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Structures of the 2′,3′-Dideoxy and 2′,3′-Didehydro-2′,3′-dideoxy Analogs of Tiazofurin

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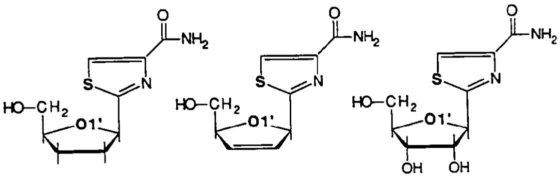
Abstract. 2′,3′-Dideoxytiazofurin [2-(2′,3′-dideoxy-β-D-*glycero*-pentafuranosyl)thiazole-4-carboxamide] hemihydrate (1), $C_9H_{12}N_2O_3S.\frac{1}{2}H_2O$, $M_r = 237{\cdot}3$, monoclinic, $C2$, $a = 23{\cdot}141\ (2)$, $b = 5{\cdot}912\ (1)$, $c = 8{\cdot}252\ (1)$ Å, $\beta = 90{\cdot}71\ (1)°$, $V = 1128{\cdot}9\ (4)$ Å^3, $Z = 4$, $D_x = 1{\cdot}396$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}54178$ Å, $\mu = 25{\cdot}0$ cm^{-1}, $F(000) = 500$, $T = 295$ K, $R = 0{\cdot}0319$ for all 1316 unique reflections. 2′,3′-Didehydro-2′,3′-dideoxytiazofurin [2-(2′,3′-dideoxy-β-D-*glycero*-pent-2-enofuranosyl)thiazole-4-carboxamide] (2), $C_9H_{10}N_2O_3S$, $M_r = 226{\cdot}3$, orthorhombic, $P2_12_12_1$, $a = 22{\cdot}172\ (2)$, $b = 8{\cdot}019\ (1)$, $c = 5{\cdot}991\ (1)$ Å, $V = 1065{\cdot}2\ (4)$ Å^3, $Z = 4$, $D_x = 1{\cdot}411$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}54178$ Å, $\mu = 25{\cdot}9$ cm^{-1}, $F(000) = 472$, $T = 295$ K, $R = 0{\cdot}0344$ for all 957 unique reflections. Both structures show a close contact between the thiazole S and the pentose O(1′) atoms. S···O(1′) distances are 2·834 (2) Å in (1) and 2·835 (1) Å in (2), resulting from *C*-glycosidic torsion angles of 14·1 (2) and 5·2 (3)° respectively. This unusual feature is conserved in the crystal structures of other thiazole nucleosides [Goldstein, Mao & Marquez (1988). *J. Med. Chem.* **31**, 1026–1031].

Introduction. Tiazofurin (3) is an antitumor drug whose crystal structure exhibits an unusual close sulfur–furanose-oxygen contact (Goldstein, Takusagawa, Berman, Srivastava & Robins, 1983). Similar close contacts are observed in crystal structures of other thiazole nucleosides (Goldstein, Mao & Marquez, 1988). Observation of this feature in a variety of packing environments suggests that it is due to an intramolecular interaction rather than to external packing forces. Computational studies (Burling & Goldstein, 1989) indicate that electrostatic interactions between the thiazole S and ribose O(1′) atoms may be responsible for the observed close contacts. These computations suggest that the S···O(1′) interaction may limit rotation of the thiazole ring about the *C*-glycosidic bond. This constraint would have important consequences with regard to the antitumor activity of tiazofurin (Goldstein, Bell & Marquez, 1990). In this study we examine the structures of two tiazofurin analogs: 2′,3′-dideoxytiazofurin (1) and 2′,3′-didehydro-2′,3′-dideoxytiazofurin (2). These structures provide further evidence for a sulfur–oxygen interaction in thiazole nucleosides.



(1) (2) (3)

Experimental. The preparation of (1) and (2) is described by Upadhya, DaRe, Schubert, Chmurny & Gabrielsen (1990). Compound (1) was crystallized by slow evaporation at 277 K from isopropanol. Compound (2) was obtained by slow evaporation at 277 K from ethyl acetate. Both crystals were colorless. Crystal dimensions along with other data-collection and refinement variables are given in Table 1. Full-sphere data were collected at room temperature for both structures and corrected for Lorentz and polarization factors. Corrections were applied for variations in beam intensity *via* a polynomial fit to three standards. Data were corrected for absorption using the semiempirical ψ-scan technique (North, Phillips & Mathews, 1968). Both structures were solved using standard Patterson and Fourier

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Table 1. *Data collection and refinement*

	(1) Dideoxy-tiazofurin	(2) Didehydrodideoxy-tiazofurin
Crystal size (mm)	0·09 × 0·24 × 0·29	0·13 × 0·26 × 0·37
Diffractometer	CAD-4	CAD-4
Scan type	ω–2θ	ω–2θ
Scan width in ω ( )	0·8 + 0·14tanθ	0·8 + 0·14tanθ
Lattice-parameter refinement	25 reflections ($40·5 < \theta < 42·0$)	24 reflections ($4·0 < \theta < 19·0$)
Transmission factor (on I)	0·782–1·000	0·724–1·000
Net variation in standards (%)	+1·5	−0·8
Data-collection range ( )	$1·5 < \theta < 78·0$	$1·5 < \theta < 60·0$
$\pm h_{\max}$, $\pm k_{\max}$, $\pm l_{\max}$	±29, ±7, ±10	±24, ±9, ±6
Total reflections	4778 [$4667 > 3\sigma(I)$]	3258 [$3137 > 3\sigma(I)$]
Unique reflections	1316	957
$R_{\rm merge}$	0·02	0·02
R (all data)	0·0319	0·0344
wR (all data)	0·0367	0·0397
No. of variables	192	177
S	1·187	1·179
$(\Delta/\sigma)_{\max}$	0·03	0·01
$(\Delta\rho)_{\max}$ (e Å^{-3})	0·43	0·26
Extinction parameter g ($\times 10^{-4}$)	1·7 (2)	0·9 (2)

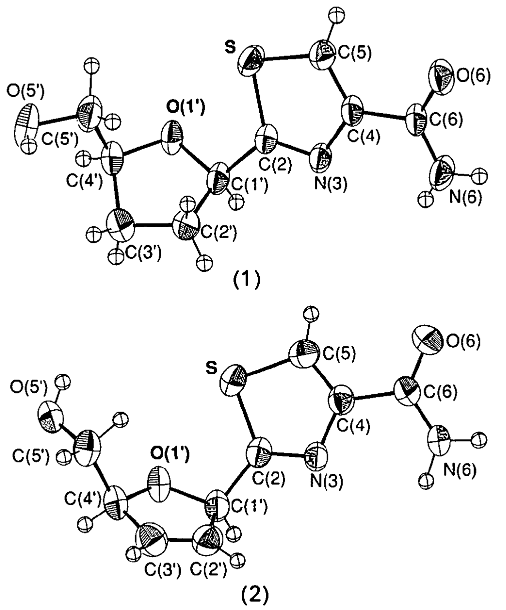

Fig. 1. Molecular structures of dideoxytiazofurin (1) and didehydrodideoxytiazofurin (2). Thermal ellipsoids of non-H atoms are drawn at the 50% probability level.

Table 2. *Fractional coordinates and equivalent isotropic thermal parameters for non-*H *atoms*

$$B_{\rm eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$(Å^2)
(1) Dideoxytiazofurin				
S	0·21387 (2)	0·29961	0·18658 (6)	3·76 (2)
C(2)	0·22549 (7)	0·5292 (4)	0·3152 (2)	2·77 (9)
N(3)	0·27573 (6)	0·6254 (3)	0·3032 (2)	2·89 (8)
C(4)	0·30757 (7)	0·5182 (4)	0·1852 (2)	2·83 (9)
C(5)	0·28146 (9)	0·3423 (4)	0·1102 (3)	3·5 (1)
C(6)	0·36748 (7)	0·5929 (4)	0·1462 (2)	2·97 (8)
N(6)	0·38942 (8)	0·7526 (4)	0·2390 (3)	4·3 (1)
O(6)	0·39259 (6)	0·5069 (4)	0·0315 (2)	4·08 (8)
C(1′)	0·17841 (8)	0·6038 (4)	0·4277 (2)	3·1 (1)
C(2′)	0·14628 (9)	0·8166 (5)	0·3721 (3)	3·9 (1)
C(3′)	0·08411 (9)	0·7701 (5)	0·4268 (3)	4·2 (1)
C(4′)	0·07883 (7)	0·5190 (4)	0·4022 (2)	3·22 (9)
C(5′)	0·0584 (1)	0·4417 (6)	0·2373 (3)	4·2 (1)
O(1′)	0·13596 (5)	0·4268 (3)	0·4328 (2)	3·26 (7)
O(5′)	−0·00150 (6)	0·4931 (5)	0·2160 (2)	4·81 (9)
O(*W*)	0·0	0·8455 (5)	0·0	5·02 (3)
(2) Didehydrodideoxytiazofurin				
S	0·21877 (3)	0·1668 (1)	0·2127 (1)	4·24 (3)
C(2)	0·2286 (1)	0·3109 (3)	0·0029 (5)	3·1 (1)
N(3)	0·28184 (9)	0·3104 (3)	−0·0904 (4)	3·1 (1)
C(4)	0·3171 (1)	0·1897 (3)	0·0062 (5)	3·2 (1)
C(5)	0·2912 (1)	0·1012 (4)	0·1694 (6)	4·0 (2)
C(6)	0·3809 (1)	0·1622 (4)	−0·0682 (5)	3·4 (1)
N(6)	0·4021 (1)	0·2715 (4)	−0·2130 (5)	4·4 (1)
O(6)	0·40952 (9)	0·0436 (3)	0·0051 (4)	4·8 (1)
C(1′)	0·1782 (1)	0·4256 (4)	−0·0608 (5)	3·4 (1)
C(2′)	0·1520 (2)	0·3962 (4)	−0·2866 (6)	4·0 (2)
C(3′)	0·0947 (1)	0·3611 (4)	−0·2724 (5)	4·3 (2)
C(4′)	0·0748 (1)	0·3557 (4)	−0·0339 (5)	3·6 (1)
C(5′)	0·0523 (1)	0·1871 (5)	0·0373 (6)	4·3 (2)
O(1′)	0·12878 (8)	0·4009 (3)	0·0906 (3)	3·5 (1)
O(5′)	0·03075 (9)	0·1904 (3)	0·2604 (4)	4·5 (1)

methods. All hydrogen positions were determined from difference Fourier maps computed from low-angle data ($\sin\theta/\lambda < 0·4$ Å^{-1}). Full-matrix least-squares anisotropic refinement of all non-H atoms and isotropic refinement of all H atoms minimized the function $\sum w(\Delta F)^2$. Weights were $w = 1/\sigma'^2$ with $\sigma'^2 = \{\sigma^2 + 0·5A|F_o|^2 + 0·5B[(\sin\theta)/\lambda]^2\}^{1/2}$. σ^2 were from counting statistics and A and B were obtained from least-squares minimization of the function $|\Delta F|^2 - \sigma'^2$ in 20 separate segments in $|F_o|$ and $(\sin\theta)/\lambda$ for each data set. Final refinements utilized all data and included a type I isotropic extinction correction (Coppens & Hamilton, 1970). Atomic scattering factors and f' and f'' for S atoms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99–102, 149–150). All programs were from the *DNA* system (Takusagawa, 1981). The conformations of (1) and (2) are illustrated in Fig. 1. Atomic coordinates are given in Table 2. Bond lengths, bond angles and selected torsion angles are listed in Table 3.*

Discussion. *Close* S···O(1′) *contacts* are observed in each structure. As with the other thiazole nucleosides, in both (1) and (2) the thiazole S is *cis* to the ribose O atom. The S—O distances [2·834 (2) for (1), 2·835 (1) Å for (2)] are less than the sum of the van der Waals radii (3·30 Å) of S and O (Bondi, 1964) and are in fact smaller than that reported for tiazofurin [2·958 (1) Å] (Goldstein *et al.*, 1983). Structures (1) and (2) show the smallest values of the *C*-glycosidic torsion angle χ [O(1′)—C(1′)—C(2)—S] seen thus far in the thiazole nucleosides [χ = 14·1 and 5·2° for (1) and (2) respectively]. Close S···O(1′) contacts have now been observed in seven thiazole nucleosides (Goldstein *et al.*, 1988), suggesting that

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* Lists of structure factors, anisotropic thermal parameters, least-squares planes and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53666 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Bond distances* (Å), *bond angles* (°) *and selected torsion angles* (°)

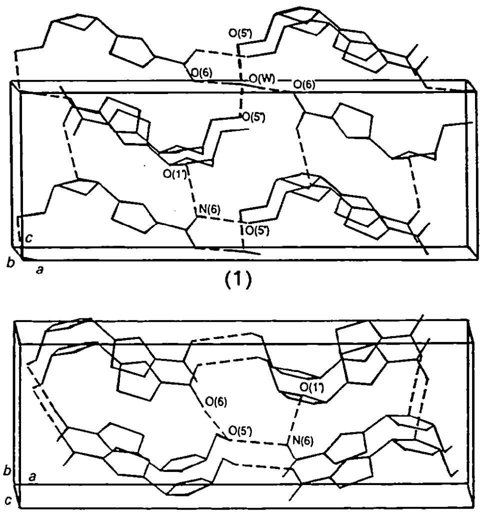
	(1)	(2)
C(2)—N(3)	1·299 (2)	1·307 (3)
N(3)—C(4)	1·382 (2)	1·372 (3)
C(4)—C(5)	1·349 (3)	1·338 (4)
C(5)—S	1·712 (2)	1·710 (3)
S—C(2)	1·742 (2)	1·721 (3)
C(2)—C(1′)	1·506 (3)	1·496 (4)
C(4)—C(6)	1·494 (2)	1·500 (4)
C(6)—O(6)	1·227 (3)	1·224 (4)
C(6)—N(6)	1·314 (3)	1·320 (4)
C(1′)—C(2′)	1·529 (4)	1·490 (5)
C(2′)—C(3′)	1·538 (2)	1·305 (5)
C(3′)—C(4′)	1·503 (4)	1·496 (5)
C(4′)—O(1′)	1·449 (2)	1·457 (3)
C(4′)—C(5′)	1·506 (3)	1·502 (5)
O(1′)—C(1′)	1·436 (3)	1·436 (3)
C(5′)—O(5′)	1·427 (3)	1·420 (4)

	(1)	(2)
C(2)—S—C(5)	88·5 (1)	88·7 (1)
S—C(5)—C(4)	110·5 (2)	110·5 (2)
C(5)—C(4)—N(3)	115·9 (2)	116·0 (2)
C(4)—N(3)—C(2)	109·8 (2)	109·7 (2)
N(3)—C(2)—S	115·2 (1)	115·1 (2)
C(1′)—C(2)—N(3)	125·0 (2)	124·6 (2)
C(1′)—C(2)—S	119·8 (1)	120·3 (2)
C(6)—C(4)—N(3)	121·4 (2)	121·1 (2)
C(6)—C(4)—C(5)	122·7 (2)	122·9 (3)
C(4)—C(6)—O(6)	119·5 (2)	119·8 (3)
C(4)—C(6)—N(6)	116·1 (2)	115·6 (3)
O(6)—C(6)—N(6)	124·3 (2)	124·6 (3)
C(4′)—O(1′)—C(1′)	110·1 (2)	109·8 (2)
O(1′)—C(1′)—C(2′)	106·1 (2)	104·7 (2)
C(2)—C(1′)—C(2′)	114·1 (2)	115·1 (3)
C(1′)—C(2′)—C(3′)	102·6 (2)	110·8 (3)
C(2′)—C(3′)—C(4′)	102·2 (2)	110·8 (3)
C(3′)—C(4′)—O(1′)	106·0 (2)	103·8 (3)
C(3′)—C(4′)—C(5′)	116·5 (2)	113·3 (3)
C(5′)—C(4′)—O(1′)	108·6 (2)	110·5 (3)
C(4′)—C(5′)—O(5′)	109·9 (2)	111·2 (3)

	(1)	(2)
χ [O(1′)—C(1′)—C(2)—S]	14·1 (2)	5·2 (3)
κ [N(6)—C(6)—C(4)—N(3)]	5·3 (3)	6·8 (4)
φ [O(5′)—C(5′)—C(4′)—C(3′)]	288·6 (3)	183·4 (2)

Table 4. *Hydrogen-bond distances and angles*

D—H···*A*	*D*···*A* (Å)	H···*A* (Å)	*D*—H···*A* (°)	Symmetry of acceptor *A*
(1) Dideoxytiazofurin				
N(6)—H′(N6)···O(1′)	2·963 (3)	2·13 (4)	160 (4)	$\frac{1}{2}-x, \frac{1}{2}+y, 1-z$
N(6)—H(N6)···O(5′)	2·906 (3)	2·13 (4)	156 (4)	$\frac{1}{2}+x, \frac{1}{2}+y, z$
O(5′)—H(O5′)···O(*W*)	2·742 (3)	1·89 (3)	171 (3)	x, y, z
O(*W*)—H(O*W*)···O(6)	2·678 (2)	1·72 (4)	153 (5)	$\frac{1}{2}-x, \frac{1}{2}+y, -z$
(2) Didehydrodideoxytiazofurin				
O(5′)—H(O5′)···O(6)	2·725 (3)	1·90 (3)	176 (3)	$\frac{1}{2}-x, -y, \frac{1}{2}+z$
N(6)—H(N6)···O(5′)	2·883 (3)	2·00 (3)	170 (3)	$\frac{1}{2}+x, \frac{1}{2}-y, -z$
N(6)—H′(N6)···O(1′)	2·959 (4)	2·02 (5)	160 (4)	$\frac{1}{2}-x, 1-y, -\frac{1}{2}+z$



(1)

(2)

Fig. 2. Crystal packing of dideoxytiazofurin (1) and didehydrodideoxytiazofurin (2). Dashed lines represent hydrogen bonds.

this feature results from a true intramolecular S···O interaction rather than packing forces. Quantum chemical calculations (Burling & Goldstein, 1989) reveal a positive charge on the S atom of the thiazole ring and a negative charge on the O(1′) atom. A net attractive electrostatic interaction exists between the oppositely charged thiazole S and ribose O atoms. In addition, the calculations show that the thiazole N is negatively charged, creating a repulsive electrostatic interaction with the ribose O atom.

Thiazole rings in both compounds are planar. Bond lengths and bond angles in the two thiazole rings are similar and are within two standard deviations of the mean of those seen in six previously determined structures of thiazole nucleosides. In each structure, the thiazole S—C(2) and S—C(5) bond lengths are shorter than single S—C(sp^2) bond lengths in thiazolidine and thiazolidinone structures which show little electron delocalization (Adman, Jensen & Warrener, 1975; Espenbetov, Yanovskii, Struchkov, Tsoi & Cholpankulova, 1981). This finding is consistent with computational results which show that the thiazole S atom exhibits conjugation with the flanking C atoms (Burling & Goldstein, 1989).

Carboxamide groups in both structures are rotated slightly with respect to the thiazole ring plane. The torsion angle κ [N(6)—C(6)—C(4)—N(3)] is 5·3 (3) and 6·8 (4)° for structures (1) and (2) respectively. There is some bending out of the N(6)—C(6)—O(6) plane by the amino H atoms. As in all previous thiazole nucleoside structures, the carboxamide N atom in (1) and (2) is located *cis* to the thiazole ring N(3) atom and *trans* to C(5). In this configuration the carboxamide group avoids an unfavorable steric interaction between the carboxamide H′(N6) and the thiazole ring H(C5) atoms. At the same time, any potentially favorable electrostatic interactions between the carboxamide O and thiazole H(C5) atoms and between the thiazole N(3) and the carboxamide H′(N6) atoms are optimized.

Furanose ring bond lengths and angles in (1) are similar to those seen in the structures of 2′,3′-dideoxyadenosine and 2′,3′-dideoxycytidine (Silverton, Quinn, Haugwitz & Todaro, 1988). Bond lengths and angles in (2) are within two standard deviations of four structures having similar unsaturated pentafuranose rings without substituents on the 2′ and 3′ C atoms (Criado, Conde & Márquez, 1985; Moreno, López-Castro & Márquez, 1985; Shiro, Nakai, Tori, Nishikawa, Yoshimura & Katagiri, 1980; Hutcheon & James, 1974). The amplitude τ_m and phase angle P of pseudorotation (Altona & Sundaralingam, 1972) for compound (1) are 36·2 and −171·6° respectively. Compound (1) thus assumes a C(2′)-*endo*–C(3)′-*exo* (${}_3T^2$) pucker. The presence of the double bond in (2) constrains the furanose ring to be approximately planar. The largest deviation from the least-squares plane for this ring is 0·0137 (4) Å by the C(3′) atom. If the concept of pseudorotation is applied to the unsaturated pentafuranose ring, compound (2) exhibits a slight twist pucker ($\tau_m = 2{\cdot}6°$, $P = -143{\cdot}7°$). Conformations about the C(4′)—C(5′) bonds are *trans–gauche* and *gauche–trans* for (1) and (2) respectively.

Packing interactions are listed in Table 4 and illustrated in Fig. 2. The only close intermolecular contacts are those resulting from hydrogen bonding. In (1), a water of hydration sits on the twofold axis, accepting and donating H bonds to two symmetry-related pairs of molecules.

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Structure of Dimethyl Dibenzobarrelene-11,12-dicarboxylate

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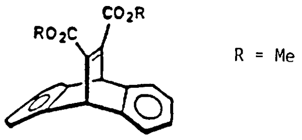
Abstract. Dimethyl 9,10-dihydro-9,10-ethenoanthracene-11,12-dicarboxylate, $C_{20}H_{16}O_4$, $M_r = 320{\cdot}35$, triclinic, $P\bar{1}$, $a = 8{\cdot}699\,(2)$, $b = 12{\cdot}992\,(3)$, $c = 8{\cdot}190\,(2)$ Å, $\alpha = 100{\cdot}78\,(1)$, $\beta = 111{\cdot}72\,(1)$, $\gamma = 97{\cdot}01\,(1)°$, $V = 825{\cdot}8\,(3)$ Å^3, $Z = 2$, $D_x = 1{\cdot}289$ g cm^{-3}, Mo $K\alpha_1$, $\lambda = 0{\cdot}70930$ Å, $\mu = 0{\cdot}84$ cm^{-1}, $F(000) = 336$, $T = 295$ K, $R = 0{\cdot}043$ for 2599 unique observed reflections. The two ester groups have different orientations, C=C—C=O torsion angles 115·7 (2) and 164·2 (2)°, and hence different amounts of conjugation with the C=C double bond [cos²(angle) = 0·19 (nearly non-conjugated) and 0·93 (conjugated)].

Introduction. As part of a crystal and photochemical study of the di-π-methane rearrangement of dibenzobarrelene derivatives (Garcia-Garibay, Scheffer, Trotter & Wireko, 1990), the structure of the symmetrical dimethyl ester has now been determined.



Experimental. Crystal from methanol, approximate dimensions 0·30 × 0·35 × 0·25 mm. Nonius CAD-4F diffractometer, monochromatized Mo $K\alpha$ radiation, lattice parameters from 25 reflections with $\theta = 16$–$22°$. Intensities for $\theta \leq 27{\cdot}5°$, *hkl*: 0 to 11, −16 to 16, −10 to 9; ω–2θ scan, ω-scan width $(0{\cdot}65 + 0{\cdot}35\tan\theta)°$ at 1·1–10° min^{-1}, extended 25% on each side for background measurement; three standard reflections showed no significant variation. Lp but no absorption corrections. 3795 unique reflections, 2599

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