polarization correction was applied, but no absorption or extinction corrections. The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1985) with refinement by full-matrix least squares on F using programs from the *SDP-Plus* (B. A. Frenz & Associates, Inc., 1984) package with non-H atoms refined anisotropically. H atoms which were located from the difference map were refined isotropically. With a weighting scheme of $w = 1/\sigma^2(F)$, $\sum w(|F_o| - |F_c|)^2$ minimized, the final residuals were $R = 0{\cdot}030$ and $wR = 0{\cdot}039$ for 2003 observed reflections; $S = 1{\cdot}5$, $(\Delta/\sigma)_{\max} = 0{\cdot}11$, $(\Delta\rho)_{\max,\min} = 0{\cdot}26$, $-0{\cdot}16$ e Å^{-3} in the final difference map. f'' multiplier $\eta = 1{\cdot}001$ (1) from least squares. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Drawings prepared using *PLUTO* (Motherwell, 1976).

Atomic parameters are given in Table 1,* intramolecular bond distances and angles in Table 2. Fig. 1 shows the molecular structure and the numbering scheme.

Related literature. The synthesis (Korpiun, Lewis, Chickos & Mislow, 1968) and characterization of this and its diastereoisomer by spectroscopic means has been reported (Lewis, Korpiun & Mislow, 1968). It has been widely used for the synthesis of chiral phosphine oxides and phosphines (Vineyard, Knowles, Sabacky, Bachman & Weinkauff, 1977). The structures of the related compounds S-methyl (PS)-phenylphosphonothioate and menthyl methyl (PR)-phenylphosphonate have been reported (Donohue & Mandel, 1981)

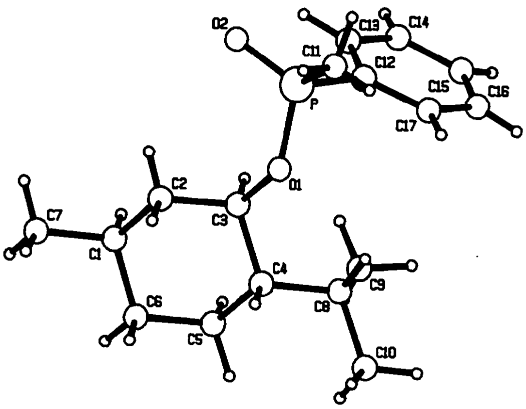

Fig. 1. Molecular structure and numbering scheme for (2).

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* Lists of structure factors, anisotropic temperature factors, H-atom parameters, torsion angles and a packing diagram have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54192 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1991). C**47**, 2242–2245

1:2 Product of 2,7-Dimethyl-2*H*-benzothieno[2,3-*c*]pyrrole with Dimethyl Acetylenedicarboxylate

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(*Received* 17 *February* 1991; *accepted* 19 *April* 1991)

Abstract. $C_{24}H_{23}NO_8S$, tetramethyl 1,9-dimethyl-3a,10c-dihydro-1*H*-benzothieno[2,3-*g*]indole-2,3,3a,4-tetracarboxylate, $M_r = 485{\cdot}51$, orthorhombic, $Iba2$, $a = 23{\cdot}358$ (8), $b = 24{\cdot}250$ (7), $c = 8{\cdot}360$ (1) Å, $V = 4735$ (2) Å^3, $Z = 8$, $D_x = 1{\cdot}362$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}18$ mm^{-1}, $F(000) = 2032$, $T = 291$ (1) K, final $R = 0{\cdot}043$ for 3486 unique observed [$F \geq 4{\cdot}0\sigma(F)$] diffractometer data. The constitution and configuration of the crystalline 1:2 product has been elucidated from

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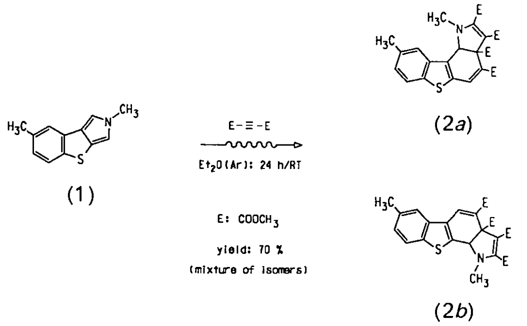
structure analysis. In agreement with this result the preferred direction of the bridge-opening reaction is established. Formation of the substituted 3a,10c-dihydro-1*H*-benzothieno[2,3-*g*]indole with the substituted N atom at the opposite site of the S atom can be explained, based on theoretical calculations [Palmer & Kennedy (1974). *J. Chem. Soc. Perkin Trans.* 2, pp. 1893–1903], by the inductive and steric effect of the S atom; the donating activity can be neglected. The crystal structure establishes the formation of the *cis*-configurated diastereomer with absolute configuration C(3a)-*R* and C(10c)-*R* at the chiral centres. The benzo[*b*]thiophene fragment is nearly planar; the dihedral angle between the thiophene and pyrrole unit has a value of 64·87 (9)°.

Experimental. 2,7-Dimethyl-2*H*-benzothieno[2,3-*c*]-pyrrole (1) and dimethyl acetylenedicarboxylate (1:1 molar ratio) were reacted in ether for 24 h at room temperature. After concentration a mixture of the isomeric annellated indoles (2*a*) and (2*b*) (ratio of isomers 67%:33%; determined by ^{1}H NMR) was isolated and crystallized from ether/*n*-pentane (1:1). The ^{1}H NMR signals are not significant in order to determine the definite configuration.



The isomers were separated by column chromatography (silica gel; *n*-hexane/ethyl acetate 2:1) and characterized by analytical and spectroscopic means. X-ray analysis was carried out for the major product (2*a*) (m.p. 491–493 K from ether/*n*-pentane), which was crystallized from ethyl acetate solution by slow concentration (four days) at 298 K. A crystal platelet 1·20 × 0·13 × 0·32 mm was used and its quality checked with optical polarizing microscopy. The crystal was mounted on a glass fibre. Intensity data collected with $\omega/2\theta$ scans, variable scan speed 1·5–10·2° min^{-1} in θ, scan width 1·2° + dispersion on a Nicolet *R*3*m*/*V* diffractometer with graphite-monochromated Mo $K\alpha$ radiation. Lattice parameters determined from symmetry-constrained least-squares fit of 24 reflections with $2\theta_{\max} = 34{\cdot}42°$. ω scans of low-order reflections along the three crystal axes showed acceptable mosaicity. Six standard reflections (400, 080, 0,0,10, $\bar{4}00$, $0\bar{8}0$, $0,0,\overline{10}$) recorded every 300 reflections, only random deviations detected during 156·72 h of X-ray exposure; 7898 reflections with $2{\cdot}0 \leq 2\theta \leq 50{\cdot}0°$ (16 680 accessible), $-10 \leq h \leq 6$, $0 \leq k \leq 28$, $-29 \leq l \leq 29$. Data corrected for Lorentz–polarization but not for absorption effects and averaged ($R_{\rm int} = 0{\cdot}048$) to 3781 unique reflections, 3486 of which had $F \geq 4{\cdot}0\sigma(F)$. 10 reflections were omitted because of suspected extinction. Systematic absences (hkl) $h + k + l = 2n + 1$, ($0kl$) $k = 2n + 1$ and ($h0l$) $h = 2n + 1$ conform to space group *Iba*2. Structure solved *via* direct methods and $\Delta\rho$ maps. It was refined (on *F*) using full-matrix least squares with anisotropic displacement parameters for all non-H atoms and a common isotropic one for the H atoms, which were placed in geometrically calculated positions (C—H 0·96 Å). 308 parameters refined. Weights $w = 1{\cdot}0/[\sigma^2(F) + (0{\cdot}0125F^2)]$ led to a featureless analysis of variance in terms of $\sin\theta$ and F_o. Refinement converged to $R = 0{\cdot}043$, $wR = 0{\cdot}048$, $S = 0{\cdot}54$, $(\Delta/\sigma)_{\max} = 0{\cdot}007$ (no extinction correction). Correctness of the space-group choice was checked by using *MISSYM* (Le Page, 1987). An η-refinement [$\eta = 1{\cdot}5\,(2)$; Rogers (1981)] confirmed the proposed chirality. Largest peaks in final $\Delta\rho$ map were $\pm 0{\cdot}4\,(1)$ e Å^{-3}. Atomic scattering factors for neutral atoms and real and imaginary dispersion terms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Programs used: *PARST* (Nardelli, 1983), *SHELXTL-Plus* (Sheldrick, 1987), *PCK*83 (Williams, 1984), *PLATON* (Spek, 1982), *MISSYM* (Le Page, 1987). The molecule and the numbering scheme are shown in Fig. 1 and a stereoscopic view of the unit-cell content in Fig. 2. Positional parameters and the equivalent values of the anisotropic displacement parameters for the non-H atoms are given in Table 1.* Bond lengths, bond angles, torsion angles, least-squares planes, dihedral angles and possible hydrogen bonds are given in Table 2.

Related literature. Formation of 1:2 products from dimethyl acetylenedicarboxylate by reaction with *N*-methylpyrrole was first observed by Diels, Alder & Winckler (1931); the constitution as substituted indole was further established by Acheson & Vernon (1962) and a reaction mechanism was suggested. This observation was extended to isoannellated pyrroles (alkyl = H, Me, Et, *n*-Bu, benzyl) by Schmitt (1967), Bender, Bonnett & Smith (1970) and Kricka & Vernon (1971, 1972). In contrast to the general

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* Lists of structure factors, anisotropic displacement parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54184 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates and equivalent isotropic displacement parameters* (Å^2 × 10^4)

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

	x	y	z	$U_{\rm eq}$
S(1)	0·75759 (3)	0·99614 (2)	0·80300	490
O(1)	0·6595 (1)	0·71289 (9)	0·7773 (3)	693
O(2)	0·67428 (9)	0·68161 (7)	1·0236 (3)	492
O(3)	0·5707 (1)	0·7381 (1)	1·1092 (4)	732
O(4)	0·58892 (9)	0·79498 (8)	1·3144 (4)	603
O(5)	0·63989 (8)	0·92773 (7)	1·3073 (3)	515
O(6)	0·69543 (8)	0·85614 (8)	1·3726 (3)	447
O(7)	0·54408 (9)	0·8651 (1)	1·0749 (5)	913
O(8)	0·5440 (1)	0·9351 (1)	0·9082 (5)	1014
N(1)	0·72457 (9)	0·80863 (8)	0·9257 (3)	320
C(2)	0·6799 (1)	0·77662 (9)	0·9837 (4)	324
C(3)	0·64662 (9)	0·80249 (9)	1·0884 (3)	329
C(3a)	0·66711 (8)	0·86263 (8)	1·1018 (3)	294
C(4)	0·6332 (1)	0·9011 (1)	0·9918 (4)	404
C(5)	0·6592 (1)	0·9388 (1)	0·8973 (4)	427
C(5a)	0·7208 (1)	0·9433 (1)	0·8964 (4)	370
C(6a)	0·8222 (1)	0·9676 (1)	0·8615 (4)	441
C(7)	0·8769 (1)	0·9877 (1)	0·8284 (5)	596
C(8)	0·9233 (1)	0·9596 (1)	0·8855 (5)	669
C(9)	0·9180 (1)	0·9118 (1)	0·9807 (5)	515
C(10)	0·8639 (1)	0·8910 (1)	1·0099 (4)	378
C(10a)	0·8149 (1)	0·9187 (1)	0·9502 (3)	326
C(10b)	0·75583 (9)	0·90551 (9)	0·9660 (3)	293
C(10c)	0·73005 (8)	0·85513 (8)	1·0404 (3)	252
C(11)	0·7783 (1)	0·7813 (1)	0·8828 (4)	462
C(12)	0·6694 (1)	0·7205 (1)	0·9147 (4)	392
C(13)	0·6559 (2)	0·6273 (1)	0·9753 (6)	717
C(14)	0·5973 (1)	0·7758 (1)	1·1689 (5)	459
C(15)	0·5423 (2)	0·7705 (2)	1·4013 (8)	1033
C(16)	0·66491 (9)	0·88633 (9)	1·2703 (4)	334
C(17)	0·6885 (2)	0·8704 (2)	1·5377 (5)	725
C(18)	0·5694 (1)	0·8976 (1)	0·9992 (6)	650
C(19)	0·4818 (1)	0·9326 (2)	0·910 (1)	1296
C(20)	0·9700 (1)	0·8832 (2)	1·0470 (7)	819

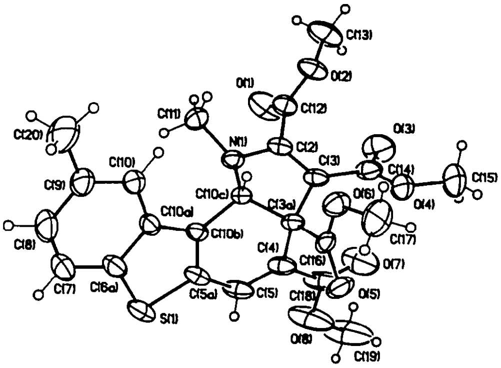

Fig. 1. General view (*SHELXTL-Plus*; Sheldrick, 1987) of the molecule, showing the atom-numbering scheme. Anisotropic ellipsoids represent 50% probability boundaries.

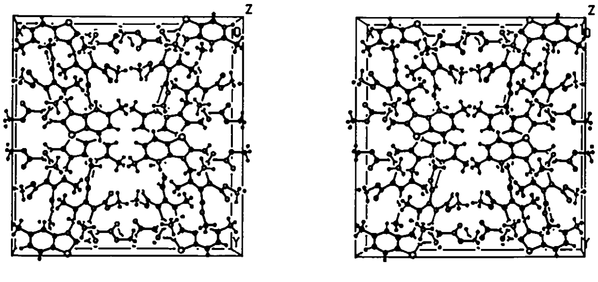

Fig. 2. Stereoscopic view (*SHELXTL-Plus*; Sheldrick, 1987) of the unit cell.

Table 2. *Bond distances* (Å), *bond angles* (°), *torsion angles* (°), *least-squares planes and dihedral angles* (°)

S(1)—C(5a)	1·730 (3)	C(3)—C(3a)	1·539 (3)
S(1)—C(6a)	1·730 (3)	C(3)—C(14)	1·482 (4)
O(1)—C(12)	1·186 (4)	C(3a)—C(4)	1·531 (4)
O(2)—C(12)	1·316 (4)	C(3a)—C(10c)	1·568 (3)
O(2)—C(13)	1·443 (4)	C(3a)—C(16)	1·522 (4)
O(3)—C(14)	1·212 (4)	C(4)—C(5)	1·352 (4)
O(4)—C(14)	1·317 (5)	C(4)—C(18)	1·493 (4)
O(4)—C(15)	1·438 (6)	C(5)—C(5a)	1·443 (4)
O(5)—C(16)	1·202 (3)	C(5a)—C(10b)	1·360 (4)
O(6)—C(16)	1·332 (3)	C(6a)—C(7)	1·396 (4)
O(6)—C(17)	1·432 (5)	C(6a)—C(10a)	1·408 (4)
O(7)—C(18)	1·171 (5)	C(7)—C(8)	1·367 (5)
O(8)—C(18)	1·326 (5)	C(8)—C(9)	1·412 (5)
O(8)—C(19)	1·456 (4)	C(9)—C(10)	1·382 (4)
N(1)—C(2)	1·388 (3)	C(9)—C(20)	1·505 (5)
N(1)—C(10c)	1·486 (3)	C(10)—C(10a)	1·417 (4)
N(1)—C(11)	1·463 (4)	C(10a)—C(10b)	1·424 (3)
C(2)—C(3)	1·328 (4)	C(10b)—C(10c)	1·497 (3)
C(2)—C(12)	1·498 (4)		

C(5a)—S(1)—C(6a)	90·5 (1)	C(7)—C(6a)—C(10a)	120·5 (3)
C(12)—O(2)—C(13)	115·7 (3)	C(6a)—C(7)—C(8)	118·9 (3)
C(14)—O(4)—C(15)	115·7 (3)	C(7)—C(8)—C(9)	122·4 (3)
C(16)—O(6)—C(17)	115·2 (3)	C(8)—C(9)—C(20)	120·9 (3)
C(18)—O(8)—C(19)	114·4 (3)	C(8)—C(9)—C(10)	118·6 (3)
C(10c)—N(1)—C(11)	115·3 (2)	C(10)—C(9)—C(20)	120·4 (3)
C(2)—N(1)—C(11)	118·5 (2)	C(9)—C(10)—C(10a)	120·2 (3)
C(2)—N(1)—C(10c)	105·3 (2)	C(6a)—C(10a)—C(10)	119·2 (2)
N(1)—C(2)—C(12)	119·8 (2)	C(10)—C(10a)—C(10b)	130·0 (2)
N(1)—C(2)—C(3)	113·9 (2)	C(6a)—C(10a)—C(10b)	110·8 (2)
C(3)—C(2)—C(12)	125·9 (2)	C(5a)—C(10b)—C(10a)	113·1 (2)
C(2)—C(3)—C(14)	123·2 (2)	C(10a)—C(10b)—C(10c)	127·8 (2)
C(2)—C(3)—C(3a)	108·3 (2)	C(5a)—C(10b)—C(10c)	119·0 (2)
C(3a)—C(3)—C(14)	128·5 (2)	C(3a)—C(10c)—C(10b)	114·7 (2)
C(3)—C(3a)—C(16)	114·5 (2)	N(1)—C(10c)—C(10b)	112·7 (2)
C(3)—C(3a)—C(10c)	99·1 (2)	N(1)—C(10c)—C(3a)	102·6 (2)
C(3)—C(3a)—C(4)	111·9 (2)	O(2)—C(12)—C(2)	111·7 (3)
C(10c)—C(3a)—C(16)	112·2 (2)	O(1)—C(12)—C(2)	123·1 (3)
C(4)—C(3a)—C(16)	108·0 (2)	O(1)—C(12)—O(2)	125·2 (3)
C(4)—C(3a)—C(10c)	111·1 (2)	O(4)—C(14)—C(3)	112·4 (3)
C(3a)—C(4)—C(18)	117·1 (2)	O(3)—C(14)—C(3)	122·7 (3)
C(3a)—C(4)—C(5)	122·0 (2)	O(3)—C(14)—O(4)	124·7 (3)
C(5)—C(4)—C(18)	120·7 (3)	O(6)—C(16)—C(3a)	111·6 (2)
C(4)—C(5)—C(5a)	120·1 (3)	O(5)—C(16)—C(3a)	124·7 (2)
S(1)—C(5a)—C(5)	123·6 (2)	O(5)—C(16)—O(6)	123·6 (3)
C(5)—C(5a)—C(10b)	123·2 (3)	O(8)—C(18)—C(4)	112·6 (3)
S(1)—C(5a)—C(10b)	113·2 (2)	O(7)—C(18)—C(4)	124·4 (3)
S(1)—C(6a)—C(10a)	112·4 (2)	O(7)—C(18)—O(8)	123·0 (3)
S(1)—C(6a)—C(7)	127·1 (2)		

C(2)—N(1)—C(10c)—C(3a)	−30·4 (2)	C(10c)—C(3a)—C(4)—C(5)	−23·6 (3)
C(10c)—N(1)—C(2)—C(3)	17·5 (3)	C(3a)—C(4)—C(5)—C(5a)	0·8 (4)
N(1)—C(2)—C(3)—C(3a)	4·0 (3)	C(4)—C(5)—C(5a)—C(10b)	11·3 (4)
C(2)—C(3)—C(3a)—C(10c)	−22·1 (3)	C(5)—C(5a)—C(10b)—C(10c)	3·2 (4)
C(3)—C(3a)—C(10c)—N(1)	30·8 (2)	S(1)—C(5a)—C(10b)—C(10c)	−175·3 (2)
C(3)—C(3a)—C(10c)—C(10b)	153·4 (2)	C(5)—C(5a)—C(10b)—C(10a)	−179·9 (3)
C(4)—C(3a)—C(10c)—C(10b)	35·6 (3)	C(5a)—C(10b)—C(10c)—C(3a)	−27·3 (3)
C(10b)—C(10c)—N(1)—C(2)	−154·3 (2)		

No.	Plane through atoms	Equation of the plane	χ^2
1	C(6a),C(7),C(8),C(9),C(10),C(10a)	$0·040x - 0·546y - 0·8365z = -18·07$Å	72·1
2	S(1),C(5a),C(6a),C(10a),C(10b)	$0·031x - 0·5300y - 0·8474z = -17·94$Å	25·9
3	N(1),C(2),C(3),C(3a),C(10c)	$-0·480x + 0·410y - 0·7755z = -6·22$Å	17622·1

Dihedral angles: 1,2 1·23(8); 1,3 66·08(10); 2,3 64·87(9)°.

Possible hydrogen bonds

Donor—H···Acceptor	$D\cdots A$	H···A	D—H···A
C(5)—H(5a)···O(8^0)	2·693 (4)	2·351 (4)	100·3 (2)
C(10c)—H(10b)···O(6^0)	2·893 (4)	2·476 (3)	106·1 (2)
C(19)—H(19a)···O(5^1)	2·971 (4)	2·644 (4)	100·3 (3)
C(19)—H(19c)···O(5^1)	2·971 (4)	2·717 (4)	95·7 (3)

Symmetry codes: (0) x, y, z; (1) $-x + 1, y, z - \frac{1}{2}$.

behaviour of these *o*-quinoid hetarenes the reaction of 1-alkoxy-2-alkyl-2*H*-isoindoles with *N*-alkylmaleimides leads to the formation of 1:2 adducts *via* Michael-type addition and subsequent [4 + 2] cycloaddition (Kreher, Hennige, Jelitto & Preut, 1989). In order to prove the predicted reaction pathway and to determine the preferred direction of the bridge-opening reaction, a crystal structure determination was performed. For further details see Lee, Hahn & Noland (1978), Sewarte-Roß (1989) and Sha & Tsou (1990). For preparation and spectroscopic data of tricyclic 14π-hetarenes see Dyker (1988) and Götte (1991).

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Acta Cryst. (1991). C**47**, 2245–2247

Structure of 1-(2-Deoxy-2-fluoro-α-D-arabinopyranosyl)-5-iodouracil*

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(*Received* 3 *March* 1991; *accepted* 29 *April* 1991)

Abstract. 1-(2-Deoxy-2-fluoro-α-D-arabinopyranosyl)-5-iodouracil, $C_9H_{10}FIN_2O_5$, $M_r = 372{\cdot}09$, triclinic, $P1$, $a = 5{\cdot}098\ (2)$, $b = 6{\cdot}470\ (2)$, $c = 9{\cdot}293\ (6)$ Å, $\alpha = 81{\cdot}10\ (2)$, $\beta = 84{\cdot}91\ (3)$, $\gamma = 79{\cdot}38\ (2)°$, $V = 297{\cdot}1\ (2)$ Å^3, $Z = 1$, $D_m = 2{\cdot}08$, $D_x = 2{\cdot}080$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 2{\cdot}692$ mm^{-1}, $F(000) = 180$, $T = 293$ K, final $R = 0{\cdot}024$ for 1702 unique observed [$F \geq 4\sigma(F)$] reflections. The pyranose ring adopts a chair conformation with $\varphi_2 = -19\ (4)°$, $\theta_2 = 8{\cdot}9\ (5)°$ and $Q_t = 0{\cdot}596\ (5)$ Å. The N-glycosidic torsion angle χ [O(5′)—C(1′)—N(1)—C(2)] between this ring and the pyrimidine base is oriented $+ac$ [95·1 (4)°]. The conformational parameters are in accordance with the IUPAC–IUB Joint Commission on Biochemical Nomenclature [*Pure Appl. Chem.* (1983), **55**, 1273–1280] guidelines.

Experimental. Colourless crystals were crystallized from a methanol solution, 0·38 × 0·15 × 0·23 mm. Density measured by flotation in 1,1,2,2-tetrabromoethane/CCl_4. Weissenberg photographs showed no systematically absent reflections. Stoe STADI-4 computer-controlled diffractometer, cell constants by least-squares refinement of the θ angles of 28 reflections with $20 \leq 2\theta \leq 30°$, $\omega/2\theta$ scan, $[(\sin\theta)/\lambda]_{\max} = 0{\cdot}7034$ Å^{-1}, $-7 \leq h \leq 7$, $-9 \leq k \leq 9$, $-12 \leq l \leq 12$. Intensities of two standard reflections ($1\bar{1}\bar{1}$, $1\bar{1}1$) monitored every two hours showed no

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* Structural Studies on Modified Nucleosides. XII. Part XI: Van Aerschot, Everaert, Gosselin, Peeters, Blaton, De Ranter, Imbach, Balzarini, De Clercq & Herdewijn (1990).

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0108-2701/91/102245-03$03.00