Table 3. *Deviations* (Å) *of atoms from the least-squares planes through the five atoms of the γ-lactone rings*

	Eupalmerin acetate[a]	Lobophytolide[b]	Jeunicin iodobenzoate[c]	Peunicin[d]	Cueunicin acetate[e]
O(1)	0·104	0·030	0·127	0·097	0·047
C(1)	0·159	0·074	0·181	0·127	0·051
C(14)	−0·161	−0·064	−0·188	−0·137	−0·059
C(15)	−0·105	−0·061	−0·115	−0·080	−0·028
C(16)	0·004	0·021	0·004	−0·007	−0·011
C(17)	−0·388	−0·271	−0·459	−0·285	−0·122
O(2)	0·027	−0·044	0·015	0·009	−0·029
Mean e.s.d.'s (Å)	0·002	0·01	0·01	0·002	0·002

References: (*a*) Ealick *et al.* (1975); (*b*) Karlsson (1977); (*c*) van der Helm *et al.* (1976); (*d*) Chang *et al.* (1980); (*e*) present communication.

The γ-lactone ring deviates from the ideal envelope conformation. However, the sum of endocyclic torsion angles ($\sum|\gamma|$) of 34 (3)° is significantly smaller than that found in other cembranolides [*i.e.* 76° in peunicin (Chang, Ciereszko, Hossain & van der Helm, 1980), 106° in jeunicin (van der Helm *et al.*, 1976)]. The lactone ring is *cis*-fused with the cembrane ring and its plane lies nearly perpendicular to the plane of the cembrane ring (dihedral angle is 86·2°). The bond distances and angles of the γ-lactone are nearly identical with those observed in other cembranolides, eupalmerin acetate (Ealick, van der Helm & Weinheimer, 1975), eunicin (Hossain, Nicholas & van der Helm, 1968), jeunicin, peunicin and lobophytolide (Karlsson, 1977). But unlike in other cembranolides, the five-membered lactone ring is nearly flat in the present structure (Table 3). The r.m.s. deviation of the atoms is 0·043 Å in cueunicin and this value is about 1/3 of the r.m.s. deviation in the other molecules. The only exception is lobophytolide, where the lactone ring is *trans*-fused with the cembrane ring. Cueunicin is the only example where a *cis*-fused γ-lactone assumes such a planar geometry. Even the deviation of the *exo* methylene group [atom C(17)] in the present structure is quite small, 0·122 Å, as compared to 0·459 Å in jeunicin, 0·388 Å in eupalmerin acetate and 0·285 Å in peunicin.

We thank Dr R. A. Gross for a sample of cueunicin acetate. This work was supported by grant CA 17562, awarded by the National Cancer Institute.

References

AHMED, F. R. (1966). *SFLS* program *NRC*-10. National Research Council, Ottawa, Canada.

CHANG, C. Y., CIERESZKO, L. S., HOSSAIN, M. B. & VAN DER HELM, D. (1980). *Acta Cryst.* B**36**, 731–733.

EALICK, S. E., VAN DER HELM, D. & WEINHEIMER, A. J. (1975). *Acta Cryst.* B**31**, 1618–1626.

GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

GROSS, R. A. (1974). PhD thesis, Univ. of Oklahoma.

HOSSAIN, M. B., NICHOLAS, A. F. & VAN DER HELM, D. (1968). *Chem. Commun.* pp. 385–386.

International Tables for X-ray Crystallography (1974). Vol. IV, p. 73. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

KARLE, J. & KARLE, I. L. (1966). *Acta Cryst.* **21**, 849–859.

KARLSSON, R. (1977). *Acta Cryst.* B**33**, 2032–2034.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

TURSCH, B., BRAEKMAN, J. C., DALOZE, D. & KAISIN, M. (1978). *Marine Natural Products*, Vol. II, edited by P. J. SCHEUER, pp. 247–296. New York: Academic Press.

VAN DER HELM, D., ENWALL, E. L., WEINHEIMER, A. J., KARNS, T. K. B. & CIERESZKO, L. S. (1976). *Acta Cryst.* B**32**, 1558–1560.

WEINHEIMER, A. J., MATSON, J. A., HOSSAIN, M. B. & VAN DER HELM, D. (1977). *Tetrahedron Lett.* pp. 2923–2926.

Acta Cryst. (1986). C**42**, 436–440

Structure of Methyl 2,3′-*O*-Anhydro-1-β-D-fructofuranosylorotate

BY SEIICHIRO YAWATA, YASUO HATA AND YUKITERU KATSUBE

Institute for Protein Research, Osaka University, Suita, Osaka 565, *Japan*

AND TOSHIO TATSUOKA

Suntory Institute for Biomedical Research, Suntory Ltd, 1–1–1 *Wakayamadai, Shimamoto-Cho, Mishima-Gun, Osaka* 618, *Japan*

(*Received* 24 *September* 1985; *accepted* 30 *October* 1985)

Abstract. $C_{12}H_{14}N_2O_8$, $M_r = 314{\cdot}25$, orthorhombic, $P2_12_12_1$, $a = 13{\cdot}601$ (1), $b = 20{\cdot}533$ (1), $c = 9{\cdot}497$ (1) Å, $V = 2652{\cdot}3$ (2) Å^3, $Z = 8$, $D_m = 1{\cdot}579$, $D_x = 1{\cdot}574$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 11{\cdot}1$ cm^{-1}, $F(000) = 1312$, room temperature, final $R = 0{\cdot}040$ for 2391 independent observed reflections.

0108-2701/86/040436–05$01.50

One of the two independent molecules has a C(5')-*endo*–C(4')-*exo* sugar conformation ($_4T^5$), and the other has a C(4')-*endo* sugar conformation (^4E). The orientation about the C(5')–C(6') bond is *gauche*–*gauche* (*gauche* ≡ –synclinal) in both molecules, and that about the glycosidic bond is high-*anti* for the former molecule and *anti* for the latter. In the crystal structure, the two kinds of helical arrangement of each molecule along the **c** axis are alternately packed in the **a** and **b** directions, through the network of intermolecular hydrogen bonds.

Introduction. In the biosynthesis of the pyrimidine nucleotides, the synthesis of orotidylic acid by the condensation reaction of orotic acid and phosphoribosyl pyrophosphate is known to be catalyzed by orotate phosphoribosyltransferase. The properties and catalytic mechanism of this enzyme have been studied with various methods. An interesting method for such kinds of studies is that of investigating the reactivity of the enzyme by using substrate analogues formed in the transition state of the enzymatic reaction. Cyclonucleosides can be used as the analogue for this purpose, because they are expected to have similar conformations to those adopted in the transition state through the restriction imposed on the sugar ring system by the cyclization. Various kinds of cyclonucleosides have been synthesized so far (Holý, 1974, 1975), and their conformations have been examined by X-ray crystal-structure analysis (Suck & Saenger, 1973; Delbaere & James, 1973). The comparison of the conformation between non-cyclonucleosides and cyclonucleosides has shown that the former prefer a C(2')-*endo* or C(3')-*endo* ribose puckering (Low, 1983; Srikrishnan, Parthasarathy, De & Chheda, 1983) while the latter adopt a C(4')-*endo* or C(4')-*endo*–C(3')-*exo* ribose puckering which is not preferred by the former. However, the structure of an analogue of orotidylic acid has not yet been determined.

In order to obtain information on the structure of the substrate analogue formed in the transition state in the biosynthetic reaction, we have determined the molecular structure of methyl 2,3'-*O*-anhydro-1-β-D-fructofuranosylorotate by X-ray crystal-structure analysis.

Experimental. The present compound was prepared by the Michaelis–Arbuzov reaction; details will be published elsewhere. Crystals obtained by recrystallization from an ethanol–dichloromethane solution. Colorless needle-shaped crystal. Crystal dimensions 0·75 × 0·15 × 0·13 mm. D_m by flotation in n-C_6H_{14}/CCl_4 solution. Rigaku C-5 automated four-circle diffractometer, Ni-filtered Cu $K\alpha$ radiation from Rigaku RU-200 rotating-anode X-ray generator (operating conditions: 40 kV, 200 mA). Lattice parameters determined by least-squares method from 2θ values for 18 strong reflections with $19 < 2\theta < 47°$. Data collection with $3 < 2\theta < 125°$; θ–2θ scan mode, scan speed 6° min^{-1} (θ), scan width $(1{\cdot}2 + 0{\cdot}15\tan\theta)°$, background counts 4 s before and after each scan, three standard reflections monitored every 100 reflections, intensities of 2414 unique reflections obtained by averaging symmetry-related intensities of 4877 observations (h: −16 to 16, k: 0 to 24, l: 0 to 11), $R_{int} = 0{\cdot}015$; Lorentz–polarization corrections, no absorption corrections. Almost complete structure including H atoms ($R = 0{\cdot}043$) solved by 'the automatic analysis program on a microcomputer' developed in our laboratory; program based on *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978); only a few input data required at the beginning of the job; details will be published elsewhere. Positions of H atoms rechecked in a difference Fourier map. All non-H atoms with anisotropic thermal factors refined by full-matrix least-squares *FMLS* (Ashida, 1973) using 2391 reflections with $|F_o| \geq \sigma(F_o)$, H atoms not refined. $\sum w(|F_o| - |F_c|)^2$ minimized; $w = 1/[\sigma^2(F_o) + 0{\cdot}001|F_o|^2]$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Final $R = 0{\cdot}040$, $wR = 0{\cdot}064$, $(\Delta/\sigma)_{max} = 0{\cdot}138$, $|\Delta\rho|_{max} = 0{\cdot}2$ e Å^{-3}. All calculations carried out with an ACOS S-850 computer at the Crystallographic Research Center, Institute for Protein Research, Osaka University.

Discussion. The final atomic coordinates and equivalent isotropic thermal parameters for non-H atoms are given in Table 1.* Bond distances and angles are listed in Table 2. The molecular structure with the numbering scheme of the atoms is shown in Fig. 1. The bond distances of one of the two crystallographically independent molecules, *A* and *B*, are in good agreement with the corresponding distances of the other molecule with a maximum difference of 0·022 Å in the C(3')–O(3') length. The glycosidic C(2')–N(1) bond length [1·505 (5) and 1·517 (5) Å] is significantly longer than those (*ca* 1·48 Å) observed in nucleosides, which reflects the fact that the glycosidic bond length should increase almost linearly to a maximum (1·52 Å) as the glycosidic torsion angle approaches 180° (Lin, Sundaralingam & Arora, 1971; Lo, Shefter & Cochran, 1975). Some disagreements between the *A* and *B* molecules are observed in the exocyclic bond angles, C(1')–C(2')–N(1), C(1')–C(2')–O(5'), C(3')–C(4')–O(4') and C(4')–C(5')–C(6'), which reflect the difference in the conformation of the furanose ring between molecules *A* and *B*, as shown in Fig. 2.

* Lists of structure factors, selected torsion angles, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42615 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å²) *with e.s.d.'s in parentheses*

$B_{eq} = \frac{4}{3}(\beta_{11}^2 a^2 + \beta_{22}^2 b^2 + \beta_{33}^2 c^2)$.

	x	*y*	*z*	B_{eq}
Molecule *A*				
N(1)	0·8038 (3)	0·5878 (1)	0·3852 (4)	2·16 (7)
C(2)	0·8084 (3)	0·5643 (2)	0·5186 (4)	2·41 (8)
N(3)	0·8480 (3)	0·5104 (2)	0·5618 (4)	2·94 (8)
C(4)	0·8899 (3)	0·4711 (2)	0·4608 (5)	2·90 (9)
O(4)	0·9263 (3)	0·4186 (1)	0·4967 (4)	3·87 (8)
C(5)	0·8881 (3)	0·4932 (2)	0·3152 (4)	2·55 (8)
C(6)	0·8466 (3)	0·5506 (2)	0·2801 (4)	2·28 (8)
C(7)	0·8495 (3)	0·5763 (2)	0·1328 (4)	2·32 (8)
O(7)	0·8292 (3)	0·6310 (1)	0·1000 (4)	3·25 (7)
O(8)	0·8765 (3)	0·5304 (1)	0·0429 (3)	3·06 (7)
C(9)	0·8864 (4)	0·5513 (3)	−0·1016 (5)	4·04 (12)
O(1′)	0·6049 (3)	0·6040 (2)	0·2979 (4)	4·03 (8)
C(1′)	0·6664 (3)	0·6590 (2)	0·2852 (5)	2·86 (9)
C(2′)	0·7538 (3)	0·6532 (2)	0·3832 (4)	2·24 (8)
C(3′)	0·7248 (3)	0·6579 (2)	0·5409 (4)	2·54 (8)
O(3′)	0·7657 (3)	0·6031 (1)	0·6121 (3)	3·22 (7)
C(4′)	0·7690 (3)	0·7223 (2)	0·5926 (5)	2·99 (9)
O(4′)	0·6910 (3)	0·7680 (2)	0·5877 (4)	4·49 (9)
C(5′)	0·8476 (3)	0·7391 (2)	0·4833 (5)	2·59 (9)
O(5′)	0·8204 (2)	0·7036 (1)	0·3556 (3)	2·73 (6)
C(6′)	0·9534 (3)	0·7260 (3)	0·5230 (5)	3·28 (10)
O(6′)	0·9661 (3)	0·6611 (2)	0·5747 (4)	3·66 (7)
Molecule *B*				
N(1)	0·1801 (3)	0·4102 (1)	0·5197 (4)	2·15 (7)
C(2)	0·1878 (3)	0·4378 (2)	0·3909 (4)	2·50 (8)
N(3)	0·1651 (3)	0·4968 (2)	0·3541 (4)	3·06 (8)
C(4)	0·1284 (3)	0·5360 (2)	0·4578 (5)	3·00 (9)
O(4)	0·1057 (3)	0·5935 (1)	0·4292 (4)	4·30 (9)
C(5)	0·1203 (3)	0·5108 (2)	0·5996 (4)	2·44 (8)
C(6)	0·1440 (3)	0·4486 (2)	0·6280 (5)	2·26 (8)
C(7)	0·1343 (3)	0·4208 (2)	0·7740 (5)	2·50 (9)
O(7)	0·1482 (3)	0·3652 (1)	0·8048 (4)	3·96 (8)
O(8)	0·1082 (3)	0·4669 (1)	0·8638 (3)	3·18 (7)
C(9)	0·0950 (4)	0·4468 (3)	1·0085 (5)	4·35 (13)
O(1′)	0·0431 (3)	0·3177 (2)	0·4025 (4)	4·23 (8)
C(1′)	0·1049 (3)	0·2995 (2)	0·5155 (6)	3·47 (11)
C(2′)	0·2013 (3)	0·3379 (2)	0·5089 (5)	2·48 (8)
C(3′)	0·2513 (3)	0·3374 (2)	0·3634 (4)	2·72 (8)
O(3′)	0·2230 (3)	0·3973 (1)	0·2940 (3)	3·32 (7)
C(4′)	0·3608 (3)	0·3353 (2)	0·3979 (5)	2·75 (9)
O(4′)	0·4192 (3)	0·2998 (1)	0·3029 (4)	3·79 (7)
C(5′)	0·3634 (3)	0·3030 (2)	0·5413 (5)	2·33 (8)
O(5′)	0·2694 (2)	0·3166 (1)	0·6066 (3)	2·59 (6)
C(6′)	0·4451 (3)	0·3263 (2)	0·6342 (5)	2·88 (9)
O(6′)	0·4438 (3)	0·3948 (1)	0·6458 (4)	3·56 (7)

The conformation of the furanose ring can be described in terms of the pseudorotation phase angle, *P*, and the maximum torsion angle, τ_m, which are calculated from the endocyclic torsion angles (deposited) (Altona & Sundaralingam, 1972). The values of *P* and τ_m for molecule *A* are 212·2 and 22·2° respectively. The furanose ring of molecule *A* has an unsymmetrical C(5′)-*endo*–C(4′)-*exo* twist conformation ($_4T^5$) where the deviations of the C(4′) and C(5′) atoms from the O(5′)–C(2′)–C(3′) plane are 0·23 (8) and 0·13 (8) Å respectively. The C(5′)-*endo*–C(4′)-*exo* conformation in this compound is equivalent to the C(4′)-*endo*–C(3′)-*exo* in ribonucleoside. This conformation, though not preferred in non-cyclonucleosides, is dominant in cyclonucleosides and their analogues, and seems to reflect the rigidity imposed on the sugar ring by the additional oxazolidine ring formation. The values of *P* and τ_m for molecule *B* are 15·7 and 27·3° respectively. In contrast to molecule *A*, molecule *B* adopts the C(4′)-*endo* envelope conformation (^4E), corresponding to the C(3′)-*endo* in ribose, which is one of the preferred conformations in non-cyclonucleosides. The displacement of the C(4′) atom from the best plane of the other four atoms is 0·43 (1) Å which is larger than that in molecule *A*. It is interesting that in spite of the restriction imposed by the ring formation, the present compound has the C(4′)-*endo* conformation preferred in non-cyclonucleosides. However, the maximum amplitude of pucker (τ_m) is small in both molecules, compared with an average of 39° for normal furanose rings (Altona & Sundaralingam, 1972). The orientation of the C(6′)–O(6′) bond about the exocyclic C(5′)–C(6′) bond is *gauche–gauche* in both molecules. The glycosidic torsion angle χ[O(5′)–C(2′)–N(1)–C(2)] is 108·7 (3)° for molecule *A* and 129·9 (3)° for molecule *B* which corresponds to a high-*anti* and an *anti* conformation respectively. This conformational difference may be due to that in the furanose ring between the two molecules. The difference in the torsion angle O(3′)–C(3′)–C(2′)–N(1) [*A*: 5·1 (4), *B*: −19·1 (4)°] is also dependent on that in the furanose-ring conformation. The exocyclic C(4′)–O(4′) bond in molecule *A* is oriented almost perpendicularly

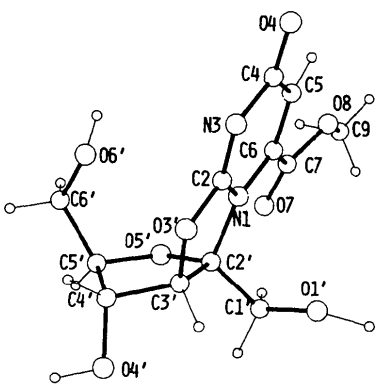

Fig. 1. A *PLUTO*78 drawing (Motherwell & Clegg, 1978) of the molecule showing the atom-numbering scheme.

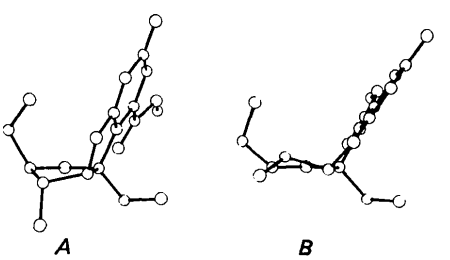

Fig. 2. Drawing of the two independent molecules showing the sugar puckering and the base stacking.

Table 2. *Bond distances* (Å) *and angles* (°) *with their e.s.d.'s in parentheses*

	A	*B*		*A*	*B*		*A*	*B*
N(1)–C(2)	1·357 (5)	1·352 (5)	C(5)–C(6)	1·349 (5)	1·344 (5)	C(2′)–C(3′)	1·552 (5)	1·540 (6)
N(1)–C(2′)	1·505 (5)	1·517 (5)	C(6)–C(7)	1·496 (5)	1·506 (5)	C(3′)–O(3′)	1·425 (5)	1·447 (5)
N(1)–C(6)	1·385 (5)	1·384 (5)	C(7)–O(7)	1·197 (5)	1·194 (5)	C(3′)–C(4′)	1·533 (6)	1·525 (6)
C(2)–O(3′)	1·328 (5)	1·330 (5)	C(7)–O(8)	1·324 (5)	1·324 (5)	C(4′)–O(4′)	1·417 (5)	1·407 (5)
C(2)–N(3)	1·296 (5)	1·297 (5)	O(8)–C(9)	1·445 (6)	1·446 (6)	C(4′)–C(5′)	1·529 (6)	1·516 (6)
N(3)–C(4)	1·377 (6)	1·366 (6)	C(1′)–O(1′)	1·410 (5)	1·414 (6)	C(5′)–C(6′)	1·511 (6)	1·497 (6)
C(4)–O(4)	1·234 (6)	1·251 (6)	C(1′)–C(2′)	1·514 (6)	1·531 (6)	C(5′)–O(5′)	1·462 (5)	1·449 (5)
C(4)–C(5)	1·455 (6)	1·447 (6)	C(2′)–O(5′)	1·400 (5)	1·380 (5)	C(6′)–O(6′)	1·432 (5)	1·412 (5)
C(2)–N(1)–C(6)	117·2 (3)	117·5 (3)	N(1)–C(6)–C(7)	119·4 (3)	120·0 (3)	O(5′)–C(2′)–C(3′)	107·4 (3)	107·8 (3)
C(2)–N(1)–C(2′)	110·5 (3)	109·5 (3)	C(6)–C(7)–O(7)	124·6 (3)	125·1 (4)	O(3′)–C(3′)–C(2′)	108·0 (3)	106·6 (3)
C(6)–N(1)–C(2′)	132·3 (3)	132·4 (3)	C(6)–C(7)–O(8)	111·0 (3)	110·2 (3)	O(3′)–C(3′)–C(4′)	112·1 (3)	112·4 (3)
N(1)–C(2)–N(3)	128·2 (4)	128·0 (4)	O(7)–C(7)–O(8)	124·3 (3)	124·7 (4)	C(2′)–C(3′)–C(4′)	105·2 (3)	103·8 (3)
N(1)–C(2)–O(3′)	112·9 (3)	113·1 (3)	C(7)–O(8)–C(9)	115·3 (3)	116·2 (3)	C(3′)–C(4′)–O(4′)	105·5 (3)	115·4 (3)
O(3′)–C(2)–N(3)	118·9 (3)	118·9 (3)	C(2)–O(3′)–C(3′)	109·2 (3)	108·2 (3)	C(3′)–C(4′)–C(5′)	104·5 (3)	103·2 (3)
C(2)–N(3)–C(4)	116·8 (4)	116·3 (4)	O(1′)–C(1′)–C(2′)	110·5 (3)	110·1 (4)	O(4′)–C(4′)–C(5′)	110·7 (3)	109·6 (3)
N(3)–C(4)–C(5)	118·2 (4)	119·2 (4)	C(1′)–C(2′)–N(1)	115·6 (3)	109·8 (3)	C(4′)–C(5′)–O(5′)	105·9 (3)	106·2 (3)
N(3)–C(4)–O(4)	119·0 (4)	119·4 (4)	C(1′)–C(2′)–O(5′)	109·6 (3)	112·6 (3)	C(4′)–C(5′)–C(6′)	117·2 (3)	114·0 (3)
O(4)–C(4)–C(5)	122·8 (4)	121·4 (4)	C(1′)–C(2′)–C(3′)	112·9 (3)	114·3 (4)	C(5′)–C(6′)–O(6′)	111·4 (3)	110·8 (3)
C(4)–C(5)–C(6)	120·9 (4)	120·5 (4)	N(1)–C(2′)–C(3′)	99·1 (3)	98·7 (3)	O(5′)–C(5′)–C(6′)	111·1 (3)	110·0 (3)
C(5)–C(6)–N(1)	118·7 (3)	118·5 (3)	N(1)–C(2′)–O(5′)	111·7 (3)	113·1 (3)	C(2′)–O(5′)–C(5′)	112·1 (3)	111·4 (3)
C(5)–C(6)–C(7)	121·9 (3)	121·6 (3)						

to the furanose-ring plane with a torsion angle C(2′)–C(3′)–C(4′)–O(4′) of 98·0 (4)°. In molecule *B*, the corresponding torsion angle is 145·9 (3)° and the C(4′)–O(4′) bond is oriented in the direction so that the O(4′) atom is kept away from the sugar plane. The C(1′)–C(2′) bonds of both molecules have similar orientations to each other with respect to the C(5′)–O(5′)–C(2′)–C(3′) plane; the torsion angles C(1′)–C(2′)–O(5′)–C(5′) are 128·5 (3) and 128·4 (4)° respectively. The C(1′)–O(1′) bond is nearly *trans* to the C(2′)–O(5′) bond in both molecules, but a comparison of the torsion angle O(1′)–C(1′)–C(2′)–O(5′) [*A*: 172·1 (3), *B*: −172·8 (3)°] in both molecules shows that they have opposite orientations of the O(1′)–C(1′) bond with respect to the O(5′)–C(2′)–C(1′) plane.

The conformation of the methyl orotate moiety can be described by the relationship between two planes; plane (I) consists of the N(1), C(2), O(3′), N(3), C(4), O(4), C(5), C(6) and C(7) atoms, and plane (II) corresponds to the carboxylate moiety. In molecule *A*, the C(2′) and C(3′) atoms are also located on plane (I) with deviations of 0·002 (6) Å for C(2′) and 0·071 (6) Å for C(3′), and the pyrimidine ring and its adjacent oxazolidine ring formed by the C(3′)–O(3′) cyclization are essentially coplanar. In molecule *B*, the C(2′) and C(3′) atoms deviate by 0·201 (9) and 0·133 (9) Å from plane (I), respectively. In both molecules, planes (I) and (II) are not coplanar. The torsion angle N(1)–C(6)–C(7)–O(7) is 10·5 (6)° in molecule *A* and −6·3 (6)° in molecule *B*. The molecules, as mentioned above, seem to adopt the two different conformations in order to adapt themselves to the environment around them in the crystalline state.

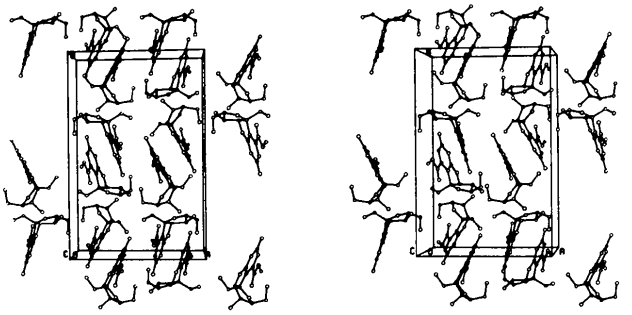

Fig. 3. A stereoscopic view of the crystal structure drawn by *PLUTO*78; viewed along **c**, **a** horizontal and **b** vertical.

In the crystal structure, as shown in Fig. 3, the two independent molecules are packed near the different crystallographic twofold screw axes parallel to the **c** axis. Molecules *A* related by one of the 2_1 axes are linked to each other by intermolecular hydrogen bonds [O(1′)–H···O(4), 2·929 (5) Å] to make an extended helical chain *A* around the 2_1 axis. In a similar way, molecules *B* are also linked by intermolecular hydrogen bonds [O(6′)–H···O(4), 2·785 (5) Å] to make a helix *B* around another 2_1 axis. Each sugar moiety in helix *A* is projected out of the surface of the helix while in helix *B* they are located on the surface of the helix. The *A* and *B* helices along the **c** axis, which are packed alternately in the **a** and **b** directions, are linked to one another by intermolecular hydrogen bonds [O(6′):*A*···O(4):*B*, 2·727 (5) Å] and [O(1′):*B*···O(4):*A*, 2·760 (5) Å] in the **a** direction, and [O(4′):*A*···O(4):*B*, 3·075 (5) Å] in the **b** direction.

References

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

ASHIDA, T. (1973). *FMLS. The Universal Crystallographic Computing System–Osaka.* The Computation Center, Osaka Univ., Japan.

DELBAERE, L. T. & JAMES, M. N. G. (1973). *Acta Cryst.* B**29**, 2905–2912.

HOLÝ, A. (1974). *Nucleic Acids Res.* **1**, 289–298.

HOLÝ, A. (1975). *Czech. Chem. Commun.* **40**, 738–752.

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

LIN, G. H.-Y., SUNDARALINGAM, M. & ARORA, S. K. (1971). *J. Am. Chem. Soc.* **93**, 1235–1241.

LO, A., SHEFTER, E. & COCHRAN, T. G. (1975). *J. Pharm. Sci.* **64**, 1707–1710.

LOW, J. N. (1983). *Acta Cryst.* C**39**, 796–798.

MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO78.* A program for plotting molecular and crystal structures. Univ. of Cambridge, England.

SRIKRISHNAN, T., PARTHASARATHY, R., DE, N. C. & CHHEDA, G. B. (1983). *Acta Cryst.* C**39**, 1441–1445.

SUCK, D. & SAENGER, W. (1973). *Acta Cryst.* B**29**, 1323–1330.

Acta Cryst. (1986). C**42**, 440–442

Structure of 1-(4-Chloro-2-fluorophenyl)-4,4-dimethyl-2-(1*H*-1,2,4-triazol-1-yl)-3-pentanone

BY S. K. BRANCH*

School of Pharmacy and Pharmacology, University of Bath, Claverton Down, Bath BA2 7AY, England

AND I. W. NOWELL

Department of Chemistry, Sheffield City Polytechnic, Pond Street, Sheffield S1 1WB, England

(*Received* 11 *January* 1985; *accepted* 19 *August* 1985)

Abstract. $C_{15}H_{17}ClFN_3O$, $M_r = 309{\cdot}8$, orthorhombic, $P2_12_12_1$, $a = 19{\cdot}813\ (8)$, $b = 13{\cdot}699\ (6)$, $c = 5{\cdot}777\ (3)$ Å, $V = 1568{\cdot}0$ Å^3, $Z = 4$, $D_m = 1{\cdot}29\ (2)$, $D_x = 1{\cdot}31$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}213$ mm^{-1}, $F(000) = 648$, room temperature, final $R = 0{\cdot}053$ for 1003 reflections classed as observed. C atom 1 of the phenyl ring and C atoms 1 to 4 of the pentanone function adopt an extended 'W' conformation with the atoms essentially coplanar. The triazolyl ring, which shows significant delocalization [N–N bond length 1·357 (9) Å; C–N 1·324–1·337 Å], and the chlorofluorophenyl ring are twisted by 86 (1) and 58 (1)°, respectively, out of this plane. The exocyclic angles of the triazolyl ring are very asymmetric with C–N–C [130·6 (7)°] significantly larger than C–N–N [119·1 (6)°]. The F atom is disordered being distributed (80,20% occupancy) between positions 2 and 6 in the phenyl ring. The title compound was recrystallized from a racemic mixture but the absolute configuration [(2*S*)-enantiomer] of the molecules in the crystal under investigation was established by comparison of *wR* values for the determined and the inverted structure.

* To whom correspondence should be addressed.

0108-2701/86/040440-03$01.50

Introduction. The title compound is one of a group of azolylmethane fungicides (Balasubramanyan & Shephard, 1975) which are related to the well established systemic fungicide triadimefon (Martin & Morris, 1979), whose structure has been reported previously (Nowell, Walker & Anderson, 1982). We have determined the structure of the title compound to establish its solid-state conformation as part of a programme investigating the relationship between conformation and biological activity in this class of compounds. A preliminary description of this structure has been published (Anderson, Branch, Loeffler, Mann, Nowell & Walker, 1984).

Experimental. Recrystallization from ethanol:water (2:1); D_m by flotation in benzene/CCl_4; crystal, clear colourless needle, approximate dimensions 0·38 × 0·24 × 0·16 mm, mounted about crystallographic *c* axis; Stoe Stadi-2 two-circle diffractometer, Mo $K\alpha$ radiation; Lorentz, polarization but no absorption corrections applied; one standard reflection monitored for each layer collected, intensity variation <4%; 1890 reflections up to $\theta = 27{\cdot}5°$; index ranges *h* 0 to 24, *k* 0 to 17, *l* 0 to 6; 1829 independent reflections of which 1003 with $I \geq 2{\cdot}0\sigma(I)$ con-