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# Structure of 1-(5-*O*-Sulfamoyl-β-D-ribofuranosyl)-1*H*-1,2,4-triazole-3-carbonitrile

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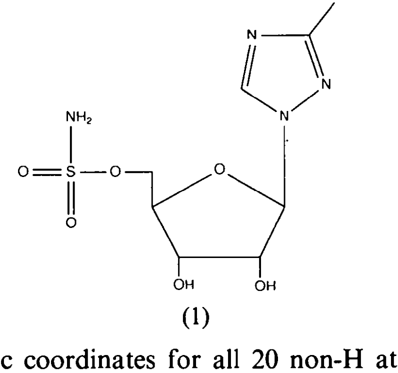
**Abstract.** $C_8H_{11}N_5O_6S$, $M_r = 305{\cdot}26$, orthorhombic, $P2_12_12_1$, $a = 6{\cdot}2993\,(13)$, $b = 7{\cdot}8161\,(13)$, $c = 25{\cdot}420\,(5)$ Å, $V = 1251{\cdot}6\,(4)$ Å$^3$, $Z = 4$, $D_x = 1{\cdot}620$ g cm$^{-3}$, Cu $K\alpha$ ($\lambda = 1{\cdot}54178$ Å), $\mu = 26{\cdot}195$ cm$^{-1}$, $F(000) = 632$, $T = 295$ K, $R = 0{\cdot}0282$ for 2394 reflections ($F \geq 4\sigma_F$). The sugar conformation and puckering parameters are $_3T^2$ (C3′*exo*/C2′-*endo*), $P = 192{\cdot}2°$ and $\tau_m = 38{\cdot}0°$. The C5′—O5′ side-chain orientation is *gauche–trans* [$\varphi_{OO} = 59{\cdot}8\,(2)$; $\varphi_{CO} = 176{\cdot}96\,(15)°$]. The glycosylic torsion angle (N2—N1—C1′—O4′) is 64·2 (2)°. The triazole ring is planar [r.m.s. deviation: 0·001 (2) Å]. The C3—C6—N7 group is linear and is 1·4° out of the triazole plane. All possible hydrogen donors (four) participate in hydrogen bonding [range of H···*A* distances is 2·14 (3)–2·40 (3) Å]. The sulfonamide amino group, which is above the furanose ring, is slightly nonplanar. The base moieties are perpendicular to the *a* axis and base stacking occurs *via* sandwiching of the carbonitrile groups between triazole rings of adjacent molecules along the $2_1$ axes parallel to the *a* axis.

**Experimental.** The title compound (1) was prepared from 1-β-D-ribofuranosyl-1*H*-1,2,4-triazole-3-carbonitrile as previously described (Kini, Henry, Robins, Larson, Marr, Berens, Bacchi, Nathan & Keithly, 1990). Crystals grew from a warm methanol/ether solution as colorless, transparent needles. Table 1 summarizes data collection and refinement. Unit-cell parameters were obtained by least-squares refinement of the setting angles of 25 reflections with $55{\cdot}0 < 2\theta < 59{\cdot}5°$. An Enraf–Nonius CAD-4 diffractometer with a graphite monochromator was used for all measurements. Data reduction, which included Lorentz, polarization, decay and absorption corrections, was accomplished with the *SDP-Plus* software (Frenz, 1985). Crystal and instrument stability were monitored by remeasurement of three check reflections every hour. A linear fit of the intensities of these reflections was used to correct the data. Absorption corrections, based on crystal-face measurements, were applied. The function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w^{-1} = (\sigma_F{}^2 + 0{\cdot}0004F^2)$, $\sigma_F = F\sigma_I/2I$, and $\sigma_I = (N_{pk} + N_{bg1} + N_{bg2})^{1/2}$.



(1)

Atomic coordinates for all 20 non-H atoms were obtained by direct methods [*SHELXS86* (Sheldrick, 1986)]. All H atoms were located in a difference map as peaks of density 0·44–0·91 e Å$^{-3}$ at $R = 0{\cdot}046$. All positional parameters, anisotropic thermal parameters for non-H atoms, isotropic thermal parameters for H atoms and an extinction parameter were refined with *SHELX76* (Sheldrick, 1976). Scattering factors and anomalous-dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974) except those of H which were taken from Stewart, Davidson & Simpson (1965). Least-squares-planes program from Cordes (1983); figures were drawn with *ORTEP*II (Johnson, 1976); parameter and geometry tables were produced with *FUER*

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Table 1. *Summary of data collection and refinement for* (1)

| | |
|---|---|
| (*a*) Data collection (295 K) | |
| Mode | $\omega$–$2\theta$ scan |
| Scan range (°) | 0·80 + 0·15 tan $\theta$ |
| Background | Scan 0·25 times scan range before and after scan |
| Scan rate (° min$^{-1}$) | 1·4–8·25 |
| Exposure time (h) | 35·8 |
| Stability correction range on $I$ | 1·000–1·015 |
| Check reflections | 1,3,11, 24$\bar{3}$, 3$\bar{3}$5 |
| $2\theta$ range (°) | 3·0–152·0 |
| Range in $hkl$, min. | 0,0, −31 |
| max. | 7,9,31 |
| Total reflections, measured, unique | 3015, 2600 |
| $R_{int}$ | 0·0132 |
| Crystal dimensions (mm) | 0·32 × 0·23 × 0·09 |
| Crystal volume (mm$^3$) | 0·00451 |
| Crystal faces | (00$\bar{1}$); ($\bar{1}$00); {011}; (102); (10$\bar{2}$) |
| Transmission-factor range | 0·584–0·828 |
| (*b*) Structure refinement | |
| Reflections used ($F \geq 4\sigma_F$) | 2394 |
| No. of variables | 226 |
| Extinction parameter | 1·07 (9) × 10$^{-6}$ |
| Goodness of fit, $S$ | 1·400 |
| $R$, $wR$ | 0·0282, 0·0414 |
| $R$ for all data | 0·0331 |
| Max., av. $\Delta/\sigma$ | 0·002, 0·0004 |
| Max., min. $\Delta\rho$ in $\Delta F$ map (e Å$^{-3}$) | 0·30, −0·36 |

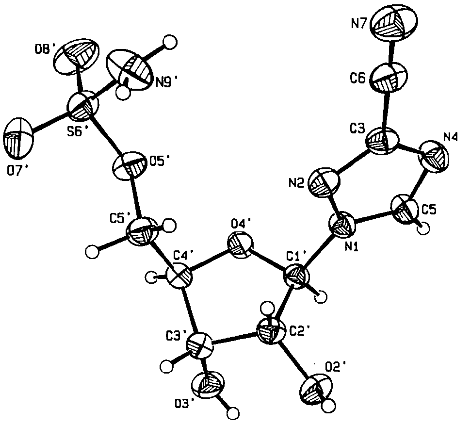

Fig. 1. Thermal-ellipsoid plot of (1) illustrating atom labeling and molecular conformation. N9′ is 0·116 (2) Å out of the plane of S6′, H9′*A* and H9′*B*. The ellipsoids are drawn at the 50% probability level.

and structure-factor-amplitude tables were produced with *LISTFC* (Larson, 1980). The atomic coordinates are listed in Table 2. Bond lengths and bond angles are given in Table 3. Hydrogen bonding is given in Table 4. Fig. 1 illustrates the atom labeling and molecular conformation; Fig. 2 illustrates the unit-cell packing and hydrogen bonding.*

Table 2. *Positional and isotropic thermal parameters for all atoms in* (1)

For non-H atoms, $U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i{}^*a_j{}^*\mathbf{A}_{ij}$, where $\mathbf{A}_{ij}$ is the dot product of the *i*th and *j*th direct-space unit-cell vectors.

| | $x$ | $y$ | $z$ | $U_{eq}/U_{iso}$(Å$^2$) |
|---|---|---|---|---|
| N1 | 0·5271 (2) | 0·5467 (2) | 0·54287 (5) | 0·0268 (4) |
| N2 | 0·5271 (3) | 0·3768 (2) | 0·55208 (6) | 0·0308 (4) |
| C3 | 0·5160 (3) | 0·3154 (2) | 0·50368 (7) | 0·0295 (4) |
| N4 | 0·5084 (3) | 0·4323 (2) | 0·46437 (6) | 0·0346 (4) |
| C5 | 0·5164 (4) | 0·5776 (2) | 0·49090 (6) | 0·0307 (5) |
| C6 | 0·5171 (3) | 0·1344 (2) | 0·49392 (8) | 0·0351 (5) |
| N7 | 0·5185 (4) | −0·0089 (2) | 0·48633 (8) | 0·0480 (6) |
| C1′ | 0·5369 (3) | 0·6707 (2) | 0·58604 (6) | 0·0263 (4) |
| C2′ | 0·3600 (3) | 0·6452 (2) | 0·62649 (7) | 0·0297 (5) |
| C3′ | 0·4712 (3) | 0·7001 (2) | 0·67732 (6) | 0·0327 (5) |
| C4′ | 0·6936 (3) | 0·6319 (2) | 0·66862 (7) | 0·0305 (5) |
| C5′ | 0·7160 (3) | 0·4464 (3) | 0·68423 (8) | 0·0371 (5) |
| O2′ | 0·1845 (2) | 0·7486 (2) | 0·61366 (6) | 0·0428 (4) |
| O3′ | 0·4856 (3) | 0·8819 (2) | 0·68008 (6) | 0·0449 (4) |
| O4′ | 0·7327 (2) | 0·6534 (2) | 0·61266 (5) | 0·0314 (4) |
| O5′ | 0·9320 (2) | 0·3965 (2) | 0·67112 (6) | 0·0374 (4) |
| S6′ | 1·00249 (8) | 0·21081 (6) | 0·68773 (2) | 0·03833 (14) |
| O7′ | 0·9355 (3) | 0·1822 (2) | 0·74039 (6) | 0·0562 (6) |
| O8′ | 1·2221 (3) | 0·2087 (3) | 0·67448 (8) | 0·0635 (7) |
| N9′ | 0·8808 (4) | 0·0805 (3) | 0·65138 (9) | 0·0562 (7) |
| H5 | 0·521 (4) | 0·685 (3) | 0·4756 (9) | 0·041 (6) |
| H1′ | 0·535 (3) | 0·790 (2) | 0·5696 (7) | 0·026 (5) |
| H2′ | 0·330 (4) | 0·526 (3) | 0·6270 (9) | 0·039 (6) |
| H3′ | 0·404 (4) | 0·650 (3) | 0·7084 (9) | 0·040 (6) |
| H4′ | 0·808 (4) | 0·700 (3) | 0·6866 (8) | 0·038 (6) |
| H5′*A* | 0·613 (4) | 0·375 (3) | 0·6673 (8) | 0·035 (6) |
| H5′*B* | 0·698 (5) | 0·428 (3) | 0·7246 (10) | 0·051 (7) |
| HO2′ | 0·074 (7) | 0·699 (5) | 0·6146 (14) | 0·101 (14) |
| HO3′ | 0·383 (5) | 0·931 (4) | 0·6713 (10) | 0·058 (9) |
| H9′*A* | 0·761 (5) | 0·052 (4) | 0·6649 (10) | 0·061 (9) |
| H9′*B* | 0·915 (6) | 0·073 (4) | 0·6165 (13) | 0·080 (10) |

Table 3. *Bond lengths* (Å) *and bond angles* (°) *in* (1)

| 1 | 2 | 3 | 1—2 | 1—2—3 |
|---|---|---|---|---|
| N2 | N1 | C5 | 1·348 (2) | 110·34 (13) |
| C5 | N1 | C1′ | 1·345 (2) | 128·23 (14) |
| C1′ | N1 | N2 | 1·466 (2) | 121·43 (12) |
| C3 | N2 | N1 | 1·322 (2) | 101·31 (13) |
| N4 | C3 | C6 | 1·355 (2) | 122·5 (2) |
| N4 | C3 | N2 | | 116·29 (14) |
| C6 | C3 | N2 | 1·436 (2) | 121·2 (2) |
| C5 | N4 | C3 | 1·322 (2) | 101·65 (14) |
| N1 | C5 | N4 | | 110·42 (14) |
| N7 | C6 | C3 | 1·137 (2) | 179·7 (4) |
| C2′ | C1′ | O4′ | 1·529 (2) | 107·53 (13) |
| C2′ | C1′ | N1 | | 112·75 (14) |
| O4′ | C1′ | N1 | 1·413 (2) | 109·39 (13) |
| C3′ | C2′ | O2′ | 1·531 (3) | 113·2 (2) |
| C3′ | C2′ | C1′ | | 101·41 (14) |
| O2′ | C2′ | C1′ | 1·408 (2) | 109·97 (15) |
| C4′ | C3′ | O3′ | 1·516 (3) | 107·4 (2) |
| C4′ | C3′ | C2′ | | 101·59 (14) |
| O3′ | C3′ | C2′ | 1·426 (2) | 110·49 (15) |
| C5′ | C4′ | O4′ | 1·510 (3) | 110·66 (15) |
| C5′ | C4′ | C3′ | | 112·7 (2) |
| O4′ | C4′ | C3′ | 1·453 (2) | 104·98 (14) |
| O5′ | C5′ | C4′ | 1·454 (2) | 106·5 (2) |
| C1′ | O4′ | C4′ | | 109·40 (13) |
| S6′ | O5′ | C5′ | 1·5756 (15) | 116·69 (12) |
| O7′ | S6′ | O8′ | 1·421 (2) | 120·64 (12) |
| O7′ | S6′ | N9′ | | 107·85 (12) |
| O7′ | S6′ | O5′ | | 108·27 (10) |
| O8′ | S6′ | N9′ | 1·424 (2) | 109·07 (13) |
| O8′ | S6′ | O5′ | | 102·76 (10) |
| N9′ | S6′ | O5′ | 1·574 (2) | 107·55 (10) |

Table 4. *Hydrogen bonding in* (1)

| $D$— | H··· | $A$ | Symmetry of $A$ relative to $D$ | $d(D\cdots A)$ (Å) | $d(\mathrm{H}\cdots A)$ (Å) | $\angle(D$—H$\cdots A)$ (°) |
|---|---|---|---|---|---|---|
| O2′ | HO2′ | O4′ | $x-1{\cdot}0,y,z$ | 2·942 (2) | 2·18 (4) | 160· (4) |
| O3′ | HO3′ | O2′ | $x,y,z$ | 2·746 (2) | 2·40 (3) | 109· (3) |
| O3′ | HO3′ | O8′ | $x-1{\cdot}0,y+1{\cdot}0,z$ | 3·050 (2) | 2·40 (3) | 141· (3) |
| N9′ | H9′*A* | O3′ | $x,y-1{\cdot}0,z$ | 3·024 (3) | 2·22 (3) | 156· (3) |
| N9′ | H9′*B* | N4 | $0{\cdot}5+x,0{\cdot}5-y,1{\cdot}0-z$ | 3·052 (3) | 2·14 (3) | 177· (3) |

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* Tables of anisotropic thermal parameters, bond lengths and angles involving H atoms, torsion angles, least-squares planes and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52437 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

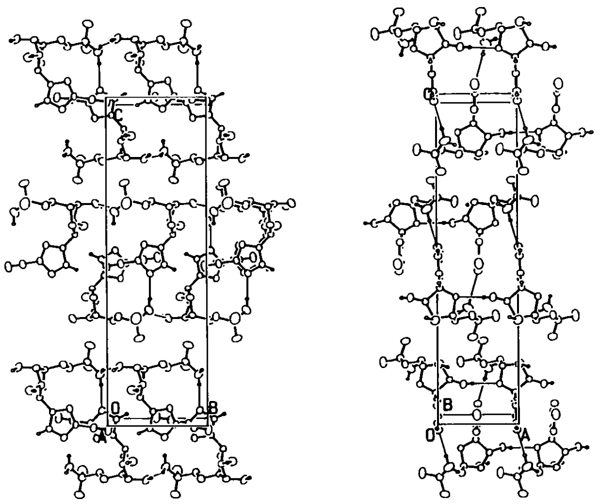
(a) (b)

Fig. 2. Crystal packing diagram of (1) with C—H H atoms of the sugar moiety omitted. Hydrogen bonds are drawn as thin lines. (*a*) View along the *a* axis illustrating the sandwiching of the cyano group between triazole rings of adjacent molecules along the $2_1$ axes parallel to **a**. Hydrogen bonding also occurs between adjacent molecules along this screw axis. (*b*) View along the *b* axis showing how the triazole planes are parallel to the *bc* plane. Hydrogen bonding only occurs between molecules along the *a*-parallel screw axes or translated in the **b** direction.

**Related literature.** The title compound (1) has shown some antiparasitic activity (Kini *et al.*, 1990). The preceding papers report the structures of the carboxamide (Larson, Kini & Robins, 1990) and thiocarboxamide (Larson, Henry, Kini & Robins, 1990) derivatives of (1). In each of these structures the amino group of the sulfamoyl moiety is over the furan ring, regardless of the C5′—O5′ orientation. Several other triazole nucleosides have been reported (Prusiner & Sundaralingam, 1976; Haines, Leonard & Wiemer, 1982; Sanghvi, Hanna, Larson, Fujitaki, Willis, Smith, Robins & Revankar, 1988; Hanna, Dimitrijevich, Larson, Robins & Revankar, 1988). A search of the Cambridge Structural Database (1989) revealed no structures containing the 5-*O*-sulfamoylribose moiety. Conformational parameters follow the conventions of Altona & Sundaralingam (1972).

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# Structure of 2-(4-Methylthien-2-yl)-2,5-dihydro-3*H*-pyrazolo[4,3-*c*]quinolin-3-one

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**Abstract.** $C_{15}H_{11}N_3OS$, $M_r = 281{\cdot}33$, monoclinic, $P2_1/c$, $a = 9{\cdot}972\,(7)$, $b = 9{\cdot}650\,(5)$, $c = 14{\cdot}000\,(3)$ Å, $\beta = 101{\cdot}28\,(3)°$, $V = 1321\,(1)$ Å$^3$, $Z = 4$, $D_x = 1{\cdot}414$ Mg m$^{-3}$, $\lambda(\text{Cu }K\alpha) = 1{\cdot}54178$ Å, $\mu = 2{\cdot}11$ mm$^{-1}$, $F(000) = 584$, $T = 295$ K, $R = 0{\cdot}038$ for 1897 observed reflections $[F_o > 3\sigma(F_o)]$. The molecule is almost planar except for H atoms of the methyl group, but torsion angles N(11)—N(12)—C(15)—

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