

2-Pyridone–tartronic acid (1/1), 3-hydroxypyridinium hydrogen tartronate and 4-hydroxypyridinium hydrogen tartronate

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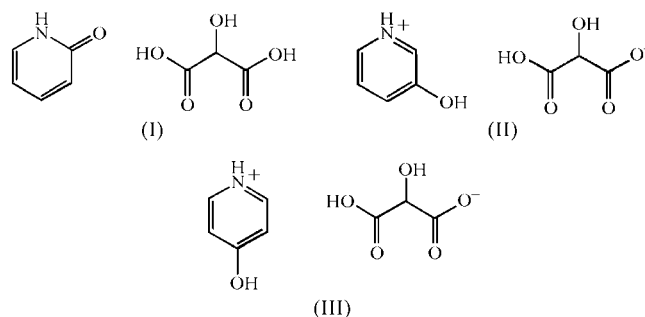
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Tartronic acid forms a hydrogen-bonded complex, C₅H₅NO·C₃H₄O₅, (I), with 2-pyridone, while it forms acid salts, namely 3-hydroxypyridinium hydrogen tartronate, (II), and 4-hydroxypyridinium hydrogen tartronate, (III), both C₅H₆NO⁺·C₃H₃O₅⁻, with 3-hydroxypyridine and 4-hydroxypyridine, respectively. In (I), the pyridone molecules and the acid molecules form R₂²(8) and R₂²(10) hydrogen-bonded rings, respectively, around the inversion centres. In (II) and (III), the cations and anions are linked by N—H···O and O—H···O hydrogen bonds to form a hydrogen-bonded chain. In each of (I), (II) and (III), an intermolecular hydrogen bond is formed between a carboxyl group and the hydroxyl group attached to the central C atom, and in (I), the hydroxyl group participates in an intramolecular hydrogen bond with a carbonyl group. No intermolecular hydrogen bond is formed between the carboxyl groups in (I), or between the carboxyl and carboxylate groups in (II) and (III).

Comment

Tartronic acid, a member of a family of hydroxycarboxylic acids, is of potential interest in crystal engineering for the formation of two- and three-dimensional aggregates with organic bases, as shown in several organic salts of tartaric acid (Aakeröy *et al.*, 1992; Aakeröy & Hitchcock, 1993; Aakeröy, Bahra & Nieuwenhuyzen, 1996), malonic acid (Aakeröy & Nieuwenhuyzen, 1994, 1996), dihydroxymalonic acid (Aakeröy, Nieuwenhuyzen & Robinson, 1998) and oxamic acid (Aakeröy, Hughes & Nieuwenhuyzen, 1996). Furthermore, tartronic acid is an interesting candidate for the selective synthesis and crystallization of optically active substances, because its acidic anion, the hydrogen tartronate ion, is optically active. Previously, we have reported the three-dimensional hydrogen-bonded structure of imidazolium hydrogen

tartronate, where the anions form a homochiral infinite chain but the chains aggregate to form a heterochiral layer (Fukunaga & Ishida, 2003). In the present study, we have selected



hydroxypyridines as the counter-base in order to extend the previous examination further, and report here the structures of the title compounds, (I)–(III). Fig. 1 shows the asymmetric units of compounds (I), (II) and (III) and Tables 1, 3 and 5, respectively, give details of selected geometric parameters.

In (I), the base molecule is in the lactam form (Yang & Craven, 1998), and no acid–base interaction involving H-atom

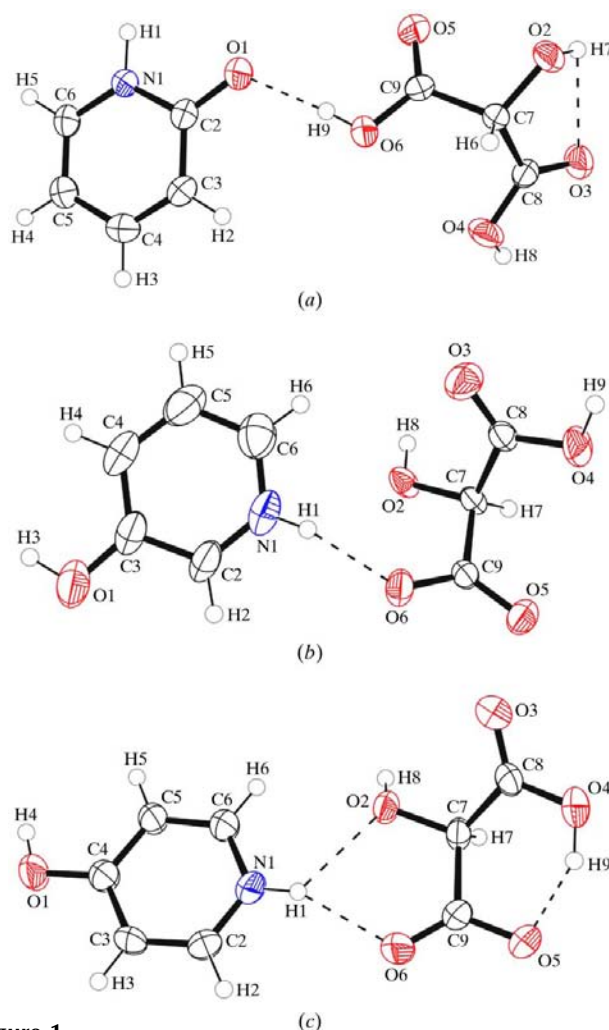


Figure 1
A view of the asymmetric units of (a) (I), (b) (II) and (c) (III). Displacement ellipsoids are drawn at the 50% probability level. The N—H···O and O—H···O hydrogen bonds are indicated by dashed lines.

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transfer is observed. Two pyridone molecules are held together by $N-H \cdots O$ hydrogen bonds, forming a centrosymmetric hydrogen-bonded ring with graph-set descriptor $R_2^2(8)$ (Bernstein *et al.*, 1995), and two molecules of tartaric acid are held together by $O-H \cdots O$ hydrogen bonds between the hydroxyl and carbonyl groups, forming a centrosymmetric $R_2^2(10)$ ring (Fig. 2*a* and Table 2). The dimeric unit composed of pyridone molecules is connected to that composed of acid molecules *via* an $O6-H9 \cdots O1$ hydrogen bond on one side, and by $C6-H5 \cdots O3^{iv}$ and $C6-H5 \cdots O5^i$ bifurcated hydrogen bonds on the other side, to afford a molecular tape running along $[01\bar{1}]$ [symmetry codes: (i) $1-x, -y, 1-z$; (iv) $x, y-1, 1+z$; Fig. 2*a* and Table 2]. The molecular tapes are stacked parallel to the c direction through an $O4-H8 \cdots O2^{iii}$ hydrogen bond, resulting in a layer parallel to (202) [symmetry code: (iii) $1+x, y, z$; Figs. 2*b* and 2*c*, and Table 2]. The closest separation between the pyridone rings in the layer is 3.428 Å, and the centre-to-centre separation is 3.735 (3) Å.

The hydrogen-bonding motif formed between the pyridone dimer and the acid moiety is similar to that reported for 2:1 co-

crystals of 2-pyridone and several dicarboxylic acids, namely oxalic acid, succinic acid, adipic acid dihydrate, suberic acid and sebacic acid (Aakerøy *et al.*, 1998), fumaric acid and *meso*-2,3-dibromosuccinic acid (Aakerøy *et al.*, 2000), oxalic acid and *trans*- β -hydromuconic acid (Kashino *et al.*, 2001), and malonic acid and pimeric acid (Edwards *et al.*, 2002). The pyridone dimer is also formed in 1:1 co-crystals of 2-pyridone and some non-centrosymmetric dicarboxylic acids, namely glutaric acid and azelaic acid (Edwards *et al.*, 2002). The pyridone dimer is not formed in the 1:1 co-crystals of 2-pyridone with *trans*-glutaconic acid and L-tartaric acid (Kashino *et al.*, 2001).

In (II) and (III), proton transfer occurs from the acid to the base moiety and a chiral hydrogen tartronate anion is induced in these crystals. In (II), a cation is linked to two neighbouring anions related by a glide plane through the $N1-H1 \cdots O6$, $N1-H1 \cdots O2$, $O1-H3 \cdots O5^v$ and $C4-H4 \cdots O5^v$ hydrogen bonds, to form a zigzag chain running along $[\bar{1}02]$ [symmetry code: (v) $1+x, -y, z-\frac{1}{2}$; Fig. 3*a* and Table 4]. On the other hand, the anions aggregate through $O-H \cdots O$ hydrogen

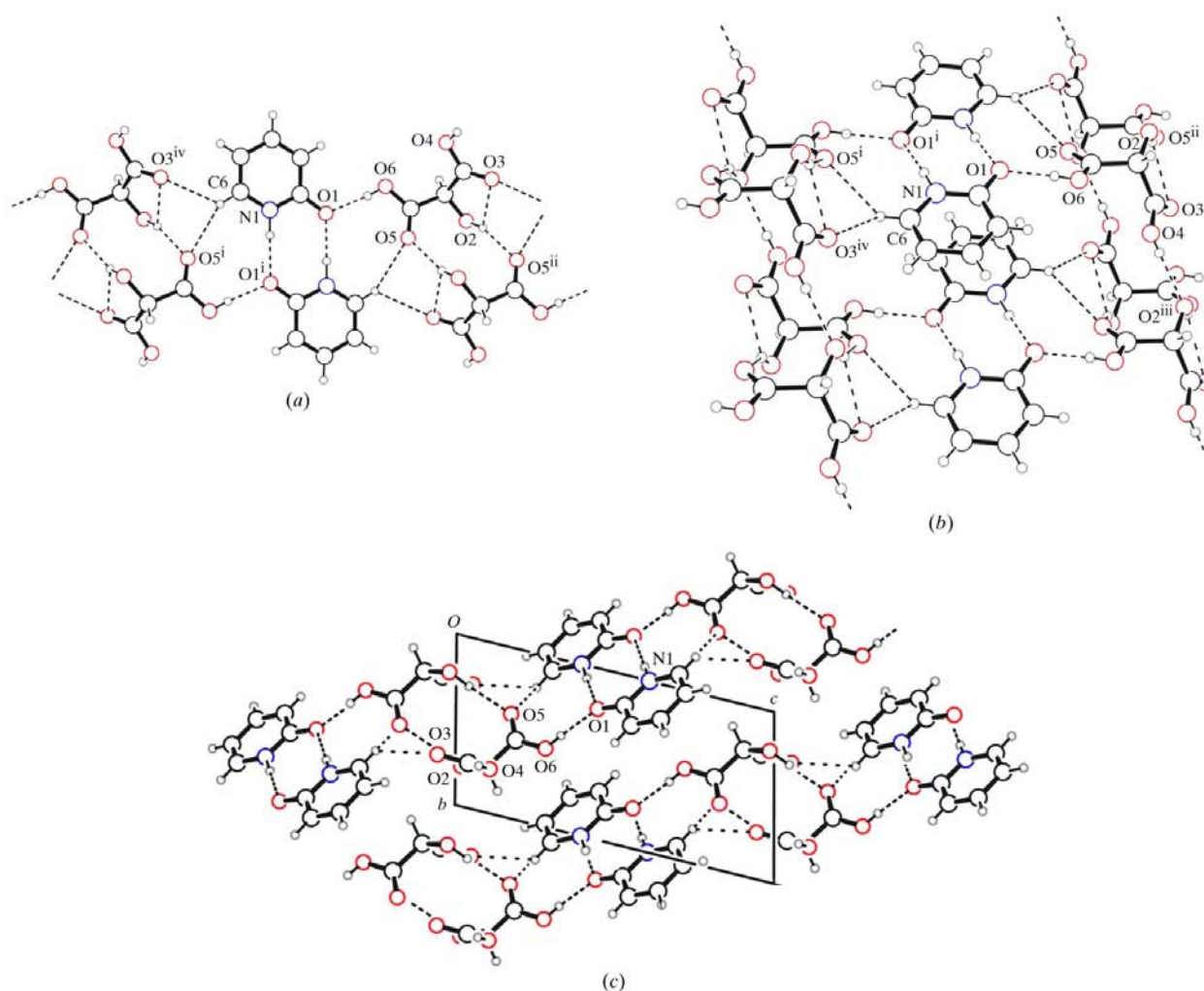


Figure 2

The crystal structure and hydrogen-bonding scheme for (I), showing (a) hydrogen-bonded rings centred on inversion centres, (b) the pyridone molecules linked to the hydrogen-bonded ribbons of the acid molecules and (c) a packing diagram viewed along the a direction, showing the $N-H \cdots O$, $O-H \cdots O$ and $C-H \cdots O$ hydrogen bonds (symmetry codes are as in Table 2).

bonds between the hydroxyl and carboxylate groups ($O2-H8 \cdots O6^{vi}$) and between the carboxyl and hydroxyl groups ($O4-H9 \cdots O2^{vii}$) to form a homochiral plane parallel to (001) [symmetry codes: (vi) $x, 1 + y, z$; (vii) $x - 1, y, z$; Fig. 3*b*]. The planes related by a glide plane are bridged by cations through the $N-H \cdots O$, $O-H \cdots O$ and $C-H \cdots O$ hydrogen bonds mentioned above, resulting in a three-dimensional hydrogen-bonded network. It is noted that such a homochiral plane has also been found in some chiral crystals, namely 3-hydroxypyridinium hydrogen L-tartrate (Tafeenko *et al.*, 1990) and

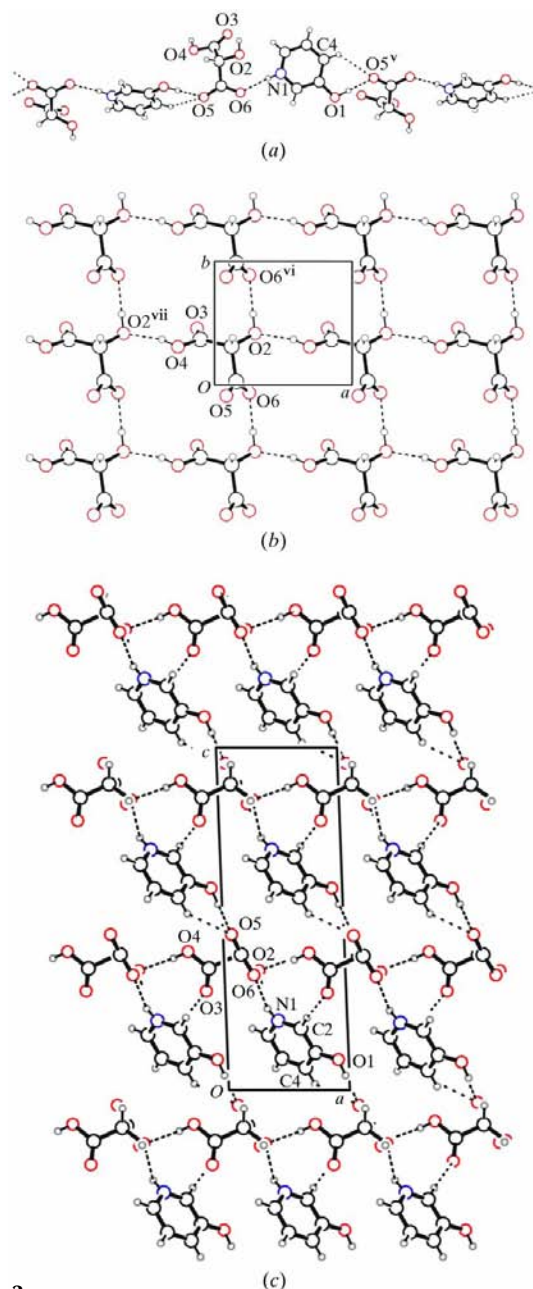


Figure 3

The crystal structure and hydrogen-bonding scheme for (II), showing (a) the hydrogen-bonded chain formed by the anion and cation, (b) the homochiral anions forming a hydrogen-bonded plane across the c axis and (c) a packing diagram viewed along the b direction, showing the $N-H \cdots O$, $O-H \cdots O$ and $C-H \cdots O$ hydrogen bonds (symmetry codes as in Table 4).

3-hydroxypyridinium hydrogen L-maleate (Aakerøy & Nieuwenhuyzen, 1994). In these crystals, planes with the same chirality were bridged by the cations.

In (III), an intramolecular $O-H \cdots O$ hydrogen bond is observed between the carboxyl and carboxylate groups in the anion (Fig. 1 and Table 6). A cation is linked to two neighbouring anions, which are related to each other by a translation along $[10\bar{1}]$, through $O1-H4 \cdots O5^{ix}$, $C5-H5 \cdots O5^{ix}$ and bifurcated $N-H \cdots O$ hydrogen bonds, to form a zigzag chain running along $[\bar{1}21]$ [symmetry codes: (ix) $x - 1, y, 1 + z$; Fig. 4*a* and Table 6]. The chains are linked by an $O2-H8 \cdots O3^x$ hydrogen bond between the hydroxyl and carboxyl groups, resulting in a heterochiral layer parallel to (101) [symmetry code: (x) $\frac{1}{2} + x, 1 - y, \frac{1}{2} + z$; Fig. 4*b*]. The layers are connected *via* a $C7-H7 \cdots O2^{xiii}$ hydrogen bond [symmetry code: (xiii) $1 + x, y, z$; Table 6].

In the crystal structure of tartronic acid, neighbouring molecules related by a 2_1 axis form a molecular chain through $O-H \cdots O$ hydrogen bonds between the carboxyl groups (van Eijck *et al.*, 1965). In the crystal structure of ammonium hydrogen tartrate, hydrogen tartrate anions related by a 2_1 axis form a very short $O-H \cdots O$ hydrogen bond between the carboxyl and carboxylate groups (Taka *et al.*, 1998; Moritani *et al.*, 2001). This is an indication that a strong intermolecular hydrogen bond is formed between the carboxyl and carboxylate groups of the anions. However, in the crystal structures of (I), (II) and (III), no such hydrogen bonding is observed. This fact shows that the hydroxyl group attached to

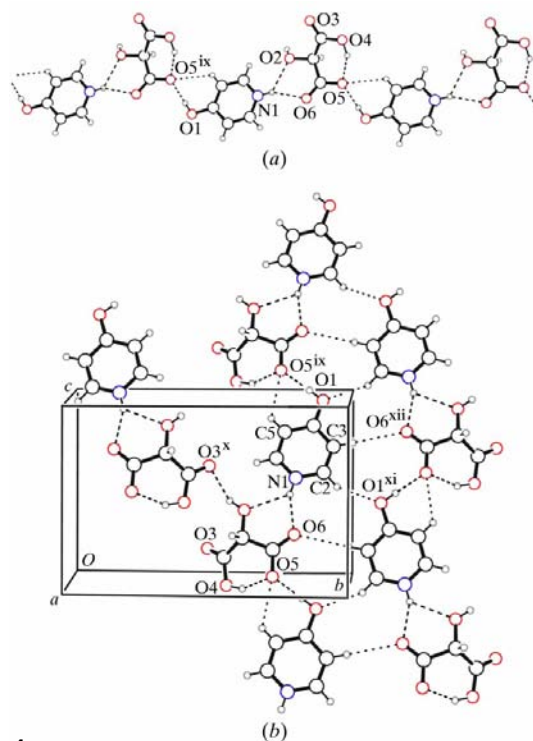


Figure 4

The crystal structure and hydrogen-bonding scheme for (III), showing (a) the hydrogen-bonded chain formed by the anion and cation, and (b) the hydrogen-bonded net formed by the $N-H \cdots O$, $O-H \cdots O$ and $C-H \cdots O$ hydrogen bonds, viewed along the (101) plane (symmetry codes as in Table 6).

the central C atom of tartronic acid or the hydrogen tartronate anion can form stable intermolecular hydrogen bond(s) in place of the carboxyl group.

Experimental

Crystals of (I), (II) and (III) were grown by slow evaporation at room temperature of ethanol solutions of tartronic acid and the respective hydroxypyridine in molar ratios of 1:1.

Compound (I)

Crystal data

$C_5H_5NO \cdot C_3H_4O_5$	$Z = 2$
$M_r = 215.16$	$D_x = 1.549 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 5.879 (4) \text{ \AA}$	Cell parameters from 25 reflections
$b = 6.859 (4) \text{ \AA}$	$\theta = 10.3\text{--}11.5^\circ$
$c = 12.588 (8) \text{ \AA}$	$\mu = 0.14 \text{ mm}^{-1}$
$\alpha = 74.61 (5)^\circ$	$T = 296 \text{ K}$
$\beta = 80.94 (6)^\circ$	Plate, colourless
$\gamma = 70.95 (5)^\circ$	$0.30 \times 0.20 \times 0.08 \text{ mm}$
$V = 461.2 (5) \text{ \AA}^3$	

Data collection

Rigaku AFC-5R diffractometer	$h = -8 \rightarrow 8$
$\omega/2\theta$ scans	$k = -9 \rightarrow 1$
3319 measured reflections	$l = -17 \rightarrow 17$
2698 independent reflections	3 standard reflections
1323 reflections with $I > 2\sigma(I)$	every 97 reflections
$R_{\text{int}} = 0.032$	intensity decay: none
$\theta_{\text{max}} = 30.0^\circ$	

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.065$	$w = 1/[\sigma^2(F_o^2) + (0.08P)^2]$
$wR(F^2) = 0.191$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{\text{max}} = 0.001$
2698 reflections	$\Delta\rho_{\text{max}} = 0.26 \text{ e \AA}^{-3}$
153 parameters	$\Delta\rho_{\text{min}} = -0.24 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$) for (I).

O1—C2	1.279 (7)	O4—C8	1.308 (4)
O2—C7	1.416 (5)	O5—C9	1.208 (7)
O3—C8	1.204 (3)	O6—C9	1.304 (4)
O1—C2—N1	118.7 (3)	O3—C8—C7	121.6 (3)
O1—C2—C3	125.1 (3)	O4—C8—C7	112.5 (2)
O2—C7—C8	110.6 (2)	O5—C9—C7	122.0 (3)
O2—C7—C9	110.9 (3)	O6—C9—C7	112.8 (3)
C8—C7—C9	112.1 (3)		

Table 2

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N1—H1 \cdots O1 ⁱ	0.96 (3)	1.85 (3)	2.809 (4)	174 (4)
O2—H7 \cdots O5 ⁱⁱ	0.93 (4)	1.96 (4)	2.825 (4)	155 (4)
O4—H8 \cdots O2 ⁱⁱⁱ	0.76 (4)	1.97 (4)	2.712 (4)	164 (4)
O6—H9 \cdots O1	0.80 (5)	1.77 (5)	2.569 (4)	175 (3)
O2—H7 \cdots O3	0.93 (4)	2.29 (4)	2.675 (4)	104 (3)
C6—H5 \cdots O3 ^{iv}	0.93	2.50	3.096 (4)	122
C6—H5 \cdots O5 ⁱ	0.93	2.52	3.286 (5)	140

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

Compound (II)

Crystal data

$C_5H_6NO^+ \cdot C_3H_3O_5^-$	$D_x = 1.467 \text{ Mg m}^{-3}$
$M_r = 215.16$	Mo $K\alpha$ radiation
Monoclinic, Pc	Cell parameters from 25 reflections
$a = 5.7563 (11) \text{ \AA}$	$\theta = 11.0\text{--}11.5^\circ$
$b = 5.1871 (7) \text{ \AA}$	$\mu = 0.13 \text{ mm}^{-1}$
$c = 16.3206 (19) \text{ \AA}$	$T = 298 \text{ K}$
$\beta = 92.186 (13)^\circ$	Plate, colourless
$V = 486.95 (13) \text{ \AA}^3$	$0.50 \times 0.50 \times 0.10 \text{ mm}$
$Z = 2$	

Data collection

Rigaku AFC-5R diffractometer	$h = -1 \rightarrow 8$
$\omega/2\theta$ scans	$k = -1 \rightarrow 7$
2353 measured reflections	$l = -23 \rightarrow 23$
1562 independent reflections	3 standard reflections
1394 reflections with $I > 2\sigma(I)$	every 97 reflections
$R_{\text{int}} = 0.016$	intensity decay: 0.2%
$\theta_{\text{max}} = 31.0^\circ$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0945P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.042$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.119$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.00$	$\Delta\rho_{\text{max}} = 0.43 \text{ e \AA}^{-3}$
1562 reflections	$\Delta\rho_{\text{min}} = -0.18 \text{ e \AA}^{-3}$
153 parameters	Extinction correction: <i>SHELXL97</i>
H atoms treated by a mixture of independent and constrained refinement	(Sheldrick, 1997)
	Extinction coefficient: 0.115 (19)

Table 3

Selected geometric parameters (\AA , $^\circ$) for (II).

O1—C3	1.342 (4)	O4—C8	1.320 (2)
O2—C7	1.404 (2)	O5—C9	1.236 (3)
O3—C8	1.203 (3)	O6—C9	1.251 (2)
O1—C3—C2	118.1 (2)	O3—C8—C7	123.47 (17)
O1—C3—C4	124.5 (2)	O4—C8—C7	111.24 (17)
O2—C7—C8	110.62 (15)	O5—C9—C7	118.45 (17)
O2—C7—C9	108.66 (13)	O6—C9—C7	116.53 (16)
C8—C7—C9	108.96 (13)		

Table 4

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1—H3 \cdots O5 ^v	0.90 (5)	1.71 (5)	2.604 (3)	180 (7)
O2—H8 \cdots O6 ^{vi}	0.86 (4)	1.74 (4)	2.5956 (19)	179 (4)
O4—H9 \cdots O2 ^{vii}	0.83 (5)	1.79 (5)	2.602 (2)	165 (4)
N1—H1 \cdots O6	0.83 (5)	1.88 (5)	2.682 (3)	162 (5)
C2—H2 \cdots O3 ^{viii}	0.93	2.50	3.422 (3)	174
C4—H4 \cdots O5 ^v	0.93	2.56	3.206 (4)	127

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

Compound (III)

Crystal data

$C_5H_6NO^+ \cdot C_3H_3O_5^-$	$D_x = 1.574 \text{ Mg m}^{-3}$
$M_r = 215.16$	Mo $K\alpha$ radiation
Monoclinic, Pn	Cell parameters from 25 reflections
$a = 4.0607 (13) \text{ \AA}$	$\theta = 10.8\text{--}11.4^\circ$
$b = 13.144 (3) \text{ \AA}$	$\mu = 0.14 \text{ mm}^{-1}$
$c = 8.696 (2) \text{ \AA}$	$T = 298 \text{ K}$
$\beta = 101.99 (2)^\circ$	Plate, colourless
$V = 454.0 (2) \text{ \AA}^3$	$0.46 \times 0.26 \times 0.17 \text{ mm}$
$Z = 2$	

Data collection

Rigaku AFC-5R diffractometer
 $\omega/2\theta$ scans
 Absorption correction: ψ scan
 (North *et al.*, 1968)
 $T_{\min} = 0.942$, $T_{\max} = 0.977$
 1985 measured reflections
 1341 independent reflections
 861 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.038$
 $\theta_{\text{max}} = 30.0^\circ$
 $h = -1 \rightarrow 5$
 $k = -1 \rightarrow 18$
 $l = -12 \rightarrow 12$
 3 standard reflections
 every 97 reflections
 intensity decay: 2.0%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.051$
 $wR(F^2) = 0.133$
 $S = 1.00$
 1341 reflections
 136 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0521P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.21 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.19 \text{ e } \text{\AA}^{-3}$

Table 5

Selected geometric parameters (\AA , $^\circ$) for (III).

O1—C4	1.336 (5)	O4—C8	1.318 (5)
O2—C7	1.400 (4)	O5—C9	1.291 (5)
O3—C8	1.196 (5)	O6—C9	1.215 (5)
O1—C4—C3	119.5 (3)	O3—C8—C7	123.0 (3)
O1—C4—C5	122.0 (3)	O4—C8—C7	116.3 (4)
O2—C7—C8	111.3 (3)	O6—C9—C7	120.9 (3)
O2—C7—C9	109.8 (3)	O5—C9—C7	112.6 (4)
C8—C7—C9	113.7 (3)		

Table 6

Hydrogen-bonding geometry (\AA , $^\circ$) for (III).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1—H4 \cdots O5 ^{ix}	0.82	1.74	2.562 (4)	177
O2—H8 \cdots O3 ^x	0.82	2.05	2.808 (4)	153
O4—H9 \cdots O5	0.82	1.79	2.538 (4)	151
N1—H1 \cdots O2	0.86	2.25	2.862 (5)	128
N1—H1 \cdots O6	0.86	2.07	2.872 (5)	156
C2—H2 \cdots O1 ^{xi}	0.93	2.29	3.149 (5)	153
C3—H3 \cdots O6 ^{xii}	0.93	2.57	3.497 (5)	173
C5—H5 \cdots O5 ^{ix}	0.93	2.54	3.179 (6)	127
C7—H7 \cdots O2 ^{xiii}	0.98	2.49	3.369 (6)	149

Symmetry codes: (ix) $x - 1, y, 1 + z$; (x) $\frac{1}{2} + x, 1 - y, \frac{1}{2} + z$; (xi) $\frac{1}{2} + x, 2 - y, z - \frac{1}{2}$; (xii) $x - \frac{1}{2}, 2 - y, \frac{1}{2} + z$; (xiii) $1 + x, y, z$.

For all three title compounds, H atoms attached to C atoms were treated as riding, with C—H = 0.98 (methine H) or 0.93 \AA (aromatic H), and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. For (I) and (II), H atoms involved in O—H \cdots O and N—H \cdots O hydrogen bonds were refined. For (III), the O—H distances and C—O—H angles were fixed at 0.82 \AA and 109.5 $^\circ$, respectively, while the torsion angles around the C—O bonds were refined, with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$. H atoms attached to N atoms were constrained in the riding model, with N—H = 0.86 \AA and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$. For (II) and (III), Friedel opposites were merged.

For all three title compounds, data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation,

1990); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN for Windows* (Molecular Structure Corporation, 1997–1999); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *TEXSAN for Windows*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1496). Services for accessing these data are described at the back of the journal.

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