Table 2. *Selected geometric parameters* (Å, °)

S—O1	1.498 (2)	C9—C10	1.507 (2)
S—C8	1.7766 (15)	C10—C14	1.532 (3)
S—C1	1.787 (2)	C10—C11	1.545 (2)
C1—C2	1.373 (2)	C11—C15	1.525 (2)
C1—C6	1.375 (3)	C11—C12	1.541 (2)
C2—C3	1.380 (3)	C12—C16	1.518 (2)
C3—C4	1.382 (3)	C12—C13	1.538 (2)
C4—C5	1.384 (5)	C15—O3	1.209 (2)
C4—C7	1.517 (3)	C15—N	1.370 (3)
C5—C6	1.383 (4)	C16—O2	1.197 (3)
C8—C9	1.325 (2)	C16—N	1.370 (2)
C8—C13	1.495 (2)		
O1—S—C8	106.49 (9)	C9—C10—C14	112.5 (2)
O1—S—C1	106.21 (8)	C9—C10—C11	109.49 (12)
C8—S—C1	98.10 (7)	C14—C10—C11	114.68 (15)
C2—C1—C6	120.3 (2)	C15—C11—C12	103.77 (13)
C2—C1—S	120.13 (13)	C15—C11—C10	114.44 (14)
C6—C1—S	119.5 (2)	C12—C11—C10	113.17 (11)
C1—C2—C3	120.0 (2)	C16—C12—C13	108.95 (14)
C4—C3—C2	121.1 (2)	C16—C12—C11	105.10 (12)
C3—C4—C5	117.7 (2)	C13—C12—C11	114.61 (12)
C3—C4—C7	120.1 (3)	C8—C13—C12	110.78 (13)
C5—C4—C7	122.2 (3)	O3—C15—N	124.2 (2)
C6—C5—C4	121.9 (2)	O3—C15—C11	127.0 (2)
C1—C6—C5	118.9 (2)	N—C15—C11	108.68 (15)
C9—C8—C13	120.42 (14)	O2—C16—N	125.6 (2)
C9—C8—S	119.56 (13)	O2—C16—C12	126.2 (2)
C13—C8—S	120.02 (12)	N—C16—C12	108.1 (2)
C8—C9—C10	118.26 (14)	C16—N—C15	114.23 (15)

The determination of the absolute configuration was possible from the known *R* configuration of the sulfoxide group in the starting diene; using the method described by Flack (1983), tha absolute configuration was confirmed by the calculations [*SHELXL93* (Sheldrick, 1993) option applied on non-centrosymmetric space groups]. H atoms were refined as rigid groups using the *AFIX* card of the *SHELXS93* program.

Data collection: *DIF4* (Stoe & Cie, 1988*a*). Cell refinement: *DIF4*. Data reduction: *REDU4* (Stoe & Cie, 1988*b*). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990), option *TREF*. Program(s) used to refine structure: *SHELXL93*. Molecular graphics: *ORTEP* (Johnson, 1965).

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5-Ethyl-2'-deoxycytidine, C$_{11}$H$_{17}$N$_3$O$_4$

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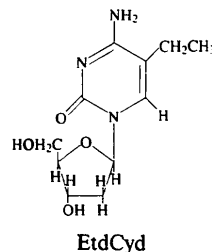
Abstract

The deoxyribose ring in the title compound adopts the twist conformation (^2_3T) with a glycosyl torsion angle of 231.0 (6)°. The pseudo-rotation parameters are P = 180.29 (5)° and τ_m = 38.3 (5)°. The exocyclic side chain at C5' has the *t* conformation [γ = 174.8 (11)°]. The ethyl group at C5 is on the same side of the pyrimidine plane as the O4' atom of the furanose ring.

Comment

5-Ethyl-2'-deoxyuridine (EtdUrd) has been shown to be a relatively potent inhibitor of herpes simplex virus (types 1 and 2) replication (De Clerq & Rosenwirth, 1985; De Clerq & Shugar, 1975; Schinazi, Scott, Peters, Rice & Nahmias, 1985). 5-Ethyl-2'-deoxycytidine (EtdCyd) was prepared as a potential antiviral agent, based on the potency of EtdUrd (De Clerq & Shugar, 1975; Kulikowski & Shugar, 1974).

EtdCyd

A perspective *ORTEP*II (Johnson, 1976) drawing of the molecule is shown in Fig. 1. The bond lengths and angles of EtdCyd are in the range reported for other 2'-deoxycytidine nucleosides (Young & Wilson, 1975; Kashino, Negishi & Hayatsu, 1988; Low, Tollin, Howie & Wilson, 1988; Sato, 1988; Silver-

ton, Quin & Haugwitz, 1988; Jia, Tourigny, Stuart, Delbaere & Gupta, 1990a). The glycosidic torsion angle [C2—N1—C1'—O4' 231.0 (6)°] has the anti conformation. EtdCyd exhibits the t conformation [γ = 174.8 (11)°]. Other deoxycytidine analogs that have the t conformation include: 5-methyl-2'-deoxycytidine (Sato, 1988), 5-methoxymethyl-N^4-methyl-2'-deoxycytidine (Jia, Tourigny, Stuart, Delbaere & Gupta, 1990b) and 5-hydroxymethyl-2'-deoxycytidine (Li, Kumar, Stuart, Delbaere & Gupta, 1995). EtdCyd adopts the C2'-endo—C3'-exo twist (^2_3T) conformation. The displacement of the C2' atom from the plane through C1', O4' and C4' is −0.308 (14) Å, and that of C3' is 0.317 (15) Å. A pseudo-rotational analysis of the furanose ring torsion angles in terms of two degrees of freedom for ring puckering (Altona & Sundaralingam, 1972) gave a phase angle (P) of 180.29 (5)° and a puckering amplitude (τ_m) of 38.3 (5)°. These results indicate that the deoxyribose moiety of EtdCyd has a typical S conformation. The ethyl group at C5 is on the same side of the pyrimidine plane as the O4' atom of the furanose ring. The pyrimidine ring is nearly planar; the largest deviation from the mean plane is for atom C4 with a displacement of 0.008 (7) Å. There are several intermolecular hydrogen bonds which are summarized in Table 3.

Fig. 2. Packing diagram for EtdCyd viewed down the a axis of the unit cell; intermolecular hydrogen bonds are represented by dashed lines.

Experimental

Crystal data

$C_{11}H_{17}N_3O_4$
$M_r = 255.27$
Orthorhombic
$P2_12_12_1$
$a = 4.600$ (3) Å
$b = 10.0410$ (10) Å
$c = 24.8350$ (3) Å
$V = 1147.1$ (8) Å^3
$Z = 4$
$D_x = 1.478$ Mg m^{-3}

Mo Kα radiation
$\lambda = 0.7107$ Å
Cell parameters from 24 reflections
$\theta = 10–12.5°$
$\mu = 0.11$ mm^{-1}
$T = 123$ K
Flat plate
$0.45 \times 0.15 \times 0.08$ mm
Colorless
Crystal source: vapour diffussion (EtOH/Et$_2$O)

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
1722 measured reflections
1224 independent reflections
918 observed reflections
[$I > 1.5\sigma(I)$]

$R_{int} = 0.018$
$\theta_{max} = 24.95°$
$h = -5 \rightarrow 5$
$k = -11 \rightarrow 11$
$l = -29 \rightarrow 29$
3 standard reflections frequency: 83.3 min intensity variation: 2.7%

Refinement

Refinement on F
$R = 0.054$
$wR = 0.052$
$S = 1.18$
918 reflections
73 parameters
$w = 1/[\sigma^2(F) + 0.0007F^2]$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.275$ e Å^{-3}
$\Delta\rho_{min} = -0.381$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from International Tables for X-ray Crystallography (1974, Vol. IV, Table 2.2B)
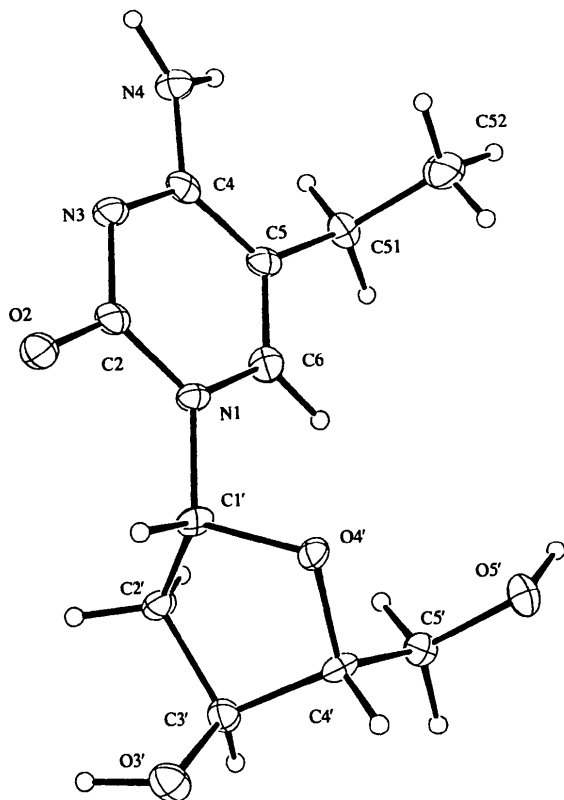

Fig. 1. Perspective ORTEPII (Johnson, 1976) view of EtdCyd showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.
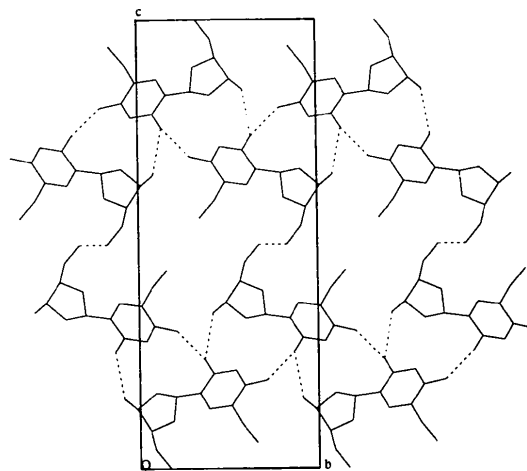
Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

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

	x	y	z	U_{eq}
O2	0.5910 (9)	0.3677 (3)	0.2409 (1)	0.019 (2)
O3′	0.5434 (9)	−0.0846 (3)	0.1547 (1)	0.020 (2)
O4′	0.5145 (9)	0.1900 (3)	0.1050 (1)	0.015 (2)
O5′	0.2877 (9)	0.1723 (3)	0.0002 (1)	0.020 (2)
N1	0.3061 (10)	0.3409 (4)	0.1671 (2)	0.014 (2)
N3	0.2985 (11)	0.5399 (4)	0.2176 (2)	0.016 (2)
N4	0.0036 (12)	0.7109 (4)	0.1951 (2)	0.018 (2)
C2	0.4042 (13)	0.4167 (5)	0.2099 (2)	0.015 (3)
C4	0.0967 (13)	0.5874 (5)	0.1843 (2)	0.014 (3)
C5	−0.0100 (13)	0.5144 (5)	0.1392 (2)	0.015 (3)
C51	−0.2254 (13)	0.5719 (5)	0.0992 (2)	0.017 (3)
C52	−0.0716 (15)	0.6589 (5)	0.0573 (2)	0.027 (3)
C6	0.0991 (13)	0.3917 (5)	0.1325 (2)	0.017 (3)
C1′	0.4154 (12)	0.2044 (5)	0.1597 (2)	0.012 (3)
C2′	0.1906 (13)	0.0953 (5)	0.1679 (2)	0.015 (3)
C3′	0.3030 (14)	−0.0143 (5)	0.1316 (2)	0.016 (3)
C4′	0.4129 (13)	0.0638 (5)	0.0836 (2)	0.013 (3)
C5′	0.1815 (13)	0.0893 (5)	0.0417 (2)	0.016 (3)
HO3′	0.471	−0.102	0.192	
HO5′	0.132	0.227	−0.017	
HN4B	0.102	0.775	0.220	

Table 2. *Selected geometric parameters* (Å, °)

O2—C2	1.255 (7)	N4—C4	1.339 (7)
O3′—C3′	1.432 (7)	C4—C5	1.425 (7)
O4′—C1′	1.439 (6)	C5—C51	1.517 (8)
O4′—C4′	1.450 (6)	C5—C6	1.341 (7)
O5′—C5′	1.413 (6)	C51—C52	1.533 (8)
N1—C2	1.384 (7)	C1′—C2′	1.520 (8)
N1—C6	1.380 (7)	C2′—C3′	1.514 (7)
N1—C1′	1.471 (6)	C3′—C4′	1.513 (7)
N3—C2	1.343 (6)	C4′—C5′	1.510 (8)
N3—C4	1.332 (7)		
C1′—O4′—C4′	109.3 (4)	C5—C51—C52	111.1 (5)
C2—N1—C6	120.0 (4)	N1—C6—C5	121.1 (5)
C2—N1—C1′	119.9 (4)	O4′—C1′—N1	108.7 (4)
C6—N1—C1′	120.1 (4)	O4′—C1′—C2′	105.7 (4)
C2—N3—C4	119.6 (4)	N1—C1′—C2′	114.9 (5)
O2—C2—N1	118.7 (4)	C1′—C2′—C3′	102.2 (4)
O2—C2—N3	121.4 (4)	O3′—C3′—C2′	112.5 (4)
N1—C2—N3	119.9 (5)	O3′—C3′—C4′	108.3 (5)
N3—C4—N4	115.5 (5)	C2′—C3′—C4′	101.9 (4)
N3—C4—C5	123.0 (5)	O4′—C4′—C3′	105.9 (4)
N4—C4—C5	121.5 (5)	O4′—C4′—C5′	109.3 (4)
C4—C5—C51	122.9 (5)	C3′—C4′—C5′	113.3 (5)
C4—C5—C6	116.2 (5)	O5′—C5′—C4′	111.1 (5)
C51—C5—C6	120.9 (4)		

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

D—H⋯A	H⋯A	D⋯A
O5′—HO5′⋯O5′ⁱ	2.08	2.78
O3′—HO3′⋯O2ⁱⁱ	1.72	2.71
N4—HN4B⋯O2ⁱⁱⁱ	1.95	2.91

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

The H atoms attached to O5′ and O3′ were found in a difference synthesis map. All other H atoms were placed by the computer program. H-atom parameters were not refined. U_{iso} of each H atom was assigned as 0.01 Å^2 + U_{eq} of the atom to which it is covalently bonded.

Data reduction: *NRCVAX DATRD2* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *NRCVAX SOLVER*. Program(s) used to refine structure: *NRCVAX LSTSQ*. Molecular graphics: *NRCVAX*. Software used to prepare material for publication: *NRCVAX TABLES* (Version of January 94).

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Two Centrosymmetric Conjugated Nitrones of the Diazabutadiene System

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Abstract

The X-ray structures of N-[2-(4-methoxyphenylimino)-2-phenylethylidene]phenylamine N-oxide (N11), $C_{21}H_{18}N_2O_2$, and N-[2-{4-(N,N-dimethylamino)phenylimino}-2-phenylethylidene]phenylamine N-oxide (N15),