

## Hydrogen-bonded complexes of 2-pyridone with centrosymmetric and non-centrosymmetric dicarboxylic acids

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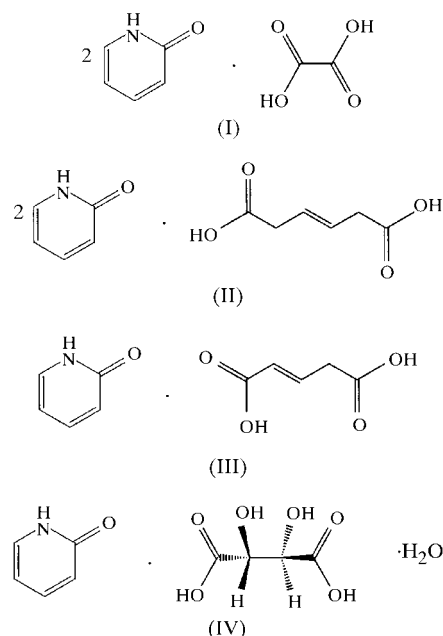
2-Pyridone (2-oxopyrimidine) forms hydrogen-bonded complexes with dicarboxylic acids, the molar ratio of 2-pyridone/dicarboxylic acid being 2:1 for the complexes with oxalic acid (ethanedioic acid),  $2C_5H_5NO \cdot C_2H_2O_4$ , (I), and *trans*- $\beta$ -hydro-muconic acid (*trans*-hex-3-enedioic acid),  $2C_5H_5NO \cdot C_6H_8O_4$ , (II), and 1:1 for the complexes with *trans*-glutaconic acid (*trans*-pent-2-enedioic acid),  $C_5H_5NO \cdot C_5H_6O_4$ , (III), and L-tartaric acid (L-2,3-dihydroxybutanedioic acid),  $C_5H_5NO \cdot C_4H_6O_6 \cdot H_2O$ , (IV). Common features in the hydrogen-bonding patterns were found for the centrosymmetric and non-centrosymmetric acids, respectively. The 2-pyridone molecule takes the lactam form in these crystals.

### Comment

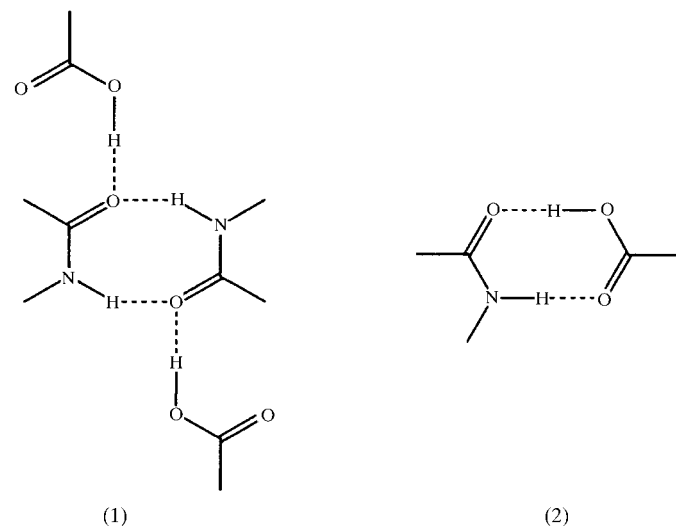
2-Pyridone has been extensively studied because it exhibits lactam–lactim tautomerism, and the lactam group can be regarded as a model of *cis*-peptides and of the purine and pyrimidine bases of nucleic acids (Yang & Craven, 1998; Field & Hillier, 1987). The crystal structure of 2-pyridone has been determined from high-resolution X-ray data at 123 K, and the molecule is in the lactam form (Yang & Craven, 1998). In the present study, the title complexes, (I)–(IV), have been prepared with the expectation that the lactam group of the 2-pyridone could be a building unit of a supramolecular synthon, and so the hydrogen-bonding patterns and the geometries of the 2-pyridone moiety in the four complexes have been elucidated.

The hydrogen-bond patterns in (I) and (II) are shown in Figs. 1 and 2, respectively. In the crystals of (I) and (II), a molecule of the acid has a centre of symmetry. Two molecules of 2-pyridone related by an inversion centre form a dimer *via* an N–H...O hydrogen bond. The dimeric unit and the acid molecule are connected by an O–H...O hydrogen bond

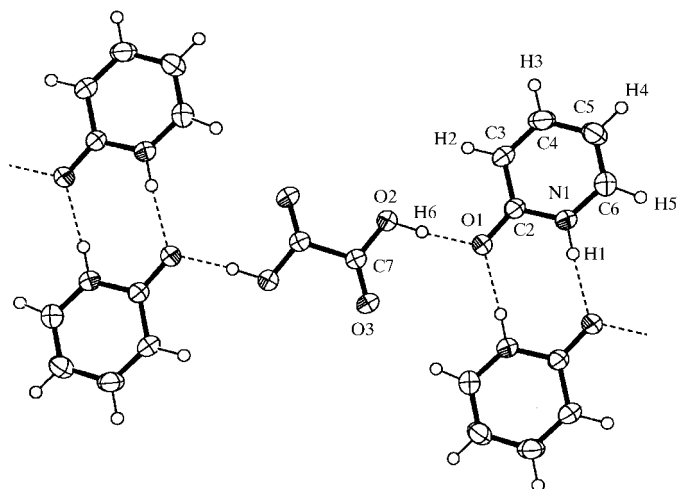
between the carboxyl group and the O atom of 2-pyridone, to form a chain (Figs. 1 and 2; Tables 2 and 4). In (I), the chains are arranged side by side to form a sheet along (111). These sheets are stacked along the *c* axis, with short contacts  $O3 \cdots C7^{ii} = 3.302(2) \text{ \AA}$  and  $O3 \cdots C7^{iii} = 3.307(2) \text{ \AA}$  [symmetry codes: (ii)  $1 - x, -y, -z - 1$ ; (iii)  $x, y, z - 1$ ]. In the chain of (II), a C–H...O interaction is observed (Table 4). The chains are arranged around the twofold screw axis, with normal van der Waals contacts and no sheet structure is



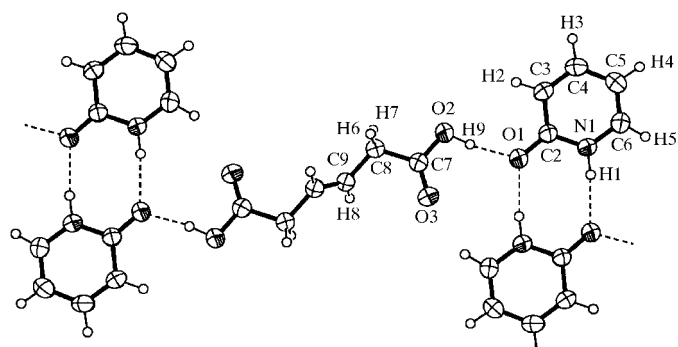
formed. In spite of the difference in the packing mode of the chains in (I) and (II), the patterns of the hydrogen bonds in the chains show a common feature in these crystals. Thus, hydrogen-bond scheme (1), as found in (I) and (II), can be recognized as the supramolecular synthon (Desiraju, 1995) possible in complexes of 2-pyridone/centrosymmetric dicarboxylic acid in the ratio 2:1.



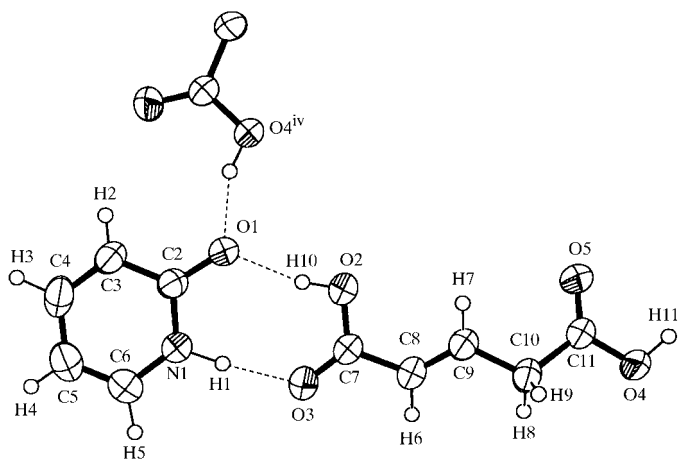
The acid molecules in (III) and (IV) have no centre of symmetry (Figs. 3 and 4). In both crystals, one carboxyl group


**Figure 1**

A molecular view of (I) showing the hydrogen-bonding pattern and the atomic numbering scheme for the asymmetric unit. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. Hydrogen bonds are indicated by dashed lines.

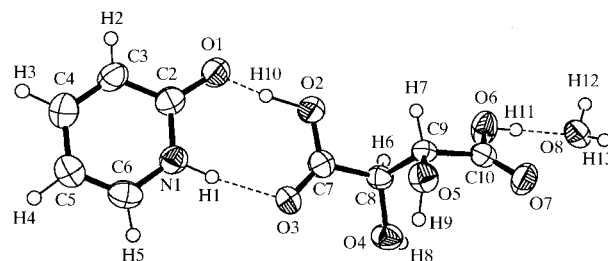

**Figure 2**

A molecular view of (II) showing the hydrogen-bonding pattern and the atomic numbering scheme for the asymmetric unit. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. Hydrogen bonds are indicated by dashed lines.

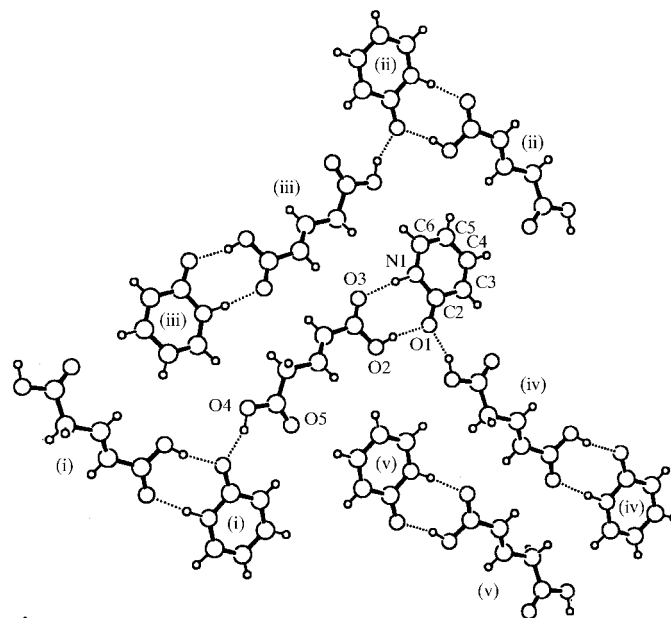

**Figure 3**

A molecular view of (III) showing the hydrogen-bonding pattern and the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. Hydrogen bonds are indicated by dashed lines.

forms an O—H...O and an N—H...O hydrogen bond with a 2-pyridone molecule (Tables 6 and 8). The O2—H10...O1 hydrogen bond in (IV) is short, the O2—H10 bond length being elongated, as found for a short non-centrosymmetric O—H...O hydrogen bond in ammonium hydrogen tartrate [O—H 1.18 (3) Å and O...O 2.443 (2) Å; Taka *et al.*, 1998]. It could not be confirmed whether atom H10 is disordered, because the difference Fourier map for H10 showed a single peak. In (III), the other carboxyl group forms an O—H...O hydrogen bond with a 2-pyridone molecule at  $(x - 1, \frac{1}{2} - y, \frac{1}{2} + z)$  to form a zigzag chain, and the chains form a sheet along (102) *via* C—H...O interactions (Fig. 5 and Table 6). The other carboxyl group in (IV) forms a hydrogen bond with a water molecule. The water molecule forms bifurcated hydrogen bonds with the acid molecules related by a translation along *a* and by the twofold screw axes along the *a* axis, forming a sheet parallel to (040) (Fig. 6 and Table 8). Between the sheets related by the twofold screw axes along the *c* axis,


**Figure 4**

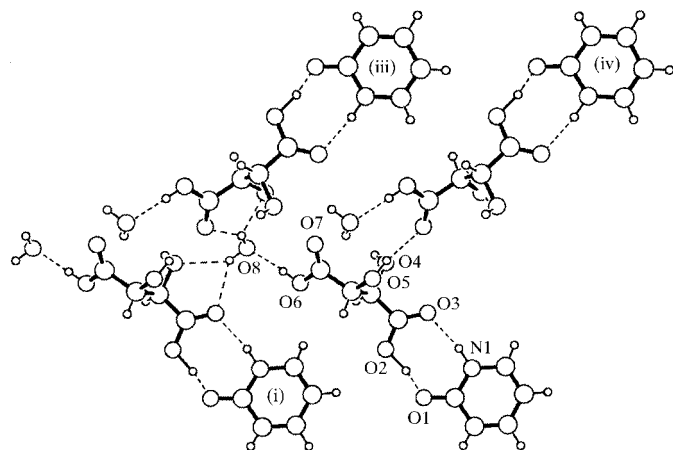
A molecular view of (IV) showing the hydrogen-bonding pattern and the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. Hydrogen bonds are indicated by dashed lines.


**Figure 5**

The molecular arrangement along (201) of (III), viewed down the *c* axis. The *a* axis points to the right and the *b* axis points upwards. Hydrogen bonds are shown by dotted lines. [Symmetry codes: (i)  $x - 1, \frac{1}{2} - y, \frac{1}{2} + z$ ; (ii)  $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$ ; (iii)  $-x, 1 - y, 1 - z$ ; (iv)  $1 + x, \frac{1}{2} - y, z - \frac{1}{2}$ ; (v)  $1 - x, y - \frac{1}{2}, \frac{1}{2} - z$ .]

there is a C—H···O interaction and some short contacts between overlapping 2-pyridone molecules: C9—H7 0.976 (12), H7···O1<sup>v</sup> 2.563 (13), C9···O1<sup>v</sup> 3.255 (2) Å, and C9—H7···O1<sup>v</sup> 127.9 (10)°; O3···C4<sup>vi</sup> 3.186 (3), O1···C6<sup>vii</sup> 3.373 (3), N1···C6<sup>vii</sup> 3.280 (3) and C2···C6<sup>vii</sup> 3.302 Å [symmetry codes: (v)  $\frac{1}{2} - x, 1 - y, z + \frac{1}{2}$ ; (vi)  $\frac{3}{2} - x, 1 - y, z - \frac{1}{2}$ ; (vii)  $\frac{3}{2} - x, 1 - y, \frac{1}{2} + z$ ]. Because hydrogen-bond scheme (2) is found in both (III) and (IV), it can be recognized as the supramolecular synthon possible in complexes of 2-pyridone/non-centrosymmetric dicarboxylic acid in the ratio 1:1.

The geometries of the 2-pyridone moieties observed in compounds (I)–(IV) (Tables 1, 3, 5 and 7) show a common feature characteristic of the lactam form, as found in the crystal of 2-pyridone (Yang & Craven, 1998). The C—C distance [1.544 (2) Å] of oxalic acid in (I) is rather long for a Csp<sup>2</sup>—Csp<sup>2</sup> bond, as observed in dimorphs of oxalic acid [1.537 (1) Å; Derissen & Smith, 1974], and close to that determined for oxalic acid dihydrate at 15 K [1.5423 (5) Å; Zobel *et al.*, 1992].



**Figure 6**

A view down the *c* axis showing the formation of a double layer in (IV) through the hydrogen bonds involving water molecules. The *a* axis points to the right and the *b* axis points upwards. Hydrogen bonds are shown by dashed lines. [Symmetry codes: (i)  $x - 1, y, z$ ; (iii)  $x - \frac{1}{2}, \frac{3}{2} - y, 1 - z$ ; (iv)  $\frac{1}{2} + x, \frac{3}{2} - y, 1 - z$ .]

## Experimental

Crystals of the four title compounds were grown by slow evaporation from ethanol solutions of 2-pyridone and the respective dicarboxylic acid, with molar ratios of 1:1. Commercially available L-tartaric acid (Aldrich) was used for the preparation of (IV).

### Compound (I)

#### Crystal data

2C<sub>5</sub>H<sub>5</sub>NO·C<sub>2</sub>H<sub>2</sub>O<sub>4</sub>  
*M<sub>r</sub>* = 280.24  
 Triclinic, *P*1̄  
*a* = 9.162 (3) Å  
*b* = 9.898 (3) Å  
*c* = 3.7672 (17) Å  
 $\alpha$  = 98.86 (4)°  
 $\beta$  = 97.66 (3)°  
 $\gamma$  = 109.61 (3)°  
*V* = 311.6 (2) Å<sup>3</sup>

*Z* = 1  
*D<sub>x</sub>* = 1.493 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
 Cell parameters from 18 reflections  
 $\theta$  = 9.4–11.5°  
 $\mu$  = 0.12 mm<sup>-1</sup>  
*T* = 298 K  
 Prismatic, colourless  
 0.50 × 0.38 × 0.35 mm

#### Data collection

Rigaku AFC-5R diffractometer  
 $\omega/2\theta$  scans  
 1645 measured reflections  
 1428 independent reflections  
 1336 reflections with *I* > 0.1σ(*I*)  
*R*<sub>int</sub> = 0.006  
 $\theta_{\max}$  = 27.5°

*h* = -11 → 11  
*k* = -12 → 12  
*l* = 0 → 4  
 3 standard reflections  
 every 97 reflections  
 intensity decay: <0.9%

#### Refinement

Refinement on *F*<sup>2</sup>  
*R*(*F*) = 0.059  
*wR*(*F*<sup>2</sup>) = 0.098  
*S* = 1.98  
 1336 reflections  
 115 parameters

All H-atom parameters refined  
 $w = 1/[\sigma^2(F_o) + 0.00031|F_o|^2]$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.24 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\min} = -0.22 \text{ e } \text{Å}^{-3}$

**Table 1**

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

O1—C2	1.2683 (15)	C3—C4	1.3573 (19)
N1—C2	1.3573 (16)	C4—C5	1.404 (2)
N1—C6	1.3554 (17)	C5—C6	1.3507 (19)
C2—C3	1.4191 (16)		
C2—N1—C6	123.88 (11)	C2—C3—C4	120.89 (12)
O1—C2—N1	118.99 (10)	C3—C4—C5	120.49 (12)
O1—C2—C3	125.19 (11)	C4—C5—C6	118.39 (12)
N1—C2—C3	115.81 (11)	N1—C6—C5	120.53 (12)

**Table 2**

Hydrogen-bonding 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>
O2—H6···O1	0.96 (2)	1.62 (2)	2.5774 (16)	174.0 (19)
N1—H1···O1 <sup>i</sup>	0.91 (2)	1.89 (2)	2.7967 (16)	173.1 (15)

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

### Compound (II)

#### Crystal data

2C<sub>5</sub>H<sub>5</sub>NO·C<sub>6</sub>H<sub>8</sub>O<sub>4</sub>  
*M<sub>r</sub>* = 334.33  
 Monoclinic, *P*2<sub>1</sub>/*n*  
*a* = 9.824 (3) Å  
*b* = 5.5720 (17) Å  
*c* = 15.556 (5) Å  
 $\beta$  = 101.31 (3)°  
*V* = 835.0 (5) Å<sup>3</sup>  
*Z* = 2

*D<sub>x</sub>* = 1.330 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
 Cell parameters from 25 reflections  
 $\theta$  = 10–11°  
 $\mu$  = 0.10 mm<sup>-1</sup>  
*T* = 298 K  
 Prismatic, colourless  
 0.43 × 0.35 × 0.20 mm

#### Data collection

Rigaku AFC-5R diffractometer  
 $\omega/2\theta$  scans  
 2802 measured reflections  
 2429 independent reflections  
 2187 reflections with *I* > 0.1σ(*I*)  
*R*<sub>int</sub> = 0.009  
 $\theta_{\max}$  = 30°

*h* = 0 → 13  
*k* = 0 → 7  
*l* = -21 → 21  
 3 standard reflections  
 every 97 reflections  
 intensity decay: <0.1%

#### Refinement

Refinement on *F*<sup>2</sup>  
*R*(*F*) = 0.059  
*wR*(*F*<sup>2</sup>) = 0.102  
*S* = 1.93  
 2187 reflections  
 146 parameters  
 All H-atom parameters refined  
 $w = 1/[\sigma^2(F_o) + 0.00022|F_o|^2]$

$(\Delta/\sigma)_{\max} = 0.010$   
 $\Delta\rho_{\max} = 0.26 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\min} = -0.30 \text{ e } \text{Å}^{-3}$   
 Extinction correction: Zachariasen (1967)  
 Extinction coefficient:  
 1.6 (4) × 10<sup>-6</sup>

**Table 3**  
Selected geometric parameters (Å, °) for (II).

O1—C2	1.2639 (13)	C3—C4	1.3488 (18)
N1—C2	1.3652 (15)	C4—C5	1.395 (2)
N1—C6	1.3540 (16)	C5—C6	1.3526 (19)
C2—C3	1.4191 (16)		
C2—N1—C6	123.57 (10)	C2—C3—C4	120.86 (11)
O1—C2—N1	119.22 (10)	C3—C4—C5	120.90 (11)
O1—C2—C3	125.04 (10)	C4—C5—C6	118.54 (12)
N1—C2—C3	115.74 (10)	N1—C6—C5	120.38 (12)

**Table 4**  
Hydrogen-bonding geometry (Å, °) for (II).

D—H...A	D—H	H...A	D...A	D—H...A
O2—H9...O1	0.94 (2)	1.68 (2)	2.6078 (14)	171.1 (16)
N1—H1...O1 <sup>i</sup>	0.93 (1)	1.93 (1)	2.8465 (15)	172.8 (12)
C6—H5...O3 <sup>i</sup>	0.93 (1)	2.30 (1)	3.1492 (19)	150.9 (11)

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

### Compound (III)

#### Crystal data

C<sub>5</sub>H<sub>5</sub>NO·C<sub>5</sub>H<sub>6</sub>O<sub>4</sub>  
M<sub>r</sub> = 225.20  
Monoclinic, P2<sub>1</sub>/c  
a = 7.889 (3) Å  
b = 16.839 (10) Å  
c = 8.288 (2) Å  
β = 100.49 (3)°  
V = 1082.5 (7) Å<sup>3</sup>  
Z = 4

D<sub>x</sub> = 1.382 Mg m<sup>-3</sup>  
Mo Kα radiation  
Cell parameters from 25 reflections  
θ = 10.8–11.4°  
μ = 0.11 mm<sup>-1</sup>  
T = 298 K  
Prismatic, colourless  
0.43 × 0.40 × 0.27 mm

#### Data collection

Rigaku AFC-5R diffractometer  
ω/2θ scans  
2298 measured reflections  
2128 independent reflections  
1848 reflections with I > 0.1σ(I)  
R<sub>int</sub> = 0.016  
θ<sub>max</sub> = 26°

h = 0 → 9  
k = 0 → 20  
l = -10 → 10  
3 standard reflections  
every 97 reflections  
intensity decay: <0.3%

#### Refinement

Refinement on F<sup>2</sup>  
R(F) = 0.079  
wR(F<sup>2</sup>) = 0.088  
S = 1.46  
1848 reflections  
189 parameters

All H-atom parameters refined  
w = 1/[σ<sup>2</sup>(F<sub>o</sub>) + 0.00022|F<sub>o</sub>|<sup>2</sup>]  
(Δ/σ)<sub>max</sub> = 0.010  
Δρ<sub>max</sub> = 0.19 e Å<sup>-3</sup>  
Δρ<sub>min</sub> = -0.25 e Å<sup>-3</sup>

**Table 5**  
Selected geometric parameters (Å, °) for (III).

O1—C2	1.271 (2)	C3—C4	1.360 (3)
N1—C2	1.359 (2)	C4—C5	1.395 (3)
N1—C6	1.357 (3)	C5—C6	1.335 (3)
C2—C3	1.407 (2)		
C2—N1—C6	123.72 (17)	C2—C3—C4	120.25 (19)
O1—C2—N1	118.53 (15)	C3—C4—C5	120.8 (2)
O1—C2—C3	125.31 (17)	C4—C5—C6	118.9 (2)
N1—C2—C3	116.13 (17)	N1—C6—C5	120.2 (2)

**Table 6**  
Hydrogen-bonding geometry (Å, °) for (III).

D—H...A	D—H	H...A	D...A	D—H...A
O2—H10...O1	0.89 (3)	1.78 (3)	2.658 (2)	168 (2)
O4—H11...O1 <sup>i</sup>	0.92 (3)	1.72 (3)	2.601 (2)	158 (3)
N1—H1...O3	1.02 (2)	1.80 (2)	2.811 (2)	168.8 (18)
C4—H3...O5 <sup>ii</sup>	0.97 (2)	2.41 (2)	3.339 (3)	160.8 (17)
C6—H5...O4 <sup>iii</sup>	1.01 (2)	2.47 (2)	3.457 (3)	165.2 (17)
C3—H2...O5 <sup>iv</sup>	0.92 (2)	2.56 (2)	3.397 (3)	150.8 (15)

Symmetry codes: (i) x - 1, ½ - y, ½ + z; (ii) 1 - x, ½ + y, ½ - z; (iii) -x, 1 - y, 1 - z; (iv) 1 + x, ½ - y, z - ½.

### Compound (IV)

#### Crystal data

C<sub>5</sub>H<sub>5</sub>NO·C<sub>4</sub>H<sub>6</sub>O<sub>6</sub>·H<sub>2</sub>O  
M<sub>r</sub> = 263.20  
Orthorhombic, P2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>  
a = 8.528 (5) Å  
b = 20.668 (8) Å  
c = 6.5438 (15) Å  
V = 1153.4 (7) Å<sup>3</sup>  
Z = 4  
D<sub>x</sub> = 1.516 Mg m<sup>-3</sup>

Mo Kα radiation  
Cell parameters from 25 reflections  
θ = 10.8–11.5°  
μ = 0.14 mm<sup>-1</sup>  
T = 295 K  
Prismatic, colourless  
0.53 × 0.28 × 0.20 mm

#### Data collection

Rigaku AFC-5R diffractometer  
ω/2θ scans  
Absorption correction: ψ scan  
(North *et al.*, 1968)  
T<sub>min</sub> = 0.952, T<sub>max</sub> = 0.999  
2660 measured reflections  
2303 independent reflections  
2064 reflections with I > 0.1σ(I)

R<sub>int</sub> = 0.011  
θ<sub>max</sub> = 32°  
h = 0 → 12  
k = 0 → 30  
l = -1 → 9  
3 standard reflections  
every 97 reflections  
intensity decay: <0.5%

**Table 7**  
Selected geometric parameters (Å, °) for (IV).

O1—C2	1.262 (2)	C3—C4	1.350 (3)
N1—C2	1.368 (2)	C4—C5	1.391 (3)
N1—C6	1.365 (3)	C5—C6	1.340 (3)
C2—C3	1.409 (3)		
C2—N1—C6	123.18 (19)	C2—C3—C4	121.5 (2)
O1—C2—N1	119.92 (18)	C3—C4—C5	120.8 (2)
O1—C2—C3	124.78 (19)	C4—C5—C6	118.4 (2)
N1—C2—C3	115.30 (18)	N1—C6—C5	120.8 (2)

**Table 8**  
Hydrogen-bonding geometry (Å, °) for (IV).

D—H...A	D—H	H...A	D...A	D—H...A
O2—H10...O1	1.20 (2)	1.30 (2)	2.487 (2)	168.2 (18)
N1—H1...O3	0.96 (2)	1.92 (2)	2.878 (2)	171.6 (15)
O6—H11...O8	0.84 (2)	1.79 (2)	2.625 (2)	173 (2)
O5—H9...O4	0.95 (2)	2.36 (3)	2.781 (2)	106.7 (19)
O8—H12...O3 <sup>i</sup>	0.86 (3)	2.22 (3)	2.914 (2)	138 (2)
O8—H12...O4 <sup>i</sup>	0.86 (3)	2.23 (3)	2.991 (3)	147 (2)
O4—H8...O8 <sup>ii</sup>	0.80 (2)	1.98 (2)	2.772 (2)	171 (2)
O8—H13...O7 <sup>iii</sup>	0.80 (2)	2.21 (2)	2.902 (2)	145 (2)
O8—H13...O5 <sup>iii</sup>	0.80 (2)	2.33 (2)	2.980 (2)	140 (2)
O5—H9...O7 <sup>iv</sup>	0.95 (2)	1.97 (2)	2.846 (2)	152 (2)

Symmetry codes: (i) x - 1, y, z; (ii) ½ + x, ¾ - y, -z; (iii) x - ½, ¾ - y, 1 - z; (iv) ½ + x, ¾ - y, 1 - z.

## Refinement

Refinement on $F^2$	$(\Delta/\sigma)_{\max} = 0.010$
$R(F) = 0.044$	$\Delta\rho_{\max} = 0.32 \text{ e } \text{\AA}^{-3}$
$wR(F^2) = 0.044$	$\Delta\rho_{\min} = -0.31 \text{ e } \text{\AA}^{-3}$
$S = 1.81$	Extinction correction:
2064 reflections	Zachariasen (1967)
216 parameters	Extinction coefficient:
All H-atom parameters refined	$1.68 (12) \times 10^{-6}$
$w = 1/[\sigma^2(F_o) + 0.00001 F_o ^2]$	

All H atoms were located from difference Fourier maps and were refined isotropically. The ranges of  $U_{\text{iso}}$  values for the H atoms were 0.051 (3)–0.084 (4)  $\text{\AA}^2$  for (I), 0.054 (3)–0.085 (4)  $\text{\AA}^2$  for (II), 0.055 (5)–0.154 (10)  $\text{\AA}^2$  for (III) and 0.015 (3)–0.146 (10)  $\text{\AA}^2$  for (IV). Values larger than 0.1  $\text{\AA}^2$  were observed for H10 and H11 in (III), and for H9, H10 and H12 in (IV). The least-squares refinement of (IV) was carried out assuming the chirality of L-tartaric acid and removing redundant diffraction data.

For all compounds, data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1990); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1999); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *TEXSAN*; molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *TEXSAN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1582). Services for accessing these data are described at the back of the journal.

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