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## Cytidine Nitrate

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**Abstract.**  $C_9H_{14}N_3O_5^+NO_3^-$ , monoclinic,  $P2_1$ ;  $a = 7.818$  (3),  $b = 7.514$  (3),  $c = 11.095$  (5) Å,  $\beta = 108.35$  (5)°,  $U = 618.6$  Å<sup>3</sup>;  $M = 306.2$ ,  $Z = 2$ ,  $D_x = 1.644$  g cm<sup>-3</sup>. The structure has been determined by direct methods and refined to an  $R$  of 0.026 for 1319 unique X-ray diffractometer data. The sugar residue is best described by the envelope conformation, with C(1'), C(2'), C(4') and O(1') defining the best plane through the ring. The glycosidic torsion angle, C(2)–N(1)–C(1')–O(1'), is  $-163.6^\circ$ .

**Introduction.** Crystals of cytidine nitrate (Fig. 1) were grown by accident in the course of a study of metal complexes of nucleic acid components. In view of the interesting pattern of hydrogen bonding we have completed the structure determination.

Intensities were determined with an automated Stoe STADI-2 two-circle diffractometer, graphite-monochromated Mo  $K\alpha$  radiation, and two crystals (layers  $h0-8l$  and  $hk0-12$  respectively). Cell dimensions were determined by least squares from 357 diffractometer zero-layer  $\omega$  measurements.  $L_p$  (but not absorption,  $\mu = 0.98$  cm<sup>-1</sup>) corrections were applied, and inter-layer scale factors obtained by a linear least-squares analysis of common reflexions. Equivalent reflexions were averaged to yield the 1319 unique observed data employed in structure refinement.

The structure (Fig. 2) was solved by multiresolution tangent refinement with the program *SHELX*. A starting set of nine reflexions was chosen on the basis of a convergence map; this defined the origin and enantiomorph and generated 2<sup>8</sup> phase permutations. One of the two phase sets with lowest figure of merit ( $R_\alpha = 0.037$ ) revealed all but two of the non-hydrogen atoms. The structure was refined by full-matrix least squares

with anisotropic temperature factors for C, N and O and an overall H atom isotropic temperature factor (which refined to  $U = 0.048$  Å<sup>2</sup>). C–H vectors were constrained to lie in geometrically idealized orientations with C–H = 1.01 Å; the coordinates of all other H atoms were allowed to refine freely. The weights were

Table 1. *Atom coordinates* ( $\times 10^4$ )

	<i>x</i>	<i>y</i>	<i>z</i>
N(1)	8243 (3)	6733 (4)	3508 (2)
C(2)	7957 (4)	5226 (6)	4128 (3)
O(2)	8120 (3)	3730 (4)	3768 (2)
N(3)	7506 (4)	5558 (5)	5210 (3)
C(4)	7243 (5)	7186 (5)	5644 (3)
N(4)	6808 (4)	7305 (6)	6690 (3)
C(5)	7451 (4)	8662 (5)	4929 (3)
C(6)	7962 (4)	8398 (5)	3893 (3)
C(1')	8863 (4)	6414 (5)	2383 (3)
C(2')	7349 (4)	5843 (4)	1215 (3)
O(2')	8159 (3)	4672 (4)	541 (2)
C(3')	6796 (4)	7589 (4)	511 (2)
O(3')	6068 (3)	7359 (4)	-824 (2)
C(4')	8556 (4)	8584 (5)	825 (3)
C(5')	8474 (5)	10592 (4)	814 (3)
O(5')	7479 (3)	11188 (4)	1625 (2)
O(1')	9566 (2)	8008	2086 (2)
N(10)	6464 (3)	2391 (5)	7218 (2)
O(10)	6431 (3)	3807 (4)	7799 (2)
O(11)	6672 (3)	2437 (4)	6144 (2)
O(12)	6258 (4)	949 (4)	7684 (2)
H(1)	7247 (45)	4693 (50)	5525 (31)
H(2)	6556 (43)	8361 (54)	6917 (33)
H(3)	6732 (42)	6396 (55)	7081 (31)
H(4)	7219	9905	5189
H(5)	8143	9460	3388
H(6)	9785	5426	2615
H(7)	6287	5221	1362
H(8)	7346 (44)	4585 (44)	-281 (34)
H(9)	5820	8215	766
H(10)	5063 (42)	6988 (45)	-959 (29)
H(11)	9101	8301	135
H(12)	7858	11020	-81
H(13)	9737	11089	1128
H(14)	7902 (42)	12016 (50)	2021 (30)

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$w = 1/\sigma^2(F)$ , which led to a mean value of  $w\Delta^2$  virtually independent of the magnitude of  $\sin \theta$  or  $F_o$ . Complex neutral atom scattering factors were employed. The refinement converged to  $R' = \sum w^{1/2} \Delta / \sum w^{1/2} F_o = 0.025$ , with a corresponding unweighted  $R$  of 0.026. Final atomic coordinates are given in Table 1, and anisotropic

temperature factors in Table 2. Bond lengths and angles are given in Tables 3 and 4.\*

\* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31846 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 2. Anisotropic temperature factors ( $\text{\AA}^2 \times 10^3$ )

The temperature factor exponent takes the form:  
 $-2\pi^2(U_{11}h^2a^{*2} + \dots + 2U_{12}hka^*b^*)$ .

	$U_{11}$	$U_{22}$	$U_{33}$	$U_{23}$	$U_{13}$	$U_{12}$
N(1)	40 (1)	30 (1)	32 (1)	4 (1)	12 (1)	-1 (1)
C(2)	40 (2)	41 (2)	33 (2)	-1 (2)	9 (2)	-1 (1)
O(2)	66 (2)	31 (1)	43 (1)	1 (1)	21 (1)	1 (1)
N(3)	53 (2)	36 (2)	33 (1)	5 (1)	15 (1)	-1 (1)
C(4)	39 (2)	47 (2)	29 (2)	-4 (2)	5 (1)	-1 (2)
N(4)	72 (2)	55 (2)	39 (2)	1 (2)	23 (1)	6 (2)
C(5)	50 (2)	34 (2)	37 (2)	-7 (2)	10 (1)	0 (2)
C(6)	38 (2)	29 (2)	37 (2)	-1 (1)	8 (1)	-1 (1)
C(1')	38 (2)	32 (2)	37 (2)	4 (1)	15 (1)	1 (1)
C(2')	37 (2)	25 (2)	36 (2)	1 (1)	16 (1)	1 (1)
O(2')	58 (2)	32 (1)	44 (1)	-4 (1)	17 (1)	11 (1)
C(3')	35 (2)	26 (2)	32 (2)	1 (1)	15 (1)	1 (1)
O(3')	45 (1)	43 (1)	31 (1)	2 (1)	9 (1)	-7 (1)
C(4')	41 (2)	28 (2)	33 (2)	4 (1)	13 (1)	0 (1)
C(5')	58 (2)	27 (2)	49 (2)	4 (2)	16 (2)	-10 (2)
O(5')	58 (2)	28 (1)	52 (1)	-9 (1)	11 (1)	-1 (1)
O(1')	33 (1)	35 (1)	41 (1)	7 (1)	9 (1)	-5 (1)
N(10)	38 (1)	47 (2)	33 (1)	5 (2)	9 (1)	1 (1)
O(10)	66 (2)	46 (2)	44 (1)	-11 (1)	23 (1)	7 (1)
O(11)	62 (1)	45 (1)	30 (1)	-2 (1)	18 (1)	-3 (1)
O(12)	92 (2)	48 (2)	53 (1)	8 (1)	37 (1)	-6 (1)

Table 3. Bond lengths ( $\text{\AA}$ )

C(2)—N(1)	1.379 (5)	C(6)—N(1)	1.362 (5)
C(1')—N(1)	1.494 (5)	O(2)—C(2)	1.213 (5)
N(3)—C(2)	1.377 (5)	C(4)—N(3)	1.354 (5)
N(4)—C(4)	1.312 (5)	C(5)—C(4)	1.402 (6)
C(6)—C(5)	1.346 (5)	C(2')—C(1')	1.516 (6)
O(1')—C(1')	1.400 (4)	O(2')—C(2')	1.425 (4)
C(3')—C(2')	1.519 (6)	O(3')—C(3')	1.420 (4)
C(4')—C(3')	1.507 (6)	C(5')—C(4')	1.510 (6)
O(1')—C(4')	1.440 (4)	O(5')—C(5')	1.434 (5)
O(10)—N(10)	1.248 (4)	O(11)—N(10)	1.253 (4)
O(12)—N(10)	1.233 (4)		

Table 4. Bond angles ( $^\circ$ )

C(6)—N(1)—C(2)	122.1 (3)	C(1')—N(1)—C(2)	115.5 (3)
C(1')—N(1)—C(6)	122.4 (3)	O(2)—C(2)—N(1)	123.2 (4)
N(3)—C(2)—N(1)	114.4 (4)	N(3)—C(2)—O(2)	122.5 (4)
C(4)—N(3)—C(2)	125.6 (4)	N(4)—C(4)—N(3)	119.1 (4)
C(5)—C(4)—N(3)	117.2 (4)	C(5)—C(4)—N(4)	123.7 (4)
C(6)—C(5)—C(4)	119.0 (4)	C(5)—C(6)—N(1)	121.6 (4)
C(2')—C(1')—N(1)	112.9 (3)	O(1')—C(1')—N(1)	108.3 (3)
O(1')—C(1')—C(2')	107.4 (3)	O(2')—C(2')—C(1')	105.5 (3)
C(3')—C(2')—C(1')	102.4 (3)	O(1')—C(4')—C(3')	104.9 (3)
O(1')—C(4')—C(5')	108.5 (3)	O(5')—C(5')—C(4')	109.7 (3)
C(4')—O(1')—C(1')	110.5 (3)	C(5')—C(4')—C(3')	117.4 (4)
C(3')—C(2')—O(2')	111.4 (3)	O(3')—C(3')—C(2')	112.8 (3)
C(4')—C(3')—C(2')	102.6 (3)	C(4')—C(3')—O(3')	109.9 (3)
O(11)—N(10)—O(10)	119.8 (4)	O(12)—N(10)—O(10)	120.4 (3)
O(12)—N(10)—O(11)	119.7 (4)		

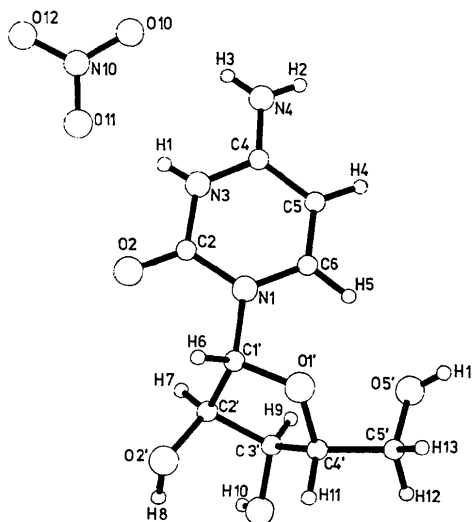


Fig. 1. The cytidinium and nitrate ions, showing the labelling of the atoms.

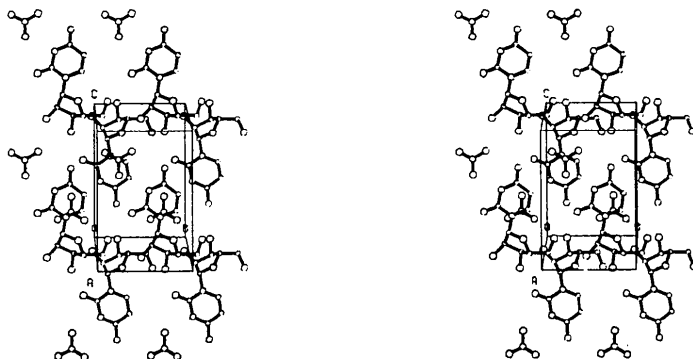


Fig. 2. Stereoscopic drawing of the cell viewed along  $a$ .

**Discussion.** The cytosine ring atoms are coplanar to within 0.026 Å. Bond lengths in the ring are similar to those reported for other protonated cytosines (Viswamitra, Reddy, Lin & Sundaralingam, 1971). The sugar ring is puckered with C(3') *endo*, i.e. lying 0.53 Å out of the mean plane through the other ring atoms, on the same side as C(5'). In terms of the mean plane through all five ring atoms, C(2') is *exo* and C(3') is *endo*, the sugar pucker can therefore be described as  $^3T_2$  (Young & Wilson, 1975). The mean plane of the sugar ring makes an angle of 107.3° with that of the cytosine ring. The conformation about C(4')–C(5') is *gauche-gauche* with torsion angles O(5')–C(5')–C(4')–

C(3') 54.0 and O(5')–C(5')–C(4')–O(1') –64.6°, in line with other structures of this type. The glycosidic torsion angle C(2)–N(1)–C(1')–O(1') is –163.6, similar to the –162.8° observed in cytidine (Furberg, Petersen & Rømming, 1965), and consistent with other structures containing the *endo* C(3') atom (Young & Wilson, 1975).

The non-bonded distances involved in possible hydrogen bonds are analysed in Table 5. Since the coordinates of the hydrogen atoms involved, except H(5), were allowed to refine freely, the O–H and N–H distances are shorter than the true internuclear separations, as normally found in X-ray diffraction studies. The hydrogen bonds link the ions into layers perpendicular to **a**.

Table 5. Possible hydrogen bonds (Å)

O(2')–H(8)	0.94	N(3)····O(11)	2.725
O(3')–H(10)	0.80	N(4) <sup>iv</sup> ····O(12)	3.033
O(5')–H(14)	0.77	N(4)····O(10)	2.955
N(3)–H(1)	0.79	C(6)····O(5')	3.205
N(4)–H(2)	0.87	H(8) <sup>i</sup> ····O(10)	2.11
N(4)–H(3)	0.82	H(10) <sup>ii</sup> ····O(5')	1.98
C(6)–H(5)	1.01	H(14)····O(2) <sup>iii</sup>	2.29
		H(1)····O(11)	1.94
O(2') <sup>i</sup> ····O(10)	2.986	H(2) <sup>iv</sup> ····O(12)	2.16
O(3') <sup>ii</sup> ····O(5')	2.775	H(3)····O(10)	2.14
O(5')····O(2) <sup>iii</sup>	2.968	H(5)····O(5')	2.27

Key to symmetry operations relating designated atoms to reference atoms at (x, y, z)

(i)	x,	y,	1.0+z	(iii)	x,	1.0+y	, z
(ii)	1.0–x,	0.5+y,	–z	(iv)	x,	y–1.0,	z

We are grateful to the Science Research Council for providing the diffractometer, and for financial support to J.J.G. and R.T. The calculations were performed on the Cambridge University IBM 370/165 computer.

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## Hexakis(imidazole)nickel(II) Chloride Tetrahydrate

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**Abstract.** Ni(C<sub>3</sub>H<sub>4</sub>N<sub>2</sub>)<sub>6</sub>(Cl)<sub>2</sub>·4H<sub>2</sub>O, *M* = 610.1; triclinic, *P* $\bar{1}$ ; *a* = 9.084 (2), *b* = 8.808 (2), *c* = 10.566 (2) Å,  $\alpha$  = 83.16 (2),  $\beta$  = 104.89 (2),  $\gamma$  = 118.28 (2)°, *V* = 719.5 Å<sup>3</sup>; *Z* = 1, *D<sub>m</sub>* = 1.41 (2), *D<sub>c</sub>* = 1.408 g cm<sup>–3</sup>. Final *R* = 0.043. The crystal structure consists of six planar imidazole rings coordinated to Ni with an extensive network of hydrogen bonding.

**Introduction.** Crystals of the title compound were prepared by slow evaporation of an aqueous solution of NiCl<sub>2</sub> and imidazole (Im) in a molecular ratio of approximately 1:6. Analysis calculated from the empirical formula: C, 35.4; H, 5.3; N, 27.6; found: C, 34.2; H, 5.0; N, 27.0. Precession photographs of several

crystals did not reveal symmetry greater than anorthic and the reduced cell matrix (No. 31, *International Tables for X-ray Crystallography*, 1969) confirmed that the metric and crystal symmetry are triclinic. The structure was successfully refined in the space group *P* $\bar{1}$ .

A spherical crystal of 0.1 mm radius was mounted on a diffractometer. With Mo *K* $\alpha$  radiation ( $\lambda$  = 0.71069 Å) and  $\theta_{\max}$  = 30°, a total of 3940 reflections were measured with the stationary-crystal stationary-counter technique. 508 of these reflections with  $I < 2\sigma(I)$  { $\sigma(I)$  = [peak + background + (0.005 peak)<sup>1/2</sup>] } were included in the refinement only if  $F_c > F_o$ . All reflections were included in the calculation of both *R* and *R<sub>w</sub>*. A graph giving peak/integrated ratio as a function of 2 $\theta$  was determined. The data were then converted to integrated intensities and corrected for Lorentz and polarization factors. No absorption correction was re-

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