Griesbaum, K., Rao, V. V. R. & Leifker, G. (1982). *J. Org. Chem.* **47**, 4975–4981.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Rubenstein, M. & Rubenstein, S. (1987). *CHEM3D. The Molecular Modelling System.* Cambridge Scientific Computing, Inc., Cambridge, Massachusetts, USA.

Stewart, J. M. (1980). Editor. *The XRAY System of Crystallographic Programs.* Technical Report TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland, USA.

Acta Cryst. (1993). C**49**, 1786–1789

X-ray Diffraction Analysis of Two Hexahydropyrido[2,1-*b*][1,3,4]oxadiazines: Evidence for the Effect of Steric Congestion on Anion Formation at the Aminonitrile Centre

A. Chiaroni, C. Riche,* L. Guerrier, J. Royer, D. S. Grierson and H.-P. Husson

Institut de Chimie des Substances Naturelles, CNRS, 91198 Gif sur Yvette CEDEX, France

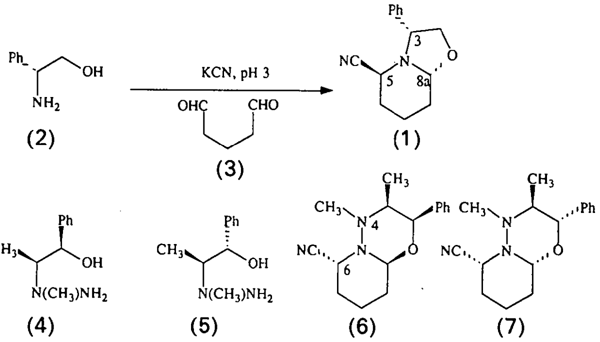
(*Received* 9 *December* 1992; *accepted* 24 *February* 1993)

Abstract

The X-ray crystal structures of (2*R*,3*S*,6*R*,9a*S*)- and (2*S*,3*S*,6*R*,9a*R*)-3,4-dimethyl-2-phenyl-3,4,7,8,9,9a-hexahydro-2*H*,6*H*-pyrido[2,1-*b*][1,3,4]oxadiazine-6-carbonitrile, (6) and (7), respectively, revealed that the six-membered rings are chair shaped and the ring junctions are *cis*. Furthermore, it was found that the C6 H atom is hindered by the axial methyl group substituted at C3 in (6) and that the C6 H atom in compound (7) is similarly protected by the presence of the equatorial methyl group on N4. These results provide insight into the reactivity of these molecules.

Comment

The versatility of 3-phenyl-2,3,6,7,8,8a-hexahydro-5*H*-oxazolo[3,2-*a*]pyridine-5-carbonitrile (1) as a monochiral 1,4-dihydropyridine equivalent is illustrated by the concise enantioselective synthesis of a variety of different piperidine alkaloids (Guerrier, Royer, Grierson & Husson, 1983; Yue, Royer & Husson, 1990; Lienard, Royer, Quirion & Husson, 1991; Rubiralta, Giralt & Diez, 1991). This compound is obtained as a crystalline solid in a high-yielding one-step procedure involving condensation of (−)-2-amino-2-phenylethanol (2) with glutaraldehyde (3) in the presence of KCN (Bonin, Grierson, Royer & Husson, 1991). An essential feature of the reactivity of (1) is that the N atom of aminophenylethanol is incorporated into the piperidine ring of the target molecule, and that once the role of the chiral benzylic carbon centre C3 has been fulfilled, the piperidine N atom can be liberated conveniently under hydrogenolysis conditions. An alternative strategy which permits both liberation of the piperidine N atom and recovery of the aminoalcohol component intact involves the use of the hydrazine based compounds (6) and (7). These monochiral compounds were prepared in an analogous fashion to (1) from the hydrazine derivatives (4) and (5) of (1*R*,2*S*)-(−)-ephedrine and (1*S*,2*S*)-(+)-pseudoephedrine, respectively (Takahashi, Senda & Higashiyama, 1991). Interestingly, problems were encountered during attempts to generate and alkylate the anion formed at C6 through reaction of (6) and (7) with alkyllithium bases or lithium diisopropylamide. To corroborate our view that the C6 position in these compounds is highly congested, thus prohibiting both efficient anion formation and subsequent approach of the electrophile to the anion centre, we have determined the structures of compounds (6) and (7) by X-ray diffraction.



In the asymmetric unit of compound (7) there are two independent molecules which adopt identical conformations. A systematic comparison of all bond distances, bond angles and torsion angles gave r.m.s. deviations of 0.006 Å, 0.5 and 3.1°, respectively.

In the structures of (6) and (7), the six-membered rings are chair shaped and the junctions of these rings are *cis*. However, the conformations of the two molecules (6) and (7) are quite different. The H atom at C9a and the lone pair at N5 point in different directions, being below the general plane of the molecule in (6) and above this plane in (7). The phenyl group is equatorial in the two molecules while the position of the cyano group at C6 is equatorial in (6) and axial in (7). The methyl groups at N4 and C3 are axial in (6) and equatorial in (7). The steric environment of H6 in compounds (6) and (7) is clearly depicted in the figures. In compound (6) (Fig. 1), H6, which is in an axial position, is in close contact with the methyl group C17 (linked to C3); H6···C17 2.61 and H6···H17a 2.09 Å.

In compound (7), H6, now in an equatorial position, is in close proximity to the methyl group C18 which is linked to N4. The short intramolecular contacts are H6···C18 (molecule *A* 2.47, molecule *B* 2.49 Å) and H6···H18*c* (molecule *A* 2.05, molecule *B* 2.08 Å). These observations confirm that C6 is in a sterically crowded environment and support our hypothesis concerning the non-reactivity of compounds (6) and (7).

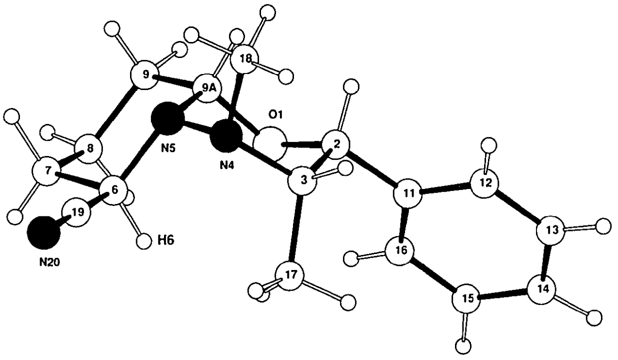


Fig. 1. Perspective view of compound (6).

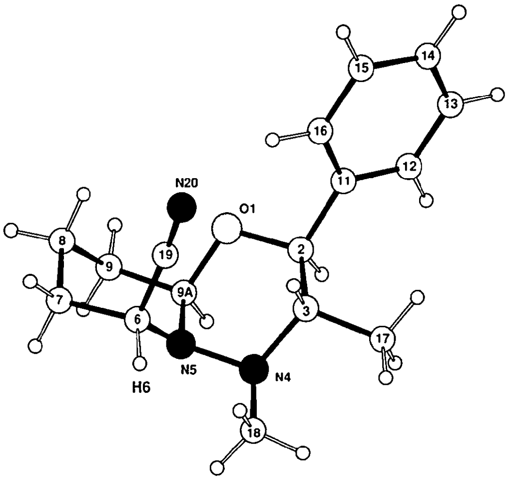


Fig. 2. Perspective view of compound (7).

Experimental

Compound (6)

Crystal data

$C_{16}H_{21}N_3O$
$M_r = 271.36$
Orthorhombic
$P2_12_12_1$
$a = 8.934\ (3)$ Å
$b = 11.556\ (4)$ Å
$c = 14.884\ (6)$ Å
$V = 1536.6\ (9)$ Å^3
$Z = 4$
$D_x = 1.17$ Mg m^{-3}

Cu *Kα* radiation
$\lambda = 1.5418$ Å
Cell parameters from 24 reflections
$\theta = 5.9\text{–}45.6°$
$\mu = 0.56$ mm^{-1}
$T = 293$ K
Prism
0.20 × 0.30 × 0.50 mm
Colourless

Data collection

Philips PW1100 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
5557 measured reflections
1618 independent reflections
1473 observed reflections $[I > 3.0\sigma(I)]$

$R_{\text{int}} = 0.044$
$\theta_{\max} = 67.94°$
$h = -10 \rightarrow 10$
$k = -13 \rightarrow 13$
$l = 0 \rightarrow 17$
3 standard reflections
frequency: 180 min
intensity variation: none

Refinement

Refinement on F
Final $R = 0.041$
$wR = 0.060$
$S = 0.82$
1473 reflections
244 parameters
Only coordinates of H atoms refined

Calculated weights $w = 1/[\sigma^2(F) + 0.0053F^2]$
$(\Delta/\sigma)_{\max} = 0.09$
$\Delta\rho_{\max} = 0.14$ e Å^{-3}
$\Delta\rho_{\min} = -0.19$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *for* (6)

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

	x	y	z	U_{eq}
O1	0.0003 (2)	0.5026 (1)	0.4570 (1)	0.058 (1)
C2	−0.1324 (3)	0.5661 (2)	0.4349 (1)	0.053 (2)
C3	−0.1913 (3)	0.6257 (2)	0.5208 (1)	0.050 (2)
N4	−0.2102 (2)	0.5411 (2)	0.5957 (1)	0.054 (2)
N5	−0.0894 (2)	0.4592 (2)	0.6050 (1)	0.058 (2)
C6	0.0293 (3)	0.5080 (2)	0.6602 (2)	0.058 (2)
C7	0.1541 (4)	0.4188 (3)	0.6768 (2)	0.077 (3)
C8	0.2169 (5)	0.3811 (4)	0.5868 (2)	0.093 (4)
C9	0.0931 (5)	0.3310 (3)	0.5297 (2)	0.087 (4)
C9a	−0.0385 (4)	0.4123 (2)	0.5189 (2)	0.065 (2)
C11	−0.1001 (3)	0.6514 (2)	0.3606 (1)	0.051 (2)
C12	−0.2189 (3)	0.6993 (2)	0.3131 (2)	0.058 (2)
C13	−0.1934 (4)	0.7825 (2)	0.2482 (2)	0.067 (2)
C14	−0.0490 (4)	0.8193 (2)	0.2303 (2)	0.070 (3)
C15	0.0702 (3)	0.7702 (3)	0.2756 (2)	0.071 (3)
C16	0.0448 (3)	0.6869 (2)	0.3406 (2)	0.062 (2)
C17	−0.0956 (3)	0.7292 (2)	0.5485 (2)	0.057 (2)
C18	−0.3539 (4)	0.4803 (3)	0.5925 (2)	0.072 (3)
C19	−0.0351 (3)	0.5475 (2)	0.7457 (2)	0.062 (2)
N20	−0.0763 (3)	0.5790 (3)	0.8138 (2)	0.084 (3)

Table 2. *Geometric parameters* (Å, °) *for* (6)

O1—C2	1.432 (3)	C6—C19	1.469 (3)
O1—C9a	1.435 (3)	C7—C8	1.516 (5)
C2—C3	1.544 (3)	C8—C9	1.510 (6)
C2—C11	1.509 (3)	C9—C9a	1.514 (5)
C3—N4	1.493 (3)	C11—C12	1.390 (3)
C3—C17	1.527 (3)	C11—C16	1.391 (3)
N4—N5	1.442 (3)	C12—C13	1.383 (4)
N4—C18	1.464 (4)	C13—C14	1.385 (5)
N5—C6	1.455 (3)	C14—C15	1.382 (4)
N5—C9a	1.464 (3)	C15—C16	1.384 (4)
C6—C7	1.539 (4)	C19—N20	1.138 (4)
C2—O1—C9a	108.7 (2)	C6—C7—C8	108.6 (3)
O1—C2—C3	108.7 (2)	C7—C8—C9	109.7 (3)
O1—C2—C11	110.2 (2)	C8—C9—C9a	113.0 (3)
C3—C2—C11	112.3 (2)	O1—C9a—N5	111.6 (2)
C2—C3—N4	111.4 (2)	O1—C9a—C9	109.4 (2)
C2—C3—C17	112.5 (2)	N5—C9a—C9	112.2 (2)
N4—C3—C17	112.0 (2)	C2—C11—C12	119.2 (2)
C3—N4—N5	114.6 (2)	C2—C11—C16	121.8 (2)
C3—N4—C18	112.9 (2)	C12—C11—C16	119.0 (2)

N5—N4—C18	110.2 (2)	C11—C12—C13	120.5 (2)
N4—N5—C6	110.2 (2)	C12—C13—C14	120.1 (3)
N4—N5—C9a	113.0 (2)	C13—C14—C15	119.8 (3)
C6—N5—C9a	114.3 (2)	C14—C15—C16	120.1 (3)
N5—C6—C7	111.1 (2)	C11—C16—C15	120.5 (2)
N5—C6—C19	108.9 (2)	C6—C19—N20	175.8 (3)
C7—C6—C19	110.6 (2)		
N5—C9a—O1—C2	−65.6 (2)	C6—C7—C8—C9	−58.1 (3)
O1—C2—C3—N4	−53.1 (2)	C7—C8—C9—C9a	54.5 (3)
C2—C3—N4—N5	42.9 (2)	C8—C9—C9a—N5	−49.3 (3)
C3—N4—N5—C9a	−43.3 (2)	C9—C9a—N5—C6	50.1 (2)
N4—N5—C9a—O1	54.1 (2)	O1—C2—C11—C12	163.1 (3)
C9a—N5—C6—C7	−55.6 (2)	N5—C6—C19—N20	169.6 (4)
N5—C6—C7—C8	59.0 (3)		

Compound (7)

Crystal data

$C_{16}H_{21}N_3O$
$M_r = 271.36$
Monoclinic
$P2_1$
$a = 11.756$ (4) Å
$b = 7.564$ (3) Å
$c = 17.261$ (8) Å
$\beta = 99.78$ (5)°
$V = 1512.6$ (10) Å^3
$Z = 4$
$D_x = 1.19$ Mg m^{-3}

Cu *Kα* radiation
$\lambda = 1.5418$ Å
Cell parameters from 24 reflections
$\theta = 5.0$–47.6°
$\mu = 0.57$ mm^{-1}
$T = 293$ K
Prism
0.60 × 0.70 × 0.50 mm
Colourless

Data collection

Philips PW1100 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
3055 measured reflections
2968 independent reflections
2861 observed reflections [$I > 3.0\sigma(I)$]

$R_{\rm int} = 0.008$
$\theta_{\rm max} = 67.93°$
$h = -14 \rightarrow 13$
$k = 0 \rightarrow 9$
$l = 0 \rightarrow 20$
3 standard reflections
frequency: 180 min
intensity variation: none

Refinement

Refinement on *F*
Final $R = 0.052$
$wR = 0.064$
$S = 0.51$
2842 reflections
180 parameters
H-atom parameters not refined

Calculated weights $w = 1/[\sigma^2(F) + 0.0004F^2]$
$(\Delta/\sigma)_{\rm max} = 0.18$
$\Delta\rho_{\rm max} = 0.24$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.24$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 3. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *for* (7)

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

	x	y	z	$U_{\rm eq}$
O1*A*	0.6912 (1)	0.4760 (1)	0.4904 (1)	0.040 (1)
C2*A*	0.6429 (2)	0.5554 (3)	0.5527 (1)	0.039 (2)
C3*A*	0.6884 (2)	0.7448 (3)	0.5694 (1)	0.042 (2)
N4*A*	0.6738 (2)	0.8424 (3)	0.4945 (1)	0.047 (2)
N5*A*	0.7228 (2)	0.7550 (3)	0.4337 (1)	0.043 (2)
C6*A*	0.8484 (2)	0.7553 (4)	0.4399 (1)	0.052 (2)
C7*A*	0.8816 (3)	0.6667 (6)	0.3670 (2)	0.071 (3)
C8*A*	0.8339 (3)	0.4809 (5)	0.3586 (2)	0.073 (3)
C9*A*	0.7042 (3)	0.4842 (4)	0.3540 (1)	0.061 (3)
C9a*A*	0.6669 (2)	0.5838 (3)	0.4209 (1)	0.043 (2)
C11*A*	0.6683 (2)	0.4366 (3)	0.6233 (1)	0.041 (2)
C12*A*	0.5817 (2)	0.3867 (4)	0.6647 (2)	0.057 (2)
C13*A*	0.6069 (3)	0.2858 (5)	0.7322 (2)	0.069 (3)
C14*A*	0.7184 (3)	0.2324 (4)	0.7596 (2)	0.066 (3)
C15*A*	0.8049 (2)	0.2750 (4)	0.7174 (2)	0.057 (2)
C16*A*	0.7797 (2)	0.3777 (4)	0.6498 (1)	0.049 (2)
C17*A*	0.6206 (3)	0.8343 (5)	0.6258 (2)	0.074 (3)
C18*A*	0.7119 (3)	1.0266 (4)	0.5011 (2)	0.069 (3)
C19*A*	0.9186 (2)	0.6717 (4)	0.5109 (1)	0.049 (2)
N20*A*	0.9795 (2)	0.6123 (4)	0.5624 (1)	0.064 (2)
O1*B*	0.8466 (1)	1.2373 (2)	0.1804 (1)	0.039 (1)
C2*B*	0.7439 (2)	1.1369 (3)	0.1822 (1)	0.040 (2)
C3*B*	0.7485 (2)	0.9561 (3)	0.1416 (1)	0.041 (2)
N4*B*	0.8590 (2)	0.8707 (3)	0.1745 (1)	0.042 (2)
N5*B*	0.9595 (2)	0.9789 (3)	0.1726 (1)	0.040 (2)
C6*B*	0.9972 (2)	1.0043 (4)	0.0969 (2)	0.055 (2)
C7*B*	1.1094 (2)	1.1100 (5)	0.1096 (2)	0.070 (3)
C8*B*	1.0913 (2)	1.2880 (5)	0.1466 (2)	0.069 (3)
C9*B*	1.0503 (2)	1.2568 (4)	0.2245 (2)	0.055 (2)
C9a*B*	0.9457 (2)	1.1386 (3)	0.2168 (1)	0.039 (2)
C11*B*	0.6417 (2)	1.2466 (4)	0.1453 (1)	0.048 (2)
C12*B*	0.5436 (2)	1.2571 (6)	0.1803 (2)	0.069 (3)
C13*B*	0.4501 (3)	1.3568 (7)	0.1470 (2)	0.087 (4)
C14*B*	0.4500 (3)	1.4439 (6)	0.0781 (3)	0.087 (4)
C15*B*	0.5461 (3)	1.4355 (5)	0.0423 (2)	0.073 (3)
C16*B*	0.6416 (2)	1.3364 (4)	0.0752 (2)	0.056 (2)
C17*B*	0.6500 (3)	0.8412 (5)	0.1579 (2)	0.066 (3)
C18*B*	0.8753 (3)	0.6951 (4)	0.1433 (2)	0.063 (3)
C19*B*	0.9141 (2)	1.0895 (4)	0.0316 (1)	0.058 (2)
N20*B*	0.8602 (2)	1.1538 (5)	−0.0209 (1)	0.075 (3)

Table 4. *Geometric parameters* (Å, °) *for* (7)

O1*A*—C2*A*	1.430 (2)	O1*B*—C2*B*	1.432 (3)
O1*A*—C9a*A*	1.438 (3)	O1*B*—C9a*B*	1.436 (3)
C2*A*—C3*A*	1.539 (3)	C2*B*—C3*B*	1.541 (3)
C2*A*—C11*A*	1.502 (3)	C2*B*—C11*B*	1.509 (3)
C3*A*—N4*A*	1.474 (3)	C3*B*—N4*B*	1.475 (3)
C3*A*—C17*A*	1.517 (4)	C3*B*—C17*B*	1.513 (4)
N4*A*—N5*A*	1.441 (3)	N4*B*—N5*B*	1.443 (3)
N4*A*—C18*A*	1.462 (4)	N4*B*—C18*B*	1.459 (4)
N5*A*—C6*A*	1.463 (3)	N5*B*—C6*B*	1.462 (3)
N5*A*—C9a*A*	1.451 (3)	N5*B*—C9a*B*	1.453 (3)
C6*A*—C7*A*	1.534 (4)	C6*B*—C7*B*	1.526 (4)
C6*A*—C19*A*	1.496 (3)	C6*B*—C19*B*	1.505 (4)
C7*A*—C8*A*	1.511 (6)	C7*B*—C8*B*	1.521 (5)
C8*A*—C9*A*	1.514 (5)	C8*B*—C9*B*	1.522 (4)
C9*A*—C9a*A*	1.505 (4)	C9*B*—C9a*B*	1.507 (3)
C11*A*—C12*A*	1.392 (3)	C11*B*—C12*B*	1.392 (4)
C11*A*—C16*A*	1.385 (3)	C11*B*—C16*B*	1.389 (4)
C12*A*—C13*A*	1.381 (4)	C12*B*—C13*B*	1.376 (5)
C13*A*—C14*A*	1.376 (5)	C13*B*—C14*B*	1.359 (6)
C14*A*—C15*A*	1.386 (4)	C14*B*—C15*B*	1.380 (5)
C15*A*—C16*A*	1.391 (4)	C15*B*—C16*B*	1.388 (5)
C19*A*—N20*A*	1.135 (3)	C19*B*—N20*B*	1.125 (4)
C2*A*—O1*A*—C9a*A*	109.9 (1)	C2*B*—O1*B*—C9a*B*	109.9 (2)
O1*A*—C2*A*—C3*A*	111.3 (2)	O1*B*—C2*B*—C3*B*	111.4 (2)
O1*A*—C2*A*—C11*A*	108.0 (2)	O1*B*—C2*B*—C11*B*	108.2 (2)
C3*A*—C2*A*—C11*A*	112.9 (2)	C3*B*—C2*B*—C11*B*	112.6 (2)
C2*A*—C3*A*—N4*A*	108.4 (2)	C2*B*—C3*B*—N4*B*	108.1 (2)
C2*A*—C3*A*—C17*A*	109.4 (2)	C2*B*—C3*B*—C17*B*	109.8 (2)
N4*A*—C3*A*—C17*A*	110.1 (2)	N4*B*—C3*B*—C17*B*	109.2 (2)
C3*A*—N4*A*—N5*A*	114.1 (2)	C3*B*—N4*B*—N5*B*	114.4 (2)
C3*A*—N4*A*—C18*A*	114.9 (2)	C3*B*—N4*B*—C18*B*	114.8 (2)
N5*A*—N4*A*—C18*A*	109.8 (2)	N5*B*—N4*B*—C18*B*	110.4 (2)
N4*A*—N5*A*—C6*A*	118.0 (2)	N4*B*—N5*B*—C6*B*	118.0 (2)
N4*A*—N5*A*—C9a*A*	107.1 (2)	N4*B*—N5*B*—C9a*B*	107.1 (2)
C6*A*—N5*A*—C9a*A*	116.0 (2)	C6*B*—N5*B*—C9a*B*	116.0 (2)
N5*A*—C6*A*—C7*A*	109.5 (2)	N5*B*—C6*B*—C7*B*	109.0 (2)
N5*A*—C6*A*—C19*A*	117.9 (2)	N5*B*—C6*B*—C19*B*	117.9 (2)
C7*A*—C6*A*—C19*A*	107.8 (2)	C7*B*—C6*B*—C19*B*	108.6 (2)
C6*A*—C7*A*—C8*A*	110.1 (3)	C6*B*—C7*B*—C8*B*	110.2 (3)
C7*A*—C8*A*—C9*A*	110.1 (3)	C7*B*—C8*B*—C9*B*	108.8 (3)
C8*A*—C9*A*—C9a*A*	113.0 (2)	C8*B*—C9*B*—C9a*B*	113.0 (2)
O1*A*—C9a*A*—N5*A*	111.0 (2)	O1*B*—C9a*B*—N5*B*	110.9 (2)
O1*A*—C9a*A*—C9*A*	108.3 (2)	O1*B*—C9a*B*—C9*B*	108.6 (2)

N5*A*—C9a*A*—C9*A*	112.3 (2)	N5*B*—C9a*B*—C9*B*	112.2 (2)
C2*A*—C11*A*—C12*A*	121.2 (2)	C2*B*—C11*B*—C12*B*	120.5 (2)
C2*A*—C11*A*—C16*A*	120.4 (2)	C2*B*—C11*B*—C16*B*	121.3 (2)
C12*A*—C11*A*—C16*A*	118.4 (2)	C12*B*—C11*B*—C16*B*	118.2 (3)
C11*A*—C12*A*—C13*A*	120.8 (3)	C11*B*—C12*B*—C13*B*	120.8 (3)
C12*A*—C13*A*—C14*A*	120.5 (3)	C12*B*—C13*B*—C14*B*	120.9 (4)
C13*A*—C14*A*—C15*A*	119.4 (3)	C13*B*—C14*B*—C15*B*	119.4 (4)
C14*A*—C15*A*—C16*A*	120.0 (3)	C14*B*—C15*B*—C16*B*	120.6 (3)
C11*A*—C16*A*—C15*A*	120.7 (2)	C11*B*—C16*B*—C15*B*	120.1 (3)
C6*A*—C19*A*—N20*A*	174.5 (3)	C6*B*—C19*B*—N20*B*	173.6 (3)

N5*A*—C9a*A*—O1*A*—C2*A*	64.0 (2)
O1*A*—C2*A*—C3*A*—N4*A*	50.5 (2)
C2*A*—C3*A*—N4*A*—N5*A*	−52.8 (2)
C3*A*—N4*A*—N5*A*—C9a*A*	58.9 (2)
N4*A*—N5*A*—C9a*A*—O1*A*	−63.0 (2)
C9a*A*—N5*A*—C6*A*—C7*A*	54.8 (2)
N5*A*—C6*A*—C7*A*—C8*A*	−57.5 (3)
C6*A*—C7*A*—C8*A*—C9*A*	57.3 (3)
C7*A*—C8*A*—C9*A*—C9a*A*	−52.9 (3)
C8*A*—C9*A*—C9a*A*—N5*A*	48.3 (2)
C9*A*—C9a*A*—N5*A*—C6*A*	−50.3 (2)
O1*A*—C2*A*—C11*A*—C12*A*	−130.7 (3)
N5*A*—C6*A*—C19*A*—N20*A*	−177.5 (4)
N5*B*—C9a*B*—O1*B*—C2*B*	64.3 (2)
O1*B*—C2*B*—C3*B*—N4*B*	50.4 (2)
C2*B*—C3*B*—N4*B*—N5*B*	−52.3 (2)
C3*B*—N4*B*—N5*B*—C9a*B*	58.6 (2)
N4*B*—N5*B*—C9a*B*—O1*B*	−62.9 (2)
C9a*B*—N5*B*—C6*B*—C7*B*	55.3 (2)
N5*B*—C6*B*—C7*B*—C8*B*	−59.0 (3)
C6*B*—C7*B*—C8*B*—C9*B*	58.7 (3)
C7*B*—C8*B*—C9*B*—C9a*B*	−53.6 (3)
C8*B*—C9*B*—C9a*B*—N5*B*	48.7 (2)
C9*B*—C9a*B*—N5*B*—C6*B*	−50.3 (2)
O1*B*—C2*B*—C11*B*—C12*B*	−135.6 (3)
N5*B*—C6*B*—C19*B*—N20*B*	171.2 (5)

Isotropic temperature factors for H atoms were assigned as 1.10 × U_{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]

References

Bonin, M., Grierson, D. S., Royer, J. & Husson, H.-P. (1991). *Org. Synth.* **70**, 54-59.

Guerrier, L., Royer, J., Grierson, D. S. & Husson, H.-P. (1983). *J. Am. Chem. Soc.* **105**, 7754-7755.

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Lienard, P., Royer, J., Quirion, J.-C. & Husson, H.-P. (1991). *Tetrahedon Lett.* **32**, 2489-2492.

Riche, C. (1981). *PHIL. Program for Data Reduction for Philips PW1100 Diffractometer*. Institut de Chimie des Substances Naturelles du CNRS, France.

Riche, C. (1983). *R3M. Représentation et Manipulation de Modèles Moléculaires*. Institut de Chimie des Substances Naturelles du CNRS, France.

Riche, C. (1992). *ACTACIF. Logiciel de Préparation des Michiers CIF*. Institut de Chimie des Substances Naturelles du CNRS, France.

Rubiralta, M., Giralt, E. & Diez, A. (1991). *Piperidine. Structure, Preparation, Reactivity and Synthetic Applications of Piperidine and its Derivatives*, ch. 8. Amsterdam: Elsevier.

Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination*. Univ. of Cambridge, England.

Sheldrick, G. M. (1986). *SHELXS86. Program for the Solution of Crystal Structures*. Univ. of Göttingen, Germany.

Takahashi, H., Senda, T. & Higashiyama, K. (1991). *Chem. Pharm. Bull.* **39**, 836-842.

Yue, C., Royer, J. & Husson, H.-P. (1990). *J. Org. Chem.* **55**, 1140-1141.

Acta Cryst. (1993). C**49**, 1789–1792

β-Cytidine, $C_9H_{13}N_3O_5$

Donald L. Ward

Department of Chemistry, Michigan State University, East Lansing, MI 48824-1322, USA

(*Received* 20 *April* 1992; *accepted* 7 *April* 1993)

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

Acta Crystallographica Section C
ISSN 0108-2701 ©1993