sortent de ce plan de façon à peu près similaire [respectivement −0,757 (2) et −0,781 (2) Å], tandis que S(5) en est distant de −0,905 (1) Å. Le cycle pyridine est plan avec des écarts inférieurs à 0,024 (3) Å tandis que le benzène est plan dans la limite 0,020 (3) Å. L'angle entre ces deux cycles aromatiques vaut 110,6 (3)° [dans (II): 104,6 (5)°]. La distance entre leurs centres respectifs $X1$ et $X2$ est égale à 4,747 (3) Å, légèrement supérieure à celle dans (II) [4,697 (6) Å]. S(5) sort du plan pyridinique de 0,105 (1) Å et du plan benzénique de −0,174 (1) Å. N(12) s'écarte de ce dernier de −0,073 (2) Å. Les distances N(19)—$X1$ et N(19)—$X2$ qui pourraient constituer un paramètre important pour l'activité neuroleptique (Fillers & Hawkinson, 1982) valent respectivement 5,988 (2) et 7,770 (2) Å [dans (II): 6,098 (6) et 7,737 (6) Å].

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Structures of Two Dideoxynucleosides: 2′,3′-Dideoxyadenosine and 2′,3′-Dideoxycytidine

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Abstract. 2′,3′-Dideoxyadenosine, $C_{10}H_{13}N_5O_2$, $M_r = 235{\cdot}24$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}7404$ (4), $b = 9{\cdot}9843$ (9), $c = 14{\cdot}0842$ (10) Å, $V = 1088{\cdot}46$ Å^3, $Z = 4$, $D_x = 1{\cdot}435$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 0{\cdot}8326$ mm^{-1}, $F(000) = 496$, $T = 296$ K, final $R = 0{\cdot}032$ for 1088 observed reflections. 2′,3′-Dideoxycytidine, $C_9H_{13}N_3O_3$, $M_r = 211{\cdot}21$, tetragonal, $P4_12_12$, $a = 8{\cdot}6802$ (4), $c = 26{\cdot}1386$ (14) Å, $V = 1969{\cdot}44$ Å^3, $Z = 8$, $D_x = 1{\cdot}424$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 0{\cdot}8701$ mm^{-1}, $F(000) = 896$, $T = 296$ K, final $R = 0{\cdot}050$ for 1116 observed reflections. In both compounds the sugar rings have conformations intermediate between envelope and half chair but somewhat different pseudorotations. The relative orientations of the sugar and base are different in the two molecules with dideoxyadenosine being at the boundary of *syn* and *anti* and deoxycytidine being *anti*.

Introduction. Dideoxyadenosine (1) and dideoxycytidine (2) are two compounds of potential use in AIDS therapy (Mitsuya & Broder, 1986). In order to provide structural parameters and conformations of at least locally minimal energy for use in molecular-mechanics calculations, the crystal structures were investigated. At the point of completion of the structure of (2) by

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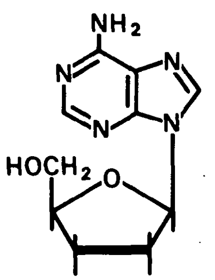
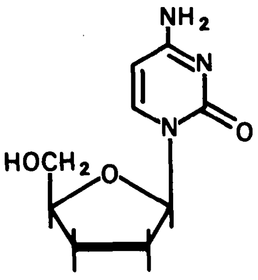
Silverton, Quinn & Haugwitz, it was brought to their attention that the structure had also been determined almost simultaneously by Todaro. The results of Todaro's investigation are not significantly different from those of Silverton, Quinn & Haugwitz and all further discussion will refer to the Silverton, Quinn & Haugwitz structures.



(1)



(2)

Table 1. *X-ray experimental data*

Both compounds	
Diffractometer and data collection method	CAD-4, graphite monochromator, Cu $K\alpha$, $\theta/2\theta$
Maximum θ (°)	74
Variation of intensity standards	3%, measured every 3 h
Phase problem solution	*MITHRIL* (Gilmore, 1983)
Refinement	Full-matrix least squares: H atoms isotropic, other atoms anisotropic
Anisotropic parameters	$\exp-\{2\pi^2[\sum_i\sum_j(U_{ij}h_ih_ja_i^*a_j^*)]\}$
Function minimized	$\sum[w(\lvert F_o\rvert - \lvert F_c\rvert)]^2$
Weights (w)	$1/\sigma(F_o)$
$\sigma(F_o)$	Statistical as Peterson & Levy (1957)
Refinement program	*XTAL* (Stewart, Hall, Alden, Olthoff-Hazekamp & Doherty, 1985)
Atomic scattering factors	From *XTAL*
Dideoxyadenosine (1)	
Crystal size (mm)	0·37 × 0·25 × 0·25
Friedel group	$2/m\ 2/m\ 2/m$
Conditions limiting reflections	hkl: all; $h00$: $h = 2n$; $k00$: $k = 2n$; $00l$: $l = 2n$
Counter aperture (mm)	3·0
Scan width (°)	1·5 + 0·35tanθ
Reflections used for lattice parameters	15 ($22 < \theta < 25°$)
Maximum $(\sin\theta)/\lambda$ (Å^{-1})	0·6230
Range of h,k,l	0:9, 0:12, 0:17
Reflections measured	1290 [1088 with $I > \sigma(I)$]
Intensity standards	401, 107, 410
R, wR	0·031, 0·030
S	1·18
$(\Delta/\sigma)_{\max}$	0·0026
Extrema of density in final difference map (e Å^{-3})	−0·22, +0·13
Dideoxycytidine (2)	
Crystal size (mm)	0·3 × 0·3 × 0·3
Friedel group	$4/m\ 2/m\ 2/m$
Conditions limiting reflections	hkl: all; $00l$: $l = 4n$; $h00$: $h = 2n$
Counter aperture (mm)	1·5
Scan width (°)	1·2 + 0·35tanθ
Reflections used for lattice parameters	18 ($22 < \theta < 30°$)
Maximum $(\sin\theta)/\lambda$ (Å^{-1})	0·6227
Range of h,k,l	0:10, 0:7, 0:32
Reflections measured	1240 [1122 with $I > \sigma(I)$]
Intensity standards	0,2,14, 407, 048
R, wR	0·050, 0·051
S	2·27
$(\Delta/\sigma)_{\max}$	0·00063
Extrema of density in final difference map (e Å^{-3})	−0·52, 0·49

Experimental. Preparation, noncrystallographic physical data and analyses for both compounds by Kulkarni (1986). Method of preparation as in literature references; dideoxyadenosine (1): Robins & Robins (1964); dideoxycytidine (2): Maramuto & Honjo (1974) and Horwitz, Chua, Noel & Donati (1967). Analyses: (1): calcd C: 51·05, H: 5·57, N: 29·77; found: 50·56, 5·45, 29·56; (2) calcd C: 51·18, H: 6·20, N: 19·89, O: 22·73; found: 51·01, 6·22, 19·79, 23·16. Physical data; colorless crystals from butanol/methanol, (1) prismatic, (2) bipyramidal, melting points: (1) 459–461 K (literature: 456–458 K); (2) 490–491 K (literature: 486–488 K); $[\alpha]_D$: (1) 75·7° (c 1·00, H_2O); (2) −28·4° (c 1·00, H_2O). Other experimental details are given in Table 1. The final atomic parameters for the non-H atoms are given in Table 2* and the bond lengths and crystal conformation are shown as *ORTEP* drawings (Johnson, 1965) in Figs. 1 and 2. The crystals of dideoxycytidine appear to exhibit extinction and six low-angle reflections were omitted from the refinement and calculation of the R factors.

Table 2. *Atomic parameters for the non-*H *atoms*

All parameters are multiplied by 10^4. The equivalent U values (Å^2) are the geometric means of the diagonal terms of the vibration tensors.

	x	y	z	U_{eq}
Dideoxyadenosine				
N1	9354 (3)	588 (2)	5688 (1)	362 (6)
C2	9091 (4)	845 (2)	4768 (2)	425 (7)
N3	9040 (3)	2014 (2)	4318 (1)	405 (6)
C4	9308 (3)	3025 (2)	4932 (1)	323 (6)
C5	9590 (3)	2920 (2)	5900 (1)	313 (6)
C6	9611 (3)	1626 (2)	6286 (2)	321 (6)
N7	9812 (3)	4185 (2)	6297 (1)	370 (6)
C8	9655 (3)	4998 (2)	5575 (2)	369 (7)
N9	9330 (3)	4370 (2)	4726 (1)	344 (6)
N10	9892 (3)	1386 (2)	7206 (1)	395 (6)
C1′	9152 (3)	4937 (3)	3773 (2)	374 (6)
C2′	8141 (4)	6245 (3)	3736 (2)	475 (8)
C3′	9018 (4)	6980 (3)	2922 (2)	506 (9)
C4′	10897 (4)	6548 (3)	3030 (2)	400 (7)
O4′	10811 (2)	5196 (2)	3391 (1)	410 (5)
C5′	11905 (3)	7413 (3)	3703 (2)	429 (8)
O6′	13665 (2)	7028 (2)	3787 (1)	417 (5)
Dideoxycytidine				
N1	1884 (3)	2518 (3)	8397 (1)	300 (6)
C2	1826 (3)	4124 (3)	8426 (1)	331 (7)
N3	1440 (3)	4936 (3)	8000 (1)	364 (7)
C4	1000 (3)	4199 (3)	7577 (1)	338 (8)
C5	942 (4)	2559 (4)	7552 (1)	371 (9)
C6	1430 (4)	1771 (3)	7965 (1)	362 (8)
N7	634 (4)	5036 (4)	7167 (1)	457 (8)
O8	2149 (3)	4762 (3)	8837 (1)	446 (7)
C1′	2458 (4)	1694 (3)	8857 (1)	362 (8)
C2′	1243 (5)	1432 (4)	9264 (1)	459 (10)
C3′	1507 (5)	−229 (4)	9434 (1)	471 (10)
C4′	2127 (4)	−974 (4)	8955 (1)	430 (9)
O4′	3015 (2)	242 (3)	8708 (1)	426 (6)
C5′	896 (5)	−1568 (4)	8601 (1)	549 (12)
O6′	1534 (6)	−1895 (3)	8111 (1)	655 (12)

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* Lists of F_o, F_c and $\sigma(F_o)$, all refinement parameters and tables of molecular dimensions have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44430 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Discussion. The molecules of both compounds use the H atoms of the amino and hydroxyl groups to form hydrogen bonds. In dideoxyadenosine (1) the molecules are linked into a three-dimensional network with bonds along all three screw axes. The bonds are O6′—H···N1, N10—H···N7 and N10—H···O6′ along the *a*, *b* and *c* screw axes, respectively. Dideoxycytidine (2) forms two intermolecular bonds; N7—H···O8 along the 4_1 axis and O6′—H···N3 along **b** but a third intermolecular bond, N7—H···O6′, is very long (3·254 Å) and is debatable. Essentially, hydrogen-bonded helices along the 4_1 axes are linked by hydrogen bonds along the *a* and *b* axes. The geometry of potential hydrogen bonds is detailed in Table 3 and packing diagrams are given as Figs. 3 and 4. Although Fig. 3 might suggest that the superposition of bases in the crystals of (1) constitutes parallel stacking, the dihedral angle between their planes is 20·4° and the distances of the atoms of one base from the plane of the next vary from 3·4 to 4·7 Å. There is no similar superposition in the crystals of (2); the bases in the hydrogen-bonded molecules related by the 4_1 screw axis are approximately perpendicular to each other.

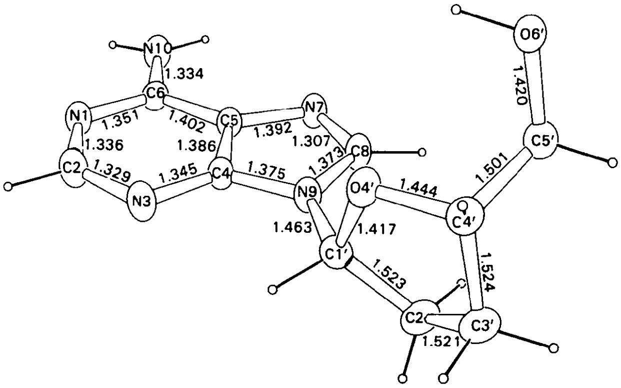

Fig. 1. *ORTEP* drawing showing the crystal conformation and bond lengths (Å) for dideoxyadenosine.

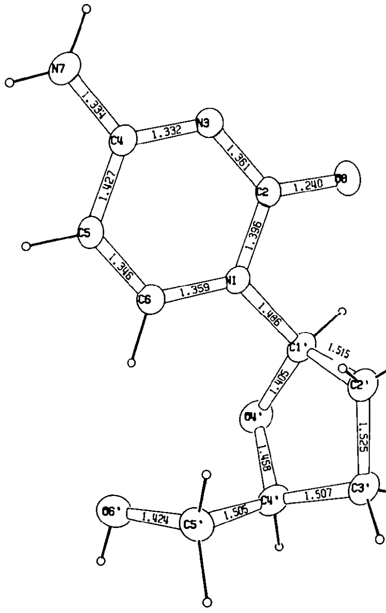

Fig. 2. *ORTEP* drawing showing the crystal conformation and bond lengths (Å) for dideoxycytidine.

The conformational notation used hereafter follows the recommendations of the IUPAC–IUB Joint Commission on Biochemical Nomenclature (1983). The degree of pucker (ψ_m), as defined by Altona & Sundaralingam (1972), is similar in both compounds being 36·5 and 34·4° for (1) and (2), respectively. The sugar torsion angles ν_0 through ν_4 (*i.e.* those for the ring bonds O4′—C1′, C1′—C2′ and clockwise around the ring) are −5·1 (2), +25·7 (2), −35·1 (3), +33·1 (2), −17·7 (2) and +5·8 (3), +15·7 (3), −30·1 (4), +33·7 (4), −25·3° for (1) and (2), respectively; the corresponding pseudorotation angles (*P*), calculated from the torsion angles, are 190·5 and 208·0°

Table 3. *Dimensions of possible hydrogen bonds*

	D—H···*A*			*D*···*A* (Å)	H···*A* (Å)	*D*—H···*A* (°)	Symmetry operation
(1)	O6′	H6′	N1	2·767 (3)	1·77 (3)	170 (3)	2_1a
	N10	H101	N7	3·054 (3)	2·29 (3)	144 (3)	2_1b
	N10	H102	O6′	2·952 (3)	2·09 (3)	152 (3)	2_1c
(2)	N7	H72	O8	2·979 (3)	2·16 (4)	169 (4)	4_1
	O6′	H6′	N3	2·767 (4)	1·99 (5)	149 (5)	*b*
	N7	H71	O6′	3·254 (6)	2·39 (4)	153 (3)	Diagonal 2

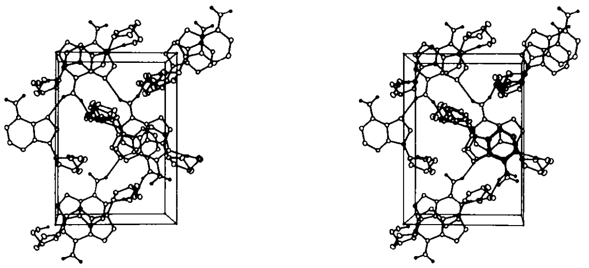

Fig. 3. Packing diagram for dideoxyadenosine. The direction of projection is **a** and hydrogen bonds are indicated by heavy lines with H atoms as filled circles.

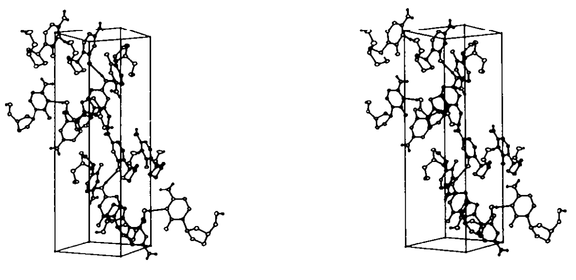

Fig. 4. Packing diagram for dideoxycytidine. The direction of projection is perpendicular to **c** and at 30° to **a**. Hydrogen bonds are indicated by heavy lines with H atoms as filled circles. The origin of the indicated cell is at (− ½, 0, 0).

indicating that both rings have approximately the ${}_3E$ (C3′-*exo*) conformation ($P = 198°$) with (1) distorted towards ${}^3_2T$ and (2) towards ${}^3_4T$. The sugar conformations of the present compounds, which lack oxygen substituents at both C2′ and C3′, are in the range which Sundaralingam (1975) has indicated as usual for 2′-monodeoxyribofuranoses. The glycosidic bonds, C1′—N9 in (1) and C1′—N1 in (2), have χ torsion angles of 264·1 (2) and 203·2 (2)°, respectively, and both molecular conformations are *anti* although that of (1) is on the borderline with the *syn* conformation ($\chi = 270°$). The C1′—O4′ bond is significantly shorter than the C4′—O4′ bond in conformity with the observations of Bugg, Thomas, Sundaralingam & Rao (1971). In general, other molecular dimensions in both molecules are comparable with similar compounds.

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Structure of 1,5-Bis(α-isopropyl-3,4-dimethoxybenzyl)-2,4-dimethoxybenzene

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Abstract. $C_{32}H_{42}O_6$, $M_r = 522·7$, monoclinic, $P2_1/c$, $a = 13·196$ (1), $b = 13·692$ (2), $c = 16·770$ (2) Å, $\beta = 104·45$ (1)°, $V = 2934·0$ (6) Å^3, $Z = 4$, $D_x = 1·18$ g cm^{-3}, $F(000) = 1128$, $T = 295$ K, $R = 0·052$ and $wR = 0·059$ for 2107 unique observed reflections (Mo $K\alpha$ radiation, $\lambda = 0·71069$ Å). This isomer is defined as *anti*, having the terminal benzyl rings on opposite sides with respect to the central benzene ring.

Introduction. The synthesis and physicochemical characterization of the title compound is a continuation of our investigations on molecules containing dimethoxy-substituted aromatic rings (Bruno, Bombieri, Rizzardi, Polizzotti & Natoli, 1983; Natoli, Agozzino, Ceraulo & Lamartina, 1983). These compounds are of particular interest because dimethoxy aromatic moieties are frequently present in synthetic and natural drugs.

By reacting 1,3-dimethoxybenzene (1) with 1-(3,4-dimethoxyphenyl)-2-methyl-1-propanol (2) in acid medium, different conformers due to the allowed rotation about C(sp^3)—C(sp^2) single bonds could be obtained as with the 1,5-bis(α-isopropyl-2,4-dimethoxybenzyl)-2,4-dimethoxybenzene derivative (Natoli, Agozzino, Ceraulo & Lamartina, 1982). In the present synthesis carried out in identical conditions only

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