47 (9)°] in order to avoid contacts between these groups that would otherwise be even shorter than the observed values



**Figure 6**

A packing diagram for the structure of (II), showing the mutual orientation of the diaqua-organic ribbons and one-dimensional (H<sub>2</sub>O)<sub>*n*</sub> chains, which results in a three-dimensional framework. Methylene H atoms have been omitted for clarity and N atoms are shaded gray. The molecules represented with filled and unfilled bonds correspond to two successive diaqua-organic ribbons. [Symmetry code: (iii)  $x, -y + \frac{1}{2}, z + \frac{1}{2}$ .]

[C3...C3<sup>iii</sup> = 2.972 (2) Å; symmetry code: (iii)  $-x + 1, y, z$ ]. At the same time, the presence of a methylene linker in (II) minimizes such intramolecular strains. Despite this strained geometry, the aromatic part in (I) retains a completely planar structure, unlike the appreciably twisted frame of octamethylnaphthalene (Sim, 1982). The latter possibility for decreasing the steric interactions could be especially relevant for  $\mu_4$ -1,4,5,8-tetramethylpyridazino[4,5-*d*]pyridazine in metal complexes, leading to a noncoplanar disposition of four metal ions.

In conclusion, the pyridazino[4,5-*d*]pyridazine tectons reveal potential as fourfold acceptors of hydrogen bonding. The system suggests a new paradigm for polyfunctional building blocks, which may find wider applications for the development of multicomponent hydrogen-bonded frameworks.

### Experimental

1,4,5,8-Tetramethylpyridazino[4,5-*d*]pyridazine was synthesized from tetraacetylene according to the method described by Adembri *et al.* (1970) and crystallized as a trihydrate, (I), from aqueous ethanol (95%). 1,2,3,6,7,8-Hexahydrocinnolino[5,4,3-*cde*]cinnoline was prepared by condensation of cyclohexane-1,3-dione and hydrazine (Stille & Ertz, 1964). Recrystallization from aqueous ethanol (95%) yielded an anhydrous material, while slow evaporation from a solution in aqueous methanol (90%) afforded the tetrahydrate (II).

#### Compound (I)

##### Crystal data

|   |   |
|---|---|
| C <sub>10</sub> H <sub>12</sub> N <sub>4</sub> ·3H <sub>2</sub> O | $V = 1209.09 (18) \text{ \AA}^3$          |
| $M_r = 242.28$  | $Z = 4$                                   |
| Orthorhombic, <i>Cmcm</i>   | Mo $K\alpha$ radiation                    |
| $a = 9.8804 (8) \text{ \AA}$                                      | $\mu = 0.10 \text{ mm}^{-1}$              |
| $b = 6.6540 (7) \text{ \AA}$                                      | $T = 213 (2) \text{ K}$                   |
| $c = 18.3908 (12) \text{ \AA}$                                    | $0.29 \times 0.22 \times 0.22 \text{ mm}$ |

##### Data collection

|                             |                                       |
|-----------------------------|---------------------------------------|
| Stoe IPDS diffractometer    | 778 reflections with $I > 2\sigma(I)$ |
| 4691 measured reflections   | $R_{\text{int}} = 0.024$              |
| 828 independent reflections |                                       |

##### Refinement

|                                 |  |
|---------------------------------|--|
| $R[F^2 > 2\sigma(F^2)] = 0.040$ | 63 parameters  |
| $wR(F^2) = 0.109$               | All H-atom parameters refined                        |
| $S = 1.13$                      | $\Delta\rho_{\text{max}} = 0.32 \text{ e \AA}^{-3}$  |
| 828 reflections                 | $\Delta\rho_{\text{min}} = -0.22 \text{ e \AA}^{-3}$ |

**Table 1**

Selected geometric parameters (Å, °) for (I).

| N1—C2                       | 1.3137 (12) | C1—C2                       | 1.4413 (10) |
|-----------------------------|-------------|-----------------------------|-------------|
| N1—N1 <sup>i</sup>          | 1.3596 (16) | C2—C3                       | 1.4977 (14) |
| C1—C1 <sup>ii</sup>         | 1.409 (2)   |                             |             |
| C2—N1—N1 <sup>i</sup>       | 121.69 (5)  | N1—C2—C3                    | 113.72 (8)  |
| C1 <sup>ii</sup> —C1—C2     | 117.50 (6)  | C1—C2—C3                    | 125.47 (9)  |
| N1—C2—C1                    | 120.81 (9)  |                             |             |
| C2 <sup>iii</sup> —C1—C2—N1 | 179.95 (8)  | C2 <sup>iii</sup> —C1—C2—C3 | 0.1 (2)     |

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

**Table 2**

Hydrogen-bond geometry (Å, °) for (I).

| <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> |
|---------------------------|-------------|---------------|-----------------------|-------------------------|
| O1—H1...N1                | 0.88 (2)    | 2.00 (2)      | 2.8716 (10)           | 169 (2)                 |
| O2—H2...O1                | 0.90 (3)    | 1.94 (3)      | 2.7881 (15)           | 157 (2)                 |
| C3—H3C...O2 <sup>iv</sup> | 0.986 (17)  | 2.50 (2)      | 3.480 (2)             | 175 (1)                 |

Symmetry code: (iv)  $x + \frac{1}{2}, y + \frac{1}{2}, z$ .

#### Compound (II)

##### Crystal data

|   |   |
|---|---|
| C <sub>12</sub> H <sub>12</sub> N <sub>4</sub> ·4H <sub>2</sub> O | $V = 703.7 (2) \text{ \AA}^3$             |
| $M_r = 284.32$  | $Z = 2$                                   |
| Monoclinic, <i>P2<sub>1</sub>/c</i>                               | Mo $K\alpha$ radiation                    |
| $a = 6.6745 (14) \text{ \AA}$                                     | $\mu = 0.10 \text{ mm}^{-1}$              |
| $b = 20.028 (4) \text{ \AA}$                                      | $T = 296 (2) \text{ K}$                   |
| $c = 5.4860 (11) \text{ \AA}$                                     | $0.25 \times 0.20 \times 0.20 \text{ mm}$ |
| $\beta = 106.352 (3)^\circ$                                       |   |

##### Data collection

|   |                                       |
|---|---------------------------------------|
| Siemens SMART CCD area-detector diffractometer              | 2866 measured reflections             |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1996) | 1438 independent reflections          |
| $T_{\text{min}} = 0.961, T_{\text{max}} = 0.980$            | 911 reflections with $I > 2\sigma(I)$ |
|   | $R_{\text{int}} = 0.028$              |

##### Refinement

|                                 |  |
|---------------------------------|--|
| $R[F^2 > 2\sigma(F^2)] = 0.049$ | 92 parameters  |
| $wR(F^2) = 0.139$               | H-atom parameters constrained                        |
| $S = 1.05$                      | $\Delta\rho_{\text{max}} = 0.37 \text{ e \AA}^{-3}$  |
| 1438 reflections                | $\Delta\rho_{\text{min}} = -0.18 \text{ e \AA}^{-3}$ |

**Table 3**

Selected geometric parameters (Å, °) for (II).

| N1—C3                    | 1.315 (2)   | C1—C6              | 1.491 (3)   |
|--------------------------|-------------|--------------------|-------------|
| N1—N2 <sup>i</sup>       | 1.374 (2)   | C2—C2 <sup>i</sup> | 1.373 (4)   |
| N2—C1                    | 1.315 (2)   | C2—C3              | 1.418 (3)   |
| C1—C2                    | 1.421 (3)   | C3—C4              | 1.488 (3)   |
| C3—N1—N2 <sup>i</sup>    | 120.86 (16) | C2—C1—C6           | 118.97 (18) |
| N2—C1—C2                 | 120.59 (19) | C3—C2—C1           | 123.07 (18) |
| N2—C1—C6                 | 120.44 (18) | N1—C3—C2           | 120.61 (18) |
| C6—C1—C2—C2 <sup>i</sup> | 178.9 (2)   | C6—C1—C2—C3        | -0.7 (3)    |

Symmetry code: (i)  $-x + 1, -y + 1, -z + 2$ .

**Table 4**

Hydrogen-bond geometry (Å, °) for (II).

| <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> |
|----------------------------|-------------|---------------|-----------------------|-------------------------|
| O1—H1W...N2                | 0.86        | 1.99          | 2.843 (2)             | 173                     |
| O1—H2W...N1 <sup>ii</sup>  | 0.86        | 2.04          | 2.870 (2)             | 164                     |
| O2—H3W...O1                | 0.85        | 1.89          | 2.744 (2)             | 175                     |
| O2—H4W...O2 <sup>iii</sup> | 0.86        | 2.02          | 2.8827 (19)           | 178                     |

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

For (I), all H atoms were found in intermediate difference Fourier maps and were refined fully with isotropic displacement parameters

[C—H = 0.960 (16)–0.986 (17) Å]. For (II), the methylene H atoms were treated as riding in geometrically idealized positions, with C—H distances of 0.97 Å and  $U_{\text{iso}}(\text{H})$  values of  $1.2U_{\text{eq}}(\text{C})$ . Water H atoms were located in difference maps and their coordinates were then fixed [with  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$ ], giving all O—H distances in the range 0.85–0.86 Å.

For (I), data collection: *IPDS Software* (Stoe & Cie, 2000); cell refinement: *IPDS Software*; data reduction: *IPDS Software*. For (II), data collection: *SMART-NT* (Bruker, 1998); cell refinement: *SAINT-NT* (Bruker, 1999); data reduction: *SAINT-NT*. For both compounds, program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *WinGX* (Version 1.70.01; Farrugia, 1999).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM3049). Services for accessing these data are described at the back of the journal.

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