

Gh. Borodi,^a A. Hernanz,^b I. Bratu,^a Mihaela Pop^{c*} and R. Navarro^b^aNational Institute for Research and Development of Isotopic and Molecular Technologies, PO Box 700, R-3400, Cluj-Napoca 5, Romania, ^bUniversidad Nacional de Educacion a Distancia, Departamento de Ciencias y Tecnicas Fisicoquimicas, Sende del Rey s/n, E-28040 Madrid, Spain, and ^cBabes-Bolyai University, Faculty of Physics, M. Kogalniceanu 1, 3400 Cluj-Napoca, Romania

Correspondence e-mail: mmpop@phys.ubbcluj.ro

Key indicators

Single-crystal X-ray study
 $T = 173\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.015\text{ \AA}$
H-atom completeness 49%
Disorder in solvent or counterion
 R factor = 0.061
 wR factor = 0.186
Data-to-parameter ratio = 7.1For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Hydrated sodium cytidine-5'-monophosphate

Hydrated sodium cytidine-5'-monophosphate, $2\text{Na}^+\cdot\text{C}_9\text{H}_{12}\cdot\text{N}_3\text{O}_8\text{P}^{2-}\cdot 6.5\text{H}_2\text{O}$, crystallizes in the monoclinic space group $P2_1$. The asymmetric unit contains two independent anions, four cations and 13 water molecules. Both molecules have approximately identical conformations.

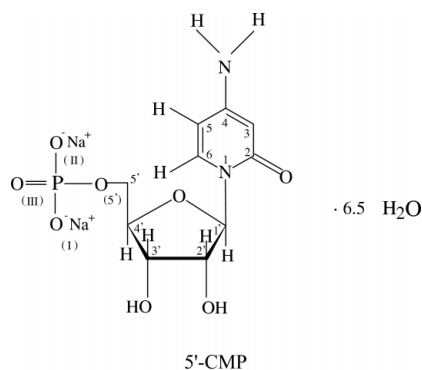
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Comment

Cytidine 5'-monophosphate (5'-CMP) is one of the four common ribonucleotides which make up ribonucleic acids (RNA's). The crystal structure of the sodium salt of cytidine 5'-monophosphate 6.5 hydrate provides valuable information on the interaction mechanisms of alkaline metal ions with water and nucleotides.



Previous studies have been performed on the barium salt of cytidine 5'-monophosphate 8.5 hydrate (Hogle *et al.*, 1980), and some information on the crystal data of disodium cytidine 5'-monophosphate was indicated by these authors. Nevertheless, to our knowledge, the molecular structure and conformational parameters have not been published. Nishimura *et al.* (1986) indicated that the crystal structure of this salt had not been solved. Difficulties in growing single crystals of this mononucleotide could be the reason. Despite these difficulties, single crystals have been obtained and the corresponding crystal structure has been determined in this study.

The two independent molecules of 5'-CMP show similar conformations. The sugar puckers (Saenger, 1984; Landolt-Bornstein, 1989) are C_2' -en (2T_1) in which the C2 and C11 atoms are displaced by 0.559 (11) and -0.576 (10) Å, respectively, from the least-squares planes of the ribose rings. The cytosine rings are nearly planar; the deviations of the ring atoms defining the least-squares planes from these planes are less than 0.025 Å. The torsion angles χ [$\text{O}7-\text{C}1-\text{N}1-\text{C}6 = -125.1$ (10) and $\text{O}15-\text{C}10-\text{N}4-\text{C}15 = -130.2$ (9)] about the glycosidic C1-N1 and C10-N4 linkages adopt values corresponding to an *anti* conformation, and the orientations γ

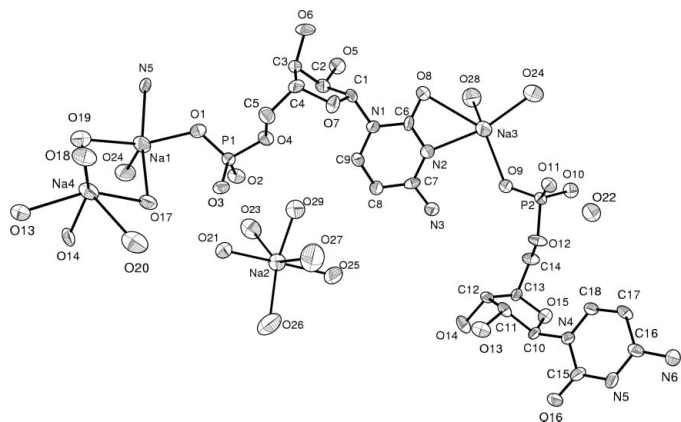


Figure 1
Molecular structure showing 50% probability displacement ellipsoids. H atoms are omitted for clarity.

[O4–C5–C4–C3 = 48.9 (12) and O12–C14–C13–C12 = 47.6 (12)] about the exocyclic C5–C4 and C14–C13 bonds are +*sc* (*gauche, gauche*). These orientations are similar to the ones observed in the barium salt of 5'-CMP (Hogle *et al.*, 1980). The ribose pseudo-rotation phase angles are $P = 161.5$ (11) (2T_1) and 157.1 (9)° (2T_1) and the degree of pucker $\tau_m = 36.9$ (6) and 38.2 (6)°, respectively.

The nucleotides are bridged *via* the phosphate groups forming chains running along the [101] direction. The unordered NaO₆ polyhedra bridge parallel chains *via* the phosphate groups. This packing leaves channels in the structure along *b* which are filled with the disordered Na ions coordinated by water molecules.

Three of the Na⁺ ions are coordinated to six ligands forming distorted octahedra, while in the case of disodium guanosine 5'-phosphate heptahydrate (Barnes & Hankinson, 1982), only two octahedra are distorted and two are fairly regular. The fourth Na⁺ ion is penta-coordinated with two waters, O9 of the phosphate group, and with the cytidine base through atoms N2 and O8. Besides atoms O8, N2 and N5 of the cytidine bases and atoms O5, O13 and O14 of the ribose rings, the O atoms O1 and O9 of the phosphate group are involved in the Na⁺ coordination. A similar situation with the O atoms of the phosphate group taking part in the Na⁺ coordination was observed in disodium guanosine 5'-monophosphate heptahydrate (Katti *et al.*, 1981). In the present hydrated crystal structure, the Na⁺ ions are either partly or completely surrounded by water-O atoms. They also show strong affinity for the negatively charged phosphate groups. The Na2⁺ ion is surrounded by six water-O atoms and was found to be disordered over two sites together with the O30 water atom.

The O–H groups of the ribose rings form intermolecular hydrogen bonds with three O atoms of the phosphate groups, while one ribose O–H group is hydrogen bonded to one water molecule. Atom N6 of the cytidine base is connected by hydrogen bonding to the positionally disordered water molecule. There are two intramolecular hydrogen bonds which connect ribose atoms C1 and C10 with the O atoms O8 and O16 which are attached to the cytidine bases. One ribose ring is stabilized by an intramolecular hydrogen bond between its

O–H groups. Details of the hydrogen-bonding system are given in Table 2.

Experimental

The disodium salt of cytidine 5'-monophosphate hexahydrate purchased from Sigma Chemical was crystallized by slow evaporation from a water–methanol solution using three recrystallization processes.

Crystal data

$2\text{Na}^+ \cdot \text{C}_9\text{H}_4\text{N}_3\text{O}_8\text{P}^{2-} \cdot 6.5\text{H}_2\text{O}$
 $M_r = 484.27$
 Monoclinic, $P2_1$
 $a = 14.042$ (2) Å
 $b = 8.924$ (2) Å
 $c = 16.091$ (3) Å
 $\beta = 94.410$ (2)°
 $V = 2010.4$ (7) Å³
 $Z = 4$

$D_x = 1.600$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 13$ – 26°
 $\mu = 0.26$ mm⁻¹
 $T = 173$ (2) K
 Prism, colourless
 $0.35 \times 0.35 \times 0.30$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
 3930 measured reflections
 3756 independent reflections
 2202 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.094$

$\theta_{\text{max}} = 25.0^\circ$
 $h = -16 \rightarrow 16$
 $k = 0 \rightarrow 10$
 $l = 0 \rightarrow 19$
 2 standard reflections
 frequency: 60 min
 intensity decay: 2%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.061$
 $wR(F^2) = 0.186$
 $S = 1.05$
 3756 reflections
 531 parameters
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.1092P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.53$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.41$ e Å⁻³
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.016 (3)
 Absolute structure: Flack (1983)
 Flack parameter = 0.1 (3)

Table 1

Selected geometric parameters (Å, °).

Na1–O1	2.334 (8)	Na3–O28	2.382 (11)
Na1–O24 ⁱ	2.334 (11)	Na3–N2	2.400 (10)
Na1–O19	2.381 (10)	Na3–O8	2.855 (10)
Na1–O17	2.434 (8)	Na4–O14 ⁱⁱⁱ	2.306 (9)
Na1–N5 ⁱⁱ	2.512 (10)	Na4–O19	2.364 (9)
Na1–O5 ^v	3.111 (10)	Na4–O18	2.384 (10)
Na2–O30	2.27 (2)	Na4–O13 ⁱⁱⁱ	2.413 (9)
Na2–O25 ^v	2.357 (14)	Na4–O17	2.455 (9)
Na2–O29	2.38 (2)	Na4–O20	2.868 (12)
Na2–O21	2.458 (13)	Na5–O29	2.24 (2)
Na2–O23	2.602 (15)	Na5–O26 ^v	2.350 (15)
Na2–O26 ^{iv}	2.643 (15)	Na5–O27	2.36 (2)
Na2–O27 ^{iv}	2.966 (19)	Na5–O25 ^v	2.410 (16)
Na3–O24	2.326 (10)	Na5–O21	2.430 (13)
Na3–O9	2.361 (9)	Na5–O30	2.44 (2)
O7–C1–N1–C6	–125.1 (10)	O15–C10–N4–C15	–130.2 (9)
C4–O7–C1–C2	–22.6 (10)	C13–O15–C10–C11	–26.0 (10)
O7–C1–C2–C3	35.2 (10)	O15–C10–C11–C12	37.0 (9)
C1–C2–C3–C4	–34.0 (10)	C10–C11–C12–C13	–34.2 (9)
C2–C3–C4–O7	22.2 (10)	C11–C12–C13–O15	20.7 (10)
C3–C4–O7–C1	0.1 (13)	C12–C13–O15–C10	3.0 (10)
O4–C5–C4–C3	48.9 (12)	O12–C14–C13–C12	47.6 (12)
C5–C4–C3–O6	144.8 (9)	C14–C13–C12–O14	146.9 (9)
P1–O4–C5–C4	–156.4 (7)	P2–O12–C14–C13	–160.1 (7)

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

Table 2
Hydrogen-bonding geometry (Å, °).

<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>
N6—H8...O29 ⁱ	0.88	2.26	3.07 (2)	153
N6—H8...O30 ⁱ	0.88	2.05	2.913 (19)	169
N3—H6...O22 ⁱⁱ	0.88	2.03	2.890 (11)	166
N3—H5...O9	0.88	2.14	2.986 (10)	161
O5—H5...O6	0.84	2.24	2.678 (12)	112
O5—H5...O23 ⁱⁱⁱ	0.84	2.49	3.288 (12)	160
O6—H6...O2 ⁱⁱⁱ	0.84	1.76	2.602 (11)	175
N6—H6B...O1 ^{iv}	0.88	2.01	2.863 (11)	163
O13—H13A...O9 ⁱ	0.95	1.95	2.651 (11)	128
O14—H14...O11 ⁱⁱ	0.95	1.88	2.573 (11)	128
C1—H1...O8	1.00	2.32	2.760 (13)	105
C5—H5B...O1	0.99	2.53	2.993 (13)	108
C10—H10...O16	1.00	2.28	2.711 (13)	105

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

The H atoms of the nucleotide molecules and H atoms belonging to three water molecules were fixed at ideal positions and refined isotropically. The disordered atoms were split into Na2 and O29 and Na5 and O30, and were refined isotropically.

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1992); cell refinement: *CAD-4-PC Software*; data reduction: *CAD-4-PC Software*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997) and *SIR97* (Altomare *et al.*, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *DIAMOND* (Brandenburg & Berndt, 1999); software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 1999).

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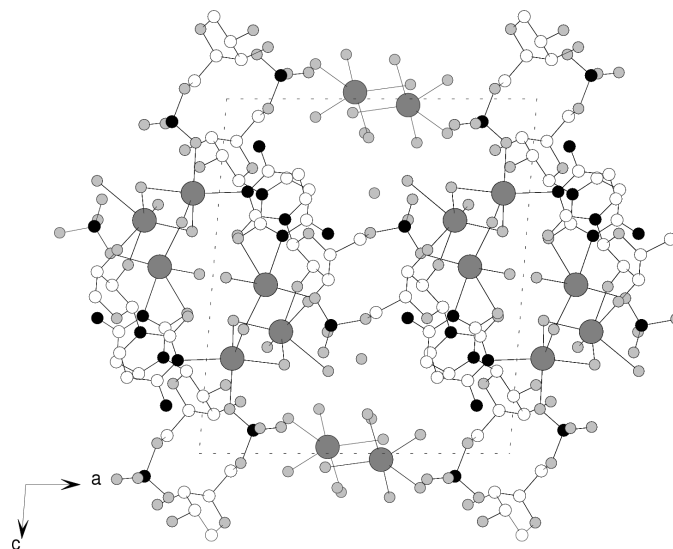


Figure 2
Packing diagram viewed down the *b* axis. Note the alternate nucleotide and solvent layers and the channels in the structure which are formed along the *b* direction.

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