N5A—C9aA—C9A	112.3 (2)	N5B—C9aB—C9B	112.2 (2)
C2A—C11A—C12A	121.2 (2)	C2B—C11B—C12B	120.5 (2)
C2A—C11A—C16A	120.4 (2)	C2B—C11B—C16B	121.3 (2)
C12A—C11A—C16A	118.4 (2)	C12B—C11B—C16B	118.2 (3)
C11A—C12A—C13A	120.8 (3)	C11B—C12B—C13B	120.8 (3)
C12A—C13A—C14A	120.5 (3)	C12B—C13B—C14B	120.9 (4)
C13A—C14A—C15A	119.4 (3)	C13B—C14B—C15B	119.4 (4)
C14A—C15A—C16A	120.0 (3)	C14B—C15B—C16B	120.6 (3)
C11A—C16A—C15A	120.7 (2)	C11B—C16B—C15B	120.1 (3)
C6A—C19A—N20A	174.5 (3)	C6B—C19B—N20B	173.6 (3)

N5A—C9aA—O1A—C2A	64.0 (2)
O1A—C2A—C3A—N4A	50.5 (2)
C2A—C3A—N4A—N5A	−52.8 (2)
C3A—N4A—N5A—C9aA	58.9 (2)
N4A—N5A—C9aA—O1A	−63.0 (2)
C9aA—N5A—C6A—C7A	54.8 (2)
N5A—C6A—C7A—C8A	−57.5 (3)
C6A—C7A—C8A—C9A	57.3 (3)
C7A—C8A—C9A—C9aA	−52.9 (3)
C8A—C9A—C9aA—N5A	48.3 (2)
C9A—C9aA—N5A—C6A	−50.3 (2)
O1A—C2A—C11A—C12A	−130.7 (3)
N5A—C6A—C19A—N20A	−177.5 (4)
N5B—C9aB—O1B—C2B	64.3 (2)
O1B—C2B—C3B—N4B	50.4 (2)
C2B—C3B—N4B—N5B	−52.3 (2)
C3B—N4B—N5B—C9aB	58.6 (2)
N4B—N5B—C9aB—O1B	−62.9 (2)
C9aB—N5B—C6B—C7B	55.3 (2)
N5B—C6B—C7B—C8B	−59.0 (3)
C6B—C7B—C8B—C9B	58.7 (3)
C7B—C8B—C9B—C9aB	−53.6 (3)
C8B—C9B—C9aB—N5B	48.7 (2)
C9B—C9aB—N5B—C6B	−50.3 (2)
O1B—C2B—C11B—C12B	−135.6 (3)
N5B—C6B—C19B—N20B	171.2 (5)

Isotropic temperature factors for H atoms were assigned as 1.10 × $U_{\rm eq}$ of the bonded atom. Data collection: Philips PW1100/20 software (1974). Data reduction: *PHIL* (Riche, 1981). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *R3M* (Riche, 1983); *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *ACTACIF* (Riche, 1992).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry including bond distances and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71197 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: PA1042]

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β-Cytidine, $C_9H_{13}N_3O_5$

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Abstract

β-Cytidine exhibits extensive three-dimensional hydrogen-bonding interactions in its crystal structure; every H atom bonded to an N or O atom (five in total) is involved and each β-cytidine molecule contributes to ten intermolecular hydrogen bonds. The O···O and O···N distances range from 2.707 (4) to 2.947 (4) Å. An intramolecular C—H···O interaction is observed with a C···O distance of 3.245 (5) Å.

Comment

The crystal structure of β-cytidine has been reported by Furberg (1950) and by Furberg, Petersen & Rømming (1965). Both studies used photographic data and reported *R* factors of 0.17–0.19 and 0.056, respectively. The present structure determination of β-cytidine was performed as part of a study of new designs and materials for X-ray collimators (Ward, 1992); it used data collected by a modern automated diffractometer and led to *R* = 0.029. As the previous studies used data collection and computational methods that are no longer considered adequate, and as no other structure report of β-cytidine was discovered, it was decided to report the present structure. The crystal structure determination of α-cytidine, which used diffractometer data and led to an *R* factor of 0.033, was reported by Post, Birnbaum, Huber & Shugar (1977).

The description of the molecular structure by Furberg *et al.* (1965) applies to the present structure, if one allows for the change in atom numbering to be consistent with the conventions of Post *et al.* (1977) and *Chemical Abstracts*, and also for the coordinate-origin translation of $-\frac{1}{2}, -\frac{1}{2}, -\frac{1}{2}$. The average difference between the C—C, C—N and C—O bond distances of Furberg *et al.* (1965), and those of the present structure is 0.009 Å; no difference exceeds three times the corresponding e.s.d. value of Furberg *et al.* For bond angles not involving H atoms, the average difference is 0.45°, again with no difference exceeding three times the corresponding e.s.d. value of Furberg *et al.*

Bond-distance differences with those of Post *et al.* (1977) average only 0.007 Å; bond-angle differences are large (average 1.5°) and are likely to be the result of the different molecular configurations and crystal packing.

The least-squares-plane analysis of the cytosine part of the molecule is also very similar to that reported by Furberg *et al.* (1965). The six-atom ring plane has a mean deviation of 0.0139 Å with N3 lying 0.022 (3) Å from the plane, whereas the five-atom plane (excluding N3) has a mean deviation of 0.0021 Å and N3 situated 0.055 Å from the plane.

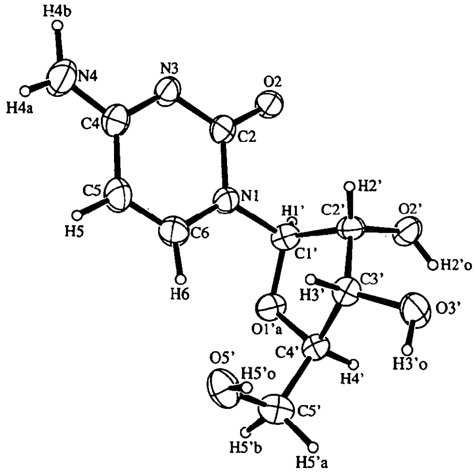

Fig. 1. β-Cytidine molecule showing the labelling of the atoms. Thermal ellipsoids are shown at 50% probability.

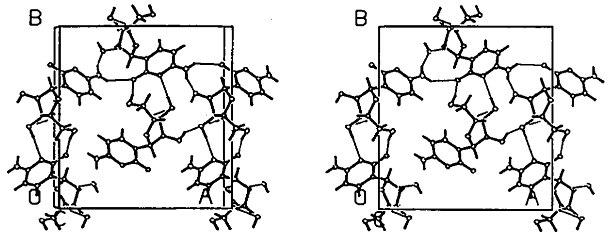

Fig. 2. Packing diagram including hydrogen bonds viewed down the *c* axis. Thermal ellipsoids are shown at 20% probability.

With the H atoms in the present structure located from difference maps and independently refined, the hydrogen-bonding system reported by Furberg *et al.* (1965) is confirmed. The N···O and O···O distances deviate by an average of only 0.0045 Å; N4···O2′ shows the largest deviation, being 0.015 Å longer in the present structure. The C···O distance of the intramolecular C—H···O interaction is also 0.015 Å longer in the present structure.

Experimental

Crystal data

$C_9H_{13}N_3O_5$
$M_r = 243.22$
Orthorhombic
$P2_12_12_1$
$a = 13.980$ (4) Å
$b = 14.788$ (3) Å
$c = 5.119$ (3) Å
$V = 1058.3$ (7) Å^3
$Z = 4$
$D_x = 1.526$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 7.8$–$9.15°$
$\mu = 0.118$ mm^{-1}
$T = 296$ (1) K
Prism
0.20 × 0.20 × 0.20 mm
Colorless

Data collection

Rigaku MSC/AFC-6*S* diffractometer
$\theta/2\theta$ scans [2θ scan width $(0.84 + 0.30\tan\theta)°$, θ scan rate 4° min^{-1}]
Absorption correction: empirical
$T_{\min} = 0.97$, $T_{\max} = 1.00$
1134 measured reflections
1134 independent reflections
805 observed reflections [$I > 3\sigma(I)$]

$\theta_{\max} = 25.0°$
$h = 0 \rightarrow 16$
$k = 0 \rightarrow 17$
$l = 0 \rightarrow 6$
3 standard reflections monitored every 150 reflections intensity variation: −1.11%

Refinement

Refinement on *F*
Final $R = 0.0287$
$wR = 0.0330$
$S = 1.28$
805 reflections
207 parameters
All H-atom parameters refined
Weighting scheme based on measured e.s.d.'s

$(\Delta/\sigma)_{\max} = 0.00$
$\Delta\rho_{\max} = 0.13$ e Å^{-3}
$\Delta\rho_{\min} = -0.15$ e Å^{-3}
Extinction correction: Zachariasen
Extinction coefficient: 0.11408×10^{-5}
Atomic scattering factors from Cromer (1974) and Cromer & Waber (1974)

Data collection: *COLLECT* (Molecular Structure Corporation, 1988). Cell refinement: *REFINE* (Molecular Structure Corporation, 1988). Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *MITHRIL* (Gilmore, 1984); *DIRDIF* (Beurskens, 1984). Program(s) used to refine structure: *TEXSAN LS*. Molecular graphics: *ORTEP* (Johnson, 1976). Software used to prepare material for publication: *TEXSAN FINISH*.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
O1′*a*	0.9864 (2)	0.6011 (1)	1.2658 (5)	0.0322
O2	0.9539 (2)	0.7915 (1)	0.7096 (5)	0.0338
O2′	0.7860 (2)	0.6300 (2)	1.0305 (6)	0.0375
O3′	0.8251 (2)	0.4480 (2)	0.9380 (6)	0.0384
O5′	1.0829 (2)	0.4248 (2)	1.1388 (6)	0.0422
N1	1.0374 (2)	0.6742 (2)	0.8809 (6)	0.0300
N3	1.1024 (2)	0.7668 (2)	0.5473 (6)	0.0315
N4	1.2468 (2)	0.7338 (2)	0.3689 (8)	0.0473
C1′	0.9552 (2)	0.6566 (2)	1.0589 (7)	0.0289
C2	1.0288 (2)	0.7465 (2)	0.7082 (7)	0.0293
C2′	0.8746 (3)	0.6055 (2)	0.9188 (8)	0.0280
C3′	0.9025 (3)	0.5081 (2)	0.9733 (7)	0.0263
C4	1.1800 (2)	0.7130 (2)	0.5405 (8)	0.0335
C4′	0.9404 (3)	0.5125 (2)	1.2503 (7)	0.0276
C5	1.1895 (3)	0.6391 (3)	0.7147 (9)	0.0417
C5′	1.0098 (3)	0.4392 (3)	1.3268 (8)	0.0384
C6	1.1181 (3)	0.6225 (2)	0.8806 (9)	0.0382
H1′	0.934 (2)	0.716 (2)	1.133 (7)	0.027 (8)
H2′*o*	0.759 (3)	0.587 (2)	1.106 (8)	0.04 (1)
H2′	0.877 (2)	0.618 (2)	0.743 (7)	0.025 (9)
H3′*o*	0.850 (3)	0.392 (3)	0.96 (1)	0.09 (1)
H3′	0.955 (2)	0.495 (2)	0.858 (7)	0.028 (9)
H4*a*	1.296 (3)	0.703 (3)	0.35 (1)	0.08 (1)
H4*b*	1.244 (3)	0.785 (3)	0.240 (9)	0.08 (1)
H4′	0.887 (2)	0.512 (2)	1.363 (7)	0.023 (9)
H5	1.247 (2)	0.604 (2)	0.706 (7)	0.031 (9)
H5′*o*	1.061 (3)	0.380 (3)	1.011 (8)	0.07 (1)
H5′*a*	0.972 (2)	0.377 (2)	1.358 (8)	0.033 (9)
H5′*b*	1.048 (3)	0.458 (3)	1.492 (8)	0.06 (1)
H6	1.117 (2)	0.571 (2)	0.988 (7)	0.04 (1)

Table 2. *Geometric parameters* (Å, °)

O1′*a*—C1′	1.410 (4)	N3—C2	1.351 (4)
O1′*a*—C4′	1.460 (4)	N3—C4	1.346 (4)
O2—C2	1.241 (4)	N4—C4	1.319 (5)
O2′—C2′	1.412 (4)	C1′—C2′	1.534 (5)
O3′—C3′	1.412 (4)	C2′—C3′	1.518 (5)
O5′—C5′	1.419 (5)	C3′—C4′	1.515 (5)
N1—C1′	1.490 (4)	C4—C5	1.417 (5)
N1—C2	1.393 (4)	C4′—C5′	1.507 (5)
N1—C6	1.363 (4)	C5—C6	1.334 (5)
C1′—O1′*a*—C4′	110.2 (3)	C1′—C2′—C3′	101.1 (3)
C1′—N1—C2	117.2 (3)	O3′—C3′—C2′	112.2 (3)
C1′—N1—C6	122.7 (3)	O3′—C3′—C4′	114.5 (3)
C2—N1—C6	120.1 (3)	C2′—C3′—C4′	102.8 (3)
C2—N3—C4	119.9 (3)	N3—C4—N4	116.8 (3)
O1′*a*—C1′—N1	108.8 (3)	N3—C4—C5	121.0 (3)
O1′*a*—C1′—C2′	107.0 (3)	N4—C4—C5	122.2 (4)
N1—C1′—C2′	111.5 (3)	O1′*a*—C4′—C3′	104.1 (3)
O2—C2—N1	118.7 (3)	O1′*a*—C4′—C5′	110.3 (3)
O2—C2—N3	121.8 (3)	C3′—C4′—C5′	115.9 (3)
N1—C2—N3	119.5 (3)	C4—C5—C6	118.2 (4)
O2′—C2′—C1′	109.2 (3)	O5′—C5′—C4′	113.3 (3)
O2′—C2′—C3′	113.2 (3)	N1—C6—C5	121.1 (4)
O1′*a*—C1′—N1—C2	−162.6 (3)	N1—C2—N3—C4	5.2 (5)
O1′*a*—C1′—N1—C6	18.1 (4)	N1—C6—C5—C4	0.9 (6)
O1′*a*—C1′—C2′—O2′	91.3 (3)	N3—C2—N1—C1′	178.3 (3)
O1′*a*—C1′—C2′—C3′	−28.3 (3)	N3—C2—N1—C6	−2.4 (5)
O1′*a*—C4′—C3′—O3′	−156.2 (3)	N3—C4—C5—C6	2.0 (6)
O1′*a*—C4′—C3′—C2′	−34.3 (3)	N4—C4—N3—C2	176.3 (4)
O1′*a*—C4′—C5′—O5′	−70.9 (4)	N4—C4—C5—C6	−179.5 (4)
O2—C2—N1—C1′	−0.5 (5)	C1′—O1′*a*—C4′—C3′	17.0 (4)
O2—C2—N1—C6	178.7 (3)	C1′—O1′*a*—C4′—C5′	142.0 (3)
O2—C2—N3—C4	−175.9 (3)	C1′—N1—C6—C5	178.6 (4)
O2′—C2′—C1′—N1	−149.8 (3)	C1′—C2′—C3′—C4′	37.6 (3)
O2′—C2′—C3′—O3′	44.4 (4)	C2—N1—C1′—C2′	79.5 (4)
O2′—C2′—C3′—C4′	−79.1 (4)	C2—N1—C6—C5	−0.7 (6)
O3′—C3′—C2′—C1′	161.1 (3)	C2—N3—C4—C5	−5.1 (5)
O3′—C3′—C4′—C5′	82.4 (4)	C2′—C1′—O1′*a*—C4′	7.3 (4)
O5′—C5′—C4′—C3′	47.1 (4)	C2′—C1′—N1—C6	−99.7 (4)
N1—C1′—O1′*a*—C4′	−113.4 (3)	C2′—C3′—C4′—C5′	−155.7 (3)
N1—C1′—C2′—C3′	90.6 (3)		

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

D	H	A	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O2′	H2′*o*	O3′^{i}	0.84 (4)	2.13 (4)	2.845 (4)	143 (3)
O3′	H3′*o*	N3′^{ii}	0.91 (4)	1.96 (5)	2.866 (3)	171 (5)
O5′	H5′*o*	O2^{ii}	0.99 (4)	1.74 (5)	2.707 (4)	168 (4)
N4	H4*a*	O2^{iii}	0.84 (4)	2.22 (4)	2.947 (4)	145 (4)
N4	H4*b*	O2′^{iii}	1.00 (4)	1.96 (5)	2.922 (5)	159 (4)
C6	H6	O5′^{iv}	0.94 (4)	2.34 (4)	3.245 (5)	160 (4)

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

The unit cell was also determined with Cu $K\alpha$ radiation (λ = 1.540598 Å at 296 K) as a = 13.9839 (10), b = 14.7714 (10), c = 5.1146 (11) Å; V = 1056.47 (23) Å^3; 25 reflections, θ = 27.60–33.17°.

Refinements of both enantiomers were made. The present enantiomer gave insignificantly better results; therefore, the absolute structure has not been determined by this work. However, the present structure is in agreement with that of the D enantiomorph of 2′-deoxycytidine hydrochloride (Subramanian & Hunt, 1970).

The β-cytidine crystal is distributed by Molecular Structure Corporation as the standard/alignment crystal for the Rigaku MSC/AFC-6S diffractometer.

The X-ray diffractometer was provided in part by the National Science Foundation Chemical Instrumentation Grant CHE-8908088.

Lists of structure factors, anisotropic thermal parameters, least-squares-planes data, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71231 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1018]

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Structure of 1-Benzoyl-4,4,5,5-tetramethyl-2-phenylimidazoline Hydrate

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Abstract

The imidazoline ring is in an envelope conformation and the two phenyl rings form a dihedral angle of 49.3° with respect to each other. An intermolecular hydrogen bond is formed between the water and the unsubstituted N atom.

Comment

Imines can be oxidized directly to nitrones by means of aqueous potassium permanganate at pH 4.1 (Christensen & Jorgensen, 1989*a*,*b*). We attempted to apply this methodology to the oxidation of *N*,*N*′-dibenzylidine-2,3-dimethyl-2,3-butanediamine and instead of obtaining the bis nitrone, the title compound was produced unexpectedly.

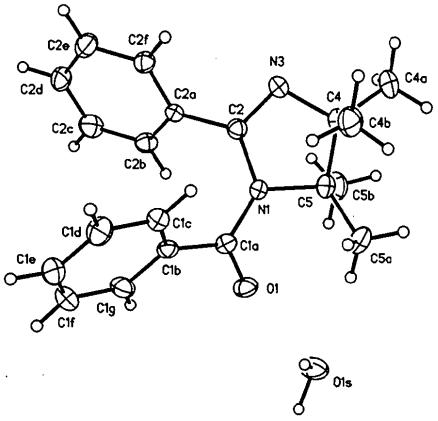

Fig. 1. Structure of the title compound showing 20% probability displacement ellipsoids.

Experimental

Crystal data

$C_{20}H_{22}N_2O.H_2O$
$M_r = 324.4$
Orthorhombic
$P2_12_12_1$
$a = 9.755$ (4) Å
$b = 10.800$ (4) Å
$c = 17.797$ (7) Å
$V = 1875.0$ (12) Å^3
$Z = 4$
$D_x = 1.149$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 11.53-17.0°$
$\mu = 0.074$ mm^{-1}
$T = 293$ (2) K
Irregular
0.52 × 0.48 × 0.22 mm
Colorless, translucent

Data collection

Siemens *R3m/V* four-circle diffractometer
Profile-fitted $\theta/2\theta$ scans
Absorption correction: none
1492 measured reflections
1438 independent reflections
1220 observed reflections [$I > 2\sigma(I)$]

$R_{\text{int}} = 0.0161$
$\theta_{\max} = 22.55°$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 11$
$l = 0 \rightarrow 19$
3 standard reflections monitored every 97 reflections intensity variation: ±2.0%

Refinement

Refinement on F^2
Final $R(F) = 0.0440$
$wR(F^2) = 0.1148$
$S = 1.111$
1436 reflections
217 parameters
H atoms: riding
Calculated weights $w = 1/[\sigma^2(F_o^2) + (0.0737P)^2 + 0.1920P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.052$
$\Delta\rho_{\max} = 0.115$ e Å^{-3}
$\Delta\rho_{\min} = -0.211$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Refinement on F^2 for all reflections except for two with very negative F^2 or those flagged by the user for potential systematic errors. Weighted R factors $wR(F^2)$ and all values of the goodness-of-fit S are based on F^2, conventional R factors $R(F)$ are based on F, with F set to zero for negative F^2. The observed criterion [$F^2 > 2\sigma(F^2)$] is used only for calculating observed R *etc.* and is not relevant to the choice of reflections for refinement. R factors based on F^2 are statistically about twice as large as those based on F; R factors based on all data will be even larger.

Data collection: *SHELXTL-Plus* (Sheldrick, 1990). Cell refinement: *SHELXTL-Plus*. Data reduction: *SHELXTL-Plus*. Program(s) used to solve structure: *SHELXTL-Plus*. Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXL93*.

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