Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

Three-dimensional hydrogen-bonded structures in the 1:1 proton-transfer compounds of L-tartaric acid with the associative-group monosubstituted pyridines 3-aminopyridine, 3-carboxypyridine (nicotinic acid) and 2-carboxypyridine (picolinic acid)

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Received 12 October 2009
Accepted 18 November 2009
Online 12 December 2009

The 1:1 proton-transfer compounds of L-tartaric acid with 3-aminopyridine [3-aminopyridinium hydrogen (2*R*,3*R*)-tartrate dihydrate, $C_5H_7N_2^+\cdot C_4H_5O_6^-\cdot 2H_2O$, (I)], pyridine-3-carboxylic acid (nicotinic acid) [anhydrous 3-carboxypyridinium hydrogen (2*R*,3*R*)-tartrate, $C_6H_6NO_2^+\cdot C_4H_5O_6^-$, (II)] and pyridine-2-carboxylic acid [2-carboxypyridinium hydrogen (2*R*,3*R*)-tartrate monohydrate, $C_6H_6NO_2^+\cdot C_4H_5O_6^-\cdot H_2O$, (III)] have been determined. In (I) and (II), there is a direct pyridinium–carboxyl $N^+—H\cdots O$ hydrogen-bonding interaction, four-centred in (II), giving conjoint cyclic $R_1^2(5)$ associations. In contrast, the $N—H\cdots O$ association in (III) is with a water O-atom acceptor, which provides links to separate tartrate anions through $O_{hydroxy}$ acceptors. All three compounds have the head-to-tail *C*(7) hydrogen-bonded chain substructures commonly associated with 1:1 proton-transfer hydrogen tartrate salts. These chains are extended into two-dimensional sheets which, in hydrates (I) and (III) additionally involve the solvent water molecules. Three-dimensional hydrogen-bonded structures are generated *via* crosslinking through the associative functional groups of the substituted pyridinium cations. In the sheet struture of (I), both water molecules act as donors and acceptors in interactions with separate carboxyl and hydroxy O-atom acceptors of the primary tartrate chains, closing conjoint cyclic $R_4^4(8)$, $R_3^4(11)$ and $R_3^3(12)$ associations. Also, in (II) and (III) there are strong cation carboxyl–carboxyl $O—H\cdots O$ hydrogen bonds [$O\cdots O$ = 2.5387 (17) Å in (II) and 2.441 (3) Å in (III)], which in (II) form part of a cyclic $R_2^2(6)$ inter-sheet association. This series of heteroaromatic Lewis base–hydrogen L-tartrate salts provides further examples of molecular assembly facilitated by the presence of the classical two-dimensional hydrogen-bonded hydrogen tartrate or hydrogen tartrate–water sheet substructures which are expanded into three-dimensional frameworks *via* peripheral cation bifunctional substituent-group crosslinking interactions.

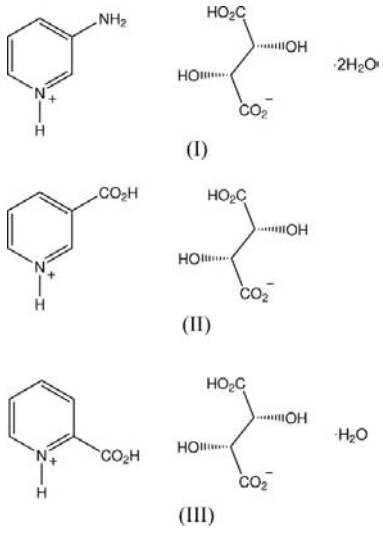
Comment

The use of common L-tartaric acid for the production of crystalline salts suitable for the single-crystal characterization of Lewis bases has been employed extensively. Such salts, particularly the 1:1 hydrogen L-tartrates, have been employed as biologically compatibile pharmaceuticals [*e.g.* carbinoxamine, dextromoramide, epinephrine, ergotamine, levorphanol and morphine (O'Neil, 2001)] and as achiral materials with potential for nonlinear optical applications (Aakeröy *et al.*, 1992; Kadirvelraj *et al.*, 1998). However, known structures of the hydrogen L-tartrate salts of the associative-group substituted monocyclic heteroaromatic base pyridine are few. These include the 1:1 proton-transfer salts with the substituted pyridines 3-hydroxypyridine (Tafeenko *et al.*, 1990), 3-methoxypyridine (Renuka *et al.*, 1995), 2-amino-5-nitropyridine (Watanabe *et al.*, 1993; Zyss *et al.*, 1993), 3,4-diaminopyridine (Koleva *et al.*, 2008), 4-(dimethylamino)pyridine (Pecaut, 1993; Parthasarathi *et al.*, 1993; Manivannan *et al.*, 2006), 4-carboxypyridine (isonicotinic acid) (Athimoolam & Natarajan, 2007*a*), 3-(aminocarboxy)pyridine (nicotinamide) (Athimoolam & Natarajan, 2007*b*) and 4-(aminocarboxy)pyridine (isonicotinamide) (Bhogala *et al.*, 2005). In the structures of the majority of the anhydrous hydrogen tartrates (Aakeröy *et al.*, 1992; Aakeröy & Hitchcock, 1993), homomeric *C*(7) (Etter *et al.*, 1990) head-to-tail carboxylic acid–carboxylate hydrogen-bonding associations form primary chain substructures. These are extended into two-dimensional sheets and then, in the majority of examples, further extended into three-dimensional framework structures. The cation species, or the solvent water molecules in the hydrated salts, are often involved in the formation of these two-dimensional sheet structures. The presence of associative functional group substituents on the pyridinium cation promotes the formation of the framework structures. In some cases, *e.g.* the isonicotinic acid salt (Athimoolam & Natarajan, 2007*a*), where there are two independent hydrogen tartrate residues in the asymmetric unit, these anions form inter-associated duplex *C*(7)-linked chains and sheet structures, as well as three-dimensional framework structures.

Considering this background, it was surprising that the structures of the 1:1 proton-transfer hydrogen L-tartrate salts of the analogous 3-carboxypyridine (nicotinic acid) or 2-carboxypyridine (picolinic acid) were not known. We therefore carried out 1:1 stoichiometric reactions of L-tartaric acid with a number of associative-group substituted pyridines, with the intention of obtaining crystalline compounds suitable for X-ray analysis to enable examination of the hydrogen-bonding systems present. Crystalline compounds were obtained with the salts from the reactions of 3-aminopyridine, nicotinic acid and picolinic acid, from aqueous ethanol or propan-2-ol solvent systems: these are 3-aminopyridinium

 doi:10.1107/S0108270109049154

hydrogen (2*R*,3*R*)-tartrate dihydrate, (I), anhydrous 3-carboxypyridinium hydrogen (2*R*,3*R*)-tartrate, (II), and 2-carboxypyridinium hydrogen (2*R*,3*R*)-tartrate monohydrate, (III), and their structures are reported here.



In compounds (I)–(III) (Figs. 1–3), not unexpectedly, the N heteroatom of the base is protonated and forms direct $N^+—H\cdots O$ hydrogen-bonding interactions (Tables 1–3). However, unlike the structures of both (I) and (II) where the interaction is with a carboxyl O-atom acceptor, in (III) one of the solvent water molecules becomes the acceptor. This is probably due to steric factors associated with the *ortho*-related carboxy group. In all three structures, the hydrogen tartrate anions form the common primary *C*(7) hydrogen-bonded chain substructures, two-dimensional sheets and overall three-dimensional framework structures (Figs. 4–8). Because of the presence of solvent water molecules in (I) and (III), the three-dimensional makeup is sufficiently different for all three compounds to be considered separately.

With the structure of the 3-aminopyridinium dihydrate salt, (I) (Fig. 1), the primary head-to-tail hydrogen tartrate anion chains form along the *a*-axis direction in the unit cell and are

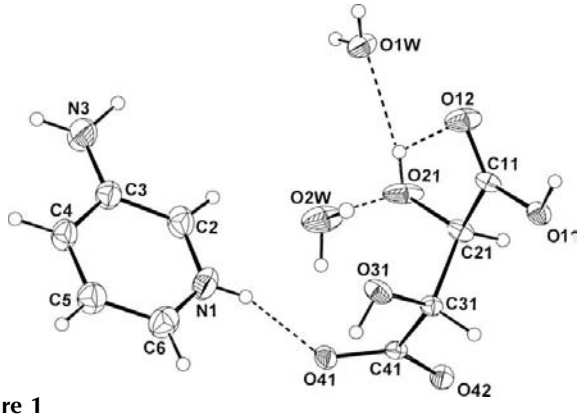


Figure 1
The molecular configuration and atom-numbering scheme for the 3-aminopyridinium cation, the hydrogen L-tartrate anion and the two solvent water molecules of (I). Displacement ellipsoids are drawn at the 40% probability level and H atoms are shown as small spheres of arbitrary radii. Dashed lines indicate hydrogen bonds.

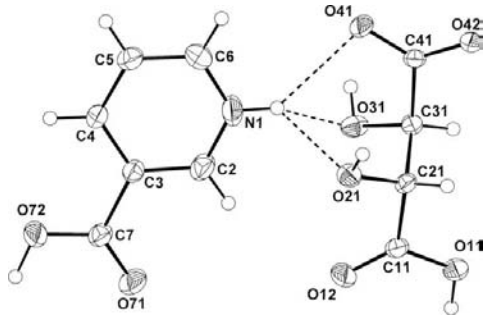


Figure 2
The molecular configuration and atom-numbering scheme for the 3-carboxypyridinium cation and the hydrogen L-tartrate anion of (II). Displacement ellipsoids are drawn at the 40% probability level and H atoms are shown as small spheres of arbitrary radii. Dashed lines indicate hydrogen bonds.

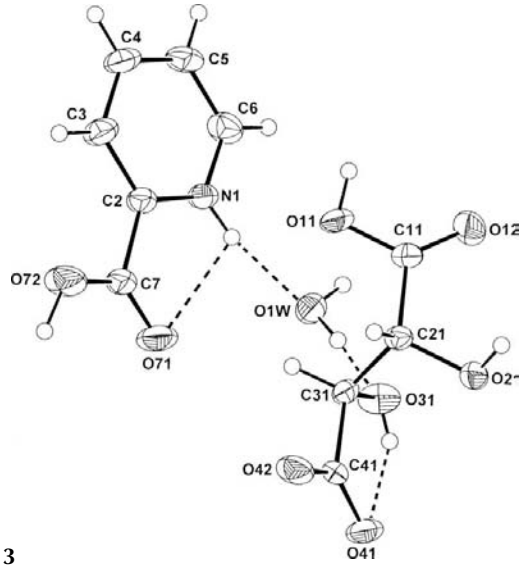


Figure 3
The molecular configuration and atom-numbering scheme for the 2-carboxypyridinium cation, the hydrogen L-tartrate anion and the solvent water molecule of (III). Displacement ellipsoids are drawn at the 40% probability level and H atoms are shown as small spheres of arbitrary radii. Dashed lines indicate hydrogen bonds.

extended into two-dimensional sheet structures through hydrogen-bonding interactions involving the two solvent water molecules (O1*W* and O2*W*) (Fig. 4). These interactions (Table 1) include the hydroxy groups acting as both donors and acceptors with both water molecules, closing conjoint $R_4^4(8)$, $R_3^4(11)$ and $R_3^3(12)$ cyclic associations. Extension into a three-dimensional framework involves both functional groups of the 3-aminopyridinium cation, which form links across the *c* cell direction with their aromatic rings layering down *a*, giving some ring overlap with weak π–π interactions [ring centroid separation = 3.719 (2) Å] (Fig. 5).

In the structure of anhydrous compound (II), with nicotinic acid, the primary hydrogen tartrate sheet structure forms through hydroxy–carboxyl $O—H\cdots O$ interchain cyclic associations [graph sets $R_2^2(11)$ and $R_4^4(19)$; Fig. 6]. The pyridinium $N^+—H$ group is involved in a four-centre cation–anion hydrogen-bonding association with a carboxylate O atom and two hydroxy O-atom acceptors of the anion (Fig. 2), enclosing

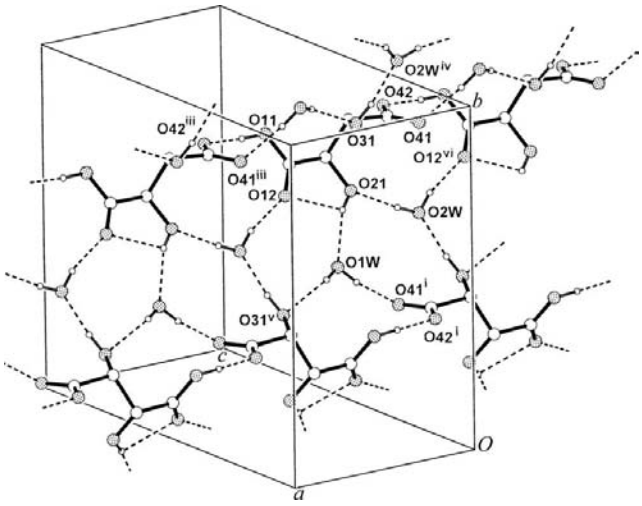


Figure 4
A view of the *C*(7) head-to-tail hydrogen-bonded chains of hydrogen tartrate anions in (I) and their extension into two-dimensional sheets through the two solvent water molecules. H atoms not involved in hydrogen bonding and the 3-amininopyridinium cations have been omitted for clarity. Hydrogen bonds are shown as dashed lines. (See Table 1 for symmetry codes.)

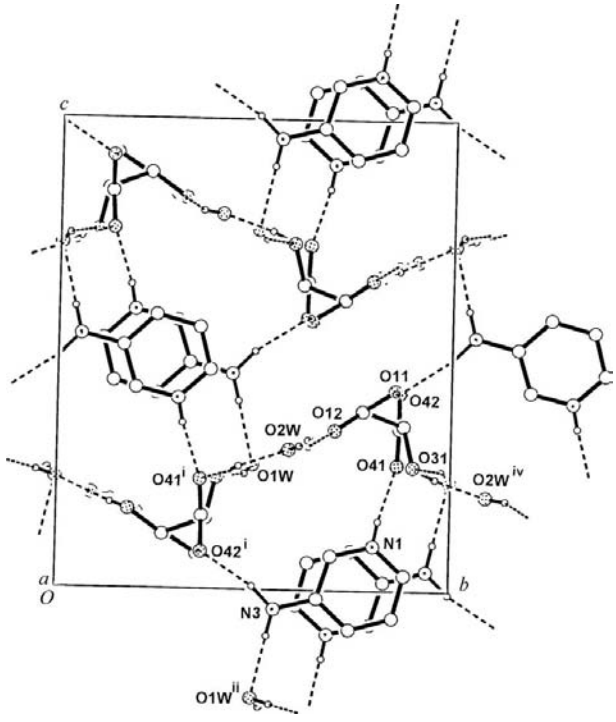


Figure 5
The three-dimensional extension of the hydrogen tartrate–water sheet substructures in (I) *via* the substituted pyridinium cations, viewed down the *a* cell direction. H atoms not involved in hydrogen bonding have been omitted for clarity. (See Table 1 for symmetry codes.)

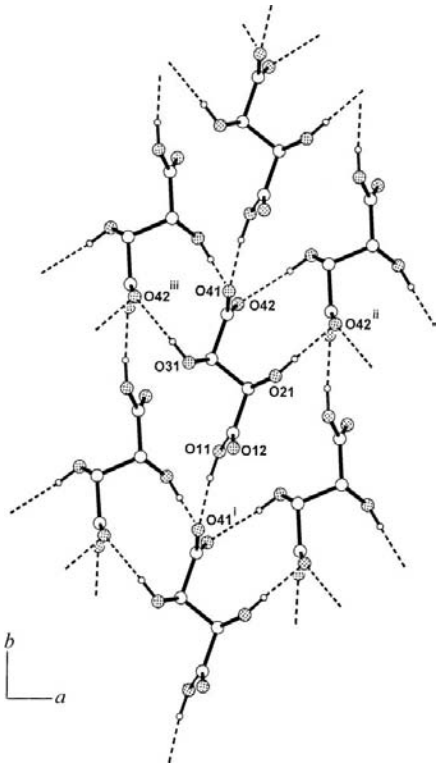


Figure 6
A view of the homomeric hydrogen-bonded two-dimensional sheet substructure in (II), comprising hydrogen tartrate anions only. H atoms not involved in hydrogen bonding and the 3-carboxypyridinium cations have been omitted for clarity. Hydrogen bonds are shown as dashed lines. (See Table 2 for symmetry codes.)

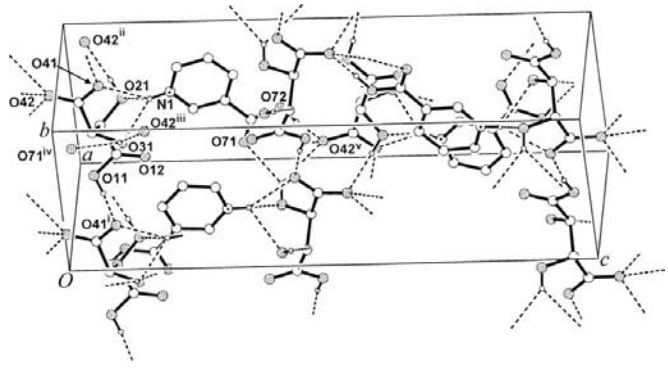


Figure 7
A perspective view of the packing of the three-dimensional structure of (II) in the unit cell, showing the peripheral extension of the primary sheet substructures across the *c* cell direction *via* the 3-carboxypyridinium cations. H atoms not involved in hydrogen bonding have been omitted for clarity. Hydrogen bonds are shown as dashed lines. (See Table 2 for symmetry codes.)

two conjoint cyclic $R_1^2(5)$ interactions. The two hydroxy H atoms and the two carboxylic acid H-atom donors (one from the anion and the other from the cation) are involved in hydrogen-bonding interactions (Table 2), expanding the primary hydrogen tartrate sheets into the three-dimensional framework structure through cation crosslinks (Fig. 7). The cation carboxyl/anion carboxylate O—H···O hydrogen bond is strong [O···O = 2.5387 (17) Å] and forms a cyclic $R_2^2(6)$ association linking separate tartrate residues in the chains *via* carboxyl and hydroxy groups.

In the monohydrate picolinate structure, (III), the solvent water molecule (O1*W*), which acts as an acceptor for the pyridinium H atom, also provides a three-centre bridge between the primary hydrogen tartrate anion chain substructures through separate hydroxy groups (Fig. 8). These tartrate

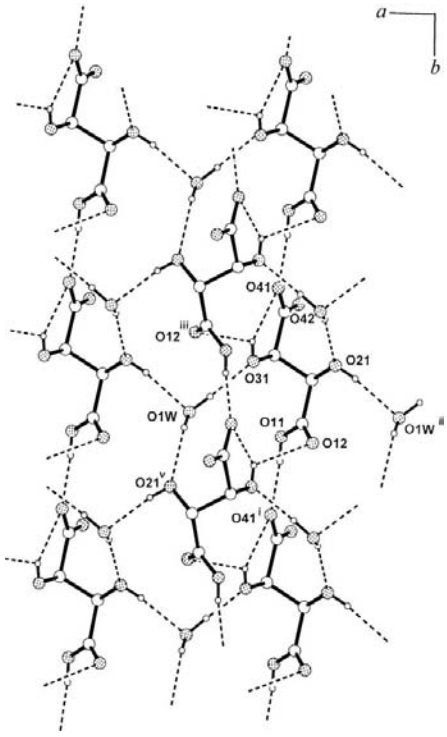


Figure 8
A view of the hydrogen-bonded two-dimensional chains of hydrogen tartrate anions in (III) and their extension into two-dimensional sheets *via* the solvent water molecule. H atoms not involved in hydrogen bonding and the 2-carboxypyridinium cations have been omitted for clarity. Hydrogen bonds are shown as dashed lines. (See Table 3 for symmetry codes.)

chains extend down the *b* cell direction of the unit cell. The bifunctional 2-carboxypyridinium cations provide the cross-links between the sheets, extending the structure along the *c* axial direction. Additional peripheral hydrogen-bonding associations (Table 3), including a strong cation carboxyl/anion carboxylate O—H···O hydrogen bond [O···O = 2.441 (3) Å], give the three-dimensional structure (Fig. 9). The picolinate cation has an intramolecular hydrogen bond [N···O = 2.682 (3) Å] between the pyridinium H atom and an O-atom acceptor of the *cis*-related carboxyl group, which essentially maintains molecular planarity [torsion angle N1—C2—C7—O72 = 175.9 (3)°].

The accepted (2*R*,3*R*) absolute configuration for the L-tartrate residues in compounds (I)–(III) (Bijvoet *et al.*, 1951; Lutz & Schreurs, 2008) was assumed and these anions adopt the common extended hydrogen tartrate conformation. The intramolecular hydroxy–carboxyl O—H···O hydrogen bond, which is common in hydrogen L-tartrates, is also found in hydrated compounds (I) (O21—H···O12) and (III) (O31—H···O41), but is absent in anhydrous compound (II). Despite this, there are no significant conformational differences in the three hydrogen tartrate anions, the characteristic O21—C21—C31—O31 torsion angle being −70.5 (2)° in (I), −65.11 (17)° in (II) and −63.5 (3)° in (III), which compare with the values of −65.1 (4) and −73.5 (4)° in the two independent anions in the asymmetric unit of 4-carboxyanilinium hydrogen L-tartrate (Athimoolam & Natarajan, 2007*a*), and −74.3 (3)° in the unsubstituted pyridinium hydrogen L-tartrate (Suresh *et al.*, 2006).

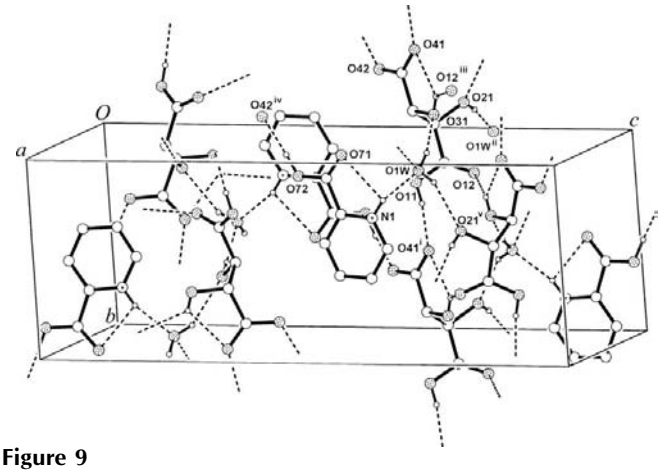


Figure 9
A perspective view of the packing of the three-dimensional structure of (III) in the unit cell, showing the extension of the sheet substructures across the *c* cell direction *via* the 2-carboxypyridinium cations. H atoms not involved in hydrogen bonding have been omitted for clarity. Hydrogen bonds are shown as dashed lines. (See Table 3 for symmetry codes.)

This series of monocyclic heteroaromatic Lewis base–hydrogen L-tartrate salts provides further examples of molecular assembly facilitated by the presence of the classical $C_1^1(7)$ hydrogen-bonded head-to-tail hydrogen tartrate chains. These chains are expanded into two-dimensional sheets, which may also contain solvent water molecules. Three-dimensional frameworks result from inter-sheet crosslinking through the interactive bifunctional substituent group of the pyridinium cations.

Experimental

Compounds (I)–(III) were synthesized by heating together for 10 min under reflux L-tartaric acid (1 mmol) and, respectively, 3-aminopyridine, pyridine-3-carboxylic acid (nicotinic acid) and pyridine-2-carboxylic acid (picolinic acid) (1 mmol) in either ethanol–water (50 ml, 1:1 *v*/*v*) for (I) or propan-2-ol–water (50 ml, 1:1 *v*/*v*) for (II) and (III). After partial room-temperature evaporation of the solvents, all compounds gave hard colourless crystals: prisms for (I) (m.p. 371–373 K), blocks for (II) (m.p. 470–471 K) and plates for (III) (m.p. 393 K).

Compound (I)

Crystal data

$C_5H_7N_2^+\cdot C_4H_5O_6^-\cdot 2H_2O$
M_r = 280.24
Orthorhombic, $P2_12_12_1$
a = 7.3073 (12) Å
b = 12.1065 (13) Å
c = 14.541 (2) Å
V = 1286.4 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 200 K
0.35 × 0.25 × 0.20 mm

Data collection

Oxford Gemini-S CCD area-detector diffractometer
4764 measured reflections
1722 independent reflections
1434 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.098$
$S = 1.02$
1722 reflections
212 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}

Compound (II)

Crystal data

$C_6H_6NO_2^+\cdot C_4H_5O_6^-$
$M_r = 273.20$
Orthorhombic, $P2_12_12_1$
$a = 6.5792$ (2) Å
$b = 7.7637$ (2) Å
$c = 21.6830$ (5) Å
$V = 1107.55$ (5) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.15$ mm^{-1}
$T = 297$ K
0.40 × 0.40 × 0.30 mm

Data collection

Oxford Gemini-S CCD area-detector diffractometer
5552 measured reflections
1537 independent reflections
1317 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.066$
$S = 1.06$
1537 reflections
192 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max} = 0.16$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}

Compound (III)

Crystal data

$C_6H_6NO_2^+\cdot C_4H_5O_6^-\cdot H_2O$
$M_r = 291.21$
Orthorhombic, $P2_12_12_1$
$a = 7.1536$ (4) Å
$b = 7.8273$ (3) Å
$c = 22.0145$ (10) Å
$V = 1232.67$ (10) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.14$ mm^{-1}
$T = 297$ K
0.45 × 0.25 × 0.08 mm

Data collection

Oxford Gemini-S CCD area-detector diffractometer
6582 measured reflections
1709 independent reflections
1416 reflections with $I > 2\sigma(I)$
$R_{int} = 0.051$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.107$
$S = 1.05$
1709 reflections
209 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}

H atoms potentially involved in hydrogen-bonding interactions were located by difference methods and their positional and isotropic displacement parameters were refined. Other H atoms were included at calculated positions, with aromatic C—H = 0.93 Å and aliphatic C—H = 0.98 Å, and treated as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$. The absolute configuration determined for the parent L-(+)-tartaric acid, (2*R*,3*R*) (Bijvoet *et al.*, 1951; Lutz & Schreurs, 2008), was invoked in all cases. Friedel pairs were averaged in all data sets used in the final refinements, meaningless values having been obtained for the absolute structure parameters (Flack, 1983) by refinement against unmerged data sets for these light-atom structures. It should also be noted that with (I) the structure was refined using diffraction data re-acquired at 200 K, necessitated because of the very large displacement parameters for all atoms in the structure obtained from room-temperature data. This problem is still apparent but considerably lessened in the low-temperature structure reported here. With (II) and (III), the problem was not significant and room-temperature data were used.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O41	0.79 (4)	1.95 (4)	2.707 (3)	161 (4)
N3—H3*A*···O42^{i}	1.00 (4)	1.95 (4)	2.934 (3)	168 (4)
N3—H3*B*···O1*W*^{ii}	0.87 (3)	2.11 (3)	2.960 (3)	164 (3)
O11—H11···O42^{iii}	0.99 (5)	1.55 (5)	2.538 (2)	175 (2)
O21—H21*A*···O1*W*	0.76 (4)	2.02 (4)	2.745 (3)	162 (4)
O21—H21*A*···O12	0.76 (4)	2.29 (4)	2.622 (3)	107 (3)
O31—H31*A*···O2*W*^{iv}	0.86 (4)	1.78 (4)	2.640 (3)	171 (3)
O1*W*—H11*W*···O41^{i}	0.87 (4)	1.85 (4)	2.711 (3)	169 (3)
O1*W*—H12*W*···O31^{v}	0.75 (5)	2.07 (5)	2.798 (3)	162 (5)
O2*W*—H21*W*···O12^{vi}	0.80 (5)	1.99 (4)	2.736 (3)	155 (4)
O2*W*—H22*W*···O21	0.86 (6)	1.87 (6)	2.724 (3)	174 (3)

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

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O21	0.89 (3)	2.33 (3)	2.874 (2)	120 (2)
N1—H1···O31	0.89 (3)	2.15 (3)	2.893 (2)	141 (2)
N1—H1···O41	0.89 (3)	2.34 (3)	3.0624 (19)	138 (2)
O11—H11···O41^{i}	0.91 (3)	1.70 (3)	2.5951 (18)	171 (3)
O21—H21*A*···O42^{ii}	0.89 (3)	1.91 (3)	2.8064 (18)	178 (3)
O31—H31*A*···O42^{iii}	0.87 (3)	2.48 (3)	3.1542 (18)	135 (2)
O31—H31*A*···O71^{iv}	0.87 (3)	2.30 (3)	3.014 (2)	139 (2)
O72—H72···O42^{v}	0.99 (3)	1.55 (3)	2.5387 (17)	176.3 (19)

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

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1*W*	0.85 (4)	1.96 (4)	2.779 (3)	162 (4)
N1—H1···O71	0.85 (4)	2.37 (4)	2.682 (3)	102 (3)
O11—H11···O41^{i}	0.85 (5)	1.83 (5)	2.669 (3)	171 (4)
O21—H21*A*···O1*W*^{ii}	0.80 (4)	2.00 (4)	2.800 (3)	171 (4)
O31—H31*A*···O41	0.74 (5)	2.18 (5)	2.669 (3)	124 (4)
O31—H31*A*···O12^{iii}	0.74 (5)	2.49 (5)	2.883 (4)	115 (4)
O72—H72···O42^{iv}	0.94 (5)	1.50 (5)	2.441 (3)	177 (6)
O1*W*—H11*W*···O31	0.89 (5)	1.90 (5)	2.781 (3)	168 (4)
O1*W*—H12*W*···O21^{v}	0.82 (5)	2.22 (5)	2.975 (3)	154 (4)

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

For all compounds, data collection: *CrysAlis CCD* (Oxford Diffraction, 2008); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2008); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008) within *WinGX* (Farrugia, 1999); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON*.

The authors acknowledge financial support from the Australian Research Committee, and the School of Physical and Chemical Sciences, Queensland University of Technology.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SF3121). Services for accessing these data are described at the back of the journal.

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