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Charge-assisted hydrogen-bonded supramolecular networks in acetoguanaminium hydrogen phthalate, acetoguanaminium hydrogen maleate and acetoguanaminium 3-hydroxypicolinate monohydrate

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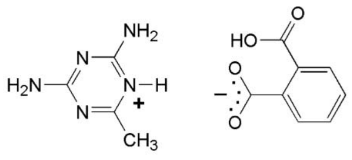
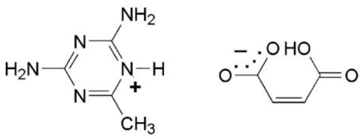
In 2,4-diamino-6-methyl-1,3,5-triazin-1-ium (acetoguanaminium) hydrogen phthalate, $C_4H_8N_5^+ \cdot C_8H_5O_4^-$, (I), acetoguanaminium hydrogen maleate, $C_4H_8N_5^+ \cdot C_4H_3O_4^-$, (II), and acetoguanaminium 3-hydroxypicolinate monohydrate, $C_4H_8N_5^+ \cdot C_6H_4NO_3^- \cdot H_2O$, (III), the acetoguanaminium cations interact with the carboxylate groups of the corresponding anions *via* a pair of nearly parallel N—H···O hydrogen bonds, forming $R_2^2(8)$ ring motifs. In (II) and (III), N—H···N base-pairing is observed, while there is none in (I). In (II), a series of fused $R_3^2(8)$, $R_2^2(8)$ and $R_3^2(8)$ hydrogen-bonded rings plus fused $R_2^2(8)$, $R_6^2(12)$ and $R_2^2(8)$ ring motifs occur alternately, aggregating into a supramolecular ladder-like arrangement. In (III), $R_2^2(8)$ motifs occur on either side of a further ring formed by pairs of N—H···O hydrogen bonds, forming an array of three fused hydrogen-bonded rings. In (I) and (II), the anions form a typical intramolecular O—H···O hydrogen bond with graph set $S(7)$, whereas in (III) an intramolecular hydrogen bond with graph set $S(6)$ is formed.

Comment

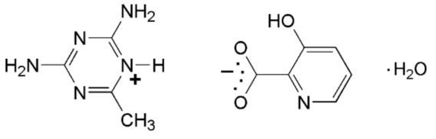
Aminopyrimidinium and aminotriazinium ions and their derivatives easily form acid–base complexes with carboxylate anions, which are linked by strong $N^+—H \cdots O^-$ (±) charge-assisted hydrogen bonds (Gilli *et al.*, 2000; Ferretti *et al.*, 2004). Triazine derivatives show antitumour activity, as well as a broad range of biological activities, such as anti-angiogenesis and antimicrobial effects (Bork *et al.*, 2003). The organic and inorganic complexes of triazine form well defined noncovalent supramolecular architectures *via* multiple hydrogen bonds, because of the presence of arrays of hydrogen-bonding sites (MacDonald & Whitesides, 1994). 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 (Sebenik *et al.*, 1989; Tashiro & Oiwa, 1981). Hydrogen-bonded carboxylic acid adducts with 2-amino heterocyclic ring systems frequently form a graph-set motif (Etter, 1990; Bernstein *et al.*, 1995) of $R_2^2(8)$ (Lynch & Jones, 2004).



(I)



(II)



(III)

The crystal structures of 2,4-diamino-6-methyl-1,3,5-triazine (Aoki *et al.*, 1994), 2,4-diamino-6-methyl-1,3,5-triazin-1-ium trifluoroacetate (Perpétuo & Janczak, 2007), 4-(dimethylamino)benzaldehyde and 6-phenyl-1,3,5-triazine-2,4-diamine (Habibi *et al.*, 2007), acetoguanaminium chloride–acetoguanamine (Portalone & Colapietro, 2007), acetoguanaminium *N*,*N*-dimethylformamide solvate (Portalone, 2008), 2,4-diamino-6-methyl-1,3,5-triazine methanol solvate (Kaczmarek

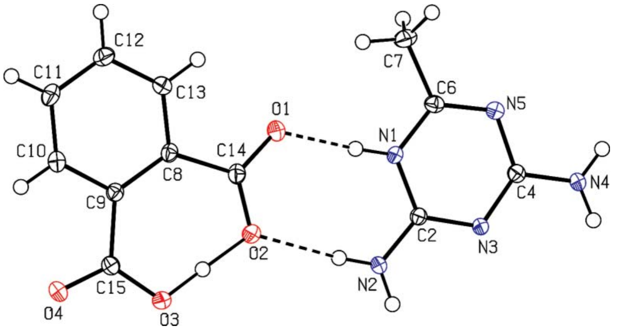


Figure 1
The asymmetric unit of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. Dashed lines indicate hydrogen bonds.

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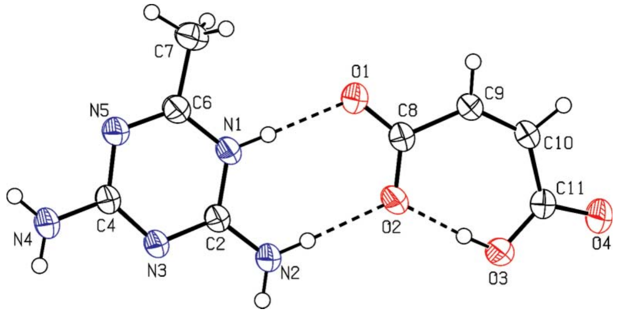

Figure 2
The asymmetric unit of (II), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. Dashed lines indicate hydrogen bonds.

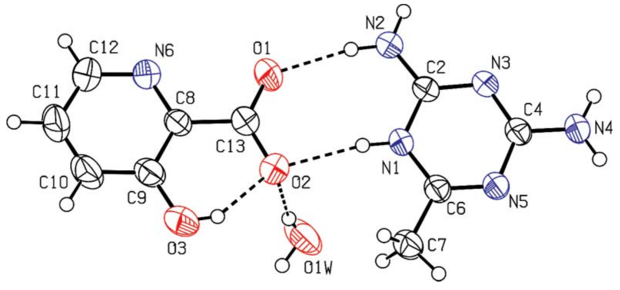

Figure 3
The asymmetric unit of (III), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. Dashed lines indicate hydrogen bonds.

et al., 2008) and 2,4-diamino-6-methyl-1,3,5-triazine ethanol solvate (Xiao, 2008) have been reported. Salts and cocrystals involving 2,4-diamino-6-methyl-1,3,5-triazine and various aliphatic dicarboxylic acids have been reported (Delori *et al.*, 2008). In this context, we present the synthesis and crystal structures of three organic salts, namely acetoguanaminium hydrogen phthalate, (I), acetoguanaminium hydrogen maleate, (II), and acetoguanaminium 3-hydroxypicolinate monohydrate, (III).

Views of compounds (I)–(III) are shown in Figs. 1–3. In (I) and (II), the asymmetric unit contains one acetoguanaminium cation and a hydrogen phthalate/hydrogen maleate anion. In (III), the asymmetric unit contains one acetoguanaminium cation, a 3-hydroxypicolinate anion and a water molecule. As expected for all three structures, the acetoguanaminium (AceguH^+) moieties are protonated at N1. In (I)–(III), the carboxylate groups of the anions (hydrogen phthalate, hydrogen maleate and 3-hydroxypicolinate, respectively) interact with the protonated triazinium moiety of the acetoguanaminium cations through a pair of linear N—H···O hydrogen bonds to form an eight-membered $R_2^2(8)$ ring motif (Figs. 1–3). This is one of the 24 most frequently observed bimolecular cyclic hydrogen-bonded motifs in organic crystal structures (Allen *et al.*, 1998). In (II) and (III), the triazine moieties of the AceguH^+ cations are centrosymmetrically paired through N—H···N hydrogen bonds, *viz.* N4—H4*A*···N3$^{\text{ii}}$ in (II) [symmetry code: (ii) $-x + 1, -y, -z + 1$] and N4—H4*B*···N5$^{\text{iv}}$ in (III) [symmetry code: (iv) $-x, -y + 1, -z$].

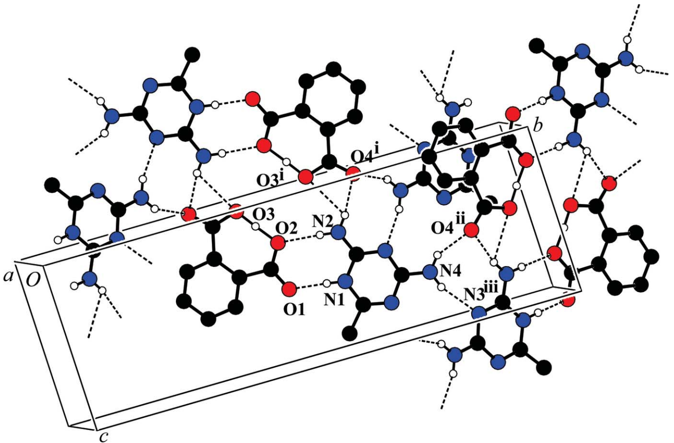

Figure 4
Hydrogen-bonding patterns in (I). Dashed lines indicate hydrogen bonds. H atoms not involved in hydrogen bonding have been omitted. [Symmetry codes: (i) $-x + 1, -y + 1, -z$; (ii) $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$; (iii) $x, -y + \frac{3}{2}, z + \frac{1}{2}$.]

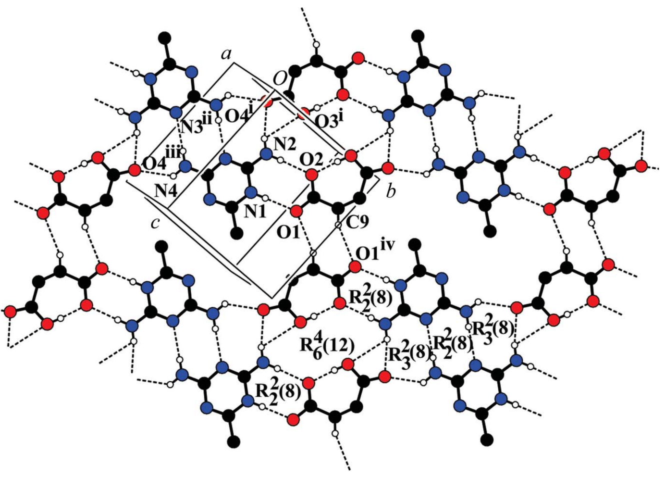

Figure 5
The *DADA* array of hydrogen bonds in (II), leading to a supramolecular ladder. Dashed lines indicate hydrogen bonds. H atoms not involved in hydrogen bonding have been omitted. [Symmetry codes: (i) $-x, -y + 1, -z$; (ii) $-x + 1, -y, -z + 1$; (iii) $x + 1, y - 1, z + 1$; (iv) $-x, -y + 2, -z + 1$.]

In (I) and (II), inversion-related $R_2^2(8)$ ring motifs (triazinium–carboxylate) lie on either side of an $R_6^4(12)$ motif formed by N—H···O and O—H···O (intramolecular) hydrogen bonds (Tables 1 and 2). The graph-set notation of the ring system is $R_2^2(8)$, $R_6^4(12)$ and $R_2^2(8)$ (Fig. 4). Similar types of interactions were also observed in trimethoprim hydrogen phthalate (Muthiah *et al.*, 2006). One of the H atoms of the 2-amino group forms a bifurcated hydrogen bond with the carboxyl O atoms of the hydrogen phthalate and hydrogen maleate anions, represented by graph-set $R_1^2(4)$. In the hydrogen phthalate anion of (I), there is a very strong intramolecular $[\text{O—H}\cdots\text{O}]^-$ hydrogen bond, graph-set notation *S*(7) [O2···O3 = 2.393 (13) Å], which is a result of the negative charge-assisted effect described by Gilli *et al.* (1994).

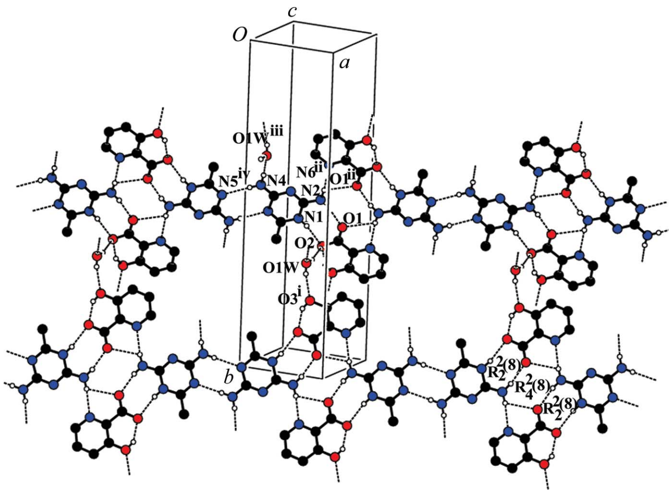

Figure 6
The *DDAA* hydrogen-bonding pattern in (III). Dashed lines indicate hydrogen bonds. H atoms not involved in hydrogen bonding have been omitted. [Symmetry codes: (i) $x, -y+\frac{3}{2}, z-\frac{1}{2}$; (ii) $-x+2, -y+1, -z+1$; (iii) $-x+1, -y+1, -z$; (iv) $-x, -y+1, -z$.]

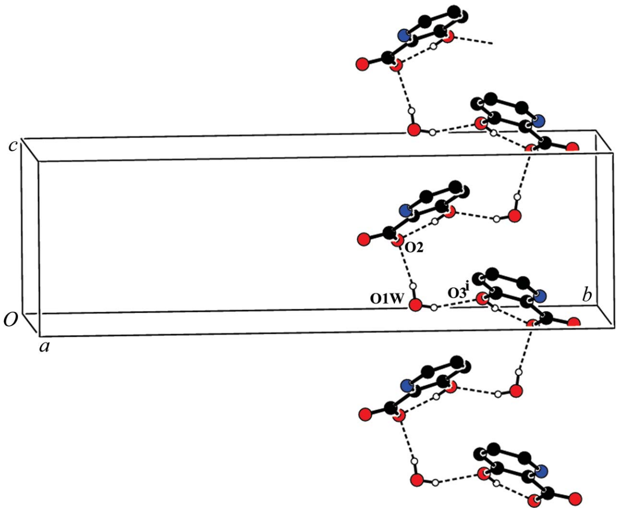

Figure 7
The supramolecular chain of 3-hydroxypicolinate anions and water molecules in (III). Dashed lines indicate hydrogen bonds. H atoms not involved in hydrogen bonding have been omitted. [Symmetry code: (i) $x, -y+\frac{3}{2}, z-\frac{1}{2}$.]

In (II), the 2- and 4-amino groups of the paired triazinium cations further interact with one of the carboxyl O atoms (O4) of the adjacent anions, leading to a complementary *DADA* array (*D* is a hydrogen-bond donor and *A* is a hydrogen-bond acceptor) of four hydrogen bonds. This type of interaction has already been reported in trimethoprim hydrogen maleate (Prabakaran *et al.*, 2001), trimethoprim trifluoroacetate (Francis *et al.*, 2002), pyrimethaminium formate (Stanley *et al.*, 2002), trimethoprim tetrafluroborate (Hemamalini *et al.*, 2005) and trimethoprim hydrogen phthalate (Muthiah *et al.*, 2006). The *DADA* hydrogen-bonding motif can be represented in the form of fused $R_3^2(8)$, $R_2^2(8)$ and $R_3^2(8)$ rings. The *DADA* arrays and $R_2^2(8)$, $R_6^4(12)$ and $R_2^2(8)$ ring motifs occur alternately, aggregating into a supramolecular ladder-like arrangement (Fig. 5). Adjacent ladders are crosslinked by $R_2^2(8)$ ring motifs of the inversely paired hydrogen maleate anions *via* C9—H9···O1^{iv} hydrogen bonds [symmetry code: (iv) $-x, -y+2, -z+1$]. The crosslinked motifs generate a large 26-membered [$R_6^4(26)$] ring motif (Fig. 5). The hydrogen maleate anion posseses nearly planar geometry and displays a strong intramolecular O3—H3···O2 hydrogen bond [Table 2; graph-set notation *S*(7)] (Lah & Leban, 2003).

In (III), the $R_2^2(8)$ motifs are linked by N2—H2*A*···O1^{ii} hydrogen bonds (symmetry code as in Table 3), forming a ring spanning the centre of symmetry at $(1, \frac{1}{2}, \frac{1}{2})$ to produce a *DDAA* array of four hydrogen bonds. This set of fused rings can be represented by the graph-set notations $R_2^2(8)$, $R_4^2(8)$ and $R_2^2(8)$ (Fig. 6). This type of motif has been reported in the crystal structures of trimethoprim hydrogen glutarate (Robert *et al.*, 2001), trimethoprim formate (Umadevi *et al.*, 2002) and pyrimethamine 3,5-dinitrobenzoate (Subashini *et al.*, 2007). The 2-amino group at N2 forms a bifurcated hydrogen bond (Table 3) with carboxyl atom O1^{ii} and atom N6^{ii} of a 3-hydroxypicolinate anion [graph-set $R_1^2(5)$]. These types of interactions are extended along the *a* axis to form a supramolecular ribbon. The ribbons are linked by the bridging water molecule into a two-dimensional net in the $(10\overline{2})$ plane. These two-dimensional nets are further linked into the three-dimensional structure by the interactions shown in Fig. 7. An intramolecular hydrogen bond between the hydroxy and carboxylate groups of the 3-hydroxypicolinate anion forms a six-membered hydrogen-bonded ring [*S*(6); Fig. 3].

In (I) and (III), π–π stacking interactions between the aromatic rings are observed. In (I), there are two distinct stacking interactions. Using the phthalate ring of the base asymmetric unit as reference, this ring has an interaction with the cation located at $(1-x, 1-y, 1-z)$, with a dihedral angle of 1.76 (6)°, a centroid-to-centroid distance of 3.6545 (8) Å, a perpendicular distance of 3.314 (6) Å and a slip angle of 24.94°. The second interaction, which has the same dihedral angle, is with the cation located at $(2-x, 1-y, 1-z)$, with a perpendicular separation of 3.367 (5) Å, a centroid-to-centroid distance of 3.5929 (8) Å and a slip angle of 20.45°. A stack is thus formed parallel to the *a* axis. In (III), a π–π interaction is observed between two 2,4-diamino-6-methyl-1,3,5-triazinium cations, with a centroid-to-centroid distance of 3.5906 (12) Å, an interplanar distance of 3.2875 (6) Å, a ring offset of 1.444 Å and a slip angle of 23.71°. These are in agreement with typical aromatic stacking values (Hunter, 1994).

In conclusion, in all three title crystal structures, the acetoguanaminium cation interacts with the carboxylate O atoms *via* N—H···O hydrogen bonds to form the frequently observed hydrogen-bonded eight-membered $R_2^2(8)$ ring motif. In (II) and (III), N—H···N base pairing is observed, while there is none in (I). In (II), the *DADA* [fused $R_3^2(8)$, $R_2^2(8)$ and $R_3^2(8)$ rings] array and $R_2^2(8)$, $R_6^4(12)$ and $R_2^2(8)$ ring motifs occur alternately, aggregating into a supramolecular ladder-like arrangement. In (III), the $R_2^2(8)$ motifs lie on either side of a ring formed by N—H···O hydrogen bonds, forming fused rings through a *DDAA* array of four hydrogen bonds.

Experimental

Compounds (I)–(III) were prepared by mixing hot methanolic solutions (20 ml) of 2,4-diamino-6-methyl-1,3,5-triazine (acetoguanamine) (31 mg, Aldrich) with hot aqueous solutions (20 ml) of the corresponding acids [phthalic acid (41 mg, Loba Chemie), maleic acid (29 mg, Loba Chemie) or 3-hydroxypicolinic acid (35 mg, Loba Chemie), respectively] in a 1:1 molar ratio, and warming the mixtures for 30 min over a water bath. Each solution was cooled slowly and kept at room temperature. After a few days, colourless crystals of the title compounds were obtained.

Compound (I)

Crystal data

$C_4H_8N_5^+ \cdot C_8H_5O_4^-$
$M_r = 291.27$
Monoclinic, $P2_1/c$
$a = 7.0743\ (2)$ Å
$b = 23.4468\ (5)$ Å
$c = 8.1134\ (1)$ Å
$\beta = 107.063\ (1)°$
$V = 1286.53\ (5)$ Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.12$ mm^{-1}
$T = 120$ K
0.54 × 0.30 × 0.25 mm

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer
14262 measured reflections
2918 independent reflections
2586 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.032$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.127$
$S = 1.12$
2918 reflections
196 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\rm max} = 0.39$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.54$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °) for (I).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1	0.86	1.80	2.6587 (14)	173
N2—H2*A*···O3$^{\rm i}$	0.86	2.59	3.0541 (14)	115
N2—H2*A*···O4$^{\rm i}$	0.86	2.08	2.9223 (14)	167
N2—H2*B*···O2	0.86	2.06	2.9074 (15)	168
O3—H3···O2	1.12 (2)	1.28 (2)	2.3916 (13)	175 (2)
N4—H4*A*···O4$^{\rm ii}$	0.86	2.10	2.8748 (15)	149
N4—H4*B*···N3$^{\rm iii}$	0.86	2.14	2.9555 (15)	158

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

Table 2
Hydrogen-bond geometry (Å, °) for (II).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1	0.86	1.88	2.7423 (15)	179
N2—H2*A*···O3$^{\rm i}$	0.86	2.59	3.0650 (15)	116
N2—H2*A*···O4$^{\rm i}$	0.86	2.09	2.9406 (16)	169
N2—H2*B*···O2	0.86	1.93	2.7852 (16)	177
O3—H3···O2	0.82	1.62	2.4375 (15)	176
N4—H4*A*···N3$^{\rm ii}$	0.86	2.21	3.0660 (17)	173
N4—H4*B*···O4$^{\rm iii}$	0.86	2.19	2.8346 (17)	132
C9—H9···O1$^{\rm iv}$	0.93	2.58	3.4143 (17)	150

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

Table 3
Hydrogen-bond geometry (Å, °) for (III).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*W*···O3$^{\rm i}$	0.796 (18)	2.112 (18)	2.875 (2)	161 (3)
O1*W*—H2*W*···O2	0.82 (2)	2.07 (2)	2.880 (3)	166 (3)
N1—H1···O2	0.86	2.04	2.890 (2)	169
N2—H2*A*···O1$^{\rm ii}$	0.86	2.44	2.912 (2)	115
N2—H2*A*···N6$^{\rm ii}$	0.86	2.11	2.965 (2)	172
N2—H2*B*···O1	0.86	1.94	2.787 (2)	169
O3—H3···O2	0.82	1.80	2.526 (2)	147
N4—H4*A*···O1*W*$^{\rm iii}$	0.86	2.06	2.898 (2)	164
N4—H4*B*···N5$^{\rm iv}$	0.86	2.14	2.994 (2)	175

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

Compound (II)

Crystal data

$C_4H_8N_5^+ \cdot C_4H_3O_4^-$
$M_r = 241.22$
Triclinic, $P\overline{1}$
$a = 7.1921\ (3)$ Å
$b = 8.2359\ (3)$ Å
$c = 9.4775\ (4)$ Å
$\alpha = 98.287\ (2)°$
$\beta = 104.656\ (2)°$
$\gamma = 91.032\ (3)°$
$V = 536.58\ (4)$ Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 0.12$ mm^{-1}
$T = 293$ K
0.22 × 0.18 × 0.16 mm

Data collection

Bruker Kappa APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (Blessing, 1995)
$T_{\rm min} = 0.974$, $T_{\rm max} = 0.981$
13516 measured reflections
3779 independent reflections
2607 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.032$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.168$
$S = 1.04$
3779 reflections
156 parameters
H-atom parameters constrained
$\Delta\rho_{\rm max} = 0.37$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.26$ e Å^{-3}

Compound (III)

Crystal data

$C_4H_8N_5^+ \cdot C_6H_4NO_3^- \cdot H_2O$
$M_r = 282.27$
Monoclinic, $P2_1/c$
$a = 7.0397\ (16)$ Å
$b = 24.680\ (5)$ Å
$c = 7.5047\ (19)$ Å
$\beta = 94.462\ (7)°$
$V = 1299.9\ (5)$ Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.11$ mm^{-1}
$T = 296$ K
0.56 × 0.42 × 0.28 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\rm min} = 0.939$, $T_{\rm max} = 0.969$
10842 measured reflections
3233 independent reflections
2090 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.050$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.154$
$S = 1.03$
3233 reflections
191 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\rm max} = 0.24$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.19$ e Å^{-3}

In (I), atom H3 (on O3) was located in a difference Fourier map and refined freely. The H atoms of the water molecule in (III) were located in a difference Fourier map and their positional parameters were initially refined with O—H distance restraints of 0.82 Å and H···H restraints of 1.297 Å. The other H atoms in (I)–(III) were positioned geometrically and refined using a riding model, with N—H = 0.86 Å and C—H = 0.93–0.96 Å in (I)–(III), and O—H = 0.82 Å in (II) and (III), and with $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C,O) for OH groups and all methyl groups, or 1.2$U_{\rm eq}$(C,N) for other H atoms.

Data collection: *COLLECT* (Hooft, 1998) for (I); *APEX2* (Bruker, 2004) for (II) and (III). Cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* for (I); *APEX2* and *SAINT* (Bruker, 2004) for (II); *SAINT* for (III). Data reduction: *DENZO* and *COLLECT* for (I); *SAINT* and *XPREP* (Bruker, 2004) for (II); *SAINT* for (III). For all three compounds, program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPII* (Johnson, 1976) and *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FA3215). Services for accessing these data are described at the back of the journal.

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