

Fig. 2. The unit cell, showing the packing of the molecules.

Table 2 shows that the intermolecular contacts N(2)—O(4) and O(3)—O(4) are similar to the corresponding H-bond values. On the other hand, the distances N(2)—H(2b) (2.20 Å) [calculated from N(2)—O(4) minus O(4)···H(3); and close to the N—H values reported by Ernst & Cagle (1977) and Ernst (1977)] and O(4)—H(4w) (0.83 Å) suggest that H

atoms from the water bridge with the SO₃ group and that the H from N(2) links with the biguanide.

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Structure of N⁴-Aminocytidine Hemihydrate

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Abstract. C₉H₁₄N₄O₅·0.5H₂O, *M_r* = 267.24, orthorhombic, *P*2₁2₁2₁, *a* = 14.573 (3), *b* = 17.894 (3), *c* = 8.722 (2) Å, *V* = 2274.4 (8) Å³, *Z* = 8, *D_m* = 1.509 (2), *D_x* = 1.561 Mg m⁻³, λ(Cu Kα) = 1.5418 Å, μ = 1.07 mm⁻¹, m.p. = 411–413 K, *F*(000) = 1128, *T* = 295 K, final *R* = 0.041 for 2082 unique reflections. The structure of N⁴-aminocytidine, a nucleoside analog having a potent mutagenicity, has been determined. In two independent molecules the conformations around the C(1′)—N(1) and C(4′)—C(5′) bonds are *anti* and *gauche-gauche*, respectively, and the sugar puckerings are C(2′)-*endo* (²*E*) type. The two independent cytosine bases are overlapped in nearly parallel to form a pair of molecules. The molecules are held together by 11 kinds of hydrogen bonds to form a three-dimensional network.

Introduction. N⁴-Aminocytidine is a compound of biological interest because it is a potent mutagen in phages and bacteria (Negishi, Harada, Ohara, Ohara, Nitta & Hayatsu, 1983), in cultured mammalian cells (Nomura, Negishi, Hayatsu & Kuroda, 1987; Takahashi, Nishizawa, Negishi, Hanaoka, Yamada & Hayatsu, 1988), and in *Drosophila* (Negishi, Negishi, Ryo, Kondo & Hayatsu, 1988). The synthesis of the compound as a crystalline hemihydrate has recently been reported (Negishi, Kawakami, Kayasuga, Odo & Hayatsu, 1987). In the present article the precise molecular conformations in the crystals, as determined by X-ray diffraction analysis, are reported.

Experimental. Crystals were prepared by slow recrystallization from an aqueous-ethanol solution (*ca*

2:1 in volume): colorless plates with developed faces {010} elongated along *c*. D_m was measured by flotation in CCl_4 -benzene. Systematic absences: $h00$ for h odd, $0k0$ for k odd, $00l$ for l odd. Orthorhombic space group $P2_12_12_1$. A crystal with dimensions $0.35 \times 0.28 \times 0.38$ mm was mounted on a Rigaku AFC-5 four-circle diffractometer. Lattice parameters were determined with 20 reflections in the range $19 < 2\theta < 51^\circ$ by the least-squares method. Intensities were measured up to $(\sin\theta)/\lambda = 0.5753 \text{ \AA}^{-1}$ by the ω - 2θ scan method [scan speed 4° min^{-1} in ω , scan range (2θ): $1.2^\circ + 0.15^\circ \tan\theta$, Ni-filtered $\text{Cu } K\alpha$, 40 kV, 200 mA, rotating anode]. Background was measured for 4 s on either side of the peak. Three standard reflections were measured for every 57 reflections; the fluctuation was within 2.7% in F . Lorentz and polarization corrections were applied, but no absorption correction. All 2082 unique reflections (ranging over $h = 0$ to 16, $k = 0$ to 20, $l = 0$ to 10) were used for refinement; the $|F_o|$ values of 2068 reflections were larger than $\sigma(F_o)$. Structure was solved by *RANTAN* (Yao, 1981) incorporated in *MULTAN84* (Main, Germain & Woolfson, 1984). An E map calculated using the best set of phases gave 18 atoms including those of the cytosine moieties of the two crystallographically independent molecules. The positions of the remaining non-H atoms were located by successive Fourier synthesis. The structure was refined (anisotropically for non-H atoms) by block-diagonal least squares. The quantity minimized was $\sum w(|F_o| - |F_c|)^2$, where $w = 1.0/[\sigma(F_o)^2 + 0.0151|F_o| + 0.0013|F_o|^2]$ for $|F_o| > 0$, $w = 0.2461$ for $|F_o| = 0$. H-atom positions were determined from difference Fourier map, and refined isotropically by least squares. Extinction correction was applied for 16 strongest reflections [$I_{\text{corr}} = I_o(1 + 3.8 \times 10^{-6}I_o)$]. The final R and wR were 0.041 and 0.061, respectively, for 2082 unique reflections ($S = 1.26$). $(A/\sigma)_{\text{max}}$ in final refinement cycle was 0.1 for non-H atoms and 0.9 for H atoms. $\Delta\rho$ in final difference Fourier map was $\pm 0.23 \text{ e \AA}^{-3}$. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Calculations were carried out at the Crystallographic Research Center, Institute for Protein Research, Osaka University, and at the Okayama University Computer Center, using the programs of the *Universal Crystallographic Computation Program System—Osaka* (1979) and their modifications.

Discussion. Final atomic parameters are listed in Table 1.* The thermal ellipsoids drawn by *ORTEP* (Johnson,

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond lengths and angles involving H atoms and selected torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44946 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Final atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters with e.s.d.'s in parentheses

$$B_{\text{eq}} = \frac{4}{3} \sum \beta_{ii} / a_i^2.$$

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{\text{eq}}(\text{\AA}^2)$
Molecule A				
N(1)	3145 (2)	2389 (1)	3373 (3)	1.99 (8)
C(2)	2966 (2)	3006 (1)	2508 (3)	2.08 (9)
N(3)	3650 (2)	3402 (1)	1862 (3)	2.25 (9)
C(4)	4525 (2)	3184 (2)	2117 (3)	2.4 (1)
C(5)	4740 (2)	2562 (2)	3045 (4)	3.0 (1)
C(6)	4031 (2)	2181 (2)	3664 (4)	2.7 (1)
O(2)	2138 (1)	3189 (1)	2281 (3)	2.84 (9)
N(4)	5178 (2)	3587 (2)	1462 (3)	3.2 (1)
N(7)	5014 (2)	4214 (2)	516 (3)	3.3 (1)
C(1')	2378 (2)	1952 (1)	4059 (3)	2.2 (1)
C(2')	2084 (2)	1291 (2)	3054 (3)	2.3 (1)
C(3')	1793 (2)	722 (2)	4265 (4)	2.6 (1)
C(4')	2415 (2)	885 (2)	5592 (3)	2.6 (1)
O(4')	2674 (1)	1665 (1)	5462 (2)	2.67 (8)
O(2')	1373 (1)	1449 (1)	2032 (2)	2.94 (9)
O(3')	870 (1)	856 (1)	4742 (3)	3.4 (1)
C(5')	3253 (2)	398 (2)	5687 (4)	3.3 (1)
O(5')	3706 (2)	393 (1)	4256 (3)	3.9 (1)
Molecule B				
N(1)	8204 (2)	2425 (1)	1756 (3)	2.03 (8)
C(2)	7865 (2)	3016 (2)	914 (3)	2.09 (9)
N(3)	8451 (2)	3497 (1)	233 (3)	2.33 (9)
C(4)	9351 (2)	3399 (2)	432 (3)	2.4 (1)
C(5)	9724 (2)	2812 (2)	1335 (4)	2.7 (1)
C(6)	9117 (2)	2330 (2)	1962 (4)	2.7 (1)
O(2)	7011 (1)	3088 (1)	765 (3)	2.98 (8)
N(4)	9939 (2)	3859 (1)	-244 (3)	3.0 (1)
N(7)	9730 (2)	4414 (1)	-1317 (3)	2.66 (9)
C(1')	7565 (2)	1888 (2)	2472 (3)	2.2 (1)
C(2')	7476 (2)	1158 (2)	1552 (3)	2.3 (1)
C(3')	7226 (2)	604 (2)	2815 (3)	2.6 (1)
C(4')	7829 (2)	877 (2)	4127 (3)	2.27 (9)
O(4')	7914 (1)	1679 (1)	3903 (2)	2.57 (8)
O(2')	6820 (1)	1182 (1)	390 (2)	2.93 (9)
O(3')	6301 (1)	674 (1)	3300 (2)	3.2 (1)
C(5')	8750 (2)	529 (2)	4292 (4)	3.0 (1)
O(5')	9300 (1)	545 (1)	2950 (3)	3.16 (9)
Water				
O(W)	798 (2)	4200 (2)	3539 (3)	4.7 (1)

1971) are shown in Fig. 1 with atomic numbering. Bond lengths and angles are listed in Table 2.

In the two independent molecules *A* and *B*, an H atom is attached to N(4) but not to N(3). Thus, both N^4 -aminocytosine moieties take the amino form as found in N^4 -aminocytosine (Takayanagi, Ogura & Hayatsu, 1980). Corresponding bond lengths and angles for the molecules *A* and *B* are in agreement within the experimental errors. The cytosine rings are planar with the maximum deviation at N(1) of 0.017 (3) Å in *A* and 0.044 (3) Å in *B*. C(1') deviates by 0.025 (5) Å in *A* and 0.107 (5) Å in *B* from the respective ring plane, while O(2) and N(4) lie in the plane. The bond lengths and angles found in the sugar moieties are in agreement with those found in cytidine (Furberg, Petersen & Rømming, 1965).

Both molecules take an *anti* conformation about the C(1')-N(1) bond, $\chi[\text{O}(4')\text{-C}(1')\text{-N}(1)\text{-C}(6)] = 31.0 (3)^\circ$ for *A* and $44.2 (3)^\circ$ for *B*, as found usually in pyrimidine nucleosides (Sundaralingam, 1969; Ts'o, 1974). The N(7) amino group in both molecules takes a *cis* conformation to N(3) [N(3)-C(4)-N(4)-N(7)

= -0.8 (5)° for *A*, -7.4 (5)° for *B*]. The *cis* conformation appears to be favored because of the steric hindrance that would arise in the *trans* conformation between H atoms attached to C(5) and N(7). It would be of interest to study whether this particular conformation of the amino group plays some role in the mutagenic action of this compound.

The conformations around the exocyclic C(4')-C(5') bond are *gauche-gauche* for both molecules [C(3')-C(4')-C(5')-O(5') = 50.6 (4)° for *A*, 53.3 (4)° for *B*; O(4')-C(4')-C(5')-O(5') = -70.0 (4)° for *A*, -67.4 (3)° for *B*] as found most frequently (Sundaralingam, 1969; Yamagata & Tomita, 1987). Both molecules have the sugar conformation of C(2')-*endo* (²*E*) type. The pseudorotation parameters (Altona & Sundaralingam, 1972) are $P = 177.1^\circ$, $\tau_m = 32.1^\circ$ for *A* and $P = 176.3^\circ$, $\tau_m = 38.8^\circ$ for *B*. The torsion angle O(3')-C(3')-C(4')-C(5') is 146.1 (3)° for *A* and 149.4 (3)° for *B*, as expected for the C(2')-*endo* (²*E*) puckering of the sugar ring (Sundaralingam, 1969).

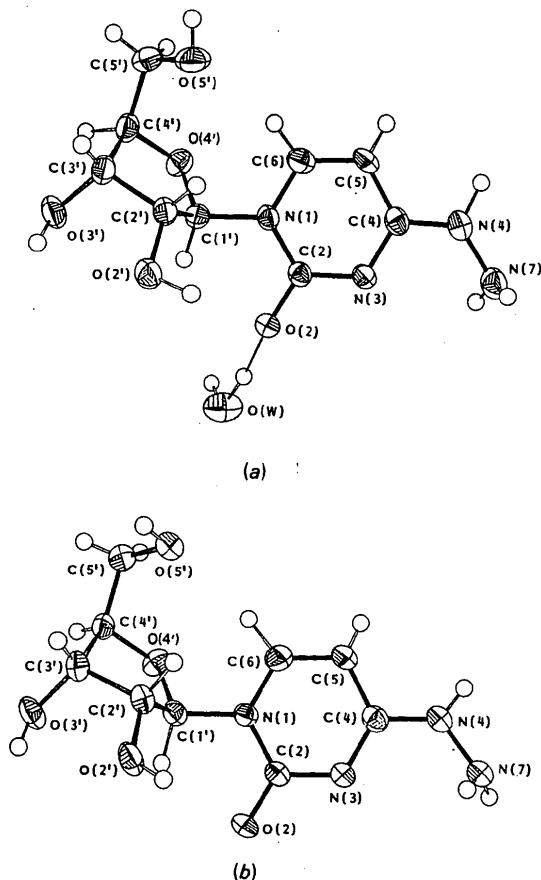


Fig. 1. The thermal ellipsoids (50% probability) of the molecules with atomic numbering of non-H atoms. The H atoms are represented as spheres equivalent to $B = 1.0 \text{ \AA}^2$. (a) Molecule *A* and water molecule, (b) molecule *B*.

Table 2. Bond lengths (Å) and angles (°) with e.s.d.'s in parentheses

	Molecule <i>A</i>	Molecule <i>B</i>
N(1)-C(2)	1.362 (4)	1.379 (4)
N(1)-C(6)	1.368 (4)	1.353 (4)
N(1)-C(1')	1.490 (4)	1.477 (4)
C(2)-N(3)	1.347 (4)	1.350 (4)
C(2)-O(2)	1.266 (4)	1.258 (4)
N(3)-C(4)	1.352 (4)	1.335 (4)
C(4)-C(5)	1.411 (5)	1.421 (4)
C(4)-N(4)	1.324 (4)	1.326 (4)
C(5)-C(6)	1.350 (5)	1.351 (4)
N(4)-N(7)	1.413 (4)	1.398 (4)
C(1')-C(2)	1.533 (4)	1.539 (4)
C(1')-O(4')	1.395 (4)	1.399 (4)
C(2')-C(3')	1.527 (4)	1.526 (4)
C(2')-O(2')	1.396 (4)	1.394 (4)
C(3')-C(4')	1.499 (4)	1.523 (4)
C(3')-O(3')	1.428 (4)	1.418 (4)
C(4')-O(4')	1.450 (4)	1.454 (4)
C(4')-C(5')	1.503 (5)	1.487 (4)
C(5')-O(5')	1.412 (5)	1.419 (4)
C(2)-N(1)-C(6)	120.3 (2)	121.3 (2)
C(2)-N(1)-C(1')	120.3 (2)	119.9 (2)
C(6)-N(1)-C(1')	119.4 (2)	118.8 (2)
N(1)-C(2)-N(3)	121.1 (3)	119.8 (3)
N(1)-C(2)-O(2)	118.6 (3)	119.2 (3)
N(3)-C(2)-O(2)	120.3 (3)	121.0 (3)
C(2)-N(3)-C(4)	118.5 (2)	118.7 (3)
N(3)-C(4)-C(5)	122.1 (3)	123.0 (3)
N(3)-C(4)-N(4)	116.7 (3)	119.7 (3)
C(5)-C(4)-N(4)	121.2 (3)	117.3 (3)
C(4)-C(5)-C(6)	117.2 (3)	116.5 (3)
N(1)-C(6)-C(5)	120.7 (3)	120.7 (3)
C(4)-N(4)-N(7)	124.3 (3)	126.7 (3)
N(1)-C(1')-C(2)	112.6 (2)	112.7 (2)
N(1)-C(1')-O(4')	108.3 (2)	108.8 (2)
C(2')-C(1')-O(4')	107.7 (2)	105.6 (2)
C(1')-C(2)-C(3')	101.3 (2)	101.3 (2)
C(1')-C(2)-O(2')	114.6 (2)	114.3 (2)
C(3')-C(2)-O(2')	111.8 (2)	112.4 (2)
C(2')-C(3')-C(4')	103.7 (2)	101.3 (2)
C(2')-C(3')-O(3')	110.6 (2)	112.6 (3)
C(4')-C(3')-O(3')	108.2 (2)	107.2 (3)
C(3')-C(4')-O(4')	106.5 (2)	105.4 (2)
C(3')-C(4')-C(5')	114.9 (3)	117.3 (3)
O(4')-C(4')-C(5')	110.5 (3)	110.5 (2)
C(1')-O(4')-C(4')	110.0 (2)	110.7 (2)
C(4')-C(5')-O(5')	109.6 (3)	114.9 (2)

Table 3. Geometry of hydrogen bonds

Donor (D)	Acceptor (A)	D...A (Å)	H...A (Å)	D-H...A (°)
(1) O(2 ^A)	O(2 ^{Bⁱⁱⁱ})	2.739 (3)	1.63 (3)	176 (3)
(2) O(2 ^{Bⁱⁱⁱ})	O(2 ^A)	2.628 (3)	1.79 (4)	177 (4)
(3) O(^{H^v})	O(2 ^A)	2.879 (4)	1.68 (4)	173 (3)
(4) O(^{H^v})	O(3 ^{B^{iv}})	2.862 (4)	1.86 (4)	165 (3)
(5) N(4 ^A)	O(2 ^B)	2.881 (4)	2.10 (6)	130 (5)
(6) O(3 ^A)	O(5 ^{Bⁱⁱⁱ})	2.826 (3)	1.88 (3)	148 (2)
(7) N(4 ^{Bⁱⁱⁱ})	O(2 ^B)	2.745 (4)	1.94 (4)	165 (4)
(8) O(3 ^B)	N(7 ^{Bⁱⁱⁱ})	2.874 (4)	1.98 (5)	163 (5)
(9) O(5 ^A)	N(7 ^A)	2.823 (4)	2.03 (7)	168 (7)
(10) O(5 ^B)	O(^{H^v})	2.739 (4)	1.69 (6)	174 (5)
(11) N(7 ^B)	O(3 ^A)	3.051 (4)	1.91 (6)	167 (5)

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

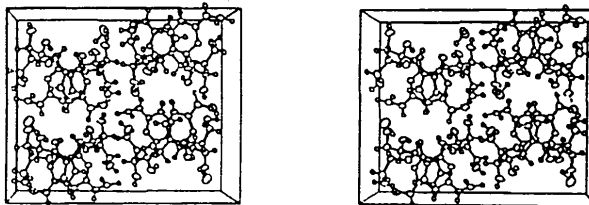


Fig. 2. Stereoscopic view of the molecular packing in the unit cell. The *a* axis points downward, the *b* axis from left to right, and the *c* axis into the plane of the paper.

The crystal structure viewed down the *c* axis is shown in Fig. 2. All the functional groups except N(3) of molecules *A* and *B* participate in hydrogen bonding. The geometry of the hydrogen bonds together with the symmetry code are summarized in Table 3. The cytosine bases of molecules *A*ⁱ and *B*ⁱⁱⁱ are overlapped in nearly parallel to form a pair of molecules: base separation is about 3.3 Å, the dihedral angle being 0.8 (1)°. The molecules *A*ⁱ and *B*ⁱⁱⁱ are related by a pseudo-twofold axis along *a*, and are joined together by hydrogen bonds (1) and (2) (Table 3). The pairs are stacked along *c* to form a column through the hydrogen bonds (3) and (4). The columns are held together by the hydrogen bonds (5) to (8) along *a* and (9) to (11) along *b* to complete the whole structure.

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Acta Cryst. (1988). **C44**, 1457–1459

Structure of α -2-Norbornyl-4-chloroacetophenone*

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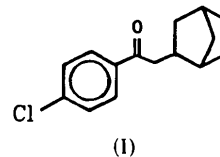
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Abstract. $C_{15}H_{17}ClO$, $M_r = 248.75$, monoclinic, $P2_1/a$, $a = 8.836$ (2), $b = 10.222$ (1), $c = 14.578$ (2) Å, $\beta = 95.828$ (7)°, $V = 1309.9$ (3) Å³, $Z = 4$, $D_x = 1.261$ g cm⁻³, $Mo K\alpha$, $\lambda = 0.70930$ Å, $\mu = 2.6$ cm⁻¹, $F(000) = 528$, $T = 295$ K, $R = 0.037$ for 1227 reflections. Bond lengths and angles are normal, and torsion angles are very similar to those in related cycloalkylacetophenones. H...O abstraction distances are favourable for photochemical reaction, and photo-product ratios can be rationalized in terms of angles between biradical *p* orbitals.

Introduction. Previous structural and photochemical studies of α -cycloalkylacetophenones have yielded correlations between photochemical behaviour and molecular parameters, and have in particular indicated

the dependence of photoproduct ratios on cycloalkyl ring size (Evans & Trotter, 1988*a,b*). As an example of a molecule with a conformationally rigid ring system an α -norbornylacetophenone (I) has now been examined.



Experimental. Colourless crystals, $0.20 \times 0.25 \times 0.30$ mm, Enraf–Nonius CAD-4F diffractometer, lattice parameters from 24 reflections with $\theta = 12$ – 18 °. Intensities for $\theta \leq 25$ °, hkl 0 to 10, 0 to 12, -17 to 17 , ω - 2θ scan, ω -scan width $(0.90 + 0.35 \tan \theta)$ ° at 1.1 – 6.7 min⁻¹, extended 25% on each side for background measurement, three standard reflections (no

* 2-(*exo*-2-Bicyclo[2.2.1]heptyl)-1-(4-chlorophenyl)ethanone.