

Lists of structure factors and anisotropic displacement parameters have been deposited with the IUCr (Reference: LI1092). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

- Bernstein, J. & Leiserowitz, L. (1972). *Isr. J. Chem.* **10**, 601–612.  
 CSSR (1993). *Crystal Structure Search and Retrieval Instruction Manual*. Chemical Classes 1 and 2. SERC, Daresbury Laboratory, Warrington, England.  
 Davies, K. (1983). *SNOOPI. CHEMGRAF User Guide*. The CHEMGRAF Suite. Chemical Crystallography Laboratory, Oxford, England.  
 Kashino, S. & Haisa, M. (1980). *Acta Cryst.* **B36**, 346–353.  
 Ladd, M. F. C. & Palmer, R. A. (1993). *Structure Determination by X-ray Crystallography*, 3rd ed., p. 434. New York: Plenum.  
 Martindale (1993). *The Extra Pharmacopoeia*. 30th ed. London: Pharmaceutical Press.  
 Rabinovich, D. & Schmidt, G. M. J. (1967). *J. Chem. Soc. B*, pp. 286–289.  
 Richards, R. M. E. (1972). *Pharm. J.* **2**, 91.  
 Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.  
 Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. Univ. of Göttingen, Germany.  
 Speakman, J. C. (1972). *Structure and Bonding*, Vol. 12, pp. 141–199. Berlin: Springer-Verlag.

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## $\gamma$ -Resorcylic Acid, its Monohydrate and its Pyridinium Complex

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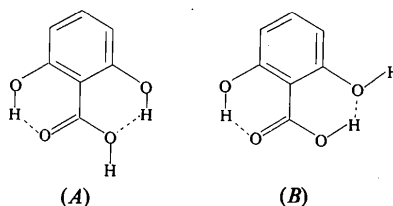
## Abstract

The crystal structures of  $\gamma$ -resorcylic acid (2,6-dihydroxybenzoic acid), C<sub>7</sub>H<sub>6</sub>O<sub>4</sub>, (I),  $\gamma$ -resorcylic acid monohydrate, C<sub>7</sub>H<sub>6</sub>O<sub>4</sub>·H<sub>2</sub>O, (II), and pyridinium  $\gamma$ -resorcyate, C<sub>5</sub>H<sub>5</sub>N<sup>+</sup>·C<sub>7</sub>H<sub>5</sub>O<sub>4</sub><sup>−</sup>, (III), have been determined. In (I) and (II), the carboxyl group of the  $\gamma$ -resorcylic acid molecule is in an *anti* conformation and the carboxylic OH group is involved as a donor in an O—H...O intramolecular hydrogen bond. This bond is part of an infinite hydrogen-bonded chain with mixed  $\sigma$ - and  $\pi$ -bond cooperativity. Structure (II) is disordered and may be considered to result from the superposition

of two lower-symmetry structures related by a mirror plane. Structure (III) consists of discrete complex units formed from the anion and cation by means of an N—H...O hydrogen bond.

## Comment

$\gamma$ -Resorcylic acid is one of the strongest carboxylic acids ( $pK_a = 1.3$ ). It is also an interesting example of a molecule with two possible intramolecular hydrogen-bond systems, one with two phenolic OH groups and another with one phenolic and one carboxylic OH group involved as donors in intramolecular hydrogen bonds; these two forms, A and B, are shown in the scheme below.



Solution <sup>1</sup>H NMR studies of this acid in the presence of bases of different proton-acceptor ability show that structure A prevails and that an intermolecular hydrogen-bond interaction with the base occurs through the carboxylic OH group (Golubev & Denisov, 1992). Some strengthening of the intramolecular hydrogen bonds was observed as the proton-acceptor ability of the base increased. In order to study the hydrogen-bond interactions of  $\gamma$ -resorcylic acid in the solid state, it was recrystallized from solvents of different proton-acceptor and proton-donor ability. The only forms obtained were anhydrous  $\gamma$ -resorcylic acid,  $\gamma$ -resorcylic acid monohydrate and, where a strong base was present in the crystallization mixture, an A<sup>−</sup>...H—B<sup>+</sup> complex. This paper presents X-ray structure analyses of  $\gamma$ -resorcylic acid (I), its monohydrate (II) and its pyridinium salt (III).

The carboxyl group of the  $\gamma$ -resorcylic acid molecule in (I) is in an *anti* conformation as a result of the intramolecular hydrogen bond with the hydroxyl group at O4. Unlike the structure in solution, the molecular structure in the crystal corresponds to form B, in which one phenolic OH group acts as an intra- and the other as intermolecular hydrogen-bond donor. The molecules are linked by hydrogen bonds to form infinite chains along [011]. Intra- and intermolecular hydrogen bonds form a chain with alternating  $\sigma$ - and  $\pi$ -bond cooperativity (Jeffrey & Saenger, 1991) (Fig. 1). The energy of the H atoms involved in the hydrogen bonds along this chain can be considered to be characterized by double-minimum potentials. A concerted proton-transfer process induced along this chain would cause a transformation from form B to form A. The two minima are of different energies and only molecules of form B

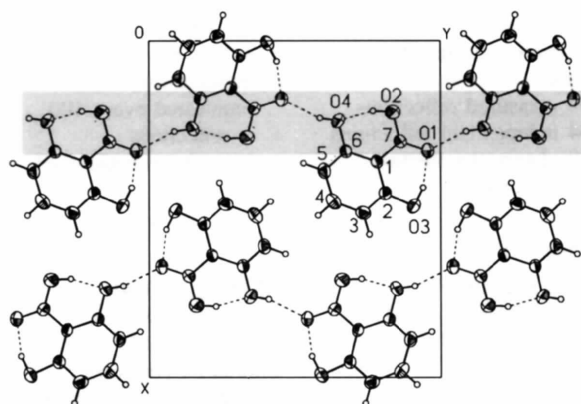


Fig. 1. View of the crystal packing of (I) along [001] showing the hydrogen-bonded chains of alternating  $\sigma$ - and  $\pi$ -bond cooperativity. Hydrogen bonds are shown by dotted lines. Displacement ellipsoids are plotted at the 50% probability level.

are found in the crystal. The hydrogen-bond geometry is given in Table 3.

The observed structure of (II) may be considered to be either disordered  $\gamma$ -resorcylic acid monohydrate or hydronium  $\gamma$ -resorcyate in which complete proton transfer from the carboxylic acid to the water molecule has occurred. In the latter case, the structure would be consistent with the space-group symmetry  $Pnma$ , while in the former the crystal structure would be a superposition of two lower symmetry ( $Pn2_1a$ ) structures related by a mirror plane perpendicular to **b** (Fig. 2), which average in space to the corresponding  $Pnma$  symmetry, a result that would otherwise be physically and chemically impossible. The  $\gamma$ -resorcylic acid molecule is situated on a mirror plane through C4, C1 and C7 which is not a symmetry element of this molecule; therefore, disorder has to be taken into account. While the non-H atoms obey the mirror symmetry approximately, the H atoms of the OH groups do not. Therefore, with the exception of H3, H4 and H1W, the H atoms were treated as disordered and were refined with an assumed site occupancy of 0.5. As in (I), the carboxyl group is in an *anti* conformation and acts as a donor in the formation of an intramolecular hydrogen bond. The phenolic OH groups, the water molecules and the carboxyl groups form an infinite hydrogen-bonded chain along [010] with mixed  $\sigma$ - and  $\pi$ -bond cooperativity (Fig. 3). As the mirror plane is perpendicular to **b**, the hydrogen-bonded chains run simultaneously in both directions along the *b* axis. The observed crystal symmetry is therefore the result of either a dynamic process in which proton transfer occurs along the hydrogen-bonded chains [in contrast to (I), proton transfer would not cause a transformation from form *B* to form *A* in this case] or, more probably, of the domain structure of the crystals. The water molecule, which is located on a mirror plane, forms additional bifurcated hydrogen bonds with the carboxylic O atoms leading to sheets of hydrogen-bonded molecules. The

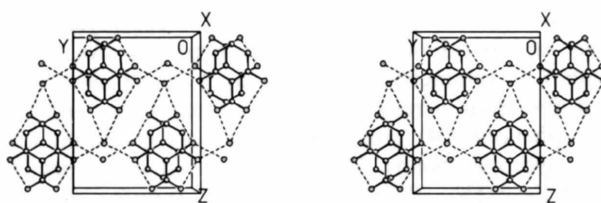


Fig. 2. Stereoview of the crystal packing of (II) viewed along [100]. H atoms have been omitted and hydrogen bonds are shown by dotted lines. This model is compatible with both the disordered structure of  $\gamma$ -resorcylic acid monohydrate and the ordered structure of hydronium  $\gamma$ -resorcyate.

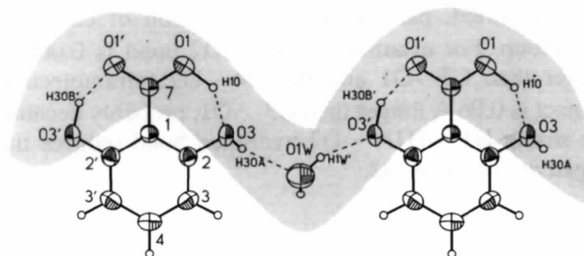


Fig. 3. The hydrogen-bonded chain with mixed  $\sigma$ - and  $\pi$ -bond cooperativity proposed for the disordered structure of  $\gamma$ -resorcylic acid monohydrate. In the crystal, hydrogen-bonded chains run simultaneously in both directions along the *b* axis but only one direction of the chain is shown here. Displacement ellipsoids are plotted at the 50% probability level.

structure is also stabilized by stacking interactions between the aromatic rings in neighbouring sheets, adjacent rings being 3.39 Å apart.

As the H atoms involved in the hydrogen bonds in (II) have not been located unambiguously, the possibility of proton transfer from the carboxylic acid to the water molecule needs to be considered. If this occurred, the  $\gamma$ -resorcyate anion and the hydronium cation would have their molecular mirrors coincident with the mirror plane of the space group. The H atoms involved in the O1...O3 and O3...O1W hydrogen bonds would then have full site occupancies. When H10 and H30A were assigned zero site occupancy and H30B and H1W, placed in their calculated positions, were refined with full site occupancy, H1W was shifted towards O3 and H30B towards the middle of the vector between O1 and O3. This was accompanied by a nearly twofold increase in the isotropic displacement factors of H30B and H1W and by an increase in *R* from 0.065 to 0.068 [ $wR(F^2)$  changed from 0.154 to 0.161]. Indirect proof that (II) has a hydrate structure is provided by the fact that the hydrogen-bonded O...O distances for a hydronium ion would be expected to be shorter than the observed values (Table 3). Moreover, the O1—C7—O1' angle [117.3 (4) compared with analogous angles in (I) and (III) of 117.8 (2) and 122.5 (2)°, respectively] also indicates a carboxylic group rather than carboxylate anion geometry.

The structure of (III) consists of  $\gamma$ -resorcyate anions and pyridinium cations, as expected. An anion with  $C_{2v}$  symmetry is formed as a result of proton transfer from  $\gamma$ -resorcylic acid to the base, and the two phenolic OH groups act as donors in intramolecular hydrogen bonds. The anion and cation interact via an N1'—H1N...O2 hydrogen bond, forming discrete hydrogen-bonded units in the crystal (Fig. 4). Some further stabilization of the complex, via a C2'—H2'...O1 interaction as in other pyridine—carboxylic acid complexes (Dega-Szafran, Gdaniec, Grunwald-Wyspianska, Kosturkiewicz, Koput, Krzyzanowski & Szafran, 1992), is also observed. The  $C_{2v}$  symmetry of the anion is quite distorted, particularly in the region of carboxylate group. For example, the C7—O2 bond is 0.009 Å longer than C7—O1 and the O4...O2 intramolecular contact is 0.06 Å longer than O3...O1, probably because the strong N1'—H1N...O2 hydrogen bond reduces the negative charge at O2.

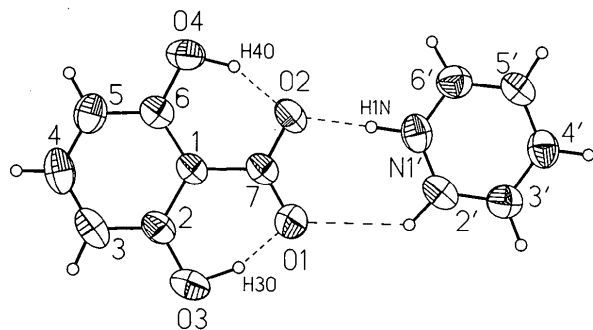


Fig. 4. The structure of the hydrogen-bonded complex (III). Displacement ellipsoids are plotted at the 50% probability level.

## Experimental

Compound (I) was obtained by recrystallization of  $\gamma$ -resorcylic acid from chloroform, (II) from a methanol/water mixture and (III) from a methanol/pyridine mixture.

### Compound (I)

#### Crystal data

C<sub>7</sub>H<sub>6</sub>O<sub>4</sub>  
 $M_r = 154.12$   
 Orthorhombic  
*Pna*2<sub>1</sub>  
 $a = 14.174$  (3) Å  
 $b = 12.132$  (2) Å  
 $c = 3.8280$  (10) Å  
 $V = 658.3$  (2) Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.555$  Mg m<sup>−3</sup>

#### Data collection

KM-4 four-circle diffractometer

Cu  $K\alpha$  radiation  
 $\lambda = 1.54178$  Å  
 Cell parameters from 24 reflections  
 $\theta = 6$ –25°  
 $\mu = 1.124$  mm<sup>−1</sup>  
 $T = 293$  (2) K  
 Transparent plate  
 0.4 × 0.4 × 0.08 mm  
 Colourless

$\theta_{\max} = 64.97^\circ$   
 $h = 0 \rightarrow 16$

$\omega/2\theta$  scans

Absorption correction:

none

654 measured reflections

654 independent reflections

610 observed reflections

$[I > 2\sigma(I)]$

### Refinement

Refinement on  $F^2$

$R[F^2 > 2\sigma(F^2)] = 0.0326$

$wR(F^2) = 0.0901$

$S = 1.189$

654 reflections

115 parameters

Only coordinates of H atoms refined except H3, H4, H5 for which only  $U$ 's refined

$w = 1/[\sigma^2(F_o^2) + (0.0599P)^2 + 0.0081P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

### Compound (II)

#### Crystal data

C<sub>7</sub>H<sub>6</sub>O<sub>4</sub>·H<sub>2</sub>O

$M_r = 172.13$

Orthorhombic

*Pnma*

$a = 6.778$  (1) Å

$b = 9.411$  (1) Å

$c = 11.890$  (2) Å

$V = 758.4$  (2) Å<sup>3</sup>

$Z = 4$

$D_x = 1.507$  Mg m<sup>−3</sup>

#### Data collection

KM-4 diffractometer

$\omega/2\theta$  scans

Absorption correction:

none

750 measured reflections

750 independent reflections

665 observed reflections

$[I > 2\sigma(I)]$

### Refinement

Refinement on  $F^2$

$R[F^2 > 2\sigma(F^2)] = 0.0652$

$wR(F^2) = 0.1544$

$S = 1.235$

750 reflections

88 parameters

All H-atom parameters

refined

$w = 1/[\sigma^2(F_o^2) + (0.0406P)^2 + 0.0081P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.004$

$k = -14 \rightarrow 0$

$l = 0 \rightarrow 4$

3 standard reflections

monitored every 100

reflections

intensity variation: 2%

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.197$  e Å<sup>−3</sup>

$\Delta\rho_{\min} = -0.138$  e Å<sup>−3</sup>

Extinction correction:

*SHELXL93* (Sheldrick, 1994)

Extinction coefficient: 0.017 (3)

Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Cu  $K\alpha$  radiation

$\lambda = 1.54178$  Å

Cell parameters from 25 reflections

$\theta = 12$ –23°

$\mu = 1.136$  mm<sup>−1</sup>

$T = 293$  (2) K

Transparent prisms

0.5 × 0.4 × 0.2 mm

Colourless

$\theta_{\max} = 69.94^\circ$

$h = 0 \rightarrow 8$

$k = 0 \rightarrow 11$

$l = 0 \rightarrow 14$

2 standard reflections

monitored every 100

reflections

intensity variation: 2%

$\Delta\rho_{\max} = 0.198$  e Å<sup>−3</sup>

$\Delta\rho_{\min} = -0.360$  e Å<sup>−3</sup>

Extinction correction:

*SHELXL93* (Sheldrick, 1994)

Extinction coefficient: 0.034 (3)

Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

**Compound (III)***Crystal data* $\text{C}_5\text{H}_6\text{N}^+\cdot\text{C}_7\text{H}_5\text{O}_4^-$  $M_r = 233.22$ 

Monoclinic

 $P2_1/n$  $a = 8.206 (2) \text{ \AA}$  $b = 10.182 (2) \text{ \AA}$  $c = 13.313 (3) \text{ \AA}$  $\beta = 100.94 (3)^\circ$  $V = 1092.1 (4) \text{ \AA}^3$  $Z = 4$  $D_x = 1.418 \text{ Mg m}^{-3}$ Cu  $K\alpha$  radiation $\lambda = 1.54178 \text{ \AA}$ 

Cell parameters from 25 reflections

 $\theta = 9-26^\circ$  $\mu = 0.906 \text{ mm}^{-1}$  $T = 293 (2) \text{ K}$ 

Needle

 $0.3 \times 0.2 \times 0.05 \text{ mm}$ 

Colourless

*Data collection*

KM-4 diffractometer

 $\omega/2\theta$  scans

Absorption correction:

none

1732 measured reflections

1672 independent reflections

1172 observed reflections

 $[I > 2\sigma(I)]$  $R_{\text{int}} = 0.0177$  $\theta_{\text{max}} = 62.49^\circ$  $h = -9 \rightarrow 9$  $k = 0 \rightarrow 11$  $l = 0 \rightarrow 15$ 

2 standard reflections

monitored every 100

reflections

intensity variation: none

*Refinement*Refinement on  $F^2$  $R[F^2 > 2\sigma(F^2)] = 0.0412$  $wR(F^2) = 0.1047$  $S = 1.193$ 

1672 reflections

199 parameters

All H-atom parameters

refined

 $w = 1/[\sigma^2(F_o^2) + (0.0801P)^2 + 0.0081P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\text{max}} = 0.005$  $\Delta\rho_{\text{max}} = 0.191 \text{ e \AA}^{-3}$  $\Delta\rho_{\text{min}} = -0.184 \text{ e \AA}^{-3}$ 

Extinction correction:

*SHELXL93* (Sheldrick, 1994)

Extinction coefficient:

0.032 (2)

Atomic scattering factors

from *International Tables for Crystallography* (1992,

Vol. C, Tables 4.2.6.8 and

6.1.1.4)

Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

$U_{\text{iso}}$ for H atoms; $U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$ for others.				
	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}/U_{\text{eq}}$
(I)				
O1	0.32734 (11)	0.95654 (12)	0.1199 (11)	0.0477 (6)
O2	0.21241 (12)	0.83726 (15)	0.1133 (11)	0.0510 (7)
O3	0.49314 (12)	0.90902 (13)	0.3657 (10)	0.0438 (6)
O4	0.23830 (12)	0.65025 (14)	0.3845 (11)	0.0482 (6)
C1	0.36031 (14)	0.7831 (2)	0.3743	0.0295 (6)
C2	0.45459 (15)	0.8111 (2)	0.4524 (10)	0.0310 (6)
C3	0.5129 (2)	0.7363 (2)	0.6217 (11)	0.0372 (6)
C4	0.4792 (2)	0.6338 (2)	0.7091 (12)	0.0409 (7)
C5	0.3880 (2)	0.6030 (2)	0.6354 (11)	0.0380 (6)
C6	0.32897 (15)	0.6764 (2)	0.4665 (10)	0.0320 (6)
C7	0.29946 (15)	0.8631 (2)	0.1948 (11)	0.0335 (6)
H2O	0.2062 (24)	0.7675 (27)	0.1833 (123)	0.068 (10)
H3O	0.4423 (25)	0.9487 (29)	0.2756 (121)	0.078 (12)
H4O	0.2294 (19)	0.5899 (24)	0.4268 (105)	0.043 (8)
(II)				
O1W	0.8192 (10)	5/4	0.1923 (5)	0.093 (2)
O1	0.8952 (4)	0.8649 (2)	0.4839 (2)	0.0503 (8)
O3	0.8957 (4)	1.0032 (2)	0.3005 (2)	0.0519 (8)
C1	0.8946 (6)	3/4	0.3046 (3)	0.0325 (9)

C2	0.8937 (4)	0.8777 (3)	0.2437 (3)	0.0355 (8)
C3	0.8906 (5)	0.8770 (3)	0.1274 (2)	0.0426 (8)
C4	0.8877 (7)	3/4	0.0707 (4)	0.0446 (12)
C7	0.8958 (6)	3/4	0.4285 (3)	0.0352 (10)
H1W	0.8159 (156)	1.1844 (106)	0.2417 (85)	0.093 (34)
H2W	0.7220 (143)	5/4	0.1437 (81)	0.130 (34)
H1O	0.9216 (186)	0.9607 (151)	0.4277 (108)	0.124 (52)
H3OA	0.8558 (85)	1.0566 (70)	0.2647 (53)	0.020 (16)
H3OB	0.8639 (129)	0.9816 (103)	0.3865 (86)	0.063 (27)

## (III)

C1	0.4607 (2)	0.6901 (2)	0.09301 (12)	0.0401 (4)
C2	0.5093 (2)	0.7151 (2)	0.19854 (13)	0.0492 (5)
C3	0.6107 (3)	0.8211 (2)	0.2327 (2)	0.0626 (6)
C4	0.6639 (3)	0.9016 (2)	0.1632 (2)	0.0634 (6)
C5	0.6194 (3)	0.8804 (2)	0.0601 (2)	0.0568 (5)
C6	0.5173 (2)	0.7748 (2)	0.02472 (12)	0.0459 (4)
C7	0.3481 (2)	0.5781 (2)	0.05733 (12)	0.0424 (4)
O1	0.2963 (2)	0.50787 (12)	0.12215 (10)	0.0589 (4)
O2	0.3062 (2)	0.55873 (12)	−0.03787 (8)	0.0527 (4)
O3	0.4575 (2)	0.6364 (2)	0.26752 (9)	0.0711 (5)
O4	0.4748 (2)	0.75672 (14)	−0.07751 (10)	0.0658 (4)
N1'	0.0942 (2)	0.3550 (2)	−0.07506 (12)	0.0507 (4)
C2'	0.0552 (2)	0.2871 (2)	0.00242 (14)	0.0526 (5)
C3'	−0.0525 (2)	0.1828 (2)	−0.01629 (15)	0.0542 (5)
C4'	−0.1172 (2)	0.1506 (2)	−0.11560 (15)	0.0542 (5)
C5'	−0.0749 (2)	0.2205 (2)	−0.19398 (14)	0.0540 (5)
C6'	0.0314 (2)	0.3233 (2)	−0.17244 (15)	0.0539 (5)
H4O	0.4049 (41)	0.6704 (32)	−0.0841 (22)	0.124 (11)
H1N	0.1653 (32)	0.4313 (26)	−0.0620 (19)	0.087 (7)
H3O	0.3875 (37)	0.5642 (26)	0.2173 (23)	0.108 (9)
H2'	0.1055 (26)	0.3195 (19)	0.0664 (16)	0.061 (6)

Table 2. *Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )*

(I)			
O1—C7	1.234 (3)	C2—C3	1.388 (3)
O2—C7	1.311 (3)	C3—C4	1.373 (3)
O3—C2	1.349 (3)	C4—C5	1.375 (3)
O4—C6	1.360 (3)	C5—C6	1.382 (3)
C1—C2	1.411 (3)	O2—H2O	0.89 (3)
C1—C6	1.414 (3)	O3—H3O	0.93 (4)
C1—C7	1.470 (3)	O4—H4O	0.76 (3)
C2—C1—C6	117.7 (2)	O4—C6—C5	122.0 (2)
C2—C1—C7	119.7 (2)	O4—C6—C1	116.9 (2)
C6—C1—C7	122.5 (2)	C5—C6—C1	121.1 (2)
O3—C2—C3	116.7 (2)	O1—C7—O2	117.8 (2)
O3—C2—C1	122.9 (2)	O1—C7—C1	121.8 (2)
C3—C2—C1	120.3 (2)	O2—C7—C1	120.4 (2)
C4—C3—C2	120.0 (2)	C7—O2—H2O	104.4 (23)
C3—C4—C5	121.5 (2)	C2—O3—H3O	103.5 (22)
C4—C5—C6	119.4 (2)	C6—O4—H4O	109.4 (23)
(II)			
C1—C2	1.403 (3)	O1—H1O	1.14 (14)
C1—C7	1.473 (5)	O3—H3OA	0.71 (7)
C2—C3	1.384 (4)	O3—H3OB	1.06 (10)
C3—C4	1.372 (4)	O1W—H1W	0.85 (10)
O1—C7	1.267 (3)	O1W—H2W	0.88 (10)
O3—C2	1.361 (3)		
C2 <sup>i</sup> —C1—C2	117.9 (4)	O1—C7—O1 <sup>i</sup>	117.3 (4)
C2—C1—C7	121.1 (2)	O1—C7—C1	121.3 (2)
O3—C2—C3	120.0 (3)	C7—O1—H1O	111.8 (63)
O3—C2—C1	119.2 (3)	C2—O3—H3OA	108.2 (49)
C3—C2—C1	120.8 (3)	C2—O3—H3OB	108.0 (51)
C4—C3—C2	119.7 (3)	H1W—O1W—H2W	115.7 (78)
C3—C4—C3 <sup>i</sup>	121.2 (4)		
(III)			
C1—C6	1.395 (2)	C7—O2	1.264 (2)
C1—C2	1.409 (2)	N1'—C2'	1.330 (2)
C1—C7	1.487 (2)	N1'—C6'	1.341 (2)
C2—C3	1.385 (3)	C2'—C3'	1.374 (3)
C3—C4	1.367 (3)	C3'—C4'	1.368 (3)
C4—C5	1.370 (3)	C4'—C5'	1.361 (3)
C5—C6	1.389 (3)	C5'—C6'	1.358 (3)
C2—O3	1.347 (2)	O3—H3O	1.08 (3)

C6—O4	1.352 (2)	O4—H4O	1.04 (3)
C7—O1	1.255 (2)	N1'—H1N	0.97 (3)
C6—C1—C2	118.1 (2)	O1—C7—C1	119.20 (14)
C6—C1—C7	121.94 (15)	O2—C7—C1	118.3 (2)
C2—C1—C7	119.9 (2)	C2'—N1'—C6'	121.3 (2)
O3—C2—C3	119.2 (2)	N1'—C2'—C3'	120.2 (2)
O3—C2—C1	120.4 (2)	C4'—C3'—C2'	118.6 (2)
C3—C2—C1	120.5 (2)	C5'—C4'—C3'	120.5 (2)
C4—C3—C2	119.6 (2)	C6'—C5'—C4'	119.2 (2)
C3—C4—C5	121.7 (2)	N1'—C6'—C5'	120.2 (2)
C4—C5—C6	119.4 (2)	C2—O3—H3O	100.5 (14)
O4—C6—C5	118.0 (2)	C6—O4—H4O	103.4 (16)
O4—C6—C1	121.2 (2)	C2'—N1'—H1N	120.2 (15)
C5—C6—C1	120.8 (2)	C6'—N1'—H1N	118.4 (15)
O1—C7—O2	122.5 (2)		

Symmetry code: (i)  $x, \frac{3}{2} - y, z$ .

Table 3. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
(I)				
O3—H3O...O1	0.93 (4)	1.74 (4)	2.596 (3)	152
O2—H2O...O4	0.89 (4)	1.68 (4)	2.522 (3)	156
O4—H4O...O1 <sup>i</sup>	0.76 (3)	1.95 (3)	2.683 (3)	161
(II)				
O1—H1O...O3	1.14 (14)	1.57 (13)	2.540 (3)	139 (10)
O3—H3OB...O1	1.06 (10)	1.61 (11)	2.540 (3)	143 (8)
O3—H3OA...O1W	0.71 (7)	2.03 (7)	2.705 (4)	159 (6)
O1W—H1W...O3	0.85 (10)	1.92 (11)	2.705 (4)	152 (10)
O1W—H2W...O1 <sup>ii</sup>	0.88 (10)	2.33 (9)	3.069 (6)	143 (5)
(III)				
O3—H3O...O1	1.08 (3)	1.46 (3)	2.497 (2)	158 (2)
O4—H4O...O2	1.04 (3)	1.59 (3)	2.556 (2)	152 (3)
N1'—H1N...O2	0.97 (3)	1.73 (3)	2.692 (2)	175 (2)
C2'—H2'...O1	0.93 (2)	2.50 (2)	3.211 (3)	133 (2)

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

The  $\gamma$ -resorcylic acid molecule in (II) lies on a mirror plane through C7, C1 and C4, therefore the carboxyl and phenolic OH groups are disordered. The molecule adopts two overlapping orientations related by the mirror plane. The positions of all H atoms except for H1W were determined from a  $\Delta F$  map. H1O and H3OB were identified as a broad peak between O1 and O3, which was probably the superposition of the electron densities of these two closely situated H atoms, and were both given a site occupancy of 0.5 and refined. The position of H1W was calculated assuming hydrogen-bond formation with O(3) and refined.

Data collection, cell refinement, and data reduction: Kuma KM-4 software (Kuma, 1991). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1994). Molecular graphics: *Stereochemical Workstation* (Siemens, 1989). Software used to prepare material for publication: *SHELXL93*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1067). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

- Dega-Szafran, Z., Gdaniec, M., Grunwald-Wypianska, M., Kosturkiewicz, Z., Koput, J., Krzyzanowski, P. & Szafran, M. (1992). *J. Mol. Struct.* **270**, 99–124.
- Golubev, N. S. & Denisov, G. S. (1992). *J. Mol. Struct.* **272**, 263–276.
- Jeffrey, G. A. & Saenger, W. (1991). *Hydrogen Bonding in Biological Structures*. Berlin, Heidelberg: Springer-Verlag.
- Kuma (1991). *Kuma KM-4 User's Guide*. Version 3.2. Kuma Diffraction, Wroclaw, Poland.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1994). *J. Appl. Cryst.* In preparation.
- Siemens (1989). *Stereochemical Workstation Operation Manual*. Release 3.4. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

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## 3'-Azido-2',3'-dideoxy-5-hydroxymethyl-uridine

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## Abstract

In the title compound, C<sub>10</sub>H<sub>13</sub>N<sub>5</sub>O<sub>5</sub>, the furanose ring adopts a C(2')-endo envelope conformation (<sup>2</sup>E), with the glycosyl linkage *anti* [ $\chi = 219.0 (2)^\circ$ ]. The