

that the exocyclic angles at C(1) and C(2) are smaller for the S derivative than for the O analog; these angles are necessarily smaller, however, because of the larger endocyclic angular requirements of the S atom.

In conclusion, the only distinguishing feature of the two crystal structures which might explain the photochemical behavior is the different C(1)...C(1)' distances. Electronic effects, e.g. charge-transfer processes, may also be involved and this possibility is being explored.

Table 1. *Positional parameters and their estimated standard deviations*

For non-hydrogen atoms  $B_{eq} = \frac{4}{3}[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + ab(\cos\gamma)\beta_{12} + ac(\cos\beta)\beta_{13} + bc(\cos\alpha)\beta_{23}]$ , where  $a$ ,  $b$  and  $c$  are lattice constants. H atoms were refined isotropically.

	$x$	$y$	$z$	$B_{eq}$ or $B(\text{\AA}^2)$
S	-0.0889 (3)	0.15220 (8)	-0.0248 (1)	1.95 (2)
O	0.0616 (9)	0.2980 (2)	0.1151 (3)	3.41 (8)
C(1)	0.037 (1)	0.0396 (3)	0.0313 (3)	1.46 (8)
C(2)	0.225 (1)	0.0511 (3)	0.1511 (3)	1.62 (8)
C(3)	0.374 (1)	-0.0173 (3)	0.2267 (4)	2.1 (1)
C(4)	0.542 (1)	0.0084 (3)	0.3349 (4)	2.3 (1)
C(5)	0.558 (1)	0.1025 (3)	0.3714 (4)	2.3 (1)
C(6)	0.407 (1)	0.1708 (3)	0.2975 (4)	2.3 (1)
C(7)	0.245 (1)	0.1451 (3)	0.1886 (3)	1.74 (8)
C(8)	0.082 (1)	0.2135 (3)	0.1055 (4)	2.13 (9)
H(3)	0.37 (1)	-0.084 (3)	0.206 (3)	2.1 (9)
H(4)	0.63 (1)	-0.035 (3)	0.384 (3)	3 (1)
H(5)	0.70 (1)	0.121 (3)	0.446 (3)	2.2 (9)
H(6)	0.419 (9)	0.231 (3)	0.318 (3)	1.6 (9)

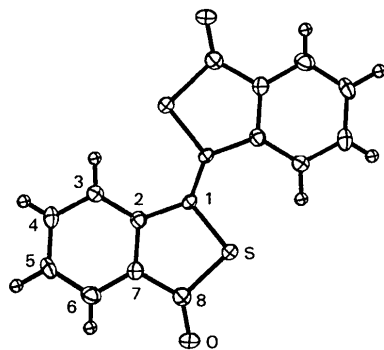


Fig. 1. Drawing of the molecule with the atomic numbering scheme. Spheres for the H atoms correspond arbitrarily to  $B = 1.0 \text{\AA}^2$ .

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### Structure of 1-(5'-O-Acetyl-2',3'-O-isopropylidene- $\beta$ -D-ribofuranosyl)-5-ditosylaminoimidazole-4-carbonitrile, $C_{28}H_{30}N_4O_9S_2$

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**Abstract.**  $M_r = 630.69$ , monoclinic,  $P2_1$ ,  $a = 11.419 (1)$ ,  $b = 13.362 (4)$ ,  $c = 9.677 (2) \text{\AA}$ ,  $\beta = 91.51 (3)^\circ$ ,  $V = 1476.1 (4) \text{\AA}^3$ ,  $Z = 2$ ,  $D_m = 1.401 (4)$ ,

0108-2701/83/111551-03\$01.50

Table 2. *Selected bond distances ( $\text{\AA}$ ) and angles ( $^\circ$ ) with estimated standard deviations*

The four C–H distances vary from 0.88 (4) to 1.00 (3)  $\text{\AA}$ .

S–C(1)	1.772 (4)	C(2)–C(7)	1.401 (5)
S–C(8)	1.793 (4)	C(3)–C(4)	1.389 (5)
O–C(8)	1.207 (4)	C(4)–C(5)	1.397 (6)
C(1)–C(1')	1.349 (7)	C(5)–C(6)	1.378 (5)
C(1)–C(2)	1.494 (4)	C(6)–C(7)	1.386 (5)
C(2)–C(3)	1.388 (5)	C(7)–C(8)	1.459 (5)
C(1)–S–C(8)	94.0 (2)	C(4)–C(5)–C(6)	119.1 (4)
S–C(1)–C(1')	121.4 (4)	C(5)–C(6)–C(7)	119.6 (4)
S–C(1)–C(2)	109.1 (2)	C(2)–C(7)–C(6)	122.1 (4)
C(1')–C(1)–C(2)	129.5 (4)	C(2)–C(7)–C(8)	115.2 (3)
C(1)–C(2)–C(3)	129.1 (4)	C(6)–C(7)–C(8)	122.7 (4)
C(1)–C(2)–C(7)	113.0 (3)	S–C(8)–O	122.3 (3)
C(3)–C(2)–C(7)	117.9 (3)	S–C(8)–C(7)	108.7 (3)
C(2)–C(3)–C(4)	120.1 (4)	O–C(8)–C(7)	129.0 (4)
C(3)–C(4)–C(5)	121.2 (4)		

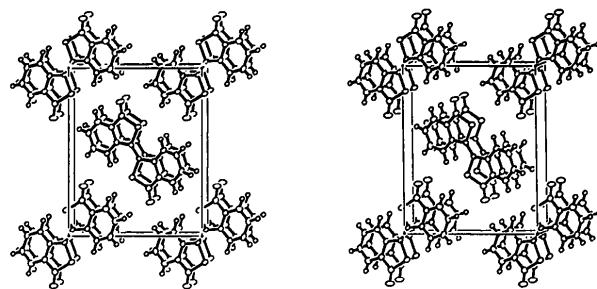


Fig. 2. Stereo packing diagram viewed nearly parallel to the  $a$  axis.

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$D_x = 1.419 \text{Mgm}^{-3}$ ,  $\lambda(\text{Mo K}\alpha) = 0.70924 \text{\AA}$ ,  $\mu = 0.242 \text{mm}^{-1}$ ,  $F(000) = 660$ ,  $T = 293 \text{K}$ . Final  $R = 0.053$  for 2631 observed reflections. The glycosyl

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torsion angle is in the *anti* range,  $\chi = 58.2 (6)^\circ$ , and the ribose ring conformation is C(3')*endo*/C(4')*exo*(<sup>3</sup>T<sub>4</sub>). The N(5)S(1)S(2) and the imidazole planes are not coplanar because of steric hindrance between the cyano group and the tosyl rings.

**Introduction.** The title compound is an intermediate for the chemical synthesis of imidazole cyclonucleosides. Although the present molecule is similar to 5-amino-1- $\beta$ -D-ribofuranosylimidazole-4-carboxamide (AICA-riboside) (Adamiak & Saenger, 1979), the conformation around the C(5)–N(5) bond (see the numbering scheme in Fig. 1) must be opposite because of the bulky tosyl substituents which do not permit coplanarity between the N(5)S(1)S(2) plane and the imidazole ring. The present analysis reveals a structure influenced by bond rotation around C(5)–N(5) which may be related to the cyclization between the imidazole and the sugar ring through the N atom.

**Experimental.** Colorless prismatic crystals from water–ethanol at room temperature; crystal 0.2 × 0.2 × 0.1 mm. Density by flotation. Cell dimensions by least-squares refinement of 19 reflections with 2 $\theta$

Table 1. Final positional and equivalent isotropic thermal parameters for the non-H atoms with their e.s.d.'s in parentheses

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

	x	y	z	B <sub>eq</sub> (Å <sup>2</sup> )
N(1)	0.8080 (4)	0.4842 (3)	0.0476 (4)	3.18 (3)
C(2)	0.8238 (5)	0.5646 (4)	-0.0336 (5)	3.91 (5)
N(3)	0.7729 (4)	0.6461 (3)	0.0105 (4)	4.06 (4)
C(4)	0.7205 (4)	0.6159 (4)	0.1293 (5)	3.29 (4)
C(5)	0.7408 (4)	0.5168 (3)	0.1544 (4)	2.63 (4)
N(5)	0.7075 (3)	0.4555 (3)	0.2636 (4)	2.71 (3)
C(6)	0.6512 (5)	0.6810 (4)	0.2096 (6)	4.12 (5)
N(6)	0.5942 (5)	0.7310 (4)	0.2734 (6)	6.41 (6)
S(1)	0.8070 (1)	0.4450 (1)	0.3977 (1)	3.14 (1)
O(11)	0.7624 (4)	0.3685 (3)	0.4839 (4)	4.48 (4)
O(12)	0.9162 (3)	0.4327 (3)	0.3322 (4)	4.33 (3)
C(11)	0.8056 (4)	0.5599 (4)	0.4830 (4)	2.92 (4)
C(12)	0.7307 (5)	0.5741 (4)	0.5904 (5)	3.60 (4)
C(13)	0.7293 (4)	0.6646 (4)	0.6550 (5)	3.81 (5)
C(14)	0.8013 (5)	0.7428 (4)	0.6134 (5)	3.96 (4)
C(15)	0.8749 (5)	0.7269 (4)	0.5058 (5)	4.12 (5)
C(16)	0.8791 (4)	0.6355 (4)	0.4406 (5)	3.63 (4)
C(17)	0.7961 (6)	0.8434 (5)	0.6866 (8)	6.01 (7)
S(2)	0.5632 (1)	0.4227 (1)	0.2763 (1)	3.08 (1)
O(21)	0.5094 (3)	0.4664 (3)	0.1574 (4)	4.37 (4)
O(22)	0.5288 (3)	0.4486 (3)	0.4105 (4)	4.50 (3)
C(21)	0.5623 (4)	0.2921 (3)	0.2572 (5)	3.22 (4)
C(22)	0.5583 (5)	0.2517 (4)	0.1258 (5)	3.81 (5)
C(23)	0.5542 (5)	0.1495 (4)	0.1112 (5)	4.17 (5)
C(24)	0.5563 (4)	0.0875 (4)	0.2256 (6)	3.85 (5)
C(25)	0.5610 (5)	0.1294 (4)	0.3561 (6)	4.09 (5)
C(26)	0.5618 (5)	0.2319 (4)	0.3737 (5)	3.62 (4)
C(27)	0.5497 (6)	-0.0249 (5)	0.2084 (8)	6.12 (8)
C(1')	0.8531 (4)	0.3838 (4)	0.0251 (5)	3.33 (4)
O(1')	0.9750 (4)	0.3893 (3)	0.0191 (4)	4.70 (4)
C(2')	0.8067 (4)	0.3368 (4)	-0.1103 (5)	3.48 (4)
O(2')	0.7828 (3)	0.2343 (3)	-0.0833 (4)	4.54 (4)
C(3')	0.9123 (4)	0.3304 (4)	-0.2015 (5)	3.50 (4)
O(3')	0.9033 (3)	0.2375 (3)	-0.2670 (4)	4.35 (3)
C(4')	1.0153 (4)	0.3377 (4)	-0.1001 (5)	3.73 (4)
C(5')	1.1240 (5)	0.3898 (5)	-0.1480 (7)	4.91 (6)
O(5')	1.0992 (4)	0.4933 (3)	-0.1750 (5)	5.63 (4)
C(6')	1.1269 (6)	0.5336 (6)	-0.2929 (7)	5.68 (7)
O(6')	1.1614 (8)	0.4858 (6)	-0.3861 (6)	11.4 (1)
C(7')	1.1166 (6)	0.6446 (6)	-0.2916 (9)	6.56 (9)
C(8')	0.8145 (5)	0.1786 (4)	-0.2021 (5)	3.78 (5)
C(9')	0.8654 (7)	0.0817 (5)	-0.1544 (7)	6.5 (1)
C(10')	0.7137 (6)	0.1665 (6)	-0.3023 (7)	5.81 (8)

= 12–29°. Rigaku–Denki AFC-4 automated diffractometer, Zr-filtered Mo K $\alpha$  radiation, 2 $\theta_{\text{max}} = 55^\circ$ ,  $\theta$ –2 $\theta$  scan technique. Of 3564 independent reflections,  $0 \leq h \leq 14$ ,  $0 \leq k \leq 5$ ,  $0 \leq l \leq 9$ , 2631 had  $|F_o| > 3\sigma(F_o)$ . Three standard reflections (300, 040, 002) monitored after every 50 reflections showed 0.4 (3)% intensity fluctuation throughout data collection. Intensities corrected for Lorentz and polarization factors but not for absorption. Direct methods (MULTAN78, Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Positional and anisotropic thermal parameters for all non-H atoms refined by block-diagonal least squares (HBL5V, Ashida, 1979).  $w(|F_o| - k|F_c|)^2$  minimized with  $w = [\sigma^2(F_o) + 0.0114|F_o| + 0.0002|F_o|^2]^{-1}$ . All H atoms located on a difference Fourier map, their positional parameters included in refinement with a fixed isotropic thermal parameter of 4.0 Å<sup>2</sup>. Final R and wR for 2631 reflections = 0.053 and 0.049 respectively, S for 479 parameters (nine for each non-H atom, four for each H atom, an overall scale and a thermal parameter) = 1.139.  $\Delta/\sigma_{\text{av}} = 0.008$ ,  $\Delta/\sigma_{\text{max}} = 0.056$ .  $\Delta\rho = -0.25 - 0.26 \text{ e \AA}^{-3}$ , no other significant features observed. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Calculations performed on an ACOS 900 computer at the Crystallographic Research Center, Institute for Protein Research, Osaka University.

**Discussion.** Final atomic coordinates for non-H atoms with their e.s.d.'s are given in Table 1, and bond distances and angles in Table 2.\* A perspective view of the molecule is shown in Fig. 1. The bond angles around N(5) indicate that the nitrogen orbital is approximately sp<sup>2</sup> hybridized. The observed dihedral angles [N(1)–C(5)–N(5)–S(1) = 84.9 (5) and N(1)–C(5)–N(5)–S(2) = -111.1 (4)°], however, suggest that the N(5) lone-pair axis is nearly perpendicular to the normal of the imidazole plane. This geometry leads to a notably poor  $\pi$  bond between N(5) and the imidazole ring system. This is consistent with the C(5)–N(5) bond distance (1.398 Å) which is considerably longer than the corresponding bonds (1.355 and 1.354 Å) in AICA-riboside and its 5'-(dihydrogen phosphate) (Adamiak & Saenger, 1979). The cyano fragment C(4)–C(6)–N(6) is significantly non-linear (178.4°) as seen in structures of tetracyanoethylene oxide (Matthews, Swanson, Mueller & Stucky, 1971) and 3-(2-methoxyphenyl)-1,1,2-cyclopropanetetra-carbonitrile (Usha & Venkatesan, 1980).

The glycosyl torsion angle C(2)–N(1)–C(1')–O(1') is in the *anti* conformation [ $\chi = 58.2 (6)^\circ$ ]. The

\* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38727 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

furanose having the fused isopropylidene ring is in the almost perfect twist conformation,  $C(3')endo/C(4')exo$ , with  $P = 37.0^\circ$  and  $\tau_m = 24.6^\circ$  defined by Altona & Sundaralingam (1972). This is opposite to the  $C(3')exo/C(4')endo$  ( ${}^4T_3$ ) conformation observed in 2',3'-*O*-isopropylideneadenosine,  $P = 215.3^\circ$  (Sprang, Rohrer & Sundaralingam, 1978), 2',3'-*O*-isopropylideneuridine,  $P = 216.3^\circ$  (Katti, Seshadri & Viswamitra, 1981) and 2',3'-*O*-isopropylidene-5-bromouridine,  $P = 211.0^\circ$  (Yamagata, Kobayashi, Okabe, Tomita & Ueda, 1983) or  $P = 216.6^\circ$  (Gautham, Seshadri & Viswamitra, 1983).

The crystal structure is stabilized only by van der Waals contacts, having no hydrogen bonding or base stacking (see Fig. 2).

Table 2. Bond distances (Å) and angles ( $^\circ$ ) for the non-H atoms

N(1)–C(2)	1.346 (7)	N(1)–C(5)	1.374 (6)
N(1)–C(1')	1.455 (7)	C(2)–N(3)	1.311 (7)
N(3)–C(4)	1.371 (7)	C(4)–C(5)	1.365 (7)
C(4)–C(6)	1.421 (8)	C(5)–N(5)	1.398 (6)
N(5)–S(1)	1.708 (4)	N(5)–S(2)	1.713 (4)
C(6)–N(6)	1.128 (8)	S(1)–O(11)	1.422 (4)
S(1)–O(12)	1.423 (4)	S(1)–C(11)	1.743 (5)
C(11)–C(12)	1.376 (7)	C(11)–C(16)	1.383 (7)
C(12)–C(13)	1.362 (8)	C(13)–C(14)	1.395 (8)
C(14)–C(15)	1.372 (8)	C(14)–C(17)	1.521 (9)
C(15)–C(16)	1.376 (8)	S(2)–O(21)	1.416 (4)
S(2)–O(22)	1.410 (4)	S(2)–C(21)	1.755 (5)
C(21)–C(22)	1.381 (7)	C(21)–C(26)	1.385 (7)
C(22)–C(23)	1.374 (8)	C(23)–C(24)	1.382 (8)
C(24)–C(25)	1.381 (8)	C(24)–C(27)	1.51 (1)
C(25)–C(26)	1.380 (8)	C(1')–O(1')	1.397 (7)
C(1')–C(2')	1.535 (7)	O(1')–C(4')	1.430 (7)
C(2')–O(2')	1.422 (6)	C(2')–C(3')	1.515 (7)
O(2')–C(8')	1.424 (7)	C(3')–O(3')	1.396 (6)
C(3')–C(4')	1.515 (7)	O(3')–C(8')	1.441 (7)
C(4')–C(5')	1.507 (8)	C(5')–O(5')	1.434 (8)
O(5')–C(6')	1.308 (9)	C(6')–O(6')	1.18 (1)
C(6')–C(7')	1.49 (1)	C(8')–C(9')	1.49 (1)
C(8')–C(10')	1.49 (1)		
C(2)–N(1)–C(5)	105.8 (4)	C(2)–N(1)–C(1')	126.5 (4)
C(5)–N(1)–C(1')	127.7 (4)	N(1)–C(2)–N(3)	113.9 (5)
C(2)–N(3)–C(4)	103.6 (4)	N(3)–C(4)–C(5)	111.0 (4)
N(3)–C(4)–C(6)	122.7 (5)	C(5)–C(4)–C(6)	126.2 (5)
N(1)–C(5)–C(4)	105.6 (4)	N(1)–C(5)–N(5)	123.5 (4)
C(4)–C(5)–N(5)	130.9 (4)	C(5)–N(5)–S(1)	115.7 (3)
C(5)–N(5)–S(2)	119.1 (3)	S(1)–N(5)–S(2)	123.1 (2)
C(4)–C(6)–N(6)	178.4 (6)	N(5)–S(1)–O(11)	105.3 (2)
N(5)–S(1)–O(12)	104.1 (2)	N(5)–S(1)–C(11)	105.9 (2)
O(11)–S(1)–O(12)	120.6 (3)	O(11)–S(1)–C(11)	110.3 (2)
O(12)–S(1)–C(11)	109.4 (2)	S(1)–C(11)–C(12)	119.5 (4)
S(1)–C(11)–C(16)	119.3 (4)	C(12)–C(11)–C(16)	121.2 (5)
C(11)–C(12)–C(13)	119.0 (5)	C(12)–C(13)–C(14)	121.2 (5)
C(13)–C(14)–C(15)	118.7 (5)	C(13)–C(14)–C(17)	119.8 (5)
C(15)–C(14)–C(17)	121.5 (5)	C(14)–C(15)–C(16)	121.0 (5)
C(11)–C(16)–C(15)	118.9 (5)	N(5)–S(2)–O(21)	103.5 (2)
N(5)–S(2)–O(22)	107.2 (2)	N(5)–S(2)–C(21)	104.5 (2)
O(21)–S(2)–O(22)	121.5 (2)	O(21)–S(2)–C(21)	108.9 (2)
O(22)–S(2)–C(21)	109.9 (2)	S(2)–C(21)–C(22)	119.1 (4)
S(2)–C(21)–C(26)	119.5 (4)	C(22)–C(21)–C(26)	121.4 (5)
C(21)–C(22)–C(23)	118.9 (5)	C(22)–C(23)–C(24)	120.9 (5)
C(23)–C(24)–C(25)	119.3 (5)	C(23)–C(24)–C(27)	120.5 (5)
C(25)–C(24)–C(27)	120.2 (5)	C(24)–C(25)–C(26)	121.0 (5)
C(21)–C(26)–C(25)	118.4 (5)	N(1)–C(1')–O(1')	108.3 (4)
N(1)–C(1')–C(2')	112.9 (4)	O(1')–C(1')–C(2')	107.9 (4)
C(1')–O(1')–C(4')	110.5 (4)	C(1')–C(2')–O(2')	107.5 (4)
C(1')–C(2')–C(3')	105.0 (4)	O(2')–C(2')–C(3')	102.2 (4)
C(2')–O(2')–C(8')	107.5 (4)	C(2')–C(3')–O(3')	105.3 (4)
C(2')–C(3')–C(4')	103.6 (4)	O(3')–C(3')–C(4')	113.4 (4)
C(3')–O(3')–C(8')	109.4 (4)	O(1')–C(4')–C(3')	107.0 (4)
O(1')–C(4')–C(5')	108.1 (4)	C(3')–C(4')–C(5')	117.5 (5)
C(4')–C(5')–O(5')	110.0 (5)	C(5')–O(5')–C(6')	120.3 (5)
O(5')–C(6')–O(6')	122.5 (8)	O(5')–C(6')–C(7')	112.5 (6)
O(6')–C(6')–C(7')	125.0 (8)	O(2')–C(8')–O(3')	105.3 (4)
O(2')–C(8')–C(9')	108.1 (5)	O(2')–C(8')–C(10')	111.9 (5)
O(3')–C(8')–C(9')	109.7 (5)	O(3')–C(8')–C(10')	108.4 (5)
C(9')–C(8')–C(10')	113.3 (6)		

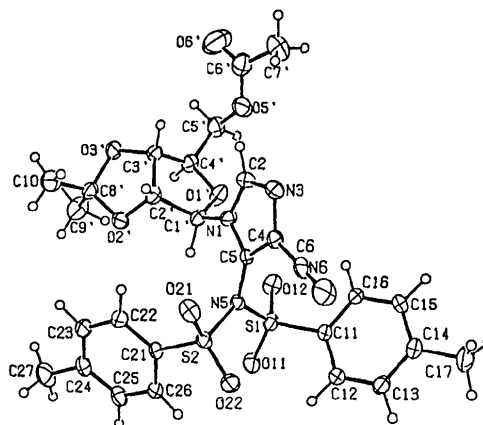


Fig. 1. Perspective drawing of the molecule (Johnson, 1976). Thermal ellipsoids correspond to 30% probability surfaces.

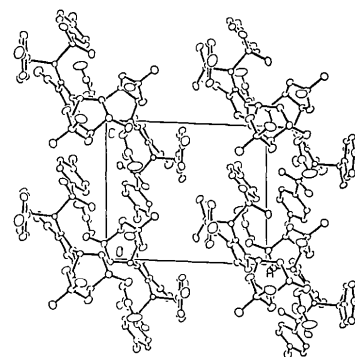


Fig. 2. Crystal structure viewed down the  $b$  axis (Johnson, 1976).

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