benzamide (Blake & Small, 1972): (*a*) N(1)–H(N2)···O(1)(1–*x*, –*y*, 2–*z*) [N···O 2·950 (4), H···O 2·02 (3) Å, N–H···O 170 (3)°]; (*b*) N(1)–H(N1)···O(1)(*x*, 1+*y*, *z*) [N···O 2·930 (4), H···O 2·09 (3) Å, N–H···O 160 (1)°].

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### References

ASHIDA, T. (1973). *HBLS*-V and *DAPH. The Universal Crystallographic Computing System, Osaka.* The Computation Center, Osaka Univ., Japan.

BLAKE, C. C. F. & SMALL, R. W. H. (1972). *Acta Cryst.* B**28**, 2201–2206.

FUJII, S. (1979). *MOLCON. The Universal Crystallographic Computing System, Osaka.* The Computation Center, Osaka Univ., Japan.

*International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

IWAMOTO, T., KASHINO, S. & HAISA, M. (1989). *Acta Cryst.* C**45**. In the press.

JOHNSON, C. K. (1971). *ORTEP*II. Report ORNL-3794, revised. Oak Ridge National Laboratory, Tennessee, USA.

KASHINO, S., OKA, H. & HAISA, M. (1989). *Acta Cryst.* C**45**, 154–157.

SAKURAI, T. (1967). *RSSFR-5. The Universal Crystallographic Computing System* (I). Tokyo: The Crystallographic Society of Japan.

SCHMIDT, G. M. J. (1964). *J. Chem. Soc.* pp. 2014–2021.

*Acta Cryst.* (1989). C**45**, 1112–1113

# Structure of 6-Amino-1,6-dideoxy-1-[3,4-dihydro-3-methyl-2,4-dioxo-1(2*H*)-pyrimidinyl]-4-thio-L-*glycero*-α-L-*ido*-heptofuranuronic Acid Monohydrate

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**Abstract.** $C_{12}H_{17}N_3O_7S.H_2O$, $M_r = 365{\cdot}36$, monoclinic, $P2_1$, $a = 15{\cdot}105\ (2)$, $b = 7{\cdot}915\ (1)$, $c = 6{\cdot}451\ (1)$ Å, $\beta = 91{\cdot}29\ (1)°$, $V = 771{\cdot}1$ Å$^3$, $Z = 2$, $D_x = 1{\cdot}574$ g cm$^{-3}$, $\lambda(\text{Cu } K\alpha) = 1{\cdot}5418$ Å, $\mu = 22{\cdot}806$ cm$^{-1}$, $F(000) = 384$, $T = 298$ K, final $R = 0{\cdot}062$ for 1665 unique reflections [$F_o^2 > 2\sigma(F_o^2)$]. The thiofuranosyl ring adopts an envelope conformation ($E_3$). The pyrimidyl moiety of the title compound is in a +*anti* orientation ($\chi = -133{\cdot}5°$), in contrast to the methyl ester of the tetraacetylsulfoxide derivative which adopts a +*syn* orientation ($\chi = 67{\cdot}7°$).

**Experimental.** Colorless prisms of title compound were grown from an aqueous ethanol solution [$H_2O$/$C_2H_5OH = 1:1$ (v/v)] at 277 K. Crystal size 0·38 × 0·20 × 0·18 mm, Enraf–Nonius CAD-4 diffractometer, Cu $K\alpha$ radiation, graphite monochromator, $\theta$–$2\theta$

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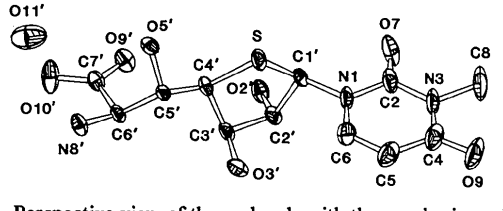


Fig. 1. Perspective view of the molecule with the numbering scheme. To show the absolute configuration correctly, the figure was drawn in the left-handed coordinate system.

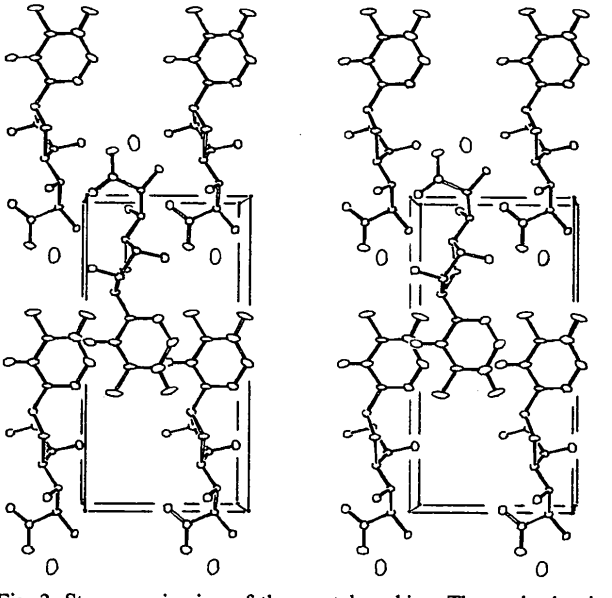

Fig. 2. Stereoscopic view of the crystal packing. The projection is down [001] and the *b* axis is horizontal.

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Table 1. *Final fractional coordinates and equivalent isotropic temperature factors for non-H atoms with e.s.d.'s in parentheses*

$B_{eq} = \frac{4}{3}\sum_i\sum_j B_{ij}\mathbf{a}_i.\mathbf{a}_j$.

| | x | y | x | $B_{eq}$(Å$^2$) |
|---|---|---|---|---|
| N(1) | 0·6099 (3) | 0·7270 (9) | 0·2690 (8) | 2·3 (1) |
| C(2) | 0·5326 (4) | 0·822 (1) | 0·251 (1) | 2·6 (1) |
| N(3) | 0·4546 (3) | 0·726 (1) | 0·2516 (9) | 3·0 (1) |
| C(4) | 0·4504 (5) | 0·554 (1) | 0·263 (1) | 3·3 (2) |
| C(5) | 0·5330 (5) | 0·466 (1) | 0·274 (1) | 3·3 (2) |
| C(6) | 0·6091 (5) | 0·555 (1) | 0·278 (1) | 2·7 (1) |
| O(7) | 0·5330 (3) | 0·9733 (9) | 0·234 (1) | 3·8 (1) |
| C(8) | 0·3701 (5) | 0·824 (2) | 0·234 (2) | 4·8 (2) |
| O(9) | 0·3776 (3) | 0·483 (1) | 0·2589 (9) | 4·6 (1) |
| C(1′) | 0·6938 (4) | 0·822 (1) | 0·2668 (9) | 1·9 (1) |
| C(2′) | 0·7421 (4) | 0·8018 (9) | 0·0530 (9) | 1·8 (1) |
| C(3′) | 0·8260 (4) | 0·6974 (9) | 0·091 (1) | 1·9 (1) |
| C(4′) | 0·8651 (4) | 0·757 (1) | 0·2973 (9) | 1·9 (1) |
| C(5′) | 0·9421 (4) | 0·6585 (9) | 0·3871 (9) | 1·6 (1) |
| C(6′) | 1·0230 (4) | 0·6657 (9) | 0·2410 (9) | 1·7 (1) |
| C(7′) | 1·0639 (4) | 0·8408 (9) | 0·2091 (9) | 1·8 (1) |
| N(8′) | 1·0931 (4) | 0·5527 (8) | 0·3244 (9) | 2·1 (1) |
| O(2′) | 0·7666 (3) | 0·9663 (7) | −0·0200 (7) | 2·22 (9) |
| O(3′) | 0·8046 (3) | 0·5237 (7) | 0·1016 (7) | 2·36 (9) |
| O(5′) | 0·9684 (3) | 0·7293 (7) | 0·5835 (6) | 2·03 (8) |
| O(9′) | 1·0128 (3) | 0·9617 (7) | 0·1732 (7) | 2·36 (9) |
| O(10′) | 1·1471 (3) | 0·8489 (8) | 0·2219 (9) | 3·5 (1) |
| O(11′) | 1·1878 (5) | 0·6961 (9) | 0·679 (1) | 4·7 (2) |
| S | 0·77011 (9) | 0·752 | 0·4719 (2) | 2·23 (3) |

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

| | | | |
|---|---|---|---|
| N(1)–C(2) | 1·392 (9) | C(2′)–C(3′) | 1·528 (9) |
| N(1)–C(6) | 1·366 (11) | C(2′)–O(2′) | 1·436 (8) |
| N(1)–C(1′) | 1·472 (8) | C(3′)–C(4′) | 1·516 (9) |
| C(2)–N(3) | 1·403 (10) | C(3′)–O(3′) | 1·413 (9) |
| C(2)–O(7) | 1·202 (12) | C(4′)–C(5′) | 1·504 (9) |
| N(3)–C(4) | 1·367 (14) | C(4′)–S | 1·844 (6) |
| N(3)–C(8) | 1·498 (11) | C(5′)–C(6′) | 1·560 (8) |
| C(4)–C(5) | 1·427 (11) | C(5′)–O(5′) | 1·433 (7) |
| C(4)–O(9) | 1·234 (10) | C(6′)–C(7′) | 1·534 (10) |
| C(5)–C(6) | 1·347 (11) | C(6′)–N(8′) | 1·478 (8) |
| C(1′)–C(2′) | 1·583 (9) | C(7′)–O(9′) | 1·248 (8) |
| C(1′)–S | 1·822 (6) | C(7′)–O(10′) | 1·259 (8) |
| | | | |
| C(2)–N(1)–C(6) | 122·4 (6) | C(1′)–C(2′)–O(2′) | 108·8 (5) |
| C(2)–N(1)–C(1′) | 116·3 (7) | C(3′)–C(2′)–O(2′) | 108·9 (5) |
| C(6)–N(1)–C(1′) | 121·2 (6) | C(2′)–C(3′)–C(4′) | 106·3 (5) |
| N(1)–C(2)–N(3) | 114·2 (8) | C(2′)–C(3′)–O(3′) | 110·1 (5) |
| N(1)–C(2)–O(7) | 122·7 (6) | C(4′)–C(3′)–O(3′) | 110·2 (6) |
| N(3)–C(2)–O(7) | 123·1 (7) | C(3′)–C(4′)–C(5′) | 117·4 (6) |
| C(2)–N(3)–C(4) | 125·5 (6) | C(3′)–C(4′)–S | 103·6 (4) |
| C(2)–N(3)–C(8) | 115·6 (8) | C(5′)–C(4′)–S | 111·1 (4) |
| C(4)–N(3)–C(8) | 118·9 (7) | C(4′)–C(5′)–C(6′) | 111·0 (5) |
| N(3)–C(4)–C(5) | 116·5 (7) | C(4′)–C(5′)–O(5′) | 109·5 (5) |
| N(3)–C(4)–O(9) | 119·6 (8) | C(6′)–C(5′)–O(5′) | 108·4 (5) |
| C(5)–C(4)–O(9) | 124· (1) | C(5′)–C(6′)–C(7′) | 115·9 (5) |
| C(4)–C(5)–C(6) | 119·6 (9) | C(5′)–C(6′)–N(8′) | 108·8 (5) |
| N(1)–C(6)–C(5) | 121·8 (7) | C(7′)–C(6′)–N(8′) | 107·9 (5) |
| N(1)–C(1′)–C(2′) | 111·8 (5) | C(6′)–C(7′)–O(9′) | 117·9 (5) |
| N(1)–C(1′)–S | 111·6 (5) | C(6′)–C(7′)–O(10′) | 116·2 (6) |
| C(2′)–C(1′)–S | 107·8 (4) | O(9′)–C(7′)–O(10′) | 125·8 (7) |
| C(1′)–C(2′)–C(3′) | 108·1 (5) | C(1′)–S–C(4′) | 92·2 (3) |

scan with scan speed 0·78–5·49° min$^{-1}$ in $\theta$, scan width (0·50 × 0·14tan$\theta$)°. Range of indices, $-18 \le h \le 18$, $0 \le k \le 9$, $0 \le l \le 8$ ($2\theta < 150°$). Lattice constants determined based on 25 $2\theta$ values ($30 < \theta < 74°$). Variation of standard < 0·1%; 1699 reflections measured; 1665 observed reflections with $F_o^2 > 2\sigma(F_o^2)$. Systematic absences $0k0$, $k$ odd. No corrections for absorption. Structure solved by direct methods with *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Refined by full-matrix least squares. The locations of 17 H atoms were found on difference Fourier maps. Non-H atoms refined with anisotropic thermal parameters, and 9 H atoms with fixed isotropic thermal parameters ($B = 5·0$ Å$^2$). $\sum w(|F_o| - |F_c|)^2$ minimized; $w = 1·0$ for $|F_o| < 72·49$, $w = (72·49/F_o)^2$ for $|F_o| \ge 72·49$. Final $R = 0·062$, $wR = 0·072$, $S = 17·1$ for 254 variables, secondary-extinction factor $g = 4·2\,(3) \times 10^{-6}$ [$|F_o| = |F_c|/(1 + gI_c)$]; $\Delta/\sigma < 0·11$ for non-H atoms, largest peak in final $\Delta F$ map +0·38 e Å$^{-3}$; atomic scattering factors from *International Tables for X-ray Crystallography* (1974); programs: Enraf–Nonius *SDP* (Frenz, 1984), *ORTEP*II (Johnson, 1976). The structure of the title compound is shown in Fig. 1 and the crystal packing in Fig. 2. Positional parameters and equivalent values of the anisotropic temperature factors are given in Table 1, bond distances and angles are listed in Table 2.*

**Related literature.** The title compound is one of the enzymatic hydrolyzation products from the antibiotic albomycin (Benz, 1984), and its absolute configuration was determined by X-ray crystal structure analysis of the methyl ester of the tetraacetylsulfoxide derivative (Benz, Born, Brieden, Grosser, Kurz, Paulsen, Sinnwell & Weber, 1984).

* Lists of anisotropic thermal parameters, H-atom coordinates, torsion angles, least-squares planes and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51788 (11 pp). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References


BENZ, G. (1984). *Justus Liebigs Ann. Chem.* pp. 1399–1407.

BENZ, G., BORN, L., BRIEDEN, M., GROSSER, R., KURZ, J., PAULSEN, H., SINNWELL, V. & WEBER, B. (1984). *Justus Liebigs Ann. Chem.* pp. 1408–1423.

FRENZ, B. A. (1984). *Enraf–Nonius Structure Determination Package.* College Station, Texas, USA, and Enraf–Nonius, Delft, The Netherlands.

*International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.