Table 3. *Torsion angles (°) of proline-containing diketopiperazines*

IUPAC designation	Atoms involved	*cyclo*-(-L-Prot-L-Prot-)*	*cyclo*-(-L-Pro-L-Pro-)	*cyclo*-(-L-Pro-L-Leu-)†	*cyclo*-(-L-Pro-Gly-)†
φ	$C_i'C_i^{\alpha}N_iC_{i+1}'$	−34·8 −32·4	−38 −37	−41·5	−44·0
ψ	$N_{i+1}C_i'C_i^{\alpha}N_i$	30·6 28·4	37 36	33·7	38·5
ω	$C_{i+1}^{\alpha}N_{i+1}C_i'C_i^{\alpha}$	1·3 4·1	0·7 −0·7	6·3	0·4
χ_1	$N_iC_i^{\alpha}C_i^{\beta}C_i^{\gamma}$	−35·8 −35·0	−34 −31	−31·5	−32·7
χ_2	$C_i^{\alpha}C_i^{\beta}C_i^{\gamma}C_i^{\delta}$	35·6 37·3	36 35	36·0	35·6
χ_3	$C_i^{\beta}C_i^{\gamma}C_i^{\delta}N_i$	−21·1 −24·5	−23 −24	−25·1	−24·0
χ_4	$C_i^{\gamma}C_i^{\delta}N_iC_i^{\alpha}$	−2·0 2·3	1 5	4·5	3·2
d_1		0·57 0·56	0·55 0·52	0·52	0·55
d_2		150·5	142	143	—

d_1 = the normal distance (Å) of the β-carbon atoms from the best plane formed by the remaining four atoms of the pyrrolidine ring.
d_2 = dihedral angle (°) between the two nearly planar amide groups.

* E.s.d.'s 0·3° for torsion angles.
† Torsion angles of the proline residues.

Close similarity of the critical C—C′ and C—N bond lengths in *cyclo*(-Prot-Prot-) and in the proline-diketopiperazines does not explain the enhanced tendency towards racemization in the course of thionation.

The similarity of the geometry between *cyclo*-(-Prot-Prot-) and related *Z* endothiopeptides (La Cour *et al.*, 1983; Jensen *et al.*, 1985) indicates no major difference in the conformation of the respective amide groups. This suggests that the thioamide unit can serve as a special 'label' in both spectroscopic and biological studies.

The authors thank Professor A. Kálmán for his advice and Mr Cs. Kertész for his technical assistance. This work was supported by the Institute for Science Management and Informatics, Ministry of Education, Budapest, Hungary.

References

BENEDETTI, E., GOODMAN, M., MARSH, R. E., RAPOPORT, H. & MUSICH, J. A. (1975). *Cryst. Struct. Commun.* **4**, 641–645.

CAVA, M. P. & LEVINSON, M. I. (1985). *Tetrahedron*, **41**, 5061–5087.

CLAUSEN, K., THORSEN, M. & LAWESSON, S.-O. (1981). *Tetrahedron*, **37**, 3635–3639.

FRENZ, B. A. (1983). *The Enraf–Nonius CAD-4 Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

JENSEN, O. E., LAWESSON, S.-O., BARDI, R., PIAZZESI, A. M. & TONIOLO, C. (1985). *Tetrahedron*, **41**, 5595–5606.

KAJTÁR, M., HOLLÓSI, M., KAJTÁR, J. & MAJER, ZS. (1986). *Tetrahedron*, **42**, 3931–3942.

KARLE, I. L. (1972). *J. Am. Chem. Soc.* **94**, 81–84.

KARLE, I. L., OTTENHEYM, H. C. J. & WITKOP, B. (1974). *J. Am. Chem. Soc.* **96**, 539–543.

LA COUR, T. F. M., HANSEN, H. A. S., CLAUSEN, K. & LAWESSON, S.-O. (1983). *Int. J. Pept. Protein Res.* **22**, 509–512.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1982). *MULTAN82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

MAJER, ZS., HOLLÓSI, M., KAJTÁR, M., KAJTÁR, J. & RADICS, L. (1987). In preparation.

MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

VON DREELE, R. B. (1975). *Acta Cryst.* B**31**, 966–970.

WALKER, N. & STUART, D. (1983). *Acta Cryst.* A**39**, 158–166.

Acta Cryst. (1987). C**43**, 2358–2361

The Structure of 3′-Deoxyformycin Hydrochloride

BY R. MCKENNA AND S. NEIDLE*

Cancer Research Campaign Biomolecular Structure Unit, The Institute of Cancer Research, Sutton, Surrey SM2 5PX, England

AND P. SERAFINOWSKI

CRC Laboratory, Drug Development Section, The Institute of Cancer Research, Sutton, Surrey SM2 5PX, England

(*Received* 28 *May* 1987; *accepted* 14 *July* 1987)

Abstract. $C_{10}H_{14}N_5O_3^+.Cl^-$, $M_r = 287{\cdot}71$, orthorhombic, $P2_12_12_1$, $a = 5{\cdot}047\ (1)$, $b = 13{\cdot}850\ (2)$, $c = 18{\cdot}321\ (3)$ Å, $V = 1280{\cdot}7$ Å^3, $Z = 4$, $D_x = 1{\cdot}492$ Mg m^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}54184$ Å, $\mu = 2{\cdot}8071$ mm^{-1}, $F(000) = 600$, $T = 298$ K, final $R = 0{\cdot}039$ for 1333 observed reflections. Formycin hydrochloride

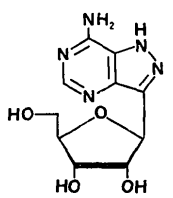
* To whom correspondence should be addressed.

0108-2701/87/122358-04$01.50

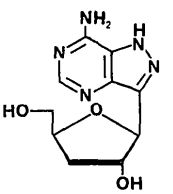
is protonated at the N(3) position on the purine base, with an intramolecular N(3)···O(5′) hydrogen bond of 2·762 (3) Å and a *syn* glycosidic angle of 14·9 (4)°. The deoxyribose sugar is in the C(3′)-*endo* class with a pseudorotation phase angle of 24·8 (9)°.

Introduction. The *C*-nucleoside antitumour antibiotic formycin (I) {(*S*)-1-*C*-(7-amino-1*H*-pyrazolo[4,3-*d*]-pyrimidin-3-yl)-1,4-anhydro-D-ribitol} was first isolated from cultures of *Norcardia interforma* (Hori, Ito, Takita, Koyama, Takeuchi & Umezawa, 1964). It is an inhibitor of purine metabolism by several pathways and is also probably an effective substrate for the enzyme adenosine deaminase, whereas conformycin is an effective inhibitor of the deamination reaction catalysed by this enzyme (Rogler-Brown, Agarwal & Parks, 1978). Formycin has growth-inhibitory properties in several experimental tumour cell lines, including S180 and L1210 (Suhadolnik, 1979). Its clinical use is hampered by its ease of deamination. Crystallographic studies have been reported on formycin itself (Prusiner, Brennan & Sundaralingam, 1973), as the hydrobromide salt (Koyama, Umezawa & Iitaka, 1974) and the 8-methyl derivative (Abola, Sims, Abraham, Lewis & Townsend, 1974).

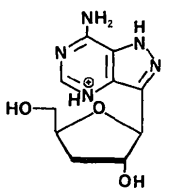
The title compound, 3′-deoxyformycin (3′-dF) (II) has been synthesized (Serafinowski, 1987) as part of a programme to examine the potential stereoelectronic requirements for inhibition of the deamination reaction. Biochemical data on 3′-dF and other modified nucleosides will be published elsewhere.



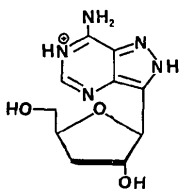
(I)



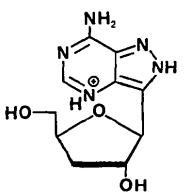
(II)



(III)



(IV)



(V)

Experimental. Recrystallization from ethanol/water solution produced colourless needle-like crystals. A specimen of dimensions 0·1 × 0·1 × 0·6mm was used. The space group is $P2_12_12_1$ (No. 19, orthorhombic) from systematic absences seen on preliminary Weissenberg photographs. Cell dimensions were obtained from least-squares refinement of 25 θ values ($10 < \theta < 25°$) measured on an Enraf–Nonius CAD-4 diffractometer; Ni-filtered Cu $K\alpha$ radiation was used. Intensity data were collected with an ω–2θ scan technique and a maximum scan time of 120s per reflection for $1·5 \leq \theta \leq 70°$ and $0 \leq h \leq 6$, $0 \leq k \leq 16$, $0 \leq l \leq 22$, 1540 unique reflections were measured of which 1333 had $I \geq 1·5\sigma(I)$.

Three intensity-standard reflections were monitored every 200 reflections during the data collection and showed no statistically significant crystal decay. An empirical absorption correction was applied (Walker & Stuart, 1983). The structure was solved by direct methods with *SHELX*84 (Sheldrick, 1984).

H atoms were located in difference Fourier syntheses, and their positional and isotropic thermal parameters refined [apart from three H atoms associated with the sugar: one of the C(5′) hydrogens and both the O(2′) and O(5′) hydrogens, all of whose thermal parameters were fixed]. Full-matrix least-squares refinement on F included anisotropic thermal parameters for non-H atoms.

The final R was 0·039 and wR was 0·054; a non-Poisson-distribution weighting scheme of the form $w = [\sigma^2(F) + 0·04|F|^2]^{-1}$ was found appropriate. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Calculations were performed on a VAX 11/750 computer using the *SDP* system (Frenz, 1980). The maximum Δ/σ for the final least-squares cycle was 0·1, with $\Delta\rho$ fluctuations in the final difference Fourier map within +0·16, −0·10 e Å^{-3}.

Discussion. The molecular structure of the hydrochloride salt of 3′-dF is shown in Figs. 1 and 2, and atomic coordinates, bond distances and angles are given in Tables 1 and 2.*

This study shows that the 3′-dF molecule is protonated at the N(3) nitrogen atom, and that the N(7) atom of the pyrazole ring also has an attached H atom. Thus, the structure corresponds to the tautomeric form (III), in striking contrast to the situation found for formycin hydrobromide (Koyama, Umezawa & Iitaka, 1974), where protonation at N(1) was reported [tautomer (IV)]. The alternative tautomer (V) is also ruled out by this structure. The relatively low-accuracy formycin

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44241 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

hydrobromide structure determination ($R = 0.083$ with photographic data) did not directly locate H atoms; instead, the position of attachment for the proton was inferred from an examination of the hydrogen bonding in the crystal lattice. Our re-examination of the packing in the hydrobromide structure indicates that protonation at N(3) would result in a more favourable hydrogen-bonding arrangement, and thus should not be excluded from consideration. Adenosine itself has been found to be protonated at N(1) in the crystal structure of its hydrochloride (Shikata, Ueki & Mitsui, 1973), in adenylyl-3′,5′-adenylyl-3′,5′-adenosine (Suck, Manor & Saenger, 1976), and in the adenylyl-3′,5′-adenosine–proflavine complex (Shieh, Berman, Neidle, Taylor & Sanderson, 1982). Solution studies have indicated that all three species co-exist in equilibrium, albeit in unknown relative proportions (Birnbaum & Shugar, 1987). The geometry of the purine ring system in several structures is compared in Table 3 with that in 3′-dF. There are several changes in bond geometry compared with the formycin structures, and with both neutral and protonated adenosine: bond length N(1)–C(2) is significantly shorter in 3′-dF, and C(2)–N(3) is longer, especially in comparison with the neutral formycins. Other bond lengths do not differ significantly from those in adenosines or formycin. In particular, the exocyclic amine distance C(6)–N(6) is normal; any significant change in length would be expected to affect susceptibility to deamination by adenosine deaminase. The C(2)–N(3)–C(4) bond angle is increased in 3′-dF by over 4° from its value in neutral formycins, indicating protonation at N(3). A similar 'opening-up' effect has been observed at the N(1) protonation site in adenosine HCl (Shikata, Ueki & Mitsui, 1973). The N(3) protonation observed in the present study is unusual, and thus the geometric changes discussed above are mostly outside the range of values given in the statistical survey of Taylor & Kennard (1982).

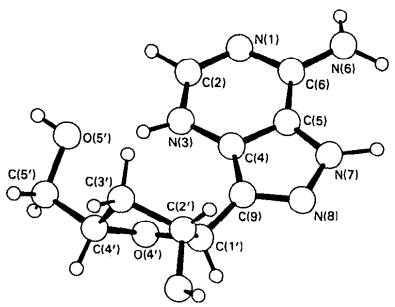


Fig. 1. View of the title compound showing atom numbering.

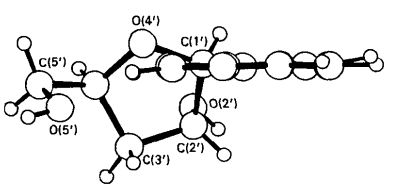


Fig. 2. Side view of the title compound.

Table 1. *Positional parameters and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

B_{eq} is in Å^2 and is defined as $\frac{1}{3}(B_{11} + B_{22} + B_{33})$.

	x	y	z	B_{eq}
C(1)	0·3149 (2)	1·07138 (6)	−0·50882 (5)	4·04 (2)
O(4′)	1·6616 (5)	1·2232 (2)	−0·1860 (1)	3·08 (5)
O(2′)	1·7805 (6)	1·4282 (2)	−0·1260 (2)	4·45 (6)
N(8)	1·3363 (7)	1·3793 (2)	−0·3137 (2)	3·45 (6)
O(5′)	1·3341 (6)	1·0991 (2)	−0·1090 (2)	4·31 (6)
N(1)	0·8426 (7)	1·1060 (2)	−0·3353 (2)	3·21 (6)
N(3)	1·1802 (6)	1·1410 (2)	−0·2501 (1)	2·68 (5)
N(7)	1·1400 (7)	1·3473 (2)	−0·3583 (2)	3·35 (6)
N(6)	0·7157 (7)	1·2138 (2)	−0·4239 (2)	3·64 (6)
C(2)	0·9904 (8)	1·0852 (2)	−0·2791 (2)	2·96 (6)
C(5)	1·0652 (8)	1·2562 (2)	−0·3398 (2)	2·69 (6)
C(4)	1·2173 (7)	1·2399 (2)	−0·2809 (2)	2·45 (6)
C(4′)	1·6668 (8)	1·2211 (3)	−0·1073 (2)	3·11 (7)
C(2′)	1·5420 (8)	1·3791 (3)	−0·1420 (2)	3·05 (7)
C(6)	0·8709 (7)	1·1923 (2)	−0·3681 (2)	2·81 (6)
C(9)	1·3830 (7)	1·3088 (2)	−0·2658 (2)	2·74 (6)
C(1′)	1·6003 (7)	1·3190 (2)	−0·2105 (2)	2·57 (6)
C(5′)	1·594 (1)	1·1201 (3)	−0·0835 (2)	4·31 (8)
C(3′)	1·4810 (9)	1·3024 (3)	−0·0850 (2)	3·58 (7)

Table 2. *Bond distances* (Å) *and angles* (°) *and details of short van der Waals contacts and hydrogen bonds, with e.s.d.'s in parentheses*

O(4′)–C(4′)	1·444 (4)	N(7)–C(5)	1·360 (4)
O(4′)–C(1′)	1·433 (4)	N(6)–C(6)	1·322 (5)
O(2′)–C(2′)	1·414 (5)	C(5)–C(4)	1·373 (5)
N(8)–N(7)	1·358 (5)	C(5)–C(6)	1·419 (5)
N(8)–C(9)	1·334 (4)	C(4)–C(9)	1·402 (5)
O(5′)–C(5′)	1·425 (6)	C(4′)–C(5′)	1·510 (6)
N(1)–C(2)	1·303 (5)	C(4′)–C(3′)	1·521 (6)
N(1)–C(6)	1·346 (4)	C(2′)–C(1′)	1·534 (5)
N(3)–C(2)	1·340 (5)	C(2′)–C(3′)	1·521 (5)
N(3)–C(4)	1·369 (4)	C(9*)–C(1′)	1·500 (5)

C(4′)–O(4′)–C(1′)	109·6 (2)	O(2′)–C(2′)–C(1′)	105·5 (3)
N(7)–N(8*)–C(9*)	106·6 (3)	O(2′)–C(2′)–C(3′)	111·5 (3)
C(2)–N(1)–C(6)	119·2 (3)	C(1′)–C(2′)–C(3′)	102·8 (3)
C(2)–N(3)–C(4)	116·9 (3)	N(1)–C(6)–N(6)	118·9 (3)
N(8)–N(7)–C(5)	110·8 (3)	N(1)–C(6)–C(5)	117·6 (3)
N(1)–C(2)–N(3)	126·5 (3)	N(6)–C(6)–C(6)	123·5 (3)
N(7)–C(5)–C(4)	106·6 (3)	N(8)–C(9)–C(4)	109·5 (3)
N(7)–C(5)–C(6)	132·8 (3)	N(8)–C(9)–C(1′)	120·3 (3)
C(4)–C(5)–C(6)	120·6 (3)	C(4)–C(9)–C(1′)	130·0 (3)
N(3)–C(4)–C(5)	119·0 (3)	O(4′)–C(1′)–C(2′)	106·8 (3)
N(3)–C(4)–C(9)	134·5 (3)	O(4′)–C(1′)–C(9*)	106·4 (3)
C(5)–C(4)–C(9)	106·5 (3)	C(2′)–C(1′)–C(9*)	117·6 (3)
O(4′)–C(4′)–C(5′)	107·6 (3)	O(5′)–C(5′)–C(4′)	108·6 (3)
O(4′)–C(4′)–C(3′)	104·0 (3)	C(4′)–C(3′)–C(2′)	102·0 (3)
C(5′)–C(4′)–C(3′)	117·3 (3)		

Symmetry translation					Angle (°)	Donor–acceptor distance (Å)	Acceptor–hydrogen distance (Å)
	x	y	z				
(i)	0	0	0	Cl···H(N6*B*)–N(6)	161 (2)	3·226 (3)	2·37 (3)
(iii)	−1	2	−1	Cl···H(N6*A*)–N(6)	157 (3)	3·259 (3)	2·36 (3)
(iii)	−1	2	−1	Cl···H(N7*A*)–N(7)	158·7 (1)	3·145 (3)	2·139 (1)
(ii)	1	2	−1	Cl···H(O5′*A*)–O(5′)	173·6 (2)	3·083 (3)	2·283 (1)
(iv)	2	−1	−1	Cl···H(O2′*A*)–O(2′)	123·9 (2)	3·204 (3)	2·615 (1)
(i)	0	0	0	O(5′)···H(N3*A*)–N(3)	165 (3)	2·762 (3)	1·87 (3)

(i) x, y, z; (ii) $0{\cdot}5-x, -y, 0{\cdot}5+z$; (iii) $0{\cdot}5+z, 0{\cdot}5-y, -z$; (iv) $-x, 0{\cdot}5+y, 0{\cdot}5-z$.

Table 3. *Comparison of aspects of the purine ring geometry in 3′-dF with that in related nucleosides*

Bond lengths (Å)	3′-dF.HCl	Ad	Ad.HCl	8-MeF	F
N(1)–C(2)	1·303 (5)	1·340 (3)	1·361 (5)	1·359 (3)	1·355 (6)
C(2)–N(3)	1·340 (5)	1·330 (3)	1·308 (5)	1·306 (3)	1·313 (5)
N(3)–C(4)	1·369 (4)	1·349 (3)	1·353 (4)	1·377 (3)	1·374 (5)
N(1)–C(6)	1·346 (4)	1·351 (3)	1·353 (4)	1·332 (3)	1·336 (6)
C(6)–N(6)	1·322 (5)	1·332 (3)	1·325 (4)	1·329 (3)	1·334 (5)
C(5)–N(7)	1·360 (4)	1·385 (3)	1·375 (4)	1·345 (3)	1·359 (5)
N(7)–N(8)	1·358 (5)	—	—	1·348 (3)	1·363 (5)
N(8)–C(9)	1·334 (4)	—	—	1·355 (3)	1·324 (5)
Bond angles (°)					
N(1)–C(6)–C(5)	117·6 (3)	117·4 (2)	113·5 (3)	117·3 (2)	116·4 (3)
C(2)–N(1)–C(6)	119·2 (3)	119·3 (3)	124·2 (3)	118·7 (2)	119·1 (3)
C(2)–N(3)–C(4)	116·9 (3)	110·4 (2)	111·6 (3)	112·6 (2)	112·5 (3)
C(5)–N(7)–N(8)	110·8 (3)	—	—	103·2 (2)	110·9 (3)

3′-dF.HCl: this study; Ad: adenosine (Lai & Marsh, 1972); Ad.HCl: adenosine hydrochloride (Shikata, Ucki & Mitsui, 1973); 8-MeF: 8-methylformycin (Abola *et al.*, 1974); F: formycin (Prusiner, Brennan & Sundaralingam, 1973).

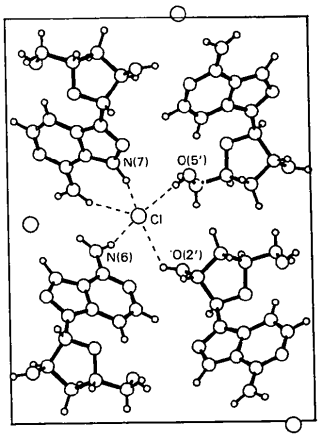


Fig. 3. Molecular packing, showing hydrogen-bond and electrostatic interactions.

The conformation about the glysosidic bond is *syn* in 3′-dF, with a χ of 14·9 (4)°. Formycin itself (Prusiner, Brennan & Sundaralingam, 1973) has a high *anti* χ of −70·2°, whereas the hydrobromide salt (Koyama, Umezawa & Iitaka, 1974) and 8-methylformycin (Abola *et al.*, 1974) are both in the *syn* range with χ values of 30·7 and 25·2° respectively. This *syn* conformation is stabilized in 3′-dF and in both earlier structures by an intramolecular hydrogen bond between the sugar O(5′) and the base N(3) atom. Only in the case of 3′-dF, however, does N(3) act as a hydrogen-bond donor, with N(3)···O(5′) 2·762 (3), N(3)–H(3)···O(5′) 1·87 (3) Å and an angle of 165 (3)° at the H atom (Table 2). The glycosidic conformation in formycins has been extensively studied by both theoretical and solution NMR methods (Birnbaum & Shugar, 1987). The relative populations of *syn* and *anti* conformations in solution are not yet clear, although the absence of a hydrogen atom at the 8-position in formycins compared with normal purines does suggest less steric hindrance, and hence a lower barrier to glycosidic bond rotation. The intramolecular hydrogen bonding observed for 3′-dF can thus readily occur, with consequent domination of the *syn* conformation for protonated formycins.

The deoxyribose sugar moiety of 3′-dF has a C3′-*endo* N-type pucker, with a pseudorotation phase angle *P* (Altona & Sundaralingam, 1972) of 24·8 (9)°. *Syn* glycosidic angles are commonly correlated with C(2′)-*endo* puckers (Saenger, 1984); the situation found both for 3′-dF here, and in 8-methylformycin (Abola *et al.*, 1974) suggests that the intramolecular hydrogen bond has more favourable geometry with C(3′)-*endo* puckering of the sugar. The conformation about the C(4′)–C(5′) bond is *gauche*$^+$ with respect to a torsion angle [O(5′)–C(5′)–C(4′)–C(3′)] of 55·5 (4)°, and is therefore in the most commonly observed range for this variable (Saenger, 1984).

There is an extensive network of hydrogen-bond and electrostatic interactions (Fig. 3). The chloride ion is probably involved in five interactions, although the one to O(2′) is weak.

References

ABOLA, J. E., SIMS, M. J., ABRAHAM, D. J., LEWIS, A. F. & TOWNSEND, L. B. (1974). *J. Med. Chem.* **17**, 62–65.

ALTONA, C. & SUNDARALINGAM, M. (1972). *J. Am. Chem. Soc.* **94**, 8205–8212.

BIRNBAUM, G. & SHUGAR, D. (1987). In *Topics in Nucleic Acid Structure*, Vol. 3, edited by S. NEIDLE. London: Macmillan. In the press.

FRENZ, B. A. (1980). *Enraf–Nonius Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.

HORI, M., ITO, E., TAKITA, T., KOYAMA, G., TAKEUCHI, T. & UMEZAWA, H. (1964). *J. Antibiot. Ser. A*, **17**, 96–99.

International Tables for X-ray Crystallography. (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

KOYAMA, G., UMEZAWA, H. & IITAKA, Y. (1974). *Acta Cryst.* B**30**, 1511–1516.

LAI, T. F. & MARSH, R. E. (1972). *Acta Cryst.* B**28**, 1982–1989.

PRUSINER, P., BRENNAN, T. & SUNDARALINGAM, M. (1973). *Biochemistry*, **12**, 1196–1202.

ROGLER-BROWN, T., AGARWAL, R. P. & PARKS, R. E. (1978). *Biochem. Pharmacol.* **27**, 2289–2296.

SAENGER, W. (1984). *Principles of Nucleic Acid Structure*. Berlin: Springer.

SERAFINOWSKI, P. (1987). In preparation.

SHELDRICK, G. M. (1984). *SHELX*84. Program for crystal structure determination. Univ. of Göttingen, Federal Republic of Germany.

SHIEH, H.-S., BERMAN, H. M., NEIDLE, S., TAYLOR, G. & SANDERSON, M. (1982). *Acta Cryst.* B**38**, 523–531.

SHIKATA, K., UEKI, T. & MITSUI, T. (1973). *Acta Cryst.* B**29**, 31–38.

SUCK, D., MANOR, P. C. & SAENGER, W. (1976). *Acta Cryst.* B**32**, 1727–1737.

SUHADOLNIK, R. J. (1979). *Nucleosides as Biological Probes*. New York: John Wiley.

TAYLOR, R. & KENNARD, O. (1982). *J. Mol. Struct.* **78**, 1–28.

WALKER, N. L. & STUART, D. I. (1983). *Acta Cryst.* A**39**, 158–166.